**Analytical Chemistry**

Analytical chemistry is that branch of chemistry which concerned with the separation, identification and determination of the amounts of the elements present in a sample.

Analytical chemistry can be divided into two branches .**Qualitative analysis(** identify the elements or compounds present in a sample**)** and **Quantitative analysis(** determine the amounts of elements or compounds present in the sample**)**.

Quantitative analysis divided into two methods:

1. Gravimetric method ( change in weight).
2. Volumetric method (change in volume).

**Titration**

Is a procedure use to determine the concentration of some substance by the controlled addition of a solution into a reaction flask from a burette.

**Types of titration:**

1. acid – base (neutralization tit.)
2. oxidation – reduction titration (redox tit.)
3. complex formation titration.
4. Precipitation titration.

**Standard solution**

Is a solution with accurately known concentration.

A primary standard compounds should fulfill these requirements:

1. It should be 100% pure.
2. It should be stable to drying temperature, light and air.
3. It should be readily available and low cost.
4. It should have a high formula weight.

**Equivalence point**

Is the point in the titration in which enough standard solution has been added to react with the substance being determined. The reactants are mixed in exact molar proportions represented by the balanced equation.

**End point**

The point at which the indicator changes colour.

**Indicator**

A compound which changes colour at a specific pH value or in the presence of a particular substance, and can be used to monitor acidity, alkalinity, or the progress of a reaction.

Solutions

A mixture say to be solution if it is 1- homogeneous mixture of two or more substances. 2- consist of a solvent and one or more solutes.

Preparation of solutions:

1. *solid in liquid solution*

molarity(M) = no. mole of solute / volume of solution (litter)

M

***Wt = M \* M.wt \* vml / 1000***

Normality (N)= no. equivalent of solute / volume of solution (litter)

N = wtsolute \* 1000 / eq.wtsolute \* vsolution (ml)

Equivalent weight (eq.wt) = M.wt / equivalent no.(n)

Equivalent number differ from a compound to another depending on the nature of the compound and the reaction.

***For acidic compound n= no. acidic proton for basic compound n= no. OH group***

|  |  |
| --- | --- |
| Acid | N |
| HCl | 1 |
| H2SO4 | 2 |
| H3PO4 | 3 |
| CH3COOH | 1 |

|  |  |
| --- | --- |
| Base | n |
| NaOH, KOH, NH3 | 1 |
| Ca(OH)2 , Mg(OH)2 | 2 |
| Fe(OH)3 , Al(OH)3 | 3 |

***for salts compounds***

|  |  |
| --- | --- |
| Salt | N |
| NaCl , KBr | 1 |
| CaCl2 , MgBr2 , (NH4)2SO4 | 2 |
| CaSO4 , MgCO3 | 2 |
| AlCl3 , FeCl3 , FePO4 | 3 |

*example*

1. prepare 0.1M of NaCl in 250ml distilled water.
2. Prepare 5M of CaSO4 in 500ml D.W.
3. Prepare 3.2M of KOH in 100ml D.W.
4. Prepare 0.6M of Mg(OH)2 in 1000ml distilled water.
5. *liquid in liquid solution*

*M(bottle) = sp.gr \* % \* 10 / M.wt*

Sp.gr = specific gravity of the liquid

% = percentage of the liquid in the bottle

*( M1 \* V1) before dilution = (M2 \* V2) after dilution*

*N(bottle) = sp.gr \* % \* 10 / eq.wt*

*( N1 \* V1) before dilution = (N2 \* V2) after dilution*

*example*

1. prepare 0.1M of HCl in 250ml distilled water.(sp.gr = 1.19 , % = 37)
2. Prepare 5M of H2SO4 in 500ml D.W. ( sp.gr = 1.85 , % = 98 )
3. Prepare 3M of H3PO4 in 100ml D.W. ( sp.gr = 1.69 , % = 85

Experiment:

**Preparation and standardization of 0.1N of sodium hydroxide**

HCl + NaOH NaCl + H2O

***Procedure***

1. A- prepare 0.1N of hydrochloric acid in 250 ml d.w.

B- prepare 0.1N of sodium hydroxide in 250 ml d.w.

1. Pipette 10 ml of NaOH solution to a conical flask, and then add 2-3 drops of methyl orange indicator.
2. Titrate the solution with HCl solution in burette until the color change from yellow to red.
3. Repeat the titration process.
4. Calculate the concentration of NaOH solution.

( N x V ) HCl = ( N x V) NaOH

1. Write all Chemical Compounds and all Apparatus used in this experiment.

Experiment :

**Purification of solid compounds by Sublimation**

**Sublimation** is the transition of a substance directly from the [solid](https://en.wikipedia.org/wiki/Solid) to the [gas](https://en.wikipedia.org/wiki/Gas) phase without passing through the intermediate [liquid](https://en.wikipedia.org/wiki/Liquid) phase.

 Sublimation is an [endothermic](https://en.wikipedia.org/wiki/Endothermic) [phase transition](https://en.wikipedia.org/wiki/Phase_transition) that occurs at temperatures and pressures below a substance's [triple point](https://en.wikipedia.org/wiki/Triple_point) in its [phase diagram](https://en.wikipedia.org/wiki/Phase_diagram).

For some substances, such as [carbon](https://en.wikipedia.org/wiki/Carbon) and [arsenic](https://en.wikipedia.org/wiki/Arsenic), sublimation is much easier than [evaporation](https://en.wikipedia.org/wiki/Evaporation) from the melt, because the pressure of their [triple point](https://en.wikipedia.org/wiki/Triple_point) is very high, and it is difficult to obtain them as liquids.

The term *sublimation* refers to a *physical* change of state and is not used to describe transformation of a solid to a gas in a chemical reaction. For example, the dissociation on heating of solid [ammonium chloride](https://en.wikipedia.org/wiki/Ammonium_chloride) into hydrogen chloride and ammonia is *not* sublimation but a chemical reaction.

Sublimation is a technique used by chemists to purified solid compounds, like menthol, naphthalene, iodine, ammonium chloride, etc.

The reverse process of sublimation is desublimation or [deposition](https://en.wikipedia.org/wiki/Deposition_(phase_transition)), in which a substance passes directly from a gas to a solid phase.

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| [Pfeil SO.svg](https://en.wikipedia.org/wiki/File:Pfeil_SO.svg) | | **To** | | |
| [**Solid**](https://en.wikipedia.org/wiki/Solid) | [**Liquid**](https://en.wikipedia.org/wiki/Liquid) | [**Gas**](https://en.wikipedia.org/wiki/Gas) |
| **From** | **Solid** | Solid-solid transformation | [Melting](https://en.wikipedia.org/wiki/Melting) | **Sublimation** |
| **Liquid** | [Freezing](https://en.wikipedia.org/wiki/Freezing) | — | [Boiling](https://en.wikipedia.org/wiki/Boiling) / [evaporation](https://en.wikipedia.org/wiki/Evaporation) |
| **Gas** | [Deposition](https://en.wikipedia.org/wiki/Deposition_(phase_transition)) | [Condensation](https://en.wikipedia.org/wiki/Condensation) | — |

***Procedure:***

1. Weight 1g of impure ammonium chloride compound and put it in a crucible.
2. Weight a pure, dry and empty cover.
3. Cover the crucible, heat for half hour.
4. Cool in desiccators. Weight the cover with pure sublimate compound.
5. Calculate the percentage of purity in that sample.
6. Write all Chemical Compounds and all Apparatus used in this experiment.

Calculation

%purity = (wt. of pure / wt. sample) \* 100

%impurity = (wt. of impure / wt. sample) \* 100

Wt. pure + Wt. impure = wt. sample

%purity + %impurity = 100

Experiment: **Determination of Acetic Acid content Vinegar**

Vinegar is formed by aerobic bacteria oxidizing grain alcohol to acetic acid and water.

More generally, vinegar can be defined as a solution composed of acetic acid [ CH3COOH], water and perhaps other substances. To be sold in stores as vinegar, this solution must contain at least four grams of acetic acid per 100 ml solution.

The objective of this experiment is to measure the total acid concentration in a specific brand of vinegar and to determine if the vinegar meets the minimum acetic acid content.

CH3COOH (aq) + NaOH(aq) CH3COONa (aq) + H2O(aq)

The end point of titration will be detect using a phenolphthalein indicator.

Acetic acid a carbon based compound with a single ionized proton, making it an organic acid of the larger class of organic acids called Carboxylic acid ( organic compounds with a –COOH functional group)

***Procedure:***

1. Fill the burette with 0.1N of NaOH solution to be used as a titrant.
2. Pipette 5 ml of commercial vinegar into the 250 ml volumetric flask and complete the volume by distilled water.
3. Put 10 ml of solution (vinegar solution) in a conical flask and add 2 drops of phenolphthalein indicator.
4. Titer with NaOH solution until formed pink color.
5. Repeat the titration process.
6. Calculate the concentration of acetic acid in 250 ml

( N x V ) CH3COOH = ( N x V) NaOH

NCH3COOH x eq.wt = wt. of CH3COOH in 1 liter

NCH3COOH x eq.wt x (250/1000) = wt. of CH3COOH in 250 ml

1. Calculate the % acetic acid in vinegar

%(w/w) = (wt. acetic acid / wt. of vinegar ) x 100

1. Write all Chemical Compounds and all Apparatus used in this experiment.

Experiment : **Precipitation Titration:**

**Determination of Chloride by the Mohr Method**

Volumetric methods based upon the formation of slightly soluble precipitate are called **" precipitation titration "** .

This method depends on formation of second highly colored precipitate for detect end point.

*Mohr’s method* in which alkaline or alkaline earth chlorides react with silver nitrate in the presence of a few drops of potassium chromate solution as indicator,

is a simple, direct and accurate method for chloride determination.

This method has been widely applied to the titration of chloride ion and bromide ion with silver nitrate , and the indicator is chromate ion (CrO4-2).The end point being signaled by the appearance of red silver

chromate(Ag2CrO4) .

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

Ag+(aq) + Cl–(aq) → AgCl(s)

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

2 Ag+(aq) + CrO4-2 (aq)→ Ag2CrO4(s)

This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water.

The pH of the sample solutions should be between *6.5 and 10*. If the solutions are acidic, *the gravimetric method or Volhard’s method* should be used.

The titration was carried out at a pH between 6.5 and 10 because chromate ion is the conjugate base of the weak chromic acid. Therefore, when the pH is lower than 7, chromate ion is protonated and the chromic acid form predominates in the solution. Consequently, in more acidic solutions the chromate ion concentration is too low to produce the precipitate at the equivalence point. If the pH is above 10, brownish silver hydroxide forms and masks the end point.

The titration is performed in neutral or slightly basic medium pH = (6.5-10) to prevent silver hydroxide formation at pH > 10 or formation of chromic acid at pH<6.5 .

2CrO4-2 + 2H+ 2HCrO4 -1 Cr2O7-2 + H2O

2Ag+ + 2OH- 2AgOH Ag2O(s) + H2O

***Procedure:***

1. Fill the burette with 0.1N of AgNO3.
2. Pipette a 10 mL aliquot of water into a conical flask and add 1 mL of chromate indicator( 5% K2CrO4).
3. Titrate the sample with silver nitrate solution. Although the silver chloride that forms is a white precipitate, the chromate indicator initially gives the cloudy solution a faint lemon-yellow colour.
4. The endpoint of the titration is identified as the first appearance of a red-brown colour of silver chromate.
5. Repeat the titration process.
6. Calculate the chloride concentration in the sample in ppm unit.
7. Write all Chemical Compounds and all Apparatus used in this experiment.

Experiment : **Purification of sodium chloride salt**

Rock salt is sodium chloride mixed with insoluble impurities as SiO2 , Ca++ and Mg++ ions.

***Procedure:***

1. Weight a beaker.
2. Add 5g of the sample to 15ml of distilled water in the beaker and stir until the salt is dissolve, then filtrate .
3. Add few drops of BaCl2 2% solution until the precipitate appears then filtrate again.

Repeat this step until no precipitate appears.

1. Add Na2CO3 5% solution until notice appearance the precipitate, filtrate the mixture.

Repeat this step until no precipitate appears.

1. Add to the last solution drops of HCl 0.2N, until the solution being neutralize by using litmus paper.
2. Evaporate the solution until white precipitate appear.
3. Calculate the percentage of NaCl in the sample .
4. Write all Chemical Compounds and all Apparatus used in this experiment.

Experiment :

**Determination of water of hydration in a hydrate compound**

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| A **Hydrate** is an ionic compound that contain water molecules in its fundamental solid structure.   |  | | --- | | This water in the hydrate (referred to as "*water of hydration*") can be removed by heating the hydrate. When all hydrating water is removed, the material is said to be anhydrous and is referred to as an anhydrate.  http://nobel.scas.bcit.ca/chem0010/labs/4-waterofhydration/images/cuso45h2o.gif copper (II) sulphate **penta**hydrate, http://nobel.scas.bcit.ca/chem0010/labs/4-waterofhydration/images/mgso47h2o.gif magnesiumsulphate **hepta**hydrate,  http://nobel.scas.bcit.ca/chem0010/labs/4-waterofhydration/images/cocl26h2o.gif cobalt (II) chloride **hexa**hydrate  http://nobel.scas.bcit.ca/chem0010/labs/4-waterofhydration/images/sncl22h2o.gif tin (II) chloride **di**hydrate  The dot indicates an attractive force between the polar water molecules and the positively charged metal ion. On heating, the attractive forces are overcome and the water molecules are released leaving behind the anhydrous salt. | |
| Gypsum is a hydrate in which two water molecules are present for every formula unit of CaSO4 in the solid. The chemical formula for gypsum is CaSO4·2 H2O and the chemical name is calcium sulfate dihydrate.  http://nobel.scas.bcit.ca/chem0010/labs/4-waterofhydration/images/cuso45h2otocuso4.gif |

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| http://nobel.scas.bcit.ca/chem0010/labs/4-waterofhydration/images/percentwater.gif |  |  |

Percent error = ( theoretical - experimental / theoretical value) x 100

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| *example* : When a 1.000 g sample of CuSO4 ·5 H2O(s) was heated so that the waters of hydration were driven off, the mass of the anhydrous salt remaining was found to be 0.6390 g.   1. What is the experimental value of the percent water of hydration?   B- Calculate the percent error. ( Cu = 63.55 , S = 32 , O =16 , H =1 g / mol)  ***Procedure:***   1. Weight a pure, dry and empty crucible. 2. put 1g of copper(II) sulphate pentahydrate (hydrate compound)in the crucible. 3. heat the crucibleand its content for half hour(the color change from blue to white and finally to grey) . 4. Cool in desiccators. Weight the crucible with anhydrous compound. 5. Calculate the percentage of water in that sample. 6. Write all Chemical Compounds and all Apparatus used in this experiment. |