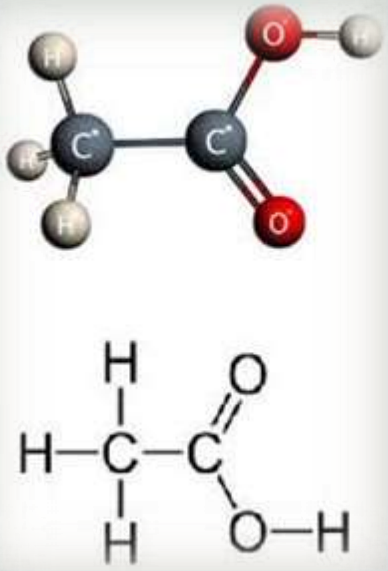
Determination of the %w/v Acetic Acid in a Vinegar Sample

Vinegar is a liquid consisting mainly of **Acetic Acid** (CH_3COOH) and water.



Glacial Acetic Acid

It is a water-free (anhydrous) acetic acid, the name comes from the ice - like crystals that form slightly below room temperature at 16.6 °C .

An abbreviation for acetic acid is **HAc** where **Ac** stands for acetate, CH_3COO^-

Acetate is the ion resulting from loss of H^+ from acetic acid.

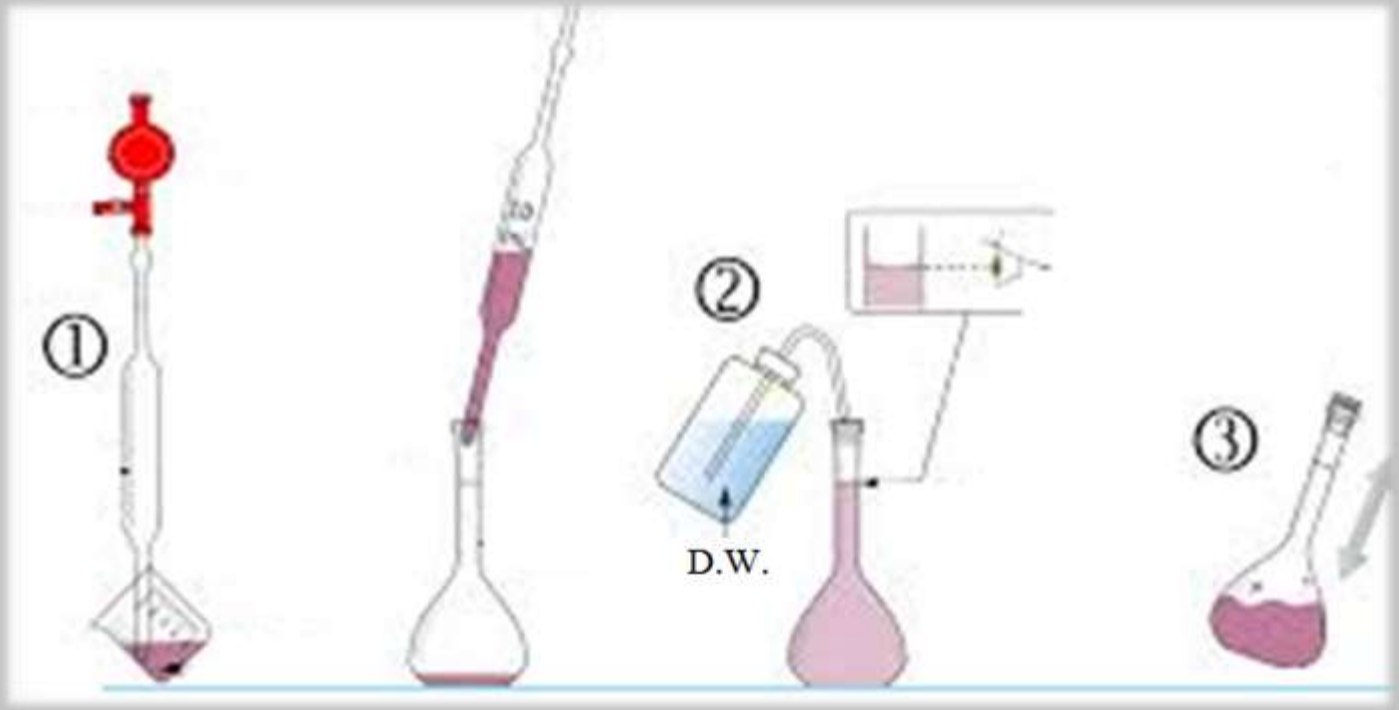
Ice - like crystals of Glacial Acetic Acid

Preparation of unknown acetic acid solution:

1- Transfer 10 ml of unknown by using 10 ml bulb pipette into a 100 ml volumetric flask.

2- Complete the volume with distilled water.

3- Stopper the flask and shake well.



Name of Experiment: Acid - Base Titration.

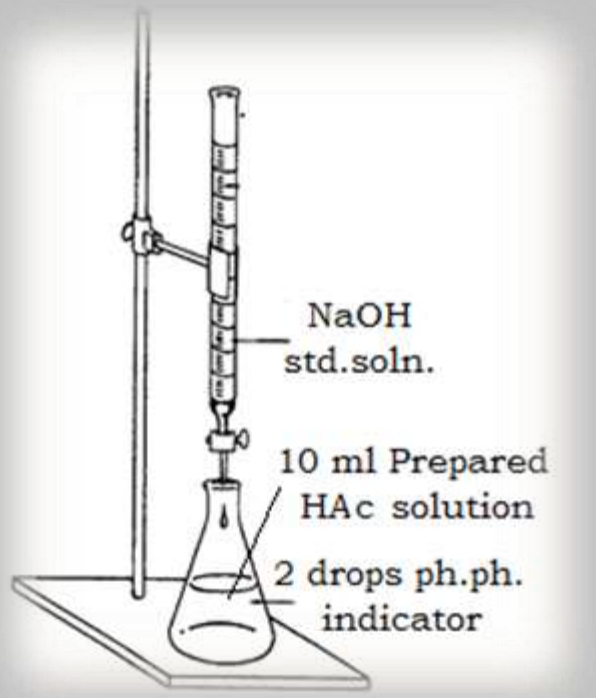
Aim of Experiment: Determination of %w/v Acetic Acid in an unknown Vinegar sample .

Principle:

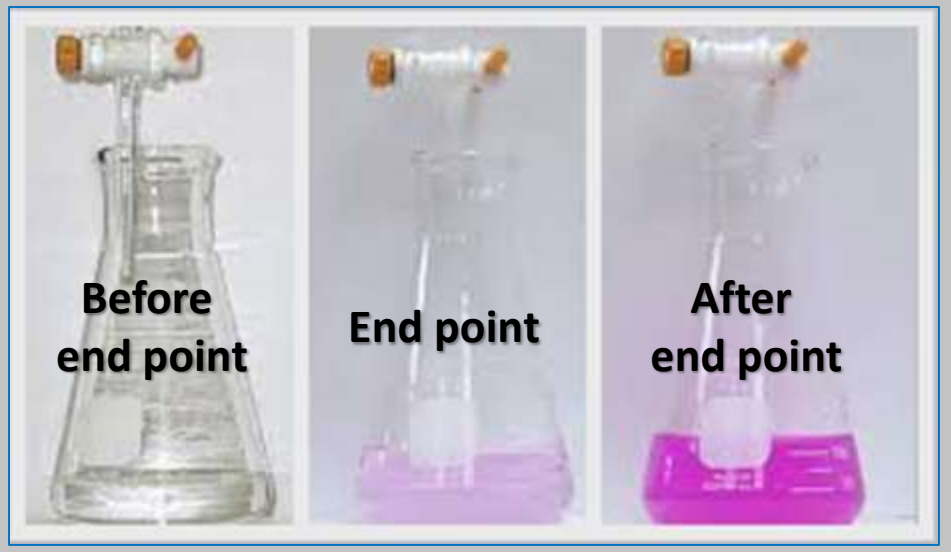
Acetic acid is a weak acid Ka = 1.8×10^{-5} , So titration of acetic acid against NaOH is a titration of a weak acid against a strong base. The end point will be not very sharp.

Procedure:

1- Using a 10 ml bulb pipette transfer 10 ml of the prepared solution to a conical flask.
2- Add 2 drops of ph.ph. indicator.
3- Fill the burette with 0.1 N NaOH standard solution.



4- Titrate the prepared acetic acid solution with standard NaOH soln. until the color of the indicator becomes faint pink.



Calculations:

$$N_1 \quad V_1 \quad = \quad N_2 \quad V_2$$
$$\text{NaOH} \qquad\qquad \text{HAc}$$

$$N_1 \quad V_1 \quad = \quad \frac{\text{Mass}_{\text{HAc}}}{\text{Eq.mass}_{\text{HAc}}} \quad * \quad 1000$$
$$\text{NaOH}$$

Example

Calculate the % w/v HAc for 10 ml sample of a vinegar that required 30ml of 0.2N NaOH soln.? Knowing that the atomic masses for Na= 23 , O = 16 , H = 1 and for C = 12.

$$N_1 \; V_{1\,NaOH} = N_2 \; V_{2\,HAc}$$

$$N_1 \; V_{1\,NaOH} = \frac{Mass_{HAc}}{Eq.mass_{HAc}} * 1000$$

$$0.2 * 30 = \frac{Mass_{HAc}}{60} * 1000$$

$$Mass_{HAc} = 0.36 \text{ g HAc in } 10 \text{ ml sample.}$$

0.36 g	10 ml
X	100 ml

$$X = \frac{0.36 g * 100 \cancel{ml}}{10 \cancel{ml}}$$

$$X = 3.6 \text{ g} = 3.6 \% \; w/v$$

Notes

In general,

For weak acid titrations,

the pH at the end point is above 7, So ph.ph. is the most suitable indicator which can be used.

For weak bases ,

the pH at the end point is below 7, So M.R. (4.2 – 6.2) or M.O. (3.1 – 4.4) are widely used.

For strong acids & strong bases,

M.R. , Bromothymole blue , ph.ph. are most suitable.

Phenolphthalein indicator is the most frequently used indicator in the titration of acetic acid with NaOH standard solution ?

The pH of the solution at the equivalence point will be above pH 7, (pH about 8.72), So ph.ph. is used (since the pH at the end point of this titration is within the pH range of ph. ph. Indicator which is 8.3 – 10).

Methyl red or methyl orange can not be used as indicators in this experiment?

The pH of the soln. at the equivalence point will be above pH 7, (pH about 8.72)

M.R (4.2–6.3) or M.O. (3.1–4.4) can not be used since they change color at much lower pH (i.e.) before the end point of titration is reached.


ANY
questions?

General Laboratory Apparatus

Basic Equipments and Instruments used in Chemistry laboratory:

Balance:

It is an instrument for measuring mass.

Pipettes:

They are used to transfer of known volumes of liquids from one container to another.

Common types are shown in the figure:

(a) Volumetric or bulb pipette delivers a single fixed volume of liquid.

(b) Mohr or graduated pipettes are calibrated in convenient units to permit the delivery of any volume up to the maximum capacity.

(a)

(b)

(c)

(d)

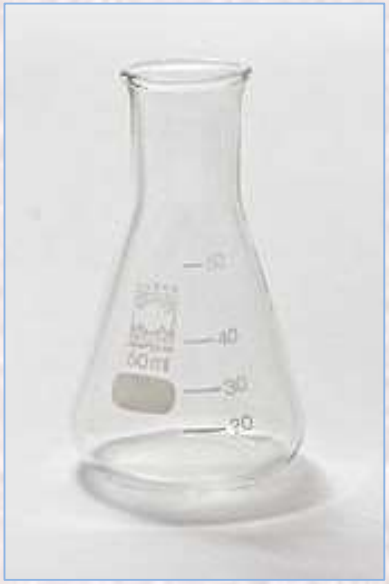
(a) Volumetric, bulbe, pipette.
(b) Graduated pipette.
(c) Digital pipette.
(d) Syringe.

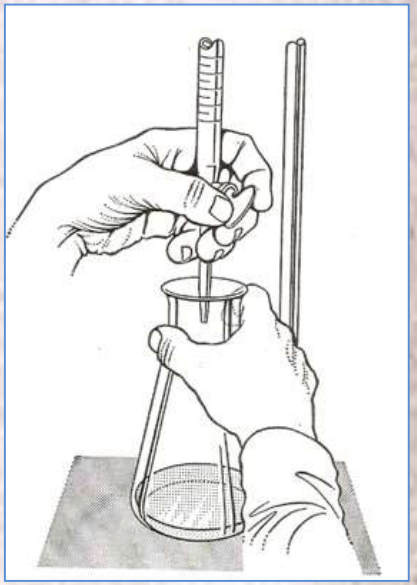
Flasks:

There are various types of flasks:

Erlenmeyer or Conical Flask:

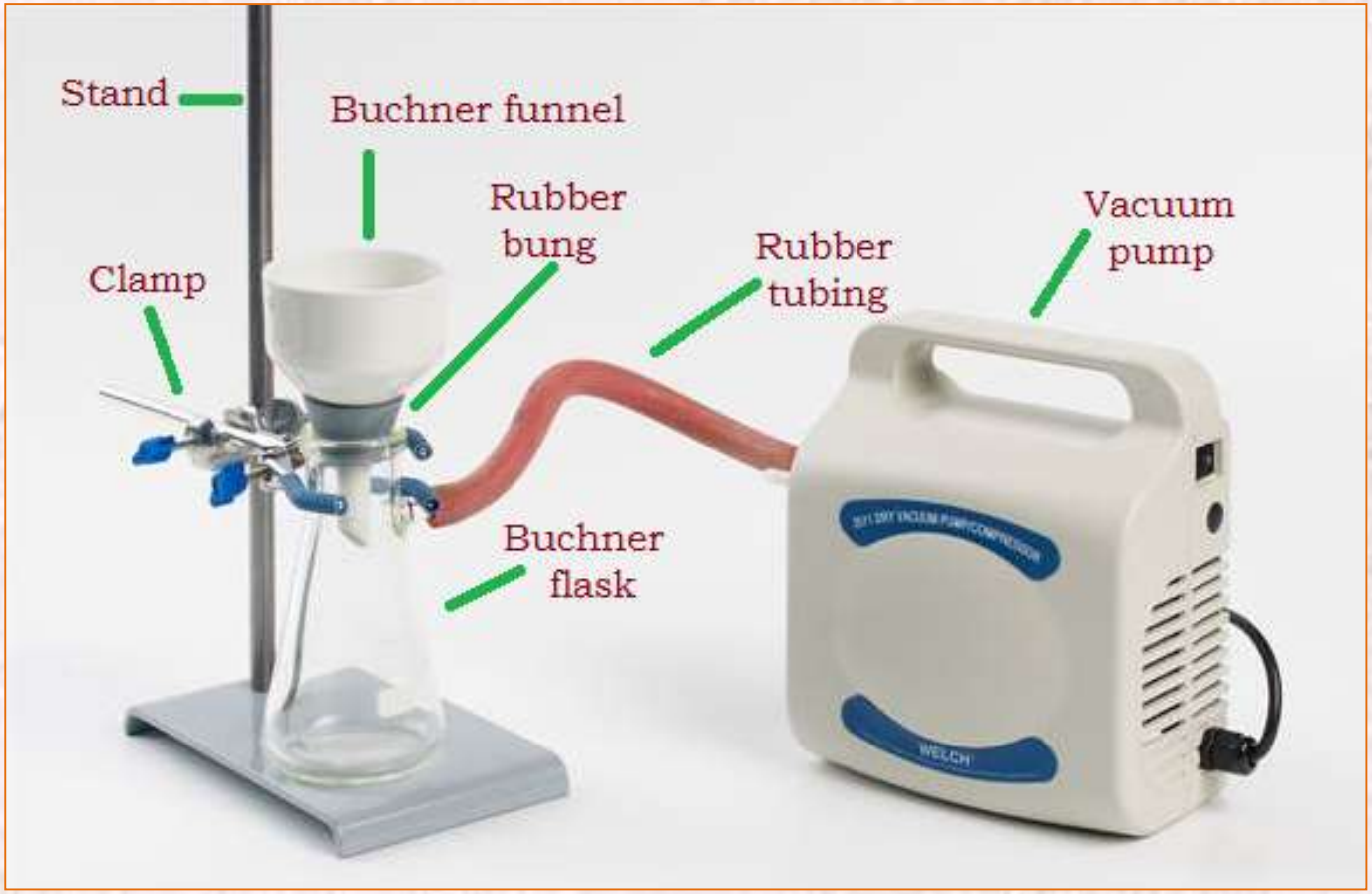
It's used in chemistry labs for titration , as they can hold the contents mixed single-handed leaving the other hand free to add the reagent .





Buchner (Vacuum) Flask:

It's a thick - walled Erlenmeyer flask with a short glass tube , used for filtration of sample under vacuum.



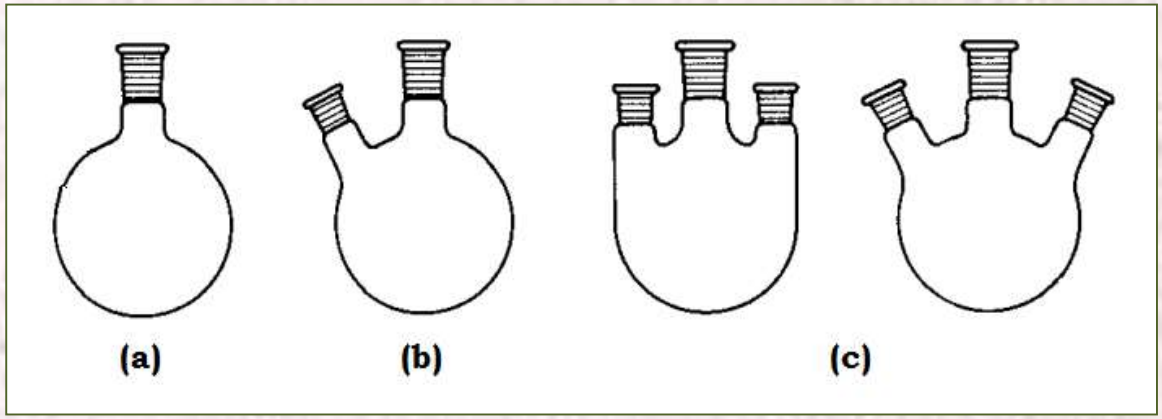
Buchner (Vacuum) Flask

Volumetric, Measuring or Graduated , Flask:

It is a pear - shaped, with a flat bottom , it's neck is elongated and narrowed with an single etched ring graduation marking . There are different sizes of volumetric flasks which are used for precise dilution and preparation of standard sol-utions.

Boiling Flask:

They are used for boiling liquids and in dist - illation apparatus.



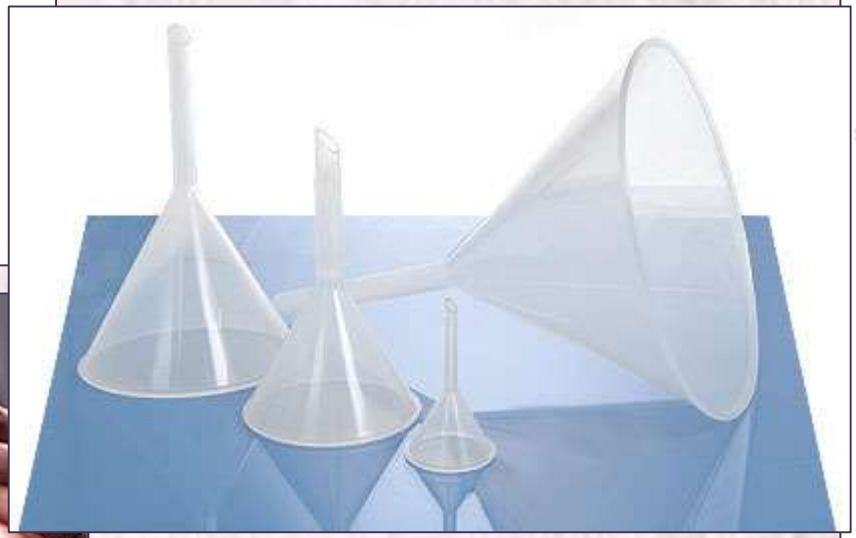
(a) is a round – bottomed boiling flask .
(b & c) are multi – necked round – bottomed Boiling flasks .

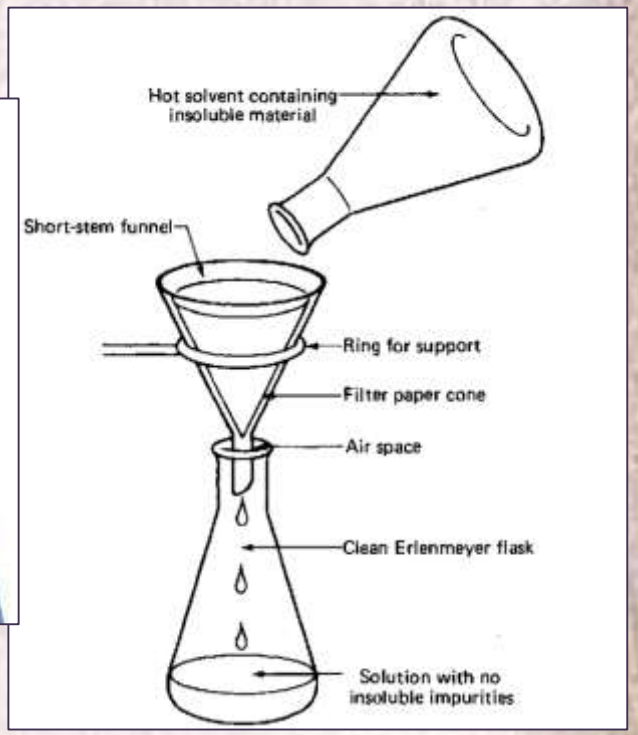
Funnels:

Various types of funnels are present:

Ordinary Funnel:

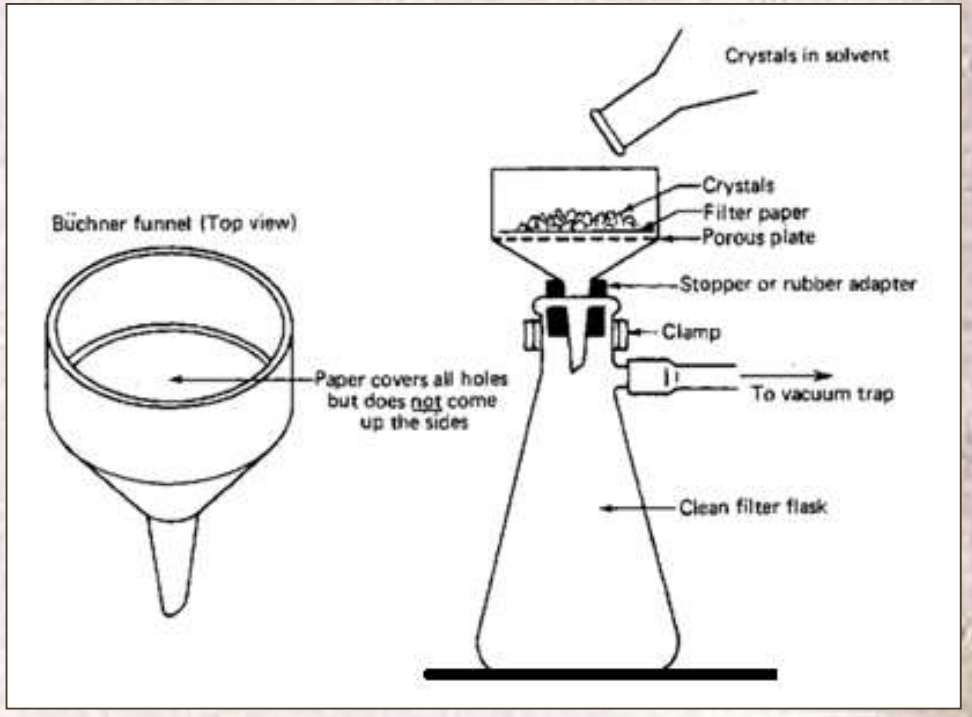
It's used for filtration and transfer of liquids and powder from one container to another.





Buchner Funnel:

It's made of porcelain and it has a perforated porcelain plate to support a filter paper.
A Buchner funnel is used in conjunction with a filter (vacuum) flask or tube for filtration by suction, (*vacuum filtration*).

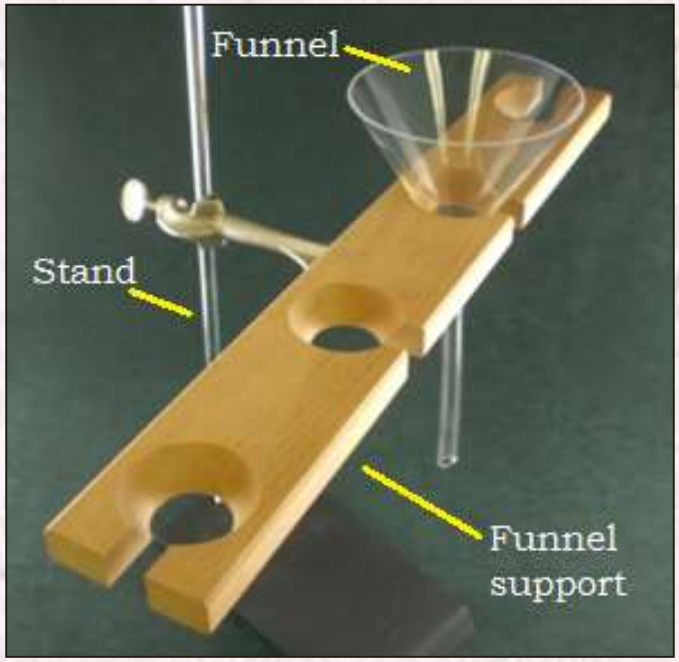


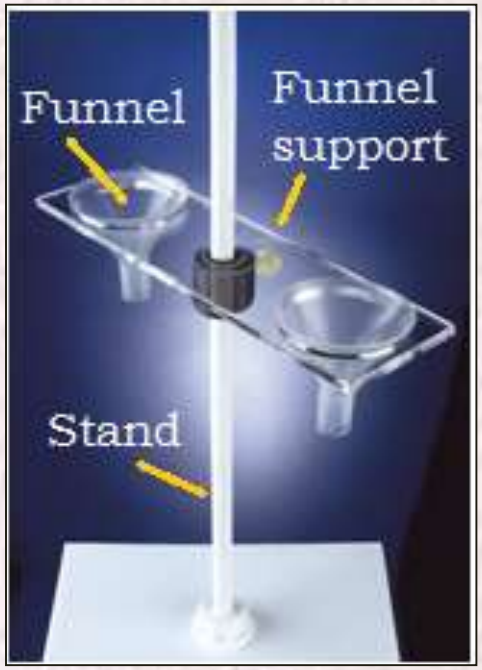
Separatory Funnel:

It is used for separation of two or more immiscible liquids, (*extraction process*).

Funnel Support:

It is used to support and hold funnels.



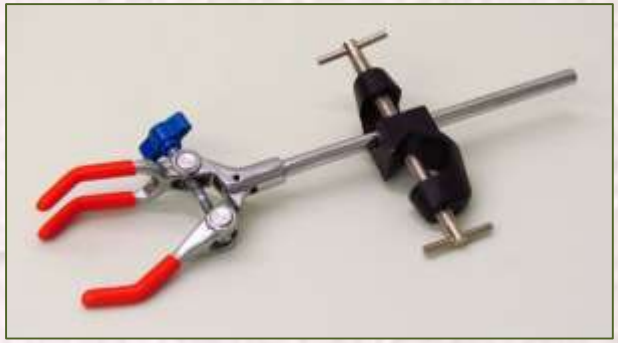


Stand:

It is used to support equipments.

Clamp:

When attached to the stand, this clamp is used to hold a large glassware above the lab table.

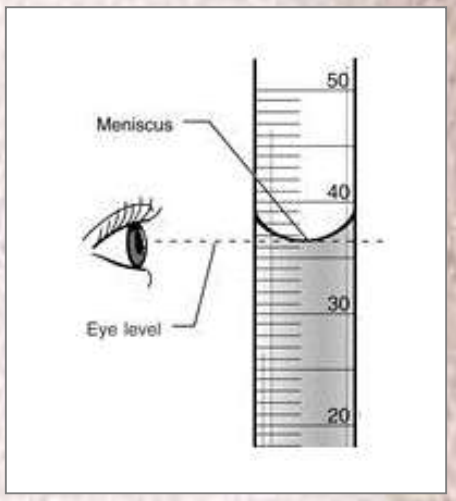


Iron Ring:

It's used to support glassware above the lab table.

Graduated Cylinder:

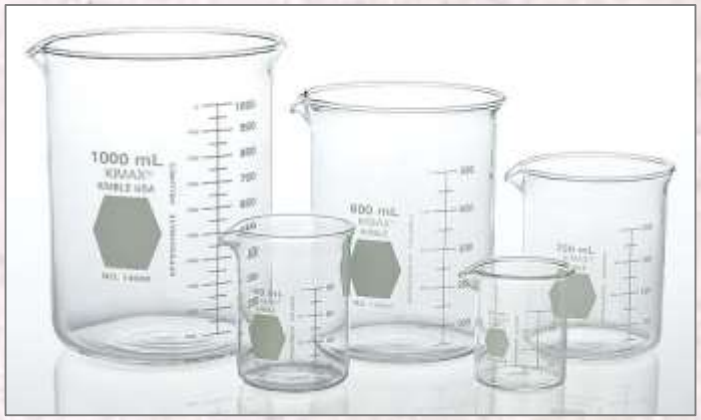
They are not highly accurate , but they are often used to measure specified quantities of liquids.



Beaker:

It is of multipurpose and essential in the lab.

Beaker is used to hold liquids.



Reagent Bottles:

They can be used for storage of chemical reagents .



Washing Bottle:

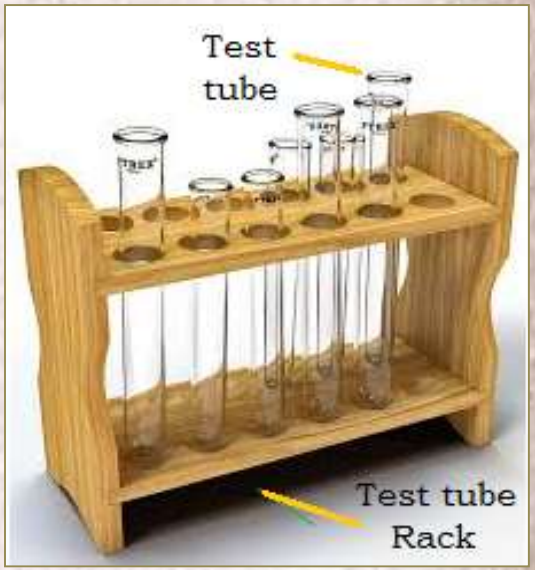
It's filled with distilled water to wash & clean laboratory glass-wares & rinsing solids out of a container when filtering.


Test Tubes:

They are widely used by chemists to hold, mix or heat small quantities of solid or liquid chemicals, especially for qualitative assays and experiments.

Test Tube Rack:

It is used to hold test tubes while reactions happen in them or while they are not needed.



Test Tube Holder:

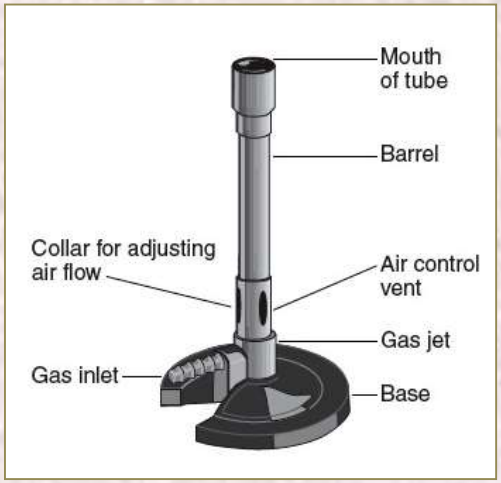
It is used to hold test tubes when they are hot & untouchable .

Test Tube Brush:

Test tube brush is used to easily clean the inside of a test tube.

Bunsen Burner:

It is used for heating and exposing items to flame.

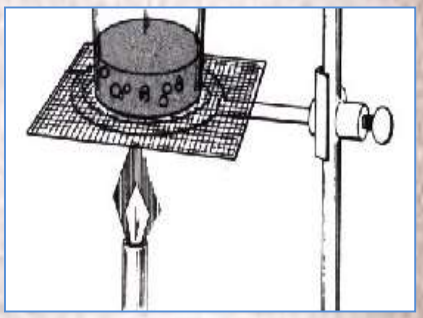


Ring Stand:

Ring or tripod stands are used to hold items being heated .

Wire Gauze:

Wire gauze, when placed between glassware & a heat source , diffuses the heat somewhat and is therefore safer than a direct flame .



Spatula:

Stainless steel and nickel spatula is used for handling of small quantities of material.

Filter Paper:

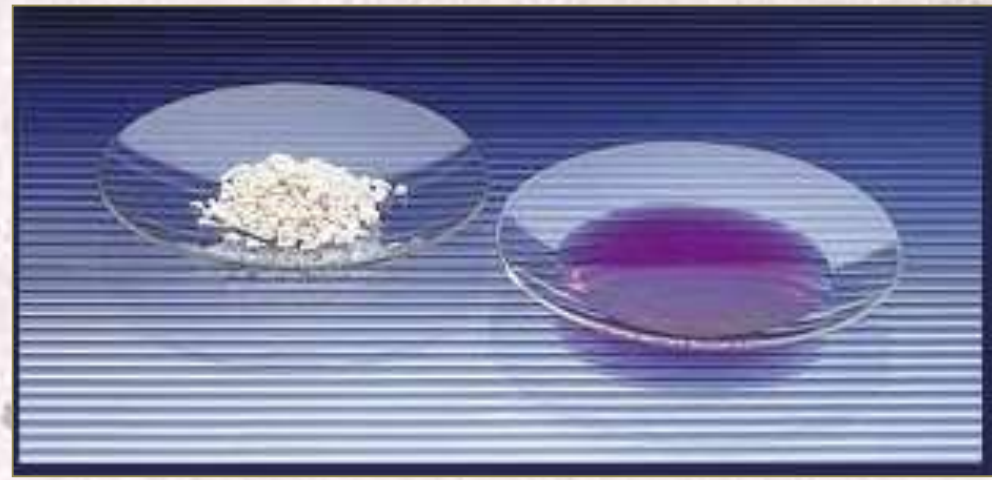
It's an important filtering medium. Ashless paper is made from cellulose fiber .

Watch Glass:

It is used to hold solids when being weighed or transported .

It can also be used to cover beakers .

It should never be heated .



Stirring Rod:

It is a glass rod used for stirring of liquids .

Rubber Policeman:

It's a small section of rubber tubing that has been crimp - ed on one end.

The open end of the tubing is fitted on to the end of a stirr - ing rod.

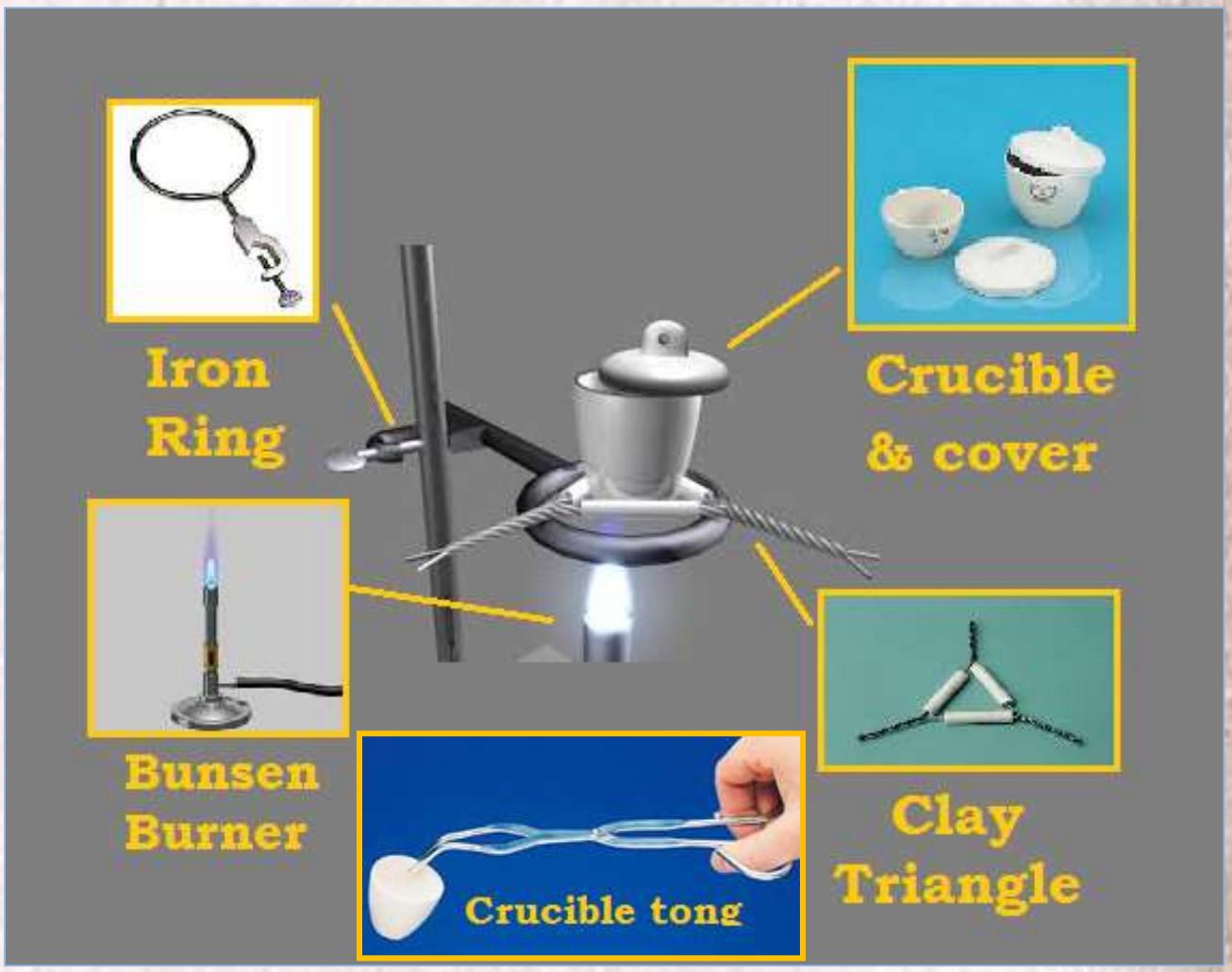
It is used in chemical lab. to transfer residues of precipitate or solid on glass surfaces when performing gravimetric analysis.

Crucible and Cover:

Crucibles are used as a container when some – thing requires "strong" heating.

Crucible Tong:

These tongs are used for picking up crucibles and crucible covers only.

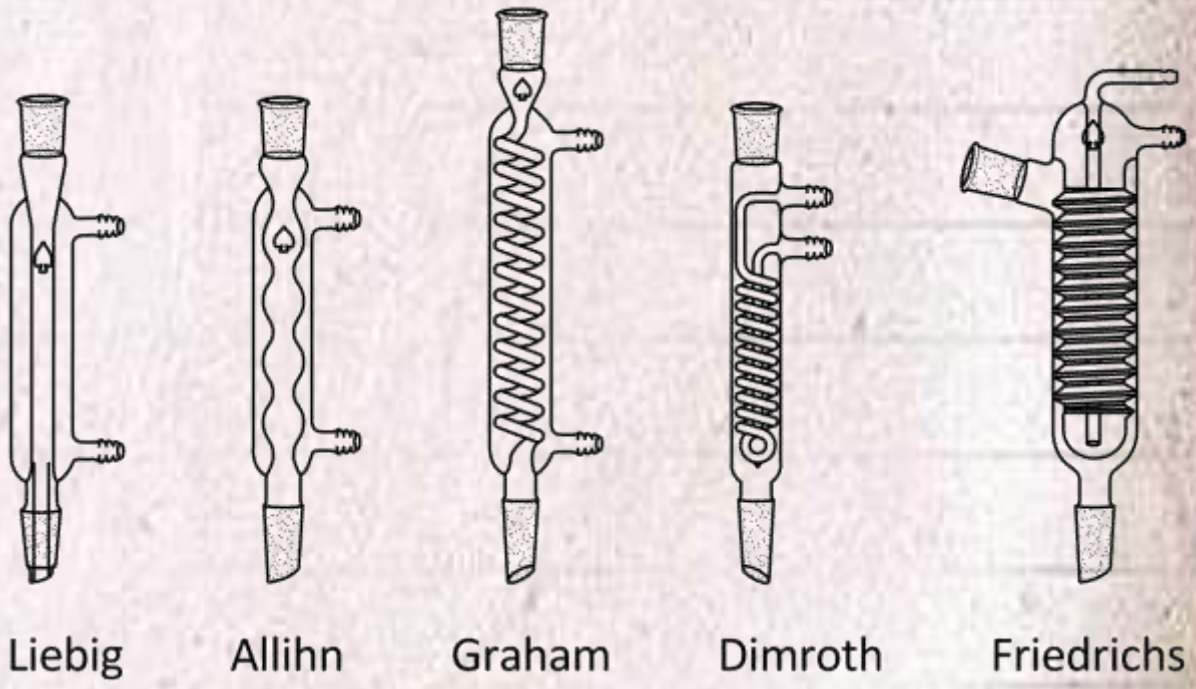


Clay Triangle:

It's used to hold crucibles when they are being heated . It usually sit on a ring stand .

Reflux Condenser:

It's employed for both reflux and for downward distillation.



Vacuum Desiccators :

Solids which are moist with either water or organic solvents are routinely dried in a vacuum desiccators at room tempera – ture .

Burettes:

Burettes, like measuring pipettes, make it possible to deliver any volume up to the maximum capacity of the device. The precision attainable with a burette is greater than the precision with a pipette . Burette is used in titrations to measure precisely how much liquid is used.

What are the rules that should be followed to prevent contamination of reagents & solutions?

What are the general rules of safety working in a chemical laboratory?

Referance:

Douglas A. Skoog , West , Holler and Crouch, Fundamentals of Analytical Chemistry, 9th edition, page 14 - 47, 2014.

Preparation & Standardization of 0.1 N HCl Solution

A reagent bottle of concentrated HCl has the following informations on it's lable:

Molar mass = 36.5 g.mol⁻

sp.gr = 1.18 g.L⁻

37% HCl (*w/w*)

How could you prepare: 1L of 0.1 N HCl solution from this conc. HCl ?

1- Calculation of the Normality of the concentrated HCl:

$$N_{HCl} = \frac{\text{Specific gravity} * \%\,(w/w) * 1000}{\text{Eq.mass of HCl}}$$

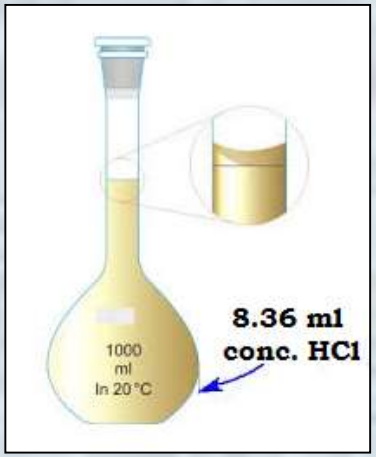
$$N_{HCl} = 11.961\ N$$

2- Calculation of the volume of 11.961N HCl that should be taken to prepare 1L of 0.1N HCl soln.

$$N_1 * V_{1\ \text{concentrated}} = N_2 * V_{2\ \text{diluted}}$$

$$11.961 * V_1 = 0.1 * 1000$$

V_1 = 8.36 ml of concentrated HCl should be taken and diluted to the mark with distilled water in a 1000 ml volumetric flask.



Standardization of the prepared HCl solution

If the chemical is available in a pure state, e.g. anhydrous Na_2CO_3, weigh out an exact quantity, dissolve it in water up to volume.

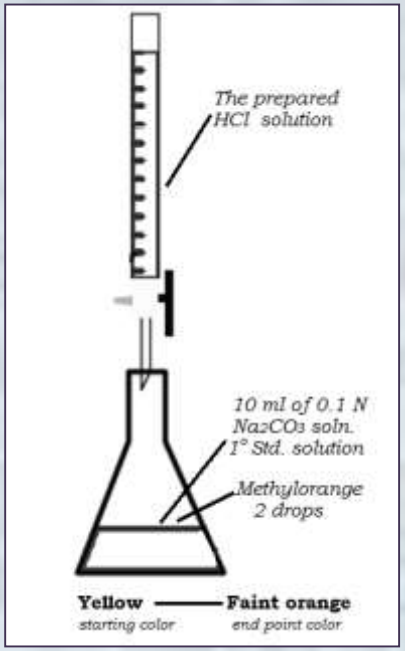
How could you prepare 0.5 L of 0.1 N Na_2CO_3? Knowing that, atomic masses of Na = 23, O = 16 and C = 12.

Substances which are not usually obtained in a pure state , e.g. mineral acids and caustic alkali, are prepared as approximate solutions and standardized against a known pure std., e.g. Na2CO3 as a primary std. soln.

Procedure:

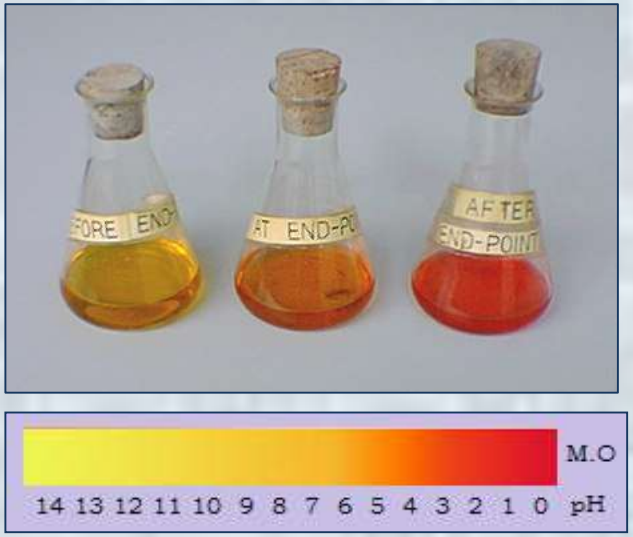
1- Fill the burette with the prepared HCl soln.

2- Transfer 10 ml of exactly 0.1 N Na_2CO_3 solution (1° - standard) in to a conical flask by using a 10 ml bulb pipette.



3- Add 2 drops of methyl orange as indicator. Yellow color is obtained.

4- Titrate with HCl soln. drop by drop from the burette in to the conical flask until a faint orange color is obtained.

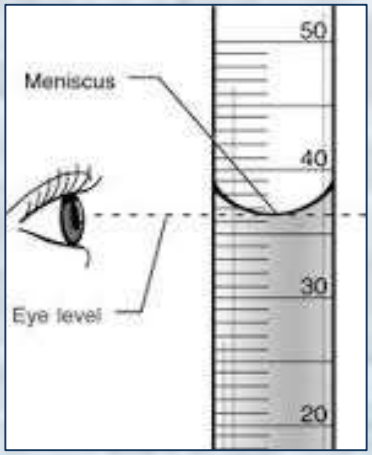


5- The exact normality can be calculated from the following equation,

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

$$N_1 * V_{1\ Na_2CO_3} = N_2 * V_{2\ HCl}$$

Directions for reading a volumetric equipment:



Avoiding a parallax:

The top surface of a liquid confined in a narrow tube exhibits a marked curvature, meniscus.

It's common practice to use the bottom of the meniscus.

Post Lab Exercise:

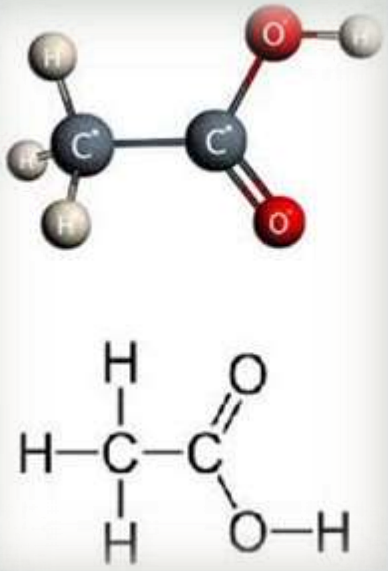
A bottle of concentrated HCl has the following informations on it's label: molar mass is 36.5 g/mol, sp.gr. 1.18 g/L and 40% HCl (*w/w*) .

a- What is the normality of the HCl in the bottle?

b- How could you prepare 2 liters of about 0.1 N HCl solution from the concentrated reagent?

Determination of the %w/v Acetic Acid in a Vinegar Sample

Vinegar is a liquid consisting mainly of **Acetic Acid** (CH_3COOH) and water.



Glacial Acetic Acid

It is a water-free (anhydrous) acetic acid, the name comes from the ice - like crystals that form slightly below room temperature at 16.6 °C .

An abbreviation for acetic acid is **HAc** where **Ac** stands for acetate, CH_3COO^-

Acetate is the ion resulting from loss of H^+ from acetic acid.

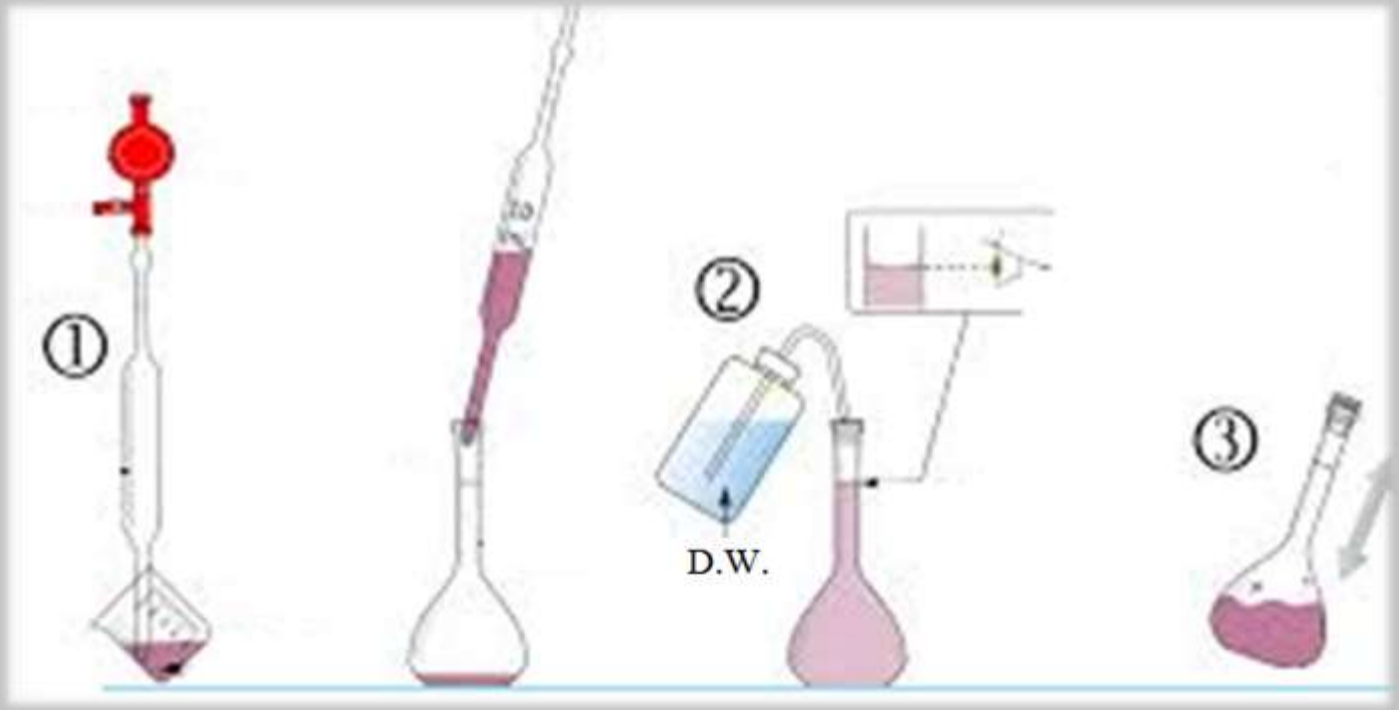
Ice - like crystals of Glacial Acetic Acid

Preparation of unknown acetic acid solution:

1- Transfer 10 ml of unknown by using 10 ml bulb pipette into a 100 ml volumetric flask.

2- Complete the volume with distilled water.

3- Stopper the flask and shake well.



Name of Experiment: Acid - Base Titration.

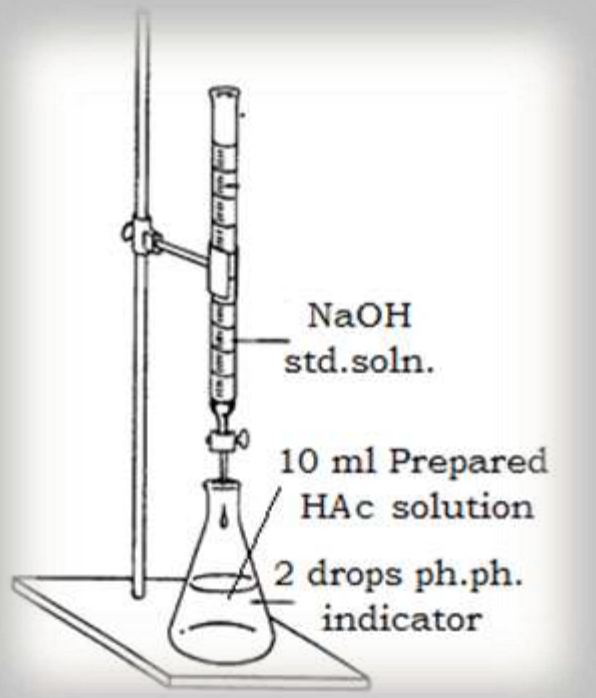
Aim of Experiment: Determination of %w/v Acetic Acid in an unknown Vinegar sample .

Principle:

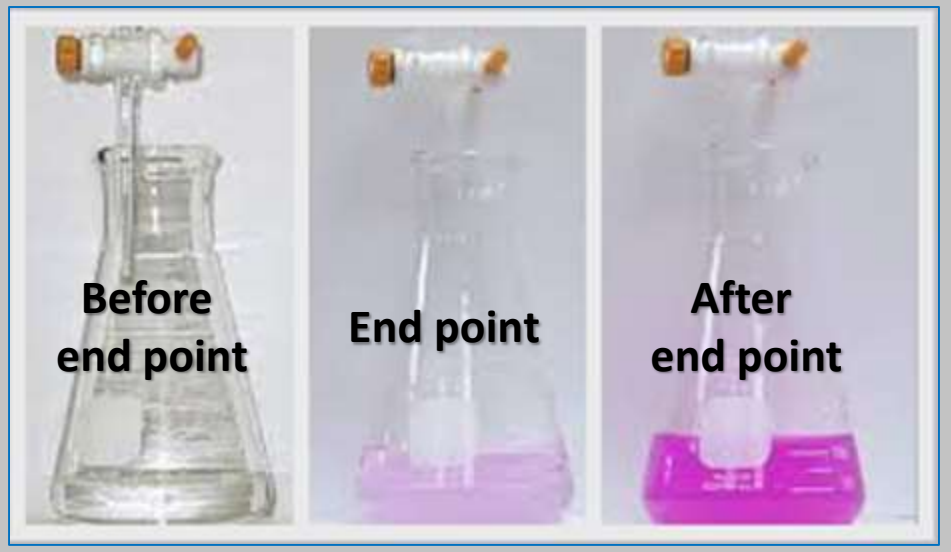
Acetic acid is a weak acid Ka = 1.8×10^{-5} , So titration of acetic acid against NaOH is a titration of a weak acid against a strong base. The end point will be not very sharp.

Procedure:

1- Using a 10 ml bulb pipette transfer 10 ml of the prepared solution to a conical flask.
2- Add 2 drops of ph.ph. indicator.
3- Fill the burette with 0.1 N NaOH standard solution.



4- Titrate the prepared acetic acid solution with standard NaOH soln. until the color of the indicator becomes faint pink.



Calculations:

$$N_1 \quad V_1 \quad = \quad N_2 \quad V_2$$
$$\text{NaOH} \qquad\qquad \text{HAc}$$

$$N_1 \quad V_1 \quad = \quad \frac{\text{Mass}_{\text{HAc}}}{\text{Eq.mass}_{\text{HAc}}} \quad * \quad 1000$$
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Example

Calculate the % w/v HAc for 10 ml sample of a vinegar that required 30ml of 0.2N NaOH soln.? Knowing that the atomic masses for Na= 23 , O = 16 , H = 1 and for C = 12.

$$N_1 \; V_{1\,NaOH} = N_2 \; V_{2\,HAc}$$

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$$Mass_{HAc} = 0.36 \text{ g HAc in } 10 \text{ ml sample.}$$

0.36 g	10 ml
X	100 ml

$$X = \frac{0.36 g * 100 \cancel{ml}}{10 \cancel{ml}}$$

$$X = 3.6 \text{ g} = 3.6 \% \; w/v$$

Notes

In general,

For weak acid titrations,

the pH at the end point is above 7, So ph.ph. is the most suitable indicator which can be used.

For weak bases ,

the pH at the end point is below 7, So M.R. (4.2 – 6.2) or M.O. (3.1 – 4.4) are widely used.

For strong acids & strong bases,

M.R. , Bromothymole blue , ph.ph. are most suitable.

Phenolphthalein indicator is the most frequently used indicator in the titration of acetic acid with NaOH standard solution ?

The pH of the solution at the equivalence point will be above pH 7, (pH about 8.72), So ph.ph. is used (since the pH at the end point of this titration is within the pH range of ph. ph. Indicator which is 8.3 – 10).

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Common types are shown in the figure:

(a) Volumetric or bulb pipette delivers a single fixed volume of liquid.

(b) Mohr or graduated pipettes are calibrated in convenient units to permit the delivery of any volume up to the maximum capacity.

(a)

(b)

(c)

(d)

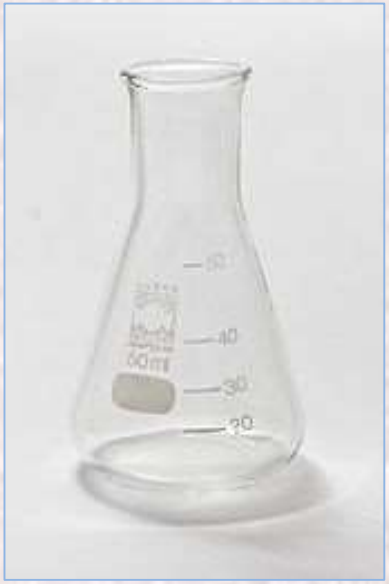
(a) Volumetric, bulbe, pipette.
(b) Graduated pipette.
(c) Digital pipette.
(d) Syringe.

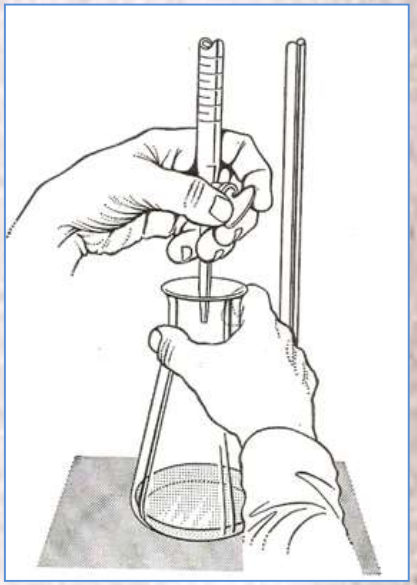
Flasks:

There are various types of flasks:

Erlenmeyer or Conical Flask:

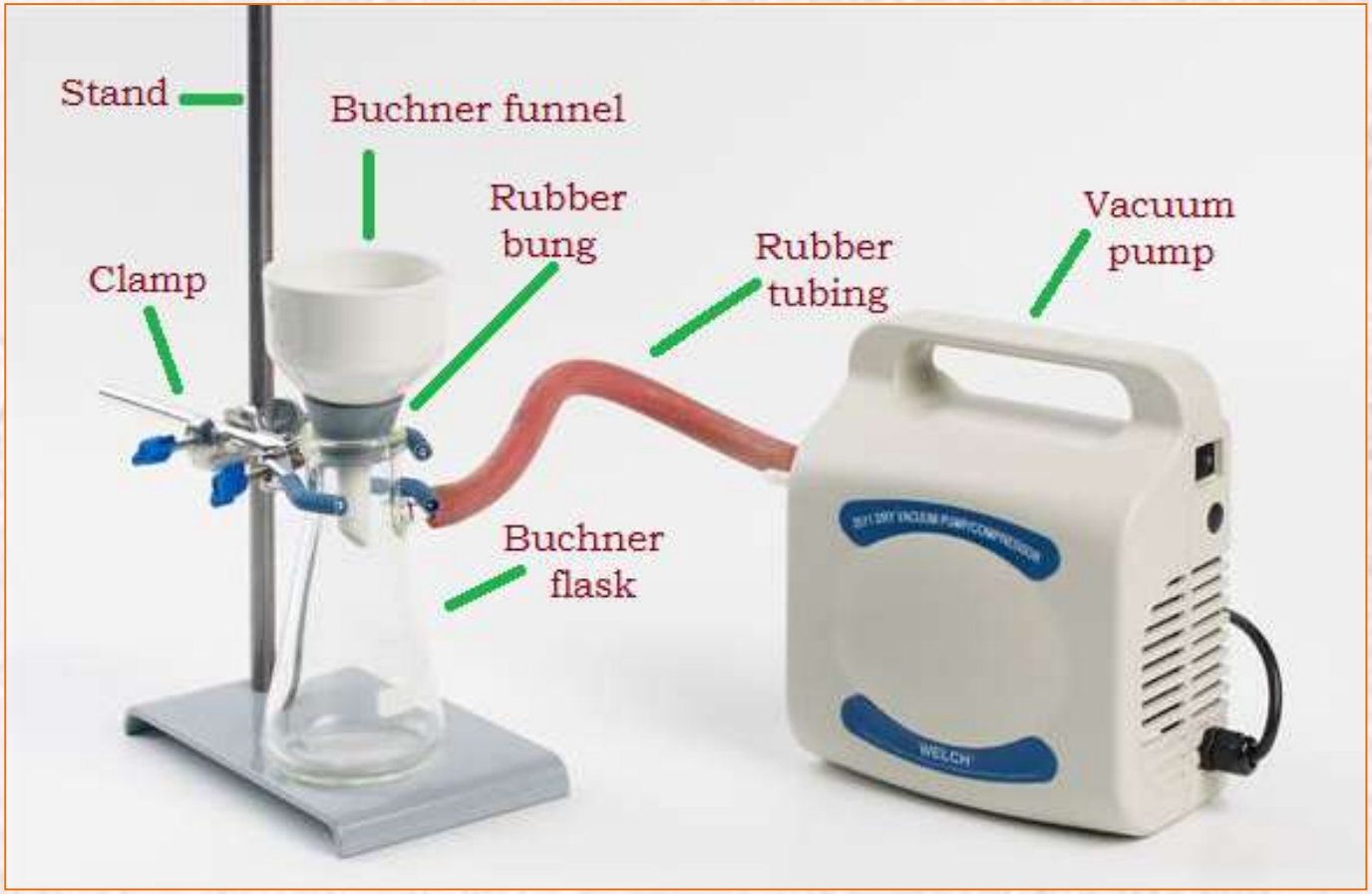
It's used in chemistry labs for titration , as they can hold the contents mixed single-handed leaving the other hand free to add the reagent .





Buchner (Vacuum) Flask:

It's a thick - walled Erlenmeyer flask with a short glass tube , used for filtration of sample under vacuum.



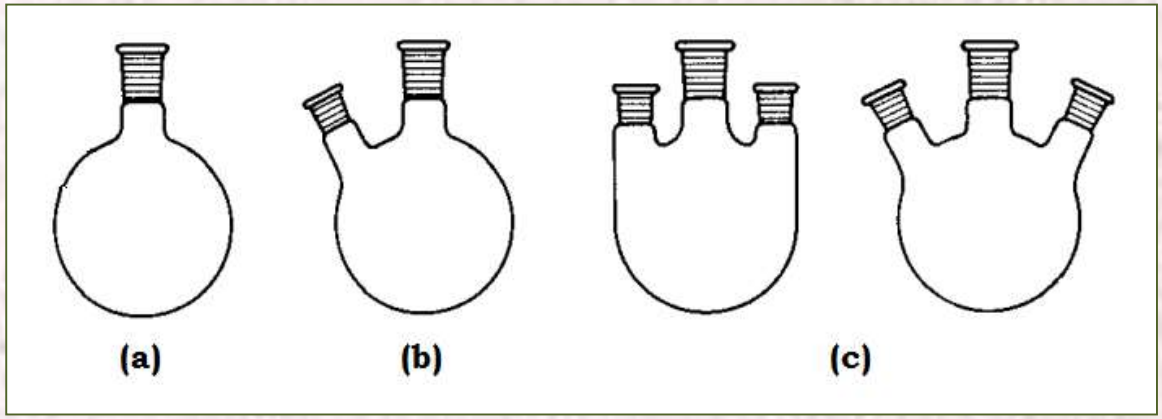
Buchner (Vacuum) Flask

Volumetric, Measuring or Graduated , Flask:

It is a pear - shaped, with a flat bottom , it's neck is elongated and narrowed with an single etched ring graduation marking . There are different sizes of volumetric flasks which are used for precise dilution and preparation of standard sol-utions.

Boiling Flask:

They are used for boiling liquids and in dist - illation apparatus.



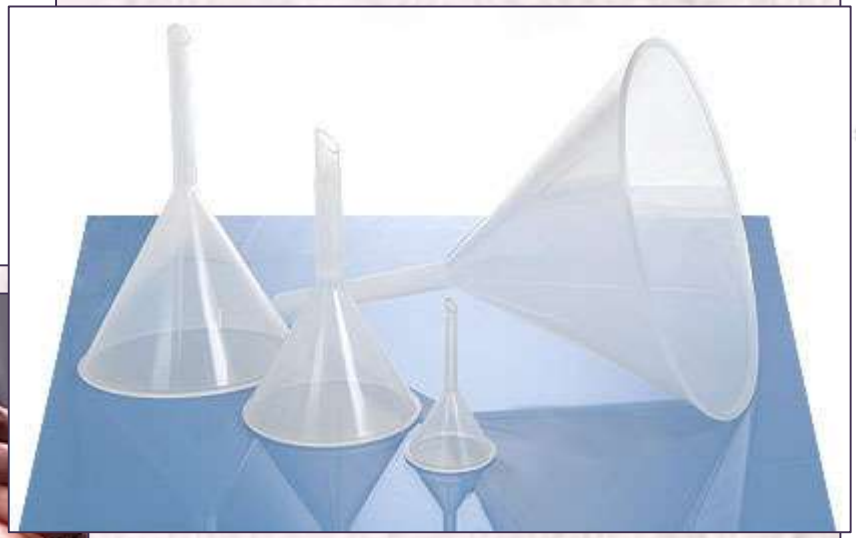
(a) is a round – bottomed boiling flask .
(b & c) are multi – necked round – bottomed Boiling flasks .

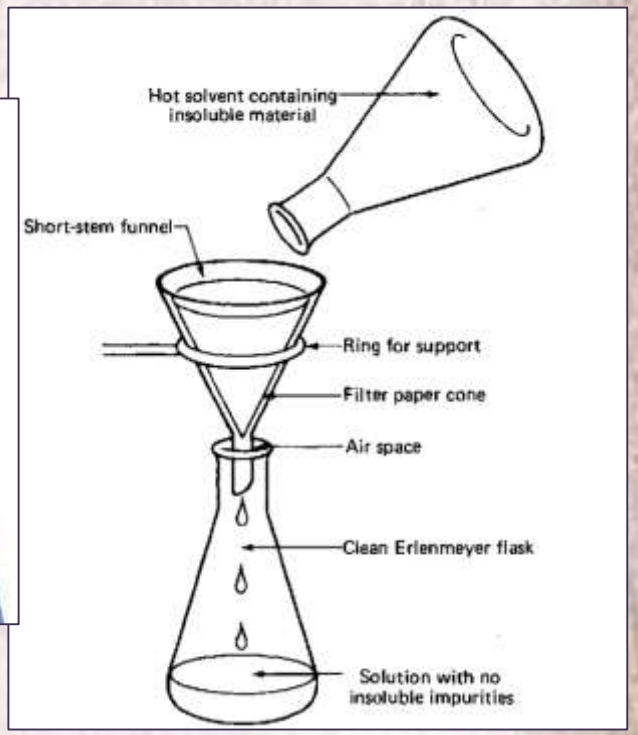
Funnels:

Various types of funnels are present:

Ordinary Funnel:

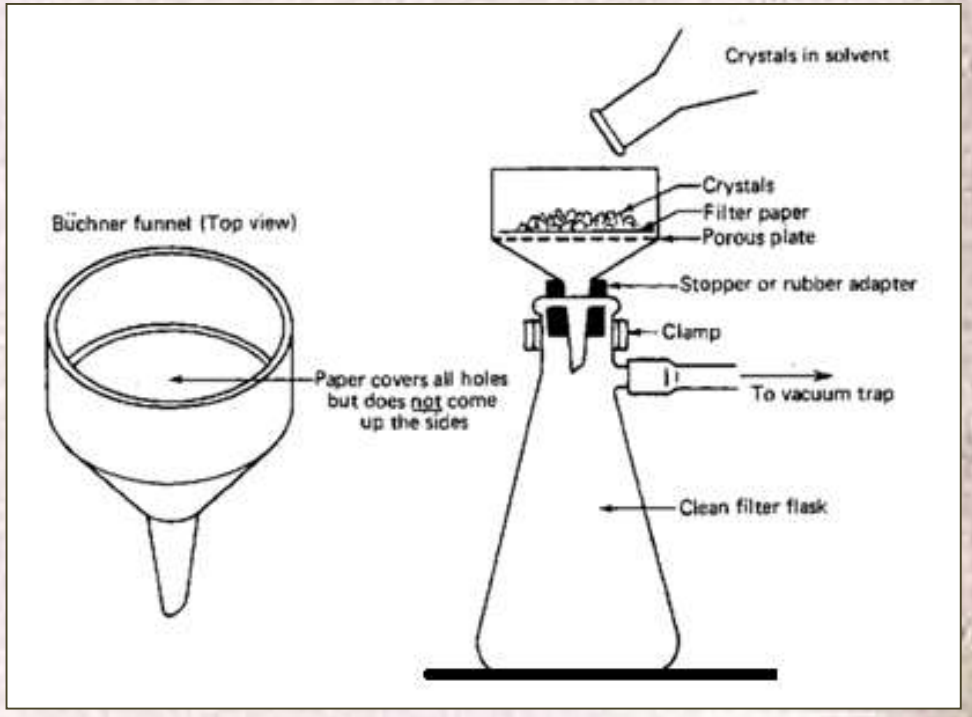
It's used for filtration and transfer of liquids and powder from one container to another.





Buchner Funnel:

It's made of porcelain and it has a perforated porcelain plate to support a filter paper.
A Buchner funnel is used in conjunction with a filter (vacuum) flask or tube for filtration by suction, (*vacuum filtration*).

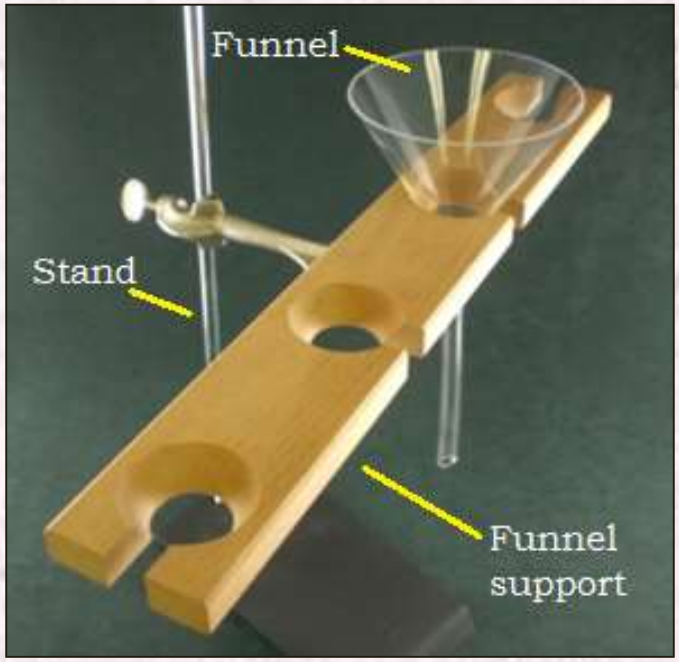


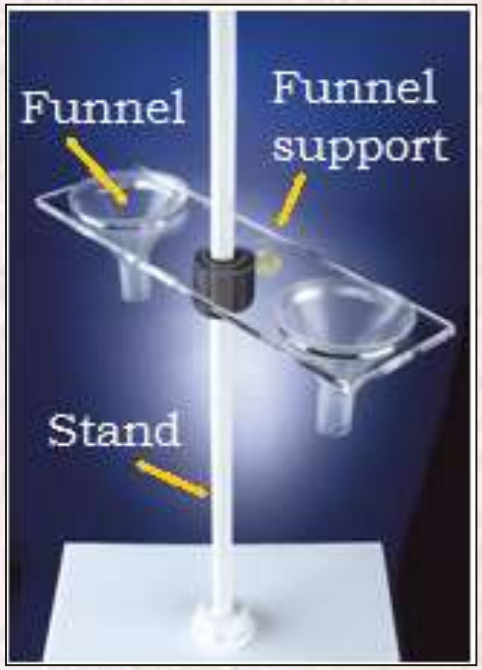
Separatory Funnel:

It is used for separation of two or more immiscible liquids, (*extraction process*).

Funnel Support:

It is used to support and hold funnels.



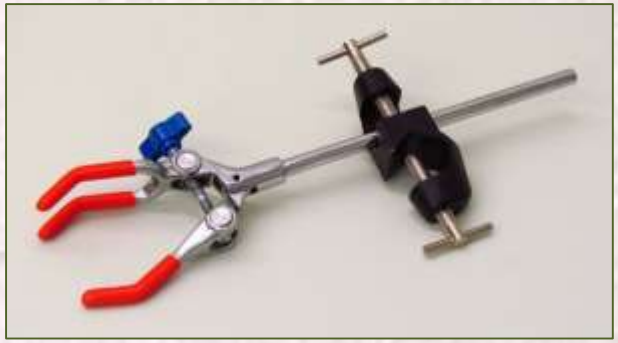


Stand:

It is used to support equipments.

Clamp:

When attached to the stand, this clamp is used to hold a large glassware above the lab table.

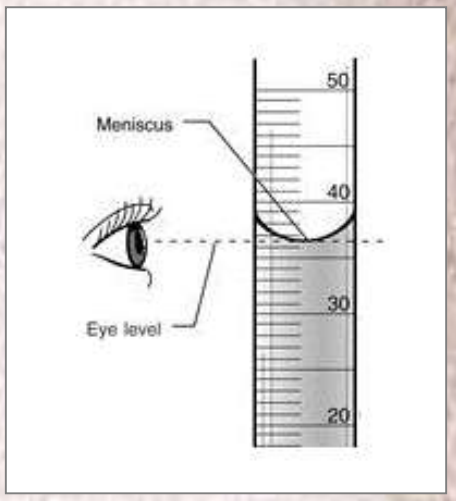


Iron Ring:

It's used to support glassware above the lab table.

Graduated Cylinder:

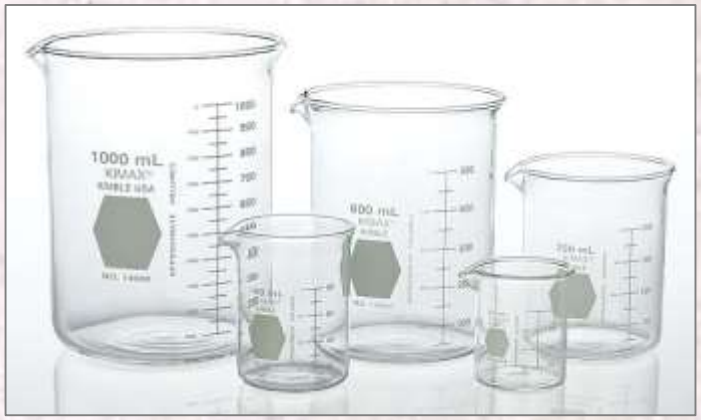
They are not highly accurate , but they are often used to measure specified quantities of liquids.



Beaker:

It is of multipurpose and essential in the lab.

Beaker is used to hold liquids.



Reagent Bottles:

They can be used for storage of chemical reagents .



Washing Bottle:

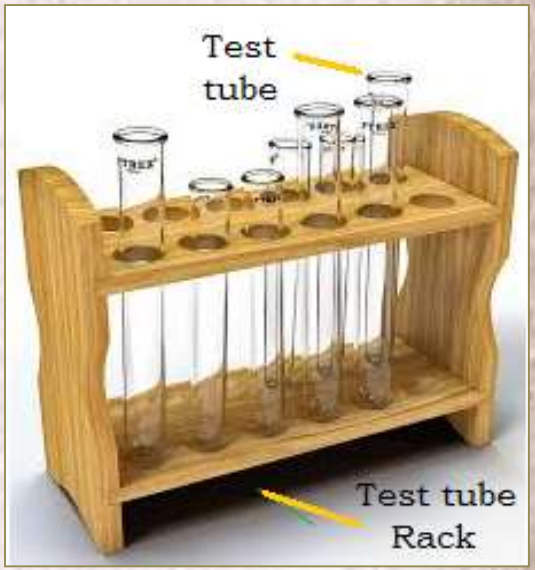
It's filled with distilled water to wash & clean laboratory glass-wares & rinsing solids out of a container when filtering.


Test Tubes:

They are widely used by chemists to hold, mix or heat small quantities of solid or liquid chemicals, especially for qualitative assays and experiments.

Test Tube Rack:

It is used to hold test tubes while reactions happen in them or while they are not needed.



Test Tube Holder:

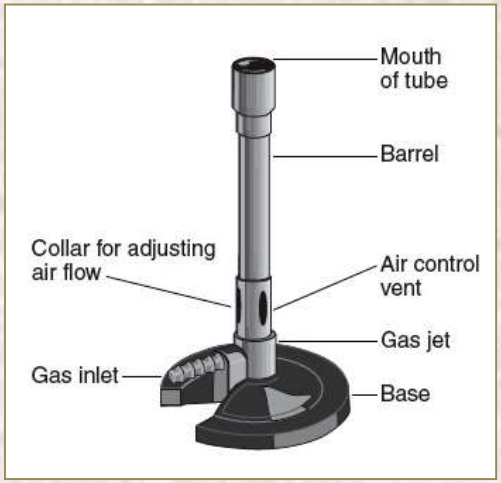
It is used to hold test tubes when they are hot & untouchable .

Test Tube Brush:

Test tube brush is used to easily clean the inside of a test tube.

Bunsen Burner:

It is used for heating and exposing items to flame.

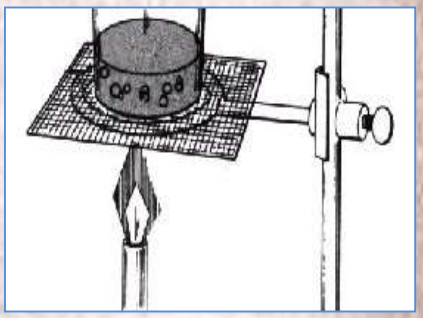


Ring Stand:

Ring or tripod stands are used to hold items being heated .

Wire Gauze:

Wire gauze, when placed between glassware & a heat source , diffuses the heat somewhat and is therefore safer than a direct flame .



Spatula:

Stainless steel and nickel spatula is used for handling of small quantities of material.

Filter Paper:

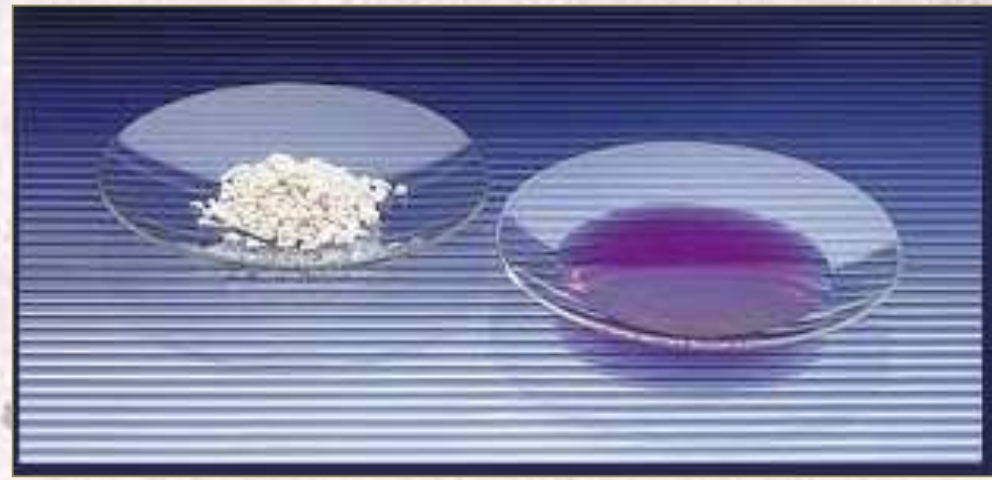
It's an important filtering medium. Ashless paper is made from cellulose fiber .

Watch Glass:

It is used to hold solids when being weighed or transported .

It can also be used to cover beakers .

It should never be heated .



Stirring Rod:

It is a glass rod used for stirring of liquids .

Rubber Policeman:

It's a small section of rubber tubing that has been crimp - ed on one end.

The open end of the tubing is fitted on to the end of a stirr - ing rod.

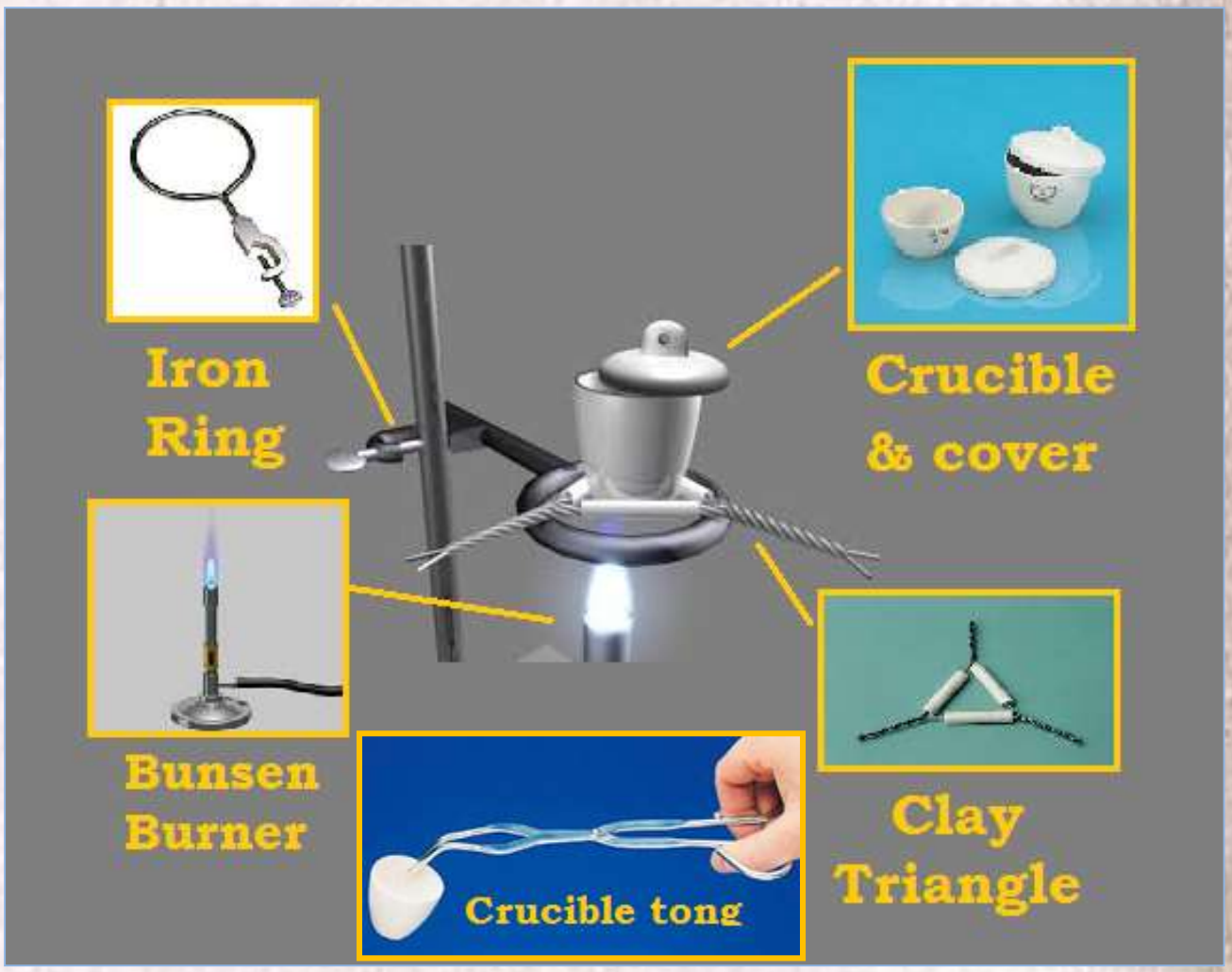
It is used in chemical lab. to transfer residues of precipitate or solid on glass surfaces when performing gravimetric analysis.

Crucible and Cover:

Crucibles are used as a container when some – thing requires "strong" heating.

Crucible Tong:

These tongs are used for picking up crucibles and crucible covers only.

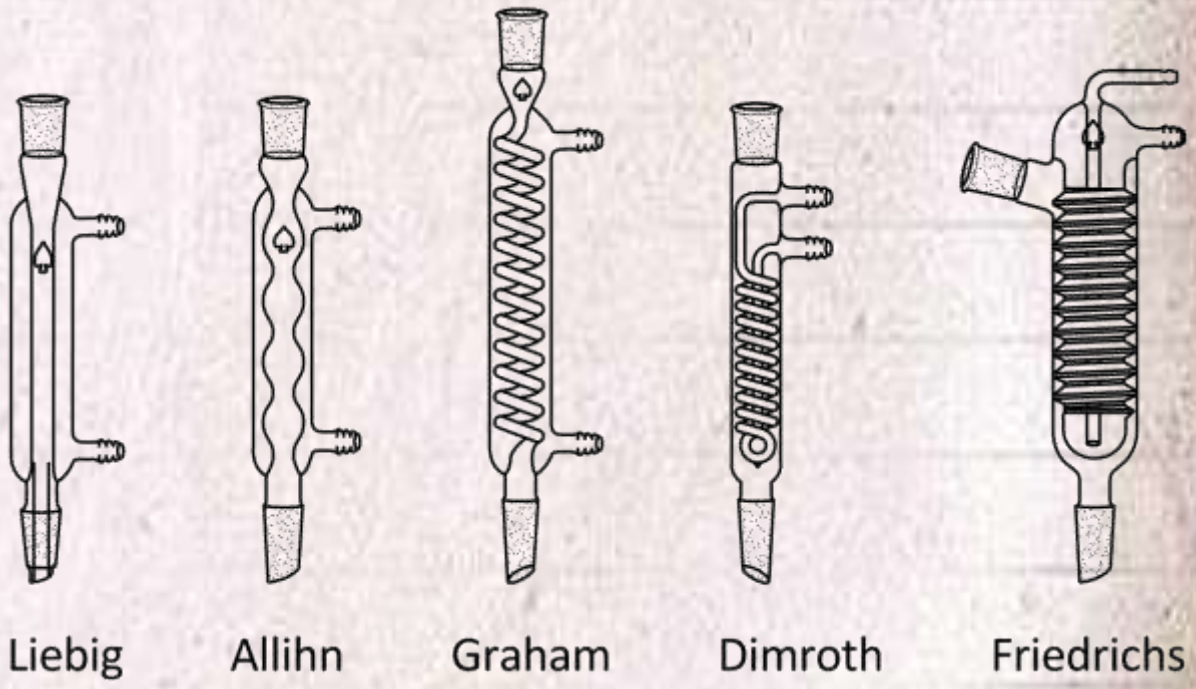


Clay Triangle:

It's used to hold crucibles when they are being heated . It usually sit on a ring stand .

Reflux Condenser:

It's employed for both reflux and for downward distillation.



Vacuum Desiccators :

Solids which are moist with either water or organic solvents are routinely dried in a vacuum desiccators at room tempera – ture .

Burettes:

Burettes, like measuring pipettes, make it possible to deliver any volume up to the maximum capacity of the device. The precision attainable with a burette is greater than the precision with a pipette . Burette is used in titrations to measure precisely how much liquid is used.

What are the rules that should be followed to prevent contamination of reagents & solutions?

What are the general rules of safety working in a chemical laboratory?

Referance:

Douglas A. Skoog , West , Holler and Crouch, Fundamentals of Analytical Chemistry, 9th edition, page 14 - 47, 2014.

Preparation & Standardization of 0.1 N HCl Solution

A reagent bottle of concentrated HCl has the following informations on it's lable:

Molar mass = 36.5 g.mol⁻

sp.gr = 1.18 g.L⁻

37% HCl (*w/w*)

How could you prepare: 1L of 0.1 N HCl solution from this conc. HCl ?

1- Calculation of the Normality of the concentrated HCl:

$$N_{HCl} = \frac{\text{Specific gravity} * \%\,(w/w) * 1000}{\text{Eq.mass of HCl}}$$

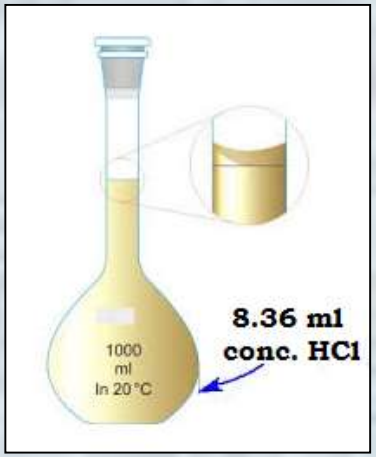
$$N_{HCl} = 11.961\ N$$

2- Calculation of the volume of 11.961N HCl that should be taken to prepare 1L of 0.1N HCl soln.

$$N_1 * V_{1\ \text{concentrated}} = N_2 * V_{2\ \text{diluted}}$$

$$11.961 * V_1 = 0.1 * 1000$$

V_1 = 8.36 ml of concentrated HCl should be taken and diluted to the mark with distilled water in a 1000 ml volumetric flask.



Standardization of the prepared HCl solution

If the chemical is available in a pure state, e.g. anhydrous Na_2CO_3, weigh out an exact quantity, dissolve it in water up to volume.

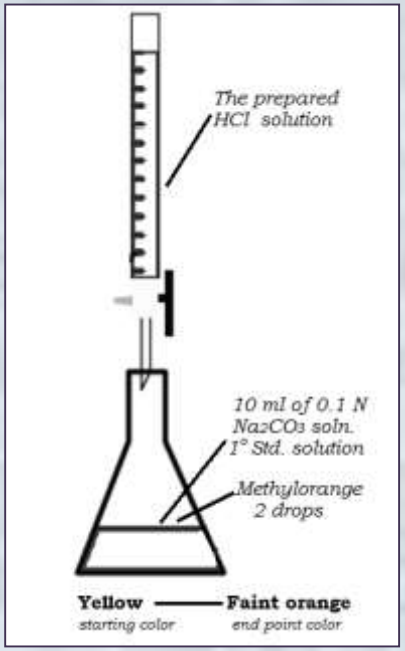
How could you prepare 0.5 L of 0.1 N Na_2CO_3? Knowing that, atomic masses of Na = 23, O = 16 and C = 12.

Substances which are not usually obtained in a pure state , e.g. mineral acids and caustic alkali, are prepared as approximate solutions and standardized against a known pure std., e.g. Na2CO3 as a primary std. soln.

Procedure:

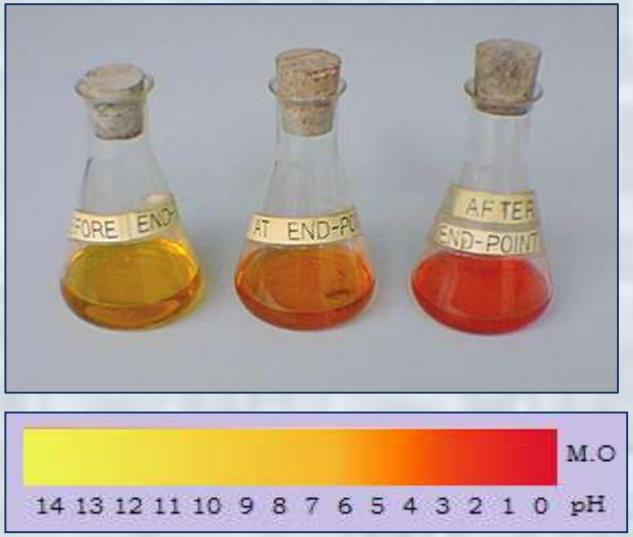
1- Fill the burette with the prepared HCl soln.

2- Transfer 10 ml of exactly 0.1 N Na_2CO_3 solution (1° - standard) in to a conical flask by using a 10 ml bulb pipette.



3- Add 2 drops of methyl orange as indicator. Yellow color is obtained.

4- Titrate with HCl soln. drop by drop from the burette in to the conical flask until a faint orange color is obtained.

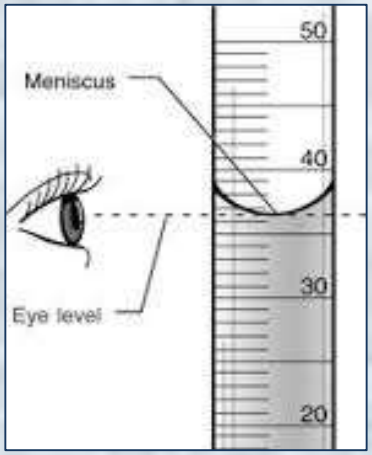


5- The exact normality can be calculated from the following equation,

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

$$N_1 * V_{1\ Na_2CO_3} = N_2 * V_{2\ HCl}$$

Directions for reading a volumetric equipment:



Avoiding a parallax:

The top surface of a liquid confined in a narrow tube exhibits a marked curvature, meniscus.

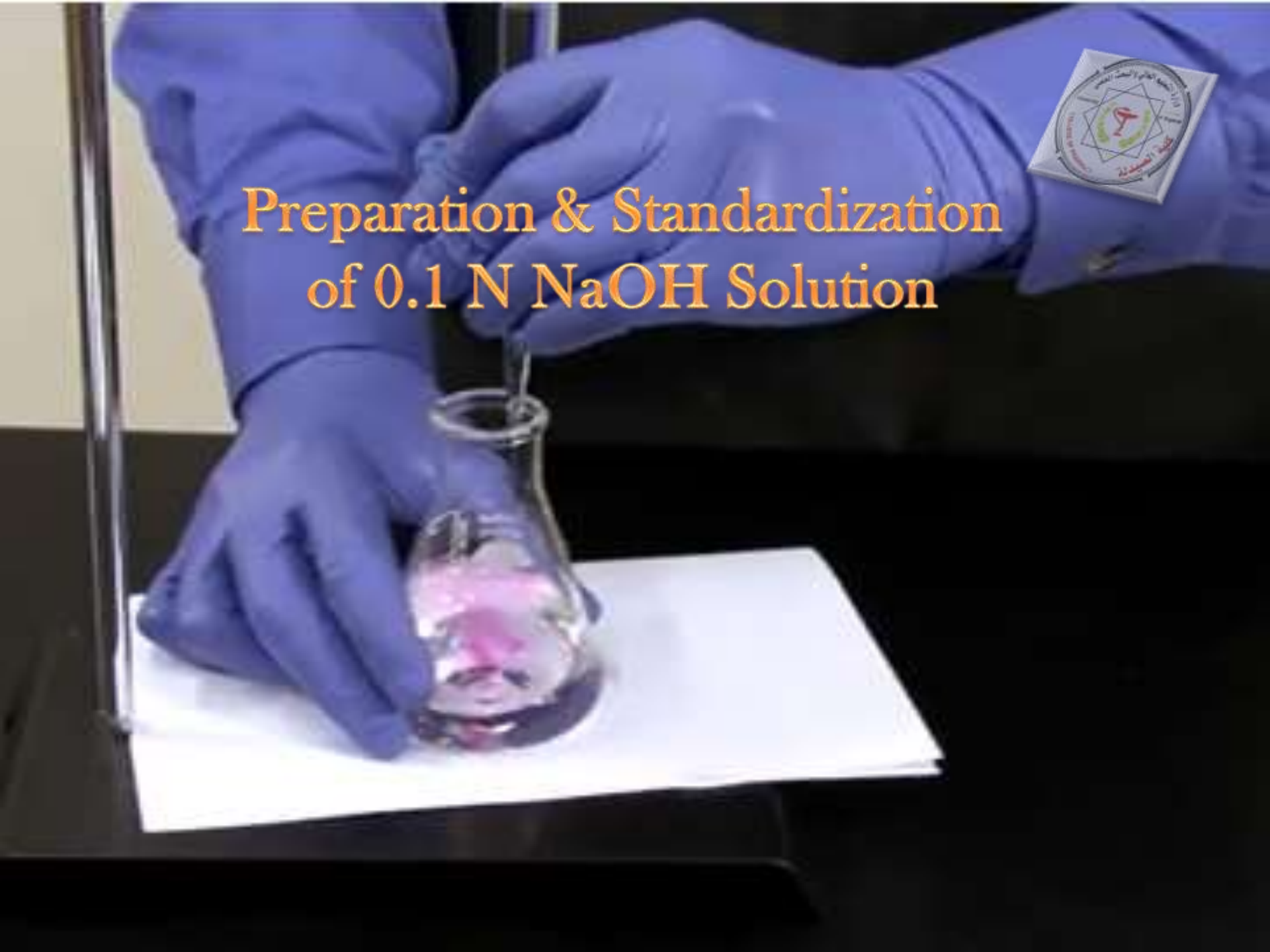
It's common practice to use the bottom of the meniscus.

Post Lab Exercise:

A bottle of concentrated HCl has the following informations on it's label: molar mass is 36.5 g/mol, sp.gr. 1.18 g/L and 40% HCl (*w/w*) .

a- What is the normality of the HCl in the bottle?

b- How could you prepare 2 liters of about 0.1 N HCl solution from the concentrated reagent?


Preparation & Standardization
of 0.1 N NaOH Solution

How could you prepare one liter of N/10 NaOH solution?

Knowing that the atomic masss of :

Na = 23 , O = 16 and H = 1.

$$N = \frac{\text{no. of equivalents}}{\text{Volume (L)}} = \frac{\text{Mass / Equivalent mass}}{\text{Volume (L)}}$$

$$= \frac{\text{Mass} * 1000}{\text{Eq. mass} * \text{V (ml)}}$$

$$0.1 = \frac{\text{Mass} * 1000}{40 * 1000}$$

Mass = 4 g of NaOH.

So, we should weigh 4 g of NaOH , dissolve it in water and make up to the mark of 1 liter volumetric flask.

Standardization of the prepared NaOH solution

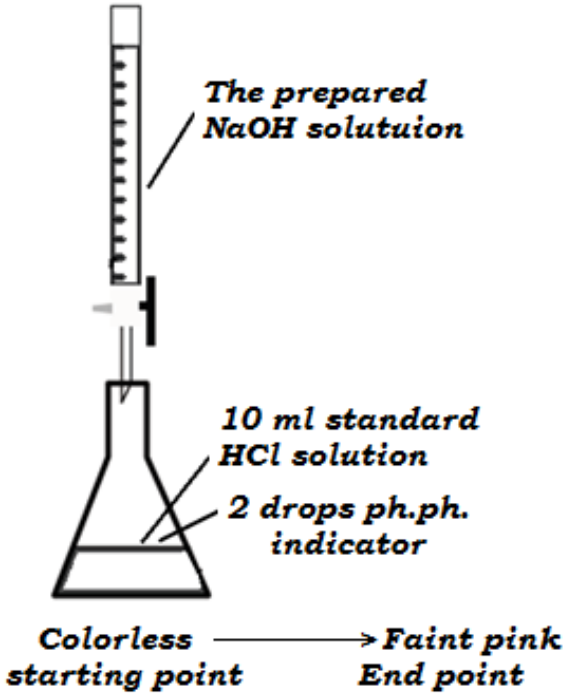
Solutions of NaOH are standardized by titration with std. acids of equivalent normality.

Method:

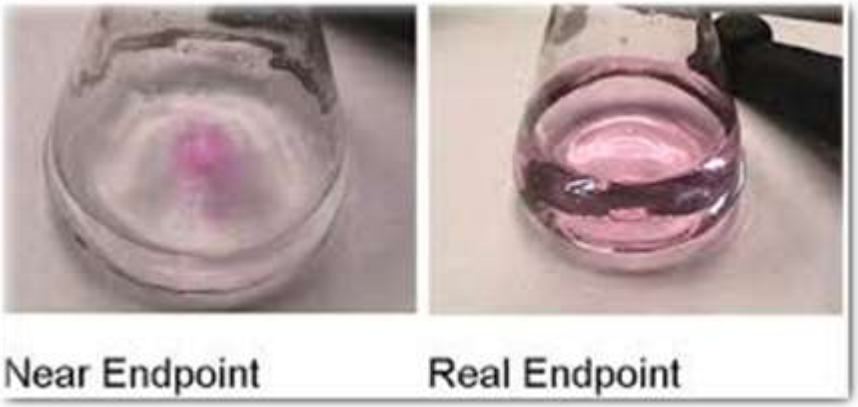
1- Using a bulb pipet, transfer 10 ml of std. HCl soln.to a conical flask.

2- Add 1 - 2 drops of ph.ph. as an indicator.

3- Fill the burette with the prepared NaOH solution.



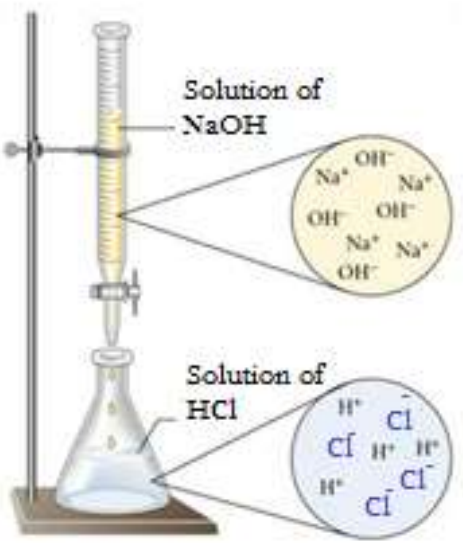
4- Add NaOH drop by drop into the conical flask until the color of the solution is faint pink.



5- The exact normality of NaOH solution is obtained from the following calculations.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

$$N_1 V_1 \underset{(NaOH)}{} = N_2 V_2 \underset{(HCl)}{}$$



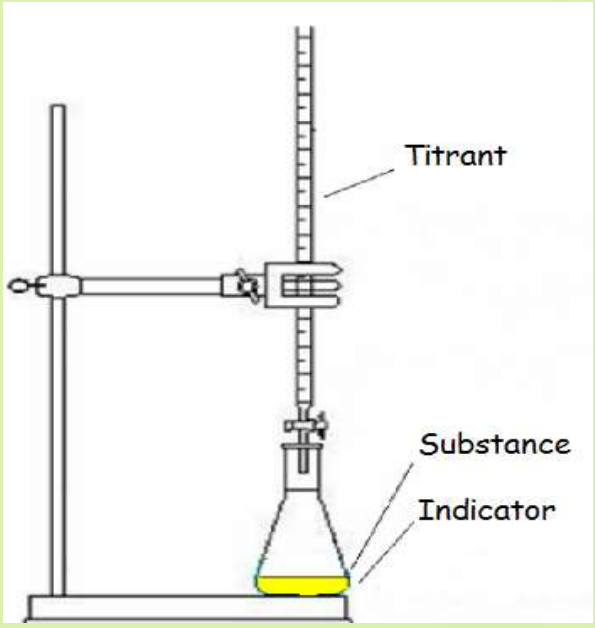
Volumetric Method of Analysis

Volumetric method:

It's one in which the analysis is completed by measuring the volume of a solution of known concentration needed to react completely with the substance being determined.

Titration:

It's a process for determining the amount of a substance by measurement of the quantity of a reagent (titrant) required to react completely with that subs..

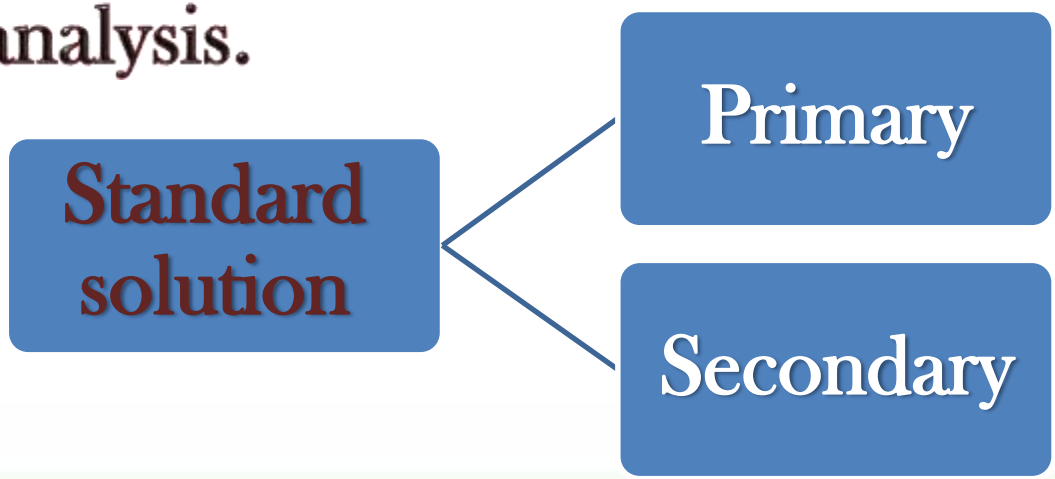


Standardization:

It's a process whereby the concentration of a std. solution is determined by titrating with a primary standard solution.

Standard solution:

It's a reagent of exactly known composition and concentration used in titrations and in many other chemical analysis.



Primary standard:

It's a highly purified chemical compound that serves as a reference material in titrations and in other analytical methods.

Requirements for a primary standard are the following:

1. It must be of the highest purity.
2. It should be stable and not attacked by atmosphere, (Atmospheric stability).
3. It should not be hygroscopic.
4. It should have high equivalence, (large molar mass), to minimize weighing errors.
5. It should be available and not too expensive.

Very few compounds meet or even approach these criteria, and only a limited number of primary – standard substances are available commercially. *As a consequence*,
less pure compounds must sometimes be used in place of a primary standard.
The purity of such a secondary standard must be established by careful analysis.

Secondary standard:

It is a compound whose purity has been determined by chemical analysis.
The secondary standard serves as the work - ing standard material for titrations and for many other analysis.

Indicator:

An indicator is a chemical compound that exhibits a change in color as a result of concentration changes occurring near the equivalence point.

Equivalence point:

It's the point in a titration when the amount of the added standard reagent is chemically equivalent to the amount of analyte.

End point:

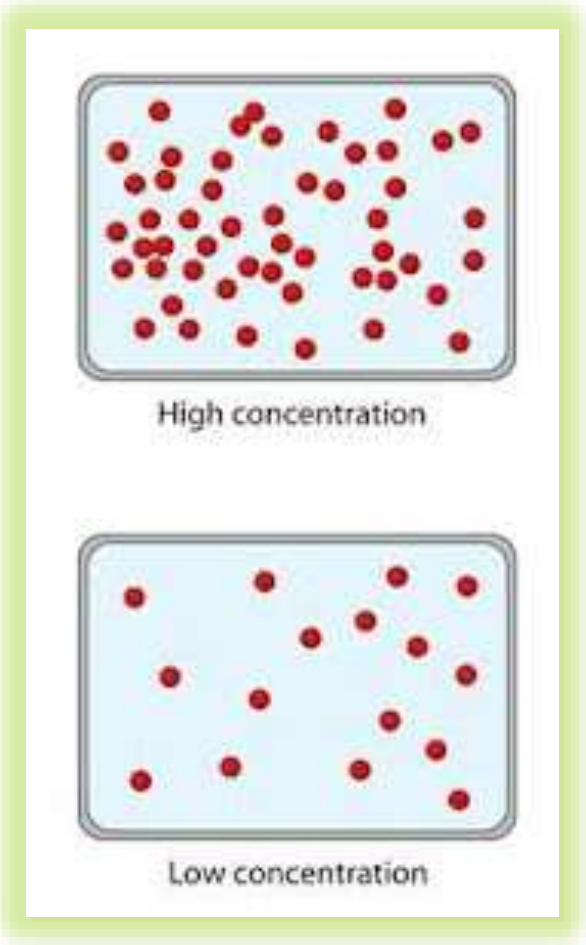
It is the point in a titration when a physical changes associated with the equivalence point can be observed.

Volumetric Calculations:

The concentration of a solution can be expressed in several ways:

Concentration of solution

- **Normality**
- **Molarity**
- **Formality**



1. Normality:

It is the number of equivalents of solute contained in one liter of solution.

Equivalent mass, *called equivalent weight in the older literature*, of an acid or base which participates in a neutralization reaction, is the mass that supplies or reacts with one mole of protons in a particular reaction.

***For example*,**

the equivalent mass of H_2SO_4 is one half of it's molar mass.

$$\text{Normality } (N) = \frac{\text{Number of equivalents of solute}}{\text{1 Liter of solution}}$$

$$\text{Number of equivalents of solute } (Eq.) = \frac{\text{Mass of solute } (g)}{\text{Equivalent mass of solute } (g/Eq)}$$

$$\text{Equivalent mass of solute } (g/Eq) = \frac{\text{Molar mass of solute } (g/mol)}{\text{Number of protons reacted } (Eq/mol)}$$

2. Molarity:

It is the number of gram molar mass or the number of moles of solute in one liter of solution.

$$\text{Molarity } (M) = \frac{\text{Number of moles of solute } (mol)}{\text{1 Liter of solution } (V_L)}$$

$$\text{Number of moles of solute } (mol) = \frac{\text{Mass of solute } (g)}{\text{Molar mass of solute } (g/mol)}$$

3. Formality:

It is the number of gram formula mass in one liter of solution.

$$\text{Formality } (F) = \frac{\text{Number of gram formula mass of solute}}{\text{Volume of solution in liter}}$$

$$\text{Number of gram formula mass of solute} = \frac{\text{Mass of solute } (g)}{\text{Gram formula mass of solute } (g/f)}$$

Volumetric methods of analysis can be divided in to four types:

- Acid – Base Titrations.
  (Neutralization Titrations).
- Oxidation –Reduction Titrations.
  (Redox Titrations).
- Complexometric Titrations.
- Precipitation Titrations.

Neutralization Titrations

Principles:

Neutralization titrations are widely used to determine the amounts of acids and bases. The std. solutions used in neutralization titrations are always strong acids or strong bases because these substances react more completely with an analyte than do weak acids & bases, and as a result, they produce sharper end points.

Weak acids & bases are never used as standard reagents because they react incompletely with analytes.

Acid/Base Indicators:

They are generally complex organic cpd.s of high molecular weight dissolved in water or other solvents where it's color depends on the pH of the medium.

An acid/base indicator , is a weak organic acid or a weak organic base whose undisso - ciated form differs in color from it's conjugate base or it's conjugate acid form.

For example,

the behavior of an acid-type indicator, HIn, is described by the equilibrium

$$HIn + H_2O \rightleftharpoons In^- + H_3O^+$$

Acid color *Base color*

The equilibrium for a base - type indicator, In, is

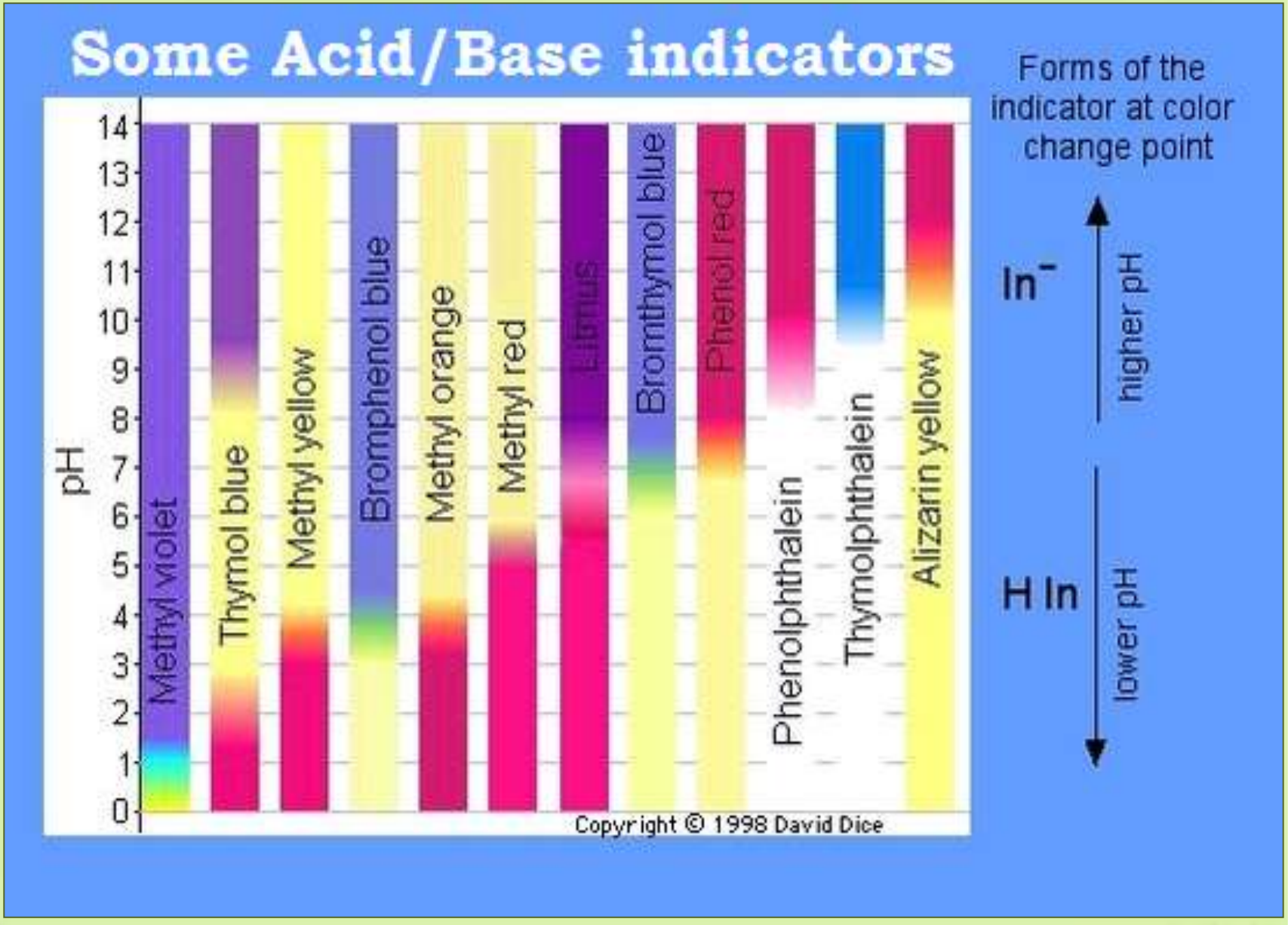
$$In + H_2O \rightleftharpoons InH^+ + OH^-$$

Base color — *Acid color*

Transition range of an indicator:

It's the pH range over which change in color of an indicator takes place.

Acid/Base indicator	Transition range	Color in Acidic medium	Color in Basic medium
Phenolphthalein (ph-ph)	8.3 - 10	Colorless	Pink
Methyl Orange (M.O)	3.1 – 4.4	Orange - Pink	Yellow
Methyl Red (M.R)	4.2 – 6.3	Red	Yellow


Some Acid/Base indicators
pH
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
Methyl violet
Thymol blue
Methyl yellow
Bromphenol blue
Methyl orange
Methyl red
Litmus
Bromthymol blue
Phenol red
Phenolphthalein
Thymolphthalein
Alizarin yellow
Forms of the indicator at color change point
In⁻
higher pH
HIn
lower pH
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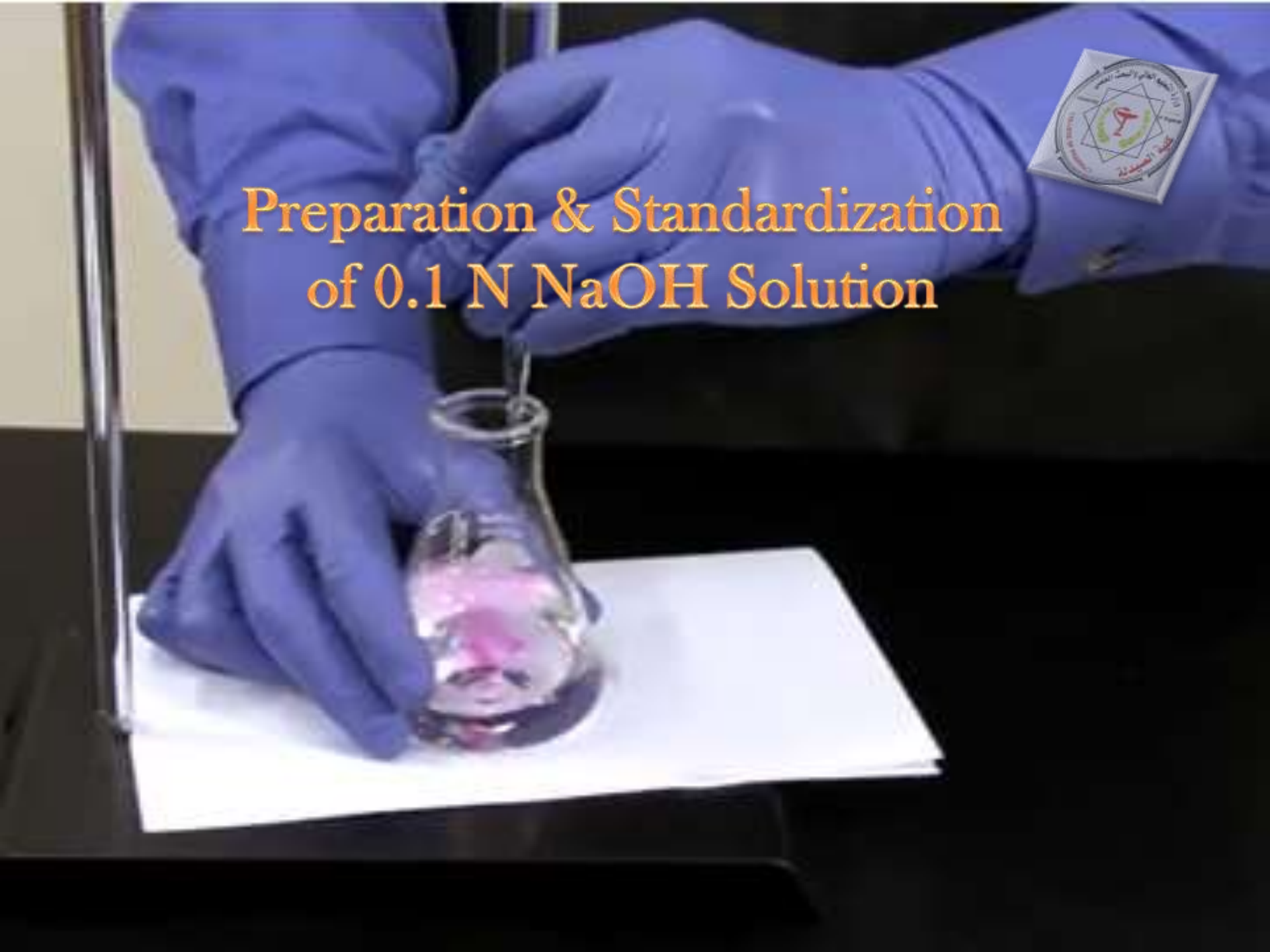
* A. H. Beckett, ***Practical Pharmaceutical Chemistry***, Quantitative Analysis, 1962.

* Douglas A. Skoog, Donald M. West , F. James Holler, Stanley R. Crouch, ***Fundamentals of Analytical Chemistry***, 9th edition , 2014.

* Vogel , Arthur , ***Vogel's Textbook of Quantitative Chemical Analysis*** , 6th edition.


Preparation & Standardization
of 0.1 N NaOH Solution

How could you prepare one liter of N/10 NaOH solution?

Knowing that the atomic masss of :

Na = 23 , O = 16 and H = 1.

$$N = \frac{\text{no. of equivalents}}{\text{Volume (L)}} = \frac{\text{Mass / Equivalent mass}}{\text{Volume (L)}}$$

$$= \frac{\text{Mass} * 1000}{\text{Eq. mass} * \text{V (ml)}}$$

$$0.1 = \frac{\text{Mass} * 1000}{40 * 1000}$$

Mass = 4 g of NaOH.

So, we should weigh 4 g of NaOH , dissolve it in water and make up to the mark of 1 liter volumetric flask.

Standardization of the prepared NaOH solution

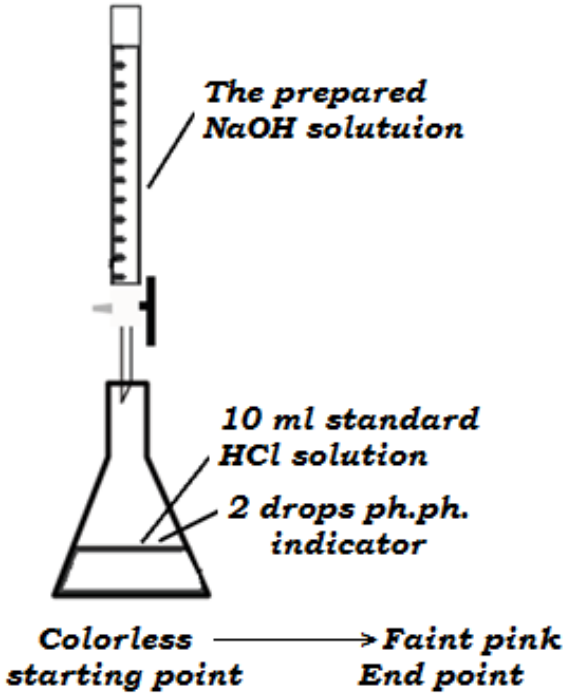
Solutions of NaOH are standardized by titration with std. acids of equivalent normality.

Method:

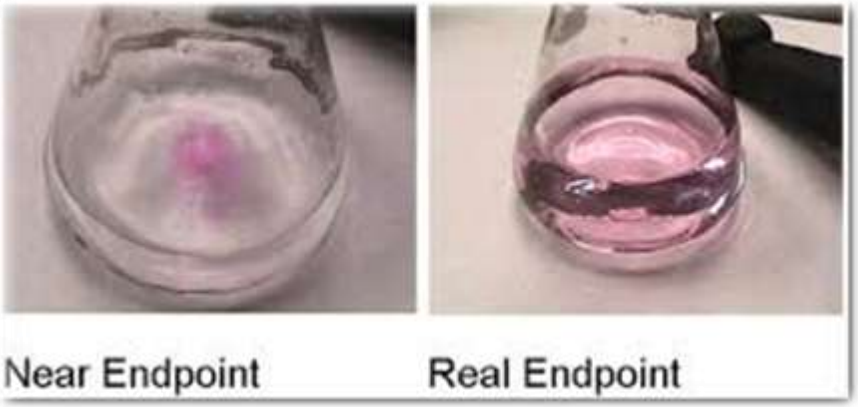
1- Using a bulb pipet, transfer 10 ml of std. HCl soln.to a conical flask.

2- Add 1 - 2 drops of ph.ph. as an indicator.

3- Fill the burette with the prepared NaOH solution.



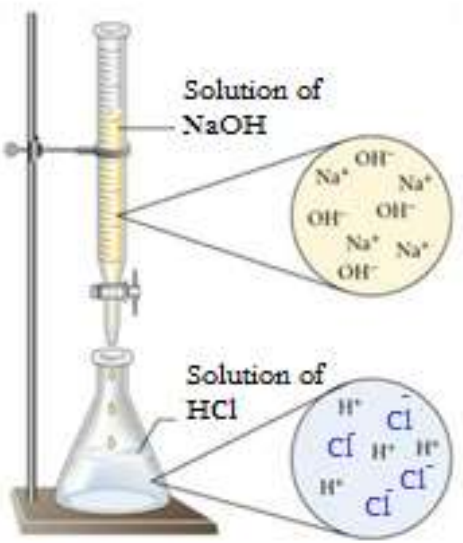
4- Add NaOH drop by drop into the conical flask until the color of the solution is faint pink.



5- The exact normality of NaOH solution is obtained from the following calculations.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

$$N_1 V_1 \underset{(NaOH)}{} = N_2 V_2 \underset{(HCl)}{}$$



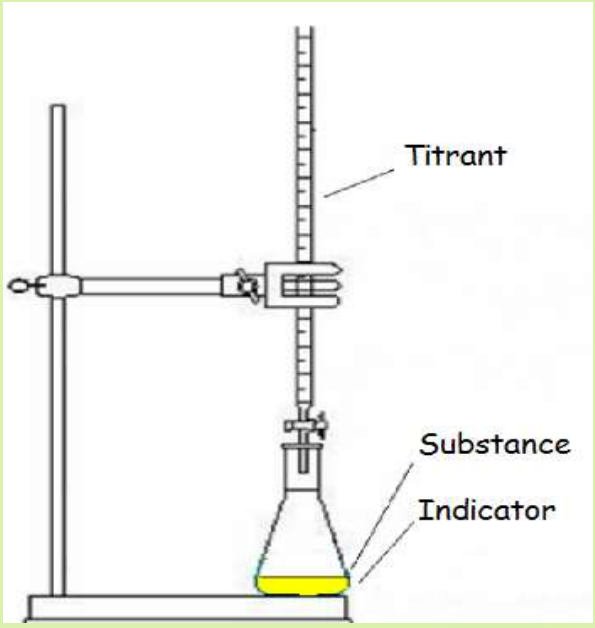
Volumetric Method of Analysis

Volumetric method:

It's one in which the analysis is completed by measuring the volume of a solution of known concentration needed to react completely with the substance being determined.

Titration:

It's a process for determining the amount of a substance by measurement of the quantity of a reagent (titrant) required to react completely with that subs..

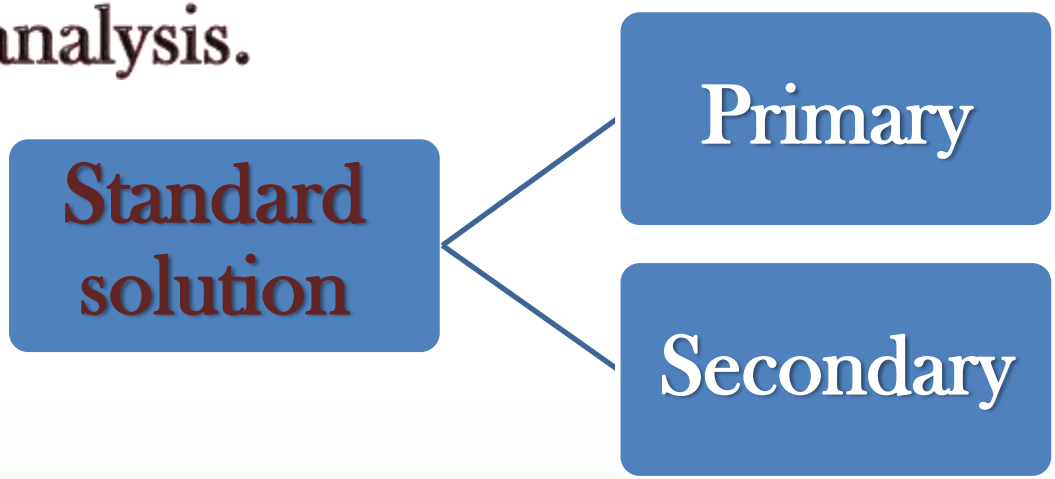


Standardization:

It's a process whereby the concentration of a std. solution is determined by titrating with a primary standard solution.

Standard solution:

It's a reagent of exactly known composition and concentration used in titrations and in many other chemical analysis.



Primary standard:

It's a highly purified chemical compound that serves as a reference material in titrations and in other analytical methods.

Requirements for a primary standard are the following:

1. It must be of the highest purity.
2. It should be stable and not attacked by atmosphere, (Atmospheric stability).
3. It should not be hygroscopic.
4. It should have high equivalence, (large molar mass), to minimize weighing errors.
5. It should be available and not too expensive.

Very few compounds meet or even approach these criteria, and only a limited number of primary – standard substances are available commercially. *As a consequence*,
less pure compounds must sometimes be used in place of a primary standard.
The purity of such a secondary standard must be established by careful analysis.

Secondary standard:

It is a compound whose purity has been determined by chemical analysis.
The secondary standard serves as the work - ing standard material for titrations and for many other analysis.

Indicator:

An indicator is a chemical compound that exhibits a change in color as a result of concentration changes occurring near the equivalence point.

Equivalence point:

It's the point in a titration when the amount of the added standard reagent is chemically equivalent to the amount of analyte.

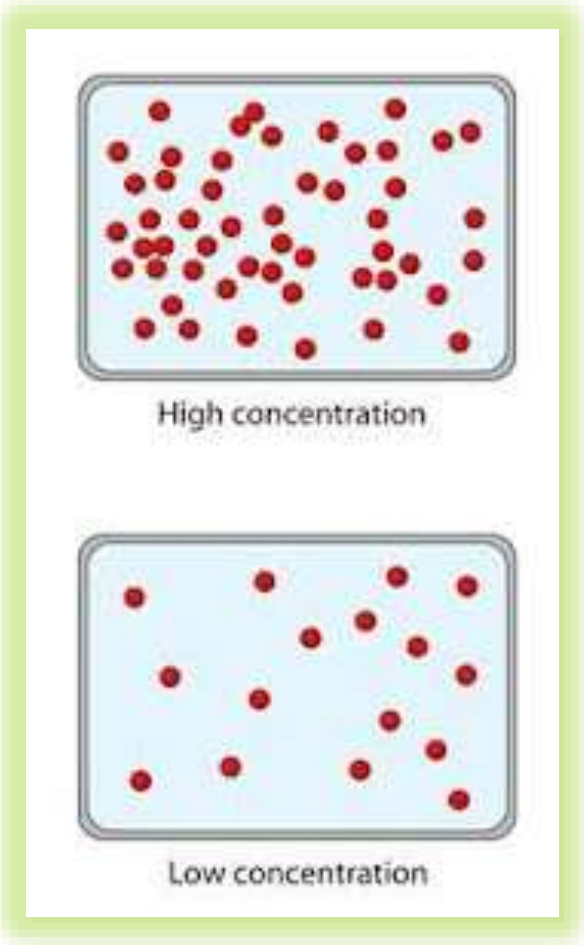
End point:

It is the point in a titration when a physical changes associated with the equivalence point can be observed.

Volumetric Calculations:

The concentration of a solution can be expressed in several ways:

- Concentration of solution
  - Normality
  - Molarity
  - Formality



1. Normality:

It is the number of equivalents of solute contained in one liter of solution.

Equivalent mass, *called equivalent weight in the older literature*, of an acid or base which participates in a neutralization reaction, is the mass that supplies or reacts with one mole of protons in a particular reaction.

***For example*,**

the equivalent mass of H_2SO_4 is one half of it's molar mass.

$$\text{Normality } (N) = \frac{\text{Number of equivalents of solute}}{\text{1 Liter of solution}}$$

$$\text{Number of equivalents of solute } (Eq.) = \frac{\text{Mass of solute } (g)}{\text{Equivalent mass of solute } (g/Eq)}$$

$$\text{Equivalent mass of solute } (g/Eq) = \frac{\text{Molar mass of solute } (g/mol)}{\text{Number of protons reacted } (Eq/mol)}$$

2. Molarity:

It is the number of gram molar mass or the number of moles of solute in one liter of solution.

$$\text{Molarity } (M) = \frac{\text{Number of moles of solute } (mol)}{\text{1 Liter of solution } (V_L)}$$

$$\text{Number of moles of solute } (mol) = \frac{\text{Mass of solute } (g)}{\text{Molar mass of solute } (g/mol)}$$

3. Formality:

It is the number of gram formula mass in one liter of solution.

$$\text{Formality } (F) = \frac{\text{Number of gram formula mass of solute}}{\text{Volume of solution in liter}}$$

$$\text{Number of gram formula mass of solute} = \frac{\text{Mass of solute } (g)}{\text{Gram formula mass of solute } (g/f)}$$

Volumetric methods of analysis can be divided in to four types:

- Acid – Base Titrations.
  (Neutralization Titrations).
- Oxidation –Reduction Titrations.
  (Redox Titrations).
- Complexometric Titrations.
- Precipitation Titrations.

Neutralization Titrations

Principles:

Neutralization titrations are widely used to determine the amounts of acids and bases. The std. solutions used in neutralization titrations are always strong acids or strong bases because these substances react more completely with an analyte than do weak acids & bases, and as a result, they produce sharper end points.

Weak acids & bases are never used as standard reagents because they react incompletely with analytes.

Acid/Base Indicators:

They are generally complex organic cpd.s of high molecular weight dissolved in water or other solvents where it's color depends on the pH of the medium.

An acid/base indicator , is a weak organic acid or a weak organic base whose undisso - ciated form differs in color from it's conjugate base or it's conjugate acid form.

For example,

the behavior of an acid-type indicator, HIn, is described by the equilibrium

$$HIn + H_2O \rightleftharpoons In^- + H_3O^+$$

Acid color *Base color*

The equilibrium for a base - type indicator, In, is

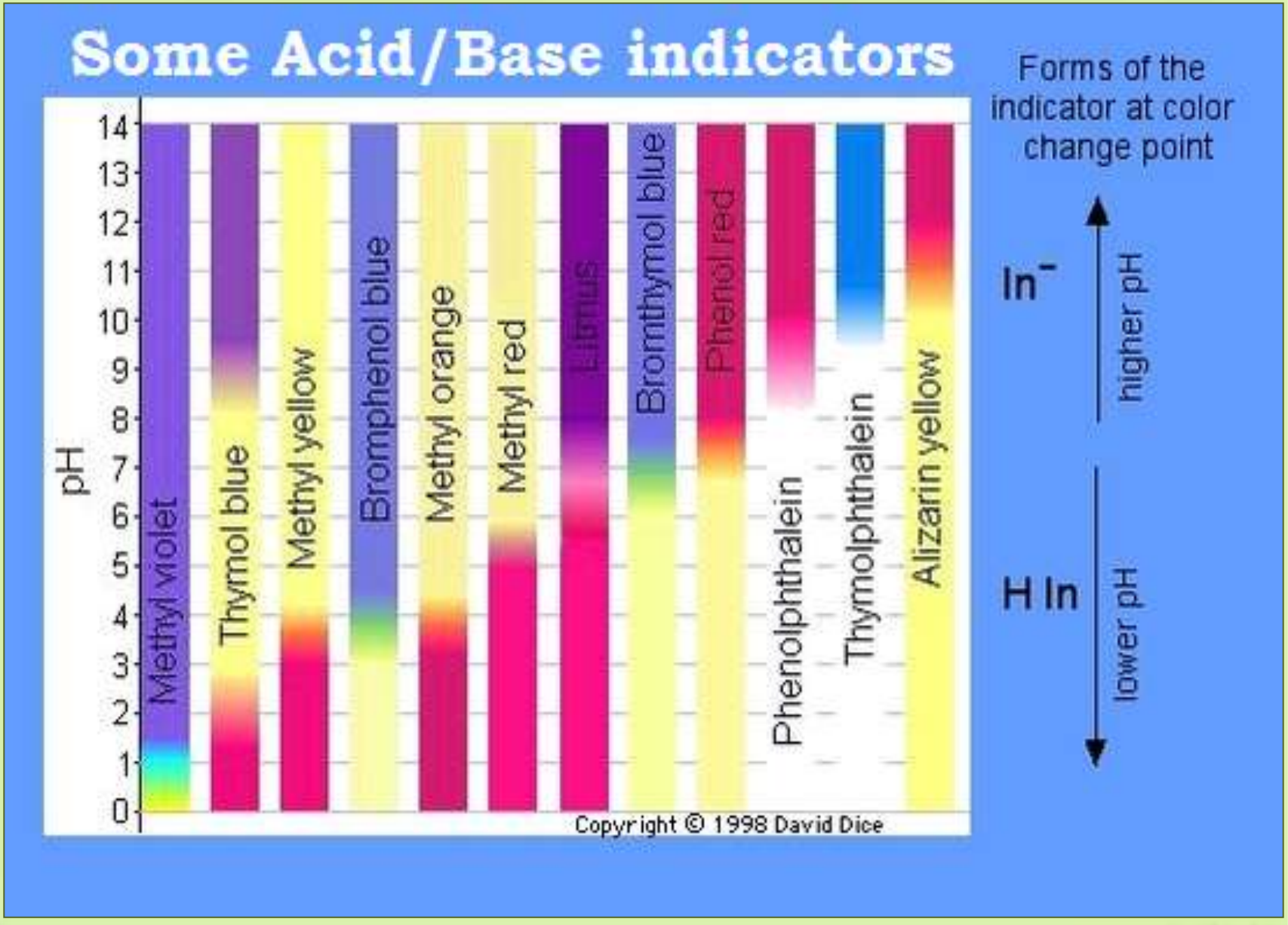
$$In + H_2O \rightleftharpoons InH^+ + OH^-$$

Base color — *Acid color*

Transition range of an indicator:

It's the pH range over which change in color of an indicator takes place.

Acid/Base indicator	Transition range	Color in Acidic medium	Color in Basic medium
Phenolphthalein (ph-ph)	8.3 - 10	Colorless	Pink
Methyl Orange (M.O)	3.1 – 4.4	Orange - Pink	Yellow
Methyl Red (M.R)	4.2 – 6.3	Red	Yellow


Some Acid/Base indicators
pH
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
Methyl violet
Thymol blue
Methyl yellow
Bromphenol blue
Methyl orange
Methyl red
Litmus
Bromthymol blue
Phenol red
Phenolphthalein
Thymolphthalein
Alizarin yellow
Forms of the indicator at color change point
In⁻
higher pH
HIn
lower pH
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REFERENCES

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* A. H. Beckett, ***Practical Pharmaceutical Chemistry***, Quantitative Analysis, 1962.

* Douglas A. Skoog, Donald M. West , F. James Holler, Stanley R. Crouch, ***Fundamentals of Analytical Chemistry***, 9th edition , 2014.

* Vogel , Arthur , ***Vogel's Textbook of Quantitative Chemical Analysis*** , 6th edition.