

Titration

Titration is done often to find out the concentration of one substance by reacting it with another substance of known concentration.

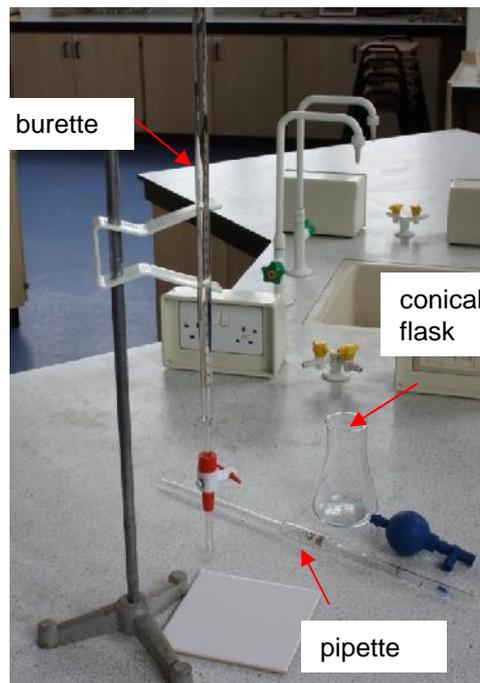
They are often done with neutralisation reactions, but can be done with redox reactions.

One substance (generally the one we don't know the concentration) is put in the conical flask. It is measured using a volumetric pipette.

The other substance is placed in the burette

However, the standard phrase: **titrate solution A with solution B** means that A should be in the conical flask and B should be in the burette.

A conical flask is used in preference to a beaker because it is easier to swirl the mixture in a conical flask without spilling the contents.

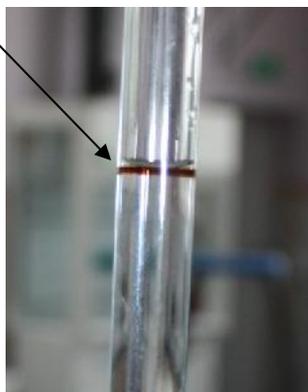


Method for Titration

Using the pipette

- **rinse** pipette with substance to go in it (often alkali).
- **pipette 25 cm³ of solution A into conical flask.** The volumetric pipette will have a mark on its neck to show the level to fill to. The bottom of the meniscus should sit on this line.
- **touch surface of solution with pipette** (to ensure correct amount is added). A small amount of solution will be left in the pipette at this stage. The calibration of the pipette will take into account this effect. It should not be forced out.

Make sure bottom of meniscus is on line on neck of pipette



Using the burette

The burette should be rinsed out with substance that will be put in it. If it is not rinsed out the acid or alkali added may be diluted by residual water in the burette or may react with substances left from a previous titration. This would lead to the concentration of the substance being lowered and a larger titre being delivered.

Don't leave the funnel in the burette because small drops of liquid may fall from the funnel during the titration leading to a false burette reading (would give a lower titre volume)

make sure the jet space in the burette **is filled** with the solution and air bubbles are removed.

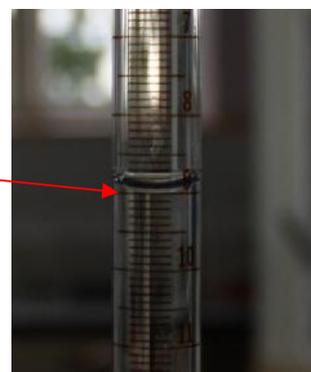
If the jet space in the burette is not filled properly prior to commencing the titration it will lead to errors if it then fills during the titration, leading to a larger than expected titre reading.



Read the bottom of the meniscus on the burette

This is reading 9.00cm^3

Even though a burette has marking reading to 0.1cm^3 , the burette readings should always be given to 2dp either ending in 0.00 or 0.05. 0.05cm^3 is the volume of 1 drop of solution delivered from a burette and so this is the smallest difference in readings that can be measured. If the bottom of the meniscus sits on a line it should end with a 0.00 as in the above example 9.00cm^3 . If the meniscus sits between two lines it should end 0.05. e.g. if the bottom of the meniscus sits between the lines marked 9.1 and 9.2, you should record 9.15



Adding indicator

Add **a few drops of indicator** and refer to colour change at end point

phenolphthalein

If acid is added from the burette the colour change would be pink (alkali) to colourless (acid): end point pink colour just disappears [use with titrations using strong alkalis e.g. NaOH]



phenolphthalein
Alkali colour



phenolphthalein acid
colour

Use a white tile underneath the flask to help observe the colour change

Methyl orange

Methyl orange is a suitable indicator for neutralisation reactions where strong acids are used.

It is red in acid and yellow in alkali. It is orange at the end point.



Methyl orange
Alkali colour



Methyl orange
end point



Methyl orange
acid colour

Add solution from burette whilst **swirling the mixture** and **add dropwise at end point**

Distilled water can be added to the conical flask during a titration to wash the sides of the flask so that all the acid on the side is washed into the reaction mixture to react with the alkali.

It does not affect the titration reading as water does not react with the reagents or change the number of moles of acid added.

note burette reading before and after addition of solution
repeats titration until at least 2 concordant results are obtained- two readings within 0.1 of each other

A single titration could be flawed. Repeating allows for anomalous titres to be spotted and discounted

Recording results

- Results should be clearly recorded in a table
- Result should be recorded in full (i.e. both initial and final readings)
- Record titre volumes to 2dp (0.05 cm³)

Titration number	1	2	3
Initial burette reading (cm ³)	0.50	2.50	1.55
Final burette reading (cm ³)	24.50	27.00	25.95
Titre (cm ³)	24.00	24.50	24.40

Safety precautions

Acids and alkalis are corrosive
(at low concentrations acids are irritants)

Wear eye protection and gloves

If spilled immediately wash affected parts after spillage

If substance is unknown treat it as potentially toxic and wear gloves.

Testing batches

In quality control it will be necessary to do titrations/testing on several samples as the amount/concentration of the chemical being tested may vary between samples.

Only distilled water should be used to wash out conical flasks between titrations because it does not add any extra moles of reagents

If **2 or 3 values are within 0.10cm³** and therefore **concordant** or close then we can say results are accurate and **reproducible** and **the titration technique is good and consistent**

Working out average titre results

Only make an average of the concordant titre results

$$\text{Average titre} = (24.50 + 24.40) / 2 = 24.45$$

Titration mixtures

If titrating a mixture to work out the concentration of an active ingredient it is necessary to consider if the mixture contains other substances that have acid base properties.

If they don't have acid base properties we can titrate with confidence.

Example 1: 23.6cm³ of H₂SO₄ neutralised 25.0cm³ of 0.150M NaOH. What is the concentration of the H₂SO₄?
 $\text{H}_2\text{SO}_4 + 2\text{NaOH} \rightarrow \text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O}$

Step 1: work out amount, in mol, of sodium hydroxide
 amount = conc x vol
 = 0.15 x 0.025
 = 0.00375 mol

Step 2: use balanced equation to give moles of H₂SO₄
 2 moles NaOH : 1 moles H₂SO₄
 So 0.00375 NaOH : 0.001875 moles H₂SO₄

Step 3 work out concentration of H₂SO₄
 conc= amount/Volume
 = 0.001875 / 0.0236
 = 0.0794 mol dm⁻³

Example 2: A 25cm³ sample of vinegar was diluted in a 250cm³ volumetric flask. This was then put in a burette and 23.10cm³ of the diluted vinegar neutralised 25.0 cm³ of 0.100 M NaOH. What is the concentration of the vinegar in gdm⁻³ ?
 $\text{CH}_3\text{CO}_2\text{H} + \text{NaOH} \rightarrow \text{CH}_3\text{CO}_2\text{Na} + \text{H}_2\text{O}$

Step 1: work out amount, in mol, of sodium hydroxide
 amount = conc x vol
 = 0.10 x 0.025
 = 0.00250 mol

Step 2: use balanced equation to give moles of CH₃CO₂H
 1 moles NaOH : 1 moles CH₃CO₂H
 So 0.00250 NaOH : 0.00250 moles CH₃CO₂H

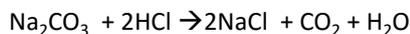
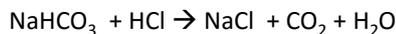
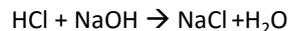
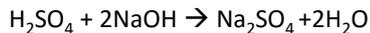
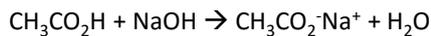
Step 3 work out concentration of diluted CH₃CO₂H in 23.1 (and 250 cm³) in moldm⁻³
 conc= amount/Volume
 = 0.00250 / 0.0231
 = 0.108 mol dm⁻³

Step 4 work out concentration of original concentrated CH₃CO₂H in 25cm³ in moldm⁻³
 conc = 0.108 x 10 = 1.08 mol dm⁻³

Step 5 work out concentration of CH₃CO₂H in original concentrated 25 cm³ in gdm⁻³
 conc in gdm⁻³ = conc in mol dm⁻³ x Mr
 = 1.08 x 60 = 64.8 g dm⁻³

To turn concentration measured in mol dm⁻³ into concentration measured in g dm⁻³ multiply by Mr of the substance
 conc in g dm⁻³ = conc in mol dm⁻³ x Mr
 The concentration in g dm⁻³ is the same as the mass of solute dissolved in 1dm³

Common Titration Equations



Example 3

950 mg of impure calcium carbonate tablet was crushed. 50.0 cm³ of 1.00 mol dm⁻³ hydrochloric acid, an excess, was then added and the mixture was transferred to a volumetric flask. The volume was made up to exactly 100 cm³ with distilled water. 10.0 cm³ of this solution was titrated with 11.1cm³ of 0.300 mol dm⁻³ sodium hydroxide solution.

What is the percentage of CaCO₃ by mass in the tablet

1. Calculate the number of moles of sodium hydroxide used

amount = conc x vol
 = 0.30 x 0.0111
 = 0.00333 mol

2. Work out number of moles of hydrochloric acid left in 10.0 cm³

use balanced equation to give moles of HCl
 1 mol NaOH : 1 mol HCl
 So 0.00333 NaOH : 0.00333 moles HCl

3. Calculate the number of moles of hydrochloric acid left in 100 cm³ of solution

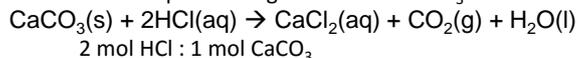
Moles in 100cm³ = 0.00333 x 10
 = 0.0333

4. Calculate the number of moles of HCl that reacted with the indigestion tablet.

In original HCl 50.0 cm³ of 1.00 mol dm⁻³ there is 0.05moles

moles of HCl that reacted with the indigestion tablet. = 0.05 - 0.0333 = 0.0167

5 Use balanced equation to give moles of CaCO₃



2 mol HCl : 1 mol CaCO₃
 So 0.0167 HCl : 0.00835 moles CaCO₃

6. work out the mass of CaCO₃ in original tablet

mass= amount x Mr
 = 0.00835 x 100 = 0.835 g

percentage of CaCO₃ by mass in the tablet = 0.835/0.950 x 100 = 87.9 %

Errors

Calculating apparatus errors

Each type of apparatus has a sensitivity error

- balance ± 0.001 g
- volumetric flask ± 0.1 cm³
- 25 cm³ pipette ± 0.1 cm³
- burette ± 0.05 cm³

Calculate the percentage error for each piece of equipment used by

$$\% \text{ error} = \pm \frac{\text{sensitivity error}}{\text{Measurement made on apparatus}} \times 100$$

e.g. for pipette

$$\% \text{ error} = 0.05 / 25 \times 100$$

To calculate the maximum percentage apparatus error in the final result add all the individual equipment errors together.

To decrease the apparatus errors you can either decrease the sensitivity error by using apparatus with a greater resolution (finer scale divisions) or you can increase the size of the measurement made.

If looking at a series of measurements in an investigation the experiments with the smallest readings will have the highest experimental errors.

If the burette used in the titration had an uncertainty for each reading of ± 0.05 cm³ then during a titration two readings would be taken so the error on the titre volume would be ± 0.10 cm³

Reducing errors in a titration

Replacing measuring cylinders with pipettes or burettes which have lower apparatus sensitivity error will lower the error

To reduce the error in a burette reading it is necessary to make the titre a larger volume. This could be done by: increasing the volume and concentration of the substance in the conical flask or by decreasing the concentration of the substance in the burette.

Reducing errors in measuring mass

Using a more accurate balance or a larger mass will reduce the error in weighing a solid

Weighing sample before and after addition and then calculating difference will ensure a more accurate measurement of the mass added.

Calculating the percentage difference between the actual value and the calculated value

If we calculated an Mr of 203 and the real value is 214, then the calculation is as follows:

Calculate difference $214 - 203 = 11$

$$\% = 11 / 214 \times 100 \\ = 5.41\%$$

If the %error due to the apparatus $<$ percentage difference between the actual value and the calculated value then there is a discrepancy in the result due to other errors.

If the %error due to the apparatus $>$ percentage difference between the actual value and the calculated value then there is no discrepancy and all errors in the results can be explained by the sensitivity of the equipment.

Errors in Titrations

- 1) A student carried out an experiment to determine the concentration of ethanoic acid in a solution of vinegar.
- The student used a measuring cylinder to measure out 25.0 cm^3 of the vinegar solution.
 - This solution was then transferred to a 250 cm^3 volumetric flask and the liquid level was carefully made up to the mark with distilled water.
 - A pipette was used to transfer 25.0 cm^3 portions of the acidic solution to conical flasks.
 - The solution was then titrated with sodium hydroxide solution, concentration $0.100 \text{ mol dm}^{-3}$, using phenolphthalein as the indicator.
- The average titre was 26.5 cm^3

- a) Suggest, with reasons, how the student's method of preparing the diluted solution could be improved.
b) The maximum total errors for the pipette and the burette in the titration are

pipette $\pm 0.05 \text{ cm}^3$

burette $\pm 0.15 \text{ cm}^3$

These errors take into account multiple measurements.

Estimate the combined maximum percentage error in using both of these pieces of apparatus.

- c) Give two changes you could make to reduce the percentage error in using the burette.
d) State why it is important to fill the space below the tap in the burette with sodium hydroxide before beginning an accurate titration.
e) Give a reason why a 250 cm^3 conical flask is preferred to a 250 cm^3 beaker for a titration.
f) During a titration, a chemist rinsed the inside of the conical flask with deionised water. The water used for rinsing remained in the conical flask.
(i) Give a reason why this rinsing can improve the accuracy of the end-point.
(ii) Explain why the water used for rinsing has no effect on the accuracy of the titre.
g) Give a reason why repeating a titration makes the value of the average titre more reliable.
h) Phenolphthalein is an acid. State how the average titre would change if a few cm^3 , rather than a few drops, of the indicator were used by mistake in this titration.
i) Vinegar contains other substances which improve flavour. Suggest one reason why these substances could affect the titration with sodium hydroxide.
j) Suggest why the student checks the concentration of the ethanoic acid in several batches of vinegar.

- k) Calculate the concentration of the vinegar in the 250 cm^3 flask.

Errors in the method will lead to an inaccurate value of the concentration of acid being calculated. How would the following situations affect the magnitude of the concentration of vinegar calculated? Explain why

- air lock below tap in burette
- air bubble in pipette of vinegar
- too much water is added so the meniscus of the diluted acid is above the line on the neck of the volumetric flask
- the concentration of the alkali has been incorrectly given. It is actually $0.095 \text{ mol dm}^{-3}$
- The conical flask contains some distilled water before the vinegar is added.
- The burette is not rinsed with sodium hydroxide
- The pipette was washed with water between titres

2) In a series of titrations a student obtained the following titres (all in cm^3).

Rough	1	2
25.7	25.20	25.35

State what this student must do in order to obtain an accurate average titre in this experiment.

3) A student wanted to test the acidity of a 100cm^3 sample of champagne. She suggested that the 100 cm^3 champagne should be divided into four portions before the titration. Explain how this change increases the reliability and decreases the accuracy of the experiment.

4) The M_r of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ is 286.0

a) The maximum percentage error in the experiment that can be due to the apparatus is $\pm 1.0\%$. If the only error is apparatus error, calculate the minimum value of the M_r of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ that could be obtained from an experiment.

Use this minimum value of the M_r to calculate a minimum experimental value for x

b) A titration was carried out with a sample of pure washing soda that had been stored for some time. A student obtained a value of 8.6 for the value of x . The container from which the hydrated sodium carbonate was taken was labelled $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$. Assume that the student carried out the titration and the calculation accurately. State a reason why the number of moles of water of crystallisation is less than 10.