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EXPERIMENT(1)

Preparation Sodium Carbonate (Na₂CO₃) and standardization it with Hydrochloric acid (HCI)

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Volumetric Analysis

Consists of essentially in determining the volume of a solution of accurately know concentration which is required to react quantitatively with the solution of the substance being determined, the end point need appearance of a signal that means the end of reaction it is possible.

It is possible to determine that through

By using indicator (it should be available which may by change a colure).
By using instrumental method for detecting the completion of the reaction i.e. conducts ometric or potentiometric titration.

Classification of reaction in volumetric analysis:

1) **Neutralization titration:** These include the titration of free basses or those formed salts of weak acid by hydrolysis, with a standard acid (an acidimetry), and the titration of free acid or those formed by the hydrolysis of salts of weak base with a standard base(a alkalimeter).

 $NaOH+ HCl \longrightarrow NaCl+ H_2O$

H+ + OH-----→H2O

2) **Precipitation titration :** These depend upon the combination of ions other than hydrogen or hydroxide ions, to form a simple precipitate as in the titration of silver ion with a solution of a chloride .

 $AgNO_3 + NaCl \longrightarrow AgCl + NaNO_3$

3) **Complex formation titration:** These depend upon the combination of ions other than hydrogen or hydroxide ions, to form a soluble slightly dissociated ion or compound .

 $Cu_{+2} + 4NH^{+3} \longrightarrow [Cu(NH3)_4]_{+2}$ $NaCN + AgNO_3 \longrightarrow [Ag(CN)_2].$

4) **Oxidation-reduction reaction :** Under this heading are included all reaction involving change in oxidation number or transfer of electrons among the reacting substance. The Standard solution are either oxidizing or reducing agents. **MnO4** .+ 5Fe₊₂ + 8H₊ \longrightarrow 5Fe₊₃ + Mn₊₂ + 4H₂O

Methods in volumetric analysis

- 1. Titration method.
- 2. Gas analysis method.
- 3. Intramural method of analysis

Titration method:

One of the quantitative volumetric analysis, it used to determine the analyte by find the volume of standard solution required to completely react with it.

Condition for the titration analysis:

- 1. The reaction between the titrant and titrate.
- 2. There is an appearance to the end of reaction.
- 3. There must be a standard solution in the burettes.

Primary standard : a highly purified substance that required as a reference material in titration , and the accuracy of volumetric analysis is dependent on it .

Properties of standard material

- a) Highest purity.
- b) Stability in air .
- c) Absence of hydrate water.
- d) High equivalent weight.
- e) Ready availability at modest cost.

Equivalence points and end point:

The equivalence point in titration is a reached when the amount of added titrant is chemically equivalent to the amount of analyte in the sample. The equivalent point is a theoretical point that cannot be determined experimentally ,instead we can only estimate it by observing some physical change associated with condition of equivalence this changes is called the end point for the titration.

Titration error:

The difference in volume between the equivalence point and end point.

Indicators:

Indicators define are weak organic compound ,some of them are acidic indicator (HIn) and basic indicator(OHIn).

Acid Base indictors:- is a chemical substance that changes color over a specified range of PH . They have two colures, each appears at a specific PH range all indicators are used in the neutralization reaction to determine the end point of the reaction between acid and base, most of the indicators are soluble in water, but some of them slightly soluble in water and in this case we have to use ethanol/water mixture to dissolve them. To determine an appropriate acid-base indicator for an acid-base titration ,you must know the equivalence point and use the indicator table.

Indicator	Acid	Base	pН
Methyl orange (M.O)	Red	Yellow	4-6
Phenolphthalein Ph- ph	Colorless	Red(pink)	8-10
Methyl red M.R	Yellow	Red	6-8
Bromphenol blue	yellow	blue	6-7

Preparation Sodium Carbonate (Na₂CO₃) and standardization it with Hydrochloric acid (HCI)

Background:

Sodium Carbonate, (also known as washing soda, soda ash and soda crystals) is the inorganic compound with the formula Na₂CO₃ and its various hydrates. Sodium carbonate is also used for other reactions that require a base to neutralize acids that form during reactions. For example, alkylation reactions often employ sodium carbonate to mop up protons (acid) that forms during the reaction. Hydrochloric acid (HCl, also known as muriatic acid) is a colorless corrosive, strong mineral acid with many industrial uses among which, when it reacts with an organic base it forms a hydrochloride salt, contain 35% of hydrogen chloride (equivalent weight 36.4) and Specific Gravity (1.18 g/cm₃), a clear, colorless, fuming poisonous, highly acidic aqueous solution of hydrogen chloride it's found in the stomach in dilute form. Sodium hydroxide is reacted with HCI according to the following formula:

 $Na_2CO_3 + 2HCl \longrightarrow NaCl + CO_2 + H_2O$

The reaction including two steps:

first step : Is the reaction of one mole of hydrochloric acid (HCl) to convert sodium carbonate (Na₂CO₃) to bicarbonate sodium (NaHCO₃)

Na₂CO₃+HCl → NaHCO₃+NaCl

Second step: a second mole from the acid used to converting sodium bicarbonate (NaHCO₃) to carbon dioxide (CO₂) and water (H₂O) as in the following equations.

NaHCO₃+ HCl NaCl+ CO₂+ H₂O

Chemical and equipment

- 1 Hydrochloric acid (HCI)
- 2 Sodium Carbonate (Na₂CO₃)
- 3 methyl orange (indicator)
- 4 Volumetric flask
- 5 Graduated cylinder
- 6 Burette
- 7 Distilled water
- 8 Watch glass

Procedure:-

A// Preparation ((0.1 N) solution from Sodium Carbonate

1) Weight accurately about (X) gm. of pure sodium carbonate in a watch glass.

2) The weight taken from sodium carbonate dissolves in a small amount of distilled water by the constant shaking.

3)Transfer the dilute solution to a 250 ml conical flask and supplement the solution with distilled water to the tip of the oval.

B. Standardization HCI solution against Na₂CO₃

1. Wash with burette water and then with distilled water two or three times and then wash with hydrochloric acid solution (HCl).

2. Fill the burette with (HCl) until the acid reaches the top and open the bottom control valve in the bowl to lower the acid level in the bowl until

zero.

3. Wash a conical flask with its capacity (250 ml) with normal water and then with distilled water.

4. Wash a 10 mL pipette with distilled water and then with sodium carbonate solution (0.1N) (Na₂CO₃).

5. Remove 10 mL of sodium carbonate Na₂CO₃ solution with the solvent and then place it fully in the conical flask. If there is a bit of solution at the end of the pipette, be sure to remove it in the beaker and gently snap it into the conical flask.

6. Add two or three drops of methyl orange to the solution with conical flask (Na₂CO₃) solution to obtain pinkish red.

7. Start calibration by gradually adding hydrochloric acid (HCl) to the sodium carbonate solution (Na_2CO_3) in the conical flask with the conical flask continuously during calibration.

Calculation:-

1. Found Weight of Na₂CO₃

$$N = \frac{Wt}{Eq.wt} \times \frac{1000}{V_{ml}}$$

2. Found Normality of HCI after standardization with Na2CO3

$$(N \times V)_{Na_2CO_3} = (N \times V)_{HCl}$$