الجامعة الألمانية الأردنية
German Jordanian University

Analytical Chemistry

Laboratory Manual

Dr. Mohammad Khanfar Dr. Nafisah Al-Rifa

Experiments Index

Exams and marks

Lab exams will be embedded in the mid and the final exams of the course.

Each lab session weighs 3 marks, two marks for the lab performance and one mark for the evaluation and personal attitude. Students are expected to answer questions during time of the lab and they will be evaluated based on their answers.

Commitment to safety in the lab

Sign the commitment during your first lab session.

Safety Notes

General Lab Rules

• Do not enter the laboratory before your instructor or TA arrives.
• Wear safety goggles and lab coat at all time when you are in the laboratory.
• Do not wear short skirts, shorts, or open-toed shoes in the laboratory.
• Do not wear scarves or neckties in the lab, because they may accidentally be ignited in the flame of a Bunsen burner
• Girls with long hair should tie it back before entering the lab.
• Never chew gum, eat, drink, or smoke in the laboratory.

Heating Safety Tips

• Never leave a lighted Bunsen burner unattended.
• Never heat solutions to dryness, this can sometimes cause an explosion.
• Never heat a "closed system" such as a stoppered flask.
• To heat liquids, add 2-3 boiling stones to help it heat evenly and boil smoother.

Waste Disposal

• Always use the smallest amount of substance required for an experiment; more is never better in chemistry.
• Never return unused portions of chemicals to their original bottle – use a waste container.
• Dispose of all reaction products as directed by your instructor. In particular, observe the special disposal techniques necessary for flammable or toxic substances.
• Dispose of all glass products in the special container provided.

Other Rules

• Never remove any chemical substance from the laboratory. This is grounds for expulsion from our class and from the university
• Keep your work area clean, and help keep the common areas of the laboratory clean. If you spill something in a common are, remember that this substance may injure someone else.
• Never fully inhale vapors of any substance. Waft a tiny amount of the vapor toward your nose if you need to smell it
• Never add water to a concentrated reagent when diluting the reagent. Always add the reagent to water. The reverse may cause it to splash out on you.
• Never perform any experiment that is not specifically authorized by your instructor. DO NOT play games with chemicals!
• Don’t use any glassware that has any cracks, chips, star fractures, or any other deformity.

Commitment to safety in the lab

- You are not allowed to enter the lab if you don't have your lab coat, eye goggles and pre-lab.
- Safety glasses and the lab coat must be worn at all times in the lab while working at or about the benches or you will be dismissed from the lab.
- Prepare for each lab by reading and studying the experiment and all associated instructions and answer the Pre-lab questions prior to coming to the lab.
- Arrive on time to the lab and listen carefully to your instructor.
- Notify the instructor (or supervisor) immediately of any accident, regardless of how minor you may think it is.
- Sturdy, closed shoes (no sandals, etc.) must be worn in the lab.
- Long hair must be tied or otherwise secured behind the head.
- No visitors in the lab, including children.
- No food, drink, tobacco products, chewing gums in the lab.
- Broken glass must be handled with proper safety precautions; all broken glass must be discarded in a designated container.
- All spills must be cleaned up immediately. Any questions about how to do so, consult with your instructor or supervisor.
- After handling chemicals, always wash your hands thoroughly, especially after a spill on your hands. In most cases, flushing with large amounts of water lessens or prevents injury in cases of cuts, burns or spills on the skin.
- Notify your instructor (or supervisor) of medical/health conditions relevant to safety before your first experiment. These include (but are not limited to): pregnancy as it becomes known, a history of seizures or fainting, neurological disorders that may compromise the safe handling of labware, chemical or latex allergies, serious vision or hearing impairments, hemophilia, hepatitis B, anosmia (inability to smell).
- Know the locations of fire extinguishers, eyewashes, emergency showers, nearest exits.
- Never work alone in the lab.
- Never leave an experiment while substances are heating or reacting.
- Carefully read the label on all chemicals before dispensing. Dispense small amounts of chemicals to avoid excess and dispose of any excess according to your instructor's directions.
- Leave your work area and glassware clean and wash your hands before leaving the lab
- If you miss the safety advisory for a particular lab experiment or assignment, you will NOT be allowed to do the experiment.

I acknowledge that I have received, read, and understand the safety information presented to me.

In doing so, I certify that I will follow the above policies, follow all instructions from University faculty and staff, follow all laboratory safety rules, and only perform exercises and activities in a safe and responsible manner. I further understand that the activities of this class (or project) will require me to perform exercises with chemicals and other potentially hazardous materials under the supervision of University faculty and staff, and that I can be dismissed if I violate this agreement.

Name: …….

Instructor : …….

TA: ……..

TA signature: ……

Basic Statistics
Mean, Mode, Median, and Standard Deviation

The Mean and Mode

The sample mean is the average and is computed as the sum of all the observed outcomes from the sample divided by the total number of events. We use x as the symbol for the sample mean. In math terms,

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x$$

where n is the sample size and the x correspond to the observed value.

The mode of a set of data is the number with the highest frequency, one that occurs maximum number of times.

Median, and Trimmed Mean

One problem with using the mean, is that it often does not depict the typical outcome. If there is one outcome that is very far from the rest of the data, then the mean will be strongly affected by this outcome. Such an outcome is called an outlier. An alternative measure is the median. The median is the middle score. If we have an even number of events we take the average of the two middles. The median is better for describing the typical value. It is often used for income and home prices.

Example

Suppose you randomly selected 10 house prices. You are interested in the typical house price. In lakhs the prices are

2.7, 2.9, 3.1, 3.4, 3.7, 4.1, 4.3, 4.7, 4.7, 40.8

If we computed the mean, we would say that the average house price is 744,000. Although this number is true, it does not reflect the price for available housing in South Lake Tahoe. A closer look at the data shows that the house valued at 40.8 x 100,000 = 40.8 million skews the data. Instead, we use the median. Since there is an even number of outcomes, we take the average of the middle two (3.7 + 4.1)/2 = 3.9. Therefore, the median house price is 390,000. This better reflects what a house shopper should have to buy a house.

There is an alternative value that also is resistant to outliers. This is called the trimmed mean which is the mean after getting rid of the outliers or 5% on the top and 5% on the bottom. We can also use the trimmed mean if we are concerned with outliers skewing the data, however the median is used more often since more people understand it.

Example

At a ski rental shop data was collected on the number of rentals on each of ten consecutive Saturdays:

44, 50, 38, 96, 42, 47, 40, 39, 46, 50.

To find the sample mean, add them and divide by 10:

(44 + 50 + 38 + 96 + 42 + 47 + 40 + 39 + 46 + 50) / 10 = 49.2

Notice that the mean value is not a value of the sample. To find the median, first sort the data: 38, 39, 40, 42, 44, 46, 47, 50, 50, 96

Notice that there are two middle numbers 44 and 46. To find the median we take the average of the two.
Median = (44 + 46) /2 = 45

Notice also that the mean is larger than all but three of the data points. The mean is influenced by outliers while the median is robust.

Variance and Standard deviation

The mean, mode, median, and trimmed mean do a nice job in telling where the center of the data set is, but often we are interested in more. For example, a pharmaceutical engineer develops a new drug that regulates iron in the blood. Suppose she finds out that the average sugar content after taking the medication is the optimal level. This does not mean that the drug is effective. There is a possibility that half of the patients have dangerously low sugar content while the other half has dangerously high content. Instead of the drug being an effective regulator, it is a deadly poison. What the pharmacist needs is a measure of how far the data is spread apart. This is what the variance and standard deviation do. First we show the formulas for these measurements. Then we will go through the steps on how to use the formulas.

We define the variance to be

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x-\bar{x})^2$$

and the standard deviation to be

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

Variance and Standard Deviation: Step by Step

1. Calculate the mean, x.
2. Write a table that subtracts the mean from each observed value.
3. Square each of the differences.
4. Add this column.
5. Divide by n -1 where n is the number of items in the sample. This is the variance.
6. To get the standard deviation, we take the square root of the variance.

Example

The owner of a restaurant is interested in how much people spend at the restaurant. He examines 10 randomly selected receipts for parties of four and writes down the following data.

44, 50, 38, 96, 42, 47, 40, 39, 46, 50

He calculated the mean by adding and dividing by 10 to get x = 49.2 Below is the table for getting the standard deviation:

x	x - 49.2	$(x - 49.2)^2$
44	-5.2	27.04
50	0.8	0.64
38	11.2	125.44
96	46.8	2190.24
42	-7.2	51.84
47	-2.2	4.84
40	-9.2	84.64
39	-10.2	104.04
46	-3.2	10.24
50	0.8	0.64
Total		**2600.4**

ŏ

Now
(2600.4) / (10-1) = 288.7

Hence the variance is 289 and the standard deviation is the square root of 289 = 17.

Since the standard deviation can be thought of measuring how far the data values lie from the mean, we take the mean and move one standard deviation in either direction. The mean for this example was about 49.2 and the standard deviation was 17. We have:
49.2 - 17 = 32.2
and
49.2 + 17 = 66.2
What this means is that most of the patrons probably spend between 32.20 and 66.20.

Practical Analytical Chemistry Glossary

- **analyte** Substance being measured or detected.

- **blank solution** A solution not intended to contain analyte. It could be made from all reagents— except unknown—that would be used in an analytical procedure. Analyte signal measured with a blank solution could be due to impurities in the reagents or, possibly, interference.

- **blank titration** One in which a solution containing all reagents except analyte is titrated. The volume of titrant needed in the blank titration should be subtracted from the volume needed to titrate unknown.

- **buffer** A mixture of a weak acid and its conjugate base. A buffered solution is one that resists changes in pH when acids or bases are added.

- **buffer capacity, _** A measure of the ability of a buffer to resist changes in pH. The larger the buffer capacity, the greater the resistance to pH change.

- **buret** A calibrated glass tube with a stopcock at the bottom. Used to deliver known volumes of liquid.

- **chromatography** A technique in which molecules in a mobile phase are separated because of their different affinities for a stationary phase. The greater the affinity for the stationary phase, the longer a molecule is retained.

- **complexometric titration** One in which the reaction between analyte and titrant involves complex formation.

- **decant** To pour liquid off a solid or, perhaps, a denser liquid. The denser phase is left behind.

- **diprotic acids and bases** Compounds that can donate or accept two protons.
- **EDTA (ethylenediaminetetraacetic acid)** $(HO_2CCH_2)_2NCH_2CH_2N(CH_2CO_2H)_2$, the most widely used reagent for complexometric titrations. It forms 1:1 complexes with virtually all cations with a charge of 2 or more.

- **effervescence** Rapid release of gas with bubbling and hissing.

- **eluate** What comes out of a chromatography column. Also called effluent.

- **eluent** Solvent applied to the beginning of a chromatography column.

- **elution** Process of passing a liquid or a gas through a chromatography column.

- **end point** Point in a titration at which there is a sudden change in a physical property, such as indicator color, pH, conductivity, or absorbance. Used as a measure of the equivalence point.
- **equivalence point** Point in a titration at which the quantity of titrant is exactly sufficient for stoichiometric reaction with the analyte.

- **equivalent** For a redox reaction, the amount of reagent that can donate or accept one mole of electrons. For an acid-base reaction, the amount of reagent that can donate or accept one mole of protons.

- **equivalent weight** The mass of substance containing one equivalent.

- **filtrate** Liquid that passes through a filter.

- **gravimetric analysis** Any analytical method that relies on measuring the mass of a substance (such as a precipitate) to complete the analysis.

- **gravimetric titration** A titration in which the mass of titrant is measured, instead of the volume. Titrant concentration is conveniently.

- **indicator** A compound having a physical property (usually color) that changes abruptly near the equivalence point of a chemical reaction.

- **median** For a set of data, that value above and below which there are equal numbers of data.

- **meniscus** Curved surface of a liquid.

- **molality, m** A measure of concentration equal to the number of moles of solute per kilogram of solvent.
- **molarity, M** A measure of concentration equal to the number of moles of solute per liter of solution.

- **neutralization** Process in which a stoichiometric equivalent of acid is added to a base (or vice versa).

- **normality** n times the molarity of a redox reagent, where n is the number of

electrons donated or accepted by that species in a particular chemical reaction. For acids and bases, it is also n times the molarity, but n is the number of protons donated or accepted by the species.

- **parts per billion, ppb** An expression of concentration denoting nanograms (10^{-9} g) of solute per gram of solution.

- **parts per million, ppm** An expression of concentration denoting micrograms (10^{-6} g) of solute per gram of solution.

- **primary standard** A reagent that is pure enough and stable enough to be used directly after weighing. The entire mass is considered to be pure reagent.

- **SI units** International system of units based on the meter, kilogram, second, ampere, kelvin, candela, mole, radian, and steradian.

- **supernatant liquid** Liquid remaining above the solid after a precipitation. Also called supernate.
- **supersaturated solution** One that contains more dissolved solute than would be present at equilibrium.

- **titer** A measure of concentration, usually defined as how many milligrams of reagent B will react with 1 mL of reagent A. One milliliter of $AgNO_3$ solution with a titer of 1.28 mg NaCl/mL will be consumed by1.28 mg NaCl in the reaction $Ag^+ + Cl^-$ □ AgCl(s). The same solution of $AgNO_3$ has a titer of 0.993 mg of KH_2PO_4/mL, because 1 mL of $AgNO_3$ solution will be consumed by 0.993 mg KH_2PO_4 to precipitate Ag_3PO_4.

- **titrant** Substance added to the analyte in a titration.

- **titration** A procedure in which one substance (titrant) is carefully added to another (analyte) until complete reaction has occurred. The quantity of titrant required for complete reaction tells how much analyte is present.

- **titration curve** A graph showing how the concentration of a reactant or a physical property of the solution varies as one reactant (the titrant) is added to another (the analyte).

- **titration error** Difference between the observed end point and the true equivalence point in a titration.

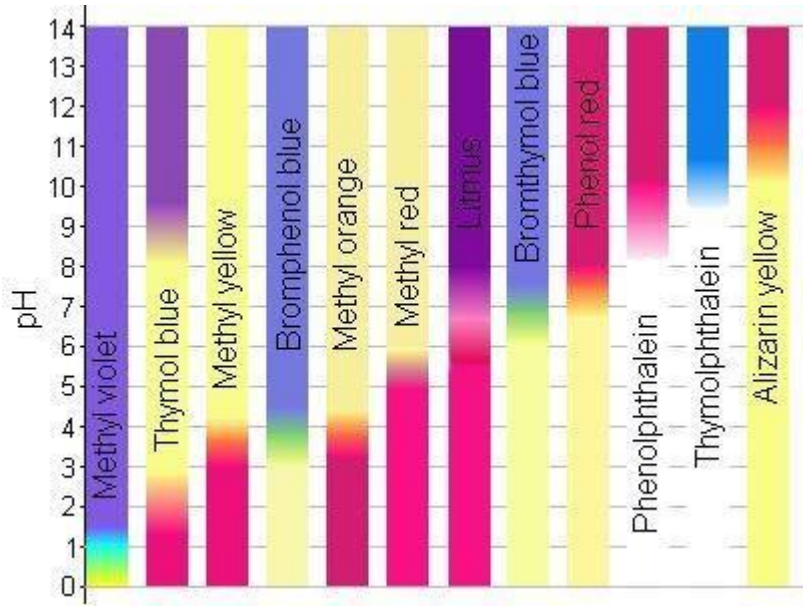
- **volume percent, vol%** Defined as (volume of solute/volume of solution) *100.

- **volumetric analysis** A technique in which the volume of material needed to react with the analyte is measured.

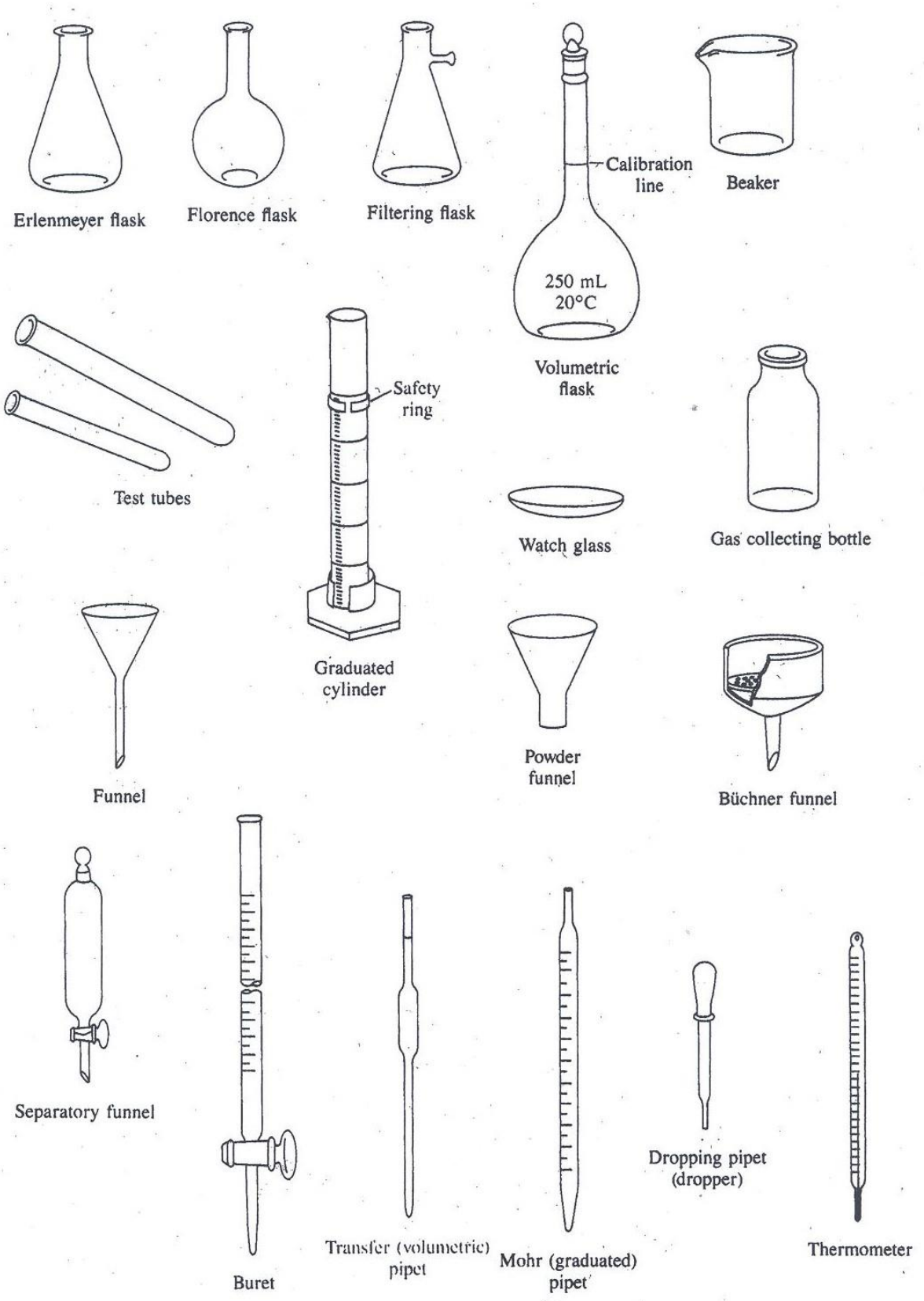
- **volumetric flask** One having a tall, thin neck with a calibration mark. When the liquid level is at the calibration mark, the flask contains its specified volume of liquid at a specified temperature.

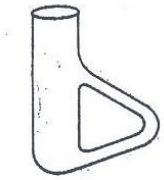
- **weight percent, wt%** (Mass of solute/mass of solution) *100.

- **weight/volume percent** [(Mass of solute, g)/(volume of solution, mL)] * 100.

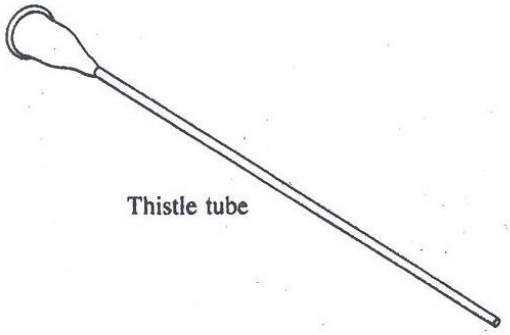


Common Laboratory Glassware and Apparatus



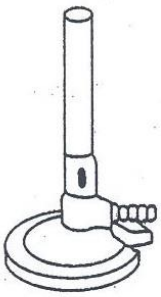


Thiele tube

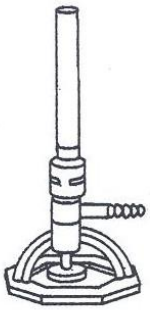


Thistle tube

Liebig condenser
(water-cooled)

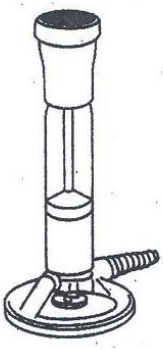


Bunsen
burner

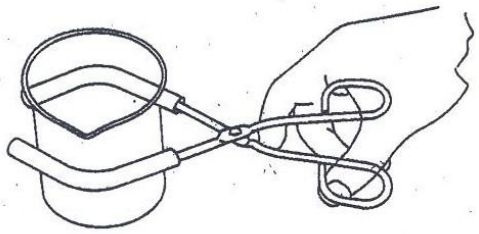


Tirrill burner

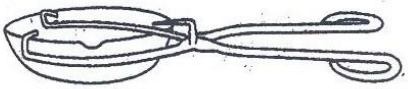
Wing top
(flame spreader)



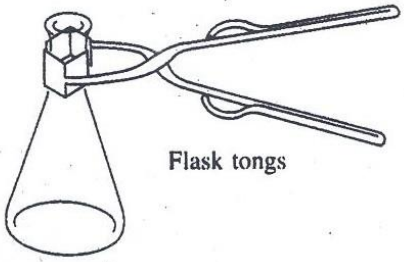
Meker burner



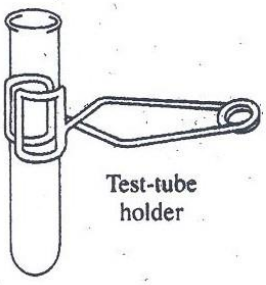
Beaker tongs



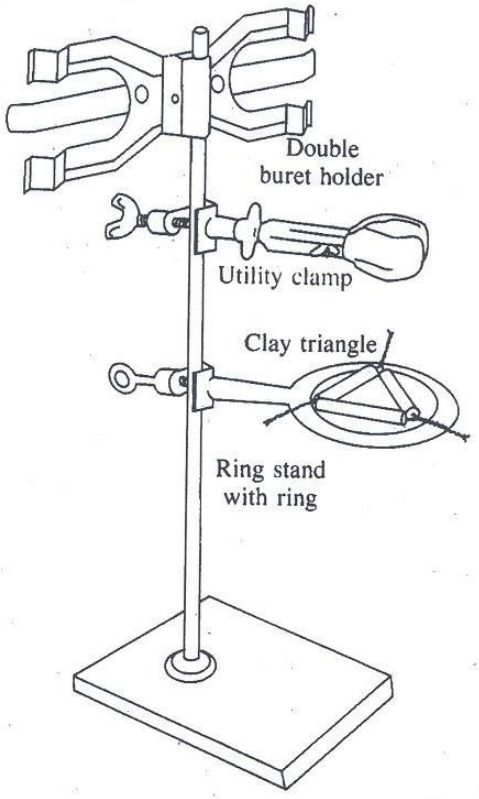
Dish tongs

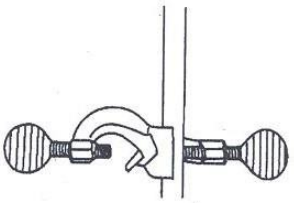


Flask tongs

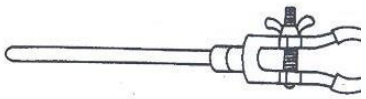


Test-tube
holder

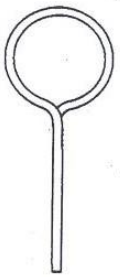




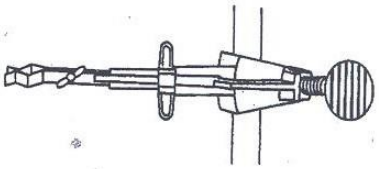
Extension clamp
holder



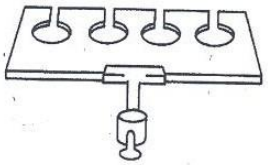
Extension clamp



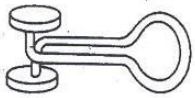
Ring
(extension type)



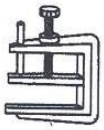
Thermometer
clamp



Funnel
rack



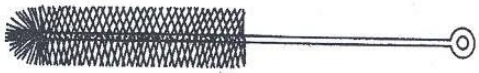
Pinchcock
clamp



Screw clamp
(Hofmann clamp)

Plastic
wash bottle

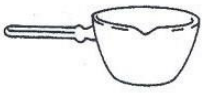
Test-tube brush



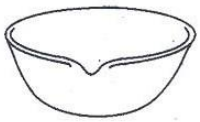
Beaker brush

Crucible and cover

Gooch crucible

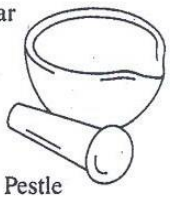


Casserole



Evaporating dish

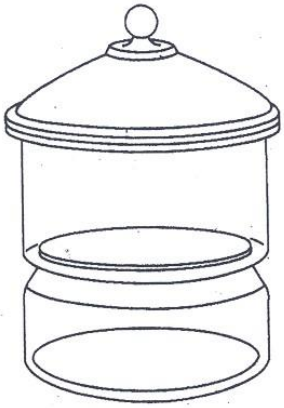
Triangular
file



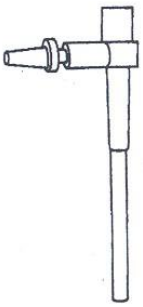
Mortar

Pestle

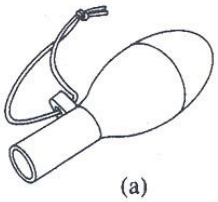
Forceps



Desiccator

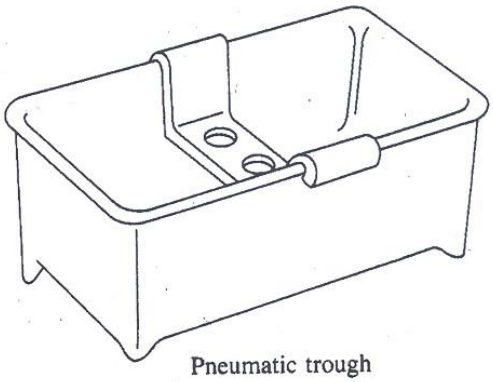


Water aspirator
(filter pump)

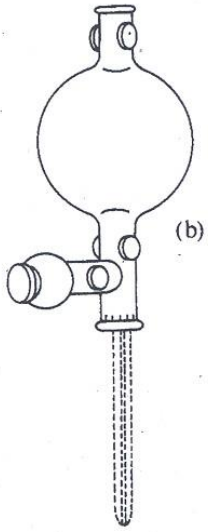


(a)

Rubber
policeman



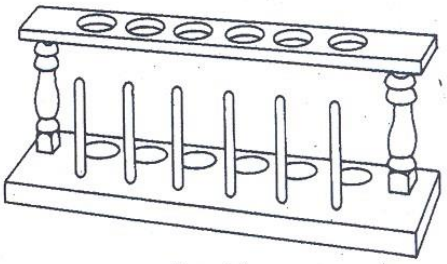
Pneumatic trough



(b)

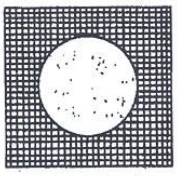
Pipet
safety bulbs

Water bath



Test tube
rack

Spatulas



Wire gauze
(ceramic center)

Lab session # 2

Gravimetric Determination of Iron as Fe_2O_3

Objective

Gravimetric analysis is a chemical analysis based on weighing a final product. The amount of iron in an unknown sample can be analyzed by precipitating the hydrated iron oxide from a basic solution. This reaction is followed by a complete dehydration to give the solid iron oxide.

$$Fe^{3+} + 3\ H_2O \rightarrow FeOOH\ x\ H_2O\ (s) + 3\ H^+$$

$$2\ FeOOH\ x\ H_2O \xrightarrow{900\ ^\circ C} Fe_2O_3\ (s) + 2$$

$$Fe^{+2} \xrightarrow[\triangle]{HNO_3} H_2O\ Fe^{3+} + e^-$$

Example – Gravimetric analysis

Chemical analysis based on weighing a final product is called **gravimetric analysis.** Iron from a dietary supplement tablet can be measured by dissolving the tablet and then converting the iron into solid Fe_2O_3. From the mass of Fe_2O_3, we can calculate the mass of iron in the original tablet.

Here are the steps in the procedure:

Step 1 Tablets containing iron(II) fumarate ($Fe^{2+}C_4H_2O_4^{2-}$) and inert binder are mixed with 150 mL of 0.100 M HCl to dissolve the Fe^{2+}. The solution is filtered to remove insoluble binder.

Step 2 Iron(II) in the clear liquid is oxidized to iron(III) with excess hydrogen peroxide:

$$\underset{\text{Iron(II) (ferrous ion)}}{2Fe^{2+}} + \underset{\text{Hydrogen peroxide FM 34.01}}{H_2O_2} + 2H^+ \rightarrow \underset{\text{Iron(III) (ferric ion)}}{2Fe^{3+}} + 2H_2O \qquad (1\text{-}5)$$

Step 3 Ammonium hydroxide is added to precipitate hydrous iron(III) oxide, which is a gel. The gel is filtered and heated in a furnace to convert it to pure solid Fe_2O_3.

$$\underset{\text{Hydroxide}}{Fe^{3+} + 3OH^- + (x-1)H_2O} \longrightarrow \underset{\text{Hydrous iron(III) oxide}}{FeOOH \cdot xH_2O(s)} \xrightarrow{900^\circ C} \underset{\text{Iron(III) oxide FM 159.69}}{Fe_2O_3(s)} \qquad (1\text{-}6)$$

The mass of Fe_2O_3 isolated at the end of the experiment was 0.277 g. What is the average mass of iron per dietary tablet?

Solution The moles of isolated Fe_2O_3 are $(0.277\ \cancel{g})/(159.69\ \cancel{g}/\text{mol}) = 1.73 \times 10^{-3}$ mol. There are 2 mol Fe per formula unit, so the moles of Fe in the product are

$$(1.73 \times 10^{-3}\ \cancel{\text{mol Fe}_2\text{O}_3})\left(\frac{2\ \text{mol Fe}}{1\ \cancel{\text{mol Fe}_2\text{O}_3}}\right) = 3.47 \times 10^{-3}\ \text{mol Fe}$$

The mass of Fe is $(3.47 \times 10^{-3}\ \cancel{\text{mol Fe}})(55.845\ \text{g Fe}/\cancel{\text{mol Fe}}) = 0.194$ g Fe. Each of the 12 tablets therefore contains an average of $(0.194\ \text{g Fe})/12 = 0.016\ 1\ \text{g} = 16.1$ mg.

Procedure

1) Weigh about 1 g of the unknown and transfer to 100 mL Flask.
2) Dissolve the sample in minimum amount of 3 M HCl, then add 5 mL 6 M HNO_3 and boil the solution.
   [The function of the nitric acid is to make sure the iron is converted into Fe^{3+} and that no iron is converted back to Fe^{2+} after the HCl is added].
3) Dilute the solution with around 10 ml distilled water and add 3 M ammonia until basic (check basicity using litmus paper (or pH paper)), then complete the reaction with boiling. **[One source of error can result from incomplete reaction, which would lead to a small percent yield].**
4) Mark a clean filter paper with your name, then weigh the paper.
5) Decant supernatant through filter paper and wash the solid with 1% NH_4NO_3.
6) Dry sample overnight in the oven.
7) Weigh the paper and its content after cooling.

Lab session # 3

Titration of a strong acid with a strong base

An acid/base titration can be monitored with an indicator or with a pH meter. In either case, the goal is to determine the equivalence point of the titration. This is the point at which enough titrant has been added to the analyte to just exactly neutralize the analyte.

When an indicator is used in a titration, the color change occurs at what is called the endpoint. If the indicator has been properly selected, this point will be the same as the equivalence point.

When a pH meter is used, the pH of the solution is recorded as the titrant is added. The pH versus the volume of titrant added can be plotted on what is called a titration curve. In this case the equivalence point occurs at the point where very small additions of titrant cause a very rapid rise in the pH. Graphically, it is also the point on the curve where the slope, $\Delta pH/\Delta V$, changes from positive to negative (called the inflection point.) Figure 1 is a titration curve for the titration of HCl by NaOH, a strong acid and strong base, where 25.0 mL of 0.1 M HCl is titrated with 0.1 M NaOH.

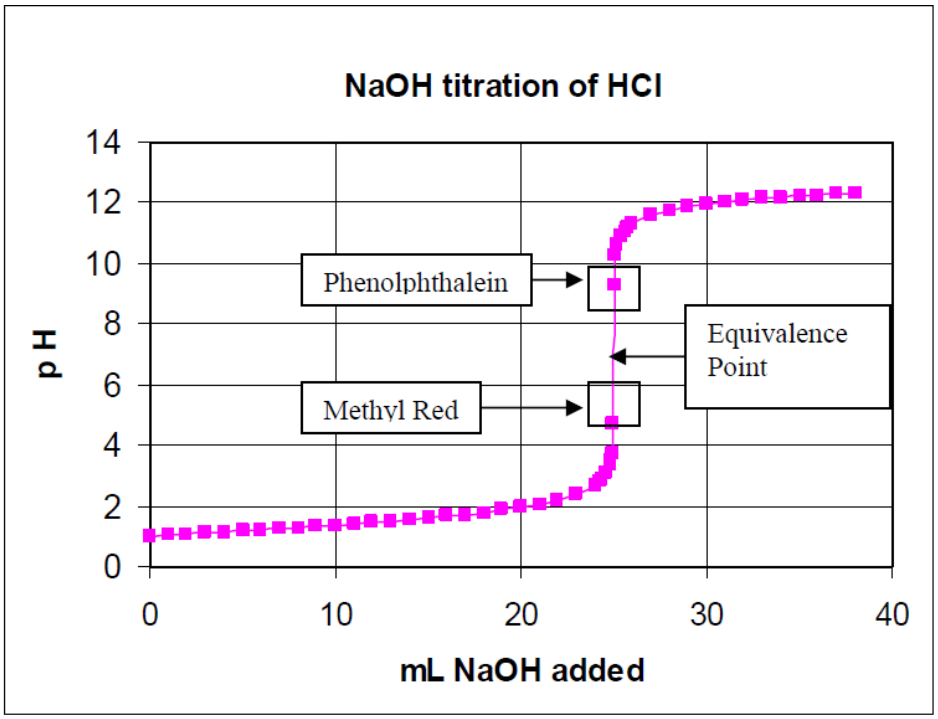


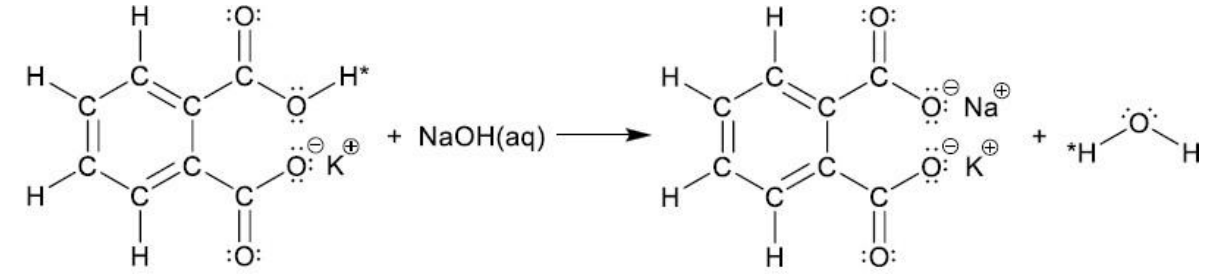
Figure 1. Titration of 25.0 mL of 0.1M HCl by 0.1 M NaOH. Blocked areas on the curve indicate the pH range in which phenolphthalein and methyl red change colors.

Note that the slope, ΔpH/ΔV, becomes large when the volume of NaOH added is at 25 mL, so this is the equivalence point. Because of this rapid rise through a range of pH values when the equivalence point is reached, a wide variety of indicators may be used to detect the endpoint visually. Either methyl red or phenolphthalein can be used for an HCl/NaOH analysis, since both will exhibit color changes in the range of pH values at the equivalence point.

Objective

In Part 1 of today's experiment, you will accurately determine the exact concentration of NaOH(aq) solution by a process known as **standardization**. To "standardize" means to accurately determine the concentration of a solution, so that solution may be used for another measurement. You can think of it as calibrating a solution. Crystalline potassium hydrogen phthalate (abbreviated KHP) will be used as the primary standard acid. By titrating a NaOH solution against a measured mass of KHP, you can accurately determine the concentration of the NaOH solution. Then, it is possible to titrate solutions of acids having unknown concentrations with the NaOH solution (whose concentration has now been determined) to find the respective unknown acid molarities.

Potassium hydrogen phthalate (KHP) is a weak, monoprotic (one acidic H, denoted by H*) organic acid that reacts with aqueous sodium hydroxide according to the reaction:



potassium hydrogen phthalate(aq)
KHP
($C_8H_5KO_4$)

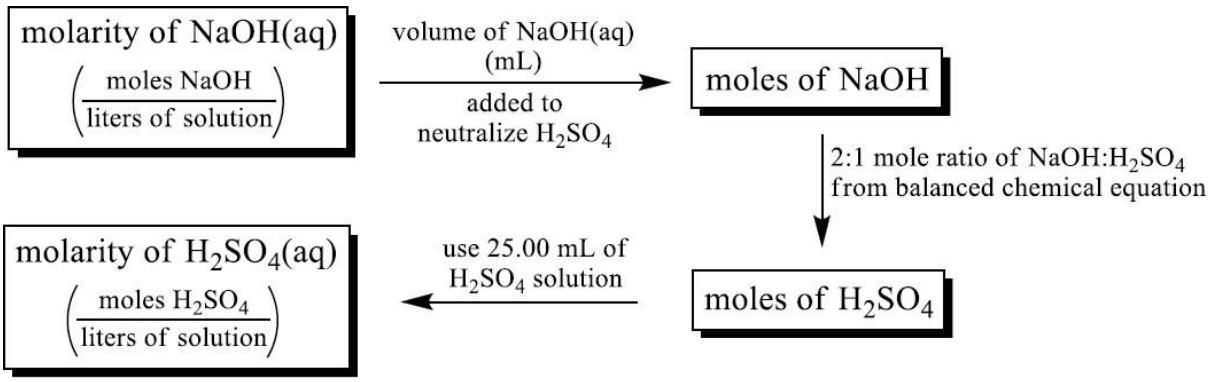
In order to detect the equivalence point (the endpoint when the reactants are exactly neutralized), an indicator dye, such as phenolphthalein, is added to the reaction mixture. The endpoints of your titrations will be signaled by the phenolphthalein color change. The indicator, in this case, is sensitive to the relative amount of hydroxide ion in solution which increases quickly once the KHP reactant is used up.

In Part 2 of today's experiment, you'll titrate your NaOH(aq) solution against a solution of hydrochloric acid (HCl) then in Part 3 against a solution of sulfuric acid (H_2SO_4) whose concentrations are unknown.

$NaOH_{(aq)} + HCl_{(aq)} \rightarrow NaCl_{(aq)} + H_2O_{(l)}$

$2NaOH_{(aq)} + H_2SO_{4(aq)} \rightarrow Na_2SO_{4(aq)} + 2H_2O_{(l)}$

Once again, phenolphthalein will be used to indicate the equivalence point of the titration; the point where enough NaOH(aq) has been added to completely consume the acid and any further addition of NaOH(aq) quickly raises the pH of the solution.



Sample Buret Readings Using a Meniscus:

In order to obtain data with good precision, you must develop good techniques with a buret, a specially designed piece of volumetric glassware. Your instructor will cover the proper use of a buret with you. You must read the buret to the proper level of precision (significant figures) as with all volumetric glassware. Below is an example of a section of a buret. Note that each of the three different examples are properly recorded to two decimal places with the last digit estimated with an "educated approximation" (commonly called the "doubtful digit").

initial volume = V_i = 20.24 mL | volume = 20.82 mL | final volume = V_f = 21.40 mL

Understand that the graduations of a buret are backward from a graduated cylinder, and burets are meant to measure the volume dispensed. Record the initial volume, record the final volume, and subtract the two values (Vf – Vi) to calculate the total volume dispensed.

Procedure:

Clean glassware and proper lab techniques are essential for success in this experiment. Your instructor will provide guidance and suggestions.

1. Standardization of NaOH solution:

1) Weigh about 0.2 g of KHP then dissolve in 50 mL of distilled water in a conical flask.
2) Add 3 drops of phenolphthalein indicator.
3) Fill the buret with NaOH and record initial volume.
4) Start titration and continue dropwise until the color of the solution turns light pink.(end point)
5) Record final volume.
6) Repeat one more time and start the calculations.
7) Calculate the average NaOH molarity and use the value for calculations in the parts below.

2. Titration of HCl with NaOH:

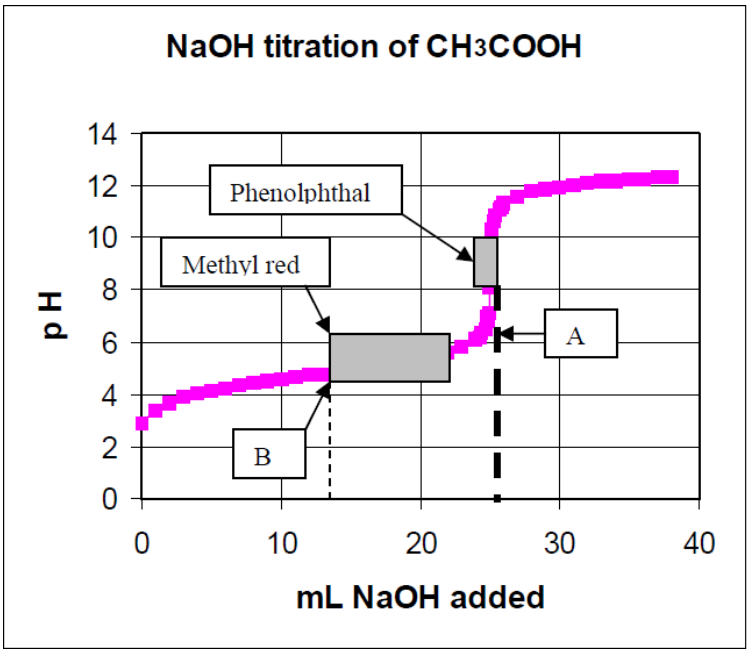
1) Fill the buret with NaOH and record initial volume.
2) Measure 10 mL of HCl solution (using a pipet or a dispenser) and pour into a flask.
3) Add 10 mL of Distilled water
4) Add 3 drops of phenolphthalein indicator.
5) Start titration and continue dropwise until the color of the solution turns light pink. (end point)
6) Record final volume.
7) Repeat one more time and start the calculations.

3. Titration of H_2SO_4 with NaOH:

1) Fill the buret with NaOH and record initial volume.
2) Measure 10 mL of H_2SO_4 solution (using a pipet or a dispenser) and pour into a flask
3) Add 10 mL of distilled water
4) Add 3 drops of phenolphthalein indicator.
5) Start titration and continue dropwise until the color of the solution turns light pink. (end point)
6) Record final volume.
7) Repeat one more time and start the calculations.

Lab session # 4
Titration of a weak acid with a strong base

When a **weak acid** is titrated by a strong base, the fact that, in aqueous solution, the weak acid dissociates into a hydrogen ion and the **conjugate base** of the acid changes the appearance of the titration curve shown for the titration of a strong acid with a strong base in the previous experiment. The curve will look similar to what is shown in the Figure below, which represents the titration of
0.1 M acetic acid with 0.1 M NaOH.



A: Equivalence point

B: Half-equivalence point

pH at B = 4.74 = pK_a

Figure: Titration of 25 mL of 0.1M acetic acid by 0.1M NaOH.

Several differences are readily apparent in the comparison of both titration curves (the one shown here and the one shown in last experiment). There are variations in the initial pH, the rate of pH change, and the pH at the equivalence point. The addition of a strong base to a weak acid creates a build up of the salt of the weak acid (in this case, $NaCH_3COO$) producing a buffering effect, which causes resistance to change in pH. Also, the pH of the equivalence point corresponds to the pH of the conjugate base, CH_3COO^-, which hydrolyzes in water. Notice that the endpoint of the methyl red does not occur at the equivalence point, and therefore it could not be used in the titration. The phenolphthalein is appropriate for this specific titration. All these effects are related to the strength (or degree of dissociation) of the acid being titrated. Because of the incomplete dissociation of the acid, the reaction is in equilibrium, with an acid dissociation constant, Ka, which is specific to that acid.

For the dissociation of any weak acid, HA:

$$HA(aq) \rightarrow H^+(aq) + A^-(aq)$$

there is an acid dissociation constant, K_a:

$$K_a = \frac{[H^+][A^-]}{[HA]}$$

This can be rearranged to solve for $[H^+]$:

$$[H^+] = \frac{K_a[HA]}{[A^-]}$$

Using the definition of pH, this equation can be rearranged as follows:

$$pH = -\log[H_3O^+] = -\log K_a - \log\left(\frac{[HA]}{[A^-]}\right), \quad \text{or}$$

$$pH = pK_a + \log\left(\frac{[A^-]}{[HA]}\right)$$

This last expression is known as the *Henderson-Hasselbach equation*. It can be used to calculate the pK_a (and thus K_a) of an acid. At the equivalence point, the volume of base added is just enough to exactly neutralize all of the acid. At one-half of this volume of added base, called the *half-equivalence point*, enough has been added to neutralize half of the acid. Since half of the acid reacted to form A^-, the concentrations of A^- and HA at the half-equivalence point are the same. Therefore, at the half-equivalence point, the pH is equal to the pK_a.

Since $\log\left(\frac{[A^-]}{[HA]}\right) = \log(1) = 0$, it follows that $pH = pK_a$.

A plot of the titration curve allows the equivalence point to be determined. At exactly one-half the volume of the equivalence point, the measured pH is equal to pKa as illustrated in the Figure above.

For polyprotic acids there are multiple dissociation steps and equivalence points, one for each acidic hydrogen present. The dissociation reactions of a weak polyprotic acid, H3A, are shown below along with the neutralization reactions that occur in a titration by a strong base.

$H_3A(aq) \rightarrow H^+(aq) + H_2A^-(aq)$ $\qquad$ $OH^- + H_3A \rightarrow H_2O + H_2A^-$

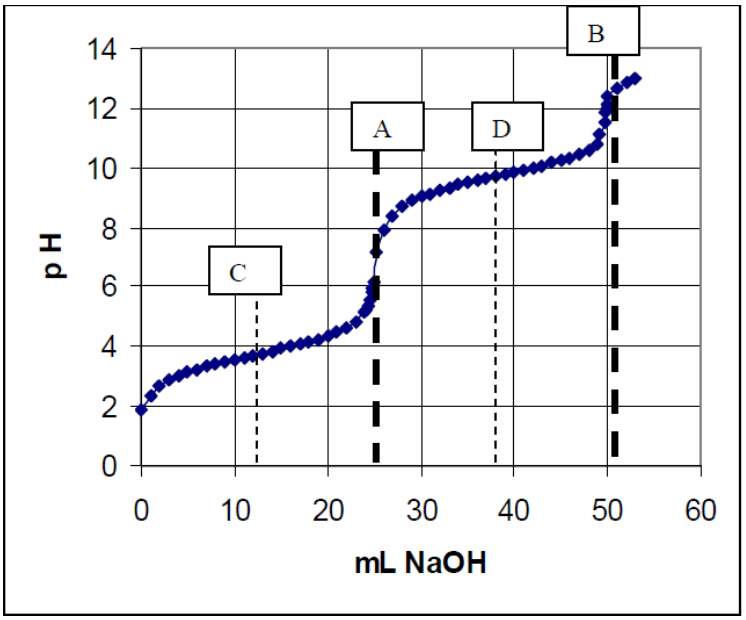
$H_2A^-(aq) \rightarrow H^+(aq) + HA^{2-}(aq)$ $\qquad$ $OH^- + H_2A^- \rightarrow H_2O + HA^{2-}$

$HA^{2-}(aq) \rightarrow H^+(aq) + A^{3-}(aq)$ $\qquad$ $OH^- + HA^{2-} \rightarrow H_2O + A^{3-}$

Each step has a separate dissociation constant: K_{a1}, K_{a2} and K_{a3}. A titration curve for a triprotic acid is thus expected to have three equivalence points, but often only

shows two. A titration curve for a diprotic acid, H_2A, would show two equivalence points, one in which OH neutralizes H_2A and a second in which OH neutralizes HA. The pK_a is obtained in the same way as for a monoprotic acid, but in this case at least two half-equivalence points are present.

One half-equivalence point occurs at one-half the volume of the first equivalence point, at which pH = pK_{a1}. The second occurs at the volume that is at the midpoint between the first and second equivalence points, and at that point, pH = pK_{a2}.



A: First equivalence point

B: Second equivalence point

C: First half equivalence point

D: Second half equivalence point

pH at C = 3.73 = pK_{a1}

pH at D = 9.68 = pK_{a2}

Figure: Titration curve of weak diprotic acid by NaOH(aq).

Objective

In this experiment you will first titrate of H_3PO_4 solution with NaOH solution using different indicators to choose the best for this titration. Then you will perform titration of CH_3COOH (AcOH) solution with NaOH solution using different indicators to choose the best for this titration, too.

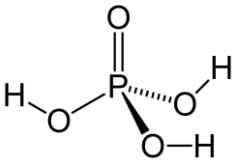
In the last part you will see how titration can be used to determine the ascorbic acid content of a Vitamin C tablets. Vitamin C (also known as ascorbic acid, $HC_6H_7O_6$) is a necessary ingredient in the human diet. The mass percentage of ascorbic acid in Vitamin C will be determined by titrating the Vitamin C samples with the standardized sodium hydroxide solution. A Vitamin C tablet contains ascorbic acid, $HC_6H_7O_6(aq)$, as well as binder material that holds the tablet together. The balanced equation for the reaction between ascorbic acid and sodium hydroxide is shown below:

$$HC_6H_7O_6\,(aq) + NaOH\,(aq) \rightarrow H_2O\,(l) + NaC_6H_7O_6\,(aq)$$

Procedure

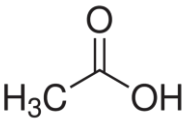
You will be provided with standarized NaOH solution to use through this experiment.

1. Titration of H_3PO_4 solution with NaOH solution:



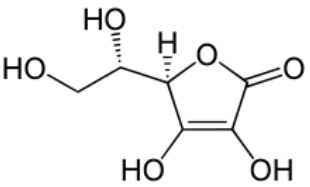
1. Fill the burette with standardized NaOH and record initial volume.
2. Measure 15 ml of H_3PO_4 and pour into a flask.
3. Add 3 drops of bromocresol indicator.
4. Start titration and continue drop wise until the color of the solution changes. (End point) and measure pH at end point using pH paper.
5. Record final volume.
6. Repeat one more time and start the calculations.
7. Repeat the same steps using methyl red then again using phenolphthalein indicator.

2. Titration of CH_3COOH (AcOH) solution with NaOH solution:



1. Fill the burette with standardized NaOH and record initial volume.
2. Measure 15 ml of AcOH and pour into a flask.
3. Add 3 drops of bromocresol indicator.
4. Start titration and continue drop wise until the color of the solution changes. (End point) and measure pH at end point using litmus paper.
5. Record final volume.
6. Repeat one more time and start the calculations.
7. Repeat the same steps using methyl red then again using phenolphthalein indicator.

3. Determination of Ascorbic acid in Vitamin C tablets:



1. Weigh 1 vitamin C tablet using analytical balance.
2. Dissolve in around 50 mL distilled water.
3. Add 3 drops of phenolphthalein indicator.
4. Titrate with standard NaOH, and continue dropwise until the color of the solution changes. (End point)
5. Record final volume.
6. Repeat one more time and start the calculations.

Lab session # 5
Analysis of a mixture of carbonate and bicarbonate

This experiment involves two titrations. First, total alkalinity (=[HCO_3^-)] + 2[CO_3^{2-}]) is measured by titrating the mixture with standard HCl to a bromocresol green end point:

$$HCO_3^- + H^+ \rightarrow H_2CO_3$$
$$CO_3^{2-} + 2H^+ \rightarrow H_2CO_3$$

A separate aliquot of unknown is treated with excess standard NaOH to convert HCO_3^- to CO_3^{2-}:

$$HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$$

Then all the carbonate is precipitated with $BaCl_2$:

$$Ba^{2+} + CO_3^{2-} \rightarrow BaCO_{3(s)}$$

The excess NaOH is immediately titrated with standard HCl to determine how much HCO_3^- was present. From the total alkalinity and bicarbonate concentration, you can calculate the original carbonate concentration.

Procedure

CO_2-free water: Boil 500 mL of distilled water to expel CO_2 and pour the water into a 500-mL plastic bottle. Screw the cap on tightly and allow the water to cool to room temperature.

Keep tightly capped when not in use. (<u>This step should be done by the TA before the lab session</u>).

1. Standardization of NaOH and HCl solutions:

1. Weigh 0.2 g of KHP then dissolve in 75 mL of distilled water in a conical flask.
2. Add 3 drops of phenolphthalein indicator.
3. Fill the burette with NaOH and record initial volume.
4. Start titration and continue dropwise until the color of the solution turns light pink.(end point)
5. Record final volume
6. Repeat one more time and start the calculations.
7. Once the NaOH solution is standardized, titrate it against 10 ml HCl (3 drops phenolphthalein indicator) 2 times.
8. Then calculate the exact concentration of HCl.

2. Total Alkalinity:

1. Weigh 2-2.5 g of the unknown mixture using analytical balance and record the unknown number on your data sheet.
2. Transfer into 250 mL volumetric flask and dissolve with CO_2 free water then dilute up to the mark.
3. Pipet 25 mL of your unknown solution into a flask, add 3 drops of bromocresol green indicator and titrate with the standardized HCl.
4. Repeat titration once more.

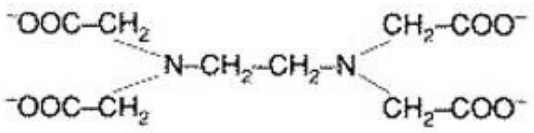
3. Bicarbonate content:

1. Pipet 25 ml of your unknown solution into a flask.
2. Add 25 ml (pipet) of standard NaOH
3. Swirl and add 10 ml $BaCl_2$ (graduated cylinder).
4. Swirl again to precipitate $BaCO_3$.
5. Add 3 drops of phenolphthalein indicator.
6. Immediately titrate with standard HCl.
7. Repeat titration once more.

Lab session # 6
Evaluation of calcium in commercial milk powder

Calcium is one of the more important minerals needed for proper nutrition. Milk and milk products are the most common sources of calcium in the diet. Different brands of dried milk powders can be compared on the basis of their calcium content.

The analysis for calcium content may be carried out by titration of the suspended milk powder sample with a solution of ethylenediaminetetraacetic acid, abbreviated as EDTA, the concentration of which is accurately known. The reaction takes place at pH 10. We use $[HY]^{3-}$ as a convenient abbreviation for the EDTA species present in a solution at pH 10. Therefore, the EDTA ion which reacts with Ca^{2+} in this titration is represented as Y^{4-} .



Structure of Y^{4-} ion

The equation for the titration reaction is:

$Ca^{2+}(aq) + Y^{4-}(aq) \rightarrow [CaY]^{2-}(aq)$ …….. **Eq. 1**

We call the $[CaY]^{2-}$ ion formed in the titration reaction a <u>complex ion</u>, or a complex. We often refer to this type of titration as a <u>complexometric titration</u>.

As shown in Eq . 1, 1 mol Ca^{2+} reacts with 1 mol of EDTA, so:

No. of moles of Ca^{2+} in sample = conc. of EDTA soln (mol/L) * volume of EDTA soln (L) needed for titration … **Eq. 2**

To calculate the mass of Ca^{2+} in the titrated sample:

Mass of Ca^{2+} in sample (g) = no. moles of Ca^{2+} in sample * molar mass of Ca^{2+}…Eq**. 3**

Then we calculate the mass % of Ca^{2+} in the sample using **Eq. 4**:

Mass% of Ca^{2+} in sample = (mass of Ca^{2+} in sample/ mass of sample) * 100 …Eq**. 4**

Today you will calculate the mass% of Ca^{2+} in two different brands of powdered milk to compare between them.

Procedure

Part A: Determination of Ca^{2+} in the 1st milk brand

1. Rinse the Buret with 10 mL EDTA.
2. Discard the rinse water and fill your Buret with EDTA.
3. Weigh 3 samples (1.35-1.56 g each) of the provided milk powder.
4. Label 3 Erlenmeyer flasks (1, 2 and 3) and put 100 mL distilled water in each.
5. Add the milk powder and stir the milk solution using a glass rod until completely dissolved, wash the remaining milk powder on the glass rod, to prevent any sample loss.
6. Add 10 mL of the NH_3-NH_4CL_2 buffer solution to each flask (under fumehood).
7. Add 15 drops of the Eriochrom Black T indicator.
8. Swirl the flasks.
9. Begin the titration and notice the color change from Magneta to Redish-purple (near end point) and to pure sky blue with no traces of red at **end point**.

Part B: Determination of Ca^{2+} in the 2nd milk brand

Repeat the steps above using the other milk brand you have.

When you are done with Experimental work calculate the mass% of Ca^{2+} in both milk brands according to data sheet.

Lab session # 7
Reduction oxidation titration; Potassium dichromate

A **redox titration** is based on an oxidation-reduction reaction between analyte and titrant. In this experiment you will use a standard solution of potassium dichromate ($K_2Cr_2O_7$) to determine the percent by weight of iron (as Fe^{2+}) in an unknown solid.

Potassium dichromate, $K_2Cr_2O_7$, is a primary standard. Its solutions are stable, and it is cheap. Dichromate ($Cr_2O_7^{2-}$) is orange and Cr^{3+} complexes range from green to violet, so indicators with distinctive color changes, such as diphenylamine sulfonic acid or diphenylbenzidine sulfonic acid, are used to find a dichromate end point. Alternatively, reactions can be monitored with Pt and calomel electrodes.

$K_2Cr_2O_7$ is not as strong an oxidant as $KMnO_4$ or Ce^{4+}. It is employed chiefly for the determination of Fe^{2+} and, indirectly, for species that will oxidize Fe^{2+} to Fe^{3+}.

In acidic solution, orange dichromate ion is a powerful oxidant that is reduced to chromic ion:

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

Only one electron is necessary to reduce Fe(III) to Fe(II)

$$Fe^{3+} + e^- \rightarrow Fe^{2+}$$

Therefore, 1 mole of $Cr_2O_7^{2-}$ (the oxidizing agent) reacts with 6 moles of Fe^{2+} (the reducing agent) to form 6 moles of Fe^{3+} and 2 moles of Cr^{3+}. Thus, in net ionic form:

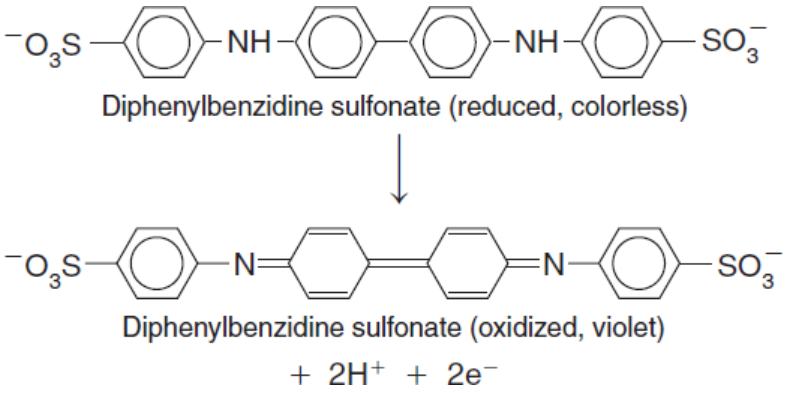
$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

The molecular form of the reaction equation can be written as:

$$K_2Cr_2O_7 + 6Fe(NH_4)_2(SO_4)2 + 7H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + Cr_2(SO_4)_3 + K_2SO_4 + 6(NH_4)_2SO_4 + 7H_2O$$

The 1:6 mole ratio with respect to the amounts of $Cr_2O_7^{2-}$ and Fe^{2+} consumed will provide the stoichiometric basis for all of the calculations in this experiment.

A **redox indicator** is a compound that changes color when it goes from its oxidized to its reduced state. Below is an example:



Today you will use sodium diphenylamine sulfonate as an indicator whose reduced form is colorless and oxidized form is purple. At the end of the titration when all of the iron is in Fe^{3+} form, a slight addition of dichromate from the buret will oxidize the indictor instead of Fe^{2+}, hence the color of the solution changes.

$$Cr_2O_7 + In(reduced) \rightarrow Cr^{3+} + In(oxidized)$$

Procedure

NOTE: Cr(VI) waste is carcinogenic and should not be poured down the drain. Cr(VI) from dichromate should be reduced to the less toxic Cr(III) with sodium hydrogen sulfite ($NaHSO_3$) and precipitated with hydroxide as insoluble $Cr(OH)_3$. The solution is evaporated to dryness and the solid is discarded in an approved landfill that is lined to prevent escape of the chemicals.

A. Preparation of a Solution of $K_2Cr_2O_7$

Weigh out 1.0-1.2 grams of $K_2Cr_2O_7$, transfer into a 250 mL volumetric flask, dissolve this sample in distilled water, and carefully dilute to the mark with additional distilled water. Mix the solution thoroughly by stoppering the flask and inverting several times.
Note: As an alternative, a larger quantity of this solution may be prepared in the stockroom and delivered to the students.

B. Titration of unknown Fe(II) solution

1. Weigh about 0.5 g of your unknown and record the exact mass you weighed and your unknown number on your data sheet.

2. In an Erlenmeyer flask; using a graduated cylinder, add 25 mL of 1 M H_2SO_4 to each flask.

3. Then add 10 mL of the l M phosphoric acid solution and 8 drops of sodium diphenylamine sulfonate indicator to the flask.

4. Swirl each flask gently to mix the contents.

5. Fill your burette with the $K_2Cr_2O_7$ solution and drain out enough so that the liquid level is just below the upper calibration mark and the buree tip is full.

6. Read the initial volume from the calibration scale on the buret.

7. Titrate the iron solution in the flask. The intense purple color produced by the first drop of excess $K_2Cr_2O_7$ signals the end point for the titration. Obtain the final volume reading from the calibration scale on the buret.

8. Repeat the titration two more times. The volume of $K_2Cr_2O_7$ solution used should agree with the first titration within 0.20 mL.

Your unknown could be either $Fe(SO_4)_2 .7H_2O$ or $FeC_2O_4.2H_2O$

Lab session # 8
Reduction oxidation titration; Potassium permanganate

Potassium permanganate ($KMnO_4$) is a strong oxidant with an intense violet color. In strongly acidic solutions (pH 1), it is reduced to colorless Mn^{2+}.

$$\underset{\text{Permanganate}}{MnO_4^-} + 8H^+ + 5e^- \rightleftharpoons \underset{\text{Manganous}}{Mn^{2+}} + 4H_2O \qquad E^\circ = 1.507\ V$$

In neutral or alkaline solution, the product is the brown solid, MnO_2.

$$MnO_4^- + 4H^+ + 3e^- \rightleftharpoons \underset{\text{Manganese dioxide}}{MnO_2(s)} + 2H_2O \qquad E^\circ = 1.692\ V$$

In strongly alkaline solution (2 M NaOH), green manganate ion is produced.

$$MnO_4^- + e^- \rightleftharpoons \underset{\text{Manganate}}{MnO_4^{2-}} \qquad E^\circ = 0.56\ V$$

Representative permanganate titrations are listed in the Table below.

Species analyzed	Oxidation reaction	Notes
Fe^{2+}	$Fe^{2+} \rightleftharpoons Fe^{3+} + e^-$	Fe^{3+} is reduced to Fe^{2+} with Sn^{2+} or a Jones reductor. Titration is carried out in 1 M H_2SO_4 or 1 M HCl containing Mn^{2+}, H_3PO_4, and H_2SO_4. Mn^{2+} inhibits oxidation of Cl^- by MnO_4. H_3PO_4 complexes Fe^{3+} to prevent formation of yellow Fe^{3+}-chloride complexes.
$H_2C_2O_4$	$H_2C_2O_4 \rightleftharpoons 2CO_2 + 2H^+ + 2e^-$	Add 95% of titrant at 25°C, then complete titration at 55°–60°C.
Br^-	$Br^- \rightleftharpoons \frac{1}{2}Br_2(g) + e^-$	Titrate in boiling 2 M H_2SO_4 to remove $Br_2(g)$.
H_2O_2	$H_2O_2 \rightleftharpoons O_2(g) + 2H^+ + 2e^-$	Titrate in 1 M H_2SO_4.
HNO_2	$HNO_2 + H_2O \rightleftharpoons NO_3^- + 3H^+ + 2e^-$	Add excess standard $KMnO_4$ and back-titrate after 15 min at 40°C with Fe^{2+}.
As^{3+}	$H_3AsO_3 + H_2O \rightleftharpoons H_3AsO_4 + 2H^+ + 2e^-$	Titrate in 1 M HCl with KI or ICl catalyst.
Sb^{3+}	$H_3SbO_3 + H_2O \rightleftharpoons H_3SbO_4 + 2H^+ + 2e^-$	Titrate in 2 M HCl.
Mo^{3+}	$Mo^{3+} + 2H_2O \rightleftharpoons MoO_2^{2+} + 4H^+ + 3e^-$	Reduce Mo in a Jones reductor, and run the Mo^{3+} into excess Fe^{3+} in 1 M H_2SO_4. Titrate the Fe^{2+} formed.
W^{3+}	$W^{3+} + 2H_2O \rightleftharpoons WO_2^{2+} + 4H^+ + 3e^-$	Reduce W with Pb(Hg) at 50°C and titrate in 1 M HCl.
U^{4+}	$U^{4+} + 2H_2O \rightleftharpoons UO_2^{2+} + 4H^+ + 2e^-$	Reduce U to U^{3+} with a Jones reductor. Expose to air to produce U^{4+}, which is titrated in 1 M H_2SO_4.
Ti^{3+}	$Ti^{3+} + H_2O \rightleftharpoons TiO^{2+} + 2H^+ + e^-$	Reduce Ti to Ti^{3+} with a Jones reductor, and run the Ti^{3+}into excess Fe^{3+} in 1 M H_2SO_4. Titrate the Fe^{2+} that is formed.
Mg^{2+}, Ca^{2+}, Sr^{2+}, Ba^{2+}, Zn^{2+}, Co^{2+}, La^{3+}, Th^{4+}, Pb^{2+}, Ce^{3+}, BiO^+, Ag^+	$H_2C_2O_4 \rightleftharpoons 2CO_2 + 2H^+ + 2e^-$	Precipitate the metal oxalate. Dissolve in acid and titrate the $H_2C_2O_4$.
$S_2O_8^{2-}$	$S_2O_8^{2-} + 2Fe^{2+} + 2H^+ \rightleftharpoons 2Fe^{3+} + 2HSO_4^-$	Peroxydisulfate is added to excess standard Fe^{2+} containing H_3PO_4. Unreacted Fe^{2+} is titrated with MnO_4^-.
PO_4^{3-}	$Mo^{3+} + 2H_2O \rightleftharpoons MoO_2^{2+} + 4H^+ + 3e^-$	$(NH_4)_3PO_4 \cdot 12MoO_3$ is precipitated and dissolved in H_2SO_4. The Mo(VI) is reduced (as above) and titrated.

For titrations in strongly acidic solution, $KMnO_4$ serves as its own indicator because the product, Mn^{2+}, is colorless. The end point is taken as the first persistent appearance of pale pink MnO_4^- . If the titrant is too dilute to be seen, an indicator such as ferroin can be used.

Potassium permanganate is not a primary standard because traces of MnO_2 are invariably present. In addition, distilled water usually contains enough organic impurities to reduce some freshly dissolved MnO_4^- to MnO_2. Potassium permanganate can be standardized by titration of sodium oxalate, $Na_2C_2O_4$.

$5C_2O_4^{2-} + 2MnO_4^- + 16H^+ \rightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$

The permanganate ion is intensely colored, while the Mn^{2+} ion is very pale in color. As the permanganate ion is added to the $C_2O_4^{2-}$ solution, it will be converted to Mn^{2+}. Once all the $C_2O_4^{2-}$ ion has reacted the continued addition MnO_4^- ion will color the solution. This reaction is a bit slower at room temperature than most acid-base reactions, making the endpoint difficult to find. As a result, the MnO_4^- solution will be heated to 60-70˚C. One drop of excess MnO_4^- ion should be sufficient to cause a color change.

In this experiment, you will analyze a solution of unknown oxalate concentration.

Procedure

Titration of unknown oxalate solution

1. Fill a buret with the permanganate solution
2. In an Erlenmeyer flask take 10 ml of your unknown and 25 ml of 3M H_2SO_4
3. Place the flask on a hotplate and heat to a temperature of 60-70 ˚C.
4. Titrate the oxalate solution with the permanganate, swirling constantly. Be careful not to add the permanganate solution too quickly. Then calculate the concentration of the unknown oxalate solution.

Lab session # 9
The determination of calcium as calcium oxalate

In common with a number of other cations, calcium is conveniently determined by precipitation with oxalate ion. The solid calcium oxalate is filtered, washed free of excess precipitating reagent, and dissolved in dilute acid. The oxalic acid liberated in this step is then titrated with standard permanganate or some other oxidizing reagent. This method is applicable to samples that contain magnesium and the alkali metals. Most other cations must be absent since they either precipitate or coprecipitate as oxalates and cause positive errors in the analysis.

Factors Affecting the Composition of Calcium Oxalate Precipitates It is essential that the mole ratio between calcium and oxalate be exactly unity in the precipitate and thus in solution at the time of titration. A number of precautions are needed to ensure this condition. For example, the calcium oxalate formed in a neutral or an ammoniacal solution is likely to be contaminated with calcium hydroxide or a basic calcium oxalate, either of which will cause low results. The formation of these compounds is prevented by adding the oxalate to an acidic solution of the sample and slowly forming the desired precipitate by the dropwise addition of ammonia. The coarsely crystalline calcium oxalate that is produced under these conditions is readily filtered. Losses resulting from the solubility of calcium oxalate are negligible above pH 4, provided that washing is limited to freeing the precipitate of excess oxalate. Coprecipitation of sodium oxalate becomes a source of positive error in the determination of calcium whenever the concentration of sodium in the sample exceeds that of calcium. The error from this source can be eliminated by reprecipitation (Reprecipitation is a method of minimizing coprecipitation errors by dissolving the initial precipitate and then reforming the solid). Magnesium, if present in high concentration, may also be a source of contamination. An excess of oxalate ion helps prevent this interference through the formation of soluble oxalate complexes of magnesium. Prompt filtration of the calcium oxalate can also help prevent interference because of the pronounced tendency of magnesium oxalate to form supersaturated solutions from which precipitate formation occurs only after an hour or more. These measures do not suffice for samples that contain more magnesium than calcium. Here, reprecipitation of the calcium oxalate becomes necessary.

Today you will determine the percentage of calcium in an unknown sample by calcium oxalate precipitation.

Procedure

Sample Preparation

1. Weigh 0.15 g samples of your calcium containing sample into three 250-mL flasks.
2. Add 10 mL of distilled water.
3. Add 10 mL of concentrated HCl drop wise, taking care to avoid losses due to spattering as the acid is introduced.

Precipitation of Calcium Oxalate

1. Dilute the sample solution by adding about 10 mL of distilled water, heat to boiling.
2. Heat at the same time about 320 mL of 6% (w/v) $(NH_4)_2C_2O_4$ solution.
3. Add 100 mL of hot 6% (w/v) $(NH_4)_2C_2O_4$ solution to each of your sample flasks.
4. Precipitate CaC_2O_4 by slowly adding 6 M NH_3. And constantly measuring the pH using litmus paper. (pH 4.5 to 5.5).
5. Allow the solutions to stand for 20 min.
6. Filter the precipitate then wash the precipitates with several 10-mL portions of cold water.

Titration of calcium oxalate

$5\ C_2O_4^{2-} + 2\ MnO_4^- + 16H^+ \rightarrow 10CO_2 + 2\ Mn^{2+} + 8H_2O$

1. Fill a burette with permanganate solution (0.05 M).
2. In an Erlenmeyer flask dissolve your precipitate in 50 ml of 3M H_2SO_4.
3. Place the flask on a hotplate and heat to a temperature of 60-70 degrees.
4. Titrate the oxalate solution with the permanganate, swirling constantly.
5. Be careful not to add the permanganate solution too quickly. The endpoint has been reached when one drop of permanganate turns the entire solution a faint pink color that does not disappear when the flask is swirled.
6. Calculate the mass percent of calcium in your sample.

Lab Session #10
Determination of Chloride Ion Concentration (The Mohr Method)

Precipitation titrimetry, which is based on reactions that yield ionic compounds of limited solubility, is one of the oldest analytical techniques. The slow rate of formation of most precipitates, however, limits the number of precipitating agents that can be used in titrations to be a handful. The most common precipitation titrations are argentometric titrations which are precipitation reactions of silver salts using $AgNO_3$ as the titrant. Argentometric titrations can be used to analyze samples for the presence of a number of anions that form precipitates with Ag^+.

Typical Argentometric Precipitation Methods

Substance Being Determined	End Point	Remarks
AsO_4^{3-}, Br^-, I^-, CNO^-, SCN^-	Volhard	Removal of silver salt not required
CO_3^{2-}, CrO_4^{2-}, CN^-, Cl^-, $C_2O_4^{2-}$, PO_4^{3-}, S^{2-}, NCN^{2-}	Volhard	Removal of silver salt required before back-titration of excess Ag^+
BH_4^-	Modified Volhard	Titration of excess Ag^+ following $BH_4^- + 8Ag^+ + 8OH^- \rightarrow 8Ag(s) + H_2BO_3^- + 5H_2O$
Epoxide	Volhard	Titration of excess Cl^- following hydrohalogenation
K^+	Modified Volhard	Precipitation of K^+ with known excess of $B(C_6H_5)_4^-$, addition of excess Ag^+ giving $AgB(C_6H_5)_4(s)$, and back-titration of the excess
Br^-, Cl^-	$2Ag^+ + CrO_4^{2-} \rightarrow Ag_2CrO_4(s)$ red	In neutral solution
Br^-, Cl^-, I^-, SeO_3^{2-}	Adsorption indicator	
$V(OH)_4^+$, fatty acids, mercaptans	Electroanalytical	Direct titration with Ag^+
Zn^{2+}	Modified Volhard	Precipitation as $ZnHg(SCN)_4$, filtration, dissolution in acid addition of excess Ag^+, back-titration of excess Ag^+
F^-	Modified Volhard	Precipitation as PbClF, filtration, dissolution in acid, addition of excess Ag^+, back-titration of excess Ag^+

There are three common chemical indicators that are associated with argentometric titrations:
1. The chromate ion, CrO_4^{2-} (the Mohr method);
2. The ferric ion, Fe^{3+} (the Volhard method);
3. Adsorption indicators such as fluorescein (the Fajans method).

In this experiment, we will be applying Mohr method; however, it is useful to briefly describe all three methods of endpoint detection.

The Mohr method

This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

$Ag^+_{(aq)} + Cl^-_{(aq)} \rightarrow AgCl_{(s)}$

The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium chromate, to form a red-brown precipitate of silver chromate.

$Ag^{+}_{(aq)} + CrO_4^{2-}{}_{(aq)} \rightarrow Ag_2CrO_4{}_{(s)}$

This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water.
The pH of the sample solutions should be between 6.5 and 10. At higher pH silver ions may be removed by precipitation with hydroxide ions, and at low pH chromate ions may be removed by an acid-base reaction to form hydrogen chromate ions or dichromate ions, affecting the accuracy of the end point. If the solutions are acidic, the gravimetric method or Volhard's method should be used.

The Volhard method

The Volhard method of Ag^+ determination is associated with argentometric titrations even though the titrating agent is actually SCN^- :

$$Ag^{+}_{(aq)} + SCN^{-}{}_{(aq)} \rightarrow AgSCN_{(s)}$$

The indicator in Volhard titrations is Fe^{3+}, which reacts with titrant to form a red colored complex:

$$Fe^{3+}_{(aq)} + SCN^{-}{}_{(aq)} \rightarrow Fe(SCN)^{2+}_{(aq)}$$

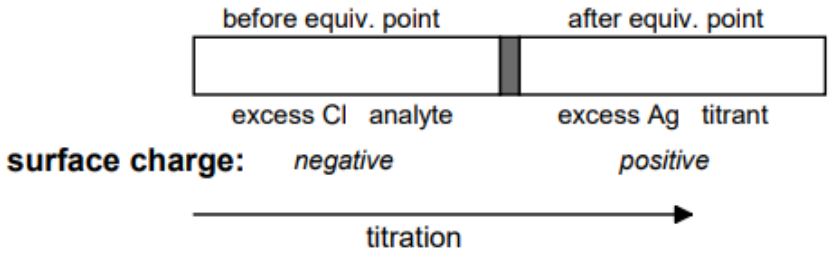
This is a good method for the analysis of Ag^+ in solution. We can extend the applicability of this method to anions such as I^- through the procedure known as back-titration. A measured excess of Ag^+ is added to the dissolved sample. After the precipitation of AgI is complete, the concentration of excess Ag^+ titrant is determined by a Volhard titration. In a similar manner, the Volhard titration method can be used to analyze for a number of anions.

The Fajans method

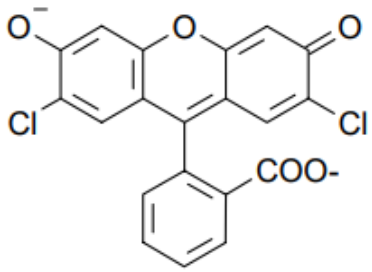
Adsorption indicators function in an entirely different manner than the chemical indicators described thus far, and they can be used in many precipitation titrations, not just argentometric methods. Let's imagine that we wish to analyze Cl^- in a sample solution by titrating with Ag^+ ;
The titration reaction would be

$Ag^{+}_{(aq)} + Cl^{-}_{(aq)} \rightarrow AgCl_{(s)}$

Silver chloride forms colloidal particles. Before the equivalence point, the surface of the precipitant particles will be negatively charged due to the adsorption of excess Cl^- to the surface of the particles. A diffuse positive counter-ion layer will surround the particles. When the equivalence point is reached, there is no longer an excess of analyte Cl^- , and the surface of the colloidal particles are largely neutral. After the equivalence point, there will be an excess of titrant Ag^+, some of these will adsorb to the solid AgCl particles, which will now be surrounded by a diffuse negative counter-ion layer. The next figure illustrates this concept.

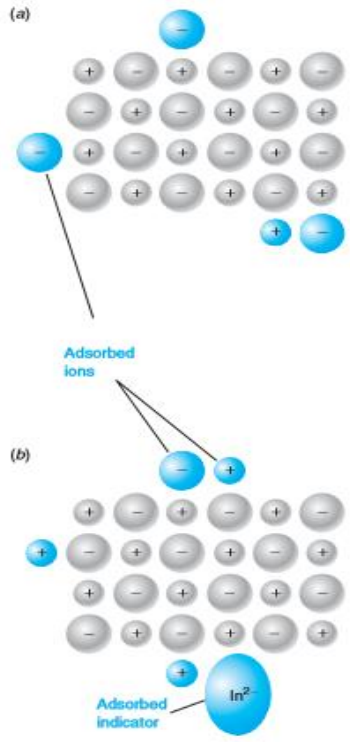


Adsorption indicators are dyes, such as dichlorofluorescein (shown below), that usually exist as anions in the titration solution.



dichlorofluoroscein

The doubly charged dichlorofluoroscein anion is attracted into the counter-ion layer immediately following the equivalence point, when the surface charge of the particles changes from negative to positive. For reasons that are not fully understood, the closer proximity of the dye to the particles changes the color of the molecule, providing a visual indication of the titration endpoint. In the case of dichlorofluorescein, the indicator changes to a pinkish color.



Ions from solution are adsorbed on the surface of a growing crystallite. (a) A crystal growing in the presence of excess lattice anions (anions that belong in the crystal) will have a slight negative charge because anions are predominantly adsorbed. (b) A crystal growing in the presence of excess lattice cations will have a slight positive charge and can therefore adsorb a negative indicator ion. Anions and cations in the solution that do not belong in the crystal lattice are less likely to be adsorbed than are ions belonging to the lattice. These diagrams omit other ions in solution. Overall, each solution plus its growing crystallites must have zero total charge.

Today you will analyze a solid chloride containing sample by Mohr method to determine the mass percent of chloride in it.

Procedure

4. Weigh 0.1 g samples of your chloride containing sample into 250-mL flask.
5. Add 20 mL of water to dissolve your sample.
6. Pipet 1 ml of potassium chromate indicator.
7. Titrate with 0.1 M silver nitrate.
8. Notice the appearance of silver chloride precipitate.
9. The endpoint of the titration is identified as the first appearance of a red-brown color of silver chromate.
10. Repeat the titration two more times.

Your unknown could be NaCl or KCl, each student must calculate the mass percent of chloride in each compound and compare their results, in order to identify their unknown.

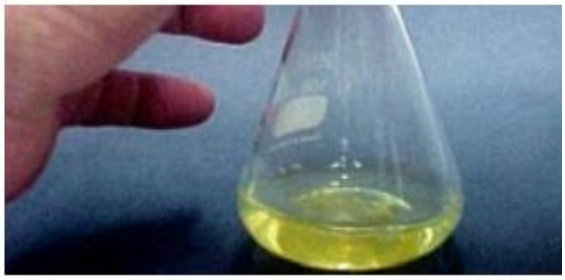


Figure 1 Before the addition of any silver nitrate the chromate indicator gives the clear solution a lemon-yellow colour.

Figure 2 Left flask: before the titration endpoint, addition of Ag^+ ions leads to formation of silver chloride precipitate, making the solution cloudy. The chromate indicator gives a faint lemon-yellow colour. Centre flask: at the endpoint, all the Cl^- ions have precipitated. The slightest excess of Ag^+ precipitates with the chromate indicator giving a slight red-brown colouration. Right flask: If addition of Ag^+ is continued past the endpoint, further silver chromate precipitate is formed and a stronger red-brown colour results. NB: The titration should be stopped when the first trace of red-brown colour is observed. Using an incompletely titrated reference flask for comparison is a helpful way to identify the first appearance of red-brown colouration.

Lab Session # 11
Application of Ion-Exchange Resins: The Separation of Cations

OBJECTIVES OF THE EXPERIMENT:
After completing this experiment, the students should be able to:

1.Describe the process of ion exchange chromatography
2.Draw the schematic diagram of the apparatus used in the experiment
3.Perform the calculations involved ion exchange chromatography

INTRODUCTION:

An ion-exchange resin or ion-exchange polymer is an insoluble matrix (or support structure) normally in the form of small (1±2 mm diameter) beads, usually white or yellowish, fabricated from an organic polymer substrate. The material has highly developed structure of pores on the surface of which are sites with easily trapped and released ions. The trapping of ions takes place only with simultaneous releasing of other ions; thus the process is called ion-exchange. There are multiple different types of ion-exchange resin which are fabricated to selectively prefer one or several different types of ions. Ion-exchange resins are widely used in different separation, purification, and decontamination processes. The most common examples are water softening and water purification. In many cases ion-exchange resins were introduced in such processes as a more flexible alternative to the use of natural or artificial zeolites. Most typical ion-exchange resins are based on crosslinked polystyrene. The required active groups can be introduced after polymerization, or substituted monomers can be used. For example, the crosslinking is often achieved by adding 0.5-25% of divinylbenzene to styrene at the polymerization process. Non-crosslinked polymers are used only rarely because they are less stable. Crosslinking decreases ion- exchange capacity of the resin and prolongs the time needed to accomplish the ion exchange processes. Particle size also influences the resin parameters; smaller particles have larger outer surface, but causes larger head loss in the column processes. Besides being made as bead-shaped materials, ion exchange resins are produced as membranes. The membranes are made of highly cross-linked ion exchange resins that allow passage of ions, but not of water, are used for electrodialysis. Several cations can form negatively charged (anionic) chloro complex with hydrochloric acid. The anion complex can be adsorbed by an anion exchanger. Every metal is adsorbed at certain pH range and property can be used as the basis for separation. For example, zinc can be adsorbed from 2 M of acidic solution but not magnesium and aluminum. Therefore, if a solution containing Zn^{2+} and Mg^{2+} being introduced to column, only Zn^{2+} will be adsorbed. Mg^{2+} is thus separated from Zn^{2+} which can then be eluted with dilute nitric acid. The amount of separated Mg^{2+} and Zn^{2+} can be determined through EDTA titration. The advantages of ion exchange processes are the very low running costs. Very little energy is required, the regenerant chemicals are cheap and if well maintained resin beds can last for many years before replacement is needed. There are, however, a number of limitations which must be taken into

account very carefully during the design stages. When itemized these limitations appear to represent a formidable list and the impression can be given that ion-exchange methods might have too many short comings to useful in practice. However, this is not the case as the advantages mentioned above are very great and compensation can readily be made for most restrictions

Procedure:

1. Regenerate the ion-exchanger column with 10 ml of 6 M HCl.
2. Add 3 drops of the ions mixture.
3. Add 5 ml of 2 M HCl
4. Collect the eluate in a 100 ml flask.
5. Replace the flask under the column with another 100 ml flask.
6. Wash the column with 5 ml of 0.5 M HNO_3. Collect the eluate.
7. Basify your solutions (in both flasks) by adding 6 M NH_4OH and add 10 ml of NH_4Cl-NH_3 buffer and 2-3 drops of Erichrome Black Tea (EBT) indicator.
8. Titrate your solutions against 0.01 M EDTA solution until the solution color changes from purple to sky blue.

Lab Session #12
Separation of mixture of $K_2Cr_2O_7$ and $KMnO_4$ by column chromatography

The separation of mixture containing both the potassium dichromate and potassium permanganate pass from the separation column containing material alumina acid, solvent using distilled water as the loyal and adsorption dichromate on the surface of alumina acid more adsorption permanganate therefore concludes permanganate first. If checked consider dichromate layer orange in the separation column you will notice a yellow minutes of chromate potassium accompanying the potassium dichromate and who is with him in equilibrium as shown in the following equation:

$$Cr_2O_7^{2-} + H_2O \leftrightarrow 2HCrO_4^-$$

Procedure:

1. Prepare slurry of alumina by mixing 5g of alumina and 5 ml of 0.5 M HNO_3.
2. Swirl the slurry well and transfer it to the column.
3. Add 4 drops of your permanganate dichromate mixture sample on the top of the pipette and let the sample to penetrate the alumina surface.
4. Wash the pipette with 5 ml 0.5M HNO_3 until the purple color approaches the end of the pipette.
5. Elute the purple fraction by washing with 0.5 M HNO_3. (this step means you have to continue adding small portions of HNO_3 until the permanganate layer is eluted and then must be collected in a flask)
6. In another flask Elute the dichromate (yellow layer) by washing with 1M H_2SO_4 (around 5-7 ml).
7. Measure the volume of each of the permanganate and dichromate layers before titration.
8. Titrate each flask with 0.02 M iron sulfate.
    a. For the permanganate, add 5ml of 3M H2SO4 then Place the flask on a hotplate and heat to a temperature of 60-70 degrees. Then titrate it against $FeSO_4$.
    b. For the Dicromate, add 5 mL of the l M phosphoric acid solution and 8 drops of sodium diphenylamine sulfonate indicator to the flask. Then titrate it against $FeSO_4$.
9. Calculate molarity and ratio of each of permanganate and dichromate in the original sample.

DATA SHEETS

Lab session # 2
Gravimetric Determination of Iron as Fe_2O_3

Name:…….

Unknown # …….

#		**Result**
1.	Mass of the iron salt	
2.	Mass of the filter paper	
3.	Mass of the filter paper and Fe_2O_3	
4.	Mass of Fe_2O_3	
5.	Moles of Fe_2O_3	
6.	Moles of iron in Fe_2O_3	
7.	Mass of iron in Fe_2O_3	
8.	% Mass of iron in the iron salt	
9.	% Mass of iron in 1- $FeSO_4 \cdot 7H_2O$ 2- $FeC_2O_4(H_2O)_2$	
10.	Formula of the iron salt	

Lab session # 3
Titration of a strong acid with a strong base

Name:…….

Unknown # ……

Standardization of NaOH solution	Trial 1	Trial 2	Trial 3
Mass of KHP			
Molar mass of KHP	204.22 g/mol		
Moles of KHP present (Show calculations for Trial 1)			
Initial NaOH buret reading			
Final NaOH buret reading			
Volume NaOH used			
Moles of NaOH used (Show calculations for Trial 1)			
Molarity of NaOH solution (Show calculations for Trial 1)			
Mean NaOH molarity (Show calculations)			

Molarity of HCl solution	Trial 1	Trial 2	Trial 3
Volume of HCl solution			
Initial NaOH buret reading			
Final NaOH buret reading			
Volume NaOH used			
Moles of NaOH present (Show calculations for Trial 1)			
Moles of HCl present (1:1 ratio)			
Molarity of HCl solution (Show calculations for Trial 1)			
Mean HCl molarity			

Molarity of H_2SO_4 solution	Trial 1	Trial 2	Trial 3
Volume of H_2SO_4 solution			
Initial NaOH buret reading			
Final NaOH buret reading			
Volume NaOH used			
Moles of NaOH present (Show calculations for Trial 1)			
Moles of H_2SO_4 present (2:1 ratio) (Show calculations for Trial 1)			
Molarity of H_2SO_4 solution (Show calculations for Trial 1)			
Mean H_2SO_4 molarity			

Lab session # 4
Titration of a weak acid with a strong base

Name:…….

1. Titration of H_3PO_4 solution with NaOH solution:

Titration of H_3PO_4 soln with NaOH	Trial 1	Trial 2
Indicator: Bromocresol Green		
Volume of H_3PO_4 solution		
Molarity of NaOH solution		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume NaOH used		
pH at the end point		
Moles of NaOH used (Show calculations for Trial 1)		
Moles of H_3PO_4 present (ratio??)		
Molarity of H_3PO_4 solution (Show calculations for Trial 1)		
Mean H_3PO_4 molarity		

Titration of H_3PO_4 soln with NaOH	Trail 1	Trial 2
Indicator: Methyl Red		
Volume of H_3PO_4 solution		
Molarity of NaOH solution		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume NaOH used		
pH at the end point		
Moles of NaOH used		
Moles of H_3PO_4 present		
Molarity of H_3PO_4 solution		
Mean H_3PO_4 molarity		

Titration of H_3PO_4 soln with NaOH	Trial 1	Trial 2
Indicator: Phenolphethalein		
Volume of H_3PO_4 solution		
Molarity of NaOH solution		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume NaOH used		
pH at the end point		
Moles of NaOH used		
Moles of H_3PO_4 present		
Molarity of H_3PO_4 solution		
Mean H_3PO_4 molarity		

2. Titration of CH_3COOH (AcOH) solution with NaOH solution:

Titration of AcOH soln with NaOH	Trial 1	Trial 2
Indicator: Bromocresol Green		
Volume of AcOH solution		
Molarity of NaOH solution		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume NaOH used		
pH at the end point		
Moles of NaOH used (Show calculations for Trial 1)		
Moles of AcOH present (ratio??)		
Molarity of AcOH solution (Show calculations for Trial 1)		
Mean AcOH molarity		

Titration of AcOH soln with NaOH	Trial 1	Trial 2
Indicator: Methyl Red		
Volume of AcOH solution		
Molarity of NaOH solution		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume NaOH used		
pH at the end point		
Moles of NaOH used		
Moles of AcOH present		
Molarity of AcOH solution		
Mean AcOH molarity		

Titration of AcOH soln with NaOH	Trial 1	Trial 2
Indicator: Phenolphethalein		
Volume of AcOH solution		
Molarity of NaOH solution		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume NaOH used		
pH at the end point		
Moles of NaOH used		
Moles of AcOH present		
Molarity of AcOH solution		
Mean AcOH molarity		

The best indicator for Titration of H_3PO_4 solution with NaOH solution:

- For the first endpoint is ----------
- For the second endpoint is ----------

The best indicator for Titration of AcOH solution with NaOH solution is:----------

Lab session # 5
Analysis of a mixture of carbonate and bicarbonate

Name:…….

NaOH standardization	Trial 1	Trial 2
Molar mass of KHP	204.22 g/mol	
Mass of KHP		
Moles KHP		
Initial buret reading		
Final buret reading		
VolumeNaOH used		
Moles of NaOH		
Molarity Of NaOH		
Mean of NaOH molarity		

Standardization of HCl	Trial 1	Trial 2
Volume of HCl		
Initial NaOH buret reading		
Final NaOH buret reading		
Volume of NaOH		
Moles of NaOH		
Moles of HCl		
Molarity Of HCl		
Mean of HCl molarity		

Total Alkalinity	Trial 1	Trial 2
Volume of (CO_3^{2-} and HCO_3^-) solution		
Mean of HCl molarity		
Initial HCl buret reading		
Final HCl buret reading		
Volume HCl used		
Moles of HCl consumed		
Mean of moles of HCl consumed		

Calculate total alkalinity (=$[HCO_3^-]$ + 2$[CO_3^{2-}]$).
(Show calculations)

Bicarbonate Concentration	Trial 1	Trial 2
Volume of (CO_3^{2-} and HCO_3^-) solution		
Concentration of NaOH added		
Volume of NaOH added		
Volume of 10% $BaCl_2$ added		
HCl concentration		
Initial HCl buret reading		
Final HCl buret reading		
Volume HCl used		
Moles of HCl consumed		
Mean of moles of HCl consumed		

Calculate the Bicarbonate concentration. Then calculate the carbonate concentration. (Show calculations)

Lab session # 6
Evaluation of calcium in commercial milk powder

Name:…….

Part A: Determination of Ca^{2+} in the 1st milk brand

	Trial 1	Trial 2	Trial 3
Brand name of milk powder sample			
Mass of milk powder sample			
Concentration of EDTA Solution			
Initial EDTA buret reading			
Final EDTA buret reading			
Volume of EDTA solution needed			
Moles of EDTA needed (Show Calculations for 1st trial)			
Moles of Ca^{2+} in milk powder sample (Show Calculations for 1st trial)			
Mass of Ca^{2+} in milk powder sample (Show Calculations for 1st trial)			
Mass % of Ca^{2+} in milk powder sample (Show Calculations for 1st trial)			
Mean mass % of Ca^{2+} in milk powder sample			

Part B: Determination of Ca^{2+} in the 2nd milk brand

	Trial 1	Trial 2	Trial 3
Brand name of milk powder sample			
Mass of milk powder sample			
Concentration of EDTA Solution			
Initial EDTA buret reading			
Final EDTA buret reading			
Volume of EDTA solution needed			
Moles of EDTA needed (Show Calculations for 1st trial)			
Moles of Ca^{2+} in milk powder sample (Show Calculations for 1st trial)			
Mass of Ca^{2+} in milk powder sample (Show Calculations for 1st trial)			
Mass % of Ca^{2+} in milk powder sample (Show Calculations for 1st trial)			
Mean mass % of Ca^{2+} in milk powder sample			

Which milk brand has Calcium?

Lab session # 7
Reduction oxidation titration; Potassium dichromate

Name:…….

	Trial 1	Trial 2	Trail 3
Concentration of $K_2Cr_2O_7$ solution (mol/L)			
Unknown No.			
mass of Fe^{2+} sample			
Initial buret reading			
Final buret reading			
Volume of $K_2Cr_2O_7$ solution needed			
Moles of $K_2Cr_2O_7$ (Show Calculations for 1st trial)			
Moles of Fe^{2+} (Show Calculations for 1st trial)			
Mass of Fe^{2+} (Show Calculations for 1st trial)			
Mean mass of Fe^{2+}			
% mass of Fe^{2+} in your sample (Show Calculations)			
Unknown Identity			

Lab session # 8
Reduction oxidation titration; Potassium permanganate

Name:…….

	Trial 1	Trial 2	Trail 3
Concentration of $KMnO_4$ solution			
Unknown No.			
Volume of $C_2O_4^{2-}$ solution			
Volume of H_2SO_4 added			
Initial MnO_4^- buret reading			
Final MnO_4^- buret reading			
Volume of MnO_4^- solution needed			
moles of MnO_4^- needed (Show Calculations for 1st trial)			
Moles of $C_2O_4^{2-}$ (Show Calculations for 1st trial)			

Molarity of $C_2O_4^{2-}$ solution (Show Calculations for 1st trial)			
Mean molarity of $C_2O_4^{2-}$ solution			

Lab session # 9
The determination of calcium as calcium oxalate

Name:…….

	Trial 1	Trial 2	Trail 3
Concentration of $KMnO_4$ solution			
Unknown No.			
Mass of Calcium Salt Sample			
Initial MnO_4^- buret reading			
Final MnO_4^- buret reading			
Volume of MnO_4^- solution needed			
Moles of MnO_4^- (Show calculations for 1st trial)			
Moles of $C_2O_4^{2-}$ (Show calculations for 1st trial)			
Moles of Ca^{2+} (Show calculations for 1st trial)			
Mass% of Ca^{2+} (Show calculations for 1st trial)			
Mean Mass% of Ca^{2+}			

Lab Session # 10
Determination of Chloride Ion Concentration (The Mohr Method)

Name----------

	Trial 1	Trial 2	Trial 3
Unknown number			
Mass of the chloride salt sample			
Concentration of $AgNO_3$ Solution (mol/L)			
Initial $AgNO_3$ buret reading			
Final $AgNO_3$ buret reading			
Volume of $AgNO_3$ solution needed			
Moles of $AgNO_3$ needed			
Moles of Cl^- in the salt sample (1:1)			
Mass of Cl^- in the salt sample (Show calculations for 1st Trial)			
Mass % of Cl^- in the salt sample (Show calculations for 1st Trial)			
Mean mass % of Cl^- in the salt sample			

Lab Session # 11
Application of Ion-Exchange Resins: The Separation of Cations

Name: …….

1- Concentration of EDTA Solution (mol/L):----------

	Trial 1
Volume of Mg^{2+} Eluate	
Volume of Mg^{2+} Eluate to be titrated with EDTA	
Initial EDTA buret reading	
Final EDTA buret reading	
Volume of EDTA solution needed	
Moles of EDTA needed	
Moles of Mg^{2+}	
Total Moles of Mg^{2+} titrated	
Volume of Zn^{2+} Eluate	
Volume of Zn^{2+} Eluate to be titrated with EDTA	
Initial EDTA buret reading	
Final EDTA buret reading	
Volume of EDTA solution needed	
Moles of EDTA needed	
Moles of Zn^{2+} titrated	
Total Moles of Zn^{2+}	
Mg^{2+}: Zn^{2+} Molar Ratio	

Lab Session #12
Separation of mixture of $K_2Cr_2O_7$ and $KMnO_4$ by column chromatography

Name:……

1-Concentration of iron sulfate Solution (mol/L):----------

	Trial 1
Volume of Cr_2O_7Eluate	
Volume of $Cr_2O_7^{-2}$Eluate	
Initial iron sulfate buret reading	
Final iron sulfate buret reading	
Volume of iron sulfate solution needed	
Moles of iron sulfate needed	
Moles of $Cr_2O_7^{-2}$	
Total Moles of $Cr_2O_7^{-2}$ titrated	
Volume of MnO_4^- Eluate	
Volume of MnO_4^- Eluate	
Initial iron sulfate buret reading	
Final iron sulfate buret reading	
Volume of iron sulfate solution needed	
Moles of iron sulfate needed	
Moles of MnO_4^- titrated	
Total Moles of MnO_4^-	
$Cr_2O_7^{-2}$: $MnO4^-$ Molar Ratio	