BASIC PURIFICATION TECHNIQUES

Chemistry is not a purely theoretical subject - most of the problems that chemists have to solve involve developing practical techniques. The problem of making pure substances is one that we have touched upon already; we understand that absolute purity is close to impossible but we must aim to get substances as pure as we can for the purpose that they are required.Many chemical reactions take place in solutions for instance, which means that the water plus some other dissolved substances may be present along with the desired product.

When a new pigment has been developed, it will be necessary to see if it is made of a mixture of more than one compound. This involves developing a technique to separate the component chemicals. When liquids are produced, it may be necessary to separate them from other contaminants, again often water.

Over the last three or four thousand years, many purification techniques have been developed, so a lot of the systems used in a modern Chemistry laboratory, or production line, are actually methods developed by ancient civilizations. The apparatus may be more sophisticated but the principles are the same. Examples of salts found from archaeological sites in ancient Egypt, metal artefacts from ancient China, or pigments from medieval Europe show a very high degree of purity.

The apparatus used for these basic principles is illustrated in **Figure 2**

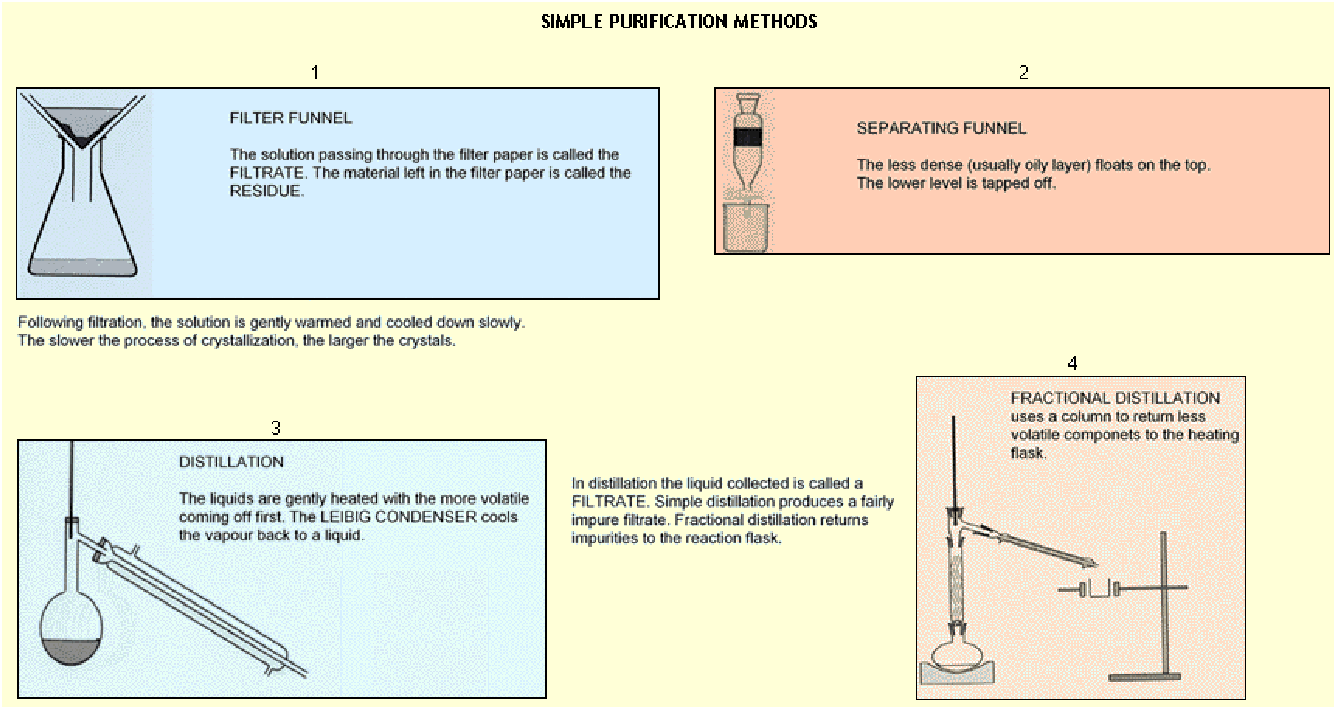

Fig. 2

Note that I have used the standard method for drawing apparatus used in chemistry which is to imagine the items are sliced through the middle and drawn as simple cross-sectional pictures. You should try to get used to this method of drawing. One point to remember is that if you look at a glass of water, the liquid is slightly raised where the sides of the glass meet the water. This is called the MENISCUS and is caused by an attraction of the water molecules to the glass (the same reason that water makes things wet); you should always include a slight meniscus on your diagrams as shown in **Figure 2.**

When a soluble substance has to be separated from an insoluble substance, FILTRATION is the accepted method. For instance, separating salt from sand would involve first adding water to the mixture, and then heating it gently to dissolve all of the salt. The mixture is then passed through a FILTER FUNNEL which contains a FILTER PAPER. The water and the dissolved salt pass through the microscopic holes in the paper but the sand remains behind. To salvage the sand it just needs to be rinsed with clean water and then dried. The remaining salt and water mixture are heated until CRYSTALLIZATION takes place with the water evaporating and pure salt crystals left behind **Fig. 2** - 1.

Usually, when two liquids are together they form a mixture with the particles of each completely dissolved in the other. Sometimes however, the two liquids do not mix but remain separate, the less dense floating on top of the more dense. Two such liquids are described as IMMISCIBLE and oil with water is a good example of this situation. Immiscible liquids can be separated using a SEPARATING FUNNEL. This apparatus allows the lower, more dense liquid to be drawn off at the bottom, leaving the less dense liquid in the funnel. An older and less satisfactory technique is the process of DECANTING where the top layer is simply poured off **Fig. 2** - 2.

Two liquids that mix completely are called miscible. Such a case is water and alcohol - two clear liquids that become completely mixed and do not form separate layers. The method of separating these two liquids is called DISTILLATION and it takes advantage of the fact that the liquids boil at different temperatures. At sea level, water boils at 100°C and ethanol has a boiling point of 78°C. (In fact, ethanol is just one of a large number of alcohols but it is the most common one that is usually produced by a process of fermentation. See Chapter 13.) If weheat up the mixture to around 80°C then all of the alcohol will be boiled off but very little of the water. The alcohol vapour is cooled down by passing it through a column called a LEIBIG CONDENSER in which the gas is surrounded by a sleeve of cold water **Fig. 2** - 3. We cannot get all of the water out of the mixture in this way as although water boils at 100°C, it begins to evaporate at much lower temperatures. One way to improve the purity of the alcohol is to use a process of FRACTIONAL DISTILLATION in which the vapour is passed up a column and condensed at various points on the surface of glass beads; this makes it much more difficult for the water to pass into the condenser **Fig. 2** - 4. Fractional distillation is of high economic importance as crude oil is distilled into gasoline by this method as we shall see in Chapter 12.

One method of separating mixtures which has been developed into a very sophisticated set of techniques is called CHROMATOGRAPHY. In its simplest form, called PAPER CHROMATOGRAPHY, a sample of ink is placed in the center of a pencil line on a strip of CHROMATOGRAPHY PAPER. The strip is placed in a solvent with the sample above the solvent line. Over a period of time, the solvent rises up the paper and separates the components of the ink. This often results in a large number of coloured components being identified. Modifications of this technique will be met later in the course including THIN LAYER CHROMATOGRAPHY (TLC) and GAS CHROMATOGRAPHY.

COLLOIDS

In Chapter 1 we visited the idea that matter exists in one of three states - solid, liquid or gas. We are now looking at mixtures and it would be a good time to consider that some mixtures appear to exist in ways that makes the state hard to decide. For instance, what about 'Jell-O' (in much of the world, Jell-O is called jelly which is very confusing!); is it a solid or a liquid - it seems to have some of the characteristics of both! We can think of many other ambiguous substances - shaving foam, mayonnaise, mud and polyurethane foam are all such examples. Chemists use the word COLLOIDS to describe mixtures that do not easily fit into the solid/liquid/gas categories.

Jell-O is in fact a liquid, held within a solid network. This means that long strands of solid material are woven together in such a way that microscopic pockets exist which hold liquid particles. Such a mixture is called a GEL. If a gas is held within a solid or liquid network it is called a FOAM; shaving foam is a gas held within a liquid network (think of it as millions of tiny bubbles of liquid containing gas), and polyurethane foam is an example of a gas held within a sold network.

Solid particles may be suspended in a liquid whereby they appear to be a uniform mixture called a SOL (an older word SUSPENSION is also in use). If you leave a sol for a period of time, the solid particles usually end up sinking to the bottom. Muddy water and many drug store indigestion mixtures are in fact sols. A bottle of prescription medicine may need shaking before use - if it is a sol, this ensures thorough mixing and is essential if you are to get the same dose of active ingredient each time.

Two immiscible liquids (see above) may be combined into a smooth mixture called an

EMULSION; they do this by the addition of a third substance called an EMULSIFYING AGENT.Mayonnaise is made by combining a water-based ingredient, vinegar, with an oil-based ingredient, olive oil, with an emulsifying agent, egg. The result is a smooth emulsion. A verycommon emulsifying agent is detergent, which causes small particles of oil to form an emulsion with water.

The most common type of colloid that we come across is when a solid or liquid becomes completely dispersed in a liquid. This is called a SOLUTION. Our experience of solutions tells us that if we leave them for any period of time, the particles do not settle out unlike a sol. If we look at the separation methods outlined above, we note that crystallization involves a solution whereby the liquid evaporates, leaving the solids behind.

The table below summarizes these types of mixture and adds a few more examples. If you come across a unusual mixture (your kitchen and bathroom will contain many), then try to add them to the list.

NAME OF COLLOID	NATURE OF COLLOID	EXAMPLES OF COLLOID
Foam	Gas held within a solid or liquid network.	Shaving foam, cake, bread, polyurethane foam, pumice stone, meringue.
Gel	Liquid held in a solid network.	Jell-O (jelly), hair gel, shaving gel.
Sol (suspension)	Solid particles held in a liquid, after a time, the solid particles usually sink to the bottom.	Muddy water, blood, many drugstore 'mixtures', water-based house paints.
Emulsion	Two or more immiscible liquids in a system involving an emulsifying agent.	Mayonnaise, milk, cream.
Solution	A solid or liquid that is	Salt water, hydrochloric

	completely mixed with a liquid. Solution do not separate with time but the liquid component may evaporate, leaving behind the solid.	acid, copper sulphate solution, urine, wine.

Source : http://www.peoi.org/Courses/Coursesen/chem/fram1.html