Module:	Chemistry		Topic:	**Titration and back titration**
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Titration

Titration is a standard laboratory method of quantitative/chemical analysis which can be used to determine the concentration of a known reactant. Because volume measurements play a key role in titration, it is also known as volumetric analysis. A reagent, called the **titrant**, of known concentration (a standard solution) and volume is used to react with a measured quantity of reactant (**analyte**). Using a calibrated burette to add the titrant, it is possible to determine the exact amount that has been consumed when the endpoint is reached. **The endpoint is the point at which the titration is stopped. This is classically a point at which the number of moles of titrant is equal to the number of moles of analyte, or some multiple thereof** (as in di- or tri- protic acids). In the classic strong acid-strong base titration the endpoint of a titration is when the pH of the reactant is just about equal to 7, and often when the solution permanently changes color due to an indicator. There are however many different types of titrations.

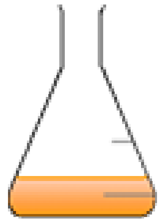
Many methods can be used to indicate the endpoint of a reaction; titrations often use visual indicators (the reactant mixture changes colour). In simple acid-base titrations a pH indicator may be used, such as phenolphthalein, which turns (and stays) pink when a certain pH is reached or exceeded. Methyl orange can also be used, which is red in acids and yellow in alkalis.

Due to the logarithmic nature of the pH curve, the transitions are generally extremely sharp, and thus a single drop of titrant just before the endpoint can change the pH by several points — leading to an immediate colour change in the indicator. That said, there is a slight difference between the change in indicator color and the actual equivalence point of the titration. This error is referred to as an indicator error, and it is indeterminate.

<u>Preparation of burette</u>

1. Fix the burette into the burette holder, taking care that it is vertical and stable.
2. Place a beaker underneath the burette.
3. Close the tap, and run some de-ionised water into the top of the burette.
4. Let the water clean the inside of the burette.
5. Open the tap, and allow the water to drain out. **Repeat**.
6. Close the tap, and (using the funnel) run some of the required reagent, e.g. acid, into the top of the burette. Open the tap, and allow the reagent to drain through into the beaker. **Repeat**.
7. Close the tap, and fill the burette to just above the 0.00 cm^3 mark with the required reagent.
8. Remove the funnel. Make sure that there are no air bubbles inside the burette.
9. Slowly open the tap, and allow the reagent to run down to (or just past) the 0.00 cm^3 mark.
10. Close the tap.

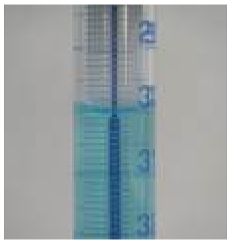
11. Remove the beaker, and place a white paper under the burette.
    The burette is now ready for use.



<u>Performing the titration</u>

1. Accurately measure a volume of the reactant into a **conical flask**.
2. Add a few drops of a suitable indicator to the flask.
3. Put the conical flask under the burette, and adjust the height of the burette so that the tip is just above the lip of the conical flask.
4. Turn the tap of the burette to allow the titrant to fall slowly into the reactant. Swirl the flask with the other hand.

5. The indicator should change colour as the titrant is added, but then quickly return to its original colour.
6. As the end-point is approached, the indicator takes longer to turn back to its starting colour. Add the titrant more slowly at this point (one drop at a time).



7. When the indicator remains at its end colour, the reaction has reached the end point. Measure the amount of titrant liquid used, as shown on the scale of the burette. As is standard for measuring any liquids in the laboratory, **measure from the bottom of the meniscus if it is concave, and from the top if it is convex.**
8. Repeat, then average the volumes.

<u>Recording the results</u>

1. Construct Results tables like the ones below. Before you start, record the reagents used.

Burette reagent	*Write in the name and concentration of the liquid in the burette*
Conical flask reagent	*Write in the name and concentration of the liquid in the flask*
Indicator	*Write in the name of the indicator used, if any*

	Run 1	Run 2	Run 3	Run 4
Initial volume (cm^3) ±0.05cm^3	*a*			
Final volume (cm^3) ±0.05cm^3	*b*			
Titre volume (cm^3)	*(b- a)*			
Mean titre volume (cm^3)				

2. In the first run, you should overshoot the end-point a little.
3. In subsequent runs, allow the burette reagent to run through more slowly as you reach the end-point to get a more accurate result. Record the start and end volumes as you go.
   Record volumes to the nearest 0.01cm^3, i.e. **all** volumes should end in .xxcm^3 .
   The uncertainty of the measurement is ±0.05cm^3.
4. If your three accurate runs are very different from each other, repeat until a more consistent result is obtained, i.e. to concordance (± 0.10cm^3). Put a pencil tick against the titres you use in your calculation of the mean titre (see below).
5. Calculate the mean volume delivered (the titre) and record it in the table.

Back titration

Back titration is an analytical chemistry technique which allows the user to find the concentration of a reactant of unknown concentration by reacting it with an excess volume of another reactant of known concentration. The resulting mixture is then titrated back, taking into account the concentration of the excess which was added.

Back titrations can be used for many reasons, including: when the sample is not soluble in water, when the sample contains impurities that interfere with forward titration, or when the end-point is more easily identified than in forward titration.

<u>Example</u>

Consider using titration to measure the amount of aspirin in a solution. Using titration it would be difficult to identify the end point because aspirin is a weak acid and reactions may proceed slowly.

Using back titration the end-point is more easily recognised in this reaction, as it is a reaction between a strong base and a strong acid. This type of reaction occurs at a high rate and thus produces an end-point which is abrupt and easily seen.

The titration curve for a strong acid with a strong alkali shows that the equivalence point occurs at pH 7. This means that the indicator phenolphthalein can be used. The end-point will be seen when the pink solution produced by the adding of phenolphthalein fades to colourless.

The first stage of this reaction is that of alkaline hydrolysis. This involves reacting the aspirin solution with a measured amount of sodium hydroxide; an amount that will exceed the amount of aspirin present. Because the hydrolysis reaction occurs at a very low rate at room temperature it will be heated to increase the reaction rate.

$CH_3COOC_6H_4COOH$ + 2NaOH -|> CH_3COONa + HOC_6H_4COONa + H_2O

2-ethanoylhydroxybenzoic acid + Sodium Hydroxide -> Sodium Ethanoate + Sodium-2-hydroxybenzoate + Water

The second stage then involves back titration of the hydrolysed sodium hydroxide solution with hydrochloric acid. This process reacts the excess sodium hydroxide with hydrochloric acid.

NaOH	+	HCl	---->	NaCl	+	H_2O
Sodium Hydroxide		Hydrochloric acid	--->	Sodium Chloride		Water

By the method of back titration the amount of hydrochloric acid needed to neutralise the unreacted sodium hydroxide in the solution can be determined. Knowing this and the amount of sodium hydroxide that was added the amount of aspirin that reacted with the sodium hydroxide can be determined.