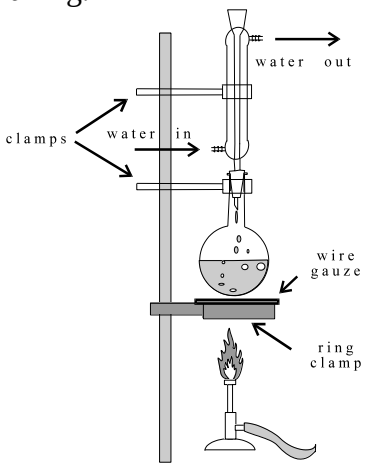
Refluxing

We can increase the rate of a chemical reaction by heating, to increase the average kinetic energy of the molecules involved, however heating a liquid sample often results in boiling off of the solvent at a rate faster than the reaction can occur, leaving you with a burnt solid sample of little use in your analysis. Refluxing is a technique which allows a sample solution to be boiled without the loss of solvent. As a solution is boiled, vapour rises into a jacketed condenser, where the vapour cools, condenses and drops back into the flask below. The following figure illustrates the apparatus setup we will use in this laboratory for refluxing.



Position your Bunsen burner and attach a ring clamp to a retort rod at a height that allows a medium size flame below the clamp. Place a piece of wire gauze on the ring clamp; this prevents excessive heating of the flask by dispersing the direct heat of the Bunsen burner flame. Next clamp your round bottom flask into position so that it is approximately one centimetre above the gauze. Once your flask is secure, you may add your reactant solution(s) to the flask, using a funnel if necessary.

Insert your jacketed reflux condenser into the flask, and clamp it securely to prevent tipping. Note that the exterior water jacket of the condenser has a very thin glass wall, therefore, do not over tighten your clamp or you may shatter the glass. Using the cooling water lines on the benches, connect the water line to the condenser such that the water flows up from the bottom, and out of the top of the condenser. This allows the jacket to fill completely with water, ensuring adequate cooling during the refluxing process. Ensure that your water outlet hose is draining to a nearby sink; you may have to clamp it to prevent it from flopping out of the sink. Now slowly turn on the water, making certain that the water is entering at the bottom of the condenser, and that water is properly draining into the sink.

Light the Bunsen burner and adjust to maintain a gentle boil of the solution. Once your solution has started to boil, you will begin to see vapour rising up the condenser, and liquid dripping back into the flask. The vapour rising into the condenser will form a “reflux ring” of condensate; below this point solvent can be seen flowing back into the flask, above this point the condenser should be dry. If the reflux ring rises more than halfway up the condenser, reduce the rate of heating to prevent the escape of solvent. If vapour is escaping from the top of your condenser, remove the heat source immediately.

At the end of the reaction period, turn of the Bunsen burner, let the apparatus cool, and then turn of the condensing water lines.