Experiments in Analytical Chemistry

by

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Introduction

The Analytical chemistry laboratory manual for the student of chemical engineering department include the most important types of chemical glassware with their nomenclature, Chemical Safety in the Laboratory Conduct and describes the experimental procedures with a brief theoretical background and chemical equations.

Laboratory Report:

1. Cover Page: Title of the experiment, identify yourself and your partners and Date of Performing the experiment.
2. Purpose or Objective
3. Theoretical Basis.
4. Apparatus and materials required.
5. Results and calculation.
6. Discussion.
8. References.
Chemical Safety in the Laboratory

There are a number of rules that must be followed in order to work safely in a chemical laboratory. Some of these rules are listed below.

1. Always wear your laboratory while working in the laboratory.
2. Always check the label on the bottle before using a chemical reagent.
3. Never taste a chemical or a solution, many laboratories chemical are poisonous
4. Keep your face at a safe distance if your experiment generates an objectionable gas. Work under the suction hood.
5. Don't pick hot objects with your bare hand. If your hand is burned, use some burn ointment
6. Avoid using bare hand in the transfer of chemicals.
7. In the event of malfunction in any tool or device within the laboratory must inform the offices and not repaired by the student.
8. Wash your hands well before leaving the laboratory
9. To avoid spattering of acids which can cause burns, always add acid to water. Never add water to acid.
10. Before obtaining any reagents, carefully read the labels on the bottles twice. Many chemicals have similar names or concentrations may vary.
11. To avoid unnecessary waste, obtain only the amount of chemicals called for in an experiment. Your instructor will tell you the proper procedure for
dispensing liquids and solids. Never insert medicine droppers or scoops into reagent bottles.

12. Never return unused chemicals to the original dispensing bottle.
e-mail: uot_chemical@hotmail.com or chemical_eng@uotechnology.edu.iq
website: http://www.uotechnology.edu.iq/dep-chem-eng/index.htm
Volumetric (Titrimetric) analysis:

Volumetric analysis (or Quantitative chemical analysis): Titration is the slow addition of one solution of a known concentration (called a titrant or titrator) to a known volume of another solution of unknown concentration (called a titrand or analyte) until the reaction reaches neutralization, which is often indicated by a color change.

- The standard solution is usually added from a graduated vessel called a burette.
- The process of adding the standard solution until the reaction is just complete is termed a titration.
- The solution of accurately known strength is called the standard solution.
- The standard solution is usually added from a graduated vessel called a burette.
- The process of adding the standard solution until the reaction is just complete is termed a titration.
- The reagent of known concentration is called the titrant, and the substance being titrated is termed the titrand.
- Equivalent point is the point in a titration where the amount of standard titrant added is chemically equivalent to the amount of analyte in sample.
- End point is the point in a titration when an observable change that signals of the titrant's amount added is chemically equivalent to the amount of analyte in sample.
- Indicator is a reagent used to indicate when the end point has been reached.
A primary standard is a highly purified compound that serves as a reference material in all volumetric titrimetric methods.

**Important requirements for primary standard compounds are:**

- Extremely pure
- Highly stable
- Anhydrous
- Less hygroscopic
- High molecular weight relatively
- Can be weighed easily
- Should be ready to use and available
- Should be preferably non toxic
- Should not be expensive

**Calculations of volumetric analysis:**

Molar solution:

Normal solution:

Part per million (ppm):

**Equivalent weight:**
(1) Equivalent weight in neutralization reaction:

- Equivalent weight of Acid = molecular weight/ number of $H^+$

- Equivalent weight of base = molecular weight/ number of $OH^-$

(2) Equivalent weight in Oxidation – Reduction:

The equivalent weight of an oxidant or a reducing is the number of electrons which 1 mol of the substance gains or losses in the reaction.

Ex. Potassium Permanganate $\text{KMnO}_4$ to $\text{MnO}_2$

(3) Equivalent weight of salt = molecular weight/ valency of the metal ion

Ex: When silver nitrate reacts with sodium chloride, to form silver chloride, the equivalent weight of $\text{AgNO}_3$ is:
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**Preparation of Standard solution**

- **Primary standard solution:**

  The primary standard is a highly purified compound that serves as a reference material in volumetric and mass titrimetric methods. The accuracy of a method is critically dependent on the properties of this compound. Important requirements for primary standard are the following:

  - High purity (e.g. 99.9% by weight), atmospheric stability, absent of hydrate water,
  - Modest cost, reasonable solubility in the titration medium and reasonable large molar mass. One of such compounds is sodium carbonate (Na$_2$CO$_3$).

- **Procedure:**

  To prepare 250ml of 0.1N Sodium carbonate (Na$_2$CO$_3$).

  - Dry Appropriate quantity of sodium carbonate in an oven set at 110°C for 1hr.
    Store the dried material in desiccators.

  - Calculate the weight of (Na$_2$CO$_3$) from the law.
• Weight (Na₂CO₃) using a microbalance (1.0 or 0.1 mg accuracy).

• Transfer this quantity into a 250 ml beaker and solve it in about 50ml distilled water (D.W). Transfer the solution into a 250ml volumetric flask. Complete the solution to the mark by distilled water with the same beaker. Stopper the volumetric flask and shake well.

**Calculation:**

\[
N = \frac{wt}{eq.wt} \times \frac{1000}{V \text{ in ml}} \text{ eq/L}
\]

\[
eq w.t = \frac{M.wt}{\text{No. of eq}}
\]

Wt = Wight of Na₂CO₃ (gm)

V = volume of volumetric flask (ml)

N = Normality of solution
Discussion:

- Determine the volume (ml) required to dilute a solution containing 48.480g of K₂SO₄ in order to prepare a 1 N of the solution.

- What are the specifications of primary standard solution?

- Secondary Standard solution

Some compounds consider as secondary standard solution due missing one of the important requirements for primary standard. For that can prepare as an approximate normal solution and then calibrate with a known standard chemical solution by volumetric analysis. Such as: hydrochloric acid HCL, sodium hydroxide NaOH.

Procedure:

Preparation of approximately (0.1N) HCl from 37% and 1.2 Sp.g

- Calculate the Normality of the concentrated HCl from the law:

- To prepare (500ml) of 0.1N HCL:

\[ N_1 \cdot V_1 = N_2 \cdot V_2 \]
\[ N_1 \times ? = 0.1 \times 500 \]

- Transfer some of distilled water to 500ml volumetric flask and add the calculated volume of conc. HCl. Mix the content thoroughly. Complete to the mark with distilled water and make a label.

**Discussion:**

- What are the specifications of secondary standard solution?
- Determine the volume (ml) required to dilute a solution of HCl from conc. 37% and Sp.g. 1.2 to 1000ml if the Normality is 0.25 eq/l

**Titration Curves**

To see the change in (pH) of solution during the process of titration points, that is known as titration curve.

**Titration Curve:** it is a graph representing the relationship between (pH) for the solution with the volume of the additive in the process of titration. When strong
acid as (HCl) titrated against strong base such as (NaOH) both will completely ionize.

This means that the (H⁺) and (OH⁻) ions be (H₂O) and other (Na⁺ & Cl⁻ ) remains in solution as ions.

**Procedure:**

- Put 20ml solution of HCl (0.25M) in the Beaker size (250ml).
- Fill the burette with (0.1M) NaOH solution.
- Add two drops of Phenolphthalein (ph.ph.) as an indicator then appointed the pH of the solution (using pH Meter).
- Add (0.1N) NaOH drop by drop until reaching equivalent point, write the volume then drop another 3 ml of (0.1N) NaOH measure the pH.

**Results & calculation:**

- Plot a curve between volume of (NaOH) (x-axis) and (pH) (y-axis).
- Compared the (pH) practical with the theoretical calculation and appointed the end point in the curve.

**Discussion:**

- What is the volume of NaOH at the end point of the reaction.

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e-mail: uot_chemical@hotmail.com or chemical_eng@uotechnology.edu.iq
website: http://www.uotechnology.edu.iq/dep-chem-eng/index.htm
• What is the color of the solution in all Cases: before and after end point of reaction.
• What is the shape of the curve and why?

**Direct Titration**

**Standardization of NaOH**

Two steps used to standardization normality of NaOH

First; accurate normality of sodium carbonate to standardize the hydrochloric acid solution as the following reaction:

\[
\text{Na}_2\text{CO}_3 + \text{HCl} \rightarrow \text{NaHCO}_3 + \text{NaCl}
\]

\[
\text{Na}_2\text{CO}_3 + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O} + \text{CO}_2
\]

In this reaction one mole of hydrochloric acid equivalent with sodium carbonate and converted into sodium bicarbonate. The second mole of hydrochloric acid converts the produced sodium bicarbonate into sodium chloride, water and carbon dioxide gas which evolves to the atmosphere.

The two equations can be assumed and re-written into the following reaction:

\[
\text{Na}_2\text{CO}_3 + 2\text{HCl} \rightarrow 2\text{NaCl} + \text{H}_2\text{O} + \text{CO}_2
\]

Second; titration the standardized HCl with NaOH

\[
\text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O}
\]
Procedure:

A) Standardization of Hydrochloric acid:

- Transfer 10 ml of 0.1 N Na₂CO₃ solutions to the 250ml conical flask by pipette.
- Add approximately 20ml distilled water to the conical flask and 2-3 drops of methyl orange as indicator.
- Full the burette with hydrochloric acid solution (unknown normality).
- Titrate the solution dropwise until the pale yellow colored solution converts to orange colored solution.
- Record the volume of hydrochloric acid.

Calculation:

\[
\text{Normality}_{(\text{Acid})} \times \text{volume}_{(\text{Acid})} = \text{Normality}_{(\text{Base})} \times \text{volume}_{(\text{Base})}.
\]

(Or: \(N_1V_1=N_2V_2\)).

Calculate the normality of hydrochloric acid from the above relation.

B) Determination the normality of Sodium hydroxide

- Transfer 10ml of sodium hydroxide solution into a 250ml conical flask by pipette.
- Add 20ml distilled water and 2-3 drops (ph.ph) indicator.
- Full the burette with hydrochloric acid standardized solution.
• Titrated dropwise with continuous shaking until one drop of the acid converts the pink color base solution into a colorless solution.

• Determine the normality of sodium hydroxide solution using the formula:

\[ N_1 V_1 = N_2 V_2 \]

Discussion:

• Why standardize the HCl before determining the NaOH normality.

• Defined the indicators.

• What are the colors of solutions when using methyl orange indicator (M.O.) and phenolphthalein indicator (ph.ph.).

• What kind of solution is: HCl, NaOH, Na\(_2\)CO\(_3\), and why?

Quantitative determination of a carbonate and hydroxide in mixture

Volumetric titration very useful to determine the quantities of mixture, for that to determine the quantities of the carbonate and hydroxide in mixture, we will use volumetric titration with two different acid-base indicators in the same solution.
Sodium hydroxide is a strong base and will be the first to react with HCl with pH as the indicator (10.0-8.3), then sodium carbonate reacts with HCl according to the following equation:

$$Na_2CO_3 + 2HCl = 2NaCl + CO_2 + H_2O$$

In other words, to neutralize all the carbonate, two equivalent of HCl should be used and as such the equivalent weight of sodium carbonate = M.wt/2 = 53. When one equivalent of HCl is added to the carbonate, it is transformed into bicarbonates.

$$Na_2CO_3 + HCl = NaHCO_3 + NaCl$$

and the pH of the solution changes from alkaline about 12 to 8.3. When phenolphthalein is used, it changes to colorless at the end of this stage as its color range falls within the same zone. pH is (8.3-10).

When another equivalent of HCl is added to the solution of bicarbonate, complete neutralization takes place and it is transformed into sodium chloride and CO₂ gas is evolved.

$$NaHCO_3 + HCl = NaCl + H_2O + CO_2$$

The pH of solution changes from 8.3 to 7.0, and only one drop of HCl will change the pH to about 3.8 which is in the range of methyl orange M.O. (3.1-4.4).

**Procedure**

- Transfer 10ml from the carbonate - hydroxide mixture into a 250ml conical flask using pipette, dilute with some 20ml distilled water.
• Add 2 drops of phenolphthalein indicator and titrate against 0.1N HCl acid from a burette until the color changes from pink to colorless.

• Record the burette reading and let the volume of acid in this stage as (A) ml.

• Now add 2 drops methyl orange indicator (to the same solution) and continue the titration with hydrochloric acid until the color change from yellow to orange. Record the burette reading and let this volume be (B) ml.

• Repeat the experiment until no appreciable difference appears in the readings.

• Calculate the concentration of sodium hydroxide and sodium carbonate in the mixture.

**Calculation**

A ml = volume of HCl in ph.ph indicator = vol. of HCl to convert NaOH TO NaCl and NaCO₃ toNaHCO₃

B ml = vol. of HCl in M.O. indicator = vol. of convert NaHCO₃ to NaCl

A-B = vol. of HCl to neutralize the hydroxide.

i.e. the volume of HCl acid for the sodium bicarbonate neutralization Completely is 2B.

Calculate the normality of NaOH and NaCO₃ in g/L using the relationship:

- \( N \times V (\text{NaOH}) = N \times V (\text{HCl}) \)

\[ N \times 10 = 0.1 \times (A-B) \]
• \[ N \text{ V} (\text{Na}_2\text{CO}_3) = N \text{ V} (\text{HCl}) \]

\[ N \times 10 = 0.1 \times (2B) \]

• Conc. = Normality × Equivalent weight

**Back titration**

Sometimes reaction is slow to go to completion and a sharp end-point cannot be obtained, a back titration will give a useful results.

To calculate a substance like:

**Procedure:**

- Put 10ml of the acid sample into a 250ml conical flask by pipet
- Add 10 ml of 0.1N NaOH solution into a 250ml conical flask by pipette.
- Add 2 drops of phenolphthalein indicator.
- Titrate against 0.1N HCl acid from a burette until the solution color changes from pink to colorless. Let this volume be \( V_1 \) ml.
- Calculate the normality of the acid solution.

**Calculation:**
To calculate the volume of NaOH which reacts with acid sample using the following relation?

\[ N_{HCl} \times V_{HCl} = N_{NaOH} \times V_{NaOH} \]

\[ 0.1 \times V_1 (\text{burette}) = 0.1 \times V \]

\[ V_1 = V_{NaOH} \text{ (the volume of NaOH which react with HCl in the titration)} \]

\[ 10 - V_1 = V_2 \text{ (the volume of NaOH which react with the acid sample)} \]

**To calculate the normality of acid sample:**

\[ N_{NaOH} \times V_{NaOH} = N_{\text{acid sample}} \times V_{\text{acid sample}} \]

\[ 0.1N \times V_2 = N_{\text{acid sample}} \times 10\text{ml} \]

**Discussion**

- Why NaOH solution is added to the HNO₃ solution "purpose of back titration"?
- Why phenolphthalein is used? Write the required chemical equation?
- Why the solution of solution of unknown acid and the base is titrated with HCl solution?
- Write three examples for substance that can be determine by titration.

**Determination of Chloride in Water**
Precipitation Titrations:

Titrations of this type involve the formation of precipitate. It is used to determine the quantity of halide (Chloride, Bromide, and Iodide) from neutral solutions, where one of the mentioned ions is precipitated as silver halide. This method is called Moher's Method. The most important application is the reaction between silver nitrate and sodium chloride using potassium chromate as indicator according to the following reactions:

\[
2\text{AgNO}_3 + \text{K}_2\text{CrO}_4 \rightarrow \text{Ag}_2\text{CrO}_4 + 2\text{KNO}_3
\]

Brown precipitate

The solubility of silver chromate is \(9.5 \times 10^6\) times larger than that of silver chloride. Therefore, if both reagents are in the same solution, and titrating against sodium chloride solution, all silver will be precipitated first and then silver chromate.
**Procedure:**

1- Take 50 ml of drinking water into a conical flask.

2- Add 4 drops of potassium chromate indicator.

3- Titrate against silver nitrate solution (0.01N) until silver chromate precipitation is formed, and then record the pipet reading.

**Calculations:**

1- Calculate the concentration of sodium chloride solution

\[
\text{Concentration} = \text{Normality} \times \text{Equivalent Weight}
\]

2- Calculate Cl\(^-\) concentration (ppm):

\[
\text{ppmCl}^- = \frac{\text{eq.wt of Cl}^- \times N_2 \times V_2 \times 1000}{V_1 \text{ of sample}}
\]

\(N_2 (\text{AgNO}_3)\) = Silver Nitrate Normality

\(V_2 (\text{AgNO}_3)\) = Sliver Nitrate Volume (from pipet)

\(\text{eq.wt. (Cl}^-)\) = Equivalent Weight of Chlorine

**Discussion:**
1- What is the name of the method applied in this experiment?

2- Why it is preferred to use dark pipets when dealing with AgNO₃ as standard solution?

3- What is the indicator used in this experiment, and Why?

4- Why the brownish precipitate is appeared? Write the chemical equation.

5- What is the standard and acceptable percentage of chlorine in drinking water? Explain the effect of high and low chlorine concentrations presented in drinking water.

**Standardization of KMnO₄ and the determination of ferrous sulfate**

A- Standardization of Potassium Permanganate (KMnO₄) Solution:

Oxalic acid reacts with potassium permanganate according to the following reaction:

\[ 5\text{H}_2\text{C}_2\text{O}_4 + 3\text{H}_2\text{SO}_4 + 2\text{KMnO}_4 \rightarrow 2\text{MnSO}_4 + 10\text{CO}_2 + \text{K}_2\text{SO}_4 + 8\text{H}_2\text{O} \]

Potassium Permanganate (KMnO₄) is considered as one of the strongest oxidizing agent when it is presented in acid media. In this media the manganese ion has initial oxidation number of (+7) and the final oxidation number is (+2) as shown in the following equation:

\[ \text{Mn}^{+7}\text{O}^- + 8\text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{+2} + 4\text{H}_2\text{O} \]
Oxalic acid is considered as reducing agent where the carbon has initial oxidation number of (+3) and the final oxidation number is (+4), as shown in the following equation:

\[
\begin{align*}
[\text{C}_2\text{O}_4]^{2-} & \quad 2 \text{CO}_2 + 2e^- \\
\text{The equivalent weight of Oxalic acid: } & \\
\text{H}_2\text{C}_2\text{O}_4 \text{ eq.wt.} = \frac{Mwt}{e^- \text{ loss}} &= \frac{80}{2}
\end{align*}
\]

B- **Standardization of Ferrous Sulfate Solution:**

Ferrous ions react with permanganate in acid media and accordingly will be converted to Ferric ions, while the Manganese of (+7) initial oxidation state will have final oxidation state of (+2), as shown below:

\[
\text{KMnO}_4 + 5\text{FeSO}_4 + 4\text{H}_2\text{SO}_4 \rightarrow \frac{1}{2}\text{K}_2\text{SO}_4 + \frac{5}{2}\text{Fe}_2\text{(SO}_4)_3 + 4\text{H}_2\text{O}
\]

**Procedure:**

**A- Determination of KMnO\textsubscript{4} Normality:**

1- Take out (10ml) of (0.1N) Oxalic Acid (H\textsubscript{2}C\textsubscript{2}O\textsubscript{4}) using a pipet to a conical flask.
2- Add (10ml) of (2N) Sulfuric Acid (H\textsubscript{2}SO\textsubscript{4}) to the same flask.

3- Heat the solution in water bath of (60°C) with gentle stirring.

4- Titrate against KMnO\textsubscript{4} solution until the color changes from colorless to light violet with keeping on both heating and stirring.

5- The reaction is ended when having stable violet color inside the flask. Then record the consumed volume of KMnO\textsubscript{4} from the burette.

**B- Determination of FeSO\textsubscript{4} Solution Normality:**

1- Transfer (10ml) of Ferrous sulfate solution into a conical flask.
2- Add (10ml) of (2N) Sulfuric Acid to the same flask.
3- Titrate against standard KMnO\textsubscript{4} solution from a burette until the solution color changes from colorless to light violet.
4- Take the reading of volume consumed from the burette.

**Calculations:**

A. \[ N \times V \text{ (KMnO}_4\text{)} = N \times V \text{ (H}_2\text{C}_2\text{O}_4\text{)} \]

\[ N_{(\text{KMnO}_4)} \times V \text{ from burette} = 0.1 \times 10 \]

B. \[ N \times V \text{ (KMnO}_4\text{)} = N \times V \text{ FeSO}_4 \]

\[ N_{(\text{KMnO}_4/A)} \times \text{ burette} = N \times 10 \]

\[ C_{\text{FeSO}_4} = N_{\text{FeSO}_4} \times \text{eq.wt.} \]

**Discussion:**
1- Why Oxalic Acid was heated to (60°) before and during titration? Why this specific temperature is applied?

2- Why H₂SO₄ is used in this type of titration?

3- Why there was no need to use any indicator in both titrations?

4- Why in the second titration FeSO₄ solution wasn't heated?

5- In calculating the equivalent weight of KMnO₄, why the molecular weight was divided by (5)?

6- Why KMnO₄ solution can be considered as secondary standard solution?

7- Why KMnO₄ solution is preserved in dark bottles?

8- From the reaction of FeSO₄ with KMnO₄, write down the reducing agent equation (ionic form) with showing the oxidation state.

**Determination of Hardness of Water**

Hardness of water, attributed to the presence of Ca²⁺ and Mg²⁺ if as HCO₃ and CO₃ salts in water, it can be removed by boiling the water to convert these anion into CO₂ and H₂O if as SO₄ or Cl it will be hard to be removed.

**Procedure:**

1) Place 100ml of tap water in a 250ml beaker.

2) Heat slowly over a Bunsen burner and then boil gently for 30 minutes
3) Cool the hot water and filter through filtered funnel directly into 250ml conical flask.

4) Add 1ml buffer solution pH=10 to 10ml Sample and then add 0.1g of Eriochrome Black-T Indicator.

5) Titrate against 0.01M EDTA solution from a burette until color changes from red to blue.

4) Add 1ml of (1N) NaOH to 10ml Sample and then add 0.1g of meroxide Indicator and measure the total hardness.

5) Titrate against 0.01M EDTA solution from a burette until color changes from blue to violet and measure the Ca and Mg hardness.

Calculate the permanent hardness of water in terms of ppm CaCO3

Determine temporary hardness in terms of ppm CaCO3

Discussion:

- Why Eriochrome black-T is used as indicator in the reactor?
- Why buffer solution of pH=10.
- Determine the amount of Mg $^{2+}$ alone? How?

Acidity of Vinegar
Vinegar contains about 5% by volume acetic acid CH₃COOH most of the remaining 95% is water plus small quantities of ethyl alcohol and other carboxylic acid. The acid contents or vinegar may determine quantitatively by volumetric titration with standard solution of sodium hydroxide solution using phenolphthalein indicator.

Part (A): Calibrate the solution of NaOH by using standard solution of 0.1N HCl.

\[ \text{NaOH} + \text{HCl} \rightarrow \text{NaCl} + \text{H}_2\text{O} \]

Procedure:

- Transfer 10.0ml of 0.1 N HCl into a clean 250ml. conical flask and 2 drops of phenolphthalein (ph.ph.) indicator.

- Titrate against sodium hydroxide solution …stop titration when the color of solution changes from colorless to pink color.

\[ N_1 \times V_1 = N_2 \times V_2 \]

Calculate the concentration of NaOH by:

\[ N_1 = \text{concentration of NaOH} \quad V_1 = \text{volume of NaOH} \]

\[ N_2 = \text{concentration of HCl} \quad V_2 = \text{volume of HCl} \]
Part (B): Titration of weak acid CH₃COOH with strong calibrated base.

\[ \text{CH}_3\text{COOH} + \text{NaOH} \rightarrow \text{CH}_3\text{COONa} + \text{H}_2\text{O} \]

- Put 5ml sample of vinegar into 25ml weighted clean beaker. Then determine the weight of net vinegar. Transfer the sample quantitatively to 100ml clean volumetric flask then dilute to the mark.

- Take 10 ml of your prepared solution in step 1, and put it into 250ml conical flask with 2 drops of ph.ph. indicator. Titrate solution with calibrated standard NaOH. Continue the titration until change of color appears from colorless to pink color.

- Determine the concentration of CH₃COOH in vinegar.

- Calculate the weight of CH₃COOH.

- Calculate the percentage of CH₃COOH wt/wt% and wt/vol.%.

\[ N_1 \times V_1 = N_2 \times V_2 \]

**Calculation:**

\[ N_1 = \text{Conc. of NaOH} \]

\[ V_1 = \text{volume of NaOH} \]
\( N_2 = \text{Conc. of Acetic acid after diluted in 100ml of dist. Water} \)

\( V_2 = \text{volume of vinegar (10ml)} \).

\( N_{Ac.} = \)

Equivalent weight of acetic acid = 60.05

**Discussion:**

- What is the pH value at the equivalent point?
- What ions are present in the solution at the equivalent point (only those in significant amounts)?
- Calculate the Molarity of NaOH, why you have to calibrate the base solution before you can use it?

**Paper Chromatography**

Paper Chromatography is a method used to separate mixtures into their different parts. Paper chromatography has been most commonly used to separate pigments, dyes and inks. To do paper chromatography you need paper with a lot of cellulose fiber (fiber found in wood) and chromatography solution, which is usually made from a mixture of water and alcohol.
Procedure

- Cut out filter paper a rectangular piece that is 8 x 15 cm in size.
- Position the filter paper so that it is longer going across.
- Draw a line using a pencil across the filter paper that is 1 cm from the top of the sheet and 1 cm from the bottom of the sheet. On the bottom line mark small lines every 2 cm and number each small line. Do not use pen for this step! You have now prepared your chromatography paper.
- In the beaker, mix 50ml of alcohol with 25 of water.
- Pour carefully into the shallow plate or bowl. The height of the liquid should be no more that 1 cm. This will be your chromatography solution.
- On each small line of your chromatography paper, make tiny dots with colored markers. Mark one pen or marker on each line. Make a key of which pen or marker goes with each number on a separate piece of paper.
- Staple the ends of your paper so that they do not touch each other.
- Carefully place the paper onto the plate or bowl so that the marker/pen dots are just above the chromatography solution.
- Allow the paper to sit in the solution until the liquid reaches the top line. Do not move or shake the set-up during this time. This may take about 1/2 an hour. Remove the paper and allow it to dry on a paper towel.
- Observe the results. What do you see? What happened to the different markers?
Qualitative analysis silver group

Reaction and separation of Group I Metals:

The common metallic ions may be divided into a number of groups which may be identified and separated according to their selective precipitation by a particular anionic functional group.

There are, for the purpose of qualitative analysis, five metal groups:

Group I (Silver Group): Ag⁺ , Pb²⁺ , Hg₂⁺

Group II (Copper Group): Hg⁺⁺ , Pb⁺⁺ , Bi⁺⁺ , Cu⁺⁺ , Cd⁺⁺ , Sn⁺⁺ , As⁺⁺⁺ , Sb⁺⁺⁺ , Sn++++

Group III A (Iron Group): Al⁺⁺⁺ , Cr⁺⁺⁺ , Fe⁺⁺⁺

Group III B (Zine Group): Ni⁺⁺ , Co⁺⁺ , Mn⁺⁺ , Zn⁺⁺

Group IV (Calcium Group): Ba⁺⁺ , Sr⁺⁺ , Ca⁺⁺

Group IV (Alkali Group): Mg⁺⁺ , Na⁺⁺ , K⁺⁺ , NH₄⁺

Each of the five groups above (except Group V) shows a distinguished affinity toward a particular reagent, which forms upon chemical reaction a ppt that can be easily separated.

In this experiment only group I of metals shall be studied.

A) Identification of Lead ion Pb²⁺⁺⁺

1) Introduce 2ml lead nitrate solution Pb(NO₃)₂ into a test tube.

2) Add few drops of hydrochloric acid HCL until all Pb²⁺ precipitates as a white PbCl₂ ppt.

3) Dissolve some of the ppt. in hot water.

4) Add few drops of potassium Chromate Solution.

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Website: http://www.uotechnology.edu.iq/dep-chem-eng/index.htm
A yellow ppt. of lead Chromate indicates the positive presence of lead ions Pb^{++}:

**B) identification of silver ion Ag^{+}**

1) Introduce 2ml of silver Nitrate Solution AgNO_{3} into a test tube.
2) Add few drops of hydrochloric acid HCL until all Silver Ag^{+} precipitates as a white ppt. of AgCL:
3) Take some of the ppt. and add to it few drops of Ammonium Hydroxide Solution NH_{4}OH (in a test tube).
4) Add few drops of Nitric Acid HNO_{3}. A white ppt. of AgCL will form again:

**C) identification of Mercurous Ion Hg_{2}^{++} :**

1) Introduce 2ml of Mercurous silver Nitrate Solution Hg_{2}(NO_{3})_{2} into a test tube.
2) Add few drops of hydrochloric acid solution HCL until all Mercurous ions precipitates as a white ppt. of Hg_{2}CL_{2}:
3) Add few drops of concentrated Ammonium Hydroxide NH_{4}OH solution HCL to the ppt. A blackish Solution of free mercury will form:

**D) Identification and Separation of Group I mixture:**

Add 2ml of dilute hydrochloric acid into a mixture of Group I ions Pb^{++}, Ag^{+}, Hg^{2+} in test tube.
Filter the ppt. and discard the ppt. Wash with few milliliters of dilute HCL. Transfer the ppt. into a test tube and heat with 5ml distilled water. Filter hot.
Residue: May contain Hg₂Cl₂ and AgCl. Wash with distilled water. Add 4ml of warm dilute NH₃ solution over p.pt.

Residue: Black Hg’Hg(NH₂)Cl. Mercury present

Filtrate: May contain PbCl₂. Add potassium chromate solution to the hot filtrate A yellow p.pt. of lead chromate PbCrO₄ mediates lead presence.

Filtrate: May contain (Ag(NH₃)₂)Cl. Acidify with dilute HNO₃. White p.pt. of AgCl Silver present

Mercury present

Discussion:

1- Why HCL is used to separate the ions of first group?

2- Why add hot water to precipitate first group?

3- What is the difference between quantitative analysis and qualitative analysis?
References:


