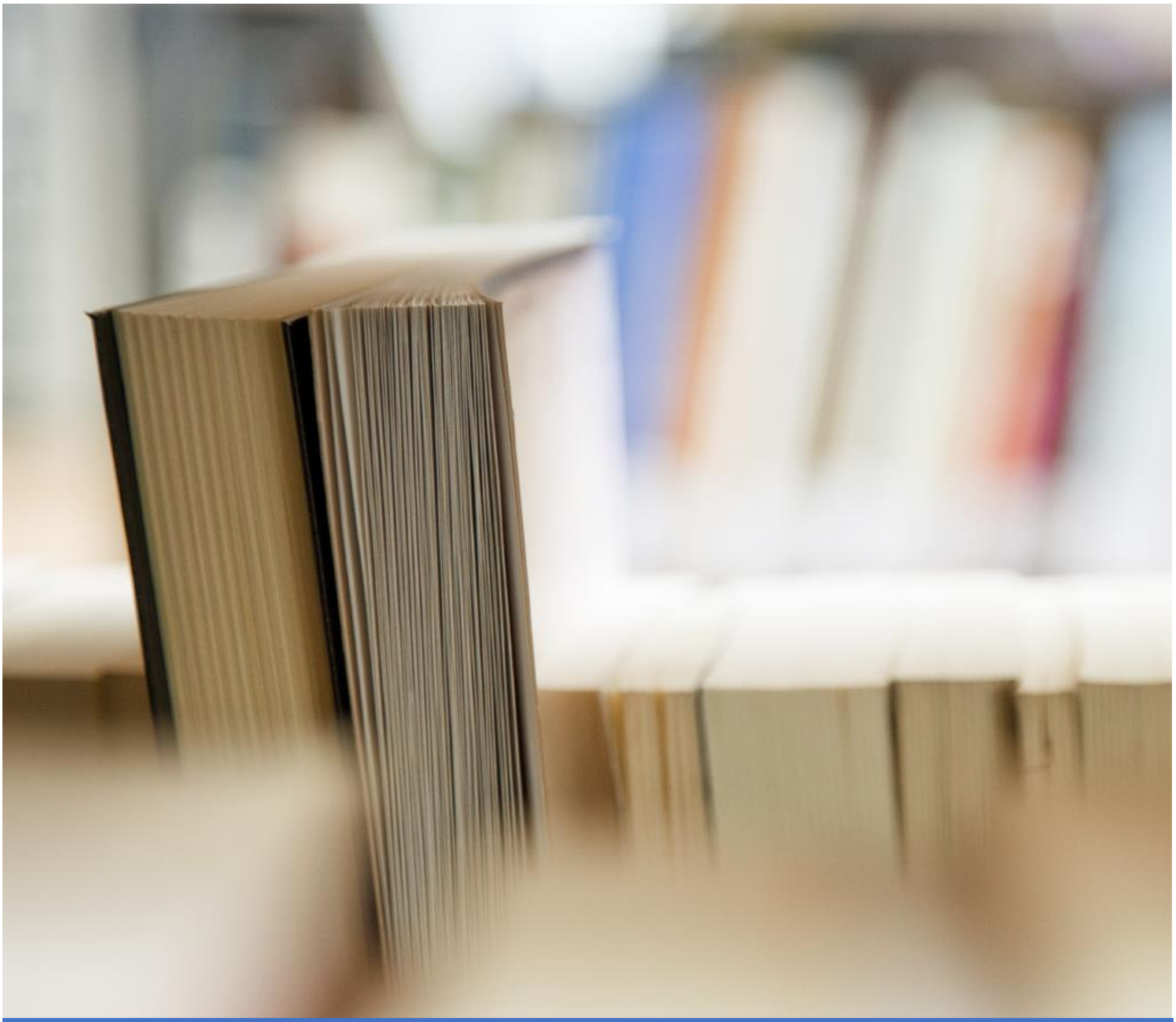


GRAVIMETRIC ANALYTICAL CHEMISTRY – LAB

Experimental 2nd Lab.

Basima A.A.Saleem

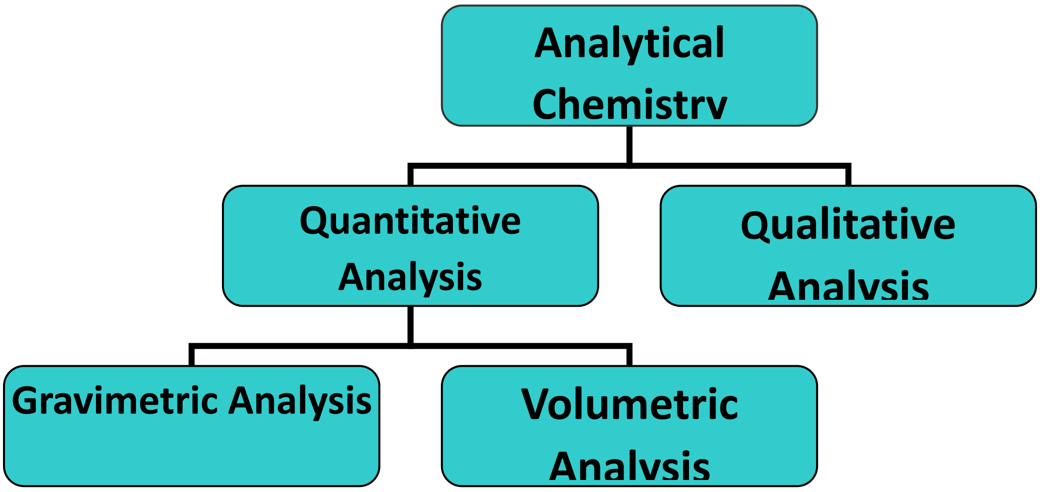
Analytical Chemistry

Introduction:

Analytical chemistry is inherently a quantitative science. Whether determining the concentration of a species in a solution, evaluating an equilibrium constant, measuring a reaction rate, or drawing a correlation between a compound's structure and its reactivity.

Analytical chemists make measurements and perform calculations. The quantitative analysis of inorganic chemical substances, determining levels of purity and contamination, constitutes the core of much of modern industrial and environmental chemistry. In gravimetric analysis, the mass of a pure solid of known composition is determined.

In volumetric analysis, the volume of an added reagent of known concentration is measured. In instrumental analysis, a physical property is measured spectroscopically, such as the absorption of some form of electromagnetic radiation.



INTRODUCTION TO GRAVIMETRIC ANALYTICAL CHEMISTRY – LAB

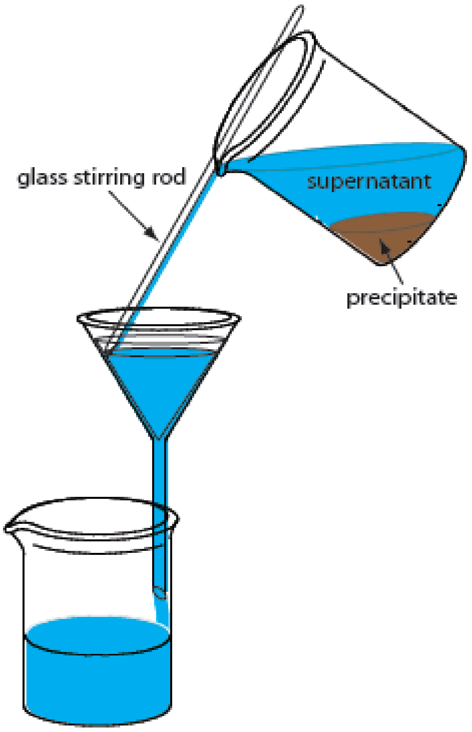
Gravimetric analysis, which by definition is based upon the measurement of mass, can be generalized into two types; precipitation and volatilization. The quantitative determination of a substance by the precipitation method of gravimetric analysis involves isolation of an ion in solution by a precipitation reaction, filtering, washing the precipitate free of contaminants, conversion of the precipitate to a product of known composition, and finally weighing the precipitate and determining its mass by difference.

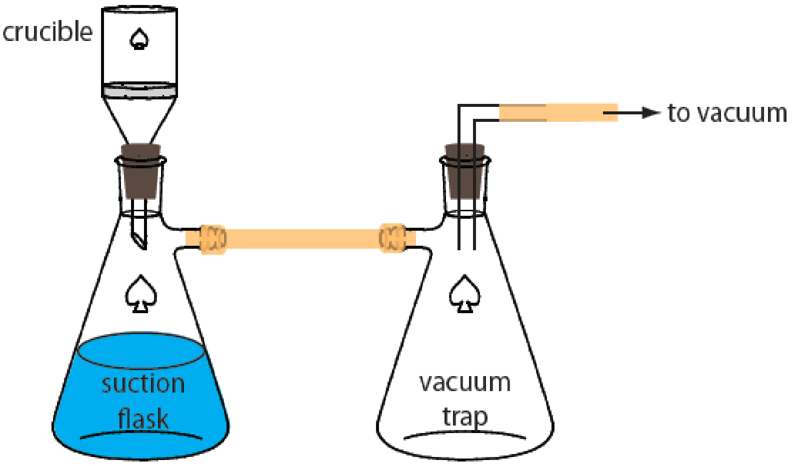
From the mass and known composition of the precipitate, the amount of the original ion can be determined.

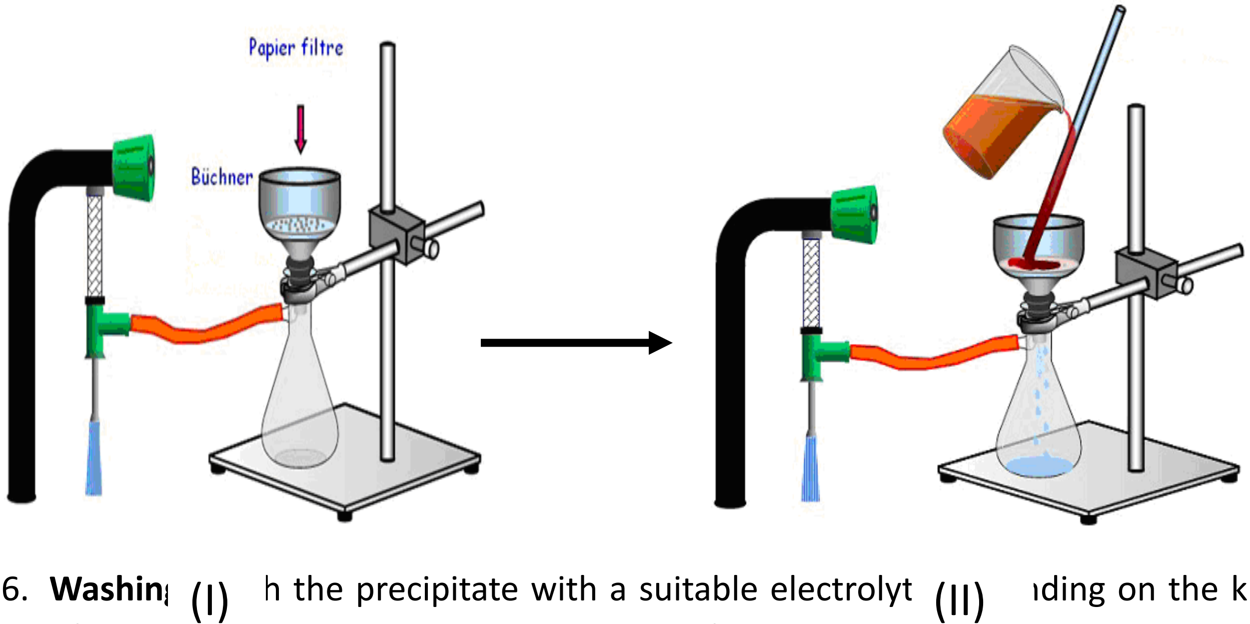
Steps of gravimetric analysis:

1. **Dissolve the analyte:** if the analyte is solid weight as your procedure then dissolve it after choosing the suitable solvent or else take a suitable volume from the liquid analyte.
2. **first treatment of the analyte:** this involve choosing pH, temperature and masking agent.
3. **precipitation:** this is most important step because the final result depends on it that mean adding reagent (precipitate agent) gradually drop by drop. Show Table 1 ,2 & 3.
4. **Digestion :** the purpose of this step is to easy filtrate and remove all grays and re-crystallization of crystals by leaving the precipitate with its filtrate in suitable place, temperature and limited time.
5. **Filtration:**

This step mean separating precipitate from its filtrate by filter paper, Buchner funnel or josh crucible.







6. **Washin** h the precipitate with a suitable electrolyt ding on the kind of preci to remove any grays must be found.

7. **Drying or burning:** dry the precipitate in an oven or burn it in furnous .

8. **Weighing the precipitate**: weighing is crucial to quantitative analysis. Very accurate weighing is performed on analytical balances. You will be shown the correct procedure by a demonstrator. Chemicals must **never** be added to containers inside the balance draft cover. Approximate weighing is carried out on bench flat-top balances. Spillages of chemicals on the balances must be cleaned up immediately.

9. **Calculations**: this step depend on the kind of the sample (solid or liquid).

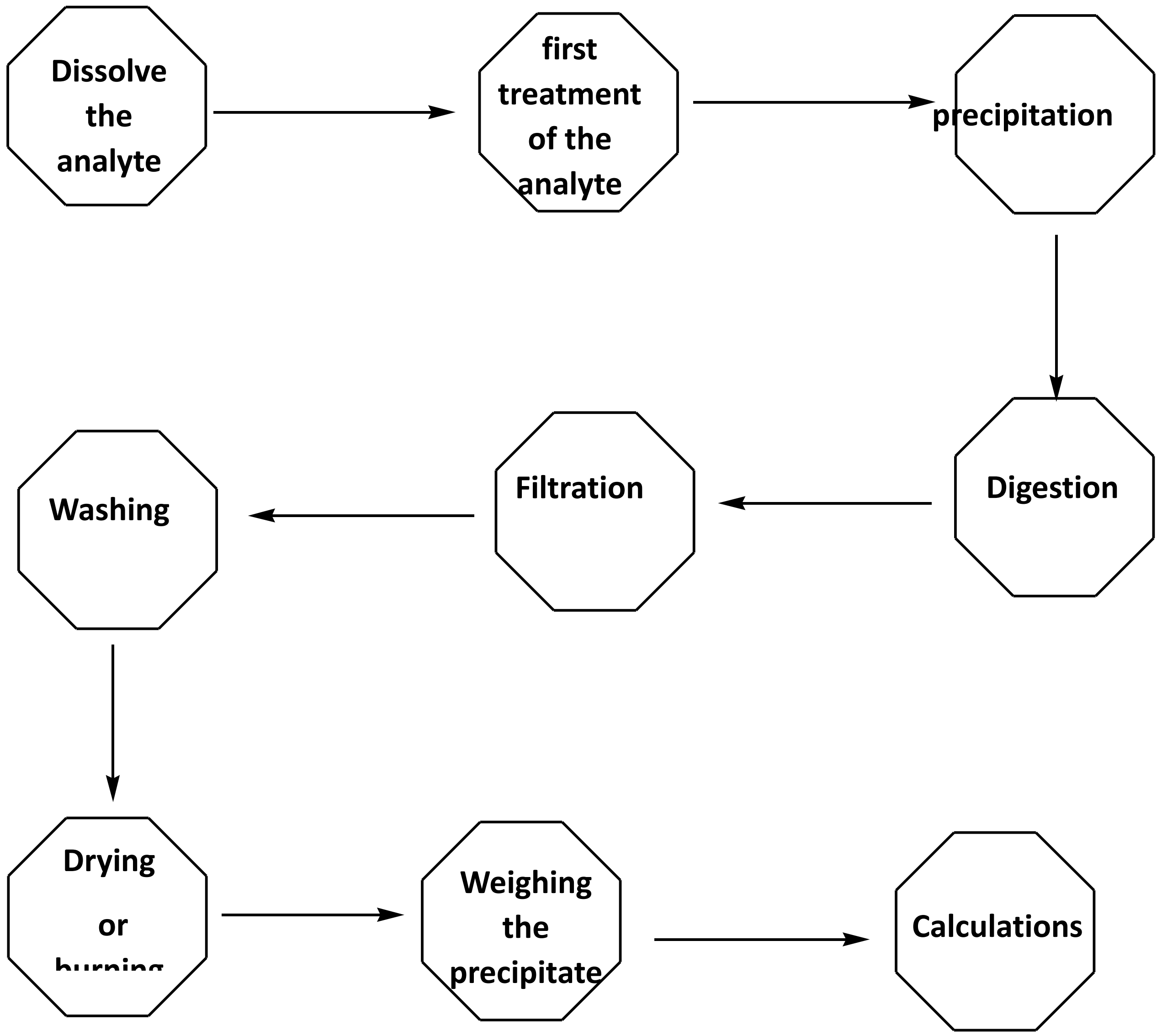


Figure 1: The steps of precipitate

Table 1: Selected precipitation gravimetric methods for inorganic cations and anions (arranged by precipitant).

Analyte	Precipitant	Precipitate Formed	Precipitate Weighed
Ba^{2+}	$(NH_4)_2CrO_4$	$BaCrO_4$	$BaCrO_4$
Pb^{2+}	K_2CrO_4	$PbCrO_4$	$PbCrO_4$
Ag^+	HCl	$AgCl$	$AgCl$
$Hg_2{}^{2+}$	HCl	Hg_2Cl_2	Hg_2Cl_2
Al^{3+}	NH_3	$Al(OH)_3$	Al_2O_3
Be^{2+}	NH_3	$Be(OH)_2$	BeO
Fe^{3+}	NH_3	$Fe(OH)_3$	Fe_2O_3
Ca^{2+}	$(NH_4)_2C_2O_4$	CaC_2O_4	$CaCO_3$ or CaO
Sb^{3+}	H_2S	Sb_2S_3	Sb_2S_3
As^{3+}	H_2S	As_2S_3	As_2S_3
Hg^{2+}	H_2S	HgS	HgS
Ba^{2+}	H_2SO_4	$BaSO_4$	$BaSO_4$
Pb^{2+}	H_2SO_4	$PbSO_4$	$PbSO_4$
Sr^{2+}	H_2SO_4	$SrSO_4$	$SrSO_4$
Be^{3+}	$(NH_4)_2HPO_4$	NH_4BePO_4	$Be_2P_2O_7$
Mg^{2+}	$(NH_4)_2HPO_4$	NH_4MgPO_4	$Mg_2P_2O_7$
Zn^{2+}	$(NH_4)_2HPO_4$	NH_4ZnPO_4	$Zn_2P_2O_7$
Sr^{2+}	KH_2PO_4	$SrHPO_4$	$Sr_2P_2O_7$
CN^-	$AgNO_3$	$AgCN$	$AgCN$
I^-	$AgNO_3$	AgI	AgI
Br^-	$AgNO_3$	$AgBr$	$AgBr$
Cl^-	$AgNO_3$	$AgCl$	$AgCl$
$ClO_3{}^-$	$FeSO_4/AgNO_3$	$AgCl$	$AgCl$
SCN^-	$SO_2/CuSO_4$	$CuSCN$	$CuSCN$
$SO_4{}^{2-}$	$BaCl_2$	$BaSO_4$	$BaSO_4$

Table 2: Selected precipitation gravimetric methods for the analysis of organic functional groups and heteroatoms.

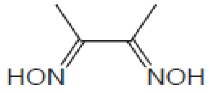
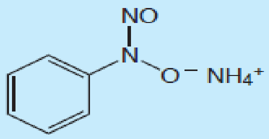
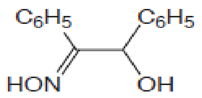
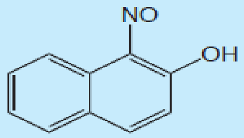
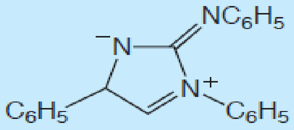
Analyte	Treatment	Precipitant	Precipitate
Organic halides R–X X = Cl, Br, I	Oxidation with HNO_3 in the presence of Ag^+	$AgNO_3$	AgX
Organic halides R–X X = Cl, Br, I	Combustion in O_2 (with a Pt catalyst) in the presence of Ag^+	$AgNO_3$	AgX
Organic sulfur	Oxidation with HNO_3 in the presence of Ba^{2+}	$BaCl_2$	$BaSO_4$
Organic sulfur	Combustion in O_2 (with Pt catalyst) with SO_2 and SO_3 collected in dilute H_2O_2	$BaCl_2$	$BaSO_4$
Alkoxy groups –O–R or –COO–R R = CH_3 or C_2H_5	Reaction with HI to produce RI	$AgNO_3$	AgI







Table 3: Selected precipitation gravimetric methods for inorganic ions using an organic precipitant.

Analyte	Precipitant	Structure	Precipitate Formed	Precipitate Weighed
Ni^{2+}	dimethylglyoxime		$Ni(C_4H_7O_2N_2)_2$	$Ni(C_4H_7O_2N_2)_2$
Fe^{3+}	cupferron		$Fe(C_6H_5N_2O_2)_3$	Fe_2O_3
Cu^{2+}	cupron		$CuC_{14}H_{11}O_2N$	$CuC_{14}H_{11}O_2N$
Co^{2+}	1-nitrso-2-napthol		$Co(C_{10}H_6O_2N)_3$	Co or $CoSO_4$
K^+	sodium tetraphenylborate	$Na[B(C_6H_5)_4]$	$K[B(C_6H_5)_4$	$K[B(C_6H_5)_4$
NO_3^-	nitron		$C_{20}H_{16}N_4HNO_3$	$C_{20}H_{16}N_4H$-NO_3

Gravimetric methods of analysis include:

1.Precipitation method:

In precipitation gravimetry an insoluble compound forms when we add a precipitating reagent, or precipitant, to a solution containing our analyte. In most methods the precipitate is the product of a simple metathesis reaction between the analyte and the precipitant; however, any reaction generating a precipitate can potentially serve as a gravimetric method.

2. Volatilization method:

A second approach to gravimetry is to thermally or chemically decompose the sample and measure the resulting change in its mass. Alternatively, we can trap and weigh a volatile decomposition product. Because the release of a volatile species is an essential part of these methods, we classify them collectively as volatilization gravimetric methods of analysis. This method includes

a- direct method (raising the weight).

b- Indirect method (decreasing the weight)

Controlling Precipitation

$$R = \frac{Q - S}{S}$$

Q = Concentration
S = Solubility

R = relative Super saturation

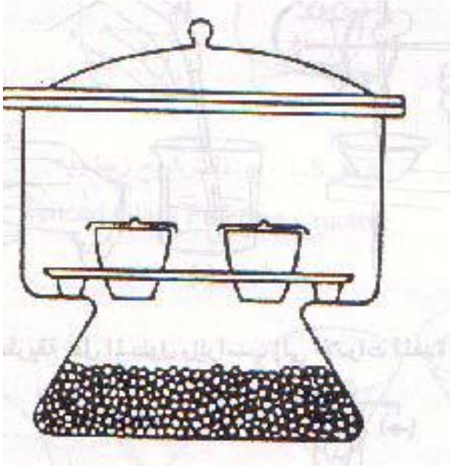
where Q is the concentration of the solute in solution at the instant the reagents are mixed, S is the concentration of the solute in a saturated solution (a saturated solution is a solution in which the solute in solution is in equilibrium with the solid solute), and Q - S , called super saturation, is the concentration of the solute in excess of that found in a saturated solution. When (Q – S / S) is large, then the rate of nucleation is much greater than the rate of growth and the particle size of the precipitate is very small.

Techniques to minimize (Q – S / S) by decreasing the value of (R), then you must increase S, increase temperature, decrease Q, dilute solution, well mixed (stirring) .

Equipments used in gravimetric analysis:

1. **balance:** An object’s mass is measured using a balance. The most common type of balance is an electronic balance in which the balance pan is placed over an electromagnet as shown below:

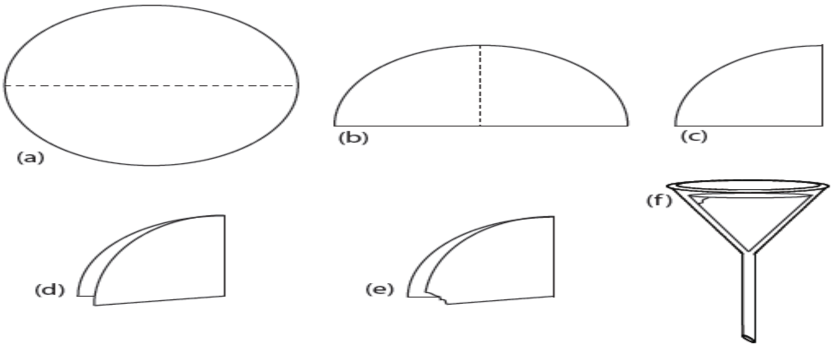
2. Desiccator: Desiccators are used to dry chemical samples, often under reduced pressure utilising an electrical air pump, in the presence of a hygroscopic material (e.g. silica gel) so that accurate weights can be recorded. When evacuation is complete the tap on the desiccator should be closed, the tube disconnected and the pump turned off – in that order! When the precipitate is dry, air can be readmitted to the desiccator *slowly* and *carefully* otherwise your sample will be blown all over the desiccator and lost - and you will have to start again! ***Analysis*** desiccator is a closed container containing a desiccant like CaO ، P_2O_5 ، $BaCl_2$ ، $CuSO_4$ ، $MgSO_4$ ، $CaCl_2$.; used to store samples in a moisture free environment.



3. Crucible of burning:

4. Ashless Filter Paper: after burn this filter paper, it have no weight can be weighted analytical balance because when it ignited, it decompose to its components as below:

$$\text{Ashless filter paper} \longrightarrow CO_2 + H_2O + \text{Cellose}$$



Preparing a filter paper cone. The filter, paper circle in (a) is folded in half (b), and folded in half again (c). The folded filter paper is parted (d) and a small corner is torn off (e). The filter paper is opened up into a cone and placed in the funnel (f).

5. Josh crucible: this crucible used for filtration to separate precipitate from its filtrate then dry the precipitate in high temperature by an oven because it made of Pyrex.

Josh crucible

Precipitation from Homogeneous Solution

Precipitation from homogeneous solution is a technique in which a precipitating agent is generated in a solution of the analyte by a slow chemical reaction. Local reagent excesses do not occur because the precipitating agent appears gradually and homogeneously throughout the solution and reacts immediately with the analyte. As a result, the relative supersaturation is kept low during the entire precipitation. In general, homogeneously formed precipitates, both colloidal and crystalline, are better suited for analysis than a solid formed by direct addition of a precipitating reagent.

Table 4: Methods for homogenous generation of precipitating agents

precipitating agents	Reagent	General reaction	Elements precipitated
OH^-	Urea	$(NH_2)_2CO + 3H_2O \longrightarrow CO_2 + 2OH^- + 2NH4^+$	Al, Ga, Th,. Bi, Fe, Sn.
PO_4^{-3}	Trimethylphosphate	$(CH_3O)_3PO + 3H_2O \longrightarrow 3CH_3OH + H_3PO_4$	Zr, Hf.
$C2O_4^{-2}$	Ethyloxalate	$(C_2H_5)_2C_2O_4 + 2H_2O \longrightarrow 2C_2H_5OH + H_2C_2O_4$	Mg, Zn, Ca.
SO_4^{-2}	Dimethyl sulphate	$(CH_3O)_2SO_2 + 4H_2O \longrightarrow 2CH_2OH + SO_4^{-2} + 2H_3O^+$	Ba, Ca, Sr, Pb.
CO_3^{-2}	Trichloro acetic acid	$CH_3COOH + 2OH^- \longrightarrow CHCl_3 + CO_3^{-2} + H_2O$	La, Ba, Ra.
H_2S	Thioacetamide	$CH_3CSNH_2 + H_2O \longrightarrow CH_3CONH_2 + H_2S$	Sb, Mo, Cu, Cd.
DMG	Biacetyl + hydroxylamine	$CH_3COCOCH_3 + 2H_2NOH \longrightarrow DMG + 2H_2O$	Ni
HOQ	8-hydroxyquinoline	$CH_3COOQ + 2H_2O \longrightarrow CH_3COOH + HOC$	Al, U, Mg, Zn.

How to write your report

The report is the culmination of all the work you as the experimenter have performed. The report is generally the only material that the outside world (your professor, supervisor, colleagues and peers) see of your work and, consequently, judgments about you and your work are generally based on the report. It is therefore critical that the report, in its presentation, organization, and content, be of the highest standard of which you are capable. A report should be typed and free of spelling, grammatical, and mathematical errors. The data presented should be clearly stated and be a compilation of information that is directly relevant to the particular conclusion(s) and/or result(s) that you are presenting to the reader. The following is a list of the important information that should be present in a scientific report:

Title Page

Include your name, section, experiment number and title, and date.

Introduction

The introduction to your report should provide the reader with background information of what has been reported in the scientific literature by previous experimenters working in the area of your experiment. You should explain how your experiment fits into previous studies and what questions you expect to answer from your study.

Experimental

A brief summary of the method that was followed and instrumentation/techniques used.

Results and Discussion

Data collected while performing the experiment must be presented (as tables/graphs). Include a single sample calculation for each different computation made. The final value for a given measurement should be an average, Describe your data and discuss its meaning. How does it relate to what you were expecting? Include a consideration of errors and any conclusions based on interpretation of the results. Discuss the implications of the results. Discuss any problems you had and how they were addressed. How could the experiment be changed to improve the data? Conclusion Briefly state the conclusions for the experiment.

References

Cite any outside sources of information used in the report.

Name of experiment :

Gravimetric determination of water in Hydrated Barium Chloride.

Number of experiment: - 1 -

The purpose of experiment:

The aim of this experiment is to determine the number of moles of water molecules of crystallisation of a sample of hydrated Barium Chloride.

Equipment: weighting crucible, spatula , washing bottle, desiccator, analytical balance, tongue.

Materials: hydrated barium chloride salt .

Theory:

The aim of this experiment is to determine the number of moles of water molecules of crystallisation of a sample of hydrated Barium Chloride. This can be calculated using Gravimetric Analysis, which involves comparing the mass of the hydrated with the mass of the anhydrous salt. Hydrated Barium Chloride is heated to constant mass. The number of moles of water of crystallisation (n in $BaCl_2. H_2O$) can be found by calculating the loss of mass in the sample.

$$BaCl_2.H_2O \xrightarrow{\Delta} BaCl_2 + H_2O$$

Procedure:

All absorbed moisture was removed from the crucible by drying in an oven and then by cooling in a dessicator. The crucible was then weighed and approximately 0.1 – 0.2 g of Hydrated Barium Chloride was added to the crucible (this was done wearing gloves and in a fume cupboard). The crucible and contents was then weighed on a balance. The crucible was then heated for 15 minutes using an oven.

The crucible was allowed to cool before being transferred to a dessicator using tongs. Once cooled, the crucible was reweighed. This process was repeated, heating for about 4 minutes each time, until two successive balance reading were within 0.002g of each other, at this point the contents were at constant mass, and all of the water had been removed.

Results and calculations:

1. calculate theoretical percentage of H_2O:

$$\% \ H_2O_{\text{Theoretical}} = \frac{2 \times \text{M.wt } H_2O}{\text{M.wt } BaCl_2.2H_2O} \times 100$$

2. calculate experimental percentage of H_2O:

$$\% \ H_2O_{\text{Experimental}} = \frac{\text{wt. of volatalize water}}{\begin{array}{c}\text{wt. of hydrated salt} \\ \text{(hydrated barium chloride)}\end{array}} \times 100$$

2. calculate the number of H_2O in the salt:

$$\frac{X \text{ M.wt } H_2O + \text{M.wt } BaCl_2}{\text{M.wt } BaCl_2} = \frac{\text{wt. } BaCl_2.2H_2O}{\text{wt. } BaCl_2}$$

X = nomber of H_2O

Discussion:

1. Discuss kind of water in chemical compounds with an example for each type.
2. what is the purpose of using desscator.
3. Calculate theoretical percentage of water in each compounds:

- $Na_2S_2O_3.5H_2O$.
- $MgSO_4.7H_2O$.
- 1,10-Phenanthroline monohydrate.

References:

Name of experiment:

Gravimetric determination of chloride in a soluble sample.

Number of experiment: - 2 -

The purpose of experiment: To determine the amount of Cl^- in a soluble sample

Equipment: filtering crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

Materials: chloride (unknown), silver nitrate, nitric acid, distilled water

Theory: The chloride ion is formed when the element chlorine, a halogen, gains an electron to form an anion (negatively-charged ion) Cl^-. The salts of hydrochloric acid HCl contain chloride ions and can also be called chlorides. The chloride ion, and its salts such as sodium chloride, are very soluble in water. It is an essential electrolyte located in all body fluids responsible for maintaining acid/base balance, transmitting nerve impulses and regulating fluid in and out of cells.

Chloride ion

The chloride content of a soluble salt can be determined by precipitation of the chloride anion as silver chloride according to the reaction:

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

If fully precipitated, the mass of the chloride (wt of Cl) or volume in the AgCl(s) precipitate (wt of AgCl) will be equal to the mass of chloride or it concentration in the unknown sample (wt. or vol. of unk). From these data, the mass % of chloride or concentration of chloride in the unknown can be calculated using from :

$$\%Cl = \frac{\text{wt of AgCl}}{\text{wt. of unk.}} \times \frac{\text{A.wt of Cl}}{\text{M.wt of AgCl}} \times 100$$

or

$$C_{Cl} = \frac{\text{wt of }_{\text{AgCl}}}{\text{volume of unknown}} \times \frac{\text{A.wt of Cl}}{\text{M.wt of AgCl}} \times 1000$$

Where A.wt.Cl and M..wt AgCl are the molar masses of Cl and AgCl, respectively. The same if the sample of chloride you have was liquid just you need the volume of unknown instead of weight. Experimentally, the AgCl precipitate is collected in a weighed filtering crucible and washed; its weight is determined after it has been dried to constant weight at 110Cº. The solution containing the sample is kept somewhat acidic during the precipitation to eliminate possible interference from anions of weak acids (such as CO_3^{2-} & PO_4^{-3}):

$$\begin{array}{lr} Ag_2CO_3 \longrightarrow 2Ag^{+} + CO_3^{=} & \\ Ag_3PO_4 \longrightarrow 3Ag^{+} + PO_3^{-3} & \text{At pH} < 7 \end{array}$$

$$\begin{array}{lr} 2Ag^{+} + CO_3^{=} \longrightarrow Ag_2CO_3 & \text{At pH} \geqslant 7 \\ 3Ag^{+} + PO_3^{-3} \longrightarrow Ag_3PO_4 & \end{array}$$

That form sparingly soluble silver salts in a neutral environment. A moderate excess of silver ion is needed to diminish the solubility of silver chloride, but an excess is avoided to minimize co-precipitation of silver nitrate.

Silver chloride forms first as a colloid and is subsequently coagulated with heat. Nitric acid and the small excess of silver nitrate promote coagulation by providing a moderately high electrolyte concentration. Nitric acid in the wash solution maintains the electrolyte concentration and eliminates the possibility of peptization during the washing step; the acid subsequently decomposes to give volatile products when the precipitate is dried. In common with other silver halides, finely divided silver chloride undergoes photodecomposition:

$$2\ AgCl_{(s)} \longrightarrow 2\ Ag_{(s)} + Cl_{2\ (g)}$$

The elemental silver produced in this reaction is responsible for the violet color that develops in the precipitate. In principle, this reaction leads to low results for chloride ion. In practice, however, its effect is negligible provided direct and prolonged exposure to sunlight is avoided. If photodecomposition of silver chloride occurs before filtration, the additional reaction:

$$3\ Cl_{2\ (g)} + 3\ H_2O_{(l)} + 5\ Ag^+_{(aq)} \longrightarrow 5\ AgCl_{(s)} + ClO_3{}^-_{(aq)}$$

tends to cause high results. Some photodecomposition of silver chloride is inevitable as the analysis is ordinarily performed. It is worthwhile to minimize exposure of the solid to intense sources of light as far as possible.

Iodide, bromide, and thiocyanate, if present, precipitate along with silver chloride and may cause high results. Additional interference can be expected from tin and antimony, which are likely to precipitate as oxychlorides under the conditions of the analysis.

Procedure:

1. Take from oven a clean and dry crucible, then weighted it after it has been cooled.
2. Transferred 10 ml of your unknown using a clean pipette.
3. place the unknown directly into a clean 250 ml beaker.
4. Label the beaker “any symbol” with a pencil and record the sample volume and the beaker number on your report sheet.
5. Add between 25 and 50 ml of distilled water and 1 ml of 6 M HNO_3 to the beaker.
6. Stir of the solutions with glass stirring rods until all of the sample has dissolved. ***Leave the stirring rods in the beakers.***
7. While stirring of the solutions add to it about 30 ml of 0.1N $AgNO_3$ solution.
8. Place a watch glass over the beaker.

9. Warm the solution gently with your water bath. And keep it warm for approximately 30 minutes (this can be done using either a Bunsen burner or a hot place). ***<u>Do not boil the solution</u>*!**

10. Completely and quantitatively transfer the precipitate and all the warm solution form the beaker onto the crucible using a rubber policeman and a wash bottle to wash out the last traces of precipitate.

11. Wash the precipitate on the filter paper with two or three ml portions of 0.1 N HNO_3 .

12. Finally, Remove the crucible and dry it in an oven for at least 30 minutes.

13. Cooled the crucible with precipitate in desiccator.

14. Weighted the crucible with precipitate and calculate the concentration of chloride in your original sample. The precipitated AgCl must be kept out of bright light because it is photosensitive and slowly decomposes in the presence of light as follows:

$$2\ AgCl_{(s)} \xrightarrow{hv} 2\ Ag_{(s)} + Cl_{2\ (g)}$$

<u>Results and calculations:</u>

$$\%Cl = \frac{\text{mass of AgCl}}{\text{mass of unk.}} \times \frac{\text{A.wt of Cl}}{\text{M.wt of AgCl}} \times 100$$

<u>or</u>

$$C_{Cl} = \frac{\text{wt of }_{AgCl}}{\text{volume of unknown}} \times \frac{\text{A.wt of Cl}}{\text{M.wt of AgCl}} \times 1000$$

<u>Discussion:</u>

1. In this experiment you avoid to using sulphuric acid & hydrochloric acid. Why?
2. Nitric acid is very important in this experiment. Why?
3. Can you apply this experiment to calculate concentration of chloride in tap water?
4. Depending on this experiment, can you determine the purity of food's salt?
5. What are –ve & +ve errors in this experiment?
6. Can you increase the selectivity of silver nitrate?

References:

***Name of experiment*:**

Gravimetric Determination of Nickel

***Number of experiment*: - 3-**

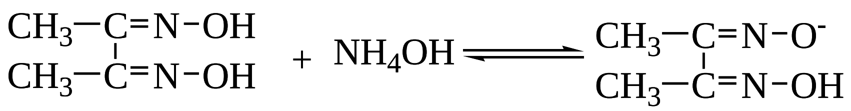
The purpose of experiment: To determine the amount of Ni .

Equipment: filtering crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

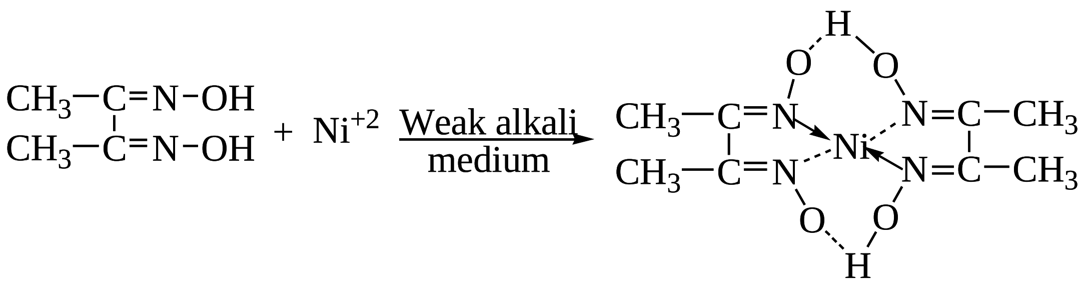
Theory:

Nickel(II) forms a precipitate with the organic compound dimethylglyoxime, $C_4H_6(NOH)_2$. Dimethylglyoxime is used as a chelating agent in the gravimetric analysis of nickel. The use of DMG as a reagent to detect nickel was discovered by L. A. Chugaev in 1905. For qualitative analysis, $DMGH_2$ is often used as a solution in ethanol. It is the conjugate base, not $DMGH_2$ itself, that forms the complexes. Furthermore, a pair of $DMGH^-$ ligands are joined through hydrogen bonds to give a macrocyclic ligand. The most famous complex is the bright red $Ni(DMGH)_2$, formed by treatment of Ni(II) sources with $DMGH_2$. This planar complex is very poorly soluble and so precipitates from solution. This method is used for the gravimetric determination of nickel, e.g. in ore

The formation of the red chelate occurs quantitatively in a solution in which the pH is buffered in the range of 5 to 9. The chelation reaction that occurs is illustrated below.



$$\begin{array}{l} CH_3-C=N-OH \\ CH_3-C=N-OH \end{array} + NH_4OH \rightleftharpoons \begin{array}{l} CH_3-C=N-O^- \\ CH_3-C=N-OH \end{array}$$



Although the loss of one proton occurs from one oxime group (NOH) on each of the two molecules of dimethylglyoxime, the chelation reaction occurs due to donation of the electron pairs on the four nitrogen atoms, not by electrons on the oxygen atoms. The reaction is performed in a solution buffered by either an ammonia or citrate buffer to prevent the pH of the solution from falling below 5. If the pH does become too low the equilibrium of the above reaction favors the formation of the nickel(II) ion, causing the dissolution of $Ni(DMG)_2$ back into the mother liquor.

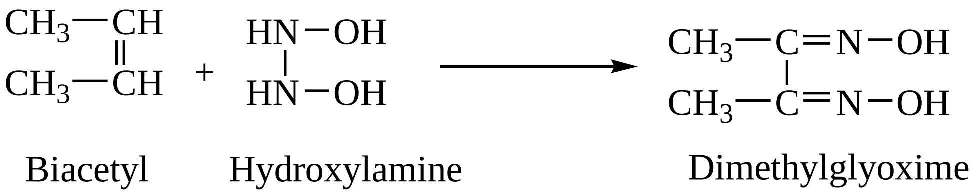
Adding tartarate or citrate ions before the precipitation of the red nickel complex prevents interference from Cr, Fe and other metals. These anions selectively form tightly bound soluble complexes with the metals and prevent the formation of insoluble metal hydroxides in the buffered solution.

An alcoholic solution of dimethyglyoxime (DMG) is used as the precipitating reagent during the experiment because DMG is only slightly soluble in water (0.063 g in 100 ml at 25 °C). It is therefore crucial to avoid the addition of too large an excess of the reagent because it may crystallize out with the chelate. It is also important to know that the complex itself is slightly soluble to some extent in alcoholic solutions. By keeping the volume added of the chelating reagent small, the errors from these sources are minimized. The amount of the reagent added is also governed by the presence of other metals such as cobalt, which form soluble complexes with the reagent. If a high quantity of these ions is present, a greater amount of DMG must be added.

The nickel dimethylglyoximate is a precipitate that is very bulky in character. Therefore, the sample weight used in the analysis must be carefully controlled to allow more convenient handling of the precipitate during transferral to the filtering crucible. To improve the compactness of the precipitate, homogeneous precipitation is often performed in the analytical scheme. This is accomplished by the adjustment of the pH to 3 or 4, followed by the addition of urea. The solution is heated to cause the generation of ammonia by the hydrolysis of the added urea, as indicated by the following reaction.

$$NH_2CONH_2 + H_2O \longrightarrow 2\ NH_3 + CO_2$$

Or by adding both of biacetyl and hydroxylamine to form dimethylgloxime gradually:



A slow increase in the concentration of ammonia in the solution causes the pH to rise slowly and results in the gradual precipitation of the complex. The result is the formation of a more dense, easily handled precipitate. Once the filtrate has been collected and dried, the nickel content of the solution is calculated stoichiometrically from the weight of the precipitate.

Procedure:

1. Quantitatively transfer the 10 ml of samples to 400 ml beakers and dilute to 150 ml with distilled water.
2. Add 1:1 ammonium hydroxide slowly until the solution is alkaline **(IN THE HOO**D). The final solution should smell distinctly of ammonia after blowing away the vapors from above the solution and its color will turn from a light green to aqua. If the addition of ammonia causes any precipitate to form, dissolve it by adding hydrochloric acid, solution, and neutralize again with ammonia.
3. Make the samples slightly acidic with hydrochloric acid (pH = 5). Add 15 ml of 1% dimethylglyoxime and heat for about an 15 min. at 80 - 85 degrees Celsius. If a red precipitate does not start forming after 5 minutes, add urea. Do not allow the solutions to heat to boiling. If the solutions begin to boil, remove them from the heat source and allow them to cool below boiling before continuing to heat on a lower setting.
4. Cool to room temperature during the lab period and check the pH with pH paper. Use the pH paper in a frugal and proper manner (see your instructor for the proper manner to use it). If the solution is not above pH 7, add a drop of ammonium hydroxide and check again. Filter the solution through one of your previously weighed crucibles.
5. Test the filtrate on all samples for the completeness of precipitation by adding a little more dimethylglyoxime (2 - 3 ml). If any red precipitate forms, reheat the solution on the hot plate after raising the pH to 7. When you are finished filtering, was the precipitate with cold water to which a few drops of ammonia have been

added (do not used distilled water without this precautionary measure). Continue washes until the washings are free form chloride ion (use acidic $AgNO_3$).

6. Dry the precipitate in the oven at 130 o C to constant weight.
7. Calculate your results and report them as concen. of Ni in the sample.
8. Clean the crucibles by dissolving the precipitate with ~3 ml of 6M hydrochloric acid. Precipitate that is adhered to the sides of the crucible can be dissolved by rolling the crucible so that the 6M HCl solution coats the crucible walls.

Results and calculations:

$$C_{Ni^{+2}} = \frac{\text{wt. of Ni(DMG)}_2}{\text{volume of unknown}} \times \frac{\text{A.wt of Ni}}{\text{M.wt of Ni(DMG)}_2} \times 1000$$

Discussion:

1. Why should the crucible be cooled before weighing?

2. Define: homogenous precipitation, then can you use it for determine nickel. How?

References:

Name of experiment **:**

Gravimetric Analysis of a Soluble Carbonate

Number of experiment: - 4 -

The purpose of experiment: To determine the amount of CO_3^{-2} gravimetrically .

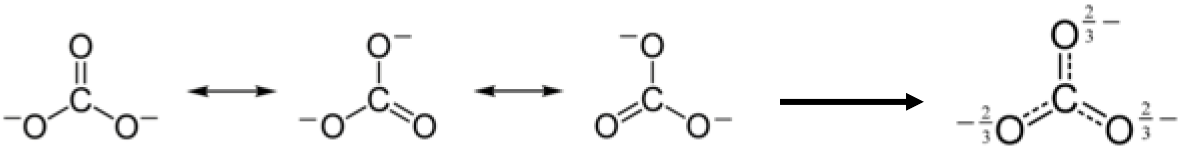
Equipment: filtering crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, analytical balance, tongue.

Materials: salt of carbonate, calcium chloride.

Theory:

In chemistry, a carbonate is a salt of carbonic acid, characterized by the presence of the carbonate ion, CO^{2-}. The name may also mean an ester of carbonic acid, an organic compound containing the carbonate group $C(=O)(O–)_2$. The term is also used as a verb, to describe carbonation: the process of raising the concentrations of carbonate and bicarbonate ions in water to produce carbonated water and other carbonated beverages — either by the addition of carbon dioxide gas under pressure, or by dissolving carbonate or bicarbonate salts into the water.

The symmetry can be achieved by a resonance between three structures



In geology and mineralogy, the term "carbonate" can refer both to carbonate minerals and carbonate rock (which is made of chiefly carbonate minerals), and both are dominated by the carbonate ion, CO^{2-}. Carbonate minerals are extremely varied and ubiquitous in chemically-precipitated sedimentary rock.

The most common are calcite or calcium carbonate, $CaCO_3$, the chief constituent of limestone (as well as the main component of mollusc shells and coral skeletons); dolomite, a calcium-magnesium carbonate $CaMg(CO_3)_2$; and siderite, or iron (II) carbonate, $FeCO_3$, an important iron ore. Sodium carbonate ("soda" or "natron") and potassium carbonate ("potash") have been used since antiquity for cleaning and preservation, as well as for the manufacture of glass. Carbonates are widely used in industry, e.g. in iron smelting, as a raw material for Portland cement and lime manufacture, in the composition of ceramic glazes, and more.

The ***quantitative analysis*** of substances and mixtures is a major part of chemistry. It has provided us with our knowledge of the compositions of chemical substances, and is used to determine the formulas of new substances. Quantitative analysis is also widely used to determine the composition of mixtures, such as the concentration of a pollutant in a water sample, or the percentage of a metal in a ore sample. The simplest way to measure the quantity of a substance is to weigh it.

There are therefore many analytical techniques, collectively know as Gravimetric Analysis, based on weighing. For each analysis, a method must be devised so that the substance of interest can be quantitatively isolated (i.e. all of it) in a pure form, or as part of a compound of known composition. In this experiment, carbonate ions from a soluble metal carbonate will be quantitatively isolated as $CaCO_3$(s) by precipitation and filtration.

$$M_2CO_{3(aq)} + CaCl_{2(aq)} \longrightarrow CaCO_{3(s)} + 2\ MCl_{(aq)}$$

The number of moles of carbonate ions in the original sample can be calculated from the mass of $CaCO_3$ obtained. The $CaCO_3$ precipitate must be collected quantitatively. Any loss of precipitate (e.g. left in the beaker or on the stirring rod) will result in an error in your analysis.

Procedure:

1. Obtain a clean dry sintered glass crucible from the oven. Place it in a desiccator and when it has cooled to room temperature (about 20 min.) make an identifying mark in pencil on the ground glass area on the side of the crucible. Then weigh it on the analytical balance.
2. Record the sample code of the alkali metal carbonate (M_2CO_3) assigned to you, and then weigh about 0.5 g of this compound into a weighing vial.
3. Weigh the vial with the metal carbonate in it on the analytical balance. Tip the contents of the vial into a 400 ml beaker. Re-weigh the vial on the same analytical balance.
4. Dissolve the metal carbonate in about 125 ml of deionized water. Do not lose any of this solution (e.g., on a glass rod used for stirring) or the final result will be

inaccurate. If the carbonate does not dissolve completely, warm the solution, crush any crystals and stir to aid dissolving.

5. Warm the solution until it begins to steam but do not boil it. Slowly add and stir about 30 ml of 0.5 mol· l^{-1} calcium chloride to the solution. This should provide an excess of calcium ions for the precipitation. Periodically stir the mixture for about 5 minutes. This will cause the calcium carbonate precipitate to coagulate and will make the filtration easier.
6. Set up your filtration apparatus and turn on the aspirator.
7. Transfer the precipitate to the pre-weighed sintered glass crucible using your glass rod to direct the flow into the crucible. Careful work is essential at this stage.
8. Wash the precipitate in the crucible with 3-5 times of distilled water to remove all traces of soluble salts.
9. Finally, wash with methanol to remove water.
10. Place the crucible in the oven and dry it for 25 minutes at about 120°C - longer if there is a lot of traffic at the oven (i.e. if the oven door is being opened and closed often).
11. Do the calculations and answer all questions for this lab in your Lab Report.

Results and calculations:

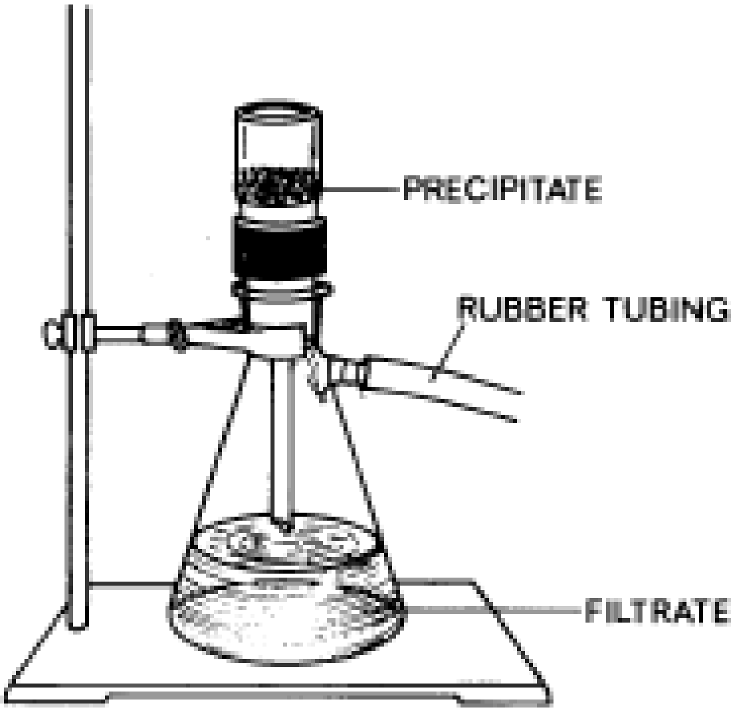
$$\%CO_3 = \frac{\text{wt of } CaCO_3}{\text{wt. of unk.}} \times \frac{\text{M.wt. of } CO_3^{-2}}{\text{M.wt of } CaCO_3} \times 100$$

Discussion:

Explain clearly:

1. What sources of error in this experiment might lead to a high value for the amount of $CaCO_3$(s) collected?.

2. What sources of error in this experiment might lead to a low value for the amount of CaCO3(s)?



References:

Name of experiment:

Gravimetric Determination of Calcium

Number of experiment: - 5-

The purpose of experiment: to calculate amount of calcium ion in different unknown samples.

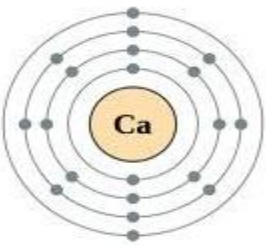
Equipment:

Filtering josh crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue, ashless filter paper.

Materials: unknown calcium solution, distilled water, hydrochloric acid, methyl red, ammonium oxalate, ammonia solution, sulphuric acid, potassium permanganate.

Theory:

Calcium is the chemical element with the symbol Ca and atomic number 20. It has an atomic mass of 40.078 amu. Calcium is a soft gray alkaline earth metal, and is the fifth-most-abundant element by mass in the Earth's crust. Calcium is also the fifth-most-abundant dissolved ion in seawater by both molarity and mass, after sodium, chloride, magnesium, and sulfate. Calcium is essential for living organisms, in particular in cell physiology, where movement of the calcium ion Ca^{2+} into and out of the cytoplasm functions as a signal for many cellular processes. As a major material used in mineralization of bones and shells, calcium is the most abundant metal by mass in many animals.



Calcium ion can be analyzed by precipitation with oxalate in basic solution to form $CaC_2O_4.H_2O$. The precipitate is soluble in acidic solution because the oxalate anion is a weak base. Large, easily filtered, relatively pure crystals of product will be obtained if the precipitation is carried out slowly. This can be done by dissolving Ca^{2+} and $C_2O_4^{2-}$ in acidic solution and gradually raising the pH by thermal decomposition of urea or by adding ammonia. Finally calcium can be precipitate gravimetrically in different ways depending on temperature as follow:

$$Ca^{+2} + C_2O_4^{-2} + H_2O \xrightarrow[25\ ^{o}C]{HCl} CaC_2O_4.H_2O$$

$$CaC_2O_4.H_2O \xrightarrow{100\ ^{o}C} CaC_2O_4 + H_2O$$

$$CaC_2O_4 \xrightarrow{475 - 525\ ^{o}C} CaCO_3 + CO$$

$$CaCO_3 \xrightarrow{1000\ ^{o}C} CaO + CO_2$$

Also calcium can be determined gravimetrically as calcium sulphate through the reaction between hydrated calcium oxalate and sulphuric acid as below:

$$CaC_2O_4 + H_2SO_4 \longrightarrow CaSO_4 + H_2C_2O_4$$

In this experiment calcium can be determined gravimetrically by weighing the precipitate after filtrate and dry it, or calcium can be determined volumetrically by dissolved the precipitate by sulphuric acid and titrate the production (oxalic acid) against potassium permanganate, when the colour is change to colourless, record the volume and calculate the concentration : $N_1 V_1 = N_2 V_2$

Important calcium compounds:

1. Calcium oxalate CaC_2O_4: Solid, salty, insoluble in water (6.7 ppm at 25 ◦C), it solubility was increased in acidic medium (14 ppm at 25 ◦C).

$$CaC_2O_4 + H^+ \rightleftharpoons HC_2O_4^- + Ca^{+2}$$

$$HC_2O_4^- + H^+ \rightleftharpoons H_2C_2O_4$$

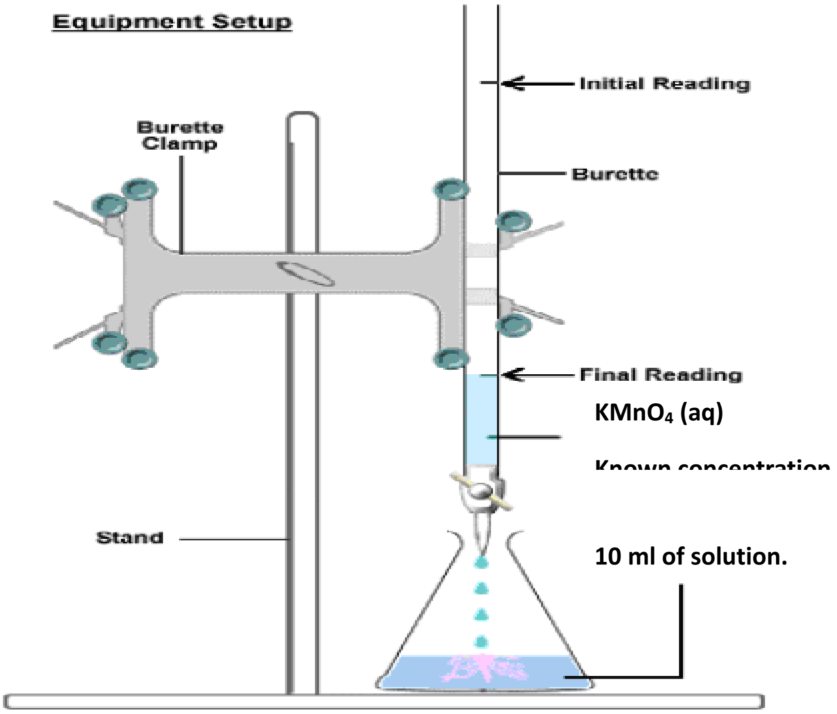
2. Calcium carbonate $CaCO_3$: white powdered precipitate, its crystal has to shape one of them is stable shape called galacite hexagonal (calcium atom was rounded by six oxygen atoms), the second shape was semi-stable which called aragonite

3. Calcium oxide CaO: white precipitate, and drying factor because its ability to absorbed water (therefore it used in desiccator) and react with water to produced calcium hydroxide:

$$CaO + H_2O \longrightarrow Ca(OH)_2 \qquad \Delta H = -76\ KJ$$

Procedure:

1. Dry josh crucible in an oven for 10 min. then cool it for 10 min. use diseccator for this purpose.
2. Use a few small portions of unknown to rinse a 25-ml transfer pipet, and discard the washings. Transfer exactly 25 ml of unknown to 400-ml beaker, and dilute with 1 ml of (1:1) HCl. Add 5 drops of methyl red indicator solution to each beaker. This indicator is red below pH 4.8 and yellow above pH 6.0.
3. Add ~25 ml of ammonium oxalate solution to each beaker while stirring with a glass rod. Remove the rod and rinse it into the beaker. Make the solution nutral to weak basic by adding drop by drop ammonia solution (or adding ~15 g of solid urea to the sample, cover it with a watchglass, and boil gently for ~30 min until the indicator turns yellow).
4. leave it for digestion 30 min.
5. Filter your solution by dry, clean and weighing josh crucible and wash it with cold distilled water or 0.1% ammonium oxalate.
6. dry it at 100◦**C**, cool it in dessecator, then calculate the concentration of calcium in the original sample.
7. Dissolve your precipitate in 50 ml hot sulphuric acid use 100 ml volumetric flask. Transferred 10 ml of this solution to conical flask and titrate it against potassium permanganate **keep the solution at 60 ◦C** until the colour is change to colourless, record the volume and repeat the titration step three times and take the average.
8. calculate the concentration of calcium gravimetrically and volumetrically.



Results and calculations:

1. $$C_{Ca^{+2}} = \frac{\text{wt. of Precipitate}}{\text{volume of unknown}} \times \frac{\text{A.wt of Ca}}{\text{M.wt of pricipitate}} \times 1000$$

2. $N_1 V_1 = N_2 V_2$ Then $N = \frac{\text{wt} \times 1000}{\text{Eq.wt} \times V(\text{ml})}$

Discussion:

1. why you need to heat the solution before titrate it against permanganate.
2. what did you add instead of base.
3. can you use distilled water as washing solution for precipitate? Why?
4. give 2 examples about aragonite and galacite?
5. define the solution which results from mixed calcium and sodium hydroxides?

References:

Name of experiment:

Assay of $SO_3^{=}$ by Gravimetric Analysis of $SO_4^{=}$

Number of experiment: - 6-

The purpose of experiment: assay of sulphite.

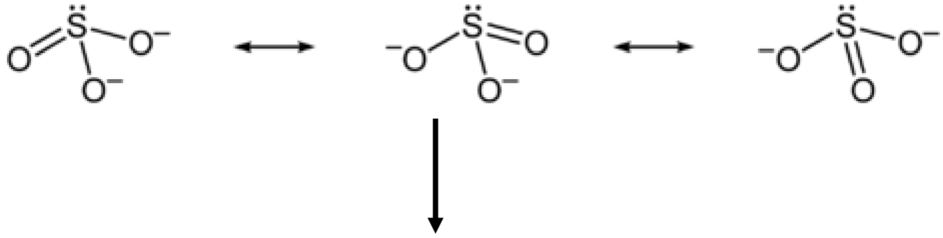
Materials: Sulphit solution, barium chloride solution, hydrochloric acid solution, nitric acid solution, silver nitrate solution, distilled water.

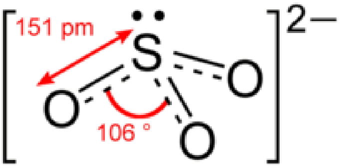
Equipment:

porcelain filter crucibles, 25-ml graduated cylinder, polyethylene wash bottle with distilled water, 150-ml beakers, stirring rods, dessicator with Drierite desiccant, filtration apparatus (500-ml filter flask, Walters adapter, 3-ft section of vacuum tubing)

Theory:

Sulphit ion SO_3^{-2}, The structure of the sulfite anion can be described with three equivalent resonance structures. In each resonance structure, the sulfur atom is double-bonded to one oxygen atom with a formal charge of zero (neutral), and sulfur is singly bonded to the other two oxygen atoms, which each carry a formal charge of −1, together accounting for the −2 charge on the anion. There is also a non-bonded lone pair on the sulfur, so the structure predicted by VSEPR theory is trigonal pyramidal, as in ammonia (NH_3). In the hybrid resonance structure, the S-O bonds are equivalently of bond order one and one-third.





Inorganic and organically-bound sulfur may be determined in a sample by conversion of all S to the SO_4^{2-} ion. Usually, this step is accomplished by ignition of the sample in oxygen, in a closed environment. Once the conversion of sulfur to sulfate has been accomplished, the sulfate can be conveniently measured by gravimetry. In this experiment, we will analyze an inorganic sulfate to avoid the need for any combustion step, which is difficult and dangerous for beginners to attempt. The most common precipitant for sulfate is the barium ion. The reaction is:

$$SO_4^{-2} + Ba^{+2} \longrightarrow BaSO_4$$

This apparently simple gravimetric determination has a few difficulties. The most significant difficulty with collecting and weighing $BaSO_4$ is the tendency for $BaSO_4$ to coprecipitate other species, especially those containing potassium and iron (III) cations. rapid precipitation from hot solution of the sample, something not usually done because it produces small particles, is advised here to minimize - but not eliminate - the coprecipitation of these cations. Another difficulty is that $BaSO_4$ has no good solvents, and it cannot be redissolved, and reprecipitated. If we could dissolve it, we could purify the precipitate by repeating the precipitation in a solution with less of the ions likely to coprecipitate.

The lack of good solvents causes another problem. The finely divided crystals of $BaSO_4$ produced must be digested to make particles large enough for collection on filter-glass mats. Filtering crucibles cannot be used, as the $BaSO_4$ cannot be removed from the crucible by any solvent. Instead, after filtering and washing the digested precipitate, the filter paper mat must be charred carefully, then ashed to remove the paper so that it does not contribute to the weight of the precipitate. **$BaSO_4$ is stable to very high heats,** and heating with a Bunsen burner has no effect on it.

In this experiment, we will report the amount of sulfur present not as %S or $\%SO_4^{2-}$ but as $\%SO_3$. This is known as an assay. In an assay, we measure one material and infer from that measurement the amount of another material. An assay presumes a stoichiometric relation between what is measured and what is sought. Here, $BaSO_4$ is measured, but the weight percent (written as %(w/w) to indicate that this is a percentage by weight of the solid) of SO_3 is sought. There is a 1:1 stoichiometric relationship between SO_3 and $BaSO_4$, so these two species are related by the gravimetric factor:

$$\%SO_3 = \frac{\text{wt of } BaSO_4}{\text{wt. of Sample}} \times \frac{\text{M.wt. of } SO_3^{-2}}{\text{M.wt of } BaSO_4} \times 100$$

where M.wt SO_3 / M.wt $BaSO_4$ is the gravimetric factor for reporting % SO_3 when one measures $BaSO_4$. A similar expression could be used to assay any other Scontainin species from measurements of the weight of $BaSO_4$. The procedure given below has shown to lead to the most accurate results for weights of $BaSO_4$.

The precipitate and filter paper are dried in a crucible. The filter paper is charred and ashed, leaving the dried sample in the crucible for ease in weighing. To reduce error, the paper ashing operation must be carried out with care.

The paper should be charred carefully at low temperature with the crucible lid in place so that escaping gases do not burst into flame. When smoke formation has ceased, the temperature is slowly increased and air is allowed ample access to the interior of the crucible. If this practice is not followed, carbon will reduce sulfate ion to sulfide ion, forming carbon monoxide:

$$BaSO_4 + 4\,C \longrightarrow BaS + 4\,CO$$

This problem should be treated by adding a little drops of sulphuric acid then heated the crucible to volatile sulphuric acid:

$$BaS + H_2SO_4 \longrightarrow BaSO_4 + H_2S$$

Also burning over than 800 ◦C produce barium oxide:

$$BaSO_4 \xrightarrow{800\ ^oC} BaO + SO_3\uparrow$$

Procedure:

1. Clean and mark porcelain filter crucible. Dry it in an oven for 10 min. then cool it for 10 min. use diseccator for this purpose**.**
2. Weigh out sample of the dried, cooled unknown to ±0.1 mg. This sample should weigh about 0.25 g, but anything in the range 0.2 to 0.3 g is acceptable.
3. Transfer sample to a 400-ml beaker, dissolve in 100 ml of distilled water, and then add 4 ml of 0.05 N HCl. Heat these solutions to near boiling.
4. Add 10 ml of 10% $BaCl_2$ and add it quickly with good stirring to the beaker containing unknown. Stir with a clean glass stirring rod. Leave the stirring rod in the solution.

Warning*: Soluble barium salts are* ***toxic!*** *Handle $BaCl_2$ carefully and wash your hands thoroughly when you complete this laboratory. Dispose of excess $BaCl_2$ as instructed.*

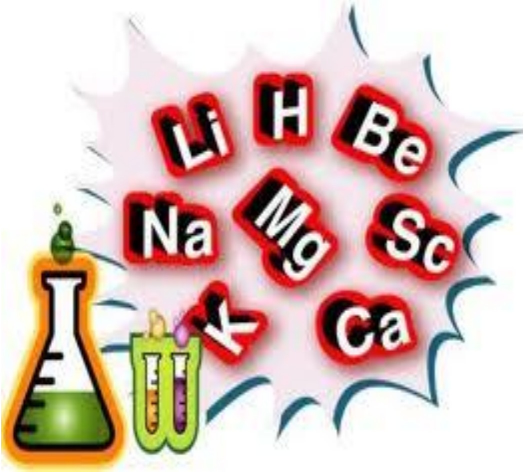
5. Heat to near boiling for 15 minutes then leave it also 15 minutes for digestion . Also heat about 50 ml of distilled water in a 150-ml beaker .
6. After allowing the solutions to cool to the point that the beaker can be safely handled, carefully filtrate it use ashless filter paper.
7. Wash the precipitate with three small (about 3 ml) portions of hot water. Acidify the filtrate with 2 drops of 8 M HNO_3.

Warning*: Even 8 M HNO3 is* ***corrosive****! Handle this acid very carefully, and avoid contact with your skin or clothes. If you do spill some, wash it off at once. Wash your hands carefully when you complete this laboratory.*

8. Check the filtrate for chloride ion by adding **1 drop** of 0.1 M $AgNO_3$ from the dropper bottle, then mixing. If the filtrate turns cloudy, chloride ion is present ($AgNO_3$ forms a white precipitate of AgCl when chloride is present) If the filtrate contains chloride, continue to wash the precipitate. Empty the filtrate containing the AgCl into the waste bottle provided, and rinse the 150-ml beaker with distilled water. The rinse may be discarded in the sink.

9. Repeat step 8 until the filtrate is clear or only very slightly cloudy upon addition of the AgNO3. Draw air through the filter for 1-2 minutes to dry the solid barium sulfate partially.
10. Heat the crucible for 5 minutes, then move it to the fernous at 800 ◦C for at least 15 minutes.
11. Cool it for 20 minutes the weight the crucible .Calculate the mean % SO_3 from your result.

Results and calculations:



$$\%SO_3 = \frac{\text{wt of } BaSO_4}{\text{wt. of Sample}} \times \frac{\text{M.wt. of } SO_3^{-2}}{\text{M.wt of } BaSO_4} \times 100$$

Discussion::

- Discuss the above procedure and
- Write about the biological important of sulphit and
- What is the relationship between sulphite and thiamine.

References:

Name of experiment:

Gravimetric determination of Phosphorus in Plant Food

***Number of experiment*:** - 7 -

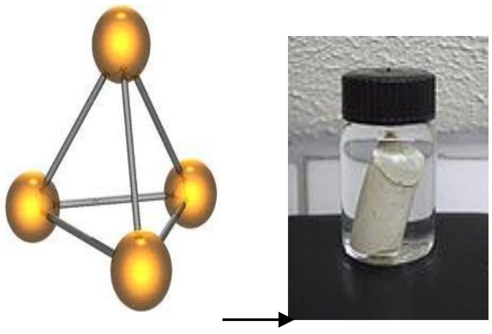
The purpose of experiment: determine the percentageof p in plant food.

Equipment: filtering crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

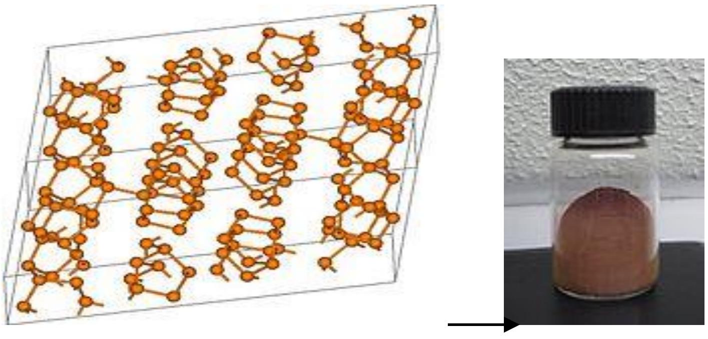
***Materials*:** ammonia solution, magnesium sulphate, plant food, distilled water.

Theory:

Phosphorus is the chemical element that has the symbol P and atomic number 15. A multivalent nonmetal of the nitrogen group, phosphorus as a mineral is almost always present in its maximally oxidized state, as inorganic phosphate rocks. Elemental phosphorus exists in two major forms—white phosphorus and red phosphorus—but due to its high reactivity, phosphorus is never found as a free element on Earth. The first form of elemental phosphorus to be produced (white phosphorus, in 1669) emits a faint glow upon exposure to oxygen – hence its name given from Greek mythology, Φωσφόρος meaning "light-bearer" (Latin *Lucifer*), referring to the "Morning Star", the planet Venus. Although the term "phosphorescence", meaning glow after illumination, derives from this property of phosphorus, the glow of phosphorus originates from oxidation of the white (but not red) phosphorus and should be called chemiluminescence



Crystal of white phosphorous



crystal of red phosphorous

Gravimetric analysis is a quantitative method of classical analysis. The element to be determined is isolated in a solid compound of known identity and definite composition. The mass of the element that was present in the original sample can be determined from the mass of this compound. Plant foods contain three essential nutrients that are not readily available from soils. These are soluble compounds of nitrogen, phosphorus, and potassium. A typical label on a plant food will have a set of numbers such as 15-30-15. These numbers mean that the plant food is guaranteed to contain at least 15% nitrogen, 30% phosphorus (expressed as P_2O_5) and 15 % potassium (expressed as K_2O). The remaining of the product is fillers, dyes and other anions and cations to balance the charge in the chemical compounds. In this experiment, we will illustrate a quality control analysis for the determination of phosphorus in plant food by gravimetric analysis. Phosphorus will be determined by precipitation of the insoluble salt magnesium ammonium phosphate hexahydrate according to the reaction:

$$5H_2O_{(l)} + HPO_4^{-2}{}_{(aq)} + NH_4^{+}{}_{(aq)} + Mg^{2+}{}_{(aq)} + OH^{-}{}_{(aq)} \longrightarrow MgNH_4PO_4.6H_2O_{(s)}$$

The % P and % P_2O_5 in the initial sample can be calculated from the mass of $MgNH_4PO_4.6H_2O$ obtained using the following method:

mass of $MgNH_4PO_4.6H_2O$ → moles of $MgNH_4PO_4.6H_2O$ → moles of P → mass of P %P →

and

mass of $MgNH_4PO_4.6H_2O$ → moles of $MgNH_4PO_4.6H_2O$ → moles of P → moles of P_2O_5 → mass of P_2O_5 → %P_2O_5

Procedure:

Prepare 2 samples per group. Rinse all your glassware thoroughly with water before using.

1. Weigh by difference, to the second decimal point, 3.00 to 3.50 g of your unknown sample using a weighing dish. Record the sample mass in your notebook. Remember to record all digits on the balance.

NOTICE: Closed toed shoes and goggles are REQUIRED for the lab

2. Transfer your sample to a 250-ml beaker and add 35 to 40 ml of distilled water and stir the mixture with a glass stirring rod to dissolve the sample. They may be a small amount of insoluble residue. If your sample does not dissolve completely, remove the insoluble material by filtration.
3. Add 45 ml of 10% $MgSO_4.7H_2O$ solution to the filtrate. Then add approximately 150 ml of 2*M* NH_3 slowly while stirring. A white precipitate of $MgNH_4PO_4.6H_2O$ will form. Allow the mixture to sit at room temperature for 15 minutes to complete the precipitation.
4. Prepare a vacuum filtration apparatus using a buchner funnel. Obtain one filter paper for each one of your samples, weigh them and record their mass in your notebook (label them with a pencil to be able to differentiate them later).
5. Wet the paper with distilled water to hold it in place in the funnel. Transfer all the solution and the precipitate from the beaker using a rubber policeman. Wash the precipitate with two or three 5-ml portions of distilled water. Do this by adding each portion to the beaker in which you did the precipitation to transfer any remaining precipitate; then pour over the solid in the funnel. Finally, pour two 10-mL portions of 75% isopropyl alcohol through the filter paper.
6. When all the liquid has gone through, disconnect the hose and turn the water off. Carefully remove the filter paper with the precipitate and place on a marked paper towel. Blot your samples dry with a second paper towel. Discard the filtrate down the sink.
7. When the $MgNH_4PO_4.6H_2O$ precipitate is thoroughly dry, weigh the filter papers with precipitate. Be careful not to lose any of the solid. Record their masses. Record your group data, this class data will be available for use in the discussion section.

Results and calculations:

$$\%P = \frac{\text{wt of Precipitate}}{\text{wt. of unk.}} \times \frac{\text{A.wt. of P}}{\text{M.wt of } MgNH_4PO_4.6H_2O} \times 100$$

$$\%P_2O_5 = \frac{\text{wt of Precipitate}}{\text{wt. of unk.}} \times \frac{\text{A.wt. of } P_2O_5}{\text{M.wt of } MgNH_4PO_4.6H_2O} \times 100$$

Discussion:

Compare the $\%P_2O_5$ that you calculated for your sample to the value of the label. Are they in good agreement? If they are not, explained what could have gone wrong in your procedure. How can we improve this analysis for future trials. Discuss any trends you see in the class data, how do your results compare?

Questions

1. The label on a plant food reads 23-19-17. What does this mean? What is the minimum percentage of potassium in this plant food? What is the minimum percentage of phosphorus in this plant food?

2. $MgNH_4PO_4.6H_2O$ has a solubility of 0.023g/100 ml in water. Suppose a 5.02- g sample were washed with 20 ml of water. What fraction of the $MgNH_4PO_4.6H_2O$ would be lost?

3. $MgNH_4PO_4.6H2O$ loses H_2O stepwise as it is heated. Between 40°C and 60°C the monohydrate ($MgNH_4PO_4.H_2O$) is formed and above 100°C the anhydrous material ($MgNH_4PO_4$) is formed. What are the phosphorus percentages of the monohydrate and anhydrous material?

4. Ignition of $MgNH_4PO_4.6H_2O$ produces NH_3, H_2O and magnesium pyrophosphate, $Mg_2P_2O_7$. Complete and balance the equation for this reaction. If 5.00 g of $MgNH_4PO_4.6H_2O$ are ignited, how many grams of $Mg_2P_2O_7$ would be formed?

$$MgNH_4PO_4.6H_2O\ (g) \xrightarrow{\text{heat}}$$

References:

Name of experiment:

Gravimetric determination of Magnesium.

Number of experiment: - 8-

The purpose of experiment: to determina the amount of magnesium gravimetrically.

Equipment: ignition crucible, glass rode, 400 ml beacker, 10 ml pipett, funnel, washing bottle, desiccator, tongue.

Materials: 6 M HCl ,methyl red indicator, $(NH_4)_2HPO_4$, NH_3, 10% v/v HCl.

Theory:

Magnesium is a chemical element with the symbol Mg, atomic number 12, and common oxidation number +2. It is an alkaline earth metal and the eighth most abundant element in the Earth's crust and ninth in the known universe as a whole. Magnesium is the fourth most common element in the Earth as a whole (behind iron, oxygen and silicon), making up 13% of the planet's mass and a large fraction of planet's mantle. The relative abundance of magnesium is related to the fact that it is easily built up in supernova stars from a sequential addition of three helium nuclei to carbon (which in turn is made from three helium nuclei). Due to magnesium ion's high solubility in water, it is the third most abundant element dissolved in seawater. Magnesium ions are sour to the taste, and in low concentrations help to impart a natural tartness to fresh mineral waters. Magnesium is the 11th most abundant element by mass in the human body; its ions are essential to all living cells, where they play a major role in manipulating important biological polyphosphate compounds like ATP, DNA, and RNA. Hundreds of enzymes thus require magnesium ions to function. Magnesium is also the metallic ion at the center of chlorophyll, and is thus a common additive to fertilizers. Magnesium compounds are used medicinally as common laxatives, antacids (e.g., milk of magnesia), and in a number of situations where stabilization of abnormal nerve excitation and blood vessel spasm is required (e.g., to treat eclampsia).

Magnesium is precipitated as $MgNH_4PO_4.6H_2O$ using $(NH_4)_2HPO_4$ as the precipitant. The precipitate's solubility in neutral solutions is relatively high (0.0065 g/100 ml in pure water at 10 ^{o}C), but it is much less soluble in the presence of dilute ammonia (0.0003 g/100 ml in 0.6 M NH_3). Because the precipitant is not selective, a preliminary separation of Mg^{2+} from potential interferents is necessary. Calcium, which is the most significant interferent, is removed by precipitating it as CaC_2O_4. The presence of excess ammonium salts from the precipitant, or the addition of too much ammonia leads to the formation of $Mg(NH_4)_4(PO_4)_2$, which forms $Mg(PO_3)_2$ after drying. The precipitate is isolated by filtering, using a rinse solution of dilute ammonia. After filtering, the precipitate is converted to $Mg_2P_2O_7$ and weighed.

Procedure:

1. Transfer a sample containing no more than 60 mg of Mg^{2+} into a 400-ml beaker.
2. Add 2–3 drops of methyl red indicator, and, if necessary, adjust the volume to 150 ml.
3. Acidify the solution with 6 M HCl and add 10 ml of 30% w/v $(NH_4)_2HPO_4$.
4. After cooling and with constant stirring, add concentrated NH_3 dropwise until the methyl red indicator turns yellow (pH > 6.3).
5. After stirring for 5 min, add 5 ml of concentrated NH_3 and continue stirring for an additional 10 min.
6. Allow the resulting solution and precipitate to stand overnight.
7. Isolate the precipitate by filtering through filter paper, rinsing with 5% v/v NH_3.
8. Dissolve the precipitate in 50 ml of 10% v/v HCl, and precipitate a second time following the same procedure.
9. After filtering, carefully remove the filter paper by charring.
10. Heat the precipitate at 500 ^{o}C until the residue is white, and then bring the precipitate to constant weight at 1100 ^{o}C.

Results and calculations:

$$\%Mg = \frac{\text{wt of Precipitate}}{\text{wt. of unk.}} \times \frac{\text{A.wt. of Mg}}{\text{M.wt of } Mg_2P_2O_7} \times 100$$

Questions

1. Why does the procedure call for a sample containing no more than 60 mg of Mg^{2+}? A 60-mg portion of Mg^{2+} generates approximately 600 mg of $MgNH_4PO_4{\bullet}6H_2O$. This is a substantial amount of precipitate. A larger quantity of precipitate may be difficult to filter and difficult to adequately rinse free of impurities.

2. Why is the solution acidified with HCl before adding the precipitant? The HCl ensures that $MgNH_4PO_4.6H_2O$ does not immediately precipitate when adding the precipitant. Because PO_4^{3-} is a weak base, the precipitate is soluble in a strongly acidic solution. If the precipitant is added under neutral or basic conditions (high *RSS*) the resulting precipitate consists of smaller, less pure particles. Increasing the pH by adding base allows the precipitate to form under more favorable (low *RSS*) conditions.

3. Why is the acid–base indicator methyl red added to the solution? The indicator's color change, which occurs at a pH of approximately 6.3, indicates when there is sufficient NH_3 to neutralize the HCl added at the beginning of the procedure. The amount of NH_3 is crucial to this procedure. If we add insufficient NH_3, then the solution is too acidic, which increases the precipitate's solubility and leads to a negative determinate error. If we add too much NH_3, the precipitate may contain traces of $Mg(NH_4)_4(PO_4)_2$, which, on drying, forms $Mg(PO_3)_2$ instead of $Mg_2P_2O_7$. This increases the mass of the ignited precipitate, giving a positive determinate error. After adding enough NH3 to neutralize the HCl, we add the additional 5 mL of NH_3 to quantitatively precipitate $MgNH_4PO_4.6H_2O$.

4. Explain why forming $Mg(PO_3)_2$ instead of $Mg_2P_2O_7$ increases the precipitate's mass? Each mole of $Mg_2P_2O_7$ contains two moles of phosphorous and each mole of $Mg(PO_3)_2$ contains only one mole of phosphorous A conservation of mass, therefore, requires that two moles of $Mg(PO_3)_2$ form in place of each mole of $Mg_2P_2O_7$. One mole of $Mg_2P_2O_7$ weighs 222.6 g. Two moles of $Mg(PO_3)_2$ weigh 364.5 g. Any replacement of $Mg_2P_2O_7$ with $Mg(PO_3)_2$ must increase the precipitate's mass.

5. What additional steps, beyond those discussed in questions 2 and 3, help improve the precipitate's purity? Two additional steps in the procedure help in forming a precipitate that is free of impurities: digestion and reprecipitation.

6. Why is the precipitate rinsed with a solution of 5% v/v NH_3? This is done for the same reason that precipitation is carried out in an ammonical solution; using dilute ammonia minimizes solubility losses when rinsing the precipitate.

Discussion:

1. Can you precipitate magnesium with another precipitate agent ? which medium you should used ?
2. What is the important biological of magnesium?
3. With the same precipitation agent of magnesium in this experiments , which element should be precipitated if it found in the magnesium solutions?

References:

Name of experiment:

Gravimetric determination of Zinc

Number of experiment: - 9-

The purpose of experiment:

Equipment: filtering crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

Materials: NH_3, M.O, CH_3COOH, 8-Hydroxy quinoline, D.W.

Theory:

Zinc or spelter (which may also refer to zinc alloys), is a metallic chemical element; it has the symbol Zn and atomic number 30. It is the first element in group 12 of the periodic table. Zinc is, in some respects, chemically similar to magnesium, because its ion is of similar size and its only common oxidation state is +2. Zinc is the 24th most abundant element in the Earth's crust and has five stable isotopes. The most common zinc ore is sphalerite (zinc blende), a zinc sulfide mineral. The largest mineable amounts are found in Australia, Asia, and the United States. Zinc is an essential mineral of "exceptional biologic and public health importance". Zinc deficiency affects about two billion people in the developing world and is associated with many diseases. In clayren it causes growth retardation, delayed sexual maturation, infection susceptibility, and diarrhea, contributing to the death of about 800,000 clayren worldwide per year. Enzymes with a zinc atom in the reactive center are widespread in biochemistry, such as alcohol dehydrogenase in humans. Consumption of excess zinc can cause ataxia, lethargy and copper deficiency.

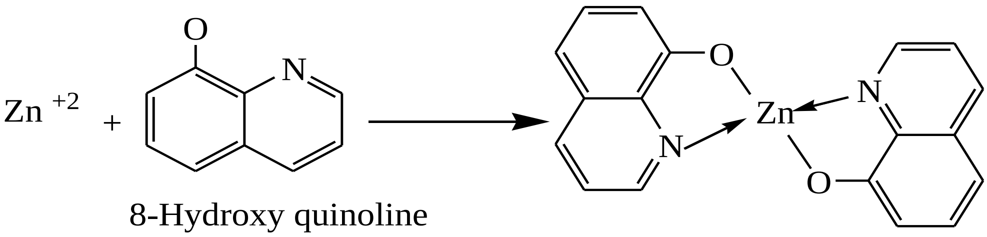
There is many different methods for gravimetric determination of zinc:

1. Quanilidin method $(Zn(C_{10}H_6O^2N)_2.H_2O$: this method is carry out in week acidic medium of acetic acid or in diluted medium of ammonia, the disadvantage of this method happened in acidic medium because of precipitating of other ions with zinc like ferrous, ferric, aluminium and titanium.

2. Pyridine method $[Zn(C_5H_5N)_2](SCN)_2$: this method is a simple and fast method, but pyridine is one of the poisoning materials also this method needs to a lot of washing solution which lead to loosing a part of precipitate as well as an error in final results.
3. Ammonium phosphate $(NH_4)_2HPO_4$: the disadvantages of this method are the interfering of ions with zinc like Be, Cd, and Co.
4. 8-Hydroxyquinolidine :It's an oldest, and , organic precipitant agent which depends on precipitating of almost metal ions with coordination numbers 4,6,8. The selectivity of precipitating agent have been controlled by pH, that means it's precipitate zinc, bismuth, cadmium and cupper in acidic medium. But in alkaline medium magnesium, lead, calcium and strontium had been precipitate which almost different of neutral medium because this precipitate agent have been precipitating of Mo, W, and V, While Al is precipitate in this medium with that ions so that adding tartarate solution to prevent the formation of aluminium hydroxide specially in alkaline medium.

Properties of precipitant agent:

It is a pell yellowish crystal , having a melting point about 72 C°. Also it is never solved in water but in hot ethanol and benzene, that made this precipitate agent prepare by dissolving of 5 gm in 12 ml of glacial acetic acid, this solution was stable for at least one weak. Then it can use to precipitating zinc in acidic medium (pH = 5.5) , then the precipitate agent had been dried between (130-145) C° as below:



Procedure:

1. Transfer a sample containing no more than 100 mg of Zn^{2+} into a 400-ml beaker.
2. Dissolve it in 30 ml of distilled water.
3. Add 3 drops of concentrated glacial acetic acid, 2 drops of methyl orange indicator (watch the solution coloure, it must be red).
4. Gradually added concentrated ammonia drop by drop until the solution colour was changed (be yellow = pH > 4.4). Then adding 2-3 drops of concentrated ammonia (pH = 5.5).

5. Adding 3 ml of precipitate agent **gradually** with constant stirring . then put your beaker in water bath of at least 15 min. at (60-80) C° , after that complete the digestion in lab. Temperature for 20 min.
6. Use josh crucible for filtrating your solution but not before washing and drying the crucible then cool it in a desicator. Wash the precipitate with hot distilled water the dry it between (130-145) C° in an oven.
7. Be sure of your correct calculations.

Results and calculations:

$$\% \text{ Zn} = \frac{\text{wt of Precipitate}}{\text{wt. of unk.}} \times \frac{\text{A.wt. of Zn}}{\text{M.wt of Zn(2-methyl oxinate)}} \times 100$$

Discussion:

1. What is the biological activity of zinc.
2. Why the compound of zinc was not coloured.
3. The produced precipitate can be drying but not ignition it? Why?

References:

Name of experiment:

Gravimetric Determination of Iron & Aluminium as oxides

Number of experiment: - 10-

Part – A : gravimetric assay of iron as Fe_2O_3 in presence of aluminium.
Part – B : gravimetric assay of aluminium as Al_2O_3 in presence of iron.

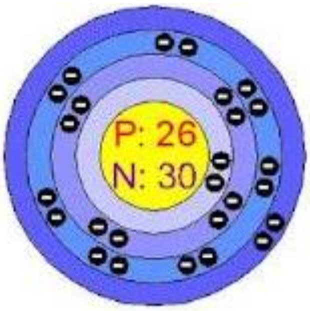
***The purpose of experiment*:** quantitative determination of both of Fe^{+3} & Al^{+3} in presence of each others.

Equipment: filtering crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

Theory:

Iron is a chemical element with the symbol Fe and atomic number 26. It is a metal in the first transition series. It is the most common element (by mass) forming the planet Earth as a whole, forming much of Earth's outer and inner core. It is the fourth most common element in the Earth's crust. Iron's very common presence in rocky planets like Earth is due to its abundant production as a result of fusion in high-mass stars, where the production of nickel-56 (which decays to iron) is the last nuclear fusion reaction that is exothermic. This allows radioactive nickel to become the last element to be produced before collapse of a supernova leads to events that scatters this precursor radionuclide of iron into space. Like other Group 8 elements, iron exists in a wide range of oxidation states, −2 to + 6, although +2 and +3 are the most common. Elemental iron occurs in meteoroids and other low oxygen environments, but is reactive to oxygen and water. Fresh iron surfaces appear lustrous silvery-gray, but oxidize in normal air to give iron oxides, also known as rust. Unlike many other metals which form passivating oxide layers, iron oxides occupy more volume than iron metal, and thus iron oxides flake off and expose fresh surfaces for corrosion.

Aluminium or aluminum is a silvery white member of the boron group of chemical elements. It has the symbol Al, and its atomic number is 13. It is not soluble in water under normal circumstances. Aluminium is the third most abundant element (after oxygen and silicon), and the most abundant metal, in the Earth's crust. It makes up about 8% by weight of the Earth's solid surface. Aluminium metal is too reactive chemically to occur natively. Instead, it is found combined in over 270 different minerals. The chief ore of aluminium is bauxite. Aluminium is remarkable for the metal's low density and for its ability to resist corrosion due to the phenomenon of passivation. Structural components made from aluminium and its alloys are vital to the aerospace industry and are important in other areas of transportation and structural materials. The most useful compounds of aluminium, at least on a weight basis, are the oxides and sulfates.



Iron

Aluminium

A sample containing iron can be analyzed by precipitation of the hydrated hydroxide from basic solution, followed by ignition to produce Fe_2O_3:

$$Fe^{3+}(aq) + 3OH^-(aq) + X\ H_2O(l) \longrightarrow Fe(OH)_3 .X\ H_2O(s)$$

$$Fe(OH)_3.X\ H_2O(s) \xrightarrow{900\ ^oC} Fe_2O_3\ (s)$$

While Aluminium is different in its assay about iron because the medium of precipitation must be neutral (pH = 7), followed by ignition (as iron) to produce Al_2O_3:

$$Al^{3+}(aq) + 3OH^-(aq) + X\ H_2O(l) \longrightarrow Al(OH)_3 .X\ H_2O(s)$$

$$Al(OH)_3.X\ H_2O(s) \xrightarrow{1000\ ^oC} Al_2O_3\ (s)$$

You will use this gravimetric technique to determine the weight percent of Fe & Al in an unknown sample.

Alminium is amphoteric in its reactions:

$$Al(OH)_3 + H^+ \rightleftharpoons Al^{+3} + H_2O$$

$$Al(OH)_3 + OH^- \rightleftharpoons AlO_2^- + H_2$$

For gravimetric determination of aluminium depending on the concept of precipitation method, aluminium reacts with precipitate agent which is (OH^-) forming a white-yellowish, colloidal precipitate with astructure of $Al(OH)_3$, dissolved in concentrated acids (pH >7), also the increased of precipitate agent dissolved it (pH <7), the medium of this reaction must be nutral (pH =7),

$$AlCl_3 + 3NH_4OH \xrightleftharpoons[\substack{100\ C^o \\ pH = 7}]{NH_4Cl} Al(OH)_3 + 3NH_4Cl$$

$$Al(OH)_3 \xrightleftharpoons[1000\ C^o]{\emptyset} Al_2O_3 + H_2O$$

Ammomium chloride was used for:

1. provide the reaction with ammonium ion which decrease the solubility of product precipitate.
2. it was formed with precipitate agent a buffer solution to control the pH of the solution at pH =7.
3. prevent the volatilization of microamount of aluminium element through the ignition.

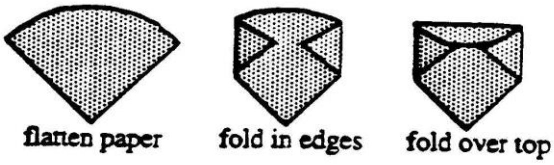
Notice: If you will make three replicate measurements, which will allow you to determine both an average and a 95% confidence interval for your data.

Procedure:

1. Bring porcelain crucibles and caps to constant weight by heating to redness for 15

min . Cool in a desiccator and weigh crucible.

2. Wash very clean 2 of 400 ml beacker , put in each one 10 ml of unknown solution, then add 25-30 ml of distilled water.
3. Labled one of them with Al symbol and the other with Fe, in order to recognized between them.
4. To Al beacker add 6 ml of 0.01 M of thiosulphate solution then, add 5 ml of ammonium chloride, followed by adding 2 drops of M.R indicator , heat to boiling.
5. add (1:1) ammonium hydroxide gradually with stirring, whatch the colour (it must be yellow).
6. use water bath for heat your solution for at least 15 min., the filtered your hot solution with ashless filter paper, wash your precipitate with washing solution of ammonium nitrate.
7. Carefully remove the filter paper from the funnel, taking care that no precipitate is lost.
8. Carefully lift the paper out of the funnel, fold it up (as shown below) and transfer to a porcelain crucible that has been brought to constant weight.



9. Dry the crucible cautiously in an oven for at least 10 min., then transferred it to furnous for at least 10 min.
10. After it is dry, cool the crucible briefly in air and then in a desiccator. Weigh the crucible the precipitate.
11. Calculate the weight percent of aluminium in each sample.
12. add to the beacker with Fe symbol 6 ml of 0.01 M acetate solution, then add 5 ml of 6 M HNO_3 to the solution, and boil for a few minutes to ensure that all iron is oxidized to Fe(III); **do this in the hood**.

1. Dilute the sample to 200 ml with distilled water and add *3 M* ammonium hydroxide with constant stirring until the solution is basic (pH indicator paper). Digest the precipitate by boiling for 5 min and allow it to settle. (The particle size increases as the solution is digested.)

2. Decant the supernatant liquid through coarse, ashless filter paper (Whatman #41). Try to retain most of the precipitate in the beaker. Test the filtrate for completeness of precipitation by adding a little 3 *M* ammonia to it.
3. Add 25-30 ml of 1% NH_4NO_3 to the beaker containing the precipitate. Heat and decant the hot solution through the same filter. Wash with the NH_4NO_3 solution once more. These washings contain cation impurities. Do not pour liquid higher than 1 cm from the top of the funnel.
4. Carefully remove the filter paper from the funnel, taking care that no precipitate is lost.
5. Carefully lift the paper out of the funnel, fold it up (as shown above) and transfer to a porcelain crucible that has been brought to constant weight.
6. Dry the crucible cautiously in an oven for at least 10 min.
7. After it is dry, char the filter paper by increasing the flame temperature (transferred it to furnous). The crucible should have free access to air to avoid reduction of iron by carbon.
8. Cool the crucible briefly in air and then in a desiccator. Weigh the crucible the precipitate.
9. Calculate the weight percent of iron in each sample.

Results and calculations:

$$\%Fe^{+3} = \frac{\text{wt of } Fe_2O_3}{\text{wt. of unk.}} \times \frac{\text{A.wt. of } Fe^{+3}}{\text{M.wt of } Fe_2O_3} \times 100$$

$$\%Al^{+3} = \frac{\text{wt of } Al_2O_3}{\text{wt. of unk.}} \times \frac{\text{A.wt. of } Al^{+3}}{\text{M.wt of } Al_2O_3} \times 100$$

***Discussion**: (**in your discussion answer these questions**)*

1. Why should the crucible be cooled before weighing?
2. What exactly happens to the filter paper during ignition?
3. Can you calculate the standard deviation, and the relative standard deviation of each ion.

4. How can you test for the completeness of precipitation? Why is this important?

References:

The cement industry

Introduction:

- The cement industry is of strategic industries, because it is directly related to the work of construction and reconstruction. The cement is used as a component of the Commonwealth of hydraulic mortar or concrete. With an industry so simple compared to the major industries, and depend on the availability of raw materials needed for that.
- The basic mixture for the cement industry consists mostly of the clay (Clay) and limestone (Limestone), which is heated at a temperature sufficient to induce the interaction between them for the production of calcium silicate (Calcium Silicate). And different pollutants from cement plants in terms of ingredients and quantity, depending on the different operations of the plant to another and hygiene procedures in place, and a variety of other factors.

- There are two basic ways in the cement industry: processes of dry and wet processes (Wet & Dry Processes). In wet processes grind the raw materials and mix with water, then separating the resulting suspension (Slurry) to the oven. In the dry process raw materials are dry prior to or during any grinding before you enter it to the oven.

Raw materials in the cement industry:

1- limestone.

2 - sand silicate.

3 - Clay.

4 - surface soils (Shale).

5 – A clay and bauxite dust (particles of medium-sized sedimentary layers such as the clay, and has a low content of water).

6 - oxides of iron: and the solvent used as catalysts or magma (Fluxing Agent) to reduce the temperature resulting from chemical reactions.

7 - Gypsum: Added in the final stage Bmills cement.

8 - other materials:

A - chemicals are used in the lab, to carry out analyzes and tests of quality control.

B - used oils and other lubricants for maintenance of the machines.

C - Use of natural gas and fuel oil to generate combustion gases are used in furnaces, ovens and be equipped so that you can burn more than one type of fuel.

Portland cement types and their characteristics:

A - Ordinary Portland Cement:

Used in construction works in general, and there are different varieties of this type, such as white cement, which contains a lower proportion of ferric oxide, and cement oil wells (Oil-Well Cement) used in the lining of oil wells, cement quick suspicion, and other varieties a multi-use particular.

B - Portland cement rigid in the high temperature-resistant and sulfate:

Is used in situations that require a moderate temperature hydrolysis, or in the construction of concrete exposed to the effects of sulfate medium.

C - a rapid hardening cement:

Different varieties of cement rapid hardening of the cement average of several aspects, including the proportion of limestone to the silicate and the proportion of silicate triple calcium in cement quick-setting are larger than those of ordinary cement. Also characterized by such a degree softer than ordinary cement, leading to the rapid sclerosis and generate a rapid heat.

Quick-setting cement is used in road construction.

D - portland cement low temperature:

Contains such a low sulfate and calcium aluminate tri tri calcium, leading to a reduction in heat generated. Ferric oxides are used to reduce the proportion of calcium aluminate trio, and thus increase the percentage of calcium aluminate four-iron in this type of cement.

E - sulfate-resistant cement:

This type of cement on the proportion of low-calcium aluminate three, and is characterized by a greater capacity to resist sulfate because of its components, or due to the processes used in industry, so it is used in situations that require high resistance to sulfate.

Production lines in the manufacture of cement:

First, the line of wet processes:

He took the use of dry processes in the cement industry to spread to gradually replace the wet processes, because of the abundance of energy which is characterized by dry processes, and accuracy in processes and in the control mixing of raw materials.

Production lines in the manufacture of cement:

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Main operations in this line:

A - crushing and mixing of raw materials:

Break the raw materials of limestone and silicate dust and a girl and superficial by the quarries, and the movement are heavily then, to be stored in the form of piles in open areas or covered.

B - milling:

Movement of raw materials in mills pendants (Slurry Mills), where mixed with water, and continue grinding until the suspense up to the required degree of smoothness. Conveys suspense after that to the storage silos, where it becomes homogeneous after setting the final of its components, taken from samples periodically to ensure matching fittings to specifications, then transported commentator to basins pendants (Slurry Basins), where the mills Hammers (Rotating Arms) by converting it to a homogeneous mixture.

C - the oven and cooled:

Commentator pulls from the bottom of ponds to feed the slot rotary furnace (Rotary Kiln). The rotary furnace is a long cylindrical furnace, lined with bricks from the inside heat, and slowly rotating about an axis inclined slightly from the horizontal plane. This allows the inclination to pay the contents of the oven during the rotation to the front. Generated at the front end (bottom) of the furnace combustion gases high heat flow to the upper part (rear) of the oven in a current opposite to the movement of the contents of the furnace surging down. The C to between 60 and°clinker is cooled (Clinker) formed by cooler air from 1400 .°200 m

D - the final grinding and packing:

Clinker is transferred to the mills Core (Ball Mills), where the gypsum is added and milled, and then packed in bags of paper or plastic.

Second, line dry processes:

Main operations in this line:

A - crushing and mixing of raw materials:

Break the raw materials of limestone and silicate dust and a girl and superficial by the quarries, and the movement are heavily then, to be stored in the form of piles in open areas or covered.

B - milling:

Is the introduction of raw materials in the dryer roundabout (Rotary Drier), where dried by hot air or exhaust resulting from the operation of the oven, then grind the raw materials in mills raw materials and transferred to storage silos before mixing (Pre-blending Silos) where they become homogeneous by air ROM, then transfer of heterogeneous raw material storage silos before mixing to storage silos. The process of mixing a 30% clay and 70% limestone.

C - the oven and cooled:

- Withdraw from heterogeneous raw material storage silos to the bottom of the slot feeding tower heating primary stages of a multi-, and height of the tower up to 120 meters, are equipped Bsakllonat calcination of the mixture, and ultimately into the rotary furnace .

- Uses natural gas or fuel oil as a source of thermal energy, also uses hot air for cooling the resulting clinker as an additional source of heat.

- Tends the oven little bit about the horizontal plane so as to allow slow motion of solids to the bottom, cut off the distance from the slot feeding at the top of the oven to the bottom, where the generated combustion gases high heat in a period of time ranging from hours to three hours, while moving gas combustion to the top of the stream opposite to the movement of solids. Operate to 900 m at°the hot combustion gases to heat the raw materials Tsgena initial the mouth of the furnace feed and provide the heat necessary for the decomposition of calcium carbonate. These The temperature of°gases then graduated from the top of the chimney at 400 m. the flame lighters emitted from about 2000 m .

- Used in dry processes stoves are shorter than those used in wet processes due to the lack of water use. Oven and rotor diameter (4-5 meters) tall and from 54 to 75 meters. And energy recovery boilers used in operations where dry exhaust temperature resulting from the operation of the furnace is greater than those generated in the wet processes, and use electrostatic precipitators (filters), electrostatic separation of particles from dust and portable ovens in the exhaust stream. Also used alumina bricks and Epsom (Magnesia Bricks) in the lining of the furnace, due to the exposure of the inner surface corrosion and chemicals at high temperatures in the region of the clinker.

- Consists of the final product (clinker) of granules ranging in size between 3 and 20 mm. Clinker and out of the oven enters the rotor cooling air to the sudden temperature drops , is also working on these refrigerants°rapidly to between 100 to 200 m exchange heat the combustion gases Tsgena preliminary.

- There are three types of refrigerant used in the clinker cooling: radiator Circle (Rotary Cooler), planetary cooler (Planetary Cooler) and the radiator network (Grate Cooler), and the latter is the species most used in the dry processes.

- Cause the dust in the oven in some problems, including: The accumulation and workshops in the cyclones and feeding slot and the sintering furnace (Claiming Zone), which may lead to stop the oven from work for several days. The evaporation of alkali consumed a great deal of energy.

* * *

D - the final grinding and packing:

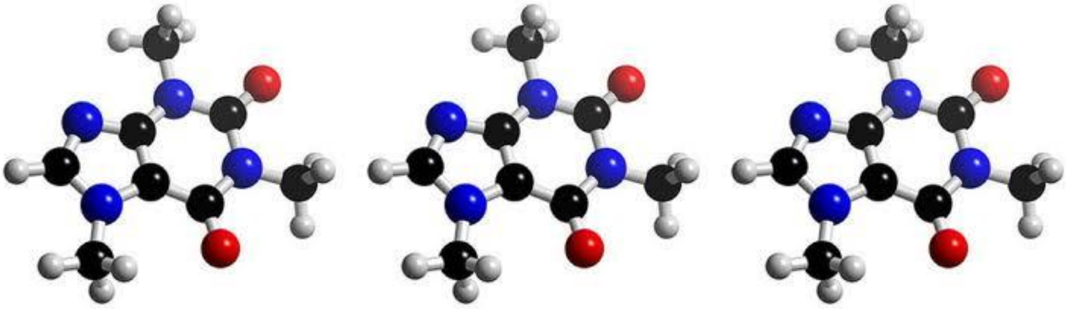
Clinker is transferred to the Core mills where gypsum is added to it and packed in bags of paper or plastic.

Third, the line of semi-dry processes:

- Operations semi-dry is a special case of processes dry, where it is used a furnace to urinate (Lepol Kiln) or oven supplier shaft (Shaft Kiln), and in both cases form the raw material grinding processes dry, in the form of granules ranging in diameter between 10 and 15 mm so that in addition to 13% of the water.

- If you use the oven to urinate dried granules and heated by the heater Tsgena elementary primary mobile network, and then transported to the furnace feed slot. And is used in this system, for the first time, a method depends on the heating of raw materials Tsgena first instance by a separate device with high thermal efficiency, a task that was carried out by the furnace in the conventional systems.

- If you use the oven supplier pole silicate are added and the girl clay during the granulation phase (Palletizing), and are drying and sintering (Sintering) and cooling in the layer vertical motion. There was the use of this system before the spread of rotary kilns, and is currently limited to use with a shaft furnace equipped with continuous discharge from the bottom of the oven to India and China. The advantage of this type of furnace heat economy, while the disadvantage of the need to continue moving the fire (Poking) in the oven to keep the situation stable combustion, in addition to the uneven quality of its products.



Name of experiment:

Study of cement properties

Number of experiment: - 11-

Part I: *Gravimetric analysis of unknown cement samples.*
Part II: *Gravimetric determination of insoluble materials in an unknown cement samples.*
Part III: *Gravimetric determination of sulphite in an unknown cement samples.*

Equipment: filtering crucible, ignition crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

Theory (part I):

The percentage of hydrated which allowed to be founded in an Iraqi cement according to Iraqi properties at the limit of 4%, as well as the kind of cement which have to be in this limits was considered to be a good cement, also having a good properties. Through the ignition method between (100-150) C◦, the sample of cement loose it hydrated , while the raising of temperatures degree to the limits of (900-950) C◦, both of calcium and magnesium carbonate convert to its oxides.

$$\text{Unknown of cement sample} \xrightarrow{\triangle} \text{Cement} + H_2O$$

$$CaCO_3 \xrightarrow[900\text{-}950\ C^o]{\varnothing} CaO + CO_2$$

$$MgCO_3 \xrightarrow[900\text{-}950\ C^o]{\varnothing} MgO + CO_2$$

Procedure (part I):

1. Accurately weigh 1g of unknown cement sample.
2. Bring porcelain crucible and cap to constant weight by heating to redness for 15 min . Cool in a desiccator and weigh crucible.
3. Heat the porcelain crucible with the sample for at least of 20 min. at (100-150) C◦.
4. Cool the crucible briefly in a desiccator. Weigh the crucible Calculate the weight percent of cement in the sample.
5. ignite the porcelain crucible for 10 min. at (900-950) C◦.
6. Again cool the crucible briefly in air and then in a desiccator. Weigh the crucible Calculate the weight percent of cement in the sample.

Results and calculations (part I):

$$\% \text{ hydrated} = \frac{m_1 - m_2}{m_1} \times 100$$

Where m_1 = Mass of sample before heating

Where m_2 = Mass of sample after heating

$$\% \text{ hydrated} = \frac{m_1 - m_2}{m_1} \times 100$$

Where m_1 = Mass of sample before ignition.

Where m_2 = Mass of sample after ignition.

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Theory (part II):

Cement contains many materials with different percentage, to make sure that the cement have a good properties , cement must contain (66-65)% of calcium oxide, (20-24)% of silicon oxide, (4-10)% of aluminium oxide, (2-4)% of iron oxide and (1-3)% of magnesium oxide, while alkaline oxides must be between (0.5-1)%, also (1-2)% of sulphite and insoluble materials which dissolved in acids must be in a limits of 1.5%.

The principle of this experiment depend on the treatment of known weight of cement sample at first with hydrochloric acid, while the precipitate will be treated with sodium hydroxide then filtered and ignite the product precipitate after cool it and weighted. The insoluble materials, calculated in that precipitate.

Procedure (part II):

1. Take the weight of sample in part I.
2. Add at first about 10 ml cold distilled water with constant stirring, then 5 ml of concentrated hydrochloric acid **(IN THE HOO**D).
3. Dilute your solution to 50 ml with distilled water.
4. Filtered your solution by using josh crucible, keep the filtrate for part III.
5. Wash the precipitate with with hot distilled water for six times in order to remove the soluble chlorides.
6. Dry in an oven at 200 C° for at least 10 min.
7. After cooling in dissicator, calculate the percentage of insoluble materials in cement.

Results and calculations (part II):

$$\% \text{ Insoluble materials} = \frac{\text{wt of precipitate}}{\text{wt of sample}} \times 100$$

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Theory (part III):

According to allowed properties, the percentage of sulphite trioxide in cement 2.5%. The concept of this method depend on determination of sulphite trioxide by precipitating it as barium sulphate by using barium chloride which finally ignite and weighted and calculated as SO_3.

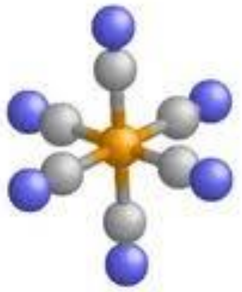
Procedure (part III):

1. Use the filtrate from part II, then dilute it with 10 ml of (10 %) hot barium chloride solution drop by drop with constant stirring.
2. Boiling the solution for 15 min. then leave it 15 min for stabilizing.
3. Filtered by using filter paper and washed with hot distilled water.
4. Ignite the precipitate between 800-900 C°.
5. Calculate the percentage of sulphite in cement, after cooling it in dissicator.

Results and calculations (part II):

$$\%SO_3 = \frac{\text{wt of precipitate}}{\text{wt. of Sample}} \times \frac{\text{M.wt. of } SO_3^{-2}}{\text{M.wt of } BaSO_4} \times 100$$

Discussion:



Write about industry of cement in Iraq.

References:

Name of experiment:

Gravimetric Determination of oxides in deferent cement samples

Number of experiment: - 12-

Part I: *Gravimetric determination of silica in an unknown cement samples.*

Part II: *Gravimetric determination of aluminium & iron oxides in an unknown cement samples.*

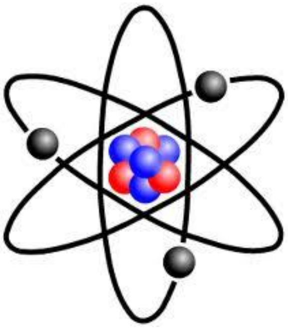
Part III: *Gravimetric determination of calcium oxide in an unknown cement samples.*

Part IV: *Gravimetric determination of magnesium oxide in an unknown cement samples.*

Equipment: filtering crucible, ignition crucible, glass rode, 400 ml beacker, 10 ml pipett, buchner funnel, washing bottle, desiccator, tongue.

Theory (part I): According to the Iraqi properties for the determination of silica in cement ,some testing must be discovered the percentage of silica in deferent cement samples which limits must be in a minimum of 19% to 24%.

The method concept depend on dissolving of different samples in a concentrated hydrochloric acid in presence of ammonium chloride . The precipitate ignited at 1150-1200 C° , while the filtrated used to determine aluminium and iron as oxides according to the following equations:



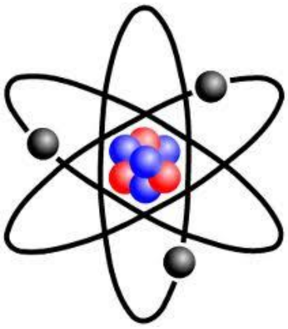
$$SiO_2 + HCl \longrightarrow \text{No. Reaction}$$

$$Al_2O_3 + HCl \longrightarrow 2AlCl_3 + 3H_2O$$

$$Fe_2O_3 + HCl \longrightarrow 2FeCl_3 + 3H_2O$$

$$CaO + HCl \longrightarrow CaCl_2 + H_2O$$

$$MgO + HCl \longrightarrow MgCl_2 + H_2O$$



Procedure(part I):(this experiments must do it in hood)

1. Accurately weigh 1g of unknown cement sample.
2. Add 1 gm of ammonium chloride, then mixed it well.
3. Add 10 ml of con. HCl slowly at your beaker wall , then stirring the mixture with glass road .
4. Put the beaker with covered in water bath for at least 30 min.
5. Dilute the solution with 50 ml with hot distilled water then filtrate it and keeping the filtration for determination oxides next lab.

6. Washing the precipitate twice with 5% HCl then with hot distilled water.
7. Ignite the precipitate at 1150-1200 C° for 30 min.

Discussion(part I):

1. What happened to the mixture if you used HF instead of HCl.
2. What is the perfect solvent for silica.
3. Why we must used NH_4Cl in this experiment.
4. Is silica which fond in cement samples useful for all kind of cement.
5. Write about silica industry in Iraq.

Results and calculations(part I):

$$\% \text{ Silica} = \frac{\text{wt of precipitate}}{\text{wt of sample}} \text{ x } 100$$

Theory (part II):

The experiment concept is depend on precipitation both of aluminium and iron oxides with ammonium hydroxide as precepitatent agent in presence of ammonium salts, which made more control the pH of the mixture and crystallization of precipitate , as well as preventing of precipitation of calcium and magnesium hydroxides by forming with it solvent complexes, the precipitate formed of:

$$AlCl_3 + 3NH_4OH \longrightarrow Al(OH)_3 + 3NH_4Cl \xrightarrow{\varnothing} Al_2O_3$$

$$FeCl_3 + 3NH_4OH \longrightarrow Fe(OH)_3 + 3NH_4Cl \xrightarrow{\varnothing} Fe_2O_3$$

Where the filtrate formed of :

$$CaCl_2 + 2NH_4OH \longrightarrow Ca(OH)_2 + 2NH_4Cl$$

$$MgCl_2 + 2NH_4OH \longrightarrow Mg(OH)_2 + 2NH_4Cl$$

Procedure(part II):

1. Dilute the filtrate (from part I) to 200 ml with distilled water.

2. Add 5 ml hydrogen peroxide.
3. Add 2-3 drops of methyl red indicator.
4. Add 1 gm of ammonium nitrate then add concentrated ammonium hydroxide solution drop by drop with stirring (the color must be yellow).
5. Leave it for at least 15 min. (Digestion).
6. Use ashless filter paper for filtration, also keep the filtrate for experiment part III;
7. Wash the precipitate three times with hot ammonium nitrate, the ignite it for 15 min. at 1100 C° .
8. Calculate the percentage of Al an Fe in cement sample.

Results and calculations(part II):

$$\% \text{ Al} = \frac{\text{wt of precipitate}}{\text{wt of sample}} \text{ x } 100$$

$$\% \text{ Fe} = \frac{\text{wt of precipitate}}{\text{wt of sample}} \text{ x } 100$$

Discussion(part II):

Theory (part III):

According to the Iraqi properties, calcium oxide limit must be between 54% - 66 % , in this experiment the precipitate agent which used for precipitating of calcium is oxalic acid . the concept of this experiment depend on precipitating of calcium from the filtrate which kept from part II (determining of Al and Fe) by adding oxalic acid as follow:

$$CaC_2O_4 + 1/2\ O_2 \longrightarrow CaO + 2CO_2$$

The concept of this experiment as bellow:

$$Ca(OH)_2 + 2\ HCl \longrightarrow CaCl_2 + 2\ H_2O$$

$$Mg(OH)_2 + 2\ HCl \longrightarrow MgCl_2 + 2\ H_2O$$

$$CaCl_2 + H_2C_2O_4 \longrightarrow CaC_2O_4 + 2\ HCl$$

$$MgCl_2 + H_2C_2O_4 \longrightarrow MgC_2O_4 + 2\ HCl$$

For dissolving of magnesium oxalate , ammonium hydroxide was added . Then the filtrate which contain magnesium ions used in part IV.

$$MgC_2O_4 + 2\ NH_4OH \longrightarrow Mg(OH)_2 + (NH_4)_2C_2O_4$$

$$CaC_2O_4 + NH_4OH \longrightarrow N.R$$

Procedure(part III):

1. Boil the filtrate from part II until 300 ml, then added 2-3 drop of methyl red indicator (solution color 's is yellow).
2. Added concentrated hydrochloric acid and wath the color (being red).
3. Add 2 gm of oxalic acid .
4. While the mixture is boiling , add concentrated ammonium hydroxide with constant stirring (color must be yellow).
5. Leave the hot mixture 15 min. for digestion (don’t cool it).
6. After filtration , wash the precipitate with 1g/l ammonium oxalate. Also keep the filtration for part IV.
7. Ignite at 1100 C°, cool it and calculate the percentage of calcium..

Results and calculations(part III):

$$\%\ Ca = \frac{\text{wt of precipitate}}{\text{wt of sample}} \times 100$$

Discussion(part III):

Theory (part IV):

Magnesium oxide is one of cement component , increasing of its percentage more than 5% in formation of cement produced a bad kind of cement. Magnesium has been precipitate from filtration which is kept from part III by adding diammoiniumhydrogen phosphate as a precepitatnt agent:

$$Mg(OH)_2 + 2\,HCl \longrightarrow MgCl_2 + 2\,H_2O$$

$$MgCl_2 + (NH_4)_2HPO_4 \longrightarrow MgHPO_4 + 2\,NH_4Cl$$

$$MgHPO_4 + 2\,NH_4Cl \longrightarrow Mg(NH_4)HPO_4 + H_2O$$

$$Mg(NH_4)HPO_4 \longrightarrow Mg_2P_2O_7 + 2\,NH + H_2O$$

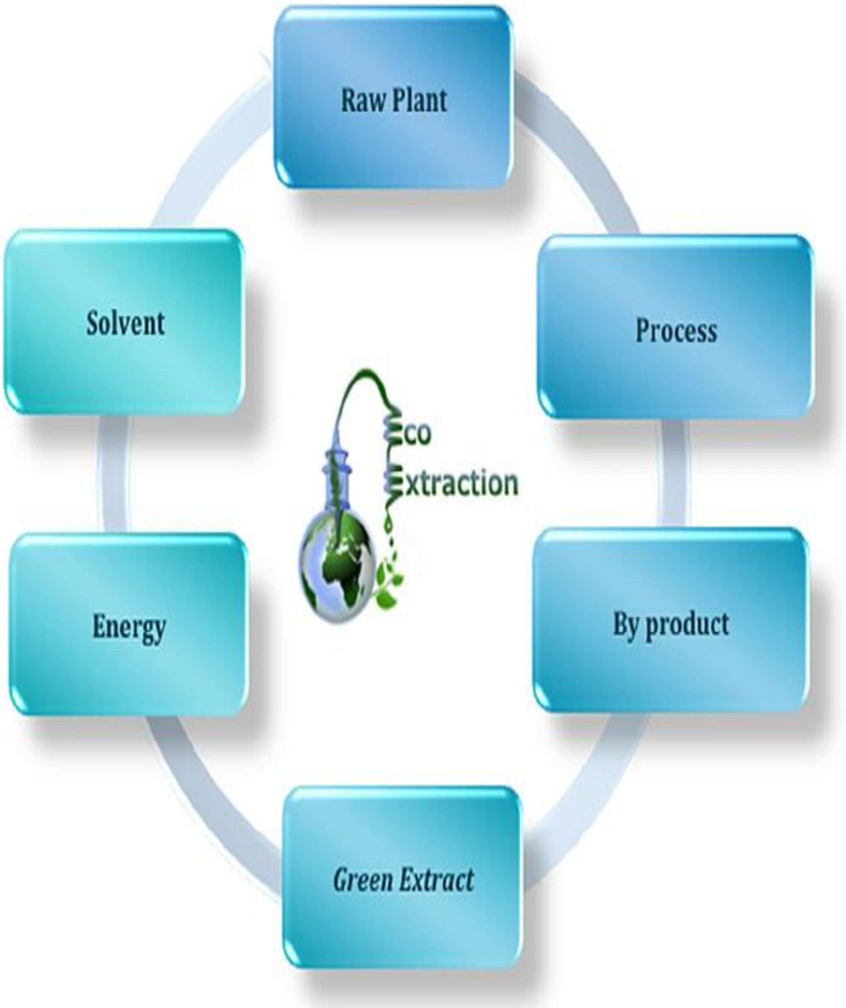
Procedure(part IV):

1. Acidified the filtration with 10% of hydrochloric acid.
2. Evaporate it till the final volume equaled to 400 ml.
3. Add 20 ml of hot and saturated diammonium hydrogen phosphate (250g/l) with constant stirring.
4. Add 35-40 ml of concentrated ammonia solution.also with constant stirring.
5. Cool it slowly under tap water for at least 20 minutes.(constant stirring with glass rod).
6. After filtration, washed it with cold distilled water containing 2.5 % ammonia.
7. Dry it between 100-120 C, then calculate the percentage of magnesium oxide.

Results and calculations(part IV):

$$\%Mg = \frac{\text{wt of precipitate}}{\text{wt of sample}} \times 100$$

Discussion(part IV):



GRAVIMETRIC ANALYTICAL CHEMISTRY – LAB

Experimental 2nd Lab.

ABSTRACT

The second stage extraction experiments focus on improving separation and concentration efficiency using techniques like evaporation and filtration…etc.

Basima A.A.Saleem
Assistant Professor

Extraction of caffeine from tea

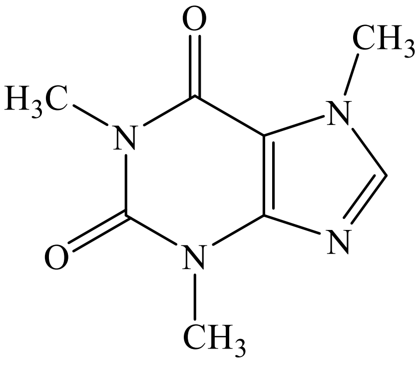
- **No. of experiment: 1**
- **Purpose of experimental**

1. To demonstrate the isolation of a natural product.

2. To learn the techniques of extraction

Theory part of experimental

Many organic compounds are obtained from natural sources through extraction. This method takes advantage of the solubility characteristics of a particular organic substance with a given solvent. In the experiment here, caffeine is readily soluble in hot water and is thus separated from the tea leaves. Caffeine is one of the main substances that make up the water solution called tea. Besides being found in tea leaves, caffeine is present in coffee, kola nuts, and cocoa beans. As much as 5% by weight of the leaf material in tea plants consists of caffeine. The caffeine structure is shown below. It is classed as an alkaloid, meaning that with the nitrogen present, the molecule has base characteristics (alkali-like). In addition, the molecule has the purine ring system, a framework which plays an important role in living systems. Caffeine is the most widely used of all the stimulants.



Structure of Caffeine

Tea leaves consist primarily of cellulose; this is the principle structural material of all plant cells. Fortunately, the cellulose is insoluble in water, so that by using a hot water extraction, more soluble caffeine can be separated. Also dissolved in water are complex substances called tannins. These are colored phenolic compounds of high molecular weight (500 to 3000) that have acidic behavior. If a basic salt such as Na_2CO_3 is added to the water solution, the tannins can react to form a salt. These salts are insoluble in organic solvents, such as chloroform or dichloromethane, but are soluble in water.

Although caffeine is soluble in water (2 g/100 g of cold water), it is more soluble in the organic solvent dichloromethane (14 g/100 g). Thus caffeine can be extracted from the basic tea solution with dichloromethane, but the sodium salts of the tannins remain behind in the aqueous solution. Evaporation of the dichloromethane yields crude caffeine; the crude material can be purified by sublimation.

➢ Chemical and apparatus

Boiling chips, Cold finger condenser, Filter paper, fast flow Hot plate, 125-mL separatory funnel with stopper, Melting point capillaries, adapter, Tea leaves, rubber stopper, Anhydrous sodium sulfate Na_2SO_4, Anhydrous sodium carbonate Na_2CO_3, Dichloromethane CH_2Cl_2.

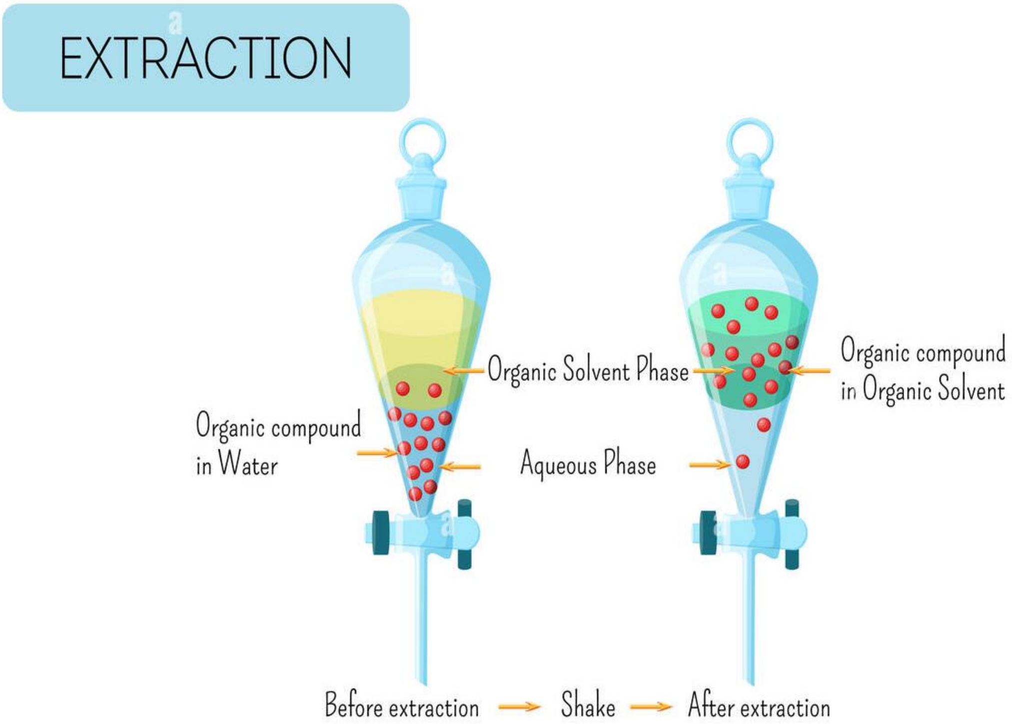
➢ Procedure of experimental

1. Into a 150-mL beaker, place the tea leaves so that they lie flat on the bottom. Add 30 mL of distilled water and 2.0 g of anhydrous Na_2CO_3; heat the contents with a hot plate, keeping a gentle boil, for 20 min. While the mixture is boiling, keep a watch glass on the beaker.
2. Decant the hot liquid into a 50-mL Erlenmeyer flask, carefully pressing the tea bag with a glass rod; add this wash water to the tea extract. (If any solids are present in

the tea extract, filter them by gravity to remove.) Cool the combined tea extract to room temperature. Transfer the cool tea extract to a 125-mL separatory funnel that is supported on a ring stand with a ring clamp.

3. Carefully add 5.0 mL of dichloromethane to the separatory funnel. Stopper the funnel and lift it from the ring clamp By holding the stopper in place with one hand, invert the funnel.
4. Return the separatory funnel to the ring clamp, remove the stopper, and allow the aqueous layer to separate from the dichloromethane layer. You should see two distinct layers form after a few minutes, with the dichloromethane layer at the bottom. Sometimes an emulsion may form at the juncture of the two layers. The emulsion often can be broken by gently swirling the contents or by gently stirring the emulsion with a glass rod. Separation of the aqueous layer and the dichloromethane layer in the separatory funnel.
5. Carefully drain the lower layer into a 25-mL Erlenmeyer flask. Try not to include any water with the dichloromethane layer.
6. Repeat the extraction with an additional 5.0 mL of dichloromethane. Combine the separated bottom layer with the dichloromethane layer obtained from step no. 5.
7. Add 0.5 g of anhydrous Na2SO4 to the combined dichloromethane extracts. Swirl the flask. The anhydrous salt is a drying agent and will remove any water that may still be present.
8. Weigh a 25-mL side-arm filter flask containing one or two boiling stones. Record this weight (2). By means of a gravity filtration, filter the dichloromethane salt mixture into the pre-weighed flask. Rinse the salt on the filter paper with an additional 2.0 mL of dichloromethane.
9. Remove the dichloromethane by evaporation in the hood. Be careful not to overheat the solvent, since it may foam over. The solid residue which remains after the solvent is gone is the crude caffeine. Reweigh the cooled flask (3). Calculate the weight of the crude caffeine by subtraction (4) and determine the percent yield (5).
10. Take a melting point of your solid. First, scrape the caffeine from the bottom and sides of the flask with a micro spatula and collect a sample of the solid in a capillary

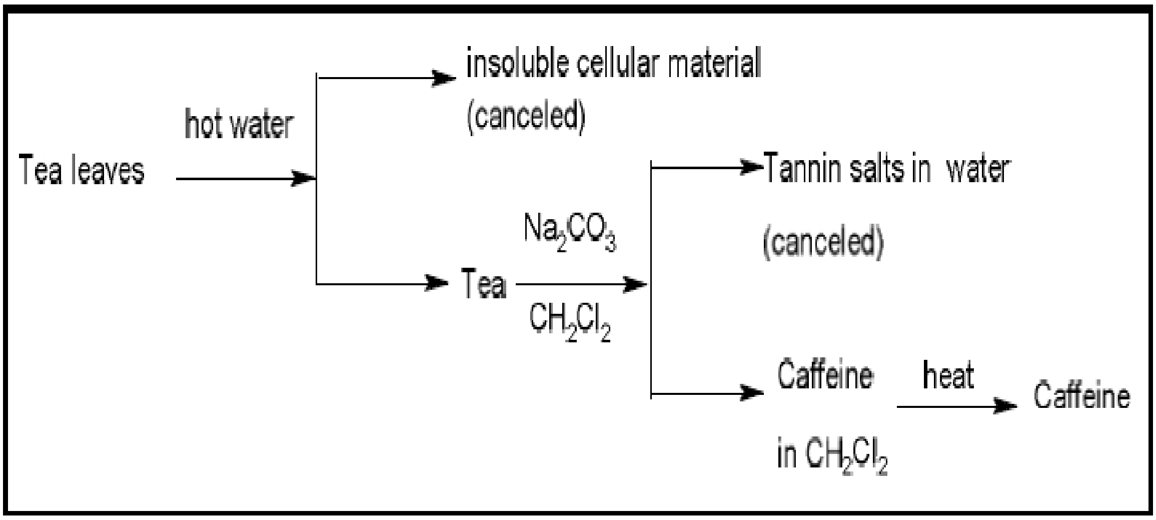
tube. Pure caffeine melts at 238 °C. Compare your melting point to the literature value.



➢ **Calculation**

$$\% \text{ Yield} = \frac{\text{Weight of product}}{\text{Weight of sample}} \times 100$$

➢ **Plan of work**



- **Questions for discussion**

1. Is methylene chloride more or less dense than water? What evidence do you have to support your answer?
2. Was your melting point the same as the reported melting point? If not, why not?
3. Why was sodium carbonate added to the coffee solution?
4. Explain why the caffeine that you obtained in this experiment may not have been white in color.
5. What method is used to obtain caffeine for tea leaves?
6. Why is caffeine classed as an alkaloid?
7. Why might an individual use a product containing caffeine?
8. Besides caffeine, what other compounds are found in tea leaves?

Analytical Chemistry Lab

GRAVIMETRIC ANALYTICAL CHEMISTRY – LAB

Experimental 2nd Lab.

Basima A.A.Saleem

Quantity of Active Component in Aspirin

- **No. of experiment: 2**
- **Purpose of experiment:**

1) **Quality Assurance:** Testing aspirin ensures that products meet required quality standards in terms of purity and concentration.
2) **Performance Comparison:** It allows for understanding the variations in effectiveness among different companies, helping to identify the best product.
3) **Contamination Detection:** Laboratory tests can reveal any contaminants or undesirable substances that could affect product safety.
4) **Regulatory Compliance Verification:** It helps verify that companies adhere to good manufacturing practices and standards set by regulatory agencies.
5) **Cost-Benefit Evaluation:** Testing assists in assessing whether different products provide the desired value for their cost.
6) **Data Provision for Future Studies:** It provides valuable data that can be used in future research and studies regarding drugs and their effects.

Introduction:

What measures may be implemented to guarantee that pharmaceuticals have the intended active ingredients? Frequently, it is not feasible to ascertain the presence or efficacy of a medication just by visual inspection. The U.S. Food and Drug Administration (FDA) has instituted Current Good Manufacturing Practices (CGMP) that define criteria for guaranteeing pharmaceutical quality. These criteria guarantee the identity, potency, quality, and purity of pharmaceutical products. Each manufacturer is accountable for determining the implementation of these criteria and for reporting their adherence. The FDA performs limited testing, mostly evaluating results submitted by pharmaceutical companies.

Suppose that you have recently been appointed as a quality control specialist . Your objective is to examine the most recent batch of aspirin to ascertain the quantity of active substance contained inside. How would you measure the active ingredients in a commercial aspirin product?

Aspirin

Aspirin, or acetylsalicylic acid, is a pharmaceutical agent utilized for the temporary alleviation of pain and fever. It has been marketed as an analgesic for many ailments, as illustrated in the label shown in Figure 1. Acetylsalicylic acid (refer to Figure 1) is classified as a weak acid. Acids are chemical substances that generate hydrogen ions H^+, in aqueous solutions, with pH serving as an indicator of hydrogen ion concentration.

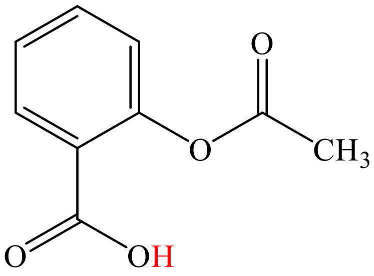


Figure 1: Chemical structure of Aspirin (Acetylsalicylic acid)

Upon ingestion, acetylsalicylic acid remains unaltered in the acidic environment of the stomach; however, in the alkaline conditions of the upper intestine, it dissociates into salicylate and acetate ions. Aspirin selectively suppresses the synthesis of cyclooxygenase-1 and cyclooxygenase-2, enzymes implicated in inflammation and discomfort.

Historical records indicate that the treatment of ailments by the consumption of extracts from salicylate-rich plants has been practiced for millennia. Approximately 2400 years ago, Hippocrates advocated the treatment of eye ailments and pain with the juices of poplar trees and willow bark, both of which contain salicylates. The varying strengths of aspirin are determined by the concentration of acetylsalicylic acid contained therein. This experiment involves quantifying the amount through a process called titration.

Titration is a method used to ascertain the concentration of an unknown acid or base by incrementally adding a precise volume of the corresponding base or acid until neutralization occurs. A neutralization or acid-base reaction occurs when an acid interacts with a base, resulting in the formation of water and a salt. The stoichiometrically balanced molecular equation for the reaction between acetylsalicylic acid and sodium hydroxide is presented in Equation 1 and illustrated in Figure 2.

Burette: contain titrant (Solution of known concentration).

Conical flask: contain the analyte with unknown concentration and indicator.

$$HC_9H_7O_{4(aq)} + NaOH_{(aq)} \longrightarrow NaC_9H_7O_{4(aq)} + H_2O_{(l)} \qquad \textbf{Equation 1}$$

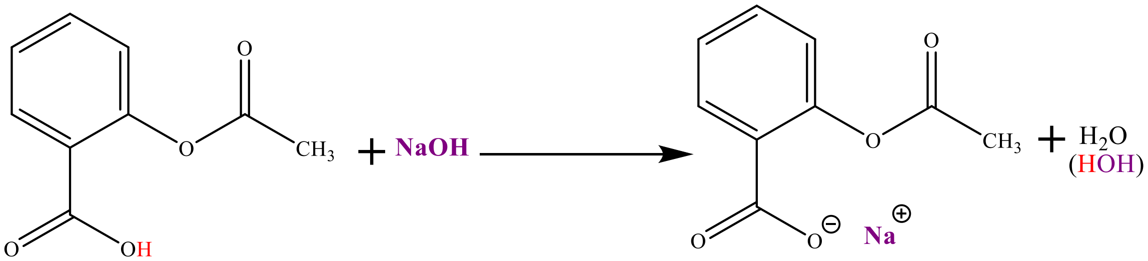


Figure 2. Structural depiction of the reaction between acetylsalicylic acid and sodium hydroxide

Experimental procedure:

A series of steps will facilitate the examination of aspirin. Initially, it is necessary to make a solution with a certain concentration of sodium hydroxide. You will receive a stock solution of roughly 1 M NaOH for this purpose. You will dilute it to roughly 0.1 M NaOH and thereafter standardize the diluted solution using titration with hydrochloric acid (HCl). A standard solution of HCl (about 0.15 M) will be supplied; its precise concentration will be indicated on the carboy or bottle. A standard solution is a solution having a precisely known concentration. Ultimately, you will dissolve the aspirin and titrate it with the NaOH solution you created to ascertain the quantity of acetylsalicylic acid per tablet. This experiment will comprise two parts.

Part A:

Determination of the concentration of NaOH(aq) using standardized HCl(aq)

Summary of the procedure: You will prepare a solution of NaOH through dilution. You will thereafter ascertain the concentration of the NaOH solution using titration, utilizing the standardized HCl solution as the titrant (see to Equation 3). Document the information from Part A Data .

1) Dilute NaOH

$$\mathbf{M_1 \times V_1 = M_2 \times V_2}$$ **Equation 2**

where: M is molarity, V is volume, in this equation it can be in L or mL as long as both volumes are in the same unit

2) Standardize NaOH using standard HCl:

$$NaOH_{(aq)} + HCl_{(aq)} \longrightarrow H_2O_{(l)} + NaCl_{(aq)}$$ **Equation 3**

Part B

Determination of the amount of acetylsalicylic acid in aspirin by titration with NaOH

1. **Prepare the aspirin sample trial.**

a. Select a brand of aspirin. Document the brand and quantity of the active substance.

b. Extract one tablet from the chosen brand, measure its weight, and document the value.
c. Grind the measured pill with a mortar and pestle.
d. Tare a vessel (or weighing paper) on the scale. Meticulously transfer the maximum quantity of powdered sample into the boat. Document the mass (including all digits displayed on the analytical balance). It is necessary to ascertain the precise mass titrated to rectify the mass loss.
e. Transfer the measured powder into a clean conical flask (125 mL or 250 mL). Rinse the flask with distilled water to guarantee the complete transfer of the sample into the flask. Designate the flask as Aspirin Trial 1.
f. Measure 10.0 mL of ethyl alcohol using a graduated cylinder, transfer it to the conical flask, and stir for approximately 30 seconds with a glass stirring rod.
g. Add 25.0 mL of deionized water to the flask and stir for an additional 30 seconds. The tablet will not fully dissolve because of inert components (fillers and binding agents); the resulting solution will be murky.
h. Filtering will be required. Ensure that your sample is appropriately labeled.
i. Rinse the filter paper and precipitate with a few milliliters of dw.

2. Titration of aspirin with NaOH

j. Add 2-3 drops of phenolphthalein indicator to the filtered aspirin sample V1.
k. Make the initial NaOH volume reading. Read the burette to a precision of ± 0.01 mL and record.
l. Start titrating: Add the NaOH into the aspirin sample while continually mixing. when the solution turns pale pink and persists for at least 30 seconds. Stop titrating. Make the final NaOH volume reading. Read and record the burette to a precision of ± 0.01 mL.
m. Repeat the steps above twice.

3. Calculation of active ingredient in the aspirin

i. Calculate the volume of NaOH added and the moles of NaOH.
ii. Calculate the moles of acetylsalicylic acid that reacted with the NaOH in the titrated sample using Equation 1.
iii. Calculate the grams of acetylsalicylic acid in the titrated sample using the molar mass acetylsalicylic acid.
iv. Determine milligrams of acetylsalicylic acid (ASA) in the whole tablet by using Equation 4.

$$mg_{ASA/tablet} = mg_{ASA\ in\ sample} \times \left(\frac{\text{mass tablet}}{\text{mass pulverized sample}}\right) \quad \text{Equation 4}$$

Read		$(X_i - \bar{X})^2$
V_1		
V_2		
V_3		
$\bar{X}$ =		$\Sigma(X_i - \bar{X})^2$ =

Then Calculate S Value:

$$S = \sqrt{\frac{\Sigma(X_i - \bar{X})^2}{n - 1}}$$

Then Calculate RSD Value:

$$RSD = \frac{S}{\bar{X}} \times 100$$, If RSD<5%, you were precise in your measurements

Then Calculate R.E Value:

$$R.E\% = \left|\frac{\text{True value - Experimental value}}{\text{True value}}\right| \times 100$$

where: true value is the expected or theoretical value experimental value is the calculated value based on data

Questions for discussion

Exercise 1: A solution of magnesium hydroxide was titrated with a hydrochloric acid solution. What was the mass of Mg(OH)2 in the solution if the titration required 71.10 mL of 0.150 M HCl?

Exercise 2: write the balanced equation for the above reaction.

Exercise 3: What volume, in milliliters, of a stock solution of 2.55 M NaOH would you have to use to prepare 1.00 L of a 0.500 M NaOH?

Exercise 4: The titration of a 25.00 mL sample of NaOH required 28.25 mL of a 0.200 M HCl solution to reach the end point. What was the molar concentration of the sodium hydroxide solution?

Exercise 5: A tablet of Pain Be Gone Aspirin, which had a mass of 1.213 g, was pulverized and 1.159 g were dissolved in 10.0 mL of ethyl alcohol and 25.0 mL of DI water. The titration of this solution with 0.1052 M NaOH required 15.62 mL to reach the phenolphthalein endpoint. a. Determine the moles of NaOH that reacted with the acetylsalicylic acid. Answer questions below.

a. Determine the moles of NaOH that reacted with the acetylsalicylic acid.

b. Determine the mass, in grams, of acetylsalicylic acid in the sample analyzed.
c. Determine the mass, in milligrams, of $HC_9H_7O_4$ in the tablet.
d. The manufacturer claims that each tablet contains 325 mg + 10 mg of acetylsalicylic acid. Is the actual amount of acetylsalicylic acid in the tablet acceptable?

Second year

GRAVIMETRIC ANALYTICAL CHEMISTRY – LAB

Basima A.A.Saleem

Second year

Separation of Food Colors in M&M Candies Via Paper Chromatography

- **No. of experiment: 3**
- **Purpose of experiment:**

- To use paper chromatography to identify dyes in candy coatings are pure substances or mixtures.
- To obtain a paper chromatogram of various dyes in candy coatings
- To identify components of dyes in candy coatings by calculating Rf values

- **Introduction:**

Chromatography is a method of physically separating *mixtures* into its individual components. It is a common laboratory technique used to identify unknown components in mixtures.

There are several types of chromatography; all types employ a *mobile phase* or *eluent* (it can be liquid or gas), which is forced through a *stationary phase* (a solid or semi-solid). Mixtures are separated because some components will be more attracted to the stationary phase (and stick to it) while some components will be more attracted to the mobile phase (and travel with it).

By eye, we cannot know if each ink color is a mixture or pure substance. Using chromatography, the components in a sample will migrate along the filter paper at different rates such that they become spread out and separated from each other. The mobile phase takes advantage of differing solubility or polarity of the components in order to separate them. This component traveling process is called *elution*. Chromatography can be used to separate inks, dyes, pigments in plants, or used to determine the chemical composition of many substances.

M&M candies are known not only for their taste but also for their vibrant colors. The use of food coloring in M&M candies contributes greatly to their appeal and brand. The colors in M&M candies are created using natural and synthetic food dyes. Some of the commonly used dyes include:

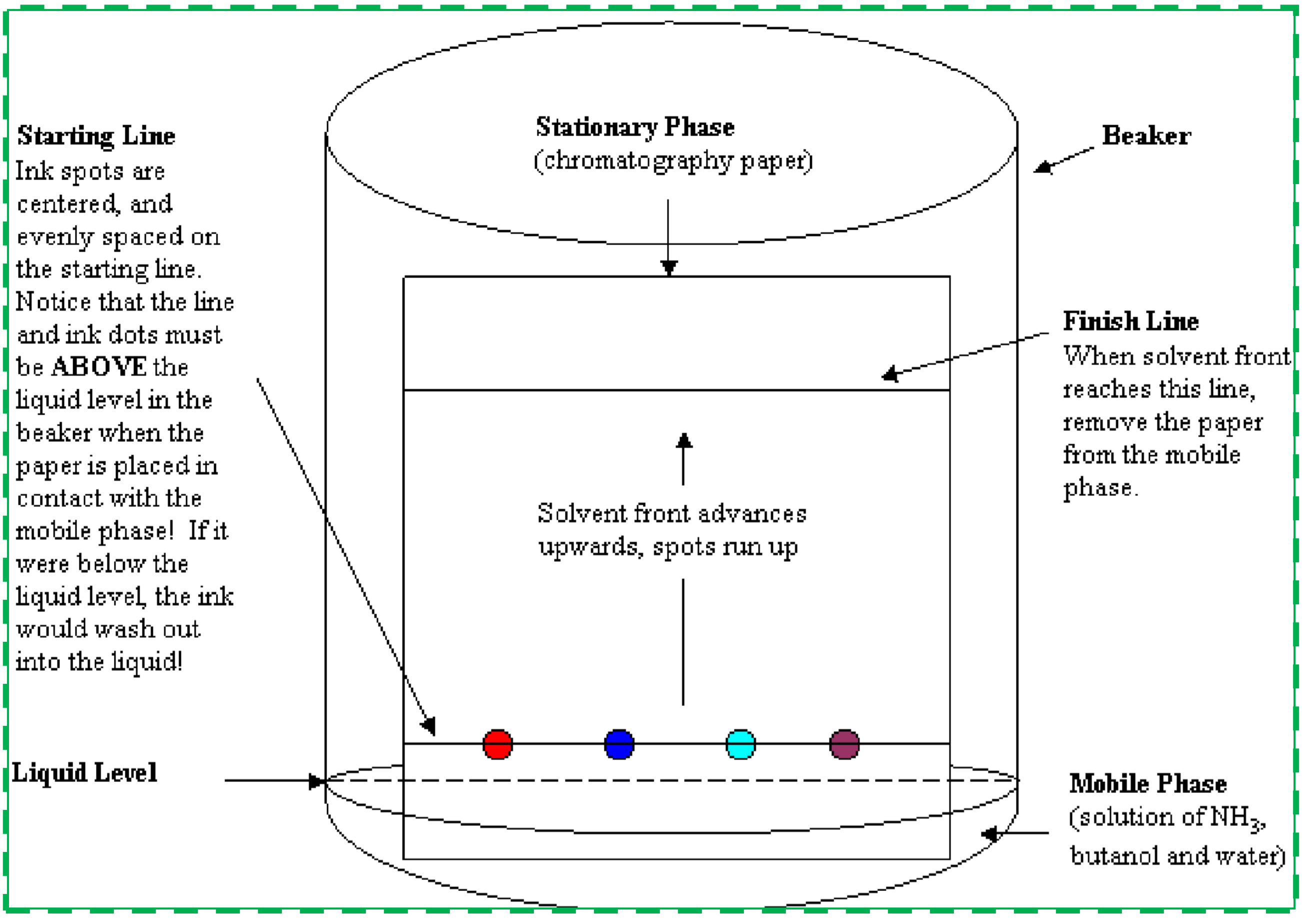
- Red 40: A synthetic dye that gives a bright red appearance.
- Yellow 5: A common yellow dye derived from coal tar.
- Blue 1: A synthetic blue dye.
- Titanium dioxide: Used for white color and as a bleaching agent.

What is Paper Chromatography?

Paper chromatography is a method used to separate mixtures of substances, typically pigments, based on their different rates of movement across a stationary phase (the paper) when dissolved in a mobile phase (the solvent).

How It Works

1. **Preparation**:Take a strip of chromatography paper and draw a light pencil line about an inch from the bottom. This is where you will apply the sample.
2. **Sample Application**: Using a toothpick or a small dropper, apply a small dot of color from the melted M&M candy onto the pencil line. Allow it to dry and apply another dot on top for a more concentrated sample.
3. **Developing the Chromatogram**: Place the bottom of the paper strip in a shallow dish containing a small amount of solvent (like water or alcohol) without immersing the dot. The solvent will travel up the paper by capillary action, carrying the pigments with it.
4. **Separation**: As the solvent moves, different pigments will travel at different rates, causing them to separate along the paper. The distance each pigment travels depends on its solubility and affinity for the paper.
5. **Analysis**: Once the solvent has nearly reached the top, remove the paper and let it dry. You will see distinct bands of color representing the different dyes used in the candy.



Materials and Equipment

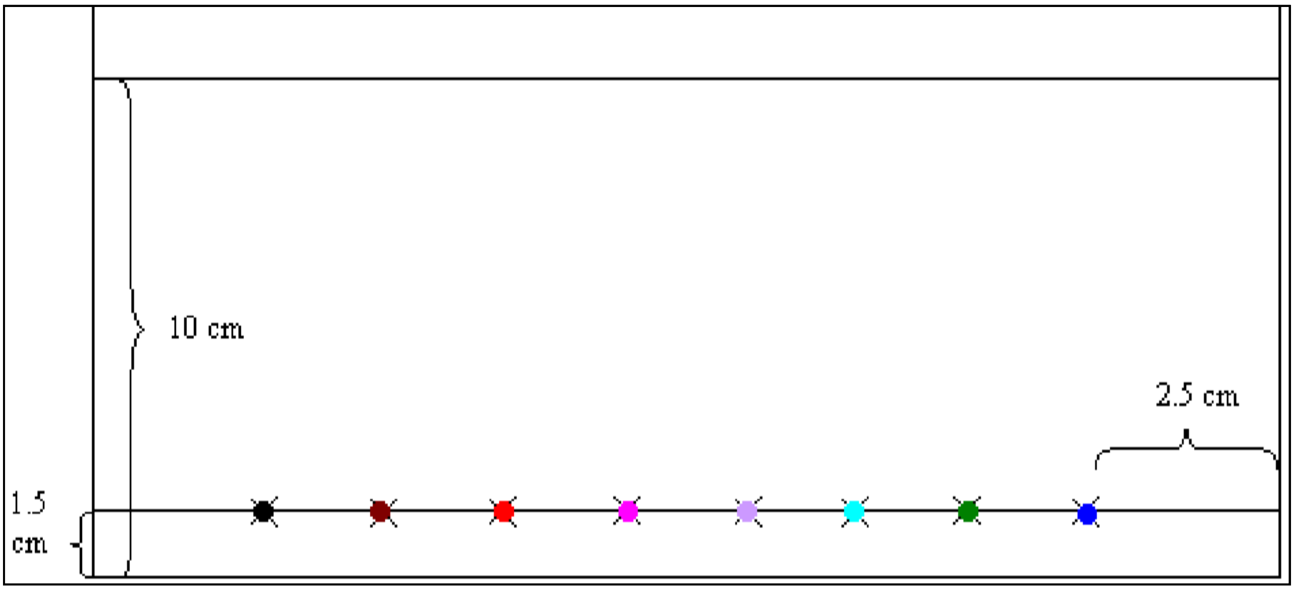
Materials: chromatography paper (filter paper cut into a rectangle), M&M candies (Red, Blue, Green, Yellow, Brown) and eluting solution (0.1g table salt dissolved in 10ml water), a wider mouth clear container or a larger beaker to keep the filter paper for elution

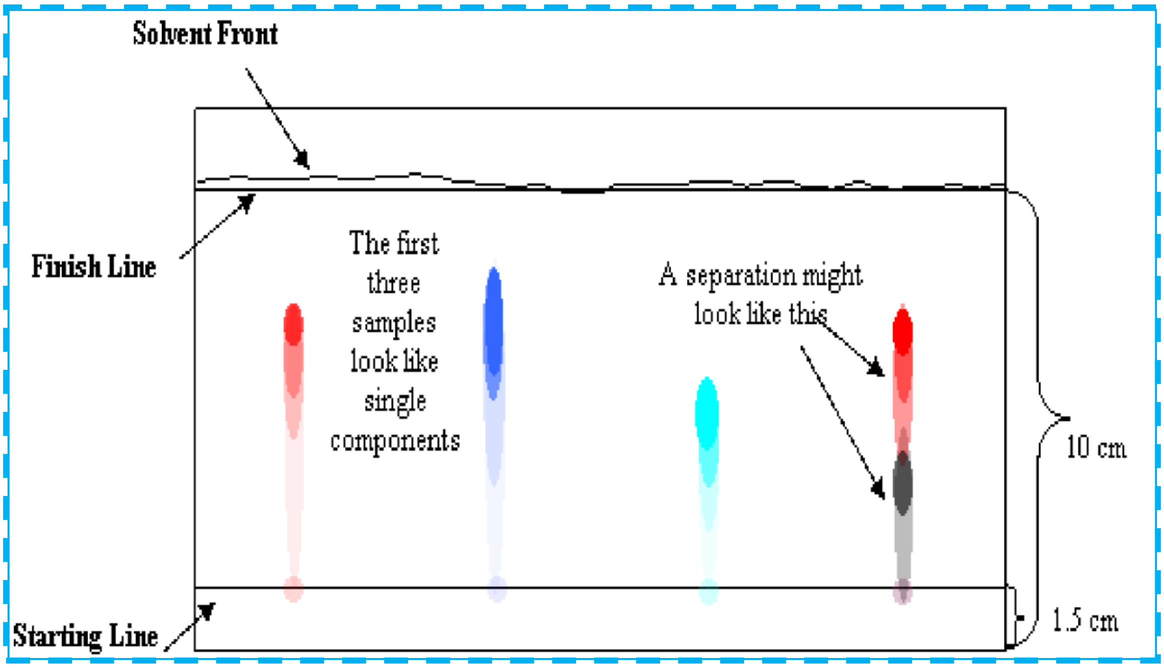
Equipment: 500 ml beaker or a similar size flat bottom container, pencil, ruler, tape, and paper towels.

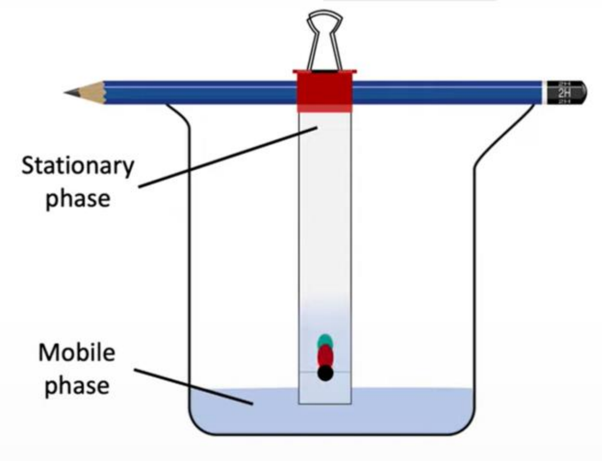
Procedure:

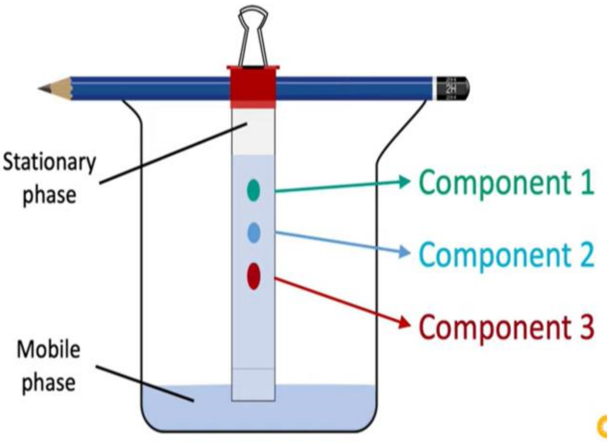
1. Prepare a 0.1% salt solution in a wide-mouth jar, ensuring the depth allows no more than 0.5 cm of the filter paper to touch the solution when suspended.
2. Draw a line about 1 cm from the bottom of the filter paper using a pencil and ruler.
3. Place the filter paper on a towel. Dip a half-toothpick in a dye and spot it onto one of the hash marks on the paper, keeping the spot about 0.2-0.3 cm in diameter. Use a different toothpick for each dye.
4. Tape the top of the filter paper to a pencil. Without touching the jar's walls, slowly lower the paper into the solution, ensuring the bottom edge touches the solution without contacting the dyes. Adjust the solution level as needed.

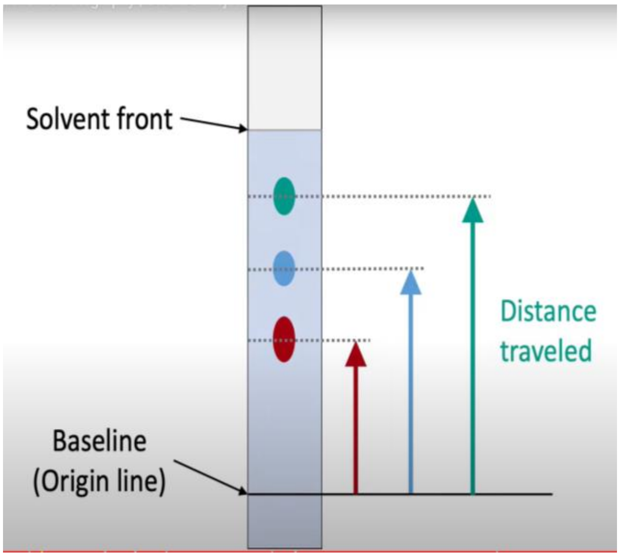
5. Remove the paper once the solution has migrated within 1-2 cm from the top and place it on a towel or napkin.











calculations

$$R_f = \frac{D}{F}$$

$$R_f = \frac{\text{Distance traveled by substance}}{\text{Distance traveled by solvent}}$$

Conclusion

This technique not only helps in educational settings to demonstrate principles of chromatography but also in quality control and food safety to ensure that the dyes used comply with regulations. Paper chromatography is a simple yet effective way to separate and analyze the colors found in M&M candies, providing insights into the variety and nature of food dyes used in these popular treats.

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