

## Week 3 Volumetric Analysis

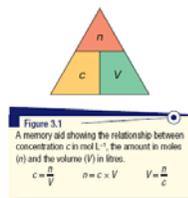
### Concentration

Is a measure of the amount of solute in a specified volume of solvent. Is often expressed as **molar concentration** referred to as **molarity**. Defined as: the amount of solute in mol, dissolved in 1 L of solution.

$$\text{concentration} = \frac{\text{amount of solute, in mol}}{\text{volume of solution, in L}}$$

$$\text{or } c = \frac{n}{V}$$

Unit = mol L<sup>-1</sup>, which is given the symbol **M**



e.g. 1.00 L of a 2.00 M solution of NaCl contains 2.00 mol of dissolved NaCl. Therefore, its concentration is 2.00 molar (M).

Another measure of concentration is mass per unit volume e.g. grams of solute dissolved in each litre of solution (g L<sup>-1</sup>).

To convert mol L<sup>-1</sup> to g L<sup>-1</sup>, multiply the molarity by the molar mass of the solute e.g.

a 2.00 M NaCl solution has a concentration of  
2.00 mol L<sup>-1</sup> X (23.0 + 35.5) g mol<sup>-1</sup> = 117 g L<sup>-1</sup>

### Standard Solutions

Are solutions with accurately known concentrations.

Substances that are so pure in that the amount in mol can be calculated accurately from their mass are called **primary standards**.

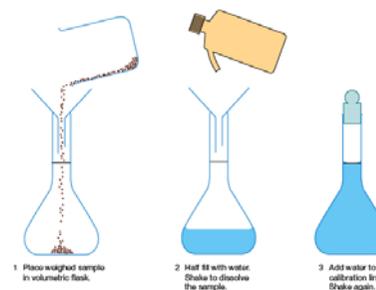
A primary standard should:

- Be readily obtainable in pure form
- Have a known formula
- Be easy to store without deteriorating or reacting with the atmosphere
- Have a high molar mass to minimise the effect of errors in weighing
- Be inexpensive

Examples of primary standards are:

- Bases  
anhydrous sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), Sodium borate (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>·10H<sub>2</sub>O)
- Acids  
Hydrated oxalic acid (H<sub>2</sub>C<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O) and potassium hydrogen phthalate (KH(C<sub>8</sub>H<sub>4</sub>O<sub>4</sub>))

Steps in preparing a standard solution. A known mass of solute is used to make a known volume of solution (in volumetric flask).

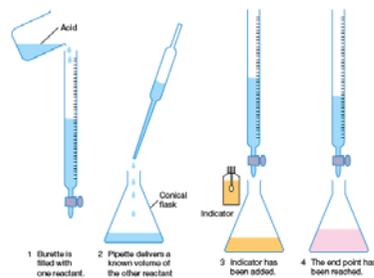


## Volumetric Analysis

Involves reacting a measured volume of a standard solution with a measured volume of the solution of unknown concentration.

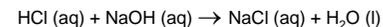
Solutions are reacted completely in the mole ratio indicated by the stoichiometric equation. This is known as performing a **titration**.

### Steps in an acid-base titration

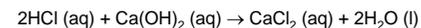


## Equivalence point

When solutions have been mixed in the mole ratio shown by the reaction equation e.g.



At the equivalence point:  $n(\text{HCl}) = n(\text{NaOH}) = 1$



At the equivalence point:  $n(\text{HCl}) : n(\text{Ca(OH)}_2) = 2:1$   
 $n(\text{HCl}) = 2n(\text{Ca(OH)}_2)$

## Identifying the equivalence point

- Solutions often colorless in acid-base titrations
- Indicator must be added
- Indicator chosen such that color change occurs at the equivalence point
- Titrations are repeated several times and the results averaged

### How accurate are our measurements?

Depends on the calibration of the equipment used

Typical uncertainties associated with volumetric analysis are:

- 20 mL pipette  $\pm 0.05$  mL
- Burette  $\pm 0.02$  mL for each reading
- 250.0 mL volumetric flask  $\pm 0.3$  mL

It is usual to repeat titrations until 3 concordant readings are obtained (readings differ by a maximum of 0.10 mL from highest to lowest – one drop from burette is approx. 0.05 mL).

TABLE 3.1 Sample titration results

Titration number	1	2	3	4	5
Final burette reading (mL)	20.20	40.82	20.64	41.78	21.86
Initial burette reading (mL)	0.00	21.00	1.00	22.00	2.00
Titre (mL)	20.20	19.82	19.64	19.78	19.86

- First reading was obviously a test to give an idea of where the end point will be
- The 3 titres in bold are concordant
- Difference between highest and lowest is  $19.86 - 19.78 = 0.08$  mL
- The mean titre is

$$\frac{19.82 + 19.78 + 19.86}{3} = 19.82$$

### Concentration

11. Calculate the molarity of :
- b. 100.0 mL solution containing 0.63 g of anhydrous sodium carbonate  
 $\text{Na}_2\text{CO}_3$  0.059 M
12. What mass of solute is required to prepare the following standard solutions?
- a. 250 mL of 0.500 M sodium oxalate  $\text{Na}_2\text{C}_2\text{O}_4$  16.7 g
14. Convert the following molar concentrations to units of  $\text{g L}^{-1}$ :
- i. 0.0024 M NaCl solution 0.14  $\text{g L}^{-1}$   
ii.  $6.3 \times 10^{-5}$  M  $\text{Pb}(\text{NO}_3)_2$  solution 0.021  $\text{g L}^{-1}$
17. To what volume of water must 10 mL of 8.0 M HCl be added in order to prepare a 0.50 M HCl solution? 0.15 L

19. Schools normally purchase concentrated (14 M) nitric acid and then dilute it for use. What volume is required to prepare 2.0 L of 0.15 M acid? 21 mL

### Volumetric analysis

23. A student is to perform an analysis of sodium hydroxide solution by titrating it with standard hydrochloric acid, as shown in Fig. 3.5. Before beginning, the student rinses the glassware that is to be used in the analysis. However, the student does not wish to wait until the glassware has dried before using it. For each of the following apparatus, a, b, and c, state if it should be:
- i. Rinsed with de-ionised water only  
ii. Rinsed with sodium hydroxide solution only  
iii. Rinsed with hydrochloric acid only
- a. Pipette  
b. Burette  
c. Conical flask
- a. ii  
b. iii  
c. i

#### summary

3.1

- Concentration is a measure of the amount of solute dissolved in a specified volume of solution.
- Molarity (M) is one measure of concentration. It is the amount, in mol, of solute dissolved in one litre of solution. The concentration,  $c$  mol  $\text{L}^{-1}$ , is given by  $c = \frac{n}{V}$  where  $n$  = number of mole and  $V$  = volume in litres.
- A primary standard has a high level of purity enabling the number of moles to be determined from its mass.
- A standard solution has a known concentration and is prepared by dissolving a known mass of a primary standard in an accurately known volume of solution.

#### summary

3.2

- In a titration, a measured volume of a standard solution is reacted with a measured volume of the solution whose concentration is to be determined.
- The equivalence point of a reaction occurs when the reagents have been mixed in the mole ratio shown by the reaction equation.
- An indicator may be used to identify the equivalence point of a reaction.
- The stage at which an indicator changes colour is called the end point.
- For accurate analysis the indicator end point must closely match the reaction equivalence point.
- Concordant titres vary within narrowly specified limits. Three concordant titres are usually obtained during a titration.