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PROFESSOR: If you haven't already viewed Distillation Part 1, Simple and Fractional Distillations, then please do so before watching this video. If you have, then you know that simple distillation is an effective technique for purifying liquids, if the liquids boil below about 150 degrees Celsius. If you have to purify a higher-boiling liquid, however, then you must perform a distillation under vacuum.

The third type of distillation is a vacuum distillation. This technique must be used if the liquid boils above about 150 degrees Celsius, where it is likely to decompose. As you know, the boiling point of a liquid occurs when its vapor pressure is equal to the external pressure. If a system is under vacuum, it has a reduced pressure and the liquid is able to boil at a lower temperature.

To set up the apparatus for the vacuum distillation, insert a Claisen adapter between the distillation flask and head. Liquids often boil violently under reduced pressure, and the Claisen adapter is used to decrease the probability that liquid will bump into the condenser. Instead of using a vacuum adapter, attach a cow or pig to the end of the condenser and place a receiving flask on each joint.

Because the system will be under vacuum during the distillation, you will not be able to replace the receiving flask as you did during atmospheric distillation. A cow or pig allows you to keep the system under vacuum but still switch to new receiving flasks. Next attach heavy-walled vacuum tubing to the outlet on the cow or pig adapter. If the walls of the tubing are too thin, then they will collapse under vacuum.

Attach the other end of the vacuum tubing to a vacuum source, such as a vacuum pump or water aspirator. The vacuum source should also be connected to manometer, a device that measures pressure. Finally, make sure that all joints are greased well. If they're not, solvent may get into the joints and freeze them shut under vacuum.

Apply a thin film of grease to the top of the male adapter and twist the female adapter onto it until this film of grease can be seen all around the joint. Do not apply too much grease to the joint and never apply grease close to the bottom of the male adapter. These actions may cause the grease to seep into the distilling flask and contaminate your liquid.

Before performing a vacuum distillation, you must check for leaks in your system. To do this, make sure that all of stopcocks are closed and turn on the vacuum. Then open the stopcocks one by one, starting from the one that is closest to the vacuum source. As each stopcock is opened, listen for a hissing noise, which implies a leak in the system. If you find the leak, turn off the vacuum, introduce air into the system, regrease the joints, and try again. The system must be free from leaks before you can proceed.

Once you are convinced that the system is free from leaks, fill the solvent trap with liquid nitrogen. This step is essential to prevent destruction of the vacuum pump by vaporized solvent. Then transfer the liquid mixture into the distilling flask and add a stir bar. Do not add boiling stones for a vacuum distillation. Boiling stones are useless because the vacuum removes the air in their pores.

Move the heating mantle up to the distilling flask and place a stir plate underneath. Do not turn on the heat. The first step in conducting a vacuum distillation is turning on the stir plate. If stirring is not occurring during a vacuum distillation, the probability of bumping increases dramatically.

Next, slowly and carefully introduce the system to vacuum. You will see the solution start to boil as low boiling impurities and air run through the apparatus without heat. After the liquid mixture has stopped boiling, turn on the heat. When the distillation has been started, you can determine the temperature at which the solution will boil under the reduced pressure conditions through the use of a nomograph.

First, find the boiling point of the compound at atmospheric pressure on line B of the nomograph. Note the current pressure of your system from the manometer and mark its location on line C of the nomograph. Using a ruler or straightedge, line up these two points and find the point of intersection on line A. This number should be a good estimate of your expected boiling point.

Carry out the vacuum distillation as normal. When finished, remove the heat source from the distillation flask, turn off the vacuum, and allow the solution to cool completely. Then introduce air into the system, stop the stirring, and remove the receiving flask.

Other things to remember when carrying out vacuum distillations are as follows. If you know that you have some low-boiling liquid present in your mixture, first carry out a simple distillation to collect it before attaching the vacuum. If you must separate compounds with similar boiling points, then you can insert a fractionating column between the distillation flask and Claisen adapter.

Unlike distillations at atmospheric pressure, where a narrow boiling point range is observed, it is not unusual to see a 10 to 20 degree Celsius temperature range during a vacuum distillation due to pressure changes. Vacuum distillation allows you to purify high-boiling liquids in a reasonable time period without having to worry about decomposition.

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