CHEMISTRY

LEAVING CERTIFICATE
HIGHER LEVEL

VOLUMETRIC ANALYSIS/
ACID & BASE
TITRATION

THE DUBLIN ACADEMY OF EDUCATION

Volumetric Analysis

Volumetric analysis is analysis using **volumes of solutions** and **their concentrations.**

Solutions

A **solution** is a **uniform mixture** of two substances.

Solute + Solvent = **Solution**

Concentrated solutions have large amounts of solute per unit solution

Dilute solutions have small amounts of solute per unit solution

Ways of expressing concentration

- **Molarity** (mols per litre) - M
  - e.g 0.5 M = 0.5 mols/L
- **g / dm^3** (g/L)
- **% w / v** (weight per volume)
- **% v / v** (volume per volume)
- **% w / w** (weight per weight)
- **p.p.m** (mg.L^{-1}) (Parts Per Million)

Molarity

- **moles per litre:** mols / dm^3, mol.dm^{-3} or mol / L
- A **1 molar (1M) solution** contains **1 mole of solute per 1 litre** of solution.

This is the most common way of expressing concentration of the solutions in the lab.

What is the molarity of these solutions?

(i) 8 g NaOH in 200 cm^3 of solution

(ii) 5 g $KMnO_4$ in 600 cm^3 of solution

(iii) 18 g $K_2Cr_2O_7$ in 400 cm^3 of solution

(iv) 20 g HNO_3 in 100 cm^3 of solution

(v) 25 g $CuSO_4.5H_2O$ in 50 cm^3 of sol^n

(vi) 40 g of NaOH in 1600 cm^3 of sol^n

2. Grams per litre (g /dm^3, g.dm^{-3} or g/L)

- **grams of solute per litre of solution**

Concentration = Molarity * Molar Mass

Eg: What is the concentration of 0.2 M NaCl in g / dm^3 ?

M_r NaCl = 23 + 35.5 = 58.5 g/mol

Conc. = Molarity x M_r

conc = 0.2 mol/dm^3 x 58.5 g / mol

= **11.7 g/dm^3 (g.dm^{-3})**

Try a few:

(i) 0.1 M NaOH

(ii) 0.5 M H_2SO_4

(ii) 0.05 M $KMnO_4$

(iv) 10 M NH_4OH

(v) 2.5 M HNO_3

(vi) 0.0125 M HCl

Moles in solutions:

$$\text{Moles} = \frac{\text{Volume (cm}^3\text{) x conc (moles/L)}}{1000}$$

Eg: How many moles of nitric acid (HNO_3) are contained in 50 cm^3 of 0.05M nitric acid.

Effect of dilutions on concentration

Eg. If 20cm^3 of a 0.1M NaOH solution is diluted to a volume of 250cm^3 with water, what is the concentration of the diluted solution.

Formula = V_{dil} x M_{dil} = V_{conc} x M_{conc}

Try a few:

(i) If 10cm^3 of a 2 M sol^n of nitric acid is diluted to a volume of 2 L, what is the conc^n of the diluted acid?

(ii) If 12cm^3 of a 0.1 M sol^n of sodium hydroxide is diluted to a volume of 500cm^3, what is the conc^n of the diluted base?

(iii) What volume of a 1M solution of sodium hydroxide is required to make up 250 cm^3 of a 0.05 M NaOH sol^n?

(iv) What volume of a 0.5 M solution of sulfuric acid is required to make up 250 cm^3 of a 0.1 M H_2SO_4 sol^n

16.2 Standard Solutions

A Primary Standard is a substance which can be obtained in a stable, pure and soluble solid form, so that it can be accurately weighed and dissolved to give a solution of known concentration.

A **Standard Solution** is a solution where the concentration is accurately known.

It is prepared by dissolving a precisely known amount of solute (primary std) in a suitable solvent (de-ionised water)

Examples of Primary Standards

- **anhydrous** Na_2CO_3
- NaCl
- $K_2Cr_2O_7$

We cannot use:

- $KMnO_4$as Primary Standard - it cannot be obtained pure and it is effected by sunlight.
- Iodine [I_2] because it sublimes and it is not soluble in water.
- KOH or NaOH they absorb CO_2 and moisture from the air.

Secondary Standard A solution whose concentration is found by titrating it against a primary standard solution.

Standardise means to find the concentration of a solution by titrating it against a solution of known concentration.

16. 3 Titrations

A titration is a laboratory procedure where a measured volume of one solution (of known concentration) is added to a known volume of another solution (where the concentration is not known) until the reaction is complete and the concentration can be calculated.

To make a standard solution:

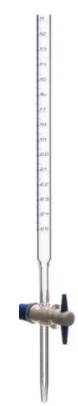
- Accurately weigh primary standard (solute) on a **clock glass** and transfer into a beaker and **dissolve** in a small amount of deionised water.
- Transfer to a **volumetric flask** using a **funnel**.
- Add **rinsings** from beaker into the volumetric flask.
- Using a water bottle add more distilled water until you are 1cm from the calibration mark.
- Add dropwise until the **bottom of the meniscus is on the line**, reading it at **eye-level.**
- **Stopper and invert** to ensure a homogenous solution.

To use a Pipette

- Rinse with **deionised water** and with the **solution** it will contain.
- Fill using **pipette filler** until bottom of the meniscus is on the graduation mark, reading at eye level.
- Empty the pipette into a conical flask, touching the tip of the pipette against the side of the flask. (There will be a drop left in the tip of the pipette, but it is calibrated to allow for this).

To use a Burette

- Rinse with **deionised water** and then with the **solution** it is going to contain.
- **Clamp vertically**
- **Fill burette above the 0 cm^3 mark** using **a funnel and remove it** *(as drops may fall from it or it may dip into the liquid giving a false level)*.
- Run the solution through the tip to **remove the air bubble.**
- Read the volume from the **bottom of meniscus** at **eye level** – Initial Volume.



*$KMnO_4$ - read from the top of the meniscus.

*Do not put NaOH in burette it may react with glass of burette or block tap

To use a Conical Flask

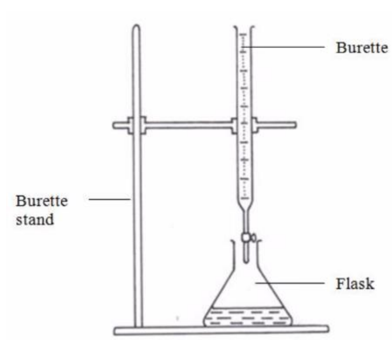
- Rinse out with **deionised water only** *(do not rinse with the solution it will contain)*
- Place on white tile - *to see colour change of indicator more easily*.
- Mix continuously by swirling
- Wash down drops on the side of the flask with deionised water if required.
  (This won't affect amount of reactant in flask or change the result as there is no change in amount of solute present)

To use a Volumetric Flask

Long thin neck to make it accurate.

- Fill, using a **funnel,** until nearly at calibration line, add final few cm^3 slowly using a dropper / water bottle.
- Read from **bottom of meniscus at eye level.**
- 20 times to ensure a homogeneous solution.

<u>To carry out a titration</u>



- Use the correct indicator for the type of titration (covered in Booklet 6.1)
- Only 3 - 4 drops of indicator should be used as they are weak acid/bases themselves so can affect the result.
- One rough and two accurate titres with the two accurate titres being within 0.1 cm^3 of each other.
- Use the **average the two accurate titres. (**Often in the LC you will just be given one value to use.)
- Mix well by swirling the conical flask, careful not to spill any mixture.
- Add the solution from burette **drop by drop** near the **end point.**
- Endpoint is identified when a readily identifiable **colour change** occurs.
- Rinse down the side of the conical flask using **de-ionised water** as this will not affect the result.

16.4 Titration Calculations:

Always identify the standard solution, the solution you know the concentration of, to allow for calculations that follow:

$$n = c \times \frac{V}{1000}$$

Unit: Mols/L

NaOH + HCl → NaCl + H_2O

1. 50.0 cm^3 of 0.10 M sodium hydroxide (NaOH) solution is titrated with hydrochloric acid (HCl) of unknown concentration. It requires 40.0 cm^3 of the HCl solution to completely neutralize the NaOH. Find
   (i) The average number of moles of NaOH used up in the titration.
   (ii) The number of moles of HCl neutralised in the titration.
   (iii) Find the concentration of the hydrochloric acid in moles / L (0.125M)

2. A 0.20 M solution of HCl is used to titrate 25.0 cm^3 of NaOH solution of unknown concentration. The volume of HCl required to reach the endpoint is 35.0 cm^3.
   (i) The average number of moles of HCl used up in the titration.
   (ii) The number of moles of NaOH neutralised in the titration.
   (iii) Find the concentration of the NaOH solution. (0.28M)

$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$

3. During a titration, 25.0 cm^3 of 0.05 M ethanoic acid (CH_3COOH) is neutralized by 22.5 cm^3 of sodium hydroxide (NaOH) solution.
   (i) The average number of moles of ethanoic acid (CH_3COOH) used up in the titration.
   (ii) The number of moles of NaOH neutralised in the titration.
   (iii) Find the concentration of the NaOH in moles / L (0.0556mol/L)

4. In a titration, 25.0 cm^3 of 0.10 M ethanoic acid (CH_3COOH) is titrated with sodium hydroxide (NaOH). The titration requires 32.5 cm^3 of NaOH to reach the endpoint.
   (i) Calculate the number of moles of ethanoic acid in 25.0 cm^3 of 0.10 M solution.
   (ii) Calculate the moles of NaOH that reacted with ethanoic acid.
   (iii) Calculate the concentration of NaOH in mol/L. (0.0769 mol/L)

LC Practice Questions:

2024:

1. A student carried out an experiment to determine the concentration of ethanoic acid in vinegar. A 10.0 cm^3 portion of the vinegar was first diluted to exactly 50.0 cm^3. The diluted vinegar solution was titrated against a 0.09 M sodium hydroxide solution in a conical flask. On average, 12.4 cm^3 of the *diluted* vinegar were required to neutralise 25.0 cm^3 of the **NaOH** solution.

The titration reaction is described by the following balanced chemical equation:

$$CH_3COOH + NaOH \rightarrow CH_3COONa + H_2O$$

(*a*) (*i*) Name the piece of apparatus used to measure accurately the 10.0 cm^3 portion of the original vinegar.

(*ii*) Describe how this piece of apparatus was rinsed before use.

(*iii*) Describe a suitable method to dilute the 10.0 cm^3 portion of the original vinegar to exactly 50.0 cm^3. (15)

(*b*) (*i*) Calculate the volume of *undiluted* vinegar which would be required to neutralise 25.0 cm^3 of the 0.09 M **NaOH** solution.

(*ii*) Outline an advantage of diluting the vinegar before carrying out the titration. (9)

(*c*) (*i*) Name a suitable indicator for this titration.

(*ii*) State the colour change observed at the end point. (9)

(*d*) (*i*) Calculate the number of moles of sodium hydroxide in each 25.0 cm^3 portion.

(*ii*) Calculate the number of moles of ethanoic acid in each cm^3 of the *diluted* vinegar.

(*iii*) Calculate the concentration of ethanoic acid in the original vinegar in moles per litre.

(*iv*) Calculate the concentration of ethanoic acid in the original vinegar in % (w/v). (17)

2020 Q1:

Washing soda is a cheap, household chemical used for laundry, removing grease and softening water. Washing soda crystals are hydrated sodium carbonate (**$Na_2CO_3.xH_2O$**). The crystals effloresce (lose some water of crystallisation) in dry air becoming powdery in the process.

To determine the average value of ***x*** in the formula **$Na_2CO_3.xH_2O$** for a sample of washing soda, 3.46 g of the crystals were dissolved in deionised water and made up to exactly 250 cm^3 of solution. 25.0 cm^3 volumes of this solution were pipetted into a conical flask and titrated with a previously standardised hydrochloric acid solution using a suitable indicator.

The balanced equation for the titration reaction is:

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

(*a*) Identify a primary standard that could have been used to standardise the hydrochloric acid solution for this analysis. (5)

(*b*) Describe how the 250 cm^3 solution of washing soda was prepared starting with 3.46 g of washing soda measured out accurately on a weighing boat. (12)

(*c*) (*i*) Name a suitable indicator for this titration.

(*ii*) Justify your choice of indicator.

(*iii*) Using this indicator what colour change was observed in the conical flask at the end point? (12)

(*d*) On average 21.5 cm^3 of 0.12 M hydrochloric acid solution were required to completely neutralise 25.0 cm^3 of the washing soda solution.

Find by calculation

(*i*) the average number of moles of **HCl** used up in a titration,

(*ii*) the number of moles of **Na_2CO_3** neutralised in each titration,

(*iii*) the number of moles of **Na_2CO_3** in 250 cm^3 of the washing soda solution,

(*iv*) the mass of **Na_2CO_3** in 250 cm^3 of the washing soda solution,

(*v*) the mass of water of crystallisation and hence the number of moles of water in 3.46 g of the crystals,

(*vi*) the ratio of moles of water of crystallisation to moles of **Na_2CO_3** in the crystals and hence the value of ***x*** to the nearest whole number. (21)