2015 - 2016

Volumetric Method of Analysis

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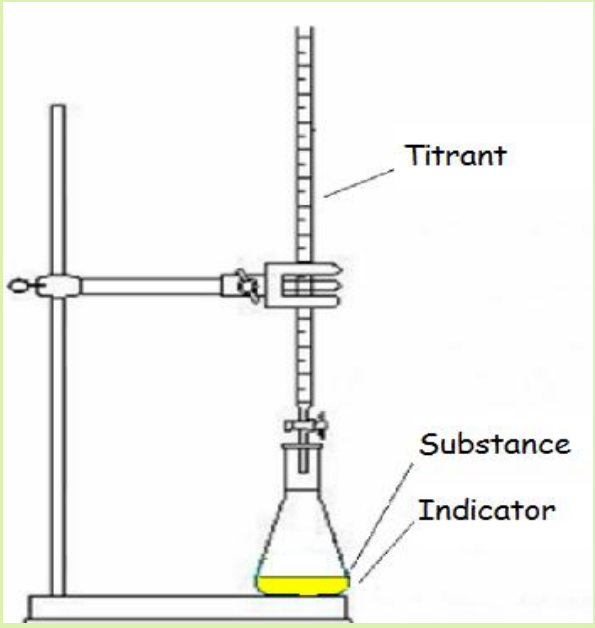
Volumetric method:

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..

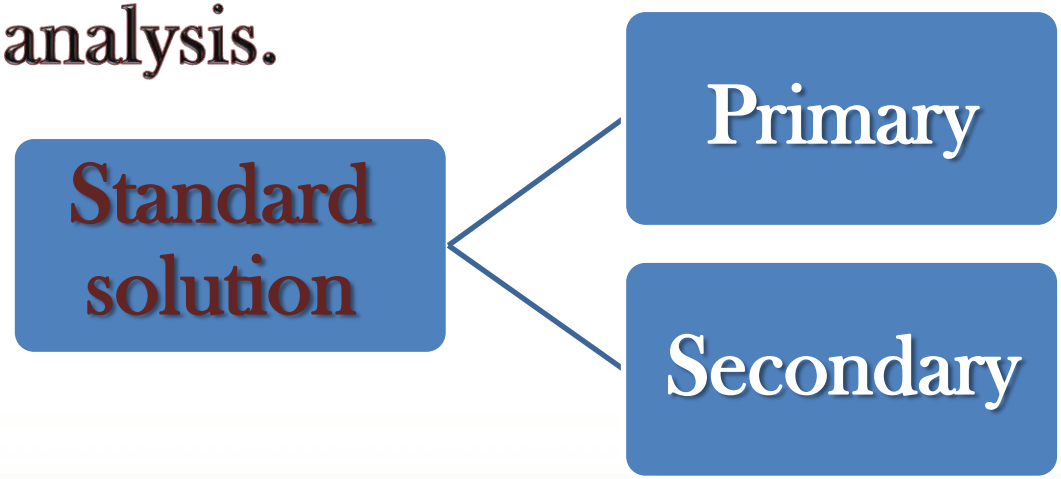


Standardization:

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.

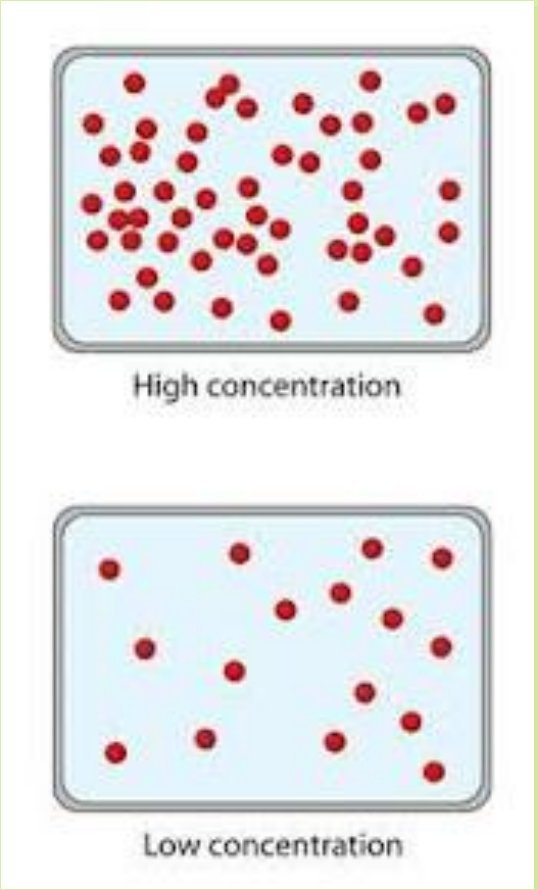
End point:

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

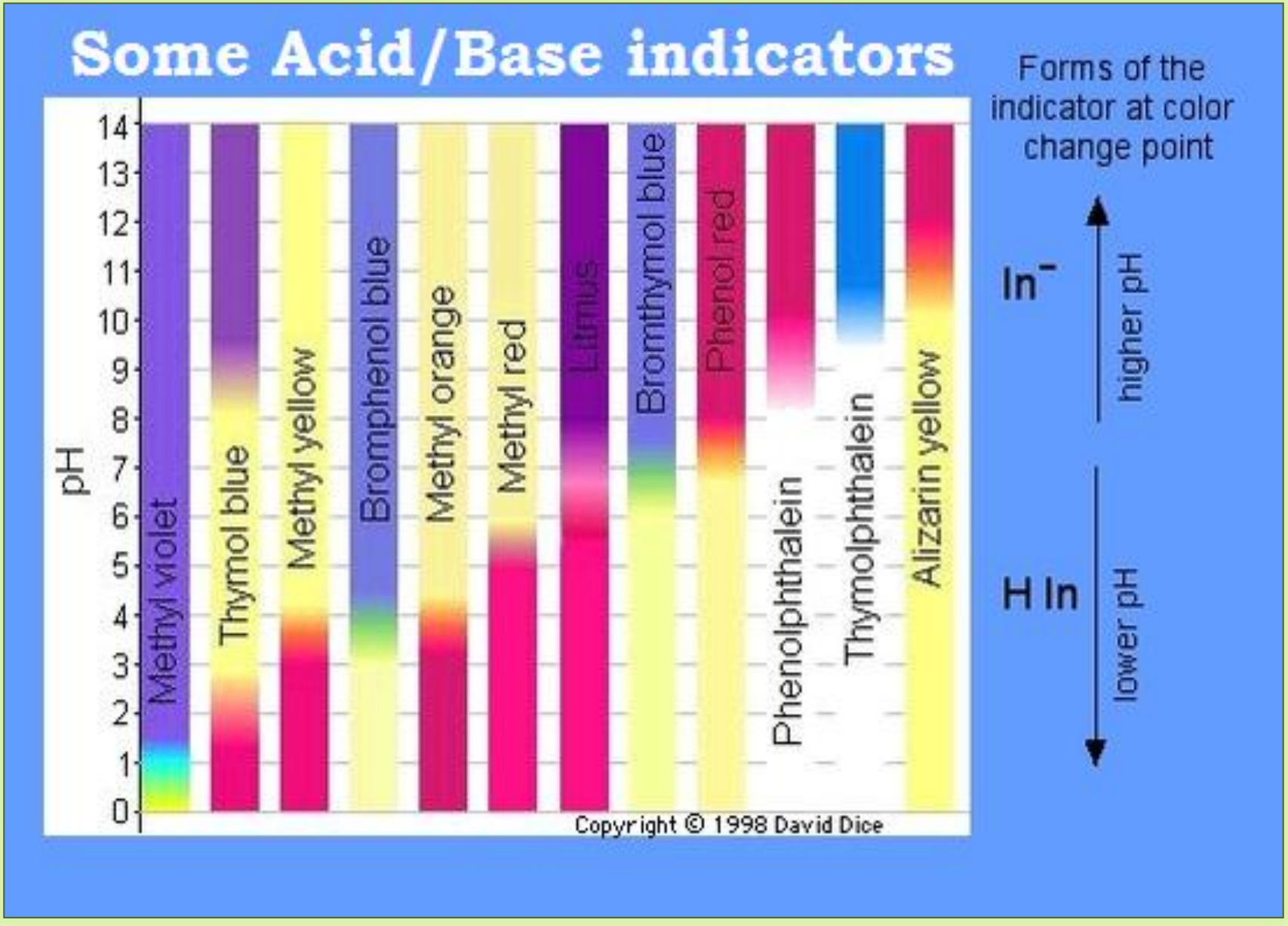
$$In + H_2O \rightleftharpoons InH^+ + OH^-$$

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
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Forms of the indicator at color change point
In⁻
higher pH
HIn
lower pH

REFERENCES

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