

Simple Distillation
Chemistry 233: Organic Lab I

Procedure:

1. Place the liquid to be distilled in a 50 mL round-bottom flask. Add 3-4 boiling chips.
2. Fit the round-bottom flask with a “Y” tube (lubricated at the ground glass joint only) equipped with a thermometer inserted to the appropriate level and a condenser (also lubricated at the ground glass joint only).
3. Clamp the neck of the flask and the condenser to two different ring stands. Clamp the assembly high enough to allow for a ceramic heater with support.
4. After establishing proper water flow through the condenser, heat the round-bottom flask until the liquid inside is boiling. Establish a distillation rate of approximately one drop of distillate per second.
5. Collect the first 1-2 mL (or more, depending on how much sample you started with) in a receiver and discard.
6. At this time, the temperature should level off. Note and record the temperature of the liquid at this point. This is the boiling point. Collect the remaining liquid in the original container (the test tube) as it distills over, careful not to let the still pot go dry. Turn off the current to the ceramic heater and quickly remove it from contact with the still pot (round-bottom flask) so as to avoid distilling to dryness.
7. Seal the test tube with parafilm, and place the test tube upright in your drawer. Turn in an unknown card with the unknown number and the boiling point of the unknown written on it.

Drying with Anhydrous MgSO₄
Chemistry 233: Organic Lab I

Procedure:

Place the liquid to be dried in a small beaker and add what you would estimate to be about $\frac{1}{4}$ mL of solid anhydrous MgSO₄ for each mL of liquid to be dried. Swirl the mixture for about 1-2 minutes, then let the solid settle. Watch the solid as it settles. If the MgSO₄ is free flowing and settles rather gently, i.e. it appears powdery rather than clumped together, then you have used enough. However, if the MgSO₄ is all clumped together, or it has dissolved, or the solution is cloudy, carefully add small additional amounts of MgSO₄.

When drying has been accomplished, carefully add a small cotton wad to the mixture and suction (long dropper with red rubber bulb) the dried liquid through the cotton and deposit it in a liquid vial or other appropriate container. If the product is to be turned in, always place a small amount of plastic wrap over the mouth of the turn-in vial before screwing on the lid. Always label the vial, giving your name, product name, and, when appropriate, the boiling range and grams or mL of the product.