LECTURE NOTES

ON

PRACTICAL

1FY2-21/ 2FY2-21 - ENGINEERING CHEMISTRY

B. Tech. I & II Semester

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SCHOOL OF AERONAUTICS

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1FY2-21/2FY2-21

ENGINEERING

CHEMISTRY

LABORATORY

School o f Aeronautic (Neemrana)

INDEX

LIST OF PROJECTS

CHAPTER-1

ENGINEERING CHEMISTRY (CHEMICAL ANALYSIS)

1.1. Introduction to Chemistry Engineering (Chemistry Analysis):

Chemical analysis**,** the study of the chemical composition and structure of substances. More broadly, it may be considered the corpus of all techniques whereby any exact chemical information is obtained. There are two branches in analytical chemistry: qualitative analysis and quantitative analysis. Qualitative analysis is the determination of those elements and compounds that are present in a sample of unknown material. Quantitative analysis is the determination of the amount by weight of each element or compound present. The procedures by which these aims may be achieved include testing for the chemical reaction of a putative constituent with an admixed reagent or for some well-defined physical property of the putative constituent. Classical methods include use of the analytical balance, gas manometer, burette, and visual inspection of color change. Gas and paper chromatography are particularly important modern methods. Physical techniques such as use of the mass spectrometer are also employed. For samples in the gaseous state, optical spectroscopy provides the best technique for determining which atomic and molecular species are present.

1.2. Qualitative chemical analysis:

Qualitative chemical analysis, branch of chemistry that deals with the identification of elements or grouping of elements present in a sample. The techniques employed in qualitative analysis vary in complexity, depending on the nature of the sample. In some cases it is necessary only to verify the presence of certain elements or groups for which specific tests applicable directly to the sample (*e.g.,* flame tests, spot tests) may be available. More often the sample is a complex mixture, and a systematic analysis must be made in order that all the constituents may be identified. It is customary to classify the methods into two classes: qualitative inorganic analysis and qualitative organic analysis.

1.3. Quantitative chemical analysis:

Quantitative chemical analysis, branch of chemistry that deals with the determination of the amount or percentage of one or more constituents of a sample. A variety of methods is employed for quantitative analyses, which for convenience may be broadly classified as chemical or physical, depending upon which properties are utilized. Chemical methods depend upon such reactions as precipitation, neutralization, oxidation, or, in general, the formation of a new compound. The major types of strictly chemical methods are known as gravimetric analysis and volumetric, or titrimetric, analysis.

Learning Objectives

1. **Scientific reasoning and quantitative analysis.** Our majors will be able to apply chemical concepts to solve qualitative and quantitative problems. They will gain proficiency in logical deduction skills through written problems and laboratory work. They will identify problems and generate hypotheses,

develop and implement experimental methods to test their hypotheses, and analyze and interpret the resulting data.

2. **Laboratory practice and safety.** In order to learn the ways in which new scientific knowledge is created, our majors will experience how chemists interpret chemical and physical phenomena through experimental investigation. They will develop and apply the appropriate lab skills and instrumentation to solve chemical problems, while recognizing the uncertainties and error in experimental measurements. Our majors will understand the concepts of safe laboratory practices. They will learn how to dispose of waste appropriately, how to comply with safety regulations, and how to recognize and minimize hazards in the laboratory.
3. **Chemical information and literature skills.** Our majors will be able to use chemical databases and retrieve peer-reviewed scientific literature. They will be able to evaluate critically chemistry-related information from a variety of sources.
4. **Communication skills.** Our majors will gain facility in both written and verbal communication. They will be able to present information in a clear and effective manner, write reports in a scientific style, and use appropriate technology in their communication. They will be able to work effectively in a diverse group to solve scientific problems.
5. **Impact and applications.** Our majors will have access to a broad education in chemistry, including the opportunity to complete a major approved by the American Chemical Society. They will understand how chemical principles are applied to address current problems in a variety of fields, and they will have the background to understand the impact of chemistry on society both locally and globally. They will understand ethical scientific behavior, including its application to their own work as proscribed by the College's Honor Code.

1.4. QUANTITATIVE CHEMICAL ANALYSIS- TITRATION

The described approach to measuring vinegar strength was an early version of the analytical technique known as titration analysis. A typical titration analysis involves the use of a burette to make incremental additions of a solution containing a known concentration of some substance (the titrant) to a sample solution containing the substance whose concentration is to be measured (the analyte). The titrant and analyte undergo a chemical reaction of known stoichiometry, and so measuring the volume of titrant solution required for complete reaction with the analyte (the equivalence point of the titration) allows calculation of the analyte concentration. The equivalence point of a titration may be detected visually if a distinct change in the appearance of the sample solution accompanies the completion of the reaction. The halt of bubble formation in the classic vinegar analysis is one such example, though, more commonly, special dyes called indicators are added to the sample solutions to impart a change in color at or very near the equivalence point of the titration. Equivalence points may also be detected by measuring some

solution property that changes in a predictable way during the course of the titration. Regardless of the approach taken to detect a titration's equivalence point, the volume of titrant actually measured is called the end point. Properly designed titration methods typically ensure that the difference between the equivalence and end points is negligible. Though any type of chemical reaction may serve as the basis for a titration analysis, the three described in this chapter (precipitation, acid-base, and redox) are most common. Additional details regarding titration analysis are provided in the chapter on acid-base equilibria.

A typical burette permits volume measurements to the nearest 0.1 mL

Example :

Titration Analysis:

The end point in a titration of a 50.00-mL sample of aqueous HCl was reached by addition of 35.23 mL of 0.250 M NaOH titrant. The titration reaction is:

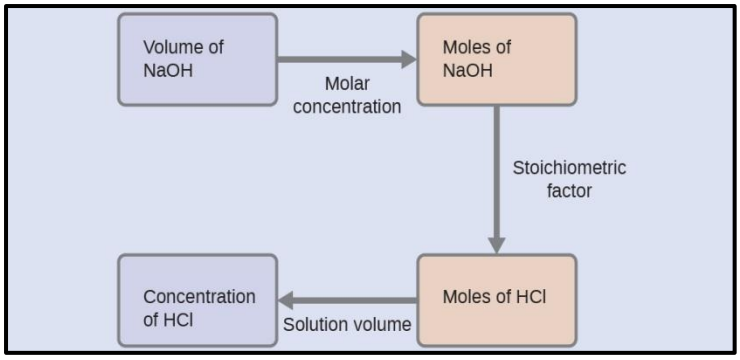
$HCl(aq)+NaOH(aq)\longrightarrow NaCl(aq)+H_2O(l)HCl(aq)+NaOH(aq)\longrightarrow NaCl(aq)+H_2O(l)$

What is the molarity of the HCl?

Solution:

As for all reaction stoichiometry calculations, the key issue is the relation between the molar amounts of the chemical species of interest as depicted in the balanced chemical equation. The approach outlined in previous modules of this chapter is followed, with additional considerations required, since the amounts of reactants provided and requested are expressed as solution concentrations.

For this exercise, the calculation will follow the following outlined steps:



The molar amount of HCl is calculated to be:

35.23 ml NaOH x 0.250 mol NaOH x 1 mol HCL / 1 mol NaOH =8.81X 10^{-3} HCL

Using the provided volume of HCL solution and definition of molarity, HCL concentration is

$M = \frac{8.81 \times 10^{-3} mol\ HCL(n-1)}{50\ ml} = 0.176\ M$

Check Your Learning

A 20.00-mL sample of aqueous oxalic acid, $H_2C_2O_4$, was titrated with a 0.09113-*M* solution of potassium permanganate.

2MnO4−(aq)+5H2C2O4(aq)+6H+(aq)⟶10CO2(g)+2Mn2+(aq)+8H2O(l)2MnO4−(aq)+5H2C2O4(aq)+6H+(aq)⟶10CO2(g)+2Mn2+(aq)+8H2O(l)

A volume of 23.24 mL was required to reach the end point. What is the oxalic acid molarity?

Answer:

0.2648 *M*

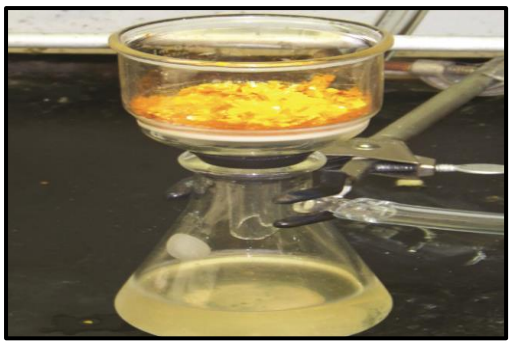
1.5. GRAVIMETRIC ANALYSIS

A gravimetric analysis is one in which a sample is subjected to some treatment that causes a change in the physical state of the analyte that permits its separation from the other components of the sample. Mass measurements of the sample, the isolated analyte, or some other component of the analysis system, used along with the known stoichiometry of the compounds involved, permit calculation of the analyte concentration. Gravimetric methods were the first techniques used for quantitative chemical analysis, and they remain important tools in the modern chemistry laboratory.

The required change of state in a gravimetric analysis may be achieved by various physical and chemical processes.

Types of Gravimetric Analysis:

- Volatilization gravimetry
- Precipitation gravimetry
- Electrogravimetry
- Thermogravimetric



Precipitate may be removed from a reaction mixture by vacuum filtration.

Example:

Gravimetric Analysis

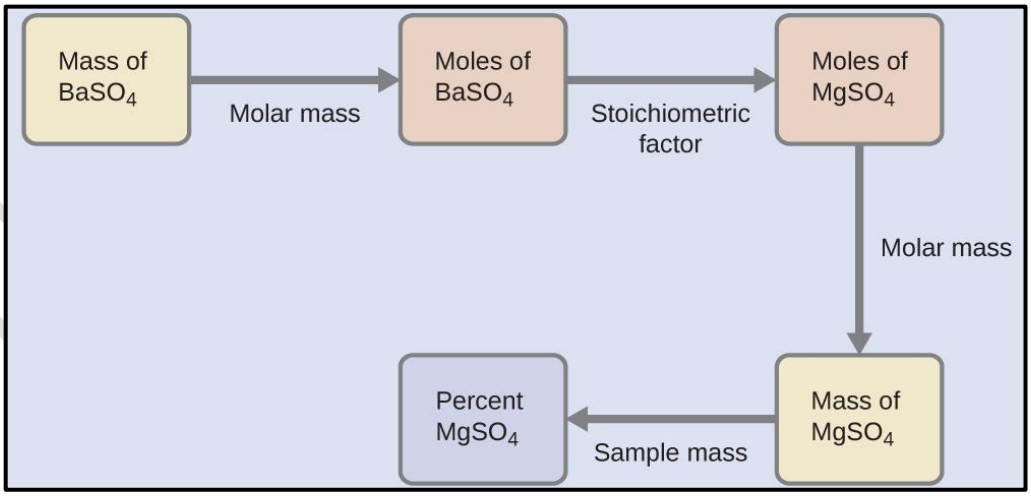
A 0.4550-g solid mixture containing $MgSO_4$ is dissolved in water and treated with an excess of $Ba(NO_3)_2$, resulting in the precipitation of 0.6168 g of $BaSO_4$.

$MgSO_4(aq)+Ba(NO_3)_2(aq)\longrightarrow BaSO_4(s)+Mg(NO_3)_2(aq)MgSO_4(aq)+Ba(NO_3)_2(aq)\longrightarrow BaSO_4(s)+Mg(NO_3)_2(aq)$

What is the concentration (percent) of $MgSO_4$ in the mixture?

Solution

The plan for this calculation is similar to others used in stoichiometric calculations, the central step being the connection between the moles of $BaSO_4$ and $MgSO_4$ through their stoichiometric factor. Once the mass of $MgSO_4$ is computed, it may be used along with the mass of the sample mixture to calculate the requested percentage concentration.



Check Your Learning

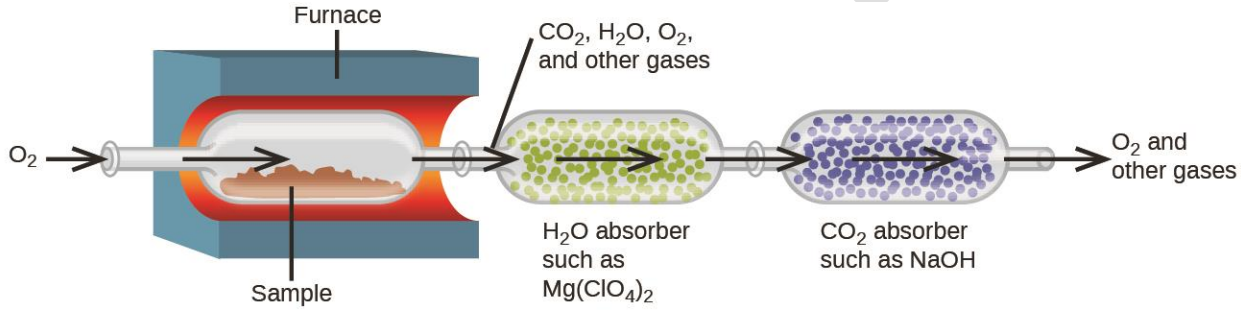
What is the percent of chloride ion in a sample if 1.1324 g of the sample produces 1.0881 g of AgCl when treated with excess Ag^+?

$Ag+(aq)+Cl−(aq)⟶AgCl(s)Ag+(aq)+Cl−(aq)⟶AgCl(s)$

Answer:

23.76%

The elemental composition of hydrocarbons and related compounds may be determined via a gravimetric method known as combustion analysis. In a combustion analysis, a weighed sample of the compound is heated to a high temperature under a stream of oxygen gas, resulting in its complete combustion to yield gaseous products of known identities. The complete combustion of hydrocarbons, for example, will yield carbon dioxide and water as the only products. The gaseous combustion products are swept through separate, preweighed collection devices containing compounds that selectively absorb each product (Figure 3). The mass increase of each device corresponds to the mass of the absorbed product and may be used in an appropriate stoichiometric calculation to derive the mass of the relevant element.



This schematic diagram illustrates the basic components of a combustion analysis device for determining the carbon and hydrogen content of a sample.

1.6. COMBUSTION ANALYSIS :

Polyethylene is a hydrocarbon polymer used to produce food-storage bags and many other flexible plastic items. A combustion analysis of a 0.00126-g sample of polyethylene yields 0.00394 g of CO_2 and 0.00161 g of H_2O. What is the empirical formula of polyethylene?

Solution

The primary assumption in this exercise is that all the carbon in the sample combusted is converted to carbon dioxide, and all the hydrogen in the sample is converted to water:

$CxHy(s)+excess\ O_2(g)⟶xCO_2(g)+y2H_2O(g)CxHy(s)+excess\ O_2(g)⟶xCO_2(g)+y2H_2O(g)$

Note that a balanced equation is not necessary for the task at hand. To derive the empirical formula of the compound, only the subscripts *x* and *y* are needed.

First, calculate the molar amounts of carbon and hydrogen in the sample, using the provided masses of the carbon dioxide and water, respectively. With these molar amounts, the empirical formula for the

compound may be written as described in the previous chapter of this text. An outline of this approach is given in the following flow chart:

Check Your Learning

A 0.00215-g sample of polystyrene, a polymer composed of carbon and hydrogen, produced 0.00726 g of CO_2 and 0.00148 g of H_2O in a combustion analysis. What is the empirical formula for polystyrene?

Answer:

CH

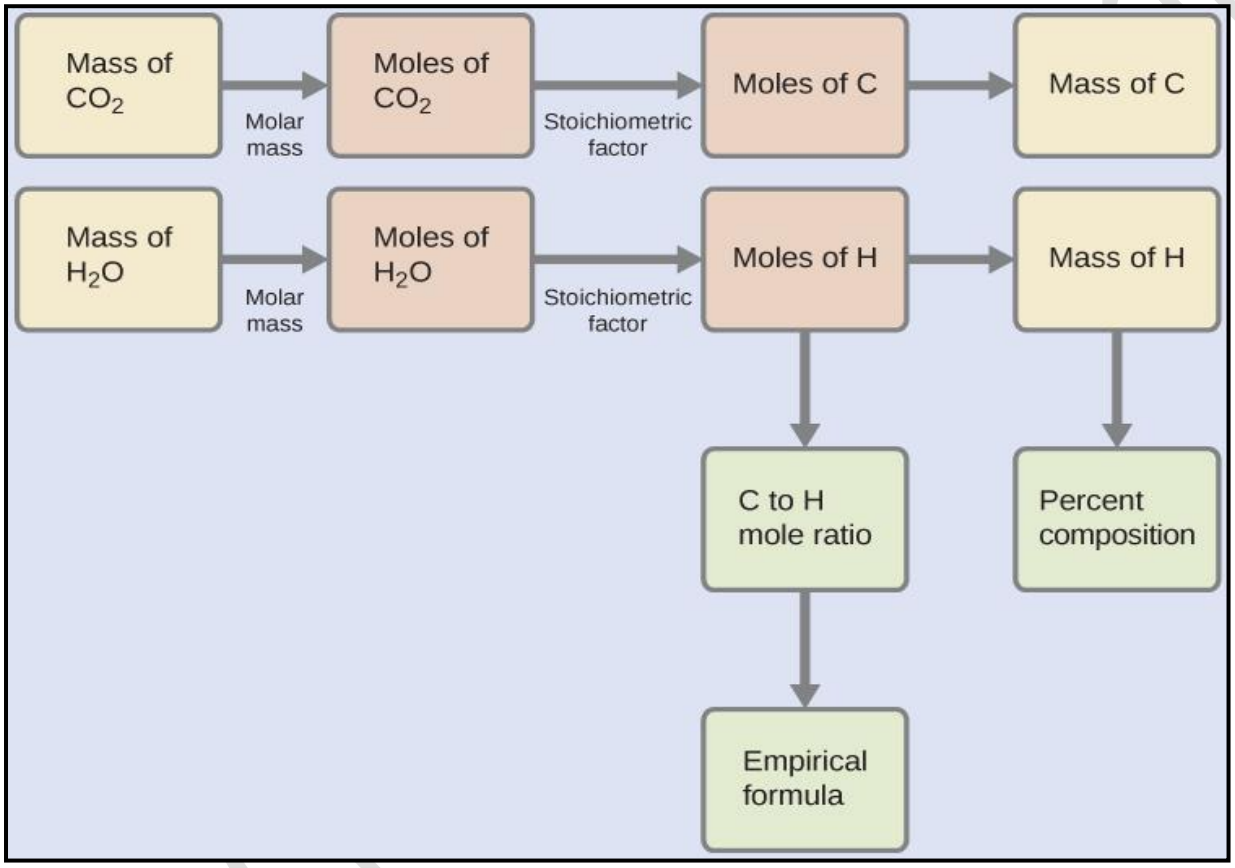


Chart for the calculation of molar amount of carbon and hydrogen in the sample.

KEY CONCEPTS AND SUMMARY

The stoichiometry of chemical reactions may serve as the basis for quantitative chemical analysis methods. Titrations involve measuring the volume of a titrant solution required to completely react with a sample solution. This volume is then used to calculate the concentration of analyte in the sample using the stoichiometry of the titration reaction. Gravimetric analysis involves separating the analyte from the sample by a physical or chemical process, determining its mass, and then calculating its concentration in the sample based on the stoichiometry of the relevant process. Combustion analysis is a gravimetric method used to determine the elemental composition of a compound by collecting and weighing the gaseous products of its combustion.

CHAPTER – 2

TITRIMETRIC ANALYSIS

2.1. Introduction:

The methods of wet chemistry such as titrimetric Analysis have an important role in model analytical chemistry.

Advantages of Titrimetric Analysis:

1. Methods are usually superior to instrumental techniques for major components analysis.
2. Methods are relatively inexpensive with low unit cost for determination.
3. There is wide scope of titrimetric procedures specially for practicing laboratory skill.

Titrimetric Analysis:

The term titrimetric Analysis: refers to quantitative chemical analysis carried out by determining the volume of the solution of accurately noon concentration which required to react quantitatively with measured volume of a solution of substance to be determine. The solution of accurately known strength is called standard solution. The weight of the substance to be determine is calculated from the volume the standard solutions.

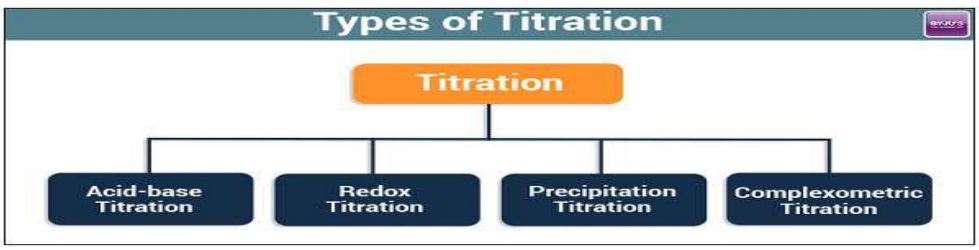
Analytical methods based on volume measuring are named titrimetric or volumetric methods. In titrimetric analyses, we add a reagent of exactly known concentration to a solution of analyzed compound until they completely react – a process called titration. They are highly accurate and precise, rapid and nowadays automated.

Titrimetric methods are one of the oldest, but still widely used in almost all laboratories worldwide in research and industry as well. If you want a rapid determination of soil organic matter or vitamin C in commonly consumed food, you will certainly use titration. In order to do this, you will need a burette – a graduated glass tube with a tap at one end. The first burette was prepared by French chemist Etienne Ossian Henry in 1845 but later was modified by German chemist Karl Friedrich Mohr.

2.2. Types of Titrimetric methods:

Titrimetric methods can be divided based on the type of reaction used into four groups:

1. Acid-base titration
2. Precipitation titration
3. Redox titration
4. Complexometric titration



If one reaction could be used in titrimetry it should meet certain criteria:

a. The reaction must be quantitative, with a high equilibrium constant.
b. The reaction must be rapid. If the reaction is slow, the time of determination is extended, possibly leading to an inaccurate determination.
c. The reaction must have known reaction stoichiometry, in order to calculate the amount of analyte present.
d. There must be some method for observing the endpoint of the reaction.

You must use a standard solution in order to perform a titration. A standard solution is a solution that contains a known concentration of a substance. The accuracy of standard solution preparation greatly affects volumetric determination accuracy. Standard solutions can be divided into two categories:

1. **Primary standard solution** – obtained by diluting the desired amount of the substance in an appropriate solvent.
2. **Secondary standard solution** – obtained by diluting the substance to approximate concentration, and then determine its concentration by titration in a process called standardization.

A primary standard solution can be prepared of stable, pure, non-hygroscopic and accessible substance. If one of these requirements is not fulfilled, a secondary standard must be prepared. In practice, only a few substances meet all of these requirements.

The concentration of the standard solution may change through time, so it must be re-determined from time to time. Also, a solution prepared by diluting standard solution must be standardized again.

2.3 Classification of reactions in Titrimetric analysis:

The reactions employed in titrimetric analysis fall into four main classes.

1. **Neutralization reaction or acidimetric and alkalimeter**: It involves the combination of hydrogen and hydroxide ions to form water.
2. **Complex formation reactions:** It depend upon the combination of ions, other than hydrogen and hydroxide ions, to form a soluble slightly dissociated ion or compound.
3. **Precipitation reaction:** It depends on the combination of ions to form a simple precipitated.
4. **Oxidation reduction reaction:** It involve the change if oxidation number or transfer of electrons among the reacting substances. The standard solutions are either oxidizing or reducing agents.

Neutralization titration

Among representative substances called acids, there are such acids as hydrochloric acid, nitric acid, and sulfuric acid.

These acids, besides both changing blue-colored litmus to red, share the properties of reacting to such metals as magnesium and producing oxygen thereby.

Acid electrolytically disassociates as follows, and produces hydrogen ions (H+).

For example, hydrochloric acid electrolitically disassociates this way:

$HCl \rightarrow H^+ + Cl^-$

Also, alkali is a substance which, in addition to changing red-colored litmus to blue, reacts with acid to cause it to lose its acid qualities.

The representative substances called alkali include sodium hydrate, potassium hydrate, and calcium hydrate.

Alkali electrolitically disassociates as follows, and produces hydroxide ions (OH^-).

For example, sodium hydrate electrolitically disassociates this way:

$NaOH \rightarrow Na^+ + OH^-$

When a sodium hydrate (NaOH) solution is gradually added to hydrochloric acid (HCl), the acidic qualities of the acid slowly weaken, and eventually both the acidic and alkaline qualities are lost. When this state is achieved, it is said to have reached the point of neutralization.

[Hydrochloric Acid] [Sodium Hydrate]

$HCl \Leftrightarrow H^+ + Cl^-$ $NaOH \Leftrightarrow Na^+ + OH^-$

$H^+ + OH^- \rightarrow H_2O$

Looking at hydrogen ion concentration and number of hydroxide ions which have reached the point of neutralization, the amount of hydrogen ions emitted from the acid, and the amount of hydroxide ions emitted from the alkali, have become equal.

The reaction between acid and alkali is called a neutralization reaction, and the method of using this reaction to find the amount of alkali or acid for which the concentration is not known is called neutralization titration.

Precipitation Titration Definition

It is a titrimetric method which involves the formation of precipitates during the experiment of titration. The titrant reacts with the analyze and forms an insoluble substance. The titration is continued till the last drop of the analyze is consumed. When the titrant is excess it reacts with the indicator and signals to terminate the titration process.

Precipitation Titration Example

Here we have discussed an example of precipitation titration.

To determine the concentration of chloride ion in a certain solution we can titrate this solution with silver nitrate solution (whose concentration is known). The chemical reaction Occurs as follows:

$Ag^+(aq) + Cl^-(aq) \rightarrow AgCl(s)$.

AgCl in the form of a white precipitate can be seen settled at the bottom of the flask during titration. The quantity of silver ion used to equivalence point is equal to the quantity of chloride ion which was originally present.

To calculate the number of moles of chloride ion or silver ion we can use n = cV …(molarity definition)

To calculate the volume of the added solution or molar concentration of ion the corresponding values of either of the ions should be known.

2.4. Redox Titration

Redox Titration is a laboratory method of determining the concentration of a given analytes by causing a redox reaction between the titrant and the analytes. These types of titrations sometimes require the use of a potentiometer or a redox indicator.

Redox titration is based on an oxidation-reduction reaction between the titrant and the analytes. It is one of the most common laboratory methods to identify the concentration of unknown analytes.

In order to evaluate redox titrations, the shape of the corresponding titration curve must be obtained. In these types of titration, it proves convenient to monitor the reaction potential instead of monitoring the concentration of a reacting species.

Redox Titration Example

An example of a redox titration is the titration of potassium permanganate ($KMnO_4$) against oxalic acid ($C_2H_2O_4$). The procedure and details of this titration are discussed below.

Titration of Potassium Permanganate against Oxalic Acid

- Prepare a standard Oxalic acid solution of about 250 ml.
- The molecular mass of oxalic acid is calculated by adding the atomic mass of each constituent atom
- The molecular mass of $H_2C_2O_4.2H_2O$ = 126
- Since the weight of oxalic acid that is required to make 1000 ml of 1M solution is 126 g. Hence, the weight of oxalic acid needed to prepare 250 ml of 0.1 M solution = 126/1000 x 250 x 0.1 = 3.15 g

Use of Redox Titration

Ores and Alloy analysis

- The process of metal extraction and refinement requires the monitoring and controlling of different chemicals by several analytical techniques. Therefore, the determination of metal content and that of the auxiliary reagents used is required at different stages in the mining process. Note that the content determination is always related to the economics of the metals mining process.
- In particular, the continuous metal content monitoring during the geological survey is a key decision factor in determining if the ore is worth mining furthermore, the applied analytical techniques aim at process efficiency and safety improvements or the quality and purity of the final products.
- Volumetric analysis is widely employed for ores and alloy analysis. The solution of ore is prepared in appropriate solvent and ore is estimated by employing is suitable titration.

Volumetric methods employed for the determination of Fe in an iron ore/ alloy by redox titration

1. Determination of percentage of Fe in an iron ore of alloy by redox titration using N- phenyl anthranelic acid as terminal indicator or di phenyl amine as internal indicator.
2. To estimate ferrous (Fe^{2+}) and Fe^{3+} iron give in the mixture using $KMnO_4$ solution
3. To determine the iron content in the given iron ore by using external indicator.

Detail of the experiment:

PROJECT-1

Determination of percentage of Fe in an iron ore of alloy by redox titration using N- phenyl anthranelic acid as terminal indicator or diphenyl amine as internal indicator.

Theory:

The estimation of iron in an iron ore or alloy can be done by potassium dichromate titration, iron ore hematite (Fe_2O_3), Magnetite (Fe_3O_40, Sidernite ($FeCO_3$). In place of these ores iron fillings can be used for the determination of Fe

Reaction involved in titration:

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

Apparatus :

Burette, pipette, conical flask , beaker etc.,

Chemicals: $K_2Cr_2O_7$, Diphenyl amine indicator , concentrated sulphuric acid, iron sample.

End Point :

Green to light purple

Procedure:

a. Preparation of the ore/alloy solution :

1. Weigh out accurately to gram of ground dry ore or iron sample a watch glass
2. Transfer it carefully into 100 ml conical flask and add about 10 to 15 ml of conc. H_2SO_4
3. Warm the content gently until the residue is not having any color

b. Preparation of titration solution :

1. Pipette out 20 ml of above solution in conical flask .
2. Add 2 to 3 ml of conc. H_2SO_4 and heat the solution on water bath for 3 minute.
3. Cool the solution and add 2 to 3 drops of diphenylamine indicator

c. Titration with $K_2Cr_2O_7$ solution

1. Prepare N/10 solution of $K_2Cr_2O_7$ by dissolving 4.9 gm of dry solid $K_2Cr_2O_7$ in distil water and make up the volume 1 litre.
2. Dissolve 1gm of diphenylamine in 100 ml of conc.H_2SO_4
3. Resins in fill W rate with 0.1 N $K_2Cr_2O_7$ solution

4. . Take 20 ml of iron solution in conical flask and add few drops of diphenylamine indicator
5. Add $K_2Cr_2O_7$ solution from w rate very slowly till the end point from green to purple appears.
6. Take 3 concrodant readings

d. Observations :

1. Weight of iron sample taken = w gram
2. Volume of iron solution prepared = 250 ml
3. Weight of iron present in 1000 ml = $\frac{1000}{250} \times w$

e. Observation table:

S.No	Volume of the solution taken in the titration flask (ml)	Burette Readings		Volume of the titrant used(Final–Initial reading)(ml)
		Initial Reading (ml)	Final Reading (ml)	
1				
2				
3				

f. Calculation :

(Fe^{2+}) $(K_2Cr_2O_7)$

$N_1V_1 = N_2V_2$

$N_1 \times 20 = \frac{1}{10} \times V_2$

$N_1 = \frac{V_2}{10 \times 20} = \frac{V_2}{200}$

Strength of Fe^{2+} in the solution = $N_1 \times Eq.wt = \frac{V_2}{200} \times 56\ g/L$

Hence, percentage if iron in the ore = $\times \frac{56V_2}{200} \times \frac{100}{x}$

g. **Result** :

The percentage of iron in iron ore = ….%

h. Precautions :

1. $SnCl_2$ solution should be added drop wise and the solution should be shaken after each addition
2. The addition of $HgCl_2$ should be in one lot.
3. If on reduction, no precipitate appears or if grey precipitate of metallic mercury is formed then reject the solution and repeat the process of reduction.

PROJECT-2

To Estimate ferrous (Fe^{2+}) and Fe $^{3+}$ ion give in the mixture using $KMnO_4$ solution

Theory:

First of all ferrous ion are estimated $KMnO_4$ solution after this we reduce ferric ion in the same amount of fresh solution to ferrous state and this time titration against $KMnO_4$ Estimates total iron present in the solution.

Reaction :

Reaction involved in this titration takes place in cold solution $KMnO_4$ behave as self indicator or medium of the reaction in acidic.

Reaction mechanism:

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

$10Fe^{2+} \rightarrow 10Fe^{3+} + 10e^-$

$2MnO_4^- + 16\ H^+ + 10Fe^{2+} \rightarrow 2Mn^{2+} + 10Fe^{3+} + 8H_2O$

Apparatus :

Burette, pipette, conical flask , beaker etc.,

Chemicals :

$KMnO_4$,S_2HO_4, sample of ferrous sulphate

End point :

Dark pink to light pink

Procedure:

i. Pipette out 10 ml of supplied solution and add 10 ml of dilute .H_2SO_4 in it and titrate against standard $KMnO_4$ taken in burette the appearance of light pink colour shows at end.

ii. Take again 10 ml of supplied solution and boil it with zinc and sulphuric acid for 10 minutes so as to convert all ferric iron to ferrous state now this is titrated as above with $KMnO_4$ .Note the end point this also. This shows the ferror iron originally present and ferrous iron obtained by reduction by ferric iron.

Observations :

1. Volume of $KMnO_4$ is used first time = V_1 ml
2. Volume of $KMnO_4$ is used with reduce solution = V_2 ml

Calculation :

V_1 volume of $KMnO_4$ used to oxidized ferrous ion already present, to ferric ion in 10 ml solution

Let N_2 be normality of $KMnO_4$ (say 1/10) and N_1 normality of ferrous ion $N_1 = \frac{\frac{1}{10} \times V_1}{10}$

Strength of ferrous ion in gm/litre

= Eq. wt. ×normlity

$= 56 \times \frac{1}{10} \times \frac{V_1}{10}$

$$= 56 \times \frac{V_1}{100}$$

Similarly for Fe^{+++} ion

Volume of $KMnO_4$ required for ferric ion $V_2 - V_1$

Strength of Ferric ion in gm/litre = $56 \times \frac{1}{10} \times \frac{V - V_1}{10}$

$$56 \times \frac{V - V_1}{100} gm/lit$$

Ferrous Sulphate Crystals

PROJECT-3

To determine the iron content in the given iron ore by using external indicator.

Theory :

Iron sample is dissolved in sulphuric acid. The solution will contain Fe^{3+} ion in form of Ferric sulphate and Fe^{2+} ion in form of ferrous sulphate. This solution is heated with zinc and concentrated sulphuric acid in order to reduce Fe^{3+} ion to Fe^{2+} ion. The resulting solution is titrated against $K_2Cr_2O_7$ solution in presence of acid.

Reaction :

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

Indicator :

Potassium ferricyanide [$K_3Fe(CN)_6$]

End point : blue to color less

Procedure :

a. Reduction of Fe^{3+} to Fe^{2+} in the solution of $FeSO_4$

1.. Take out 20ml of the solution in conical flask add Zinc and sulphuric acid H_2SO_4. Heat the solution to the boiling for 2 minutes

2. Cool the solution and dilute and make the volume again 20 ml by adding distill water.

b. Titration with $K_2Cr_2O_7$

a. Rinse and fill burette with 0.1 N $K_2Cr_2O_7$

b. Place small drops of potassium ferricyanide solution(1%) on a dry white tile by glass rod

c. Titrate the solution in conical flask with $K_2Cr_2O_7$ solution.

d. After adding 1 ml of $K_2Cr_2O_7$ solution in conical flask, withdraw a drop of solution from the flask by glass rod and mix with potassium ferricyanide . If blue colour appears it means end point not reached.

e. Continue the titration with $K_2Cr_2O_7$ until indicator – drop does not give blue colour, end point is reach note the reading

Weight of iron sample ($FeSO_4$) = w gm

Volume of solution is prepared = 250 ml

Weight of $Feso_4$ present in 1000ml = $\frac{1000}{250} \times w$

Note:

Preparation of solutions:

Eq. wt. of $K_2Cr_2O_7$ = 49

N/10 solution of $K_2Cr_2O_7$ = dissolve 4.9 gm $K_2Cr_2O_7$ in 1000 ml of distill water

Indicator solution take a small crystal of [$K_3Fe(CN)_6$] in clean test tube and dissolve the crystal in 1.5 ml of water. Indicator solution should always be prepared fresh.

Observation table :

S.No	**Volume of the solution taken in the titration flask (ml)**	**Burette Readings**		**Volume of the titrant used(Final–Initial reading)(ml)**
		Initial Reading (ml)	**Final Reading (ml)**	
1				
2				

Calculation:

(Fe^{2+}) = $K_2Cr_2O_7$

$N_1 V_1$ = $N_2 V_2$

$N_2 \text{ x } 20 \times \frac{1}{10} \times V_2$

$N_1 \frac{V_2}{10 \times 20} = \frac{V_2}{200}$

Strength if Fe^{2+} in the solution = $N_1 \times Eq.wt. = \frac{V_2}{200} \times 56\ gm/L$

Hence, percentage if iron in iron ore = $\frac{56V_2}{200} \times \frac{100}{x}$

Result the percentage of iron ore =

Related projects on oxidation and reduction process:

- To determine percentage of chromium in the given sample of chromium (III) salt.

Reference : Vogel text book of Quantitative Chemical Analysis – Pg No- 369

- To determine copper in crystallized copper sulphate

Reference : Vogel text book of Quantitative Chemical Analysis – Pg No- 372

- To determine copper in copper (I) chloride

Reference : Vogel text book of Quantitative Chemical Analysis – Pg No- 372

2.5. Iodometric titration :

Iodometric titration (indirect titration) is the titration in which iodine which is produced from a chemical reaction that is titrated with reducing agent.

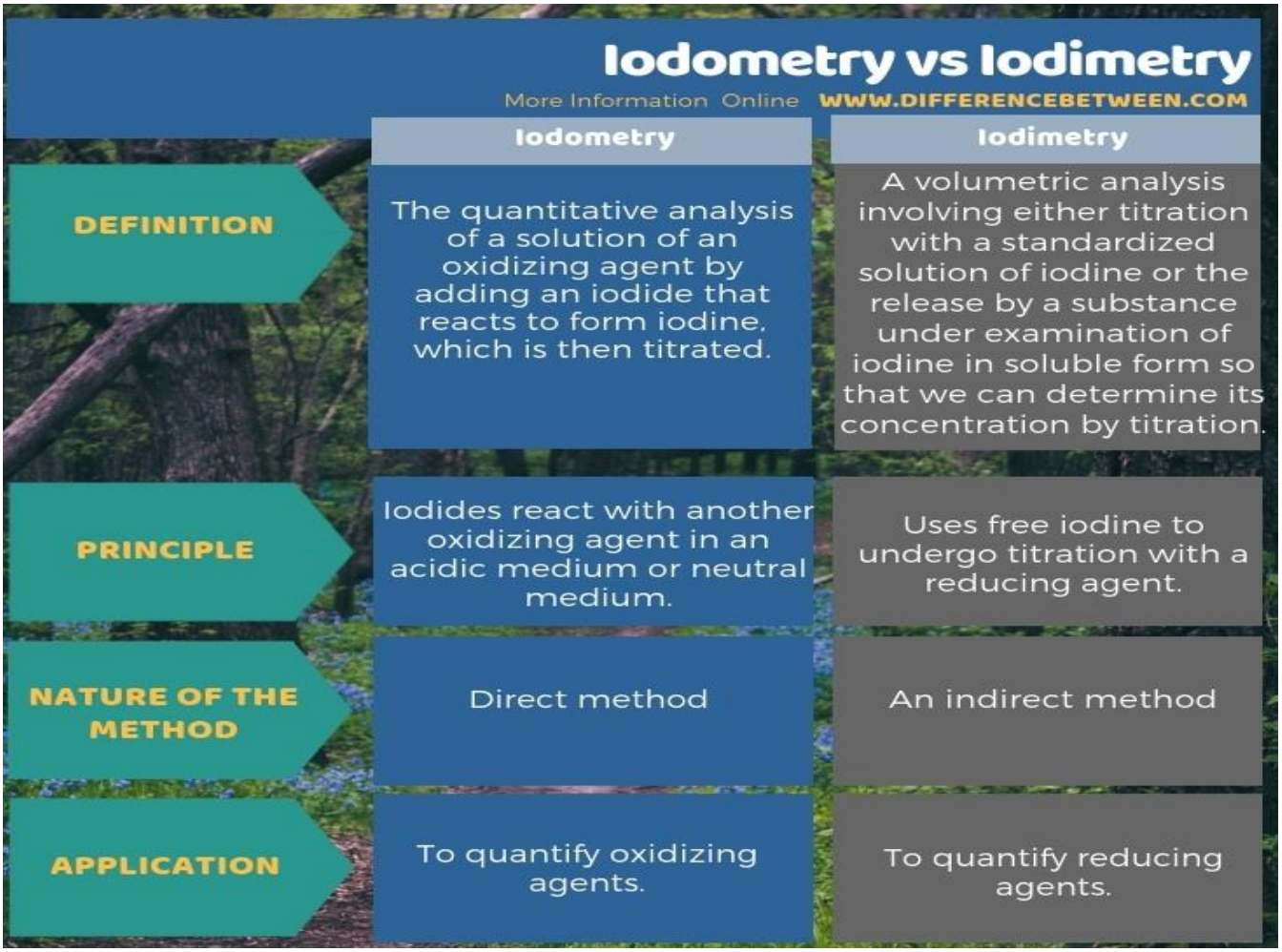
Sodium thiosulphate concentration can be determined by indirect titration as following: Potassium iodate reacts with excess potassium iodide in the presence of sulphuric acid and produce iodine.

KIO3 + 5KI + 3H2SO4 → 3K2SO4 + 3H2O + 3I2

The liberated iodine is then titrated with sodium thiosulphate in the presence of starch as an indicator.

I2 + 2Na2S2O3 → 2NaI + Na2S4O6

So the amount of sodium thiosulphate will equal the amount of iodine, which is equivalent to potassium iodate.



Related projects:

1. Iodometric determination of copper in brass

   Reference : 124 experiment in applied chemistry – Sunita Rathan- Pg No- 124 , Shashi Chawla –Pg No - 151

2. Percentage of copper in crystallized copper sulphate sample.

   Reference : Pg – 380 Vogel quantitative chemical analysis

3. Iodometric determination in copper chloride –

   Reference : pg no 372 Vogel quantitative chemical analysis

4. Determination of iodine content in iodized salts through iodimetric titration with sodium thiosulphate.- Reference : Pg -154 Shashi Chawla

5. To determine the equivalent weight of iron by chemical displacement method (Equivalent of copper weight is 6.5)

   Reference : Pg-167 , Shashi Chawla, Experimental Chemistry.

Project No -4

Iodometric determination of copper in brass

The amount of copper in brass may be found by converting the brass sample to a nearly neutral solution of copper(II) ions, and treating this with potassium iodide. Copper(II) ions are reduced to copper(I), precipitated as the iodide CuI, and iodine is liberated. This can be titrated with standard sodium thiosulphate solution.

The redox reaction between copper metal and concentrated nitric acid is complex, and depends on the concentration of the nitric acid and the temperature of the reaction mixture. Initially, with the commercial concentrated acid (about 70% HNO_3), the reaction is

$Cu + 4H^+ + 2NO_3^-$ à $Cu^{2+} + 2NO_2 + 2H_2O$.

The mixture becomes green owing to complexes of Cu^{2+} and NO_2 being formed, and brown fumes are evolved. When the acid becomes more dilute as the reaction proceeds, reduction to NO is likely instead. None of this affects the eventual outcome, which is a solution of copper(II) aquo ions and nitrate ions together with the aquo ions of the other metals found in the alloy. If this is brass, the diluted reaction product will contain $[Cu(H_2O)_6]^{2+}$ and $[Zn(H_2O)_6]^{2+}$ ions. The latter do not affect the titration.

However, excess of nitrate ions in a strongly acidic solution do; since they are oxidising agents under these conditions, and iodide ions are good reducing agents, any excess of nitric acid will oxidise the iodide ions to iodine and will give an erroneous endpoint:

$2NO_3^- + 4H^+ + 2I^-$ à $I_2 + 2NO_2 + 2H_2O$.

The acidic solution is therefore neutralized with sodium carbonate solution. Since this will cause some precipitation of the metal hydroxides, these are redissolved with a dilute solution of ethanoic acid. This gives a solution acidic enough to keep the metal ions in solution, but not so much so that oxidation of iodide ions occurs.

An excess of iodide ions react with copper(II) ions thus:

$2Cu^{2+}(aq) + 4I^-(aq)$ à $2CuI\ (s) + I_2\ (aq)$

the iodine strictly being present as $I_3^-(aq)$. You might like to look up the standard electrode potentials for the $Cu^{2+}|Cu^+$ and $½I_2|I^-$ half-reactions, and then suggest why the reaction occurs.

The reaction of iodine with thiosulphate ions is

$I_2 + 2S_2O_3^{2-}$ à $S_4O_6^{2-} + 2I^-$.

Method.

1.Weigh accurately about 2.5 g of brass into a 250 cm^3 beaker, and add about 10 cm^3 of concentrated nitric acid (very corrosive!). Allow the beaker to stand in the fume cupboard, and add more nitric acid as necessary to enable complete solution of the metal. You should aim to use as little nitric acid as possible; no more than 20 cm^3 will be needed.

2 Transfer all of the solution so obtained to a 250 cm^3 graduated flask, washing the beaker into the flask with a little pure water.

3 Add 2 mol dm^{-3} sodium carbonate solution drop by drop to the solution in the graduated flask until a faint precipitate just persists. Then add 2 mol dm^{-3} ethanoic acid solution drop by drop until the precipitate just redissolves. Then add pure water to the mark and mix well.

4 Pipette 25.0 cm^3 of this solution into a 250 cm^3 conical flask, add about 10 cm^3 of a 10% solution of potassium iodide, and swirl. Copper(I) iodide will precipitate. Titrate the liberated iodine with standard 0.100 mol dm^{-3} sodium thiosulphate solution until the iodine colour is quite pale (the mixture is of course not clear) and add a little starch indicator. Continue titration until the blue colour is discharged and the mixture is a creamy-white colour. Repeat to obtain three consistent titres.

Apparatus:

Burette , pipette , conical glass, measuring cylinder.

Chemicals:

Sodium thiosulphate solution N/30, Dil. HNO_3 (1:3), starch solution (10 %) , solid Ki , Dil. Acetic acid (1:3), aqueous ammonia sulphuric acid.

Theory:

Brass is a alloy of copper and zinc with an analysis copper in brass is used as a means of identifying any contamination in brass. Determination of copper in brass is done iodometrical for this brass is dissolve in minimum amount of nitric acid

Reaction

$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 2NO + 4\ H_2O$

Cupric ions are there in solution from cupric nitrate, KI is added. This causes release of iodine due to reduction of cupric ion to cuprous ion and cuprous iodide is also precipitated.

Reaction

$3Cu(NO_3)_2 + 4\ KI \rightarrow Cu_2I_2 \downarrow + 4KNO_3 + I_2$

The librated iodine is titrated against standard sodium thiosulphate solution using starch as indicator

$I_2 + 2\ Na_2\ S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$

Starch forms a view complex with iodine.

The end point is detected by sharp disappearance of blue colour.

Procedure :

1. **Preparation of solution of brass sample:**

   Weight out accurately about 0.2 gm of given brass sample and transfer it into a clean titration flask. Add 5 ml of 1:1 nitric acid. Cover the mouth of flask with a watch glass and boil it alloy dissolves. Add 10 ml of distill water and gently boil till the brown fuels of oxides of nitrogen are compile completely.

Destroy nitric acid by adding 1 gm of urea and continue boiling for 2 minutes to compile nitrous fumes. Cool and add 10 ml of distill water. Shake the solution to make it homogeneous.

2. Estimation of copper

Take 10 ml of brass solution in clean conical glass and add ammonia solution drop wise (to remove any mineral acid) till a pale blue precipitate is form. Dissolve in precipitate by adding acetic acid. Add about 1 gm of solid KI. Iodine is release and solution becomes brown. Start adding hypo solution from burette to conical flask. Continue to the mixture turns pennant yellow. Now at 2 ml of starch solution. The mixture becomes dark blue. Add more hypo solution till the blue color just it disappear and solution becomes milky white precipitate formed. This is end point

Observation:

Weight of brass = 0.2 gm

Normality f standard hypo solution = N/30

Observation table:

S.No	Volume of Brass solution titrate (ml)	Burette Readings		Volume of the standard hypo solution (ml)
		Initial Reading (ml)	Final Reading (ml)	

Let concordant volume of hypo solution consumed = V ml.

Calculation :

In this experiment

$2Cu \equiv I_2 \equiv 2S_2O_3^{2-}$

Thus, 1 ml of 1N sodium thiosulphate = 1 milliequivalent of copper

1 equivalent of copper = atomic mass of copper =63.54

Thus, 1 ml of 1 N $Na_2S_2O_3$ = 0.06354 gm of Cu

V ml of $\frac{N}{30}$ $Na^2S^2O^3$solution $= 0.06354 \times V \times \frac{1}{30} gm\ of\ copper$ = X g (let)

Percentage of copper of brass sample $= \frac{x}{0.2} \times 100$

Precautions :

1. It is a must to neutralized copper solution with aqueous ammonia and again acidification dilute acetic acid
2. Starch should be added near the end point when the concentration of iodine go in the solution.

Project -5

Percentage of copper in crystallized copper sulphate sample.

Theory :

Brass is a alloy of copper and zinc with an analysis copper in brass is used as a means of identifying any contamination in brass. Determination of copper in brass is done iodometrical for this brass is dissolve in minimum amount of nitric acid

Reaction:

$3Cu + 8HNO_3 \rightarrow 3Cu(NO_3)_2 + 2NO + 4\ H_2O$

Cupric ions are there in solution from cupric nitrate, KI is added. This causes release of iodine due to reduction of cupric ion to cuprous ion and cuprous iodide is also precipitated.

Reaction

$3Cu(NO_3)_2 + 4\ KI \rightarrow Cu_2I_2 \downarrow + 4KNO_3 + I_2$

The librated iodine is titrated against standard sodium thiosulphate solution using starch as indicator

$I_2 + 2\ Na_2\ S_2O_3 \rightarrow 2NaI + Na_2S_4O_6$

Starch forms a view complex with iodine.

The end point is detected by sharp disappearance of blue colour.

Apparatus:

Burette , pipette , conical glass, measuring cylinder.

Chemicals:

Sodium thiosulphate solution N/30, Dil. HNO_3 (1:3), starch solution (10 %) , $CuSo_4$ solid, solid Ki ,

Procedure :

1. Weigh out accurately about 3.0 gm of salt, dissolve in it water and make up to 250 ml . take out 50 ml of this solution in conical flask and add 1 gm solid KI in it and titrate with standard sodium thiosulphate till the solution becomes pale yellow
2. Add 2 to 3 drops of starch solution in it. And again titrate with sodium thiosulphate till disappearance of blue colour and solution becomes milky white.

Observation:

Weight of $CuSo_4$ = 3 gm

Normality of standard hypo solution = N/30

Observation table:

S.No	Volume of Brass solution titrate (ml)	Burette Readings		Volume of the standard hypo solution (ml)
		Initial Reading (ml)	Final Reading (ml)	

Calculation :

In this experiment

$2Cu \equiv I_2 \equiv 2S_2O_3^{2-}$

Thus, 1 ml of 1N sodium thiosulphate = 1 milliequivalent of copper

1 equivalent of copper = atomic mass of copper =63.54

Thus, 1 ml of 1 N $Na_2S_2O_3$ = 0.06354 gm of Cu

V ml of $\frac{N}{30}$ $Na^2S^2O^3$solution $= 0.06354 \times V \times \frac{1}{30} gm\ of\ copper$ = X g (let)

Percentage of copper of $CuSo_4$ sample $= \frac{x}{0.2} \times 100$

Note : Project 3 & 4 for practice

Project – 6

To determine the equivalent weight of iron by chemical displacement method (Equivalent of copper weight is 6.5) (Reference Pg-167 – Shashi Chawla Experimental Chemistry)

Requirements :

a. Apparatus: iron strip(3.5 cm X 1.5 cm)

b. Beaker, desiccator , burette, pipette , titration flask etc.,

Chemicals :

$CuSo_4$ solution (N/10) , $Cacl_2$, hypo solution 0.1N ,Na_2Co_3 solution , Dilute acetic acid , Solid KI , starch solution

Theory :

When iron strip is allow to stand in contact with copper sulphate solution, some of iron ions goes into solution as $FeSo_4$ and some of copper get deposited on iron strip.

$Fe + CuSo_4 \rightarrow Cu + FeSo_4$

In other words iron chemically displaces the copper from copper sulphate. And chemical displacement conditions:

$$\frac{Equivalent\ weight\ of\ copper}{\text{equivalent weight of Iron.}} = \frac{\text{weight of copper deposited on iron strip}}{\text{Weight of Iron which goes into solution}}$$

Procedure:

1. Take an iron strip and clean with sand paper
2. Weigh the clean iron strip accurately and place it in 250 ml beaker
3. Pour 100 ml (N/10) $Cuso_4$ solution into it. And allow the strip to stand in the beaker for 30 minutes.
4. With the help forceps carefully withdrawn the strip of iron and place it in the desiccators.
5. Estimate quantity of copper sulphate remaining in the solution after chemical displacement iodometricaly with the help of hypo solution
6. Carefully weigh the dried strip of iron

Observation :

1. Initial weight of iron strip = A gm
2. Weight of (iron strip + copper) after drying = B gm
3. Weight of copper deposited on iron strip = C = (initial concentration or strength of copper by titration – final concentration or strength of copper by titration .
4. The weight of iron which goes into the solution (as $FeSo_4$) D= (A+C)-B
5. Equivalent weight of copper = 63.5

Calculations:

$$\frac{Equivalent\ weight\ of\ iron}{equivalent\ weight\ of\ copper} = \frac{D}{C}$$

$$\text{Equivalent weight of iron} = 63.5 \times \frac{D}{C}$$

Result :

Equivalent weight of iron = …….

Precautions:

1. Starch solution should be freshly prepared.
2. Use anhydrous $CaCl_2$ in desiccator.

2.6. Acid base Titration :

Introduction:

An acid base titration is the determination of the concentration of an acid or base by exactly neutralizing the acid o base with an acid or base of known concentration. This allows for quantitative analysis of the concentration of an unknown acid or base solution. It makes use of the neutralization that occurs between acids and bases.

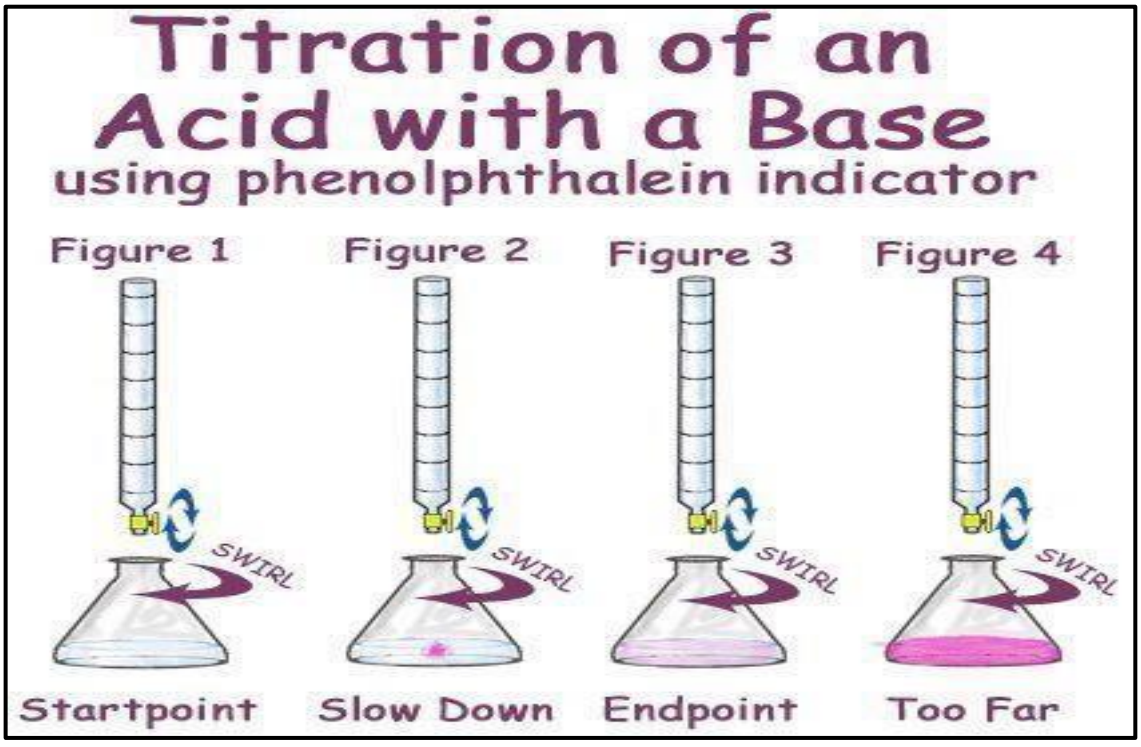
Acid – base titration can also be used to find percent purity of chemical.

Alkalimetry and Acidimetry

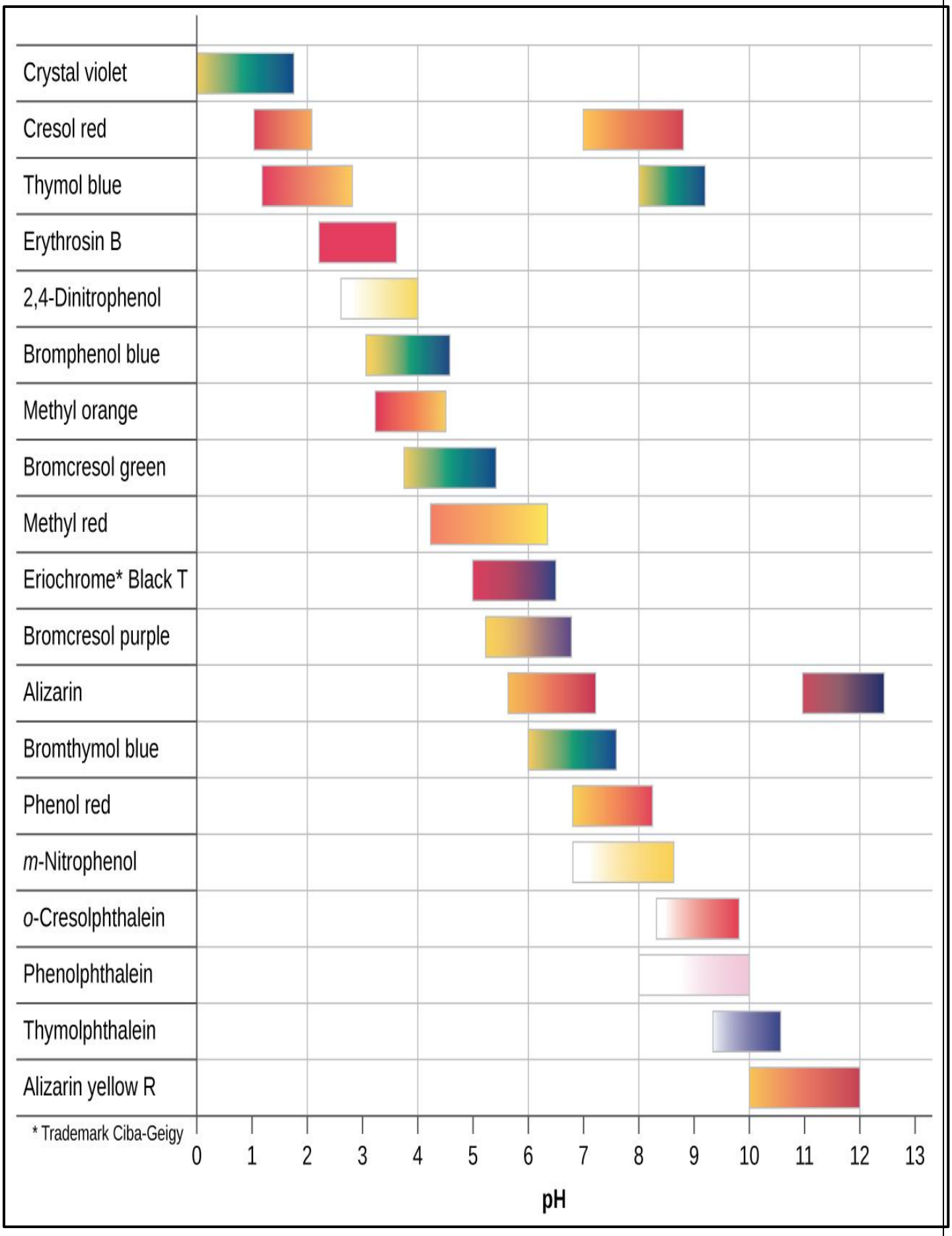
Alkalimetry and Acidimetry are a kind of volumetric analysis in which the fundamental reaction is a neutralization reaction. Alkalimetry is the specialized analytic use of a basic . Substance. Acidimetry, sometimes spelled Acidimetry, is the same concept of specialized analytic acid – base titration, but for an acidic substance.

Method:

Before starting the titration a suitable pH indicator must be chosen. The equivalence point of the reaction, the point at which equivalent amounts of the reactant have reacted, will have a ph dependent on the relative strengths of the acid and base used.



Titration of Acid with a Base



Indicators Vs pH

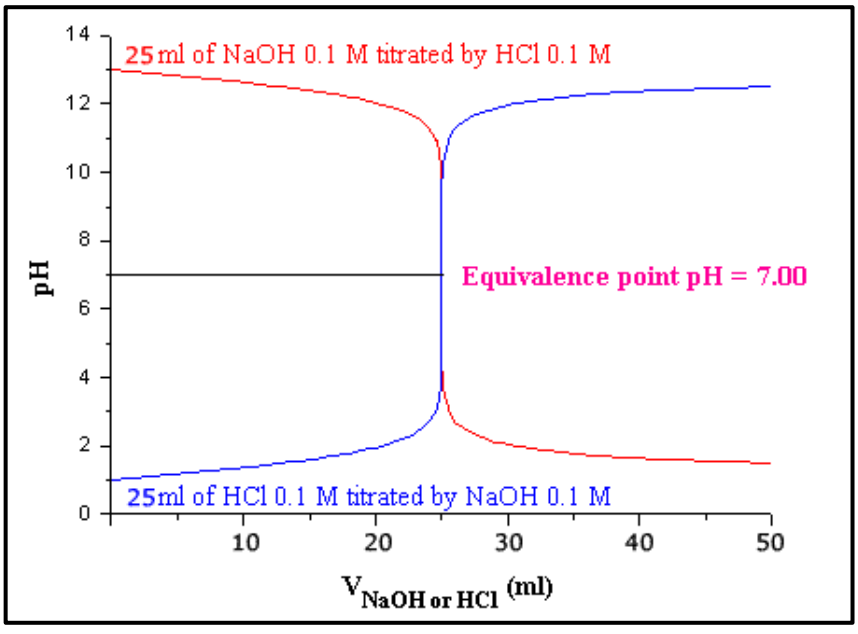
Types of Acid- base titration:

1. Strong acid – strong base titration
2. Weak acid – strong base titration
3. Strong acid – weak base titration
4. Titration of weak acid – weak base
5. Titration of Na_2CO_3 with HCL

Strong acid – strong base:

The change of pH with the volume of alkali added in case of titration of strong acid strong base. It is seen that pH changes very slowly at first, increases rapidly near the equivalence point. At the equivalence point there is sharp change in pH sharp increase in the pH solution very small amount of alkaline added. This sharp sudden change in ph from 4- 10 near equivalence point is very significant because all the indicators which color change in pH range 4 to 10 can be used to detect the end point thus methyl orange , methyl red phenolphthalein can be used as indicators for detecting end point for titration . Involving strong acid and strong base. After the equivalents points, and further addition of alkali, pH again changes slowly.

Note: it may emphasized here that if the solution or dilute (0.01m) but change in pH and equivalence point is not very sharp. pH range is limited to 5.5 – 5.8 at equivalence point. And hence indication such as methyl red or bromothimoide. The equivalence point in all cases whether solution is dilute or concentrated d in titration of strong acid and strong base remain the same. That is **7**



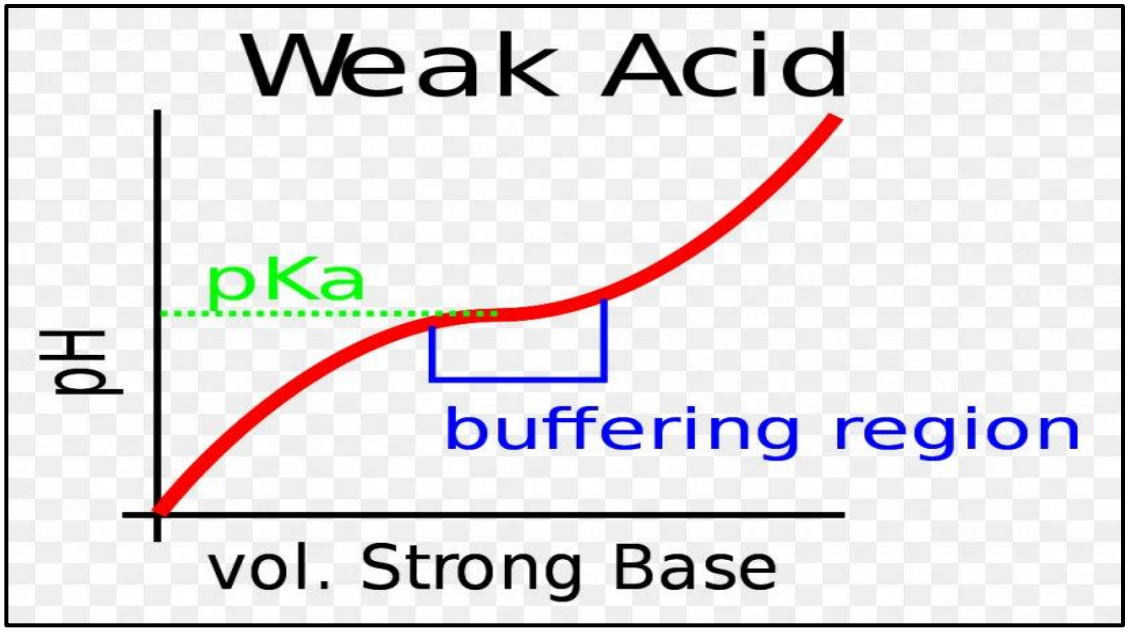
Strong acid – strong base:

Strong acid – weak base:

The titration weak base strong acid (ammonium hydroxide Vs hydrochloric acid0the pH of equvalnce point in this is lies in acid range that is below 7. This is due to formation of a salt of weak base and strong acid (NH_4Cl) which undergoes hydrolysis releasing H^+ ion in the solution. Thus the pH change at equivalence lies in the range of 6 to 4 . Thus the indicator having pH range on acid side that is methyl red or methyl orange can be used successfully.

Weak acid – strong base titration

Titration of weak acid with strong base (CH_3COOH Verses NaOH) pH at equivalence point will not be 7 because of hydrolysis or odium additate so formed. And give excess of OH^- ion in aqua solution there by increasing the pH at equivalence points. Thus indicator used should have pH range slightly on alkaline side thus phenolphthalein or thymolblue would be satisfactory indicator



weak acid – weak base

In these type of titration the pH changes during the titration are continuous that no sharp pH change is observed and hence none of the indicator is suitable. However a mixed indicator may be used

Titration of Na_2Co_3 with HCL

When a Na_2Co_3 is titrated with the strong acid such hydrochloric acid, the carbonate ions are first converted to bicarbonate ion and then to carbonic acid.

Formula :

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

$HCO_3^- + H^+ \rightarrow H_2CO_3$

As the neutralization takes place in two step , so two indicators will be used.

Step 1- the first equivalence points corresponse to neutralization of Co_3^{2-} to HCO_3^- stage. This is completed at the pH 8.5 hence phenolphthalein to use detect the conversion of Co_3^{2-} to HCO_3^- stage

Step 2 – the second equivalent point indicate the neutralization of HCO_3^- to $H_2\ CO_3$ stage. This reaction gets completed at pH 4.3. thus, methyl orange can detect the end point in this case that is complete neutralization of CO_3^{2-} ion. This behaviour of Na_2Co_3 and Na HCO_3 is used in estimation of mixture of Na_2Co_3 and Na HCO_3.

Project -7

To determine the percentage composition of a given mixture of NaCl and NaOH, 8 gms of which is dissolved per litre of solution.

Reference : Pg – No 31 – Experiment in Applied chemistry by Sunita Rattan.

Experiment :

Requirement : a standard solution of 0.1 N HCL

Apparatus : Burette , Pipette , Beaker, Conical Flask

Theory :

For the titration of solution of NaOH and NaCl , HCL solution is required. NaOH react with HCL solution and give NaCl + H_2O

Indicator : phenolphthalein (as strong acid is reacting with strong base:

End point – pink to colour less

Procedure :

1. Rinse the burette with given 0.1 N solution with HCL
2. Fill the burette with the HCl solution .
3. Pipette out 10 ml solution of Nacl + Na OH in the conical glass and add a drop of phenolphthalein indicator.
4. Titrate the solution with HCl solution till the solution changes colour pink to colourless

Observation :

Normality of HCL solution = 0.1 N

Volume of mixture solution taken = 10 ml

Observation table :

S.No	Volume of solution taken in titration flask (ml)	Burette Readings		Volume of the HCL solution used
		Initial Reading (ml)	Final Reading (ml)	

Calculation

Acid mixture (NaOH)

$N_1V_1 = N_2 V_2$

$\frac{1}{10} \times x = N_2 \times 10$

$N_2 = \frac{x}{100}$

Strength = normality × Eq.wt. = $\frac{x}{100} \times 40$ = y gm/L

The amount of NaOH in mixture = y gm/L

Amount of NaCl in the mixture = (8-y)gm/L

Percentage of NaOH = $\frac{y}{8} \times 100$

Precaution

1. Rinse the burette and pipette with respective solution.
2. A mixture NaOH and NaCl → dissolve 4 gm NaOH and 4 gm NaCl in 1000 ml distill water.

Project – 8

Percentage in ammonia in ammonium salt

Theory :

When ammonium salt(except carbon and bicarbonate) is decomposed by boiling with the excess of sodium hydroxide solution ammonia is liberated according to the equation .

If known quantity of NaOH (in excess of that required to decomposed diammonium salt) is used after decomposition is complete, an unused NaOH is determined, the difference between these quantities gives the amount of NaOH equivalent to the ammonium salt taken.

Procedure:

Weigh accurately about 1 or 2 gm of ammonium salt and transfer it into a 250 ml beaker and dissolve in about 75 ml of water. Add 50 ml N- NaOH solution. Which is more than a sufficient to decompose the ammonium salt and boil gently to expel the ammonia . at intervals about 5 minutes, test for ammonia in the escaping steel with a piece of moist litmus paper, the test paper being held outside the beaker. Boil for half an hour. When the decomposition is complete that is no ammonia is detected in the vapors. Cool the solution and transfer it to a 250 ml conical flask. Dilute the solution upto 250 ml.

Fill the burette with N/10 HCL. Pipette out 25 ml of above solution into conical flask. Add 3 drops of methyl orange as indicator. Add slowly HCL solution from burette till pink colour is just obtained note the reading.

Calculation :

Mass of salt taken = w gm

Volume of N-NaOH added = 50 ml

Volume made up after boiling the solution = 250 ml

Volume of solution taken for titration = 25 ml

Volume of N/10 HCL require to titrate unused alkali = V ml

Volume of N- NaOH left after neutralization of ammonium salt in 25 ml solution ≡ V ml of N/10 HCL

≡ V ml of N/10 NaOH

Volume of NaOH left unreacted in 250 ml of solution = $\frac{250\times V}{25\times 10} = V\ ml$

Volume of NaOH reacted with ammonium salt = (50- V)ml

1 gm equivalent NaOH ≡ 1 gm Equivalent NH_3

1000 ml N-NaOH ≡ 17 gm NH_3

(50-V) ml N-NaOH ≡ $\frac{17\ (50-\mathrm{V})}{1000}$ gm NH_3

Amount of NH_3 ammonium salt = $\frac{17\ (50-\mathrm{V})}{\mathrm{w}\times 1000}$ gm

Percentage (W/W)of NH_3 salt = $\frac{17\ (50-\mathrm{V})100}{\mathrm{w}\times 1000}$

Reference :

Practical Chemistry – O.P.Pandey – Pg No- 93

2.7. Complexometric Titration Complexation Titration

The process of complex ion formation can be described by the general term complexation (complex ion is formed by the union of simple ion or with other ions of opposite charge or with neutral molecule).

Many metals formed complexes with the reagents which are called legends may be conveniently classified on the basis of number of points of attachment to the metal ions.

Example of multi dentate legend contains more than to coordinating atoms per molecule for example Ethylene Diamine tetra acetic acid or EDTA.

EDTA has two donor nitrogen atoms and four donor oxygen atoms in the molecule, so it can be Hexadentate.

Points to be noted:

The stability of multi dentate legend is more as compare to corresponding complex with mono dentate legend. That is greater the number of points of attachment of legend to the metal ion, greater the stability of the complex.

The chelating effect is important. It is define as enhanced infinity of chelating legend for a metal ion.

Objectives:

1. Define the terms associated with metal-ligand complexes,
2. Discuss the formation and stability of metal-ligand complexes,
3. Explain the principle of complexometric titrations
4. Enlist and explain different methods of detecting the end point in complexometric titrations,

Principles of complexometric titration:

Complexometric titrations have the advantages of complex formation and at the same time suffer from the limitations of titrimetric methods. For example, although the complex formed is undissociated, it does not suffer from co-precipitation errors as in the case of precipitation titrations. The fact that a complexing agent coordinates with only certain metal ions i.e., it shows selectivity is an added feature of the complex formation. However, on the flip side, the stoichiometry of the complex is not well defined as in a redox, neutralization, or precipitation titration. Further, if the complexing titrant is an organic compound, we need to be careful about the solubility properties of the complex. As mentioned earlier, EDTA is probably the most versatile and exploited titrant in complexometric titrations.

Effect of pH:

During a complexometric titration, the pH must be constant by use of a buffer solution. Control of pH is important since the H^+ ion plays an important role in chelation. Most ligands are basic and bind to H^+ ions throughout a wide range of pH. Some of these H^+ ions are frequently displaced from the ligands (chelating agents) by the metal during chelate formation.

Equation below shows complexation between metal ion and H^+ ion for ligand:

$M^{2+} + H_2\text{- EDTA}^{2-} \leftrightarrow M\text{- EDTA}^{2-} + 2H^+$

Thus, stability of metal complex is pH dependent. Lower the pH of the solution, lesser would be the stability of complex (because more H^+ ions are available to compete with the metal ions for ligand). Only metals that form very stable complexes can be titrated in acidic solution, and metals forming weak complexes can only be effectively titrated in alkaline solution.

Mechanism of action of indicator:

Let the metal be denoted by M, indicator by I and chelate by EDTA. At the onset of the titration, the reaction medium contains the metal-indicator complex (MI) and excess of metal ion. When EDTA titrant is added to the system, a competitive reaction takes place between the free metal ions and EDTA. Since the metal-indicator complex (MI) is weaker than the metal-EDTA chelate, the EDTA which is being added during the course of the titration is chelating the free metal ions in solution at the expense of the MI complex. Finally, at the end point, EDTA removes the last traces of the metal from the indicator and the indicator changes from its complexed colour to its metal free colour. The overall reaction is given by:

MI + M + EDTA → M-EDTA + I

(Colour of metal Indicator complex) (Original colour of indicator)

Structures of some important indicators used in complexometric titrations are given in Fig. Many compounds have been used as indicators like:

a. Triphenyl methane dyes
b. Phthalein and substituted phthaleins
c. Azo dyes
d. Phenolic compounds

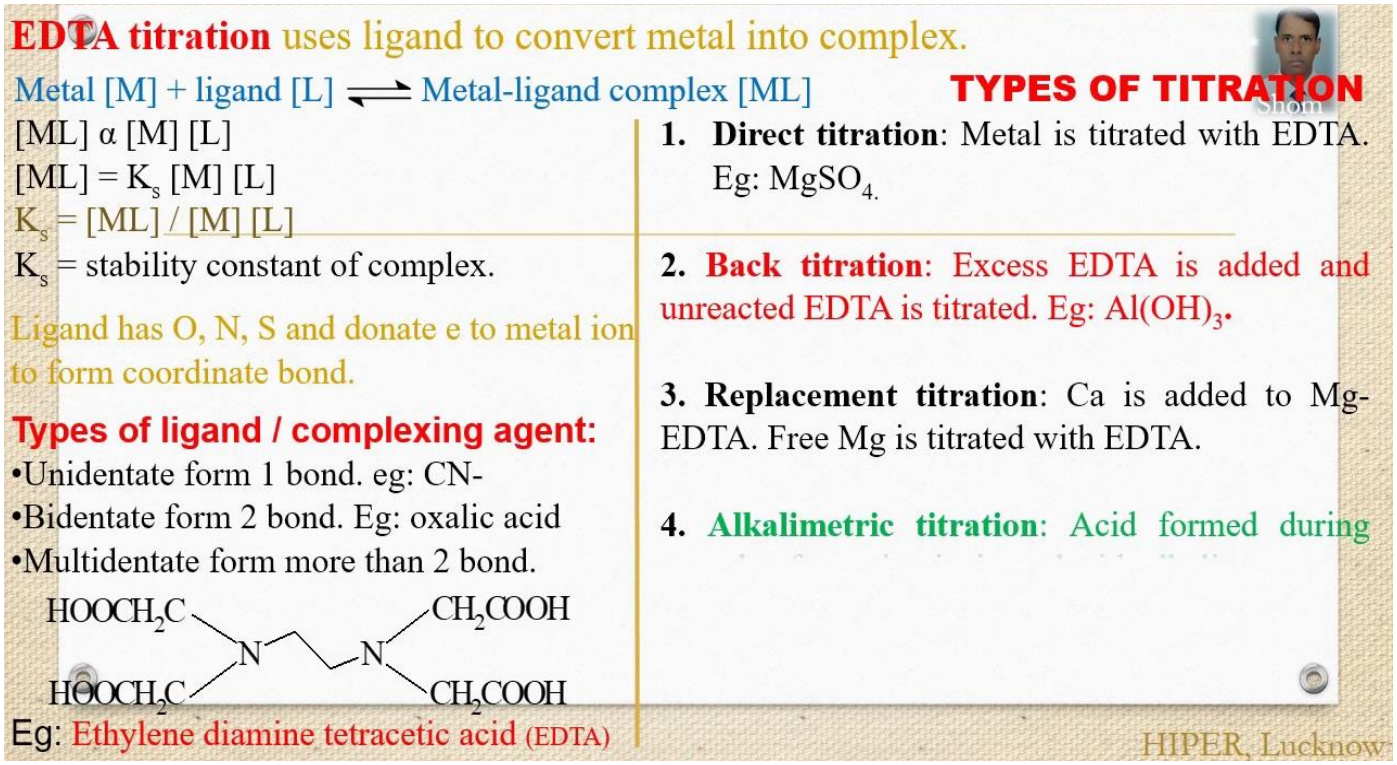
some oxidation - reduction indicators

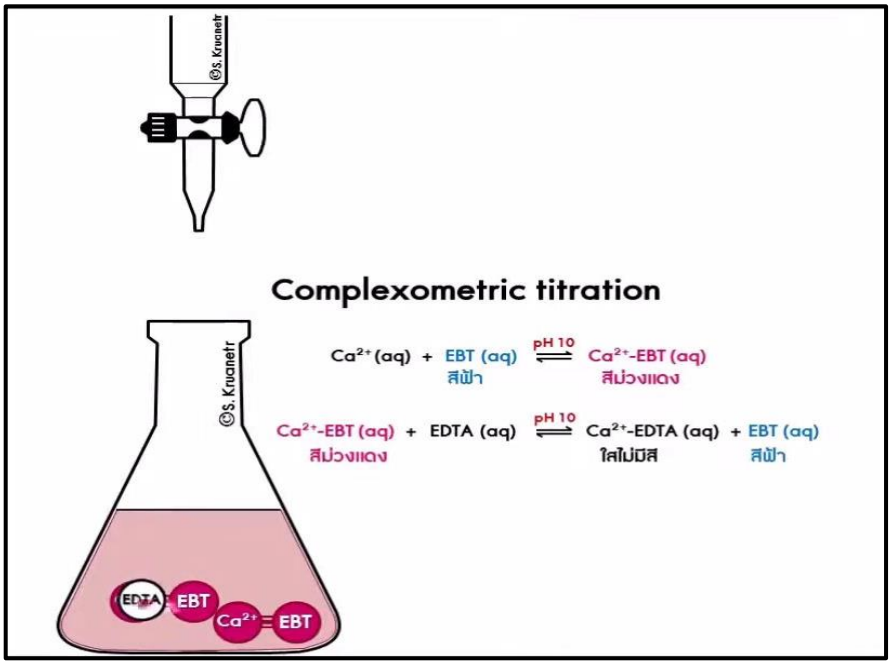
Indicator	**Colour change**		**Formal potential V at pH =0**
	Oxidized form	**Reduce form**	
5- Nitro-1, 10- Phenanthroline iron(II) sulphate (nitroferrion)	Pale blue	Red	1.25
1,10 Phenanthroline iron(II) sulphate (ferrion)	Pale blue	Red	1.06
N- Phenylanthralenic acid	Purple red	Colourless	0.89
Diphenylamine sulphonic acid	Red violet	Colourless	0.85
Diaphenylamine	Violet	Colourless	0.76

Starch- I_3^-, Ki	Blue	Colourless	0.53
Mythlene blue	Blue	Colourless	0.52

Important Indicator for Volumetric analysis

S.No	Name of the Indicator	Colour change	pH range	pH range
1.	Mordant black II	Red to Blue	6-7	Ca,Ba,Mf,Zn,Mn,Pb,Hg
	Eriochrome black T			
	Solochrome black T			
2	Murexide or Ammonia purpurate	Violet to Blue	12	Ca,Cu,Co
3	Catechol- violet	Violet to Red	8-10	Mn,Mg,Fe,Co,Pb
4	Methyle Blue	Blue to Yellow	4-5	Pb,Zn,Cd,Hg
	Thymol Blue	Blue to Grey	10-12	
5	Alizarin	Red to Yellow	4.3	Pb,Zn,Co,Mg,Cu
6	Sodium Alizarin sulphonate	Blue to Red	4	Al, Thorium
7	Xylenol orange	Lemon to Yellow	1-3	Bi, Thorium





Preparation of indicators solution:

- **Diphenylamine** : dissolve 1 gm of substance in 100 ml conc. Sulphuric acid
- **EBT** – dissolve 1 gm of pure dye in 100 ml of methanol.
- **Methyl orange** – dissolve 50 mg of methyl orange in distill water and volume 100 ml with distill water and filter.
- **Starch solution** – dissolve 1 gm of powdered starch with distill water to from a paste and add 100 ml of boiled water in it with constant stirring, boil for 5 minutes and cool.
- **Iodine solution** – dissolve 20 gm of Ki in 100 ml distill water.
- **Phenolphathalin**- dissolve 10 gm of substance in 1 litre of 50 % alcohol.
- **Alcoholic caustic potass solution** – dissolve 20gm of potassium hydroxide 1 litre of 80% alcohol.
- **Caustic potass**- dissolve nearly 31 gm potassium hydroxide in 1 litre distill water.
- **Lime water** – dissolve 1.7 gm of calcium oxide or calcium hydroxide in 1 litre distill water.
- **Caustic soda** dissolve 220 gm of sodium hydroxide in 1 litre distill water.
- **Potassium ferricyanide** – dissolve 55 gm of substance in 1 litre distill water.
- **Methylthymol Blue**- Thymolsulph&phthalein, gives a blue colour with **Ca** and **Mg** ions in alkaline solution

Equilibrium involved in EDTA Titrations:

Ethylenediaminetetraacetic acid, or EDTA, is an amino carboxylic acid that is a versatile titrant which can be used for the analysis of virtually all metal ions. It is a Lewis acid, having six binding sites (four ionised carboxylate groups and two lone pairs on the amino groups), providing six pairs of electrons. In typical analytical determinations completely deprotonated molecule of EDTA forms up to six coordination bonds

with a single metal ion. It is accomplished by donation of the lone pairs of electrons to empty orbital's existing on the metal ion.

The resulting product of this reaction is a metal-chelate complex in which EDTA forms a cage-like structure around the metal ion. The actual number of coordination sites depends on the size of the metal ion; however, all metal–EDTA complexes have a 1:1 stoichiometry, irrespective of the valency of the ion as shown below.

$M^{2+} + [H_2Y]^{2-}$ [MY]2 - + 2H+

M3+ + [H2Y]2- [MY] - + 2H+

M4+ + [H2 Y]2- [MY] + 2H+

The generalized reaction between the metal ion and the EDTA can be described as given below.

Y 4 − + Mn+ MY n−4

Where, Y4– is a shorthand notation for the fully dissociated molecule of EDTA. The formation constant for the complex will be given as following.

[MY] n 4 n 4 f + − − K

For example, the formation of a metal-EDTA complex with Cd2+ can be represented as

Cd2+ (aq) + Y4– (aq) CdY2– (aq)

The equilibrium constant (better called as formation constant) for the reaction is given as follows and has a value of 2.9 × 1016 implying that the complex is quite stable and the reaction goes far to the right.

[Cd [] Y] [CdY] 2 4 2 f + − − K

The formation constant for the complexes formed by EDTA with different metal ions are compiled in Table

The formation constants of metal-EDTA complexes

Ion	log Kf (formation constant
Li^+	+2.79
Na^+	1.66
K^+	0.8
Be^{2+}	9.2
Mg^{2+}	8.79
Ca^{2+}	10.69
Sr^{2+}	8.73
Cu^{2+}	18.81
Pb^{2+}	18.04
Fe^{3+}	25.1
Zn^{2+}	16.50

Equivalent with of substances used in volumetric Analysis

Sodium carbonate	Na_2Co_3	MW/2= 53.06
Sodium bicarbonate	$NaHCo_3$	MW/1 = 84
Potassium Carbonate	K_2CO_3	MW/2 = 69
Oxylic Acid hydrated	$H_2C_2O_4.\ 2H_2O$	MW/2= 63
Ferrous sulphate anhydrous	$FeSO_4$	MW/1= 152

Reference: Vogel's text book of Quantitative Chemical Analysis

Table 10.5 *Summarised procedures for EDTA titrations of some selected cations*

Metal[a]	Titration type[a]	pH	Buffer	Indicator[b]	Colour change[c]	
Aluminium	*back*	7–8	NH_3 (aq)	SB	B	R
Barium[d]	*direct*	12		MTB	B	Gr
Bismuth	*direct*	1		XO	R	Y
	direct	0–1		MTB	B	Y
Cadmium	direct	5	Hexamine	XO	R	Y
Calcium	direct	12		MTB	B	Gr
	substn	7–11	NH_3 (aq)/NH_4Cl	SB	R	B
Cobalt[e]	direct	6	Hexamine	XO	R	Y
Iron(III)[e]	*direct*	2–3		VB	B	Y
Lead	direct	6	Hexamine	XO	R	Y
Magnesium[f]	direct	10	NH_3 (aq)/NH_4Cl	SB	R	B
Manganese[g]	direct	10	NH_3 (aq)/NH_4Cl	SB	R	B
	direct	10	NH_3 (aq)	TPX	B	PP
Mercury	direct	6	Hexamine	XO	R	Y
	direct	6	Hexamine	MTB	B	Y
Nickel	*direct*	7–10	NH_3 (aq)/NH_4Cl	M	Y	V
	direct	7–10	NH_3 (aq)/NH_4Cl	BPR	B	R
	back	10	NH_3 (aq)/NH_4Cl	SB	B	R
Strontium	direct	12		MTB	B	Gr
	direct	10–11		TPX	B	PP
Thorium	direct	2–3		XO	R	Y
	direct	2–3		MTB	B	Y
Tin(II)	direct	6	Hexamine	XO	R	Y
Zinc	direct	10	NH_3 (aq)/NH_4Cl	SB	R	B
	direct	6	Hexamine	XO	R	Y
	direct	6	Hexamine	MTB	B	Y

[a] Items in italics are covered in Sections 10.68 to 10.73.
[b] BPR = bromopyrogallol red; M = murexide; MTB = methylthymol blue; SB = solochrome black; TPX = thymolphthalexone; VB = variamine blue; XO = xylenol orange.
[c] B = blue; Gr = grey; PP = pale pink; R = red; V = violet; Y = yellow.
[d] Can also be determined by precipitation as $BaSO_4$ and dissolution in excess EDTA.
[e] Temperature 40 °C.
[f] Warming optional.
[g] Add 0.5 g hydroxylammonium chloride (to prevent oxidation) and 3 mL triethanolamine (to prevent precipitation in alkaline solution); use boiled-out (air-free) water.

Experimentation in Complexometric titration :

Project No-9

Barium Ion detection with EDTA titration (direct titration)

Procedure:

Steps in titration:

Step 1- preparation of SHW (Standard hard water solution) dissolve 1 gram of $CaCo_3$ minimum quantity of dilute HCL then dry it by slow heating. Dissolve this dry mass in distill water and make this solution 1 litre. Strength of SHW= 1000 ppm or 1gm/L

Step 2 – Preparation of barium ion solution Dissolve 1.27 gm of $BaCl_2$ in distill water. this is m/100 solution of $BaCl_2$ molecular weight of $BaCl_2$ is 127 gm/ mole. Dissolve 1.27 gm $BaCl_2$ in 1000 ml distill water

Step 3 – 1M sodium hydroxide solution – Molecular weight of sodium hydroxide is 40gm/mole. Dissolve 40 gm sodium hydroxide in 1000 ml distill water or 4gm NaOH dissolve in 100 ml distill water it is 1M sodium hydroxide solution.

Step 4 – Indicator Methyle Thymole blue + potassium Nitrate mixture

Step 5- End point blue to grey

Step 6- Standard EDTA solution – Molecular weight of EDTA is 372 gm/mole. Prepare m/100 solution of EDTA.

Procedure –

1. Standardization of EDTA solution with SHW using EBT indicator and ammonia buffer. End point is wine red to blue. Let volume of EDTA used = V_1 ml
2. Take 25ml of barium chloride solution in the flask and dilute to about 100ml with distill water.
3. Add 3 to 4 drops of NaOH solution and adjust pH = 12 check with pH meter.
4. Add 50mg of Methyle Thymole blue + KNO_3 mixture and titrate with m/100 EDTA solution.
5. End point blue to grey.

Calculation

Let the volume of EDTA used with SHW = V_1 ml

Let volume of EDTA used with barium chloride solution = V_2 ml

or m/100 1000 ml contain 3.72 gm/L

V_2 ml EDTA contain $\frac{3.72}{1000} \times V_2 gm$ EDTA

If converting $\frac{3.72}{1000} \times V_2 gm$ EDTA $into$ mole then

$$\frac{3.72 \times V_2}{1000 \times 372} = \frac{V_2}{100 \times 1000} \text{ mole of EDTA}$$

1 mole of EDTA ≡ 1 mole of Ba^{2+}

Alternate method:

Hardness of water due to barium chloride = $\frac{V_2}{V_1} \times 1000$ mg/L or ppm

Reference :

Vogel's text book of Quantitative chemical analysis – Pg No-336

Project No-10

To determine calcium ion (Strength) in presence of Barium ion in the solution of calcium salt + barium salt.

Mixture:- 0.02 mg $BaCl_2$ + 25 mg $CaCl_2$ in 250 ml distill water.

Discussion : there is an appreciable difference between the stability constant of CDTA (1,2-(Cyclohexylenedinitrilo)Tetraacetic Acid) complex of Barium (Log K = 7.99) and calcium (Log K = 12.50) , with the result that calcium may be titrated with CDTA in the presence of barium . the stability constant EDTA complex of these two metals (Ca, Ba) are too close together to permit independent titration of calcium in the presence of barium.

Indicator : the indicator calcicrome or Solochrome is added solid in titration flask + ammonia buffer + titration solution (barium chloride and calcium chloride solution mixture).

Procedure :

1. **Preparation of CDTA solution ((1,2-Cyclohexylenedinitrilo) tetra acetic Acid) :** dissolve 6.880 gm of solid reagent (CDTA) in 50 ml of sodium hydroxide 1M solution and making up to 1 litre .
2. **Standardization of CDTA solution:** With Standard hard water (SHW) – dissolve 1gm of $CaCo_3$ in minimum quantity of dilute HCL and when $CaCo_3$ completely dissolve then heat it till dryness. Dissolve this dry mass to minimum quantity of distills water and make the volume 1 litre.
3. Indicator solution(solechrome) : dissolve 0.5 gm of solid solechrome in 100 ml water or alcohol or used solid solechrome.

Method of titration:

1) Pipette out 25ml of standard hard water in the flask and titrate with CDTA solution with solochrome indicator.

   End point pink → blue .

   Let the volume of CDTA used with SHW = V_1 ml

   (suppose burette reading = 17.7 ml CDTA)

   Step 1- Burette reading standard hard water

(CDTA +NaOH) solution

$M_1V_1 \equiv \frac{1}{111} \times 20$

$(\frac{1}{111} = \text{molarity of SHW}. V_2 = 20\text{ml}$

$M_1 = \frac{1\times20}{111\times17.7} = molarity\ of\ CDTA$

Step2- a. Pipette out 25ml of (barium calcium solution) in conical flask and dilute it to 100 ml by adding distil water in solution.

Point to be noted: the solution mixture should have 0.02 M with respective calcium and barium concentration up to 0.2M.

b. Add 10 ml sodium hydroxide solution (1M) solution in flask in step 2 and check pH of the solution. pH should lie between 11 and 12 . Add a drop of indicator (solid) in titrate the solution with CDTA solution.

End point Pink → blue

Step 3- **Calculation** :

CDTA + NaOH solution ≡ Ca^{2+} / Ba^{2+} solution

M_1V_1' = $M_2'V_2$

V' = 45.5 ml Burette reading

Taking value of M_1 from step 1 and putting and above equation $\frac{1\times20\times V_1'}{111\times17.7}$

$M_2=\frac{45.5}{111\times17.7}=0.025\ \text{gm/L}$

Strength of calcium in the presence of barium = 0.025 × 40 = 0.522 gm /L

Bibliography

Reference

1. Vogel's text book of Quantitative chemical analysis – Pg No-336
2. Essential of Experimental Engg Chemistry – Shashi Chawla –Pg

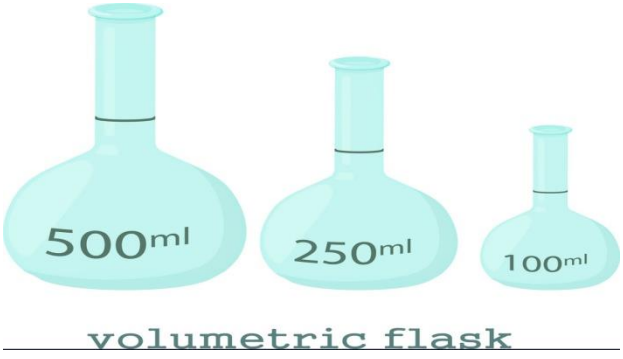
Related Projects on Complexometric titration

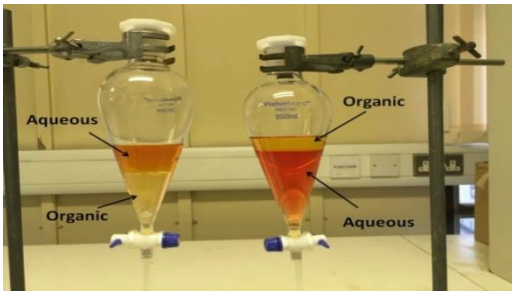
a. Detection of chromium and iron in the mixture that is chromium alloy

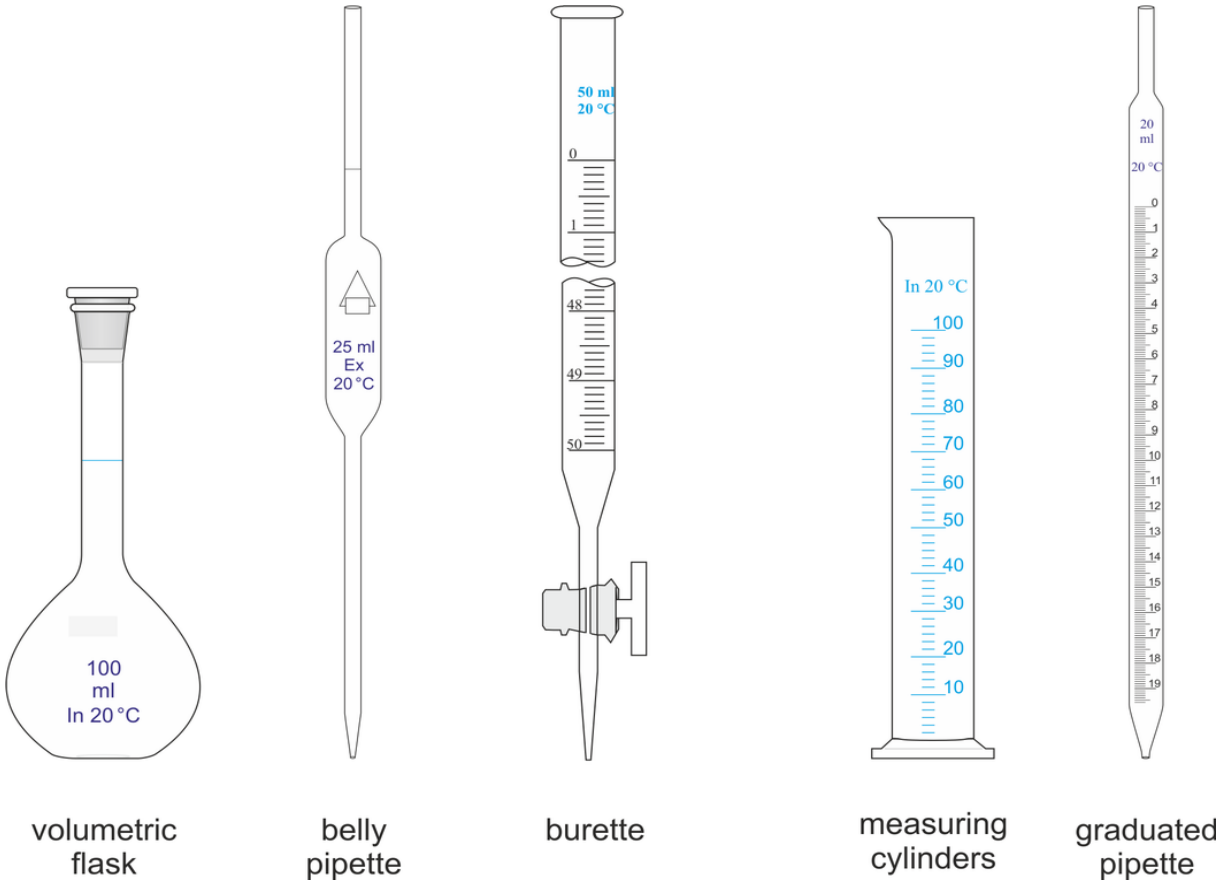
b. Lead and Tin in a mixture that is solder

c. Nickel in the presence of iron in Nickel steel

d. Calcium and lead in mixture







Volumetric Apparatus

CHAPTER- 3

LUBRICANT ANALYSIS

3.1. Introduction :

Oil analysis (OA) is the laboratory analysis of a lubricant's properties, suspended contaminants, and wear debris. OA is performed during routine predictive maintenance to provide meaningful and accurate information on lubricant.

Methods Employed for Analysis of Lubricants:

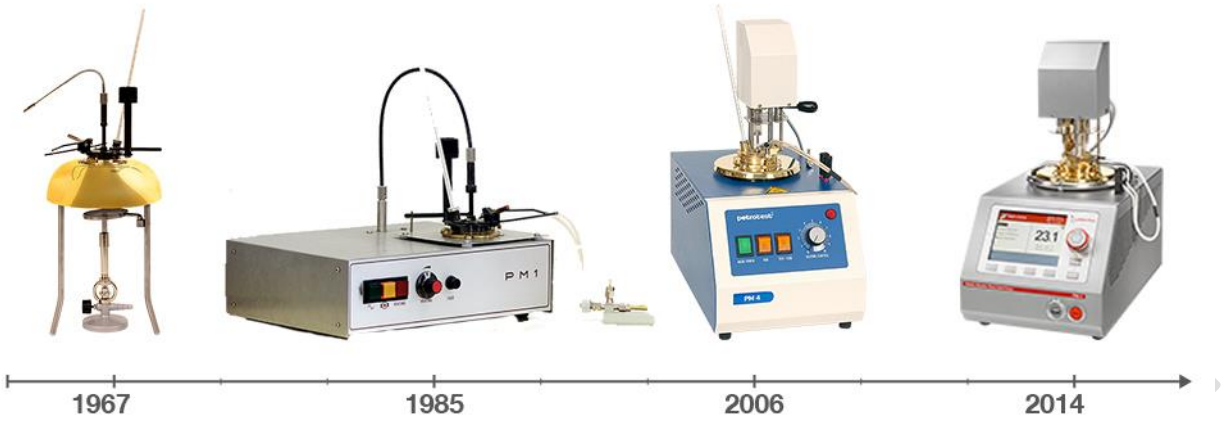
A. Flash and fire point of lubricating oil
B. Determination of carbon residue of lubricating oil
C. Determination of saponification of lubricating oil
D. Steam emulsification and Aniline point determination
E. Cloud and pour point determination of lubricating oil.
F. Viscosity and viscosity Index determination by redwood viscometer No.1

3.2. Flash and fire point of lubricating oil

Flash point and fire point: The flash point is the lowest temperature to which a lubricant must be heated before its vapor, when mixed with air, will ignite but not continue to burn. The fire point is the temperature at which lubricant combustion will be sustained.

The differences between flash point and fire points are as follows-

Flashpoint	Fire point
It has a lower value than the corresponding fire point of the same oil.	Fire point is 15^0C to 30^0C higher than the flash point of the same oil.
Comparatively less time is required to determine the flash point.	Comparatively more time is required to determine fire point.
Usually, the flash point of oil is determined.	Rarely, a fire point of oil is determined.



Pensky - Marten's Closed cup Apparatus

PROJECT -1

Flash and fire point of lubricating oil

Apparatus Requirements : Pensky Marten's closed cup apparatus, thermometer, lubricating oil (bio diesel)

Flash and Fire Point:

Flash point :

Flash point is the lowest temperature at which a liquid can gives off vapor to form an ignitable mixture in air near the surface of the liquid. The lower the flash point, the easier it is to ignite the material

Fire point:

The fire point of a fuel is the lowest temperature at which the vapour of that fuel will continue to burn for at least 5 seconds after ignition by an open flame of standard dimension.

Description of Pensky Marten's Apparatus:

For determining the flash point of fuel oils and lubricating oil, bitumen other than cutback bitumen and suspension of solids in liquids, having a flash point above 49°C. The oil cup is made of brass, fitted with a thermally insulated lifting handle. The cup lid, with insulated handle, is fitted with two opposite wedge cams, which engage under pillars secured to the cup flange. This feature facilitates easy removal of the lid by rotating the handle a few degrees to disengage the cams The shutter opening mechanism is designed for smooth operation and is operated by a spring handle with a knurled knob. A exposure device is mounted over the lid. The manually operated stirrer has a flexible shaft. The cup assembly is positioned in a cast iron air bath, fitted with a chrome plated brass top. The air bath is mounted on an upright.

Procedure:

Description of the apparatus:

The main components of the Pensky Martens Flash Point Apparatus are follows :

- OIL CUP
- AIR BATH
- ELECTRIC HEATING ARRANGEMENT
- ENERGY REGULATOR BOX
- TOP PLATE
- COVER ASSEMBLY HAVING

  (a) COVER

  (b) SHUTTER

  (c) STIRRING DEVICE

  (d) FLAME EXPOSURE DEVICE

  (e) THERMOMETER SOCKET.

Thermometers:

IP 15C -7*c TO 100* (LOW)

IP 16C 90*c TO 370*c(HIGH)

Either of the thermometers may be employed if the indicated reading falls within 93* to 110*C. For tests in which the indicated reading falls within the limits –7 to 93*C (20 to 200*F), the IF 15C thermometer shall be used and for tests in which the indicated reading falls within the limits 110 to 370*C (230 to 700*F) the IP 16C thermometer shall be used.

A blue print figure 1 is attached to show the constructional details.

Description:

The brass oil cup is meant for heating the Petroleum Products to the Flash Point. It carries a mark all round in the inside to show the required level of liquid.

The cup has a flange which rests on the brass top and the lower parts of the cup is within the Air Bath or Heating Vessel. The top plate is screwed on the Air Bath through brass spacers, so that an air gap is produced between the cup and the Air Bath by which the transmission to the cup is almost entirely by heated air. The cup flange has an indicator to fit correctly on the Top Plate. The heat is supplied from 220V A.C. Mains, by an electric heater kept below the Air Bath and the regulation of the heat is done by the Energy regulator. The function of the Air Bath and the Top Plate is to prevent external air draughts from interfering with the heating.

The cover proper of the lid is made of brass plate and has a tubular fitment which enables it to sit on the cup. The cover proper is illustrated in Figure 2/

It has three holes B,A & C in the peripheral region and also one circular hole which carries a split tube for taking brass collar of a thermometer, and one central hole admits stirrer rod.

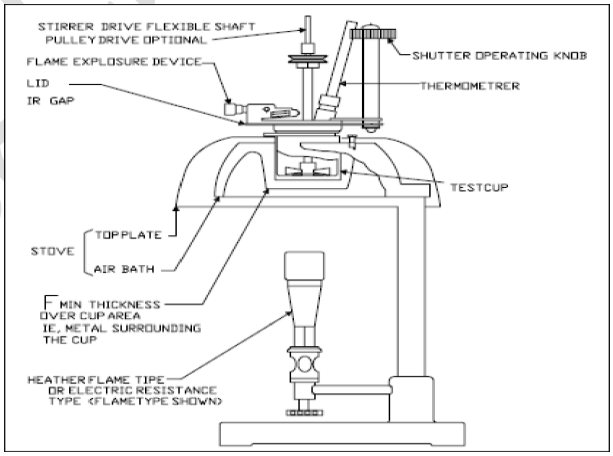
The peripherial hole. A is larger and the flame exposure to the oil under test is given through it. The flame is produced by a burning gas jet of a gas reserviour fitted close to the hole A. The gas is supplied from gas mains. The shutter is a brass disc fitted concentrically on the cover proper. It has two peripheral holes matching the hole b & A of the cover proper. The shutter can be rotated by the spring loaded handle G which is mounted on the cover proper. Initially the shutter keeps all the holes B,A, &C, closed proper . Initially the shutter keeps all the holes B,A & C closed but by slight rotation all the three holes get exposed. A lever arrangement fitted to the shutter tilts the gas Chamber in such a way that the gas jet with the flame enters the hole momentarily. The gas chamber has another ancillary jet called the Pilot flame F in figure 1. This enables the main gas jet to be relighted if extinguished during the test. The pilot flame burns continuously and is away from any of the holes of the cover proper.

Where gas supplied is not available, an oil burner replaces the gas chamber. A cotton wick produces the flame, otherwise the mechanism is similar as for the gas jet. A pilot oil flame is also provided in this case.

A light cololured ball of 4mm diameter fixed to a wire is mounted on the shutter, and which enables the test flame size to be visually adjusted to the same size as that of the ball. This is done by restricting the gas supply in the case of gas jets, and by adjusting the wick exposure in oil test flames.

The stirrer consists of a steel rod of which two pairs of brass propellers are fixed. The rod passes through the shutter.

A flexible cable protected with armoring is fitted to the external and of the rod. The flexible shaft has a brass handle which can be manually rotated, causing to the propellers to stir the test liquid.



Pensky Marten's Flash Point Apparatus

Precautions :

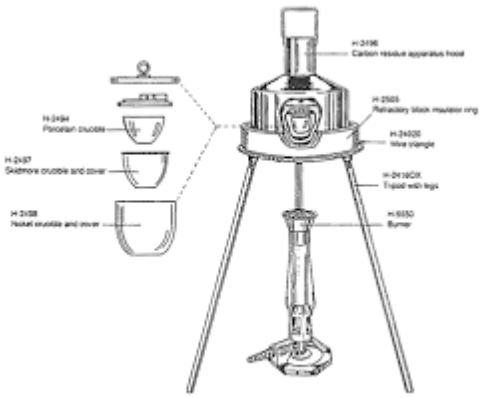
a. Properly wash and dry the oil cup. There should no moisture inside the cup.
b. Fill the supplied sample of lubricating oil up to specified film mark
   There should no oil on the outside of the cup
c. The thermometer should be Immersed in the oil cup

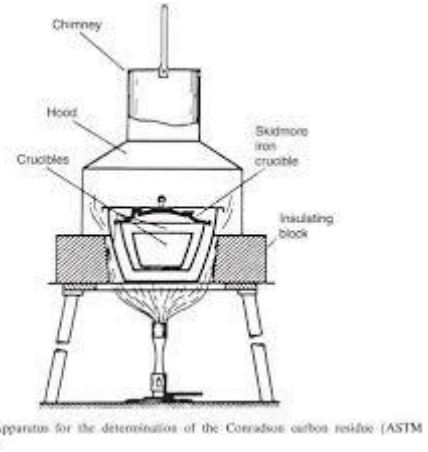
3.3. Determination of carbon residue of contain of lubricating oil conradson apparatus:

Conradson carbon residue. Conradson carbon residue, commonly known as "Concarbon" or "CCR" is a laboratory test used to provide an indication of the coke-forming tendencies of an oil. Quantitatively, the test measures the amount of carbonaceous residue remaining after the oil's evaporation and pyrolysis.

PROJECT- 2

Determination of carbon residue of contain of lubricating oil conradson apparatus





Apparatus for the determination of the Conradson carbon residue (ASTM 3)

Conradson apparatus

Significance and Use:

The carbon residue value of burner fuel serves as a rough approximation of the tendency of the fuel to form deposits in vaporizing pot-type and sleeve-type burners. Similarly, provided alkyl nitrates are absent (or if present, provided the test is performed on the base fuel without additive) the carbon residue of diesel fuel correlates approximately with combustion chamber deposits.

The carbon residue value of motor oil, while at one time regarded as indicative of the amount of carbonaceous deposits a motor oil would form in the combustion chamber of an engine, is now considered to be of doubtful significance due to the presence of additives in many oils. For example, an ash-forming detergent additive may increase the carbon residue value of an oil yet will generally reduce its tendency to form deposits.

The carbon residue value of gas oil is useful as a guide in the manufacture of gas from gas oil, while carbon residue values of crude oil residuum's, cylinder and bright stocks, are useful in the manufacture of lubricants.

Scope:

This test method covers the determination of the amount of carbon residue (Note 1) left after evaporation and pyrolysis of an oil, and is intended to provide some indication of relative coke-forming propensities. This test method is generally applicable to relatively nonvolatile petroleum products which partially decompose on distillation at atmospheric pressure.

NOTE : The term carbon residue is used throughout this test method to designate the carbonaceous residue formed after evaporation and pyrolysis of a petroleum product under the conditions specified in this test method. The residue is not composed entirely of carbon, but is a coke which can be further changed by pyrolysis. The term carbon residue is continued in this test method only in deference to its wide common usage.

NOTE :

In diesel fuel, the presence of alkyl nitrates such as amyl nitrate, hexyl nitrate, or octyl nitrate causes a higher residue value than observed in untreated fuel, which can lead to erroneous conclusions as to the coke forming propensity of the fuel. The presence of alkyl nitrate in the fuel can be detected.

Procedure :

The weighed amount of sample is placed in silica possible is put into a skidmore iron crucible having a closed fitting cover with small horizontal opening. The crucible are larger third crucible also fitted with a cover fitted loosely to cylindrical shape iron hood. Heat is applied from maker burner at certain prescribed rate till vapours all volatile metal are burn completely

$$\%\, carbon\, residue\ = \frac{weight\ of\ residue\ in\ crucible}{weight\ of\ original\ sample} \times 100$$

Precautions :

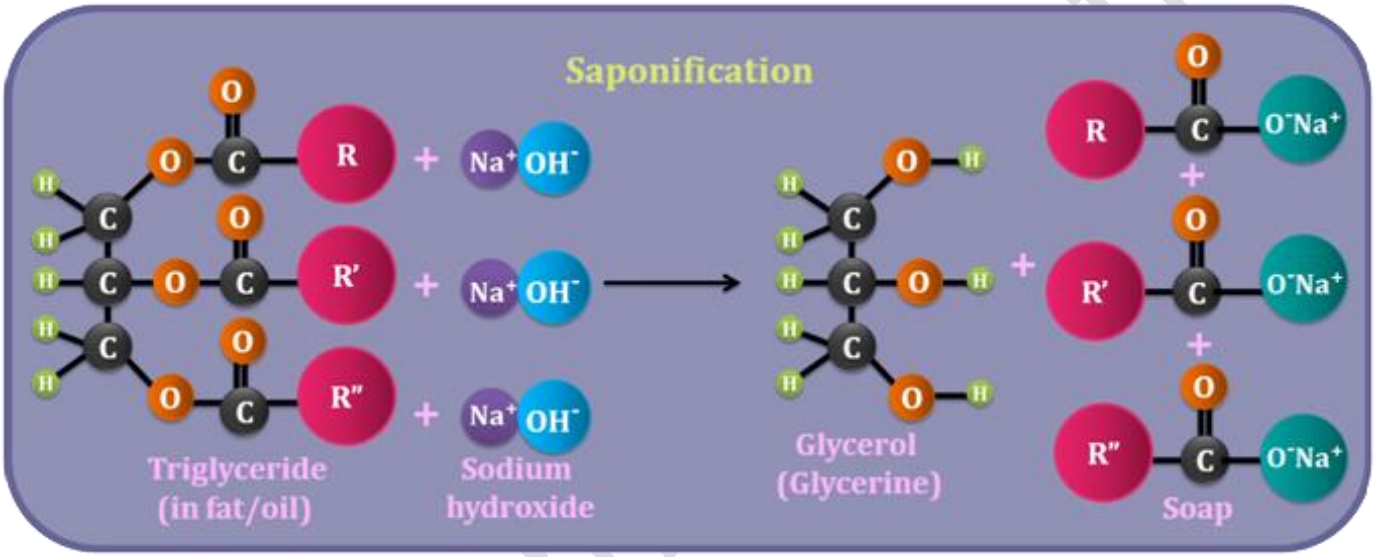
1. Lubricating oil should not spurt out of the crucible.
2. Unburnt lubricating oil should not be left in the crucible.

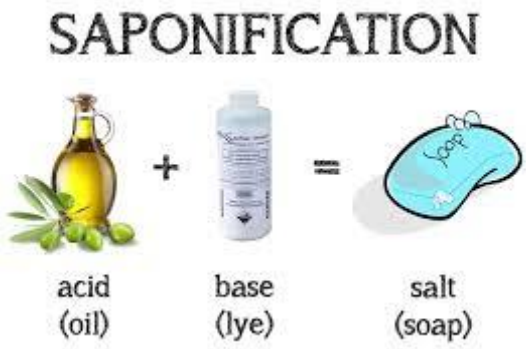
Reference :

Pg – no 205, Experimental Chemistry Shashi Chawla

3.4. Determination of saponification of lubricating oil

saponification number. saponification number - number of milligrams of potassium hydroxide (KOH) that combines with 1 gram of oil under conditions specified by test method ASTM D 94. Saponification number is an indication of the amount of fatty saponifiable material in a compounded oil.





PROJECT – 3

Determination of saponification of lubricating oil

Requirement :

a. Glass ware , conical glass , reflex condenser , burette , measuring cylinder
b. Chemicals : coconut oil, alcoholic potassium hydroxide solution, 95 % ethanol 0.5 N HCL solution, Phenothalin indicator.

Procedure :

1. Prepare an approximately 0.5N solution of potassium hydroxide by dissolving 30gm of it in 20ml of water and make the final volume to 1 litre using 95% ethanol. Leave the solution for 24 hours before decanting and filtering the solution.
2. Titrate the potassium hydroxide solution with 0.5 N HCL using phinophethalein indicator (let the volume of hCL used Y ml).
3. For Hydrolysis , accurately take about 2 gm of coconut oil in to a 250 ml conical flask and add 25 ml postassium hydroxide solution.
4. Attach a reflex condenser and heat the flask contains on a steam bath for 1 hour with occasion shaking
5. While the solution is still hot add phenothalein indicator and titrate the access KoH 0.5 N HCL

End point – Pink to colourless

Observation and calculation :

1. Value of HCL used for saponification = x ml
2. Value of HCL used for blank titration = y ml
3. The difference between the titater value of saponification titration and blank titration = (y-x) ml
4. Saponification value = $\frac{(y-x)\times 0.5 \times 56.1}{wt.of\ oil\ (g)}$

3.5. Steam emulsification and Aniline point determination

Steam Emulsification :

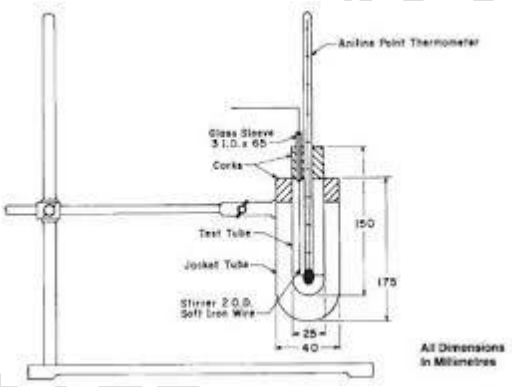
Emulsification: It is the property of oils to get intimately mixed with water, forming a mixture, called emulsion. Certain oils form emulsions with water easily. Emulsions have a tendency to collect dirt, grit,. Foreign matter etc., thereby causing abrasion and wearing out of the lubricated parts of the machinery. So, good lubricating oil should form an emulsion with water, which breaks off quickly. The tendency of lubricant-water emulsion to break is determined by A.S.T.M. test. In this, 20 ml of oil is taken in a test-tube and steam at 100°C is bubbled through it, till the temperature is raised to 90°C. The tube is then placed in a bath maintained at 90°C and the time in seconds is noted, when the oil and water separate out in distinct layers. The time in second in which

oil and water emulsion separates out in distinct layers, is called steam emulsion number (S.E.N.). A goad lubricant should possess a low steam emulsion number.

Aniline Point :

Aniline point of oil is defined as the minimum equilibrium solution temperature. For equal volume of aniline and oil sample. Aniline point gives an indication of the possible deterioration of oil in contact with rubber sealing's, pickings, etc. Aromatic hydrocarbons have a tendency to dissolve natural rubber and certain types of synthetic rubbers. Consequently, low aromatic content in the lubricants is desirable. A higher aniline-point means a higher percentage of paraffinic hydrocarbons and hence, a lower percentage of aromatic hydrocarbons.

Aniline point is determined by mixing mechanically equal volumes of the oil sample and aniline in a test-tube. The mixture is heated, till homogeneous solution is obtained. Then, the tube is allowed to cool at a controlled rate. The temperature at which the two phases (oil and aniline) separate out is recorded at the aniline point.



Aniline point apparatus

Aniline point apparatus

Theory :

The aniline point of an oil is defined as the minimum temperature at which equal volumes of aniline and lubricant oil are miscible, i.e. form a single phase upon mixing..

Requirements:

Aniline point Apparatus: aniline point thermometer, lubricating oil and aniline.

Significance :

Aniline being an aromatic compound freely mixes with aromatic so a low aniline point indicates low diesel index (because of high percentage of aromatics). Significance: High aniline point indicates that the fuel is highly paraffinic and hence has a high Diesel index and very good ignition quality.

Introduction:

In Determination of aniline point is a test to evaluate base oils that are used in oil mud. The test indicates if oil is likely to damage elastomers (rubber compounds) that come in contact with the oil. The aniline point is called the "aniline point temperature," which is the lowest temperature (°F or °C) at which equal volumes of aniline ($C_6H_5NH_2$) and the oil form a single phase. Aniline point of oil gives an indication of the possible tendency of deterioration of oil when it comes into contact with packing ,rubber sealing etc. in general oils with a high aromatic content are more detrimental to rubber products than those with a low aromatic content. The relative aromatic content of an oil is indicated by its anilinepoint and oils with a high aromatic content have a low aniline point and vice versa. The higher theaniline point of the oil ,the more desirable it is for drilling fluid usage. In our experiment, 5 ml aniline and5ml diesel were taken in a test tube provided with thermometer and heat was given until both aniline and diesel become completely miscible. The aniline point of diesel was found at the temperature of 94°C.

By definition, the aniline point is the lowest temperature at which equal volume of aniline and oil are completely miscible (clear). This method is suitable for transparent liquid samples having an initial boiling point above room temperature and where the aniline point is below the bubble point and above the solidification point of the aniline sample mixture. The procedure is useful in characterizing pure hydrocarbons. The lower the aniline point, the greater the solvency or reactivity of the oil, which in turn gives an indication of the oils

aromaticity. Paraffinic hydrocarbons have higher aniline points than aromatic types (Mair & Willingham 1936; Rossini 1937). For instance, for an aromatic oil with a 75%aromatic content, the aniline point would be between 32.2° and 48.9°C; for a naphthenic type containing 40% aromatic structures, it would be between 65.6° and 76.7°C; and for a paraffinic oil with a15% aromatic content it would be between 93.3° and 126.7°C.In a homologous series, the aniline point for mixtures of hydrocarbons such as diesel oils and mineral oils serves as a guideline for judging the aromatic hydrocarbon content of oil and for comparing oil.

Aniline being an aromatic compound freely mixes with aromatics; so a low aniline point indicates a low diesel index. Aniline point also predicts the amount of carbon present in the aromatics .

formula:

$\%C_A = 1039.4n\ d_{20} - 470.4d_{20} - 0.567AP\ (°C) - 1104.42;$ (1)

Where, n_d^{20} =refractive index at 20°C

d_{20} =density at 20°C

Diesel index is a measure of ignition quality of fuel

DIESEL

In Petroleum diesel, also called petrol, diesel, or fossil diesel is the most common type of diesel fuel. It is produced from the fractional distillation of crude oil between 200 °C (392 °F) and 350 °C (662 °F) at atmospheric pressure, resulting in a mixture of carbon chains that typically contain between 8 and 21 carbon atoms per molecule

PETROL

Gasoline also known as petrol outside North America is a transparent, petroleum-derived liquid that is used primarily as a fuel in internal combustion engines. It consists mostly oforganic compounds obtained by the fractional distillation of petroleum, enhanced with avariety of additives. On average, a 42-gallon barrel of crude oil (159 L) yields about 19 US gallons (72 L) of gasoline when processed in an oil refinery, though this varies based on the crude oil source's assay

KEROSENE

Kerosene, also known as paraffin, lamp oil and coal oil (an obsolete term), is a combustible hydrocarbon liquid which is derived from petroleum, widely used as a fuel in industry as well as households. Kerosene is widely used to power jet engines of aircraft (jet fuel) and some rocket engines, and is also commonly used as a cooking and lighting fuel and for fire toys such as poi. In parts of Asia, where the price of kerosene is subsidized, it fuels outboard motors on small fishing boats.World total kerosene consumption for all purposes is equivalent to about 1.2 million barrels per day

JET FUEL

Jet fuel, aviation turbine fuel (ATF), or avtur, is a type of aviation fuel designed for use In aircraft powered by gas-turbine engines. It is colorless to straw-colored in appearance. The most commonly used fuels for commercial aviation are Jet A and Jet A-1, which are produced to a standardized international specification. The only other jet fuel commonly used in civilian turbine-engine powered aviation is Jet B, which is used for its enhanced cold-weather performance. Jet fuel is a mixture of a large number of different hydrocarbons. The range of their sizes (molecular weights or carbon numbers) is defined by the requirements for the product, such as the freezing or smoke point. Kerosene-type jet fuel (including Jet A and Jet A-1) has a carbon number distribution between about 8 and 16 (carbon atoms per molecule); wide-cut or naphtha-type jet fuel (including Jet B), between about 5 and 15

BIO DIESEL

Biodiesel refers to a vegetable oil - or animal fat-based diesel fuel consisting of long- chain alkyl (methyl, ethyl, or propyl) esters. Biodiesel is typically made by chemically reacting lipids (e.g., vegetable oil, soybean oil, animal fat (tallow) with an alcohol producing fatty acid esters. Biodiesel is meant to be used in standard diesel engines and is thus distinct from the vegetable and waste oils used to fuel converted diesel engines. Biodiesel can be used alone, or blended with petrodiesel in any proportions. Biodiesel blends can also be used as heating oil.

Use of biodisel

The apparatus consist of the following:

1. Test Tube, approximately 25 mm in diameter and 150 mm in length, made of heat-resistant glass.

2.Jacket, approximately 37 to 42 mm in diameter and 175 mm in length, made of heat-resistant glass.

3.Stirrer, manually operated, metal, approximately 2 mm in diameter (14 B&S gage) metal wire concentric ring shall be at the bottom, having a diameter of approximately 19 mm. The length of the stirrer to a right-angle bend shall be approximately 200 mm. The right-angle bend shall be approximately 55 mm long. A

glass sleeve approximately 65 mm in length of 3-mm inside diameter shall be used as a guide for the stirrer. Any suitable mechanical device for operating the stirrer as specified is an approved alternative for the manual operation

EXPERIMENTAL PROCEDURE

Clean and dry the apparatus. Deliver 10 mL of aniline and 10 mL of the dried sample into the test tube fitted with stirrer and thermometer. If the material is too viscous for volumetric transfer, weigh to the nearest 0.01 g a quantity of the sample corresponding to 10 mL at room temperature. Center the thermometer in the test tube so That, the immersion mark is at the liquid level, making sure that the thermometer bulb does not touchthe side of the tube. Center the test tube in the jacket tube. Stir the mixture rapidly using a 50-mm (2-in.) stroke, avoiding the introduction of air bubbles. If the aniline-sample mixture is not miscible at room temperature, apply heat directly to the jacket tube so that the temperature rises at a rate of 1 to 3°C (2 to 5°F)/min by removing or reducing the heat source until complete miscibility is obtained. Continue stirringand allow the mixture to cool at a rate of 0.5 to 1.0°C (1.0 to 1.8°F)/min.

Continue cooling to a temperature of 1 to 2°C (2.0 to 3.5°F) below the first appearance of turbidity, and record as the aniline point the temperature at which the mixture suddenly becomes cloudy throughout this temperature, and not the temperature of separation of small amounts of material, is the minimum equilibrium solution temperature.

Observation table :

Sl.No	**Sample**	**Volume of sample taken(ml)**	**Volume of aniline taken (ml)**	**Aniline point(˚C)**
1	Diesel	5	5	71
2	Petrol	5	5	65
3	Kerosene	5	5	74
4	Jet fuel	5	5	80
5	Bio diesel	5	5	59.5

Aniline point of diesel = 71°C

Aniline point of petrol = 65°C

Aniline point of kerosene = 74°C

Aniline point of jet fuel = 80°C

Aniline point of bio diesel = 59.5

CONCLUSION

Specified volumes of aniline and sample, or aniline and sample plus n-heptane, are placed in a tube and mixed mechanically. The mixture is heated at a controlled rate until the two phases become miscible. The mixture is then cooled at a controlled rate and the temperature at which two phases separate is recorded as the aniline point or mixed aniline point.

REFERENCE

[1]. Oilfield Glossary

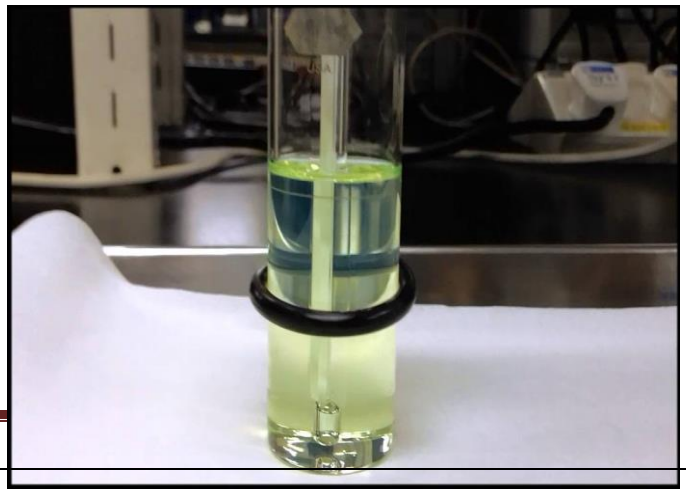
[2]. https://en.wikipedia.org/wiki/Drillingfluids.

[3]. D1500 Test Method for ASTM Color of Petroleum Products (ASTM Color Scale).

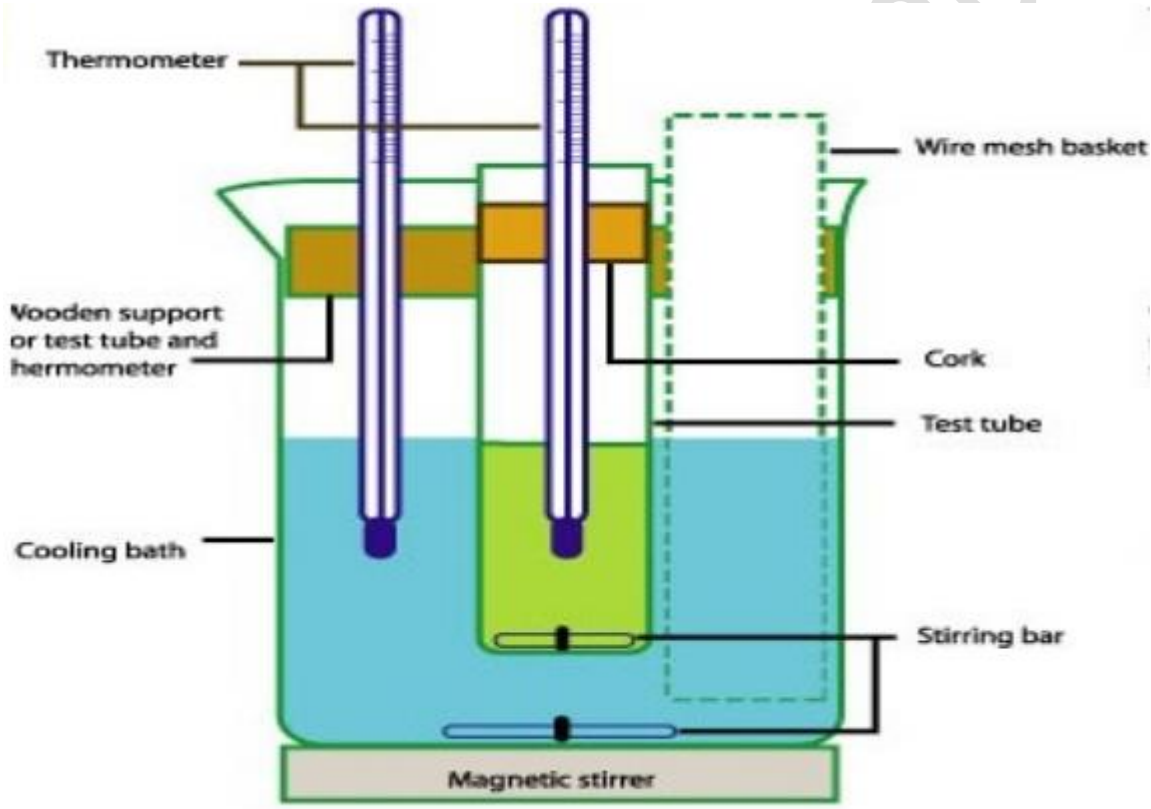
[4]. E1 Specification for ASTM Liquid-in-Glass Thermometers

3.6. Cloud and pour point determination of lubricating oil.

Cloud and pour-points: When an oil is cooled slowly, the temperature at which it becomes cloudy or hazy in appearance, is called its cloud-point while the temperature at which the oil ceases to flow or pour, is called its pour-point. Cloud and pour-points indicate the suitability of lubricants in cold conditions. Lubricant used in a machine working at low temperatures should possess low pour-point; otherwise solidification of lubricant will cause jamming of the machine. It has been found that presence of waxes in the lubricating oil raise the pout-point. Determination of pour-point is carried out with help of pour-point apparatus.



Cloud point Apparatus



Pour Point Apparatus

Introduction:

Crude oil is a naturally occurring mixture, consisting predominantly of hydrocarbons, sulphur, nitrogen and metals Crude oils are complex but mainly paraffinic, naphthenic and aromatic (Wang et al., 1994). Crude oils contain all normal alkenes. However, this percentage rises to 35% in highly paraffinic and decreases to zero in

highly biograded oils (Ali, et alDuring its transportation, because oil temperature at initial station is higher than the ambient temperature, the oil temperature continues to decrease, and when it decreases to the wax precipitation point of crude oil, wax crystal in crude oil starts to precipitate, grow, and deposit on the pipe wall finally; sediments on pipeline will reduce the effective flow area of the pipeline.

Aim and Objectives:

The specific objectives are listed below;

To determine the temperature at which the sampled crude oil becomes semi solid and loses their flow.

characteristics.

i.) To compare the cloud and pour point of the different crude samples.

ii.) To investigate the various factors that can affect the cloud and pour point of crude oils.

iii.) To make recommendations as to how wax deposition can be prevented during crude transportation.

LITERATURE REVIEW

Due to flow assurance problems encountered during crude transportation using pipelines in the oil and gas industry, it has become of paramount importance to study the different factors that enhance wax deposition in pipelines. Several researchers have done work on the subject matter and have been able to predict using scientific methods conditions that promote wax deposition while also professing ways to combat its undesirable occurrence. Stated that the main components of the heavy fraction of hydrocarbon which participate in the solid phase formation include asphaltenes, diamondoids, petroleum resins and wax. Petroleum wax consist mainly saturated paraffin hydrocarbons with number of carbon atoms in the range of 18-36. Wax may also contain small amounts of naphthenic hydrocarbons with their number of carbon atoms in the range of 30-60. Wax usually exists in intermediate crudes, heavy oils, tar sands and oil shales. There are three main factors that affect wax deposition in flow systems which according to Bott and Gudmundsson (1977) are flow rate, temperature deferential, and cooling rate, as well as surface properties.

Cloud Point Determination:

Apparatus used in cloud point determination were test jar, cork carrying thermometer, water bath with heater, cloud point chamber and crushed ice. Experimental procedures are as enumerated below;

a) Test jar was filled to the level mark, closed tightly by the cork carrying the thermometer and placed into a bath of crushed ice.

b) Test jar was removed from the jacket quickly without disturbing the specimen. Inspection for cloud point was done and jacket replaced. Operation was done without exceeding time duration of three (3) seconds.

c) Since cloud point is the temperature of a liquid specimen when the smallest observable cluster of hydrocarbon crystals first occurs upon cooling under prescribed conditions, observation was done and cloud point was reported to the nearest 1˚C. At this point, cloud is observed at the bottom of the test jar, which is confirmed by continued cooling.

Pour Point Determination:

Same apparatus that were used in cloud point determination were used in pour point determination. Experimental procedures are enumerated below; A sample of worm crude was filled to the level mark. The test jar was tightly closed by the cork carrying the test thermometer and placed in a bath of crushed ice.

a) The test jar was inspected at an interval of at three (3) minutes by holding in a horizontal position for a few seconds before returning it to cool.

b) The pour point was reached when the oil surface stayed in the vertical position for a period of 5 seconds without sagging. At this point the thermometer was inserted to cool for 10 seconds and the temperature of the oil was taken.

c) The pour point was 3˚C higher than the thermometer reading. Crude sample at pour point.

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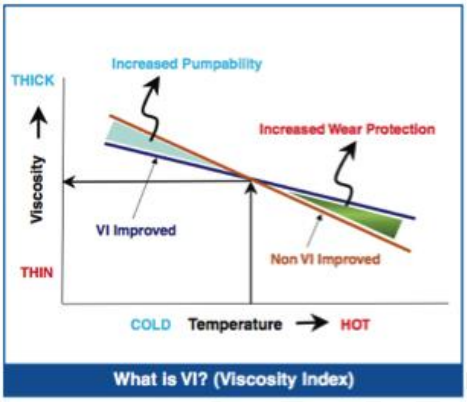
- Agrawal, K.M., Khan, H.U.,• Surianarayanan, M., Joshi, G.C., (1990).Wax Deposition of Bombay High Crude Oil under Flowing Conditions, Fuel, 69, 794-796.
- Ajienka, J.A., Ikoku, C.V., (1997).• Waxy crude oil handling in Nigeria: practices, problems and prospects, Energy sources. 12(4): 463-478.
- Al-Besharah, J.M., Salman, O.A., andAkashah, S.A., (1987). Viscosity of crude oil blends. Industrial and Engineering Chemistry Research, 26, 2445–2449.

3.6. Viscosity and viscosity Index determination by redwood viscometer No.1

Viscosity



Viscosity Index

What is Viscosity

It is a measure of the resistance offered by one layer of fluid to the other layer of the same fluid during motion. It is expressed in Pa.s unit

Redwood Viscometer is normally used for the determination of the viscosity of petroleum products.

'Redwood Viscometer':

It determines the viscosity in terms of seconds (which are terms as Redwood seconds), a time taken by oil to pass through a standard orifice and collection of the same oil in 50 cc flask.

Originally Redwood Viscometer was developed for the measurement of viscosity of petroleum products.

Redwood Viscometer' are of two types:

1. Redwood Viscometer No.1 (For fluid having viscosity corresponds to Redwood seconds less than 2000)
2. Redwood Viscometer No. 2 (For fluid having viscosity corresponds to Redwood seconds greater than 2000)

Redwood Viscometer No. 1 & 2 is used depending on the time of oil flow through an orifice at the desired temperature is greater or less than 2000 seconds. Normally viscosity of highly viscous fluids is determined by the use of Redwood Viscometer 2.

Procedure:

1. Fill the oil cup with oil to the required oil level indicated by marker point
2. Heat the water at a uniform temperature.
3. When the required temperature of the oil is attained, lift the ball above the orifice hole.
4. Allow the oil to pass through it (start the stopwatch) and collect 50 CC of oil in the volumetric flask.
5. The measured seconds are in terms of viscosity of oil measured.

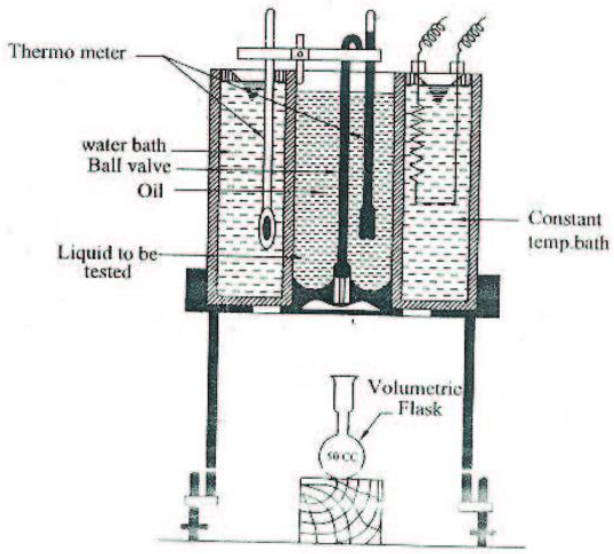
Conversion Formula:

Let the time of efflex = t seconds

Redwood viscometer : c S t = 0.26t -171 /2

Cgs physical unit for kinemetic viscosity is stokes (S t)

c S t = centi stock to get viscosity in centi poise multiple centi stock by density of lubricant at that temperature.



Redwood Viscometer

Precautions:

Before testing the viscosity filter the lubricating oil through a 100 mesh wire sieve.

Related project of Lubricant :

1. to determine iodine value of the given lubricating oil

Reference – 195 ,experimental chemistry – Shashi Chawla

2. To determine acid value of lubricating oil

Reference – 199 ,experimental chemistry – Shashi Chawla

Activities related to preparation of some Lubricants:

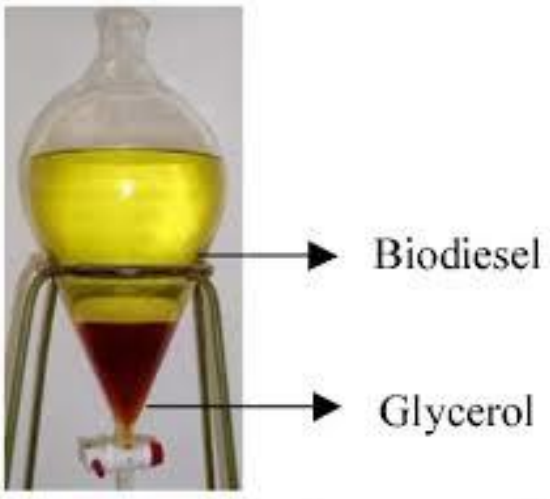
1. Preparation of bio Diesel
2. Preparation of cutting oil
3. Preparation of rust removal
4. Preparation of glycerin soap
5. Preparation of solid lubricant
    a. From bio diesel waste product
    b. From bio diesel waste from zatropha oil
    c. From petroleum jelly
    d. Paraffin oil
6. Saponification reaction and preparation or useful organic cleaning product

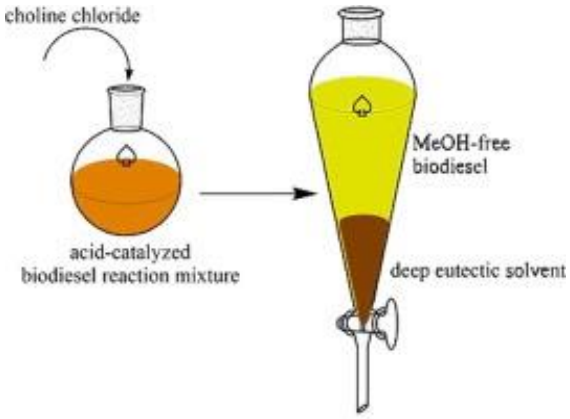
PROJECT- 4

Preparation of bio Diesel from zatropha oil

Procedure :

1. Heat the 250 ml of zatropha oil in a beaker up to temperature of 75˚C.
2. Dissolve 5 gm of sodium hydroxide in 150 ml of ethyl alcohol and heat it on water bath at 75˚C.
3. Pour the hot mixture of sodium hydroxide and ethyl alcohol in hot zatropha oil.
4. Layer of bio diesel and glycerol will separate out.
5. Put this mixture in separating funnel





Preparation of cutting oil

Material	Function	Content (% volume/volume of fixed oil)
Fixed oil	Base oil	80
Washing soap	Emulsifier	10
Phenol	Disinfectant	5
Sulphur	Extreme pressure agent	5

Cutting Oil Formula

Material	**Function**	**Content**
Mineral oil	Base oil	85
Oleic acid	Base oil	10 gm
Trienthanol amine	Emulsifier	5

Cutting Oil Formula

Preparation of rust removal

Rust remover – potassium oxalate (100 gm) + R.O water (20 ml) applied on the surface of rust it will remove the rust.

PROJECT-5

Preparation of glycerin soap:

Material Required:-

Transparent soap base – 20gm

Glycerin – 2 ml

Olive oil-2ml

Vitamin E oil-1 ml

Castor oil- 1ml

Sobitol – 1ml

Cocobitain- 1ml

Method:

Melt transparent soap base on mild heating. while soap base completely melts at the above ingredients one by one by constant stirring. After this put the mixture in soap mould and let it cool. Glycerin soap is prepared.

Pure glycerin soap prepared in Engineering Chemistry lab SOA (N)

Preparation of solid lubricant:

PROJECT-6

Preparation of axle grease in the lab .

Material Required :

beroza (pine resin) – 10 gm

Paraffin wax – 10 gm

Petroleum jelly -5 gm

Castor oil- 2 ml

Mineral oil (engine oil)- 50 ml

Method :

Melt pine resin in iron container. While it is completely melt put paraffin wax and let it also melt. After this put petroleum jelly, castor oil, engine oil with constant stirring. Heat till the mixture becomes thick. Now put off heat and let the mixture cool.

PROJECT-7

White grease formation:

Material required –

Beewax – 10 gm

Cocobutter – 5 gm

Pure tarpantine oil – 5 ml

Method :

Melt beewax and cocobutter in iron container and add tarpantine oil while the mixture is hot. Stir the mixture properly and let it cool.

White grease prepared in Engineering Chemistry lab in SOA (N)

Method - : Axil grease formation by saponification

Heat the engine oil in iron container and put flakes of washing soap in it by constant stirring. Mix the mixture till mixture becomes thick. Cool it and store.

Axle Grease prepared in Engineering Chemistry SOA (N)

PROJECT- 8

Preparation or useful organic cleaning product:

Material required;

Neem oil – 5 ml

Pine oil – 70%

Emulsifier – 30%

TRO (Turkey Red Oil)- 30%

Method :

Mix emulsifier and TRO and with the help of mixer blender. When mixing is done properly put pine oil in it and again mix with mixer blender till solution become transparent. When the mixture is ready put neem oil in it. This is white phenyl concentrate if you put white phenyl concentrate in water It will give white colour.

PROJECT-9

Preparation of Black Phenyl

Material required:-

Castor oil-125 gm

NaOH solution – 50 gm

Rosin (loban or beroza)- 25 gm

Creosot oil – 125 gm

Chloroxylene – 5 ml

Procedure :

Dissolve rosin in castor oil by heating and then add NaOH solution heating is continued in the hot mixture add cresot oil by continue heating and then add chloroxylnol. Heat the mixture for some time . At the end add pinch of Na_2CO_3 powder phenyl gets black.

Put some mixture in water, it will dissolve and give odour of phenyl disinfectant.

CHAPTER -4

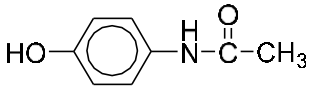
ORGANIC SYNTHESIS

4.1. Paracetamol:

What is paracetamol?

Paracetamol (acetaminophen) is a pain reliever and a fever reducer. The exact mechanism of action of is not known.

Paracetamol is used to treat many conditions such as headache, muscle aches, arthritis, backache, toothaches, colds, and fevers. It relieves pain in mild arthritis but has no effect on the underlying inflammation and swelling of the joint.



Acetaminophin or Paracetamol

Synthetic skills

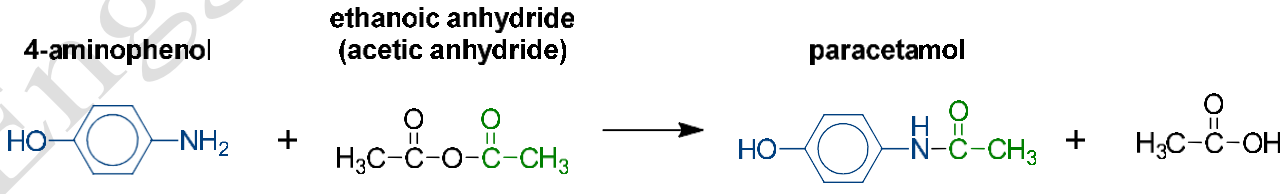
1. weighing and mixing reagents
2. vacuum filtration
3. purification by recrystallisation

Analytic Skills :

1. Thin layer chromatography
2. Melting point determination
3. Infrared (IR) spectroscopy
4. Nuclear Magnetic
5. Resonance (NMR)

Synthesis of Paracetamol (Morning)

Paracetamol is made by reacting 4-aminophenol with ethanoic anhydride (more commonly called acetic anhydride). This reaction forms an amide bond and ethanoic acid as a by- product. When the reaction is complete the paracetamol is then isolated and purified.



The synthesis of paracetamol can be broken down into 3 parts:

Part 1 – mix the reactants together to form paracetamol.

Part 2 – isolate crude paracetamol from the ethanoic acid and unreacted starting materials.

Part 3 – purify paracetamol by recrystallisation.

Analysis of Paracetamol (Afternoon)

In the afternoon you will analyze the purity of the paracetamol you have made and interpret the infrared (IR) and nuclear magnetic resonance (NMR) spectra of paracetamol to confirm you have made the correct compound.

The analysis section has 5 parts:

Part 1 – calculating the percentage yield of paracetamol.

Part 2 – determine the melting point of your sample.

Part 3 – analyze purity by thin layer chromatography (TLC).

Part 4 – measure and interpret the infrared (IR) spectrum of your compound.

Part 5 – interpret the ^{1}H nuclear magnetic resonance (NMR) spectrum.

Health and Safety

For your health and safety you will need to observe the following protocols:

- Laboratory coats and safety spectacles must be worn at all times in the laboratory.
- You must carry out your experiments in the fume hoods and not on the benches.
- Disposable gloves are available on the shelves next to where you will be working to protect your hands.
- No food or drink is allowed.
- Chemically contaminated gloves and filter papers go into the yellow bins.
- Liquid organic waste is disposed of in special containers located by each large sink in your bay. If the container is full, report it to the demonstrators.
- Acetic anhydride, hydrochloric acid and sodium hydroxide are corrosive – wear gloves!
- Ethyl acetate and petroleum ether are flammable liquids – no flames or sparks should go near these reagents.

Synthesis of Paracetamol

CHEMICAL	HAZARD	PRECAUTION	DISPOSAL
Ethyl Acetate	Highly flammable Causes eye irritation	Keep away from heat/open flames/ sparks	Acetone waste container by each sink.
Petroleum ether 40-60 °C 	Highly flammable May cause lung damage if swallowed Vapors may cause drowsiness or dizziness	Keep away from heat/ open flames/sparks Avoid inhalation	Acetone waste container by each sink.

	Toxic to aquatic organisms		
Acetic Acid	Flammable Causes burns	Wear gloves Keep away from heat, open flames and sparks	In a fume hood dilute with water and flush down sink.
4-Aminophenol	Harmful by inhalation and if swallowed Very toxic to aquatic organisms Suspected of causing genetic defects.	Wear gloves Avoid breathing dust. Handle in fume cupboard. Avoid release to the environment	Leave sample tube in your fume cupboard.
Acetic Anhydride	Flammable Harmful by inhalation and if swallowed	Keep away from heat/open flames/ sparks Wear gloves	In a fume hood dilute with water and flush down sink.
Acetaminophen (paracetamol) 	Harmful Causes skin, eye and respiratory irritation	Wear gloves Avoid breathing dust	Leave sample in your fume hood.

Synthesis of Paracetamol

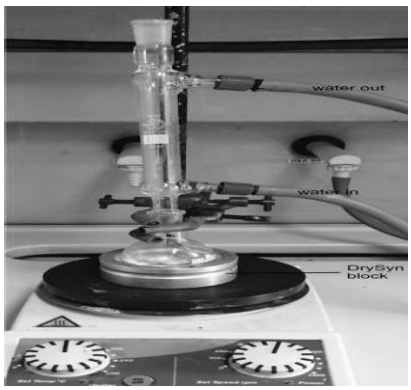
PROJECT -1

Preparation of Acetaminophin

Procedure :

1. Add 2.1 grams of 4-aminophenol (pre-weighed for you in a labeled sample tube) into the round-bottomed flask.
2. Using your 25 mL measuring cylinder, measure 18 mL of water and add this to the flask. Add a magnetic follower to the round-bottomed flask.
3. Carefully clamp the flask at the neck and position it in the metal DrySyn block which should be placed on the stirrer hotplate. Stir the reaction mixture using a magnetic follower. Do not apply heat at this stage.
4. Assemble the apparatus for reflux as shown in the diagram below. Tip: Do not clamp the condenser!
5. Using a Pasteur pipette, measure 3 mL of ethanoic anhydride (also known as acetic anhydride) into a 10 mL measuring cylinder. Add this to your mixture by lifting the condenser and adding directly to the round-bottomed flask.

6. Replace the condenser and switch on the heat to your hotplate (set the dial to about 120°C). Tip:
7. Make sure there is water going through your condenser.
8. The reaction is heated at reflux for 15 minutes, stirring continuously. The reaction mixture should become colourless.
9. After refluxing for 15 minutes, switch off the heat and carefully raise the round-bottomed flask away from the DrySyn block using the boss and clamp.
10. Allow the flask to cool to room temperature.
11. On cooling, crude paracetamol should form in the round-bottomed flask.

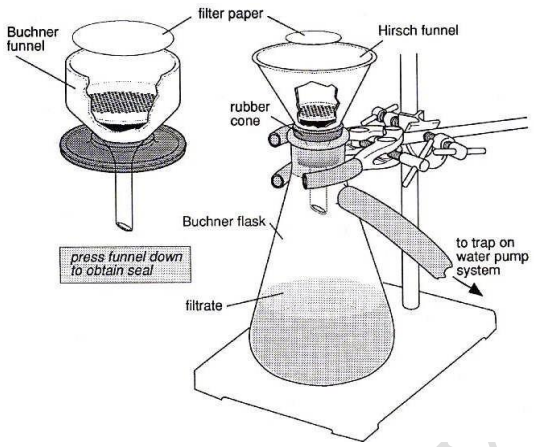


Isolate crude paracetamol from ethanoic acid and unreacted starting materials

How it works

1. The easiest way to collect or remove precipitates is by vacuum filtration. This is a faster method of filtering solids than filtration under gravity as the vacuum pump provides the suction.
2. Set up your vacuum filtration apparatus as shown in the diagram below with the Buchner flask and funnel that you have on the bench.
3. Place your Buchner flask on top of the base of the clamp stand. Clamp the flask around the neck.
4. Place a Buchner (or Hirsch) funnel on top of the flask with a rubber ring or cone in between to create a seal.
5. Place a filter paper inside the funnel. You need to use the appropriate size paper that can lay flat and covers all the holes in the
6. The orange tubing at the back of the fume cupboard is where you will connect your apparatus to the vacuum. Moisten the end of the orange vacuum tubing by dipping it in a beaker of water. This will help you place the tubing onto the side-arm of the Buchner flask.
7. A demonstrator will show you how to switch on the vacuum pump and open the taps required to apply suction to your apparatus.
8. Wet the filter paper with some cold water and gently hold down the funnel. If the water passes through quickly you have a good enough suction. A demonstrator will help you if not.

9. Filter the reaction mixture using water (approximately 5 mL) to rinse out as much of the contents of the reaction flask onto the filter paper.
10. Wash the solid with approximately 2 x 15 mL of ice cold water and leave under suction for a few minutes. This should rinse away any ethanoic acid.
11. Transfer your solid into a clean 100 mL conical flask. Empty the filtrate into the sink at the back of the fume hood and wash out with plenty of water. Clean your funnel with water and remove the used filter paper.



Paracetmole Filtration

How it works

1. Recrystallisation is a technique that will further purify your product. Paracetamol is not soluble in cold water but it is when the solvent is hot. Any impurities will also dissolve in hot water. When everything has dissolved the solution is cooled. Paracetamol will crystallise but the impurities will remain dissolved.
2. Watch the 'recrystallisation' video on the laptop provided to show how a recrystallisation is set up and performed.
3. With your crude paracetamol in the clean 100 mL conical flask, add 5 mL of water to your solid. Add another 25 mL of water to a 50 mL conical flask.
4. Place both conical flasks onto your hotplate. Switch on the hotplate to heat both flasks gently. Keep gently swirling both flasks until the water is just below its boiling point.
5. Using a Pasteur pipette, add approximately 1 mL (1 pipette full) of hot water from the conical flask to your paracetamol. Tip: do not invert the pipette whilst transferring the water! Swirl your conical flask again to try and get the solids to dissolve. Return the flask to the stirrer hotplate to keep it hot. Continue this until all the solid has dissolved. Careful! If you add a vast excess of water then your paracetamol might not recrystallise on cooling.
6. Once all the solid has dissolved in the minimum volume of hot solvent, allow the flask to cool to

room temperature slowly. Why do we want the flask to cool slowly?

7. Set up another vacuum filtration with the filtration apparatus. Pre-weigh an empty watch glass and make a note of the weight _____g.
8. When no more precipitate appears to have formed, filter your mixture by vacuum filtration. Wash the solid with a small amount (less than 5 mL) of water.
9. Transfer the solid onto the watch glass and leave to dry in a low temperature oven over lunch.
10. After lunch you will calculate the mass of your product and analyze its purity. In the meantime calculate the theoretical yield of paracetamol. You will use this to calculate the yield of your product after lunch.

Analysis of Paracetamol

In this session you will determine yield and purity of the paracetamol you made.

Part 1 – Calculating the percentage yield of paracetamol

Remove your sample from the oven. Record a weight and calculate your yield.

Weight of paracetamol + watch glass

Yield of paracetamol Percentage yield of paracetamol

Part 2 – Melting point of paracetamol

How it works

Comparing the melting point of your synthesized paracetamol to the known melting point is a good way to ascertain its purity. Any impurities present will lower the melting point and increase the range over which the solid melts.

Watch the 'melting point' video on the laptop to help you understand what to do.

When you are ready, press the open end of the capillary tube onto your solid which should transfer some solid into the tube. Invert the tube and tap the sealed end gently onto a hard surface so the solid drops down to the bottom. Repeat this until you have a depth of 2 mm of sample in the sealed end of the tube.

The full operating instructions for the melting point apparatus are provided with the instrument. First you will rapidly heat your sample to a

'plateau temperature' of 160 °C. Above the plateau, the sample will continue to be heated but at a slower rate of 2-3 degrees per minute which will allow you to record an accurate melting range of your compound.

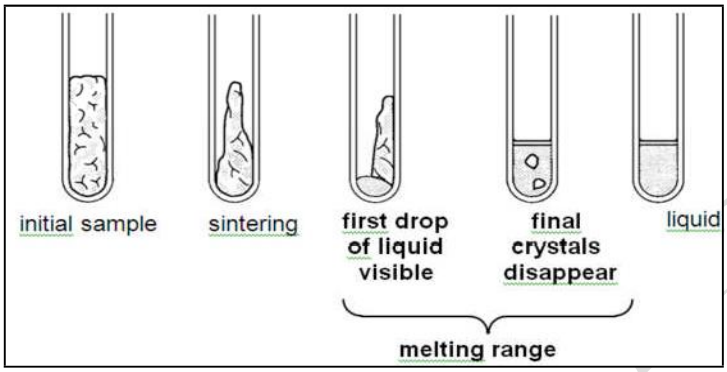
The first temperature is recorded when the first drop of

liquid is apparent. The second temperature is recorded when the entire mass of crystals has been converted into a liquid.

your first temperature here ---------- °C Record your second temperature here -------- °C

Give the melting range of your compound here - °C

Press stop to finish heating and allow the instrument to cool. Remove your sample tube and dispose in the glass waste bin.

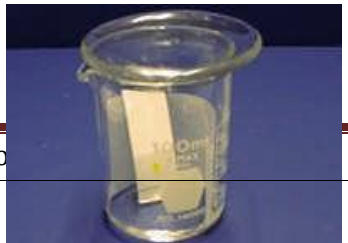


Analysis of Paracetamol

How it works

1. Thin layer chromatography (TLC) is a technique that allows you to determine if your sample contains only paracetamol or is contaminated with other compounds (e.g. starting material). A small amount of your sample and some 4-aminophenol is placed on a TLC plate coated in silica gel. A solvent is passed up the plate which causes the compounds present to travel up the plate. 4-Aminophenol and paracetamol travel up the plate at different rates due to their different structures.
2. When the solvent has nearly run to the top of the plate you will visualise the plate under UV light. You should hopefully see just one spot from your sample that corresponds to paracetamol and that it is different to your 4-aminophenol reference. This will confirm that your sample is pure.
3. Watch the 'thin layer chromatography' video on the laptop to help you understand what to do.
4. Pour at least 10 mL of TLC solvent (2:1:0.1 ethyl acetate/40-60 °C petroleum ether/acetic acid) solution into a 100 mL beaker. Line part of the beaker with a piece of filter paper so that it dips into the solvent and also sticks to the walls. Cover the beaker with a watch glass.

Dissolve a few crystals of 4-aminophenol in approximately 2 mL of ethyl acetate in a sample tube. Repeat the same procedure to prepare a TLC sample of paracetamol. Make sure each tube is properly labeled.



Faintly mark a line above the baseline in pencil. The height of this line 'X' must be higher than the depth of the solvent in the beaker.

TLC plate before elution

Mark 3 points on the baseline so that they are roughly equidistant from each other and the sides of the plate and underneath in pencil label them A, B and C.

Dip a TLC applicator into your 4-aminophenol solution. Capillary action will draw up some of the solution into the applicator. Lightly touch (once or twice) this applicator on

the TLC plates at points A and B (the smaller the applied

X spots the better).

Repeat the same process with your paracetamol solution applied to spot C first then spot B.

Place the TLC plate into the tank with the top of the plate resting against the inside of the beaker (don't forget to replace the watch glass back on top).

You should see solvent travelling up the plate. When the solvent front has reached approximately 1 cm from the top of the plate, remove the plate from the TLC tank. Mark a faint pencil line to indicate how far the solvent has travelled up the plate and allow the solvent to evaporate.

Adverse Effects of Paracetamol:

- Liver damage

Acute overdoses of paracetamol can cause potentially fatal liver damage. In 2011, the U.S. Food and Drug Administration (FDA) launched a public-education program to help consumers avoid overdose, warning: "Acetaminophen can cause serious liver damage if more than directed is used.

- Skin reactions

the U.S. Food and Drug Administration (FDA) issued a warning about paracetamol. It stated that the drug could cause rare and possibly fatal skin reactions such as Stevens–Johnson syndrome (SJS) and toxic epidermal necrolysis

- Other Factors

In contrast to aspirin, paracetamol does not prevent blood from clotting (it is not an antiplatelet), thus it may be used in people who have concerns with blood coagulation. Additionally it does not cause gastric irritation.[88] However, paracetamol does not help reduce inflammation, while aspirin does.[89] Compared with ibuprofen—whose side effects may include diarrhea, vomiting and abdominal pain—paracetamol has fewer adverse gastrointestinal effects.[90] Unlike aspirin, paracetamol is generally considered safe for

children, as it is not associated with a risk of Reye's syndrome in children with viral illnesses.[91] If taken recreationally with opioids, weak evidence suggests that it may cause hearing loss.[92] Paracetamol use may also inhibit the feeling of empathy for another's pain.[93]

4.2 Aspirin:

Aspirin, or acetylsalicylic acid (ASA), is commonly used as a pain reliever for minor aches and pains and to reduce fever. It is also an anti-inflammatory drug and can be used as a blood thinner. Aspirin is a non-steroidal anti-inflammatory drug (NSAID).

Uses

Aspirin is one of the most commonly used drugs for treating mild to moderate pain, migraines, and fever. Common uses include headaches, period pains, colds and flu, sprains and strains, and long-term conditions, such as arthritis.

For mild to moderate pain, it is used alone. For moderate to severe pain, it is often used along with other opioid analgesic and NSAIDs.

In high doses, it can treat or help reduce symptoms of:

- rheumatic fever
- rheumatic arthritis
- other inflammatory joint conditions
- pericarditis

In low doses, it is used:

- to prevent blood clots from forming and reduce the risk of a transient ischemic attack (TIA) and unstable angina
- to prevent myocardial infarction in patients with cardiovascular disease by preventing clot formation
- to prevent a stroke, but not to treat a stroke
- to prevent colorectal cancer

Precautions

Aspirin is not recommended for individuals who:

- have a peptic ulcer
- hemophilia or any other bleeding disorder
- a known allergy to aspirin
- an allergy to any NSAID, such as ibuprofen

- are at risk of gastrointestinal bleeding or hemorrhagic stroke
- drink alcohol regularly
- are undergoing dental or surgical treatment, however small

Side effects

The most common side effects of aspirin are:

- irritation of the stomach or gut
- indigestion
- nausea

The following adverse effects are possible, but less common:

- worsening asthma symptoms
- vomiting
- inflammation of the stomach
- stomach bleeding
- bruising

PROJECT -2

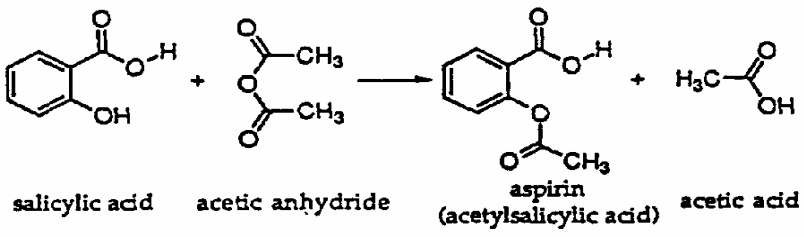
Preparation of Aspirin

Part 1: Preparation of Aspirin

How it works

The 'curly' arrow mechanism below shows the bond forming/breaking process that occurs.

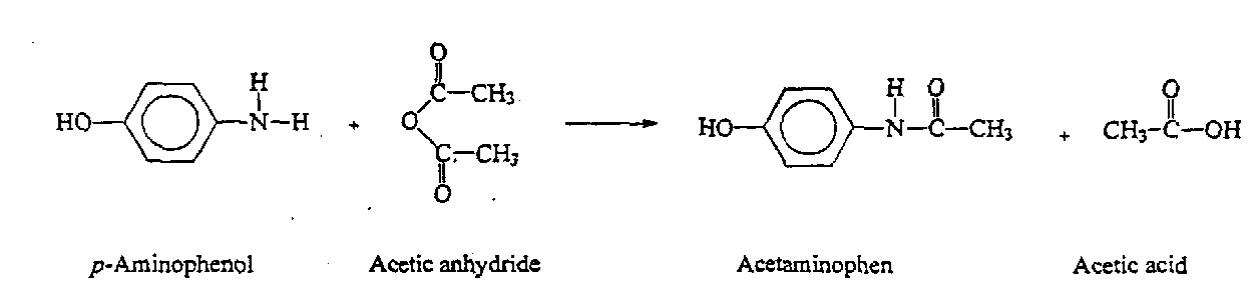
ethanoic anhydride.



Preparation of Aspirin

Safety Precautions

1. Acetic and hydride is corrosive and its vapor irragating respratory system.
2. Sulphuric acid is corrosive. Avoid skin contact.
3. Wear safety glasses in Laboratory.



p-Aminophenol Acetic anhydride Acetaminophen Acetic acid

4-aminophenol

Paracetamol ethanoic acid

The lone pair of electrons on the amine of 4-aminophenol attacks the C=O bond of acetic anhydride causing it to break.

Nitrogen has a positive charge but regains electrons by losing a proton. The negative charge on the oxygen comes back in to reform the C=O bond. This causes the other C-O bond to break.

The result is an amide bond formation and a carboxylic acid by-product.

Remember to wear gloves

4.3 Bromine Water

Bromine water is a highly oxidizing intense yellow to red mixture containing diatomic bromine (Br_2) dissolved in water (H_2O).[1] It is often used as a reactive in chemical assays of recognition for substances which react with bromine in an aqueous environment with the halogenation mechanism, mainly unsaturated carbon compounds (carbon compounds with 1 or more double or triple bond(s)). The most common compounds that react well with bromine water are phenols, alkenes, enols, the acetyl group, aniline, and glucose. In addition, bromine water is commonly used to test for the presence of an alkene which contains a double covalent bond which reacts with the bromine water which changes its color from an intense yellow to a colorless solution. Bromine water is also commonly used to check for the presence of an aldehyde group in compounds. In this reaction as well the color of bromine water is changed to colorless from yellow (oxidation process).

PROJECT-3

Preparation of Bromine Water:

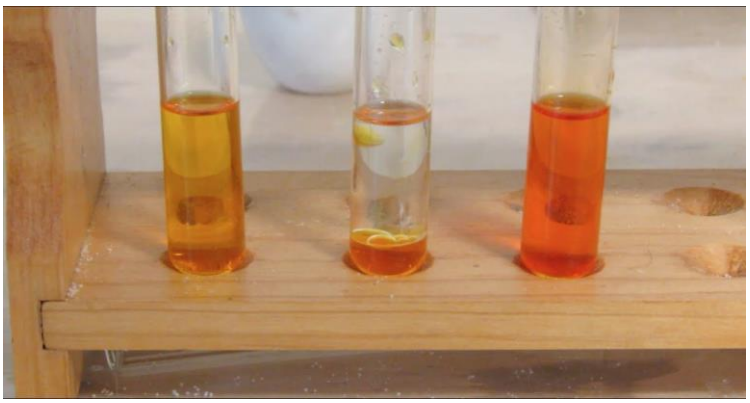
Apparatus:

Test tube, beaker, conical flask , petridate ,glass rod,thermometr, reflex condenser , water bath

Chemicals :

1. Glass bottle with screw cap, label the bottle “Br2(aq)”
2. 1.1 g sodium bromide, NaBr.
3. 10.7 mL 1 M HCl.

4. 7.6 mL sodium hypochlorite (household laundry bleach)
5. 32 mL water (preferably distilled water)



4.4 2,4,6 Tribromophenol:

2,4,6-Tribromophenol (TBP) is a brominated derivative of phenol. It is used as a fungicide, as a wood preservative, and an intermediate in the preparation of flame retardants.

PROJECT- 4

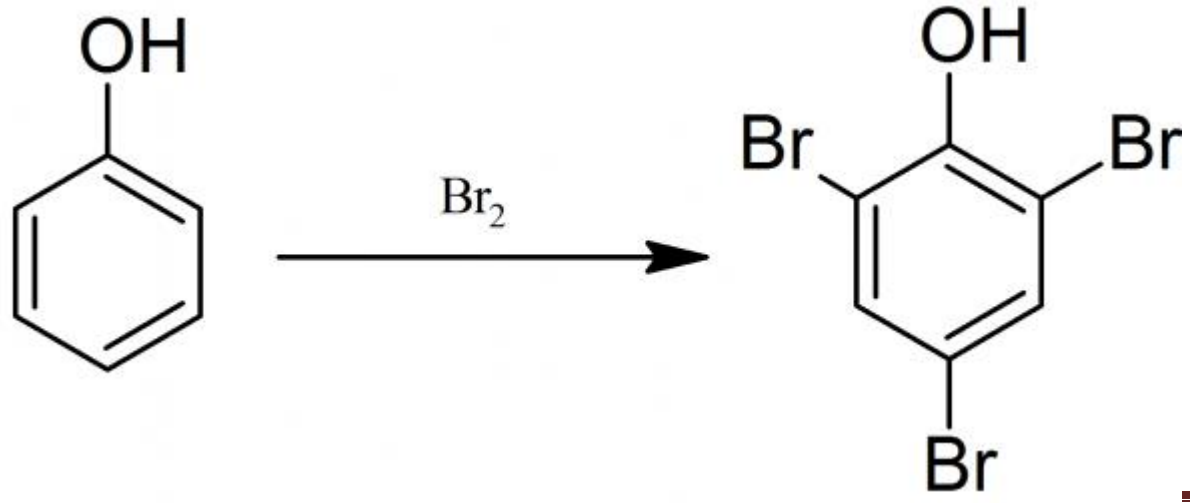
Preparation of 2,4,6 Tribromophenol:

Apparatus : Test tube, beaker, conical flask , petridate ,glass rod,thermometr, reflex condenser , water bath

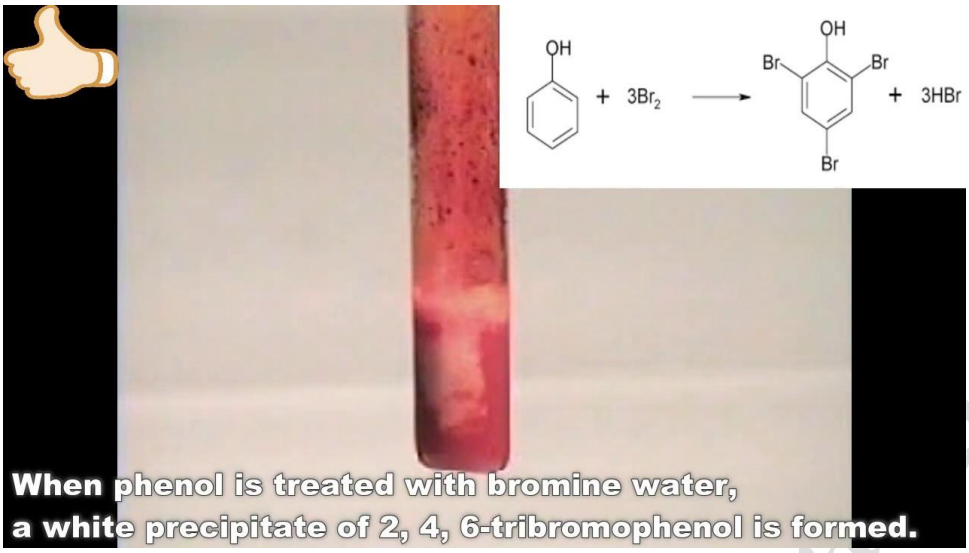
Chemical : phenol , cold water, bromine water, ethyl alcohol

Method :

To a reaction flask 10 g of phenol dissolved in 200 ml of cold water are placed. From a dropping funnel 52 g of bromine in aqueous solution are added and reaction mixture is properly stirred. The precipitated white 2,4,6-tribromophenol is collected, washed with water, and recrystallized from dilute ethyl alcohol



Phenol **2,4,6 Tribhromophenol**



4.5. Acetanilide:

Acetanilide is an odourless solid chemical of leaf or flake-like appearance. It is also known as *N*-phenylacetamide, acetanil, or acetanilide.

PROJECT- 5

Preparation of Acetanilide

Acetanilide is a white organic solid compound used primarily in organic synthesis. N-phenylacetamide, acetanilide and acetanil are other names of this compound. It was used in the past to treat fever and headache and was known as *Antifebrin* by its brand name.

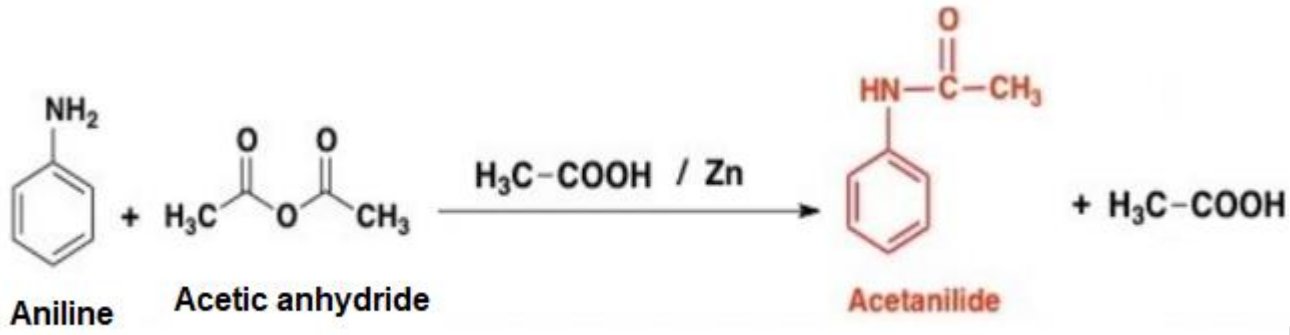
Aim:

To prepare the organic compound acetanilide from aniline, glacial acetic acid/acetic anhydride and zinc dust.

Theory:

Acetanilide is prepared from aniline when it reacts with acetic anhydride/glacial acetic acid in the presence of zinc dust. A mixture of aniline, glacial acetic acid, acetic anhydride and zinc dust is refluxed under anhydrous condition and then poured the mixture into ice cold water to get acetic anhydride precipitate. The crude precipitate of acetic anhydride is recrystallized to get pure crystals of acetanilide.

The chemical reaction is given below.



Zinc is used to prevent the oxidation of aniline during the chemical reaction. Acetanilide is medicinally important and it is used as febrifuge.

Acetanilide cal also be prepared by acetylating aniline with acetic anhydride in the presence of concentrated hydrochloric acid. Dissolve aniline in hydrochloric acid and add acetic anhydride stir well. Pour the mixture to sodium acetate in water. Acetanilide is formed which can be separated and recrystallised by ethyl alcohol.

Other names – N-phenylacetamide, N-phenylethanamide, Acetanil

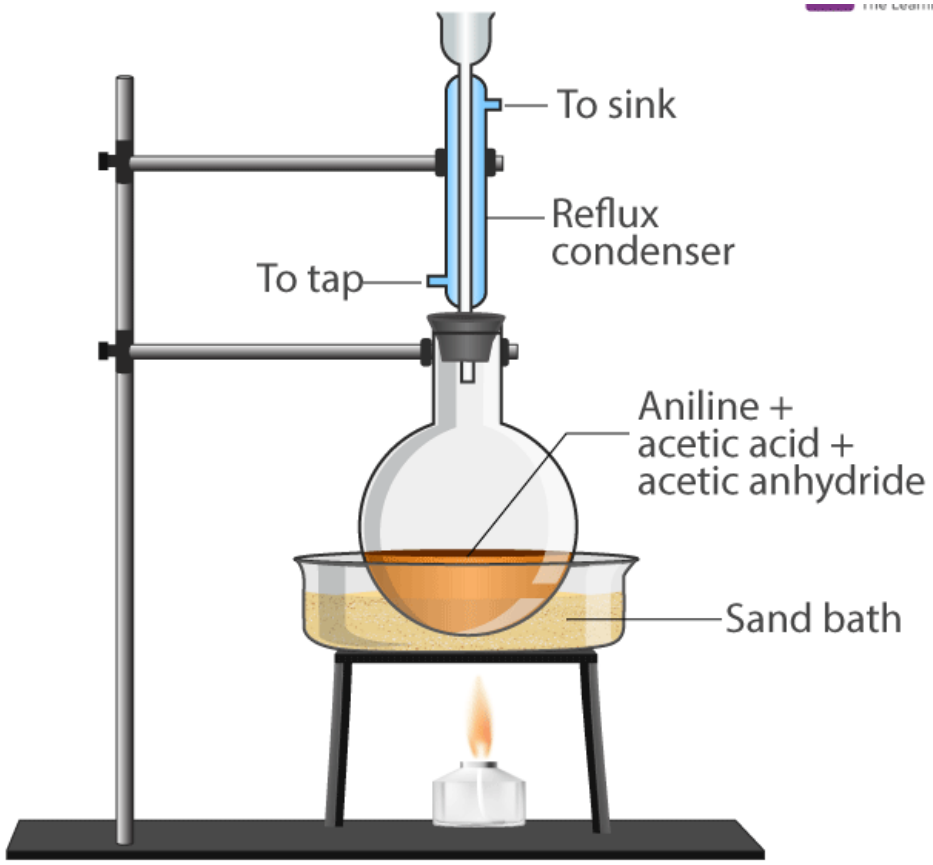
Materials Required:

1. Aniline
2. Glacial acetic acid
3. Acetic anhydride
4. Zinc dust
5. Distilled water
6. Round bottom flask
7. Beaker
8. Pipette
9. Reflux condenser
10. Funnel
11. Stirrer
12. Bunsensurner
13. Filter paper
14. Electronic balance

Chemicals:-

Zinc, Aniline , acetic acid , distill water, sulphuric acid,

Apparatus Setup:



Procedure:

1. Wash all the apparatus with distilled water before starting the experiment.
2. Take a round bottom flask in that add 10ml of aniline and 20ml of acetic anhydride and glacial acetic acid mixture and add zinc dust.
3. Fix the reflux condenser with the round bottom flask.
4. Heat the mixture gently for about 15-20 minutes on oil bath.
5. Pour the hot mixture in a beaker containing ice cold water with constant stirring.
6. Stir the mixture vigorously to hydrolyse excess of acetic anhydride.
7. Once all the acetanilide is precipitated collect and filter in buchner funnel.
8. The precipitate obtained is a crude sample of acetanilide. To get the pure crystals crystallization should be carried out.

Crystallization:

Transfer the crude sample into a beaker containing 20ml water and heat gently. If the solution is coloured then add a small amount of activated carbon. Filter the hot solution with a funnel. Cool the mixture for 30 min so that white shiny crystals of acetanilide separates out. Filter off the crystals, wash them with water and dry in the foldes of filter paper.

Observations:

Colour of the crystals	Colourless crystals
Shape of the crystals	Plate shaped
Melting point	114^{o}C

Results and Discussion:

The yield of Acetanilide is _____gm.

Precautions:

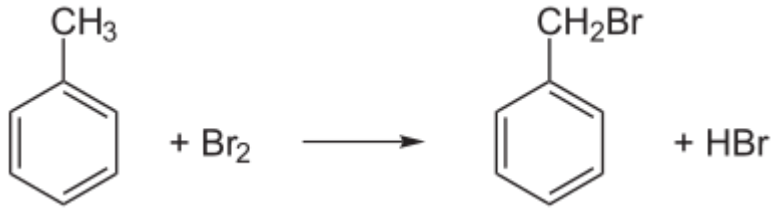
1. Do not inhale the fumes of acetic anhydride.
2. Always carry out experiments in fuming chamber or near the window.
3. Use the water condenser for refluxing the reaction mixture.
4. Dry the crystals of acetanilide before finding the weight and its melting point

4.6. Benzyl Bromide

Benzyl bromide is an organic compound with the formula $C_6H_5CH_2Br$. The molecule consists of a benzene ring substituted with a bromomethyl group. It is a colorless liquid with lachrymatory properties. The compound is a reagent for introducing benzyl groups.

Structure:

Benzyl bromide can be synthesized by the bromination of toluene under conditions suitable for a free radical halogenation:



The structure has been examined by electron diffraction

PROJECT- 6 (contd…)

Preparation of Benzyl Bromide

Chemicals : Toluene, bromine water, distill water etc**.,**

Apparatus : Test tube, beaker, conical flask , petridate ,glass rod,thermometr, reflex condenser , water bath

Method :

To the perfectly dry round bottom flask fitted with a reflux condenser, stirrer and dropping funnel 50 g toluene are placed. As the reaction flask is exposed to direct sunlight (or 500W photolamp) 75 g elemental bromine are slowly added with vigorous stirring. As the reaction proceeds the solution, which is

first colored reddish-brown, becomes colorless. When all the bromine has been added, benzyl bromide is fractionally distilled and the fraction boiling between 190-205° C. collected separately.

Bromo Benyl is a clear liquid

4.7. Preparation of P-Nitro Acetanilide

Para nitroacetanilide is also called 4-Nitroacetanilide, para nitroacetanilide is a chemical compound that is a nitroacetanilide derivative prepared from acetanilide and nitrating mixture. Along with para product a trace of ortho product is also formed.

PROJECT-7

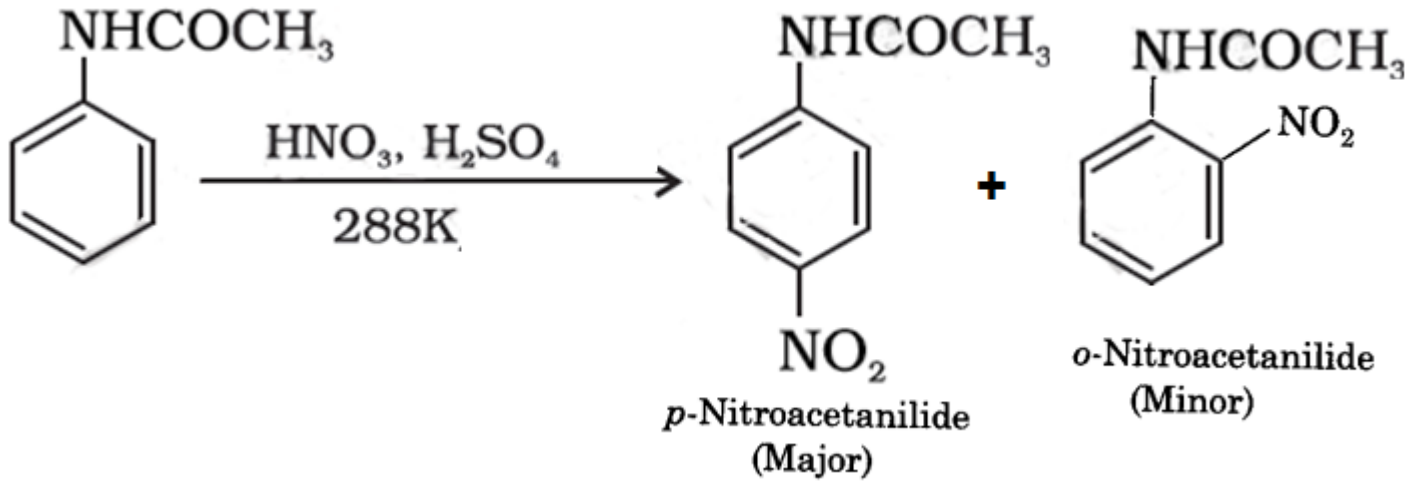
Aim:

To prepare p-Nitroacetanilide from acetanilide and acetic acid in the presence of nitrating mixture.

Theory:

The organic compound p-nitroacetanilide is prepared from acetanilide through *nitration*. When acetanilide is treated with nitrating mixture that is a mixture of nitric acid and sulfuric acid p-nitroacetanilide is formed. Along with p-nitroacetanilide, o-nitroacetanilide is also formed as a minor product. Since o-nitroacetanilide is very much soluble in alcohol it is very easy to isolate p-nitroacetanilide through crystallization.

The chemical reactions involved in this process is given below.



It is an electrophilic substitution reaction. The electrophile -NO_2 will attach the para position because the -$NHCOCH_3$ is an electron releasing group. Nitro anilines can be prepared by this type of reactions because nitration of aniline is not possible, amino group gets oxidised with nitrating mixture. In order to protect the amino group from oxidation acetanilide is first nitrated to give p-nitroacetanilide and then on hydrolysis to give p-nitroaniline which is difficult to obtain by direct nitration.

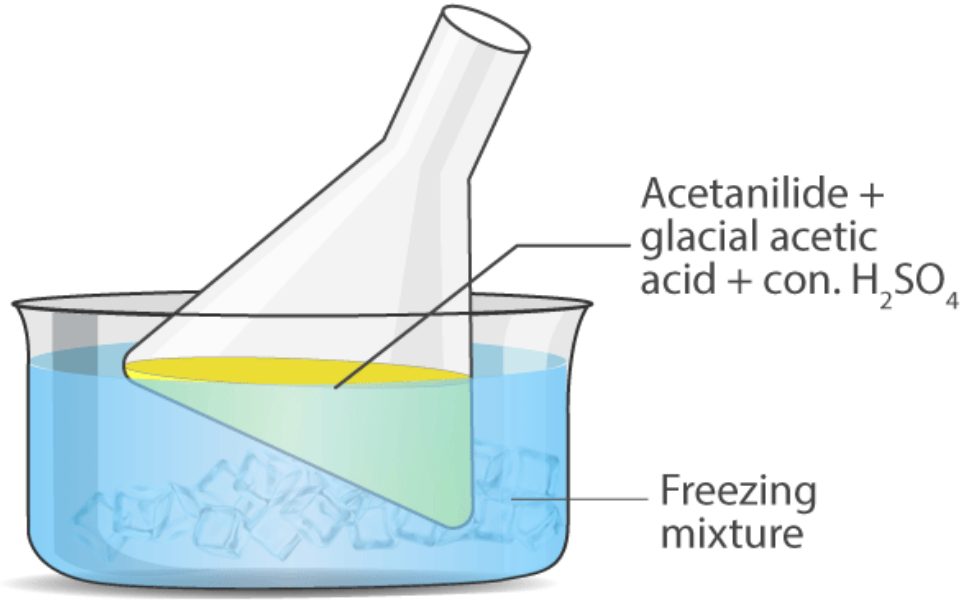
Other names – N-(4-nitrophenyl)acetamide, p-Acetamidonitrobenzene, p-Nitroacetanilide, N-Acetyl-4-nitroaniline

Materials Required:

1. Acetanilide
2. Acetic acid
3. Concentrated Sulfuric acid
4. Fuming Nitric acid
5. Ethyl alcohol
6. Conical flask
7. Beaker
8. Dropping funnel
9. Filter paper
10. Glass rod
11. Buchner funnel
12. Pipette

Chemicals– Acetinalide, concentrated nitric acid (HNO_3), concentrated H_2SO_4

Apparatus Setup:



Preparation of p-nitroacetanilide

Procedure:

1. Take 3gm of finely powdered acetanilide in a clean beaker and dissolve it by adding glacial acetic acid by stirring the content carefully at room temperature.
2. Gently warm the mixture to dissolve acetanilide completely.
3. Cool the solution and add concentrated sulfuric acid slowly with constant stirring. The solution becomes warm, keep the mixture in ice-bath and clear solution is obtained.
4. To the cool solution add fuming nitric acid dropwise through a dropping funnel with constant stirring.
5. Maintain the temperature below 20^{o}C during the whole process.
6. Once the addition of nitric acid is completed, the beaker is removed from the freezing mixture bath and allow to stand for half an hour at room temperature.
7. Pour the mixture into 100gm of crushed ice in a beaker and stir well.
8. Large crystals of p-nitroacetanilide is obtained. Filter the crystals through filter paper.
9. The separated p-nitroacetanilide is washed with cold water inorder to remove excess of acid.

10. It is crystallized from ethyl alcohol. Dry the crystals in the folds of filter paper and weigh them to know the yield.

Observations:

Colour of the crystals	Colourless
Melting point	214°C
Expected yield	4gm

Results and Discussion:

The yield of p-Nitroacetanilide is _____gm.

Precautions:

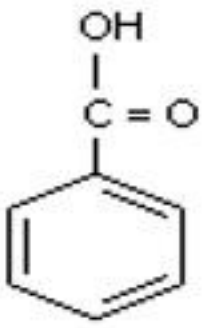
1. Temperature should not exceed more than 20°C.
2. Advisable to add nitric acid into the reaction mixture while it is immersed in ice-bath
3. Add fuming nitric acid drop by drop carefully and do not inhale the fumes of nitric acid.

4.8. Preparation of Benzoic acid from sodium benzoate:

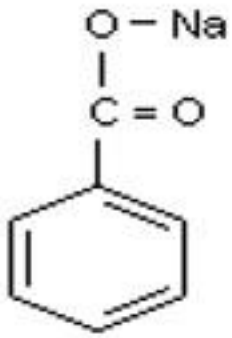
The benzoate anion is not soluble in non polar solvents because of its negative charge However, in acid solution, benzoic acid is formed. This is neutral & quite non- polar moreover it is soluble in non –polar solvents.

Chemicals: sodium Benzoate, Dilute HCL, distill water , etc.,

Apparatus: Test tube, beaker, conical flask , petridate ,glass rod,thermometr, reflex condenser , water bath



Benzoic acid



Sodium benzoate

CHAPTER-5

INORGANIC PREPARATIONS

5.1. CHROME ALUM

Chrome alum or Chromium(III) potassium sulfate is the potassium double sulfate of chromium. Its chemical formula is $KCr(SO_4)_2$ and it is commonly found in its dodecahydrate form as $KCr(SO_4)_2 \cdot 12$. It is used in leather tanning.

PROJECT- 1

PREPARATION OF CHROME ALUM

$$K_2SO_4 + Cr_2(SO_4)_3 + 24\ H_2O \longrightarrow 2KCr(SO_4)_2 \cdot 12H_2O$$

Chemicals – potassium dichromate , sulphuric acid , ethyl alcohol, distill water etc.,

Apparatus : Test tube, beaker, conical flask , petridate ,glass rod,thermometr, reflex condenser , water bath

Method:

98 grams of potassium dichromate are placed in a porcelain dish containing 400 ml of water. 76 ml of concentrated sulfuric acid are slowly added and stirred until all the salt is dissolved. If potassium dichromate has not completely dissolved the mixture is warmed gently. After all the salt has dissolved, the solution is filtered if it is not absolutely clear. Then 63 ml of ethyl alcohol are cautiously added, by stirring the reaction mixture after each addition and by allowing the heat of the reaction to raise the temperature of the solution to the boiling point. When all ethanol has been added the flask is covered and the hot solution is left to stand for a week or longer. The obtained crystals of chrome alum are filtered and if the filtrate is evaporated the additional amount of chrome alum is obtained. The crystals are dried and stored in a bottle since they are efflorescent. Chrome alum obtained by described method contains twelve molecules of crystalized water (dodecahydrate) – $KCr(SO_4)_2 \cdot 12H_2O$.

Chrome Alum

5.2. Ferrous Ammonium Sulphate

Ammonium iron(II) sulfate, or Mohr's salt, is the inorganic compound with the formula (NH4)2Fe(SO4)2(H2O)6. Containing two different cations, Fe2+ and NH4+, it is classified as a double salt of ferrous sulfate and ammonium sulfate. It is a common laboratory reagent because it is readily crystallized, and crystals resist oxidation by air. Like the other ferrous sulfate salts, ferrous ammonium sulfate dissolves in water to give the aquo complex [Fe(H2O)6]2+, which has octahedral molecular geometry. Its mineral form is mohrite.

PROJECT-2

Preparation of Ferrous Ammonium Sulphate

Ferrous Ammonium Sulphate is also called Mohr's salt a light green color sand like crystalline solid. It is a hazardous substance and preparation of ferrous ammonium sulfate is carried out from equimolar mixture of hydrated ferrous sulphate and ammonium sulphate in water; containing a little of sulphuric acid.

Aim

To prepare Mohr's salt: ferrous ammonium sulphate from ferrous sulfate and ammonium sulfate in the presence of acid.

Theory

The formula for ferrous ammonium sulfate is $FeSO_4.(NH_4)_2SO_4.6H_2O$. It is prepared by dissolving equimolar mixture of hydrated ferrous sulfate and ammonium sulfate in water containing a little sulfuric acid. The solution is subjected to crystallization, ferrous ammonium sulfate separates out from the solution.

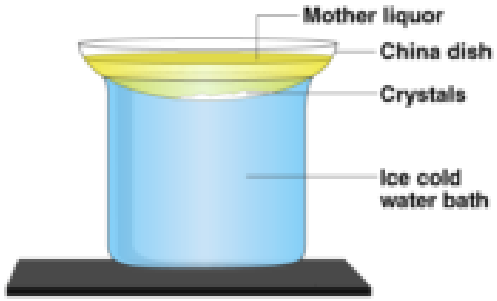
The chemical reaction is given below.

$FeSO_4 + (NH_4)_2SO_4 + 6H_2O \rightarrow FeSO_4.(NH_4)_2SO_4.6H_2O$ (Mohr's salt)

The addition of sulfuric acid in this experiment prevents the hydrolysis of this salt. Ferrous ammonium sulfate is a pale green crystalline compound which does not effloresce like ferrous sulfate. It is less readily oxidised than $FeSO_4$ and therefore, a better volumetric reagent in preference to ferrous sulfate.

Mohr's salt is also called as double salt which contain more than one simple salt. It undergoes complete dissociation in aqueous solutions. Mohr's salt dissociates into Fe^{2+}, NH_4^+, SO_4^{2-} ions and give their individual chemical test.

Mohr's salt is light green transparent octahedral crystals. It is soluble in water giving acidic solution which turns blue litmus paper red and gives effervescence with sodium bicarbonate evolving carbon dioxide.



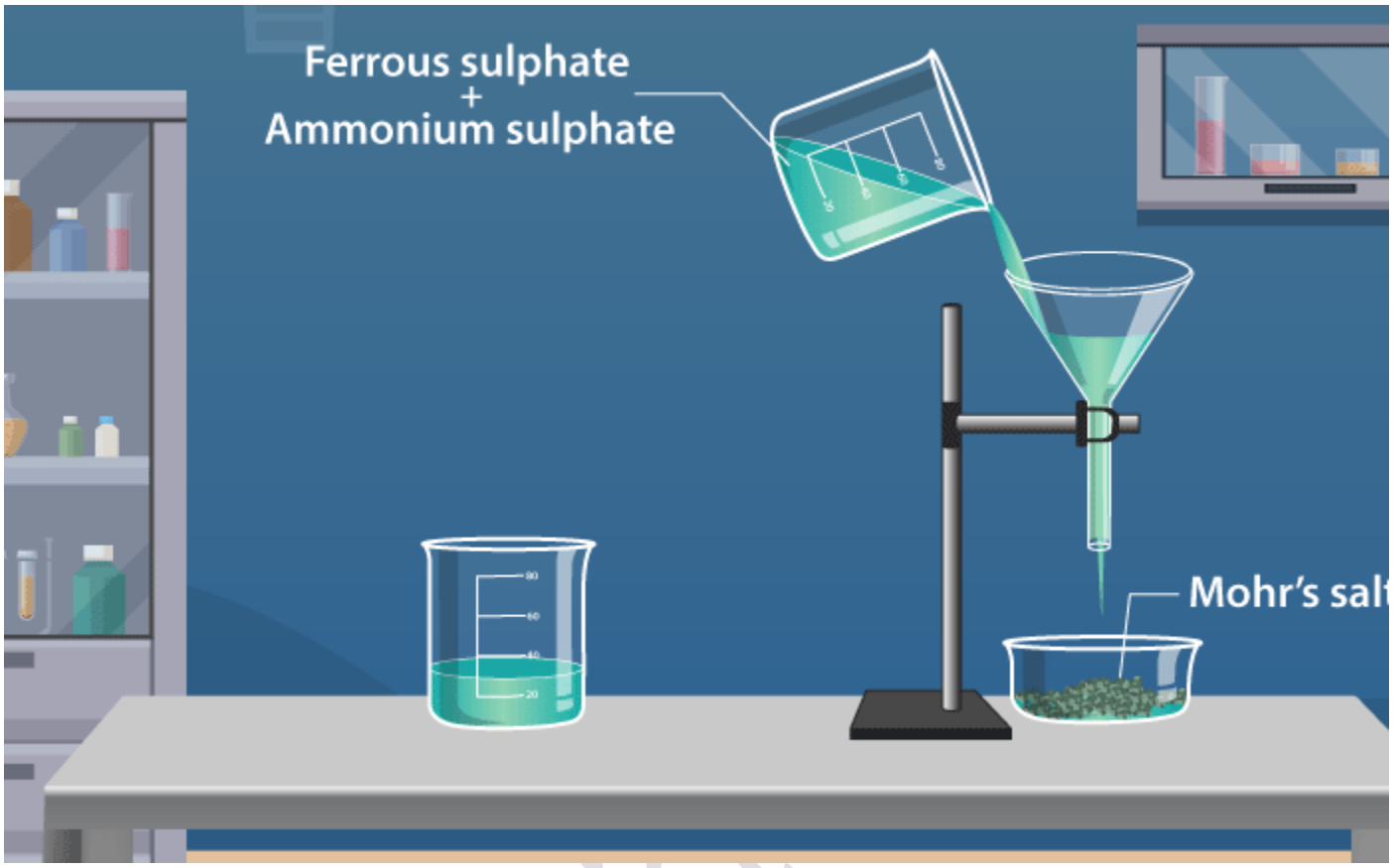
Preparation of Mohr's Salt

Ferrous Ammonium Sulphate also called mohr's salt is prepared by dissolving equimolar mixture of hydrated ferrous sulphate and ammonium sulphate in water containing a little sulphuric acid. The solution is subjected to crystallization.

Materials Required

1. Conical flask
2. Tripod stand
3. Burner
4. Funnel
5. Watch glass
6. Glass rod
7. Filter paper
8. Wire gauze
9. China dish
10. Ferrous sulfate
11. Ammonium sulfate
12. Dilute sulfuric acid

Apparatus Setup



Laboratory Preparation of Ferrous Ammonium Sulphate – Mohr's Salt

Procedure

1. Weigh 7g of ferrous sulfate and 3.5g of ammonium sulfate separately.
2. Mix ferrous sulfate and ammonium sulfate in water in a beaker containing dilute sulfuric acid.
3. Gently warm the solution in order to get a clear solution.
4. Filter the solution in order to remove suspended impurities and concentrate the clear filtrate by heating it china dish over a sand bath till the crystallization point is reached.
5. Stir the solution occasionally during heating.
6. Keep the solution undisturbed for slow cooling. After some time crystal of ferrous ammonium sulfate will get separated from the solution.
7. Separate the crystals by decantation from the mother liquor and wash the crystals with cold water.
8. Dry the crystals between the folds of filter paper or by spreading on a porous plate.

Observations

Colour of the crystal	Light green

Shape of the crystal	Octahedral
Solubility in water	Soluble
Action of blue litmus paper	Blue litmus turns red

Results and Discussion

The yield of ferrous ammonium sulfate or Mohr's salt is _____ gm.

Precautions

- Allow slow cooling and do not disturb the solution during cooling in order to get good quality crystals.
- During the process heating of the solution should be done in a short time only. Because prolonged heating forms ferric ions along with ferrous ammonium sulfate.
- Suppose if the solution is yellow in colour instead of green the experiment should be repeated.

5.3. Preparation of Potash Alum

What is Potash alum?

Potash alum is also called potassium aluminium sulfate is a chemical compound commonly encountered as the dodecahydrate. It is a double salt widely used in medicine and in the water purification process. Potash alum is not a complex salt. The chemical formula of potash alum is $K_2SO_4.Al_2(SO_4)_3.24H_2O$.

PROJECT- 3

Aim:

To prepare potash alum from potassium sulfate and aluminium sulfate through crystallization.

Theory:

The formula for potash alum is $K_2SO_4.Al_2(SO_4)_3.24H_2O$. It is prepared by crystallizing the alum from a concentrated solution containing equimolar amounts of potassium sulfate and aluminium sulfate. It is a colourless, crystalline solid with a sour taste. The crystal of potash alum is octahedral in shape. It is commonly known as 'fitkari'.

The chemical reaction is given below.

$K_2SO_4(aq) + Al_2(SO_4)_3(aq) \rightarrow K_2SO_4.Al_2(SO_4)_3.24H_2O(s)$ (Potash Alum)

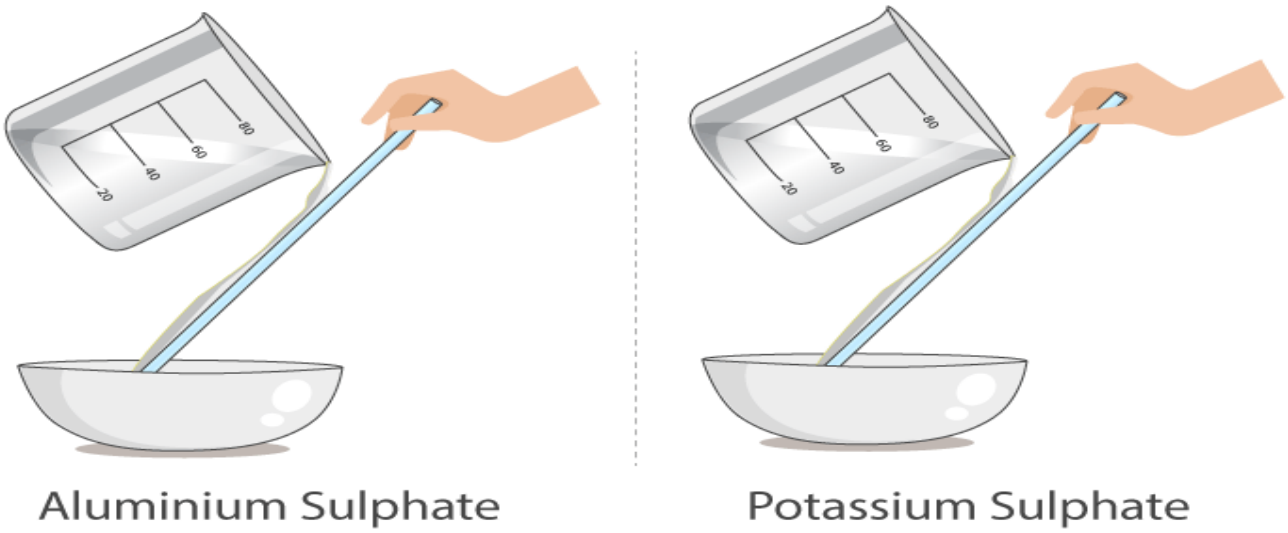
While dissolving aluminium sulphate in warm water a little amount of dilute sulfuric acid is added in order to prevent the hydrolysis of this salt.

When a solution containing two inorganic salts in a definite proportion is allowed to crystallize a double slat is said to have separated. The name alum is given to the special series of double salts. Aluminium is the most abundant metal and the recycling of aluminium products by melting and recasting into other metal products are used in the production of various aluminium compounds. In that one of the most useful compounds is potash alum.

Materials Required:

1. Potassium sulphate
2. Aluminium sulphate
3. Dil. sulfuric acid
4. Distilled water
5. Beaker
6. Conical flask
7. Tripod stand
8. Funnel
9. Burner
10. China dish
11. Wire gauze
12. Filter paper

Apparatus Setup:



Procedure:

1. Weigh 12.5g of potassium sulfate and dissolved in a minimum quantity of distilled water in a beaker. Stir to dissolve the crystals.
2. Take a conical flask, in that dissolve 50g of aluminium sulfate in warm water and add 3ml of dilute sulfuric acid to make the solution clear.
3. Filter the solutions if it is not clear.
4. Mix the two clear solutions in a china dish.
5. Place the china dish on a wire gauze over a burner.
6. Stir the solution and concentrate the solution till the crystallization point is reached.
7. Place the solution over a beaker containing cold water for a few hours.
8. Crystals of potash alum will get separated, filter then from the mother liquor and wash them with a small quantity of cold water.
9. Dry the crystals by pressing gently between the folds of the filter paper.
10. Weigh them on the chemical balance to know the yield.

Observations:

Colour of the crystal	Colourless
Shape of the crystal	Octahedral
Solubility in water	Soluble
Action of blue litmus paper	Blue litmus turns red

Results and Discussion:

1. The yield of Potash alum is _____ gm.
2. Expected yield is _____ gm.

Precautions:

- To prevent hydrolysis of aluminium sulfate, dilute sulfuric acid should be added while preparing the saturated solution.
- During crystallization do not disturb the solution.
- For dissolving salts always use warm water.
- The concentrated solution should be cooled slowly.

Chapter-6

CHEMICAL DISINFECTANTS

6.1. PREPARATION OF ALCOHOLIC DISINFECTANT :

In the healthcare setting, "alcohol" refers to two water-soluble chemical compounds—ethyl alcohol and isopropyl alcohol—that have generally underrated germicidal characteristics [482]. FDA has not cleared any liquid chemical sterilant or high-level disinfectant with alcohol as the main active ingredient. These alcohols are rapidly bactericidal rather than bacteriostatic against vegetative forms of bacteria; they also are tuberculocidal, fungicidal, and virucidal but do not destroy bacterial spores. Their cidal activity drops sharply when diluted below 50% concentration, and the optimum bactericidal concentration is 60%–90% solutions in water (volume/volume) [483, 484].

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Mode of Action.

The most feasible explanation for the antimicrobial action of alcohol is denaturation of proteins. This mechanism is supported by the observation that absolute ethyl alcohol, a dehydrating agent, is less bactericidal than mixtures of alcohol and water because proteins are denatured more quickly in the presence of water [484, 485]. Protein denaturation also is consistent with observations that alcohol destroys the dehydrogenases of Escherichia coli [486], and that ethyl alcohol increases the lag phase of Enterobacter aerogenes [487] and that the lag phase effect could be reversed by adding certain amino acids. The bacteriostatic action was believed caused by inhibition of the production of metabolites essential for rapid cell division.

Microbicidal Activity.

Methyl alcohol (methanol) has the weakest bactericidal action of the alcohols and thus seldom is used in healthcare [488]. The bactericidal activity of various concentrations of ethyl alcohol (ethanol) was examined against a variety of microorganisms in exposure periods ranging from 10 seconds to 1 hour [483]. Pseudomonas aeruginosa was killed in 10 seconds by all concentrations of ethanol from 30% to 100% (v/v), and Serratia marcescens, E, coli and Salmonella typhosa were killed in 10 seconds by all concentrations of ethanol from 40% to 100%. The gram-positive organisms Staphylococcus aureus and Streptococcus pyogenes were slightly more resistant, being killed in 10 seconds by ethyl alcohol concentrations of 60%–95%. Isopropyl alcohol (isopropanol) was slightly more bactericidal than ethyl alcohol for E. coli and S. aureus [489].

PROJECT-1

Aim :Preparation of Alcoholic Disinfectant

Apparatus :

Burette, measuring cylinder,conical flask , stirrer , pH paper etc.,

Chemicals :

1. Iso propene alcohol -99 %
2. Ethyl Alcohol -90%
3. Distill Water
4. Scented essential oil
5. Oleive oil

Method :

1. Mix iso propene alcohol 99 % 100 ml with distill water – 20 ml
2. Add 90% ethyl alcohol 20 ml
3. Add scented essential oil (rose oil ,levander oil etc.,)
4. Add oleive oil 20 drops

6.2 BLEACH SOLUTION

Bleach is a chemical compound derived from natural sources used to whiten fabrics. Bleach works by the process of oxidation, or the alteration of a compound by the introduction of oxygen molecules.

PROJECT -2

Aim: Preparation of bleach solution

Step 1 :material required

Sodium hypo chlorite

Calcium hypo chlorite + distill water

Step 2- Quantity :

Water – 4 litres

sodium hypo chlorite 300 gm

Salt 250 gms

NaOH 250 gm

$NaCO_3$ 100 gm

Solution of calcium hypo chlorite 250 ml

Step -3 Method

Stand for 3 days filter the mixture. Light yellow colour solution is the bleach solution

6.3 ROOM FRESHNER

Air fresheners are consumer products that typically emit fragrance and are used in homes or commercial interiors such as restrooms, foyers, hallways, vestibules and other smaller indoor areas, as well as larger areas such as hotel lobbies, auto dealerships, medical facilities, public arenas and other large interior spaces. There are many different methods and brands of air fresheners. Some of the different types of air fresheners include electric fan air fresheners, gravity drip hygiene odor control cleaning systems, passive non-mechanical evaporating aroma diffusers, metered aerosol time-operated mist dispensers, sprays, candles, oils, gels, beads, and plug-ins. Some air fresheners contain chemicals that provoke allergy and asthma symptoms or are toxic. Air freshening is not only limited to modern day sprays, air freshening also can involve the use of organic and everyday house hold items. Although air fresheners are primarily used for odor elimination, some people use air fresheners for the pleasant odors they emit.

PROJECT-3

Aim : Preparation of Room freshener

1. **Summer Citrus Air Freshener Spray**

- 3/4 cup water
- 2 tablespoons vodka, rubbing alcohol, or real vanilla extract
- 5 drops wild orange essential oil
- 5 drops lemon essential oil
- 5 drops lime essential oil
- 5 drops grapefruit essential oil

2. **Citrus Mint Air Freshener Spray**

- 3/4 cup water
- 2 tablespoons vodka, rubbing alcohol, or real vanilla extract
- 10 drops wild orange essential oil
- 8 drops peppermint essential oil

3. **Sweet Lavender Air Freshener Spray**

- 3/4 cup water

- 2 tablespoons real vanilla extract
- 10 drops lavender essential oil
- 5 drops chamomile essential oil

6.4. MOSQUITO REPELLENT

Mosquito Repellent is the product which protects mainly human beings from the deadly mosquitoes. Before proceeding about the mosquito repellent and about its usage and all, let us first analyze what is mosquito and repellent. Mosquito is a common flying insect that is found around the world

PROJECT-4

PREPARATION OF MOSQUITO REPELLENT

1. Lemon Eucalyptus Oil

You Will Need

- 10 mL of lemon eucalyptus oil
- 90 mL of any carrier oil (olive or coconut oil)

What You Have To Do

1. Take a 100 mL bottle and add 10 mL of lemon eucalyptus oil to it.
2. Add 90 mL of any carrier oil to the lemon eucalyptus oil and mix well.
3. Apply this mixture directly to the affected area.

2. Peppermint Oil And Coconut Oil

You Will Need

- 12 drops of peppermint oil
- 30 mL of coconut oil

What You Have To Do

1. Mix peppermint oil with coconut oil.
2. Apply this mixture directly to your hands and legs.

3. Neem Oil And Coconut Oil

You Will Need

- 10 drops of neem oil
- 30 mL of coconut oil

What You Have To Do

1. Add neem oil to coconut oil.
2. Mix well and apply this directly to the exposed areas of your body.

4. Apple Cider Vinegar With Essential Oils Spray

You Will Need

- 50 mL of apple cider vinegar
- 50 mL of water
- 10-12 drops of essential oil (clove, citronella or eucalyptus oil)

What You Have To Do

1. Mix apple cider vinegar and water in equal proportions.
2. Add a few drops of an essential oil of your choice and mix well.
3. Store this solution in a bottle with a pump.

6. Citronella Oil And Alcohol Spray

You Will Need

- 10 mL of alcohol
- 10 drops of citronella oil
- 90 mL of water

What You Have To Do

1. Mix alcohol and water in the specified proportions.
2. To this, add citronella oil and mix well.
3. Put this in a bottle and spray on the exposed areas of your body.

6.5 HAND SANITIZER

Hand sanitizers reduce levels of microorganisms by killing them chemically, just like disinfectants kill germs on environmental surfaces. The magnitude of the effect of handwashing is mainly a function of wash time and soap usage. Washing hands without soap is much less effective.

PROJECT-5

PREPARATION OF HAND SANITIZER

1. **The Quick (Gel) Recipe**

Materials Required :

- Isopropyl alcohol (also here)
- Aloe vera gel (also here)
- Tea tree oil (also here)

Method :

Mix 3 parts isopropyl alcohol to 1 part aloe vera gel. Add a few drops of tea tree oil to give it a pleasant scent and to align your chakras.

2. **The Better (Spray) Recipe**

Materials required :

1. Isopropyl alcohol (also here)
2. Glycerol or glycerin (also here)
3. Hydrogen peroxide (Walmart also has it in some stores.)
4. Distilled water (also here)
5. Spray bottle

Method :

The aloe mixture gets the job done, but aloe also leaves your skin annoyingly sticky. So, here's a recipe that's less sticky and more potent, based on the mix recommended by the WHO.

Mix 12 fluid ounces of alcohol with 2 teaspoons of glycerol. You can buy jugs of glycerol online, and it's an important ingredient because it keeps the alcohol from drying out your hands. If you can't find glycerol, proceed with the rest of the recipe anyway and just remember to moisturize your hands after applying the sanitizer.

6.6 ROOM SANITIZER

Hand sanitizer is a liquid, gel, or foam generally used to decrease infectious agents on the ... Hand sanitizers were first introduced in 1966 in medical settings such as ... There have been some rare instances where alcohol has been implicated in starting fires in the operating room

PROJECT-6

Aim : **Preparation of Room Sanitizer**

Material Require: iso Prophyl alcohol, distill water, Dettol, Hydrogen peroxide ,

Apparatus : beaker, measuring cylinder, stirrer, funnel.spray bottle etc.,

Method – Formula 1

Iso prophyl alcohol 100 ml + 30 ml distill water + 20 ml ethyl alcohol

Uses:

1. It is used for disinfecting TV screen, Laptop Screen
2. To lean looking glasses

Formula -2

Ditill Water 200ml + 10 ml 30% of hydrogen peroxide,

Uses:

1. It can be used to disinfect mirror , table top etc.,

Formula -3

200 ml distill water + 10 gm sodium hypo chloride(liquid)

Uses :

For disinfecting floor, doors etc.,

Formula 4

Tap water + few drops of dettol can be used to disinfect washroom, toilet etc.,