

Lab Techniques

Weeks 1 and 2

Melting points: <http://www.youtube.com/watch?v=T3HzRPKj1YE>

Week 3

Do microscale using directions from the book.

Weeks 4, 5, and 6

Recrystallization: <http://www.youtube.com/watch?v=Q47hTa1KvN0>

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- Do dissolving and recrystallization in reaction tubes.
- Dissolve the solute in the minimum quantity of hot solvent (or solvent mixture).
- Undissolved solids may remain with impure solutes. If this is the case, pour the hot solution into a clean reaction tube to begin recrystallization. (This step will not be needed if the solutes are pure.)
- Cool the reaction tube to begin the recrystallization.
- Use a pipet to remove the remaining solvent from reaction tube in order to recover the purified crystals.

Weeks 7 and 8

Simple, fractional and steam distillation: <http://www.youtube.com/watch?v=3JlIPnyrZMw>

Week 9

Steam distillation: <http://www.youtube.com/watch?v=1OfLPQ-29lw&feature=related>

- The product that you want distills across with the water vapor.

Week 10 - Extraction of Citral

Step 1:

Liquid-liquid extraction: <http://www.youtube.com/watch?v=kNX5wNpuw80>

<http://www.youtube.com/watch?v=vcwfhdhLiQU&feature=related>

- Extract citral from water into tert-butyl methyl ether.
- Add distillate to separatory funnel in small portions
- Separate and discard aqueous layer.
- Add the supposed aqueous layer into water to see if it dissolves, if it does, then it's the aqueous layer. If it doesn't, then it's the organic layer. Keep all layers if you are not sure.
- Dry the ether layer with saturated sodium chloride and discard the aqueous layer.
- Empty the tert-butyl methyl ether layer into a flask, and dry with anhydrous calcium chloride (see video below).

Step 2:

Vacuum Filtration: <http://www.youtube.com/watch?v=9xhwSTaLFKs>

- Remove the calcium chloride from tert-butyl methyl ether
- You want the tert-butyl methyl ether liquid in the flask which contains your product. The calcium chloride remains on the filter paper, which can be discarded.

Step 3:

Vacuum distillation: <http://www.youtube.com/watch?v=dibOJlgAicc>
<http://www.youtube.com/watch?v=mn-u-7fRQv4&feature=related>

- Can do vacuum distillation with a filter flask stopper, and aspirator.
- Remove the tert-butyl methyl ether from citral, leaving citral behind.
- You will not need the cow or the pig. As long as your compound does not boil over, all the solvent that boils off can be discarded in the disposal container. Your compound will remain in the original boiling flask.
- Your vacuum will come from a water aspirator through a vacuum adaptor connected at the end of a simple distillation adaptor.
- Work behind a shield when turning on the vacuum in case the apparatus implodes.
- We do not have a liquid nitrogen trap.