Medicinal Chemistry
The first stage
College of Dentistry

By

Assistant Lecturer

HAZIM SAAD JABBAR AL-MALIKI

University of Basra - College of Dentistry - Department of Basic Sciences

MSc. Medicinal Biochemistry from Malaysian National University (UKM) – Malaysia

Email:- hazim.saad@uobasrah.edu.iq and hazzmrn@yahoo.com

QUANTITATIVE VOLUMETRIC ANALYSIS

CONTENTS:-

INTRODUCTION

Volumetric analysis

Volumetric analysis is a general term for a method in quantitative chemical analysis in which the amount of a substance is determined by the measurement of the volume that the substance occupies. It is commonly used to determine the unknown concentration of a known reactant. Volumetric analysis is often referred to as titration.

What is the meaning of Titration?

Titration is a common laboratory method of quantitative chemical analysis that is used to determine the unknown concentration of a known reactant. Because volume measurements play a key role in titration, it is also known as volumetric analysis.

A reagent, called the titrant or titrator,[1] of a known concentration (a standard solution) and volume is used to react with a solution of the analyte or titrant,[2] whose concentration is not known. Using a calibrated burette or chemistry pipetting syringe to add the titrant.

A primary standard solution is a highly purified compound that serve as a reference material in all volumetric titrimetric methods.

Types of titrations

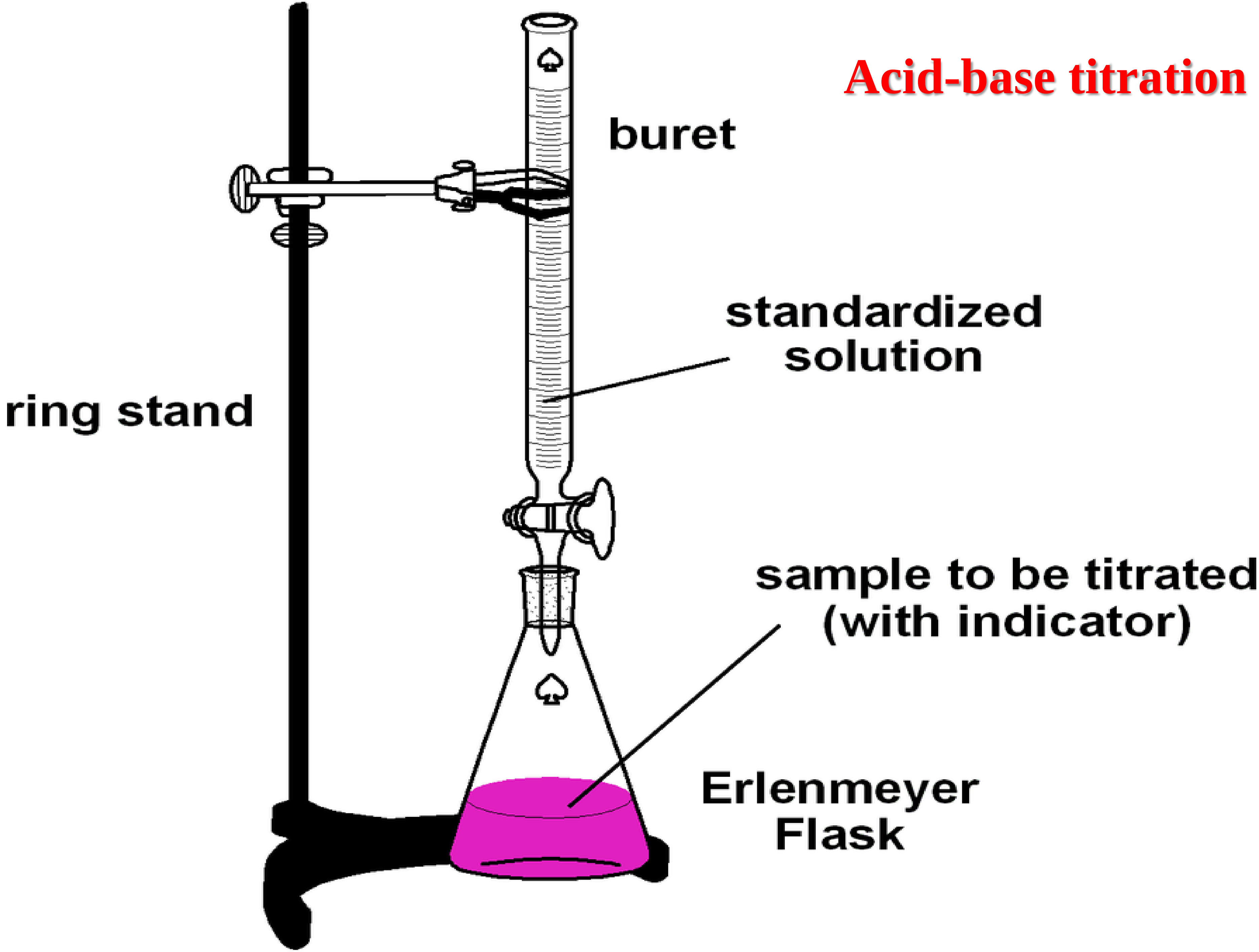
1- Acid-base titration

2- Precipitation titration

3-Redox titration

4- Complexometric titration

Acid-base titration



Calculations of volumetric analysis

Standard solution is one, which contains a known weight of the reagent in a definite volume of the solution.

Molar solution is one, which contains 1 gm molecular weight of the reagent per liter of solution.

$$M = \frac{Weight}{M.Wt} * \frac{1000}{Volume(mL)}$$

Normal solution is one that contains 1gm equivalent weight per liter of solution.

$$N = \frac{Weight}{eq.wt} * \frac{1000}{Volume(mL)}$$

Equivalent weights

(1) Equivalent weight in neutralization reactions.

The equivalent weight of acid is that weight of it which contains one-gram atom of replaceable hydrogen.

Ex: equivalent weight of H_2SO_4 =M.Wt H_2SO_4/2

equivalent weight of H_3PO_4 =M.Wt H_3PO_4/3

$$\text{eq.wt acid} = \frac{\text{M.Wt acid}}{\text{No. of active } H^+}$$

The equivalent weight of Base is that weight of it which contains one replaceable hydroxyl group.

Ex: equivalent weight of NaOH =M.Wt NaOH/1

Experiment (5)
Calibration of Hydrochloric Acid

A. Preparation hydrochloric acid solution (0.1N):-

1-Calculate the normality of the concentrated HCl :

$$N1 = \frac{SP. * Wt.\% * 1000}{Eq.\ Wt.} \quad \text{-------------} (1)$$

Where :-

N1 = hydrochloric acid calibration Centre

Sp. = Specific weight of acid (acid density)

Wt. % = The percentage of the weight of hydrochloric acid Center

$$N = \frac{1.19 * (37/100) * 1000}{36.5} = 12.0630$$

2- To prepare (500mL) of 0.1N HCl

Preparation titrated hydrochloric acid ($N_2 = 0.1$) and size ($V_2 = 500$ ML) Use the following formula to calculate the size of the acid Centre (V_1), which we take to prepare the acid diluted:

$$N_1 * V_1 = N_2 * V_2 \quad \text{-------------(2)}$$

conc. HCl dil. HCl

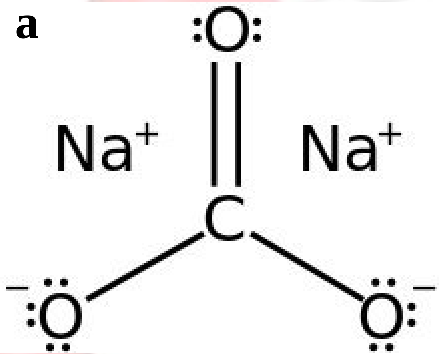
$$V_1 = \frac{N_2 * V_2}{N_1} \quad \text{----------(3)}$$

$$12.0630 \times V_1 = 500 \times 0.1$$

$$V_1 = 4.1449 \text{ mL}$$

B: Preparation of (0.1N) sodium carbonate (Na_2CO_3):

To calculate the weight of sodium carbonate needed to prepare a titration solution (0.1), the following equation is used:



$$N = \frac{\text{Weight}}{\text{equivalent weight}} * \frac{1000}{\text{Volume(mL)}}$$

$$Wt = \frac{N * V * Eq.Wt}{1000}$$

Where:

WT = weight of sodium carbonate dissolved in a volume of water

N = Standard of Sodium Carbonate to be prepared = 0.1

V = volume of the solution to be prepared (in ml)

Eq.Wt = equivalent weight to sodium carbonate

Equipment:-

1) burette of 50 ml. 2) 10 ml pipette. 3) 500ML Beaker. 4) 500 ML volumetric vial, 250 ML volumetric flask and conical flask. 5) funnel. 6) Spatule. 7) cylinder.

Procedure: -

A. Standardization of HCl solution with standard solution of Na_2CO_3

1-Clean the burette and rinse with HCl solution.

2-Fill the burette with HCl.

3-Pipet 10 ml of standard solution (Na_2CO_3) into a 250mL conical flask. Add 3 drops of **Methyl orange indicator**.

4-Titrate by adding HCl drop wise until the solution just beings to change from yellow to red.

5-Reapeat the titration a few times until you get approximate results. Take the average of the results and subtract 0.05mL. (This result represents the volume of extra drop which change the color of indicator.

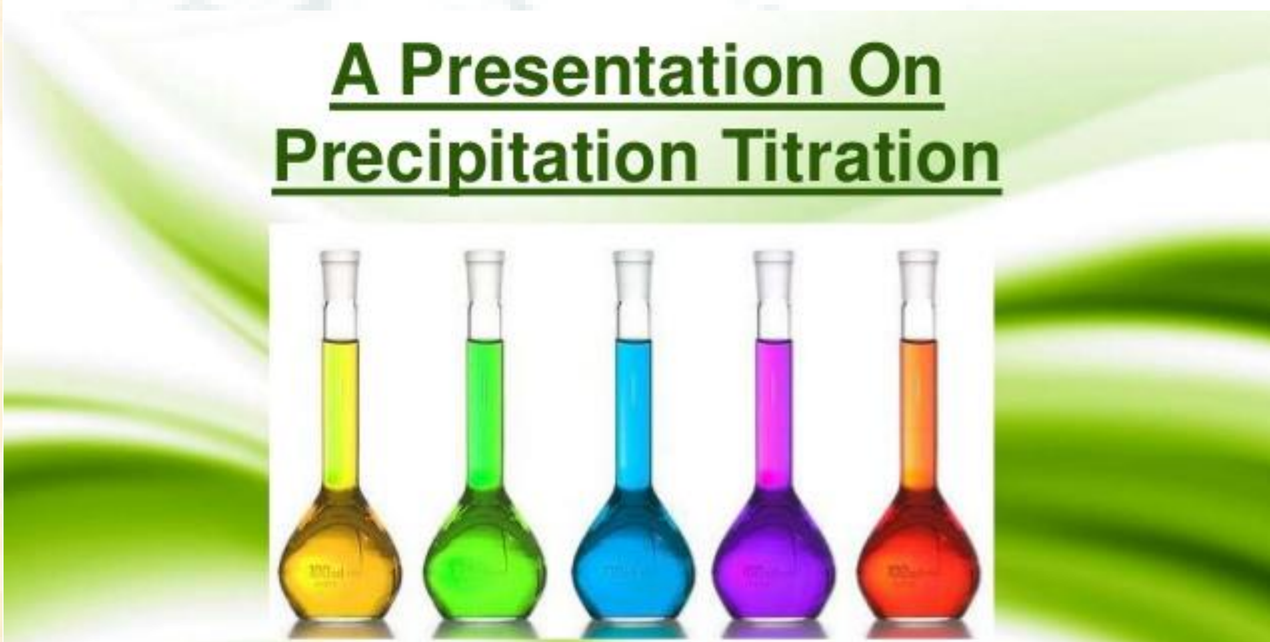
6-Calculate the normality of HCl :

$$N_{acid} \times V_{acid} = N_{base} \times V_{base}$$

7-Make label on your bottle containing your name, date of preparation and concentration of acid after standardization.

8- The equation of reaction

$$Na_2CO_3 + HCl \xrightarrow{M.O} H_2CO_3 + 2NaCl$$



B. Analysis of sodium carbonate Na_2CO_3

1-Clean the burette and rinse with standardized HCl solution and then fill it with the acid.

2-Pipet 10 ml unknown solution (Na_2CO_3) into a 250mL conical flask. Add 2 drops of **phenolphthalein indicator** the solution will be pink.

3-Titrate by adding HCl drop wise until the solution just beings to change its color from pink to **colorless** this data will be (V_1).

4- Add 1-2 drops of **Methyl orange indicator** to the above solution which became yellow then complete the titration until the color of the solution became pale **orange (onion)**, this data will be (V_2).

5-Reapeat the titration a twice time until you gets approximate results. Take the average of the results and subtract 0.05mL. (This result represents the volume of extra drop which change the color of indicator.

6- Make a table as bellow: -

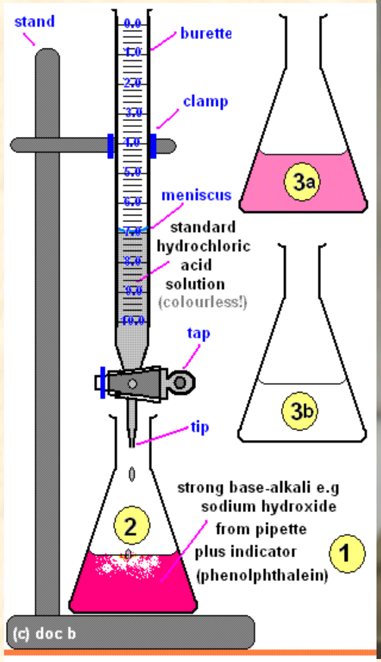
Sq		First titration	Second titration	Third titration	Average of titrations
1	Titration with ph.ph	V1	V1	V1	V1 (av.)
2	Titration with M.O	V2	V2	V2	V2 (av.)

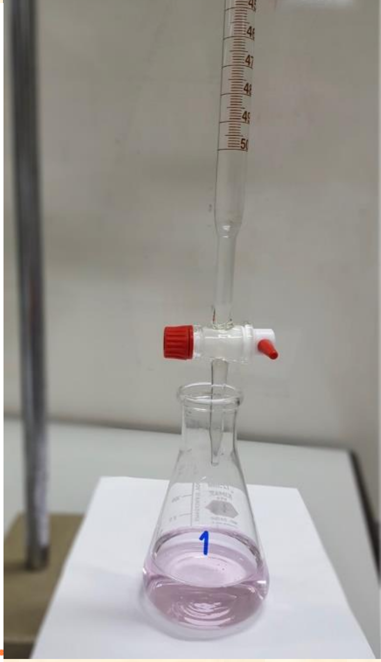
$$V1(av.) = \frac{V1+V1+V1}{3}$$

$V1(av.) = 1/2\ CO_3^{=}$

$V2(av.) = 1/2\ CO_3^{=}$

$V1+V2 = V_{tot.}$ of Na_2CO_3



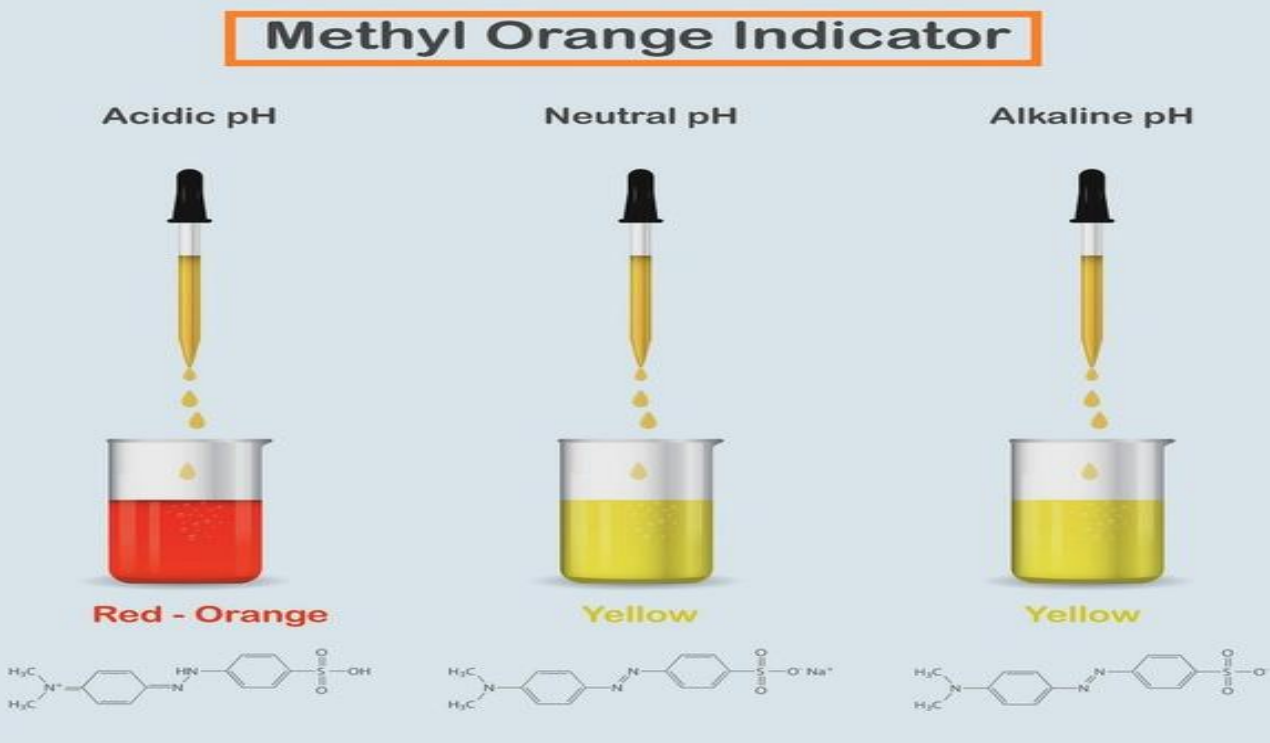


$$N_{acid} \times V_{(tot.)\ acid\ (from\ burette)} = N_{base} \times V_{base}$$

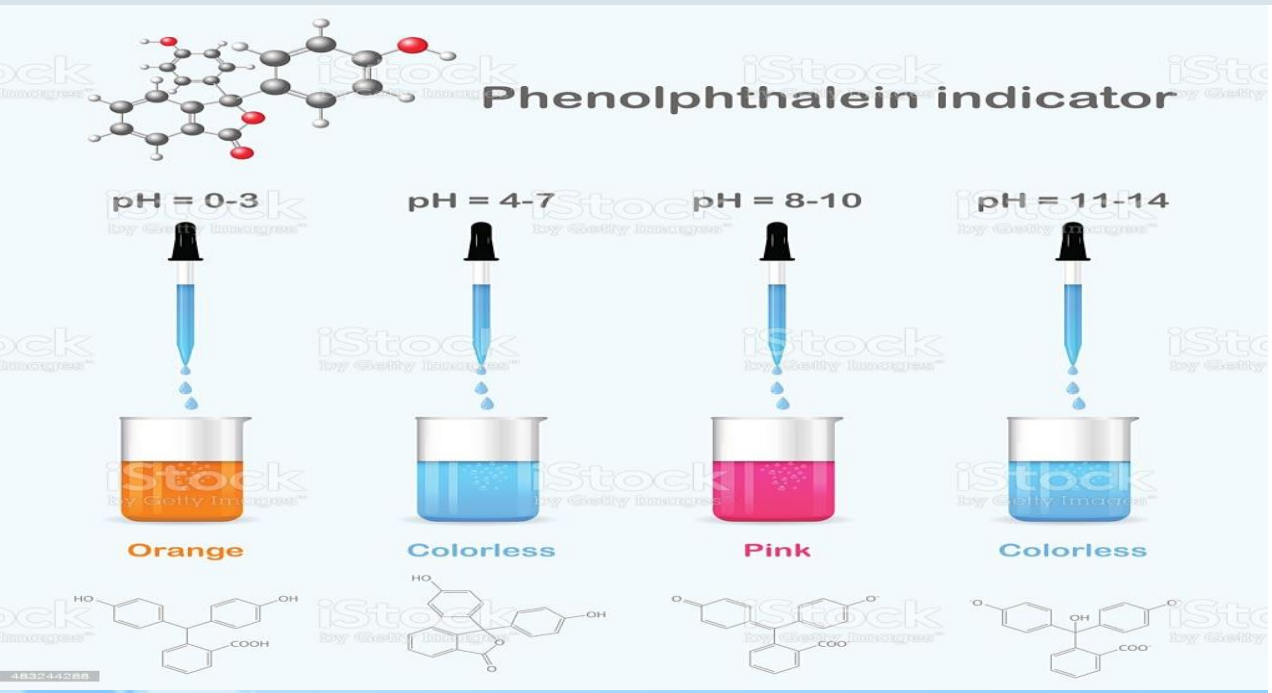
7- The equation of reaction :

$$\underset{\text{Pink}}{Na_2CO_3 + HCl} \xrightarrow{ph.ph} \underset{\text{colorless}}{NaHCO_3 + NaCl}$$

$$\underset{\text{Yellow}}{NaHCO_3 + HCl} \xrightarrow{M.O} \underset{\text{pale orange}}{H_2CO_3 + NaCl}$$


Methyl Orange Indicator
Acidic pH
Neutral pH
Alkaline pH
Red - Orange
Yellow
Yellow


Methyl Orange 0.05%
UN No. 3143
CAS No. 547-58-0
327.33 g/mol
DANGER
IF SWALLOWED: Immediately call a POISON CENTER/doctor
Consolidated Chemical & Solvents LLC


Phenolphthalein indicator
pH = 0-3
pH = 4-7
pH = 8-10
pH = 11-14
Orange
Colorless
Pink
Colorless


YOUR COMPANY
LOGO
Phenolphthalein
Indicator Solution,
0.5% w/v in Isopropanol
pH 8.3 (colorless) -10.0 (pink)
Code: PH1605-B
Size: 60mL
Lot #: 5GF852
Expires: JUN/17

Thank You For Listening