Analytical Chemistry

Analytical chemistry, as the name suggests, is a branch of Chemistry dealing with the analysis of chemical substances. When we look at food labels we generally want to know two things what ingredients it has and how much of each of these ingredients there are. We commonly check if the food has fats, sugars, and salt and if it does we generally check how much of these are ingredients are in the food. Analytical chemistry can also be split by this type of thinking, there are two types of analysis.

Quantitative analysis	Deals with "how much" of a particular chemical is in a sample. An example is determining how much lead may be in a sample of drinking water
Qualitative analysis	Deals with what is present in a chemical sample. An example may by taking a sample of soil to determine whether it has been contaminated by a local factory

Quantitative Analysis

When we are dealing with the question of "how much?" we tend to answer it in one of two ways. If you were asked how many are in your family, you would count them, whereas if you were asked how much dirt there was in the garden you would probably weigh it. This suggests that there is a connection between counting and weight.

This connection is also applies to chemistry. When we ask how much there is in terms of a chemical, we can weigh it or we can count the atoms/molecules. In reality we do both by using the unit known as the **mole.** A mole is an exact quantity like a dozen (12), it represents 6.02×10^{23} (602,000,000,000,000,000,000,000) atoms or molecules. The reason that this number is so big is that atoms and molecules are so small. To give you an idea of the size of the number. If you spent a billion dollars every minute it would take a billion years to spend a mole of dollars.

We tend not to measure how much using weight because each atom has a different weight, a sulphur atom weights twice as much as an oxygen one therefore equivalent weights do not mean equivalent numbers of atoms. Having said this though we do not count individual atoms, we generally measure the mass. However by knowing how much a mole of a particular atom weighs then we can figure out the number of atoms.

A chemical equation is something that you have probably seen a lot, but do not have a good understanding of what is going on. All chemical equations must obey the **law of conservation of mass** which states that atoms cannot be created or destroyed in a reaction the can just be moved around. Therefore when balancing equations we are making sure that the numbers we add are going to balance the atoms on each side. Those numbers also give us the more ratio, let us take the example below.

$$2H_2 + O_2 \longrightarrow 2H_2O$$

This equation can be converted into words to read as follows. Two moles of hydrogen molecules will react with one mole of oxygen molecules to produce two moles of water molecules. It tells us how many moles are reaction of each to give us our products

Molar Mass

Molar mass, as the name suggests, is the weight of 1 mole of an atom or molecule. Many experiments have been done over the years to calculate the weight of all of the elements on the periodic table, it is indicated by the mass number. Using these mass numbers we can easily calculate the mass of one mole of any substance.

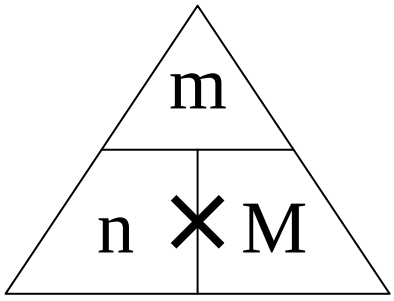
Knowing how to calculate mass numbers is simply a process of adding up the masses of each of the atoms in a chemical compound. Let us take the example of water (H_2O). The molecule contains one oxygen and two hydrogen atoms and we can therefore figure out the molar mass.

Molar mass (H_2O)	= 2 x H	+	1 x O
	= 2 x 1	+	1 x 16
	= 2	+	16
	= 18		

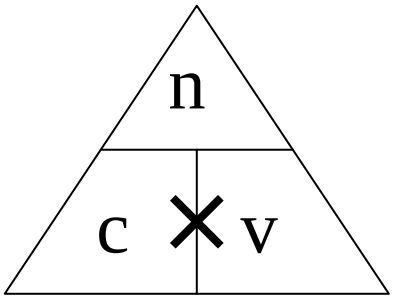
Therefore 1 mole of water weighs 18 g

Stoichiometry

When determining "how much" in chemistry we cannot see individual atoms, so instead we compare it to a known substance. To help us answer the question of how much we use two equations



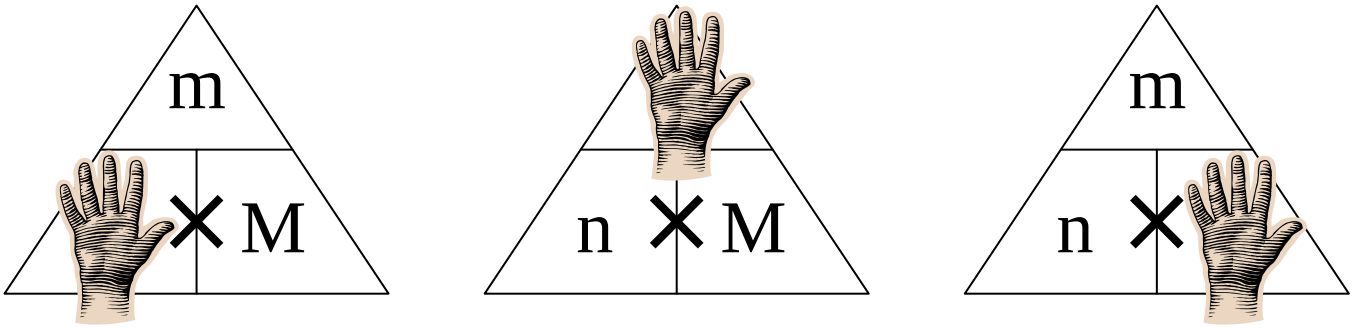
Where n = number of moles (mol)
m = mass (g)
M = molar mass (g mol^{-1})



Where n = number of moles (mol)
c = concentration (mol L^{-1})
v = volume (L)

The equations above are the two most important ones we will use in this topic and are used for almost every stoichiometric calculation. The reason for having two equations is that we are not always working with pure substances, sometimes we are dealing with solutions which have water also present. When working with solids and pure liquids we generally use the triangle on the left, whereas when working with solutions we have to use the triangle on the right.

These triangles can be use to calculate any of the three variables as long as we know the other two. By covering over the variable we are looking for we give ourselves the formula for calculating it. For example if we wanted to calculate the number of moles and we were give the mass and molar mass we would cover over n, and be left with m/M. Therefore n=m/M



$n = m/M$ $m = nM$ $M = m/n$

Let us take two examples of calculations.

a) Calculate the number of moles if have 406g of sodium chloride (NaCl)

$M_{NaCl} = 23 + 35$
$= 58$

$n = m / M$
$= 406 / 58$
$= 7$ moles

b) calculate the concentration if 1.25 moles of a substance is dissolved in 5 L of water.

$c = n / v$
$= 1.25 / 5$
$= 0.25$ mol L^{-1}

These questions are easy because you have only one substance involved. Some are much harder. Let us take the example question below

What mass of sodium hydroxide is required to neutralise a 4 L spill of 2 M sulfuric acid.

In this case you are only told one bit of information about the substance you are looking for so you cannot work it out simply by putting the numbers into one of the formulae above. In this case you need to follow a number of steps

1. Write a balanced equation for the reaction
2. Calculate the number of moles of the known substance
3. Write the mole ratio $n_{unknown} / n_{known}$
4. Calculate the number of moles of the unknown by using the more ratio
5. Calculate the missing quantity.

By following these steps for the above question we get the following

Step 1:

$$2NaOH + H_2SO_4 \longrightarrow Na_2SO_4 + 2H_2O$$

Step 2:

$$n_{(acid)} = c_{(acid)} \times v_{(acid)}$$
$$= 2 \times 4$$
$$= 8 \text{ mol}$$

Step 3: From the equation we get the following

$$n_{unknown} / n_{known} = n_{base} / n_{acid} = 2 / 1$$

Step 4: From the mole ratio above we get

$$n_{base} / n_{acid} = 2 / 1$$

$$n_{base} = 2 / 1 \times n_{acid}$$
$$= 2 \times 8 \text{ (from step 2)}$$
$$= 16 \text{ mol}$$

Step 5:

$$m_{base} = n_{base} \times M_{base}$$
$$= 16 \times 40$$
$$= 640 \text{ g}$$

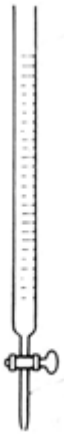
Therefore 640 g of sodium hydroxide would be required to exactly neutralise a 4 L spill of 2 M sulfuric acid.

Titrations (Volumetric Analysis)

More often than not, analysis occurs in a liquid form. There are two reasons for this, firstly reactions are much faster in a liquid form and secondly to allow the particles to move around so that they can be analysed more easily. Titrations are one of the more widely used quantitative analysis techniques that are used. They can be used to determine the acid content of orange juice, alcohol content of beer, alkali content of cleaning agents amongst others.

Titrations involve reacting a solution with another of a known concentration. Commonly they are acid-base reactions but redox reactions are also used. They use an indicator to determine the end point, the point at which the acid is neutralised by the base.

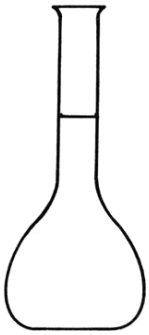
Titrations rely a lot on accuracy no only by the experimenter but also the equipment used must also have a high degree of accuracy. Below is four pieces of glassware that are used commonly for titrations and a brief explanation of each including washing procedures. Washing procedures are stated as cleanliness with titrations is vital, any impurities can alter your results dramatically.



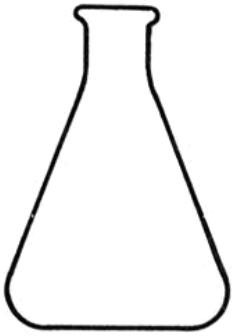
The **burette** is used to deliver a reactant in known quantities. It must be washed with the substance that is going to be used in it letting it run out through the tap.

When using the burette make sure the tip of it is full before starting as the volume markers include this amount.

The **pipette** is used to deliver an exact, known volume of liquid. The pipette should washed with the solution that you are going to be using in it



The **volumetric flask** is a vessel used for making up standard solutions, those of a known concentration. It should be cleaned only with water before use



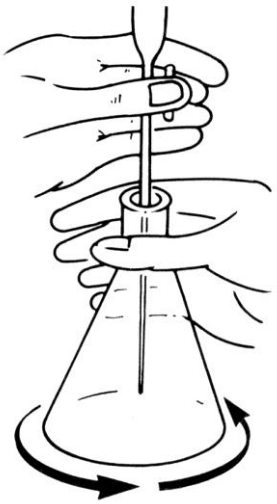
The **conical flask** is where the reaction of the titration occurs. It must be cleaned with distilled water only before a titration.

Performing a titration

1. Rinse a conical flask with distilled water and fill it with a known amount of the substance being analysed using a pipette.
2. Fill the burette with your standard solution and record the initial burette reading in your book.
3. Perform a scout titration by opening the tap of the burette so that it flows into the conical flask, swirl the flask while this is happening.
4. Close the tap when the endpoint is seen (permanent colour change)
5. Record the final burette reading.
6. Calculate the volume of titrant delivered known as the titre.
7. Perform the actual titration by setting up as with the scout titration and perform the following steps.
8. Turn the tap and let the titrant flow rapidly out of the tip until you are within 2 ml of the expected endpoint, as found above in the scout titration.
9. Rinse the walls of the flask with the wash bottle again.
10. Continue adding the titrant one drop at a time until you have reached the endpoint.
11. Turn off the tap and record the final burette reading
12. Calculate the titre.
13. Repeat steps 7-12

Concordant titres are those that agree within 0.1 ml generally the aim is to achieve two or more concordant titres.

The way in which you hold both the conical flask and the burette is shown. It is very important that it is held in this way firstly because as the drops are added you need to continuously stir it whilst being ready to stop it at any time. Secondly by holding the burette in that way you are providing stability to the apparatus because you are holding onto it. Thirdly at times the taps have been known to come out if pulled, by putting your hand around the burette in that fashion you are pulling the tap inwards not outwards and therefore eliminating the chance of pulling out the tap.



Separating Mixtures

Pure Substances and Mixtures

The differences between pure substances and mixtures in chemistry is an important one. Mixtures and pure substances are all around us and at times we want to know what something is made of, this is called qualitative analysis. In order to try and separate a sample we can do it by either physical or chemical changes. The differences between these are explained below

Physical change – When a substance changes but no new substance is formed. It is normally associated with changes in state (solid, liquid and gas).

Chemical Change – When one substance changes into another substance(s), or when other substances combine to form a new substances. It is normally paired with a change in colour, or appearance or the production of heat or light

A pure substance is one that contains only one type of substances. This group can be further broken down into two groups elements and compounds.

Elements – Substances that **can not** be broken down into two or more simpler parts by either physical or chemical changes.

Compounds – Substances that can be broken down into two or more simpler parts **only** by a chemical change, **not** a physical one. Compounds can be broken down into elements.

On the other hand mixtures contain a range (two or more) of different substances mixed together. Mixtures can be easily separated using physical changes. As with pure substances there are a few different types of mixtures

Solutions – Solutions are homogeneous mixtures, meaning their particles are distributed evenly. Their particles are microscopic and both the solute and the solvent have the same polarity. Salt water is an example of a solution

Suspensions – Suspensions have large particles which are heterogenous (unevenly distributed). Most mixtures are suspensions.

Colloids – Colloids are somewhere between solutions and suspensions. The ingredients in colloids are normally smaller than suspensions but larger than solutions, however the particles are still homogeneous or evenly distributed. Milk is an example of a colloid.

Mixtures are easily separated and a list of common techniques are shown below.

Technique	Mixtures it Separates	Physical Property it Uses
Froth Flotation	Solid – Solid	Attraction to water
Filtration	Solid – Solid (one soluble) Liquid - Solid	State change Particle size
Sedimentation	Liquid - Solid	Density difference
Decanting	Liquid - Solid	Density difference
Centrifuge	Liquid – Solid Liquid - Liquid	Density Difference Solubility
Evaporation	Liquid - Solid	Melting / Boiling Point
Distillation	Liquid – Liquid	Melting / Boiling Point
Separating Funnel	Liquid – Liquid	Solubility Density Difference
Chromatography	Solid – Liquid Liquid – Liquid Liquid – Gas Gas - Gas	Polarity Solubility

Chromatography

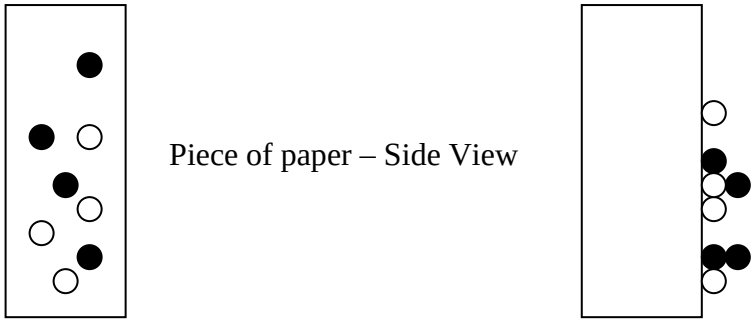
Chromatography is a form of qualitative analysis which separates components of a mixture due to differences in solubility in a solvent and adsorption or "stickability" to a surface. It is a widely used technique for identifying components of a mixture. Chromatography is used for the following purposes

- To separate mixtures of solids, liquids or gases
- To determine how many components are in a mixture
- To tell if a substance is pure
- To tell how much of each component are present
- To identify each component

There are many different types of chromatography that are used, some are still done manually however many these days are done automatically. Some of the most common types of chromatograph are shown below

- Column chromatography
- High performance liquid chromatography (HPLC)
- Thin layer chromatography (TLC)
- Paper Chromatography
- Gas Chromatography (GC)

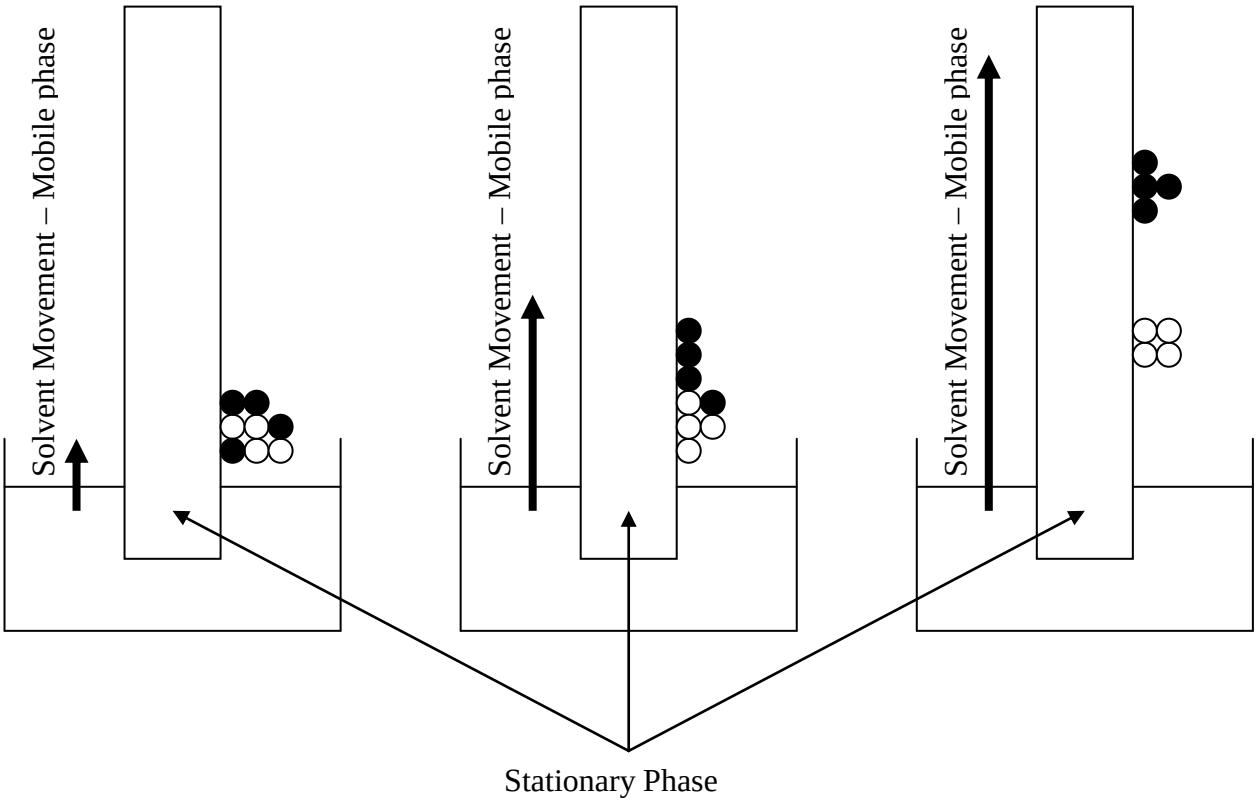
Chromatography deals with a property called **<u>adsorption</u>**, which is different to and should not be confused with absorption. The differences between them are shown below



In this case the substance has been **<u>absorbed</u>** as it has been completely drawn inside the paper.

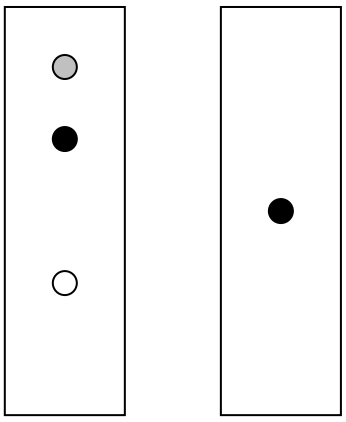
In this case the substance has been **<u>adsorbed</u>** as it has stuck to the outside surface of the paper rather than being drawn in.

Chromatography always has two components called phases. Each of the two phases has a different polarity, one is polar and the other non-polar. This allows you to easily separate substances by their polarity. The **stationary phase** is the phase which does not move. It is the surface to which the substance being analysed is adsorbed. The **mobile phase** is a solvent which dissolves the substance being analysed and carries it along the stationary phase.



As the mobile phase moves up the stationary phase the components with the same polarity as the mobile phase will be more soluble and be carried further whilst those which have the same polarity as the stationary phase will adsorb, or stick stronger and will not move as far.

One of the typical results you can see from a chromatography experiment are shown opposite. For the chromatograph on the left the substance being analysed is made up of three distinct substances as shown by the three dots or bands. Whereas the chromatograph on the right shows only one dot and therefore a pure substance.



In this case if the mobile phase was water then the grey substance would be the most polar and the white substance the least polar. This is because water is a polar substance and will carry the most polar substance furthest.