Lab 4A • 10/05/11

{directions for completing lab}

What is the source of hydroxide for part A? The aqueous layer. What is the source of benzoic acid in part B, the bicarbonate extract. Because benzoic is the stronger acid, so it will react with the weaker base, bicarbonate, but naphthol will not.

For part B, you start with ether and the three compounds dissolved together. When you add bicarbonate, you'll form three layers. It is not true that the bottom layer is always water, it's not automatically water. It's because water is more dense that it happens to be the lower layer. Since we're only using ether as an organic solvent, that's why it always on top. So when you add bicarbonate, the benzoic acid that's in there will be neutralized, become more ionic, it will go into the lower aqueous layer, which you then drain out. You'll be left with an organic layer that has two compounds in it. After you reach that point, you add sodium hydroxide. It will react with the weaker acid, the naphthol, which will then go into the lowest aqueous layer, which you drain into a separate flask. So you have two aqueous layers, one that's bicarbonate, and one that's sodium hydroxide. Those will now contain benzoic acid and the naphthol, respectively. You'll be left with an organic layer that has just the naphthalene left in it.

Recrystallization

If you did a melting point right after you recrystallized, you're still going to have water [or solvent] stuck to your compound, which would affect your results.

First, if you had an unknown compound, you'd have to figure out a solvent or a mixture of solvents that has ideal recrystallization behavior. For an ideal solvent – the whole point of recrystallization is that we're trying to remove impurities, but you also want to be able to get your compound back out again. What you're going to do is dissolve it and then make crystals form again. So if you're dissolving it and you hope that it comes back out that at room temperature, or the temperature of an ice bath, just below room temperature, that the compound effectively has zero solubility. Then, at high temperature, close to the boiling point of the solvent, you'd want to have close to infinite solubility. That way, you could heat it up, dissolve the compound, the impurities stay behind in the solvent, you cool it down, and all of it crystallizes back out, your compound, that is. But in real life, it doesn't quite work that way. You won't have infinite solubility when it's hot, and you will have some non-zero solubility when the solvent is cold. An ideal solvent is a solvent in which the solute has effectively zero solubility at or just below room temperature, and nearly infinite solubility near the boiling point of the solvent.

In reality, there will be some solubility at low temperature, which means the minimum amount of solvent should be used. RT is room temperature. In reality, the solute will have non-zero solubility at room temperature, so the minimum possible quantity of solvent should be used. Use two different containers, beakers or Erlenmeyers. Erlenmeyers I like better because it's got a constriction at the top so less chance that something's going to splash out of the container. In one of your containers, put your benzoic acid, the compound that you want to recrystallize. In the other one, you put your solvent, which for benzoic acid, we're going to use water. The reason you want to heat both of these up is reduce how much solvent you're going to use. When you get to the point when water is almost to the boiling point, transfer .5 mL or 1 mL to the solid. How do I know how much water to use? You could calculate how much based on the solubility of benzoic acid in water. When you transfer that water over, there's a few possibilities of what might occur. One, the solid might all disappear. It might dissolve. If that happens, it means you've added enough solvent already, don't add any more. Another possibility is, even with stirring, it won't all dissolve. That means you need to transfer a little more, stir it up, let it heat up again, and keep on doing that until you reach the point where it does dissolve. That last little bit where it bare all dissolves, that means you've used that minimim amount of solvent you should be using. There's a third possibility, though. You could melt your benzoic acid. That's not a problem, but then, when you add water to it, instead of looking for a solid dissolving, you might be looking for two layers in that case. If you have two layers, that means the benzoic acid's melted and won't dissolve yet in the water, there's not enough water around. In that same case, you would keep adding little bits of water unti just barely the point where it all goes into solution.

Small quantities of solvent (water) should be transfered one at a time to the target solid, what you're recrystallizing. If the solid dissolves, enough solvent has been added. If it doesn't dissolve, repeat the process until it does. Watch out for immiscible layers, which means melted, but undissolved, solid is present.

You don't just dump a bunch of solvent over because you might have used way too much, which means either you won't get all of your solid back, or you made a solution and you'll never get your solid back, until you evaporate all of the solvent. If you did, you'd get your solid back, but you'd have all of the impurities left behind, so you'd defeat the whole purpose of recrystallization.

Because you're physically picking up that solvent up with a pipette, it's cooling down for a moment as you transfer it. You want to give it a minute to heat up and try to dissolve it.

Once you've dissolved it, you need to cool the solution down. First let it cool down to room temperature, then put it in an ice bath. This is for two reason. Generally, if you more slowly cool something, the precipitate that forms will have larger, more easily-isolatable crystals. The solid formed may be fine fluffy powder, or more stout crystal, or more like a paste – all three are possible, depending on how the crystals form. [thermal shock of glassware]

After dissolving the solid, allow it to cool first to room temperature, then place the container in an ice bath. Collect the crystals on a Hirsch funnel, then wash with a small portion of cold solvent. If you use too much solvent, you're just going to be dissolving your stuff if you keep on adding. Why use it in the first place? That last little wash can help pull away the last bits of water-soluble impurities you might have sticking around with your compound.

When you're starting to evaporate your naphthalene layers, if you start seeking crystals, very fine-looking, very shimmy crystals form and they start forming up the sides of your flask, that means that you've heated away all of the ether, you've melted your compound, and it's now sublimating and recrystallizing its own self. So that means take it off the heat and stop.

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Recrystallization

Ideal solvent – A solvent in which the solute has effectively zero solubility at or just below RT and nearly infinite solubility near the boiling point of the solvent.
– In reality, the solute will have non-zero solubility at RT, so the minimum possible quantity of solvent should be used.

– small quantities of solvent should be transfered one at a time to the target solid. If the solid dissolves, enough solvent has been added. If it does not dissolve, repeat until it does. *Watch out for immiscible layers (melted but undissolved solid is present). After dissolving the solid, allow to cool first to room temperature, then place the container in an ic bath. Collect the crystals on a Hirsch funnel, then wash with a small portion of sold solvent.