

[MUSIC PLAYING]

**PROFESSOR:** Most organic reactions occur slowly at room temperature and require heat to allow them to go to completion in a reasonable time period. If these reactions were heated in a closed container, pressure would build up inside the container and may explode. If these reactions were heated in an open container, then the solvent may evaporate from the system.

The method of refluxing solves both of the above problems by allowing the chemist to heat a reaction at its boiling point, without any materials evaporating or any containers exploding. Refluxing works by heating a solution to boiling and condensing the resulting vapor by continuous cooling. Because approximately 80% of all organic reactions involve refluxing, this technique is an important one to understand.

To carry out a reflux, you will need a cork ring, a round-bottom flask, a stir plate and stir bar-- or boiling stones-- a condenser, grease, a ring stand, two extension clamps with fasteners, two pieces of thin-walled rubber tubing, three hose clips, a heating mantle, an iron ring, and a variac. The first step of refluxing involves choosing an appropriate solvent. A good refluxing solvent will dissolve all of the reagents but not react with them. And it will boil at a temperature high enough to allow the reaction to proceed rapidly.

After choosing your solvent, dissolve all of the reagents in it, and transfer the solution to round-bottom flask. The round-bottom flask should be no more than half full. Then, add either a stir bar, or boiling stones, to the solution to avoid bumping. Bumping is the eruption of large bubbles from a solution due to superheating, which results in material loss, or even a fire.

Magnetic stirrs disallow bumping because they create turbulence in the solution, which disrupt the large bubbles. Boiling stones are also able to disrupt the large bubbles because these porous materials produce a steady stream of fine air bubbles when heated in a solvent. Always place boiling stones in the solution before heating. If boiling stones are added to a hot solution, the liquid may instantaneously boil and shoot out of the flask.

To assemble the reflux apparatus, clamp the round-bottom flask containing your sample to a ring stand. Lightly grease the male end of the condenser, and attach it to the round-bottom flask. Attach the water inlet tubing to the bottom connection, and the water outlet tubing to the top connection. Secure the tubing with hose clips to prevent the tubes from popping off and flooding the lab due to pressure changes in the water.

Secure an appropriately-sized heating mantle and iron ring below the flask. If you do not have an appropriately-sized heating mantle, then use one that is too big. Never use a heating mantle that is too small because heat is not easily transferred between the mantle and glass due to poor contact, which causes the mantle to burn out. If the mantle is too big for the flask, then add sand to fill in the spaces. Finally, plug the heating mantle into a variable voltage transformer, or variac, never directly into the wall.

Before starting the reflux, make sure that the apparatus is open at the top of the condenser. Never heat a closed system because the pressure buildup may cause an explosion. To reflux a reaction, double check that all of the joints are secured with hose clips, and start the water flow. Slowly increase the setting on the very back until the solution begins a gentle boil.

You will see a ring of vapor, or condensate, form above the solution. This vapor enters the condenser, cools, and the resulting liquid drops back into the flask. Carefully adjust the amount of heat that is delivered to the solution so that the ring of condensate stabilizes about halfway up the condenser.

If the ring is too high, sample may be lost out of the condenser. If the ring is too low, then the reaction will occur over too long of a time period. After the reflux is finished, remove the heat, let the round-bottom flask cool, and shut off the water.

During your laboratory experiences, you may need to run your reflux under dry conditions. To accomplish this, simply place a clean, dry drying tube at the top of the condenser. This drying tube allows the system to be open, while preventing the atmosphere from reaching your solution.

Alternatively, insert a t-connector on the top of the condenser, and connect the other two arms of the connector to a nitrogen source and a bubbler. You may also wish to add reagents to your solution while the reflux is occurring. To accomplish this addition, you will need an addition funnel, which is a separatory funnel with a ground glass joint, a glass stopper that fits the funnel, and a Claisen adapter.

Simply fit the Claisen adapter on top of the round-bottom flask, attach the condenser to the joint directly above the round-bottom flask, and attach the addition funnel to the side arm. Remember to grease all the joints. Alternatively, you may use a three-neck flask in place of the single-neck flask and Claisen adapter.

Just remember to place a rubber septum on the remaining neck of the flask. If your addition funnel does not have a pressure equalizing arm, then you must periodically remove the glass stopper to break the vacuum. Refluxing allows a chemist to heat a reaction over an extended time period, without worrying about exploding glassware or solvent evaporation.

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