Volumetric analysis

Acid-base titrations and indicators, preparing a standard solution, titration calculations, and measurement uncertainty.

Acid–base titration

In a **titration**, a solution of known concentration (usually in the burette) is added to a measured volume of another (in a conical flask) until the **end point**, shown by an indicator, is reached.

Steps for a calculation:

1. Write the balanced equation.
2. Find the moles of the solution whose volume and concentration are known: $n = c \times V$ (with V in dm^3).
3. Use the mole ratio to find the moles of the other reactant.
4. Find the concentration: $c = n/V$.
5. If needed, convert to g dm^{-3} by multiplying by the molar mass.

> **TRY IT**
>
> 25.0 cm^3 of sodium hydroxide neutralises 21.40 cm^3 of 0.100 mol dm^{-3} hydrochloric acid. Find the concentration of the sodium hydroxide.

$NaOH + HCl \rightarrow NaCl + H_2O$. $n(HCl) = 0.100 \times 0.02140 = 2.14 \times 10^{-3}$ mol. The ratio is 1 : 1, so $n(NaOH) = 2.14 \times 10^{-3}$ mol in 25.0 cm^3. $c = 2.14 \times 10^{-3}/0.0250 = 0.0856$ mol dm^{-3}.

Indicators

Indicator	Colour in acid	Colour in alkali	Use
Methyl orange	Red	Yellow	Strong acid with weak base
Phenolphthalein	Colourless	Pink	Strong base with weak acid

For strong acid with strong base either works. Use a few drops only, because the indicator is itself a weak acid and changes the result if too much is added.

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Core Practical 3: concentration of hydrochloric acid

1. Fill a clean burette with the standard solution (rinsing first with the solution), and remove any air bubbles in the tip.
2. Use a pipette to transfer 25.0 cm^3 of the other solution to a conical flask and add the indicator.
3. Run in the solution slowly with swirling. Do one **rough** titration, then repeat accurately, adding drop by drop near the end point.
4. Repeat until **concordant** results (within 0.10 cm^3) are obtained, and use their mean.

> **NOTE**
>
> Read the burette at eye level, to the bottom of the meniscus, to ±0.05 cm^3. Rinse the pipette with the solution it will measure and the conical flask with deionised water only.

Core Practical 4: a standard solution

A **standard solution** has an accurately known concentration. To prepare it from a solid acid:

1. Weigh the solid accurately by difference: record the mass of a weighing bottle, add the solid, record again, then tip the solid into a beaker and reweigh the bottle.
2. Dissolve it in a little deionised water in a beaker, stirring.
3. Pour it into a volumetric flask through a funnel, rinsing the beaker and funnel into the flask.
4. Make up to the mark with deionised water (the bottom of the meniscus on the line), stopper and **invert** to mix.

The solution is then used to find the concentration of sodium hydroxide, as in the titration above.

A **primary standard** is stable, pure, has a high molar mass and is not hygroscopic. Hydrated ethanedioic acid and anhydrous sodium carbonate are used.

Water of crystallisation and other calculations

Titration data can be used to find the M_r of an acid, the percentage purity of a sample, the concentration in g dm^{-3} of a solution, or the number of moles of water of crystallisation, x, in a hydrated salt.

> **TRY IT**
>
> 1.43 g of hydrated sodium carbonate $Na_2CO_3 \cdot xH_2O$ is dissolved in water and made up to 250 cm^3. 25.0 cm^3 requires 20.0 cm^3 of 0.100 mol dm^{-3} HCl. Find x.

$Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$. $n(HCl) = 2.00 \times 10^{-3}$ mol, so $n(Na_2CO_3) = 1.00 \times 10^{-3}$ mol in 25.0 cm^3, so 1.00×10^{-2} mol in 250 cm^3. $M = 1.43/0.0100 = 143$. $x = (143 - 106)/18 =$

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Uncertainty

Each piece of apparatus has an uncertainty. The **percentage uncertainty** is

$$\frac{\text{uncertainty}}{\text{measurement}} \times 100$$

- A burette reading has an uncertainty of ±0.05 cm^3, and a titre is the difference between two readings, so its uncertainty is ±0.10 cm^3.
- A 25.0 cm^3 pipette typically has ±0.06 cm^3.
- A balance reading to 0.001 g has ±0.0005 g for each reading.

For the final result, **add the percentage uncertainties** of all measurements in a calculation. To reduce the uncertainty:

- use a **larger titre** (a more dilute solution, or a larger mass of solid) so that the fixed error is a smaller percentage,
- use a balance of **higher resolution**,
- make a stock solution and use **a larger volume**.

> **A single measurement can show only a resolution limit. Random errors are reduced by repeating and taking a mean, and systematic errors are not.**

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