Experimental Design and Separation Techniques

Experimental Design – Production, Collection and Testing of a Gas

If a gas is produced during a chemical reaction in a test tube, it is crucial to test the gas immediately, to establish its identity.

The chemical tests on these 6 gases will be carried out in the lab.

- Ammonia
- Carbon Dioxide
- Chlorine
- Hydrogen
- Oxygen
- Sulfur dioxide

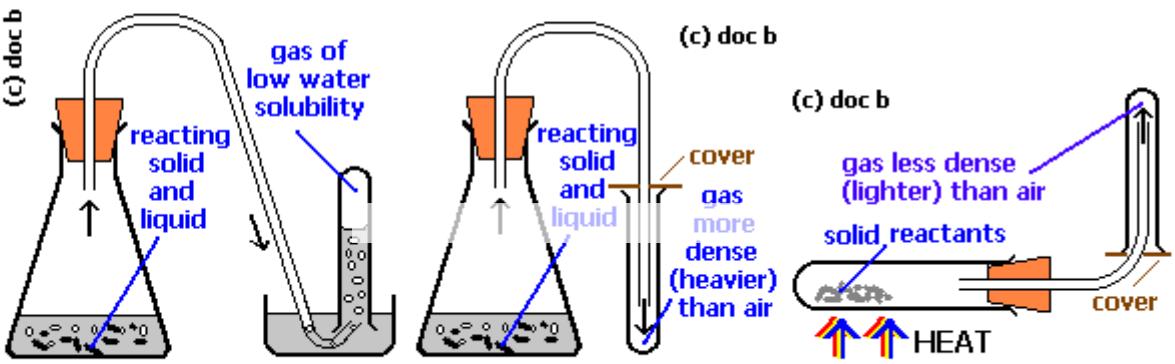
Occasionally, it is also important to collect the gas being produced.

<u>Measuring Gasses</u>

The usual apparatus of measuring volume of a gas is the gas syringe.

The method of collecting a gas may also depend on:

- Solubility in water
- Density compared to that of air



Often, the gas produced is contaminated with water vapour. The mixture is passed through a drying agent to dry the gas. Common drying agents for gasses include concentrated sulphuric acid, anhydrous calcium chloride , anhydrous copper [II] sulphate, calcium oxide and silica gel.

Anhydrous copper [II] sulphate is also used as an indicator for water vapour, because it turns from white to blue in the presence of water.

Measuring Liquids

The usual apparatus used to measure volume of a liquid are:

- Measuring cylinder
- Pipette
- Burette

The precision of the measuring cylinder is usually up to zero decimal place whereas that of the pipette is usually up to zero decimal place, and that of the burette is up to 2 decimal places (second dp is 0 or 5).

Common misconceptions:

A beaker is not to be used to measure volume of liquids. The scale lines on the side of a beaker are only approximate.

Purification Techniques

Dissolving, Filtering and Evaporating

Example: A sample of common salt is contaminated with some sand.

1. Add excess water to dissolve all the salt in a beaker [with stirring].

2. Filter the mixture and collect all the filtrate.

3. Heat the filtrate gently in an evaporating dish until all the water has been evaporated. The solid left is the pure salt.

Crystallisation

Many crystals contain water of crystallisation – this is the water molecules inside the crystal that give the crystal its shape. As such, excessive heating during crystallisation would drive off this water of crystallisation and crystals would not form.

Example: Crystallisation of copper [II] sulphate from its solution/reagent.

1. Heat Benedict's reagent in an evaporating dish.

2. When about one third of solution is left, cease all heating processes. This is called heating to saturation, and the solution after heating is said to be saturated.

3. Let the solution cool and crystals would appear. This is due to the fact that while the saturated solution is left to cool, the solubility of the copper [II] sulphate solute decreases and the undissolved solute appears as crystals.

Sublimation

Some solid substances become gaseous when heated under room conditions, without going through the liquid state.

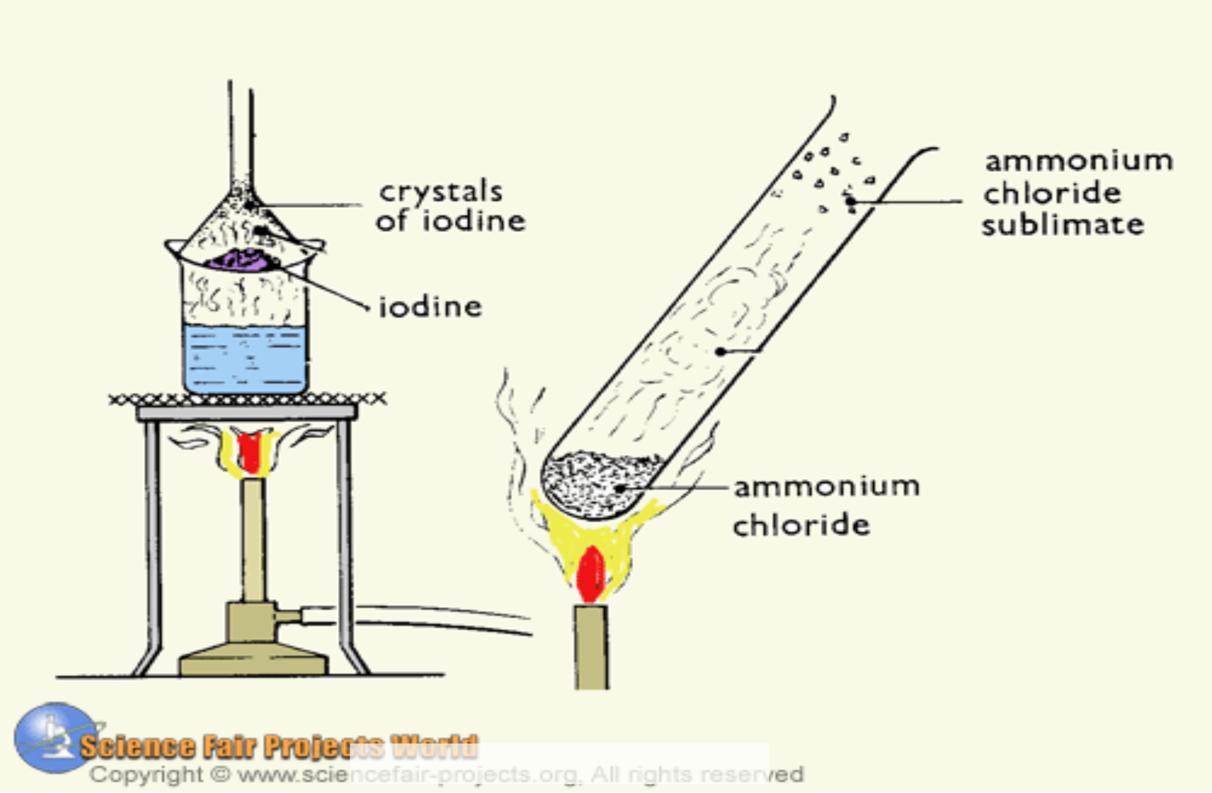
Examples of such solids that sublime are:

- Ammonium chloride
- Iodine
- Dry ice [solid carbon dioxide]
- Naphthalene [mothball]

Example: A sample of ammonium chloride is contaminated with some common salt [sodium chloride].

1. Heat the mixture in an evaporating dish and invert a filter funnel over the mixture.

2. The ammonium chloride sublimes, and its vapour solidifies as a deposit on the cooler surface of the inverted funnel.



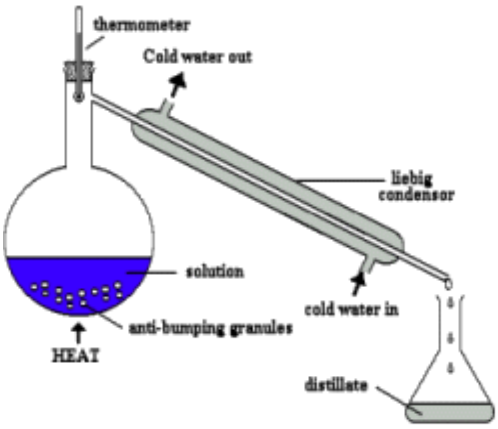
Separating Funnel

When 2 liquids, such as water and oil, cannot be easily mixed together, and tend to separate into 2 layers, we say that they are immiscible. A separating funnel is used to separate such liquids.

Simple Distillation

Distillation is used to separate/recover solvent from solution.

Example: To get pure water from sea water/contaminated water.



1. Boil the seawater [together with some broken porcelain] in a flask that is connected to a condenser, as shown in the above diagram.

2. Pump cooling water into the jacket of the condenser.

3. The water vapour/steam in the flask enters the condenser and is condensed back into the water, which is collected in the conical flask as distillate.

Side note:

- Purpose of broken porcelain is to act as anti-bumping granule or 'boiling chips' as shown in the diagram above. This ensures smoother boiling by allowing formation of smaller air bubbles.
- The thermometer is placed at the exit of the flask to measure the temperature of the pure vapour exiting the flask, thus determining the boiling point of the solvent [in this case, water] in the mixture.
- The Liebig condenser is crafted in such a way that cooling water enters from the bottom to ensure that the bottom of the condenser is the coolest part, and any vapour that initially did not condense would condense there, thus no vapour can escape.

Fractional Distillation

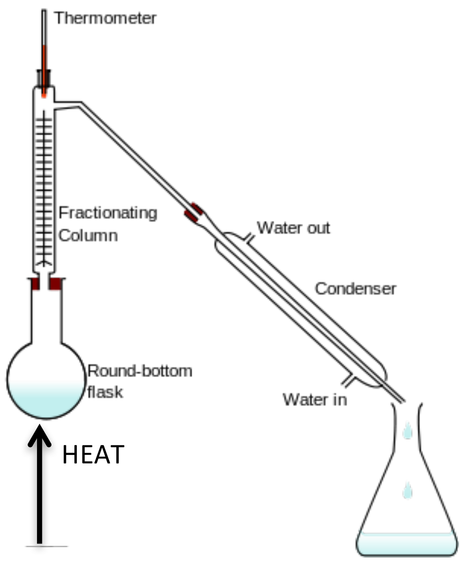
Fractional distillation is used to separate a mixture of miscible liquids with different boiling points.

Example: Separate mixture of water and ethanol.

1. Place the mixture into a conical flask connected to a fractionating column.

2. Heat the mixture with an electric heater.

3. The heat vaporises the ethanol and water and drive the vapours up the fractionating column. Water has a higher boiling point that ethanol, thus the water condenses in the fractionating



column and water drips back into the flask. The ethanol vapour reaches the top column and passes into the condenser, and is thus condensed, forming ethanol liquid as the distillate.

Side note:

- The Bunsen burner is not used to heat the mixture because ethanol is highly flammable and no naked flame should be allowed to heat it.
- Tiny bead are used to fill the fractionating column to increase surface area fir condensation, making the separation of both liquids more efficient.
- The thermometer first records the temperature of the ethanol vapour, which is 78 degrees Celsius. After all the ethanol vapour has entered the condenser the thermometer will then record the temperature of the steam, which is 100 degrees Celsius.

Revision Osmosis

High pressure is used to force impure water through a membrane which has millions of tiny holes/pores. Only water molecules can pass through the pores, leaving solute particles behind, this is one of the ways to create NeWater.