

- **REFLUX WITH ADDITION APPARATUS**



**ALWAYS** makes sure the hoses are securely attached to the condenser.

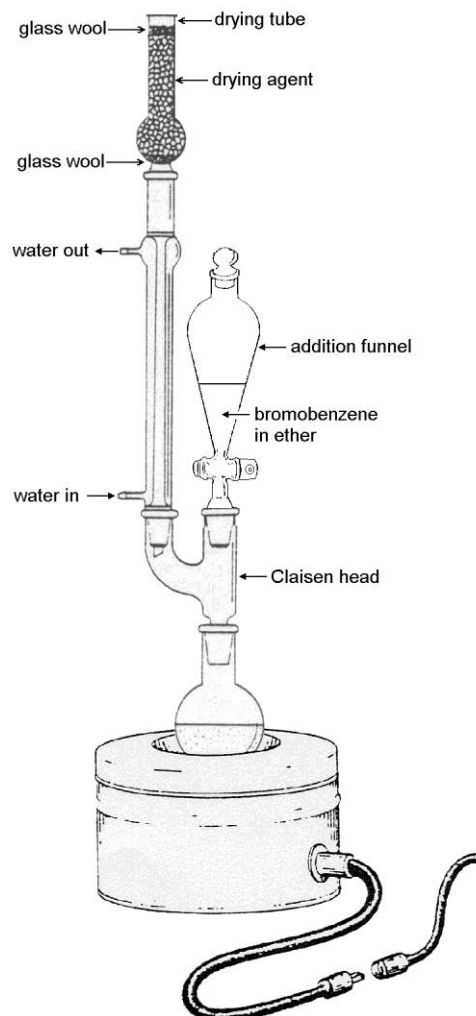
A gentle flow of cooling water is all that is needed.

**Check for leaks !**

A [basic reflux apparatus](#) was first introduced in Chem 351 and you have already used it this semester in an earlier experiment.

A more advanced set-up is often needed to facilitate adding a reagent while refluxing. For this you will use a modified reflux set-up where an addition funnel is added to the basic set-up using a Claisen head in order to allow the addition of a second reagent directly into the reaction flask without requiring the removal of the condenser. This is important if a reaction is air or moisture sensitive. Light greasing of the ground glass joints will help maintain the inert atmosphere.

As an example, Grignard reagents are very reactive intermediates and will react readily with water to form hydrocarbons (in our particular case benzene would be produced). To prevent this the reaction must be carried out taking care to exclude water until the final work-up of the product when dilute acid is added to decompose the intermediate and generate the free carboxylic acid. The typical precautions taken include: *dry* glassware; anhydrous solvents and reagents; exclusion of moisture in the air by use of a tube packed with an efficient drying agent,  $\text{CaCl}_2$ .



**NOTE:** In the equipment set up shown, a heating mantle with a heating controller is being used to heat the round bottom flask. In your equipment set up (or any of the others), the heating mantle and heating controller will be replaced by an engineered (shaped) aluminium block sitting on the top of a stirrer hot plate. This is a more modern version of a heating mantle with some distinct benefits.