Lab 3B • 10/04/11

{Discussion of lab procedure}

Hirsch funnel

The Hirsch funnel has a rubber adapter that goes along with it to make a better seal with whatever piece of glassware that you're putting that funnel down on. The flask that we're using is a vacuum flask. You're going to hook the hose connector from that vacuum flask up to the vacuum system. On top of the Hirsch funnel, you're going to put a piece of filter paper. The whole point of this is that you want to save the solid, get rid of the liquid. Benzoic acid can be washed with small quantities of ice-cold water. The colder the water is, the lower the solubility of benzoic acid is. We have to be cautious because that solubility will not be zero. The more water we use, the more chance that we're going to have in dissolving the material away. Benzoic acid has low but non-zero solubility in cold water. Small quantities of ice-cold water can be used to transfer or wash benzoic acid.

Invariably what tends to happen is that you will have some solid left behind on your original flask. This is part of joy of finding out that solid products can be a real pain to deal with. How do we get all of that solid out? It might just be that you'll have to get a spatula and manually scrape it all out. What can be done is to dissolve the solid in a solvent, transfer that mixture, and then evaporate the solvent again. Solids are just sometimes a pain to deal with.

{explanation of isolating solid}

If you need ice, you can get it from the stockroom by taking one of these buckets labeled for ice up at the front, take it to the stockroom, they'll fill it for you.

Do not use ether to [wash] benzoic acid because you'll defeat the whole purpose of this step, because you'll just dissolve the compound, you'll lose the compound.

{measuring mass of flasks ahead of precipitations or evaporations}
{taring of flasks}

It is advisable to tare – measure the mass of – a flask before use, so that an accurate mass of the product can be obtained.

Drying agents

Drying agents are generally dehydrated hydrates, particularly compounds like calcium sulfate, sodium sulfate, zinc sulfate, those are three of the most common ones. Why a dehydrated hydrate? Because those compounds end up being hygroscopic, they like absorbing water. They might even be really, really good at absorbing water, in which case we call the desiccants, a fancy term for drying agent. Drying agents are usually dehydrated hydrates, such as magnesium sulfate, sodium sulfate, calcium sulfate, and zinc sulfate.

What's the purpose of a drying agent? It's not to get rid of liquid. There's this association between the word dry and lack of liquid; but no, dry means lack of water. You're not going to see the drying agent dissolve. If it were going to dissolve, it would have be in water, because they're ionic compounds. You see the solids disappear, and they won't remove all of the liquid. It's just that, if you accidentally let some of that aqueous layer came through and got in your organic layer, that's why we're using the drying agent. It's used to remove water, not just any liquid, from an organic solution.

How do you know if you've added enough drying agent? I can't tell you because I'm not inside that solution watching the water molecules disappear. What's the evidence that it's done? Sodium sulfate, in particular, is a very fine, granular powder when dry. When it starts interacting with water, it's going to start clumping together. if you put the drying agent in and swirl it around and nothing happens, it just says a powder, that means you're lucky and maybe had a dry solution to begin with. You may never see, then, firm evidence that it's drying. If it doesn't clump, it means you might not have had enough water for the clumping to occur. If add the drying agent and it starts sticking together, you know it's absorbing water. How do you know it's done? After swirling around, if you have any of the drying agent left that didn't clump together, that's still a nice, fine powder, then that means there was enough there to finish absorbing the water and enough left over that it's a powder. If you add the drying agent and swirl it around, it clumps together and there's nothing left that's powder form, then you to add more drying agent, until you get to the point where, when after you swirl it, it is powder.

Usually, a powdered drying agent will clump together when it's exposed to water, especially when it's in a organic layer. If, after drying has occurred, if after swirling in the solution for the recommended period of time, after exposure to water, if there's till drying agent that remains in powdered form, that means there was enough excess drying agent, and so drying is completed. If you only have clumps of drying agent present, you need to add more drying agent to ensure that it's done.

You'll start with your organic layer. You'll add in the sodium sulfate and swirl it around. You're checking for clumps. If you never from clumps, that means it was dry, so after a few minutes of swirling with the drying agent, just move on. If you form clump, make sure that you till see some powdery drying agent, otherwise add more. Then, you're going to filter off the drying agent. This solid you don't want at all, trapped solid that's dissolved in the ether. You're going to use a gravity for that.

{filter paper and pipette supplies}

You grab just one of the four leaves of that fold. {refers to filter paper}
{trying to remove drying agent to get an accurate mass measurement}

{evaporation of solvents}
{auto-ignition}

If you get it up to that temperature, it just spontaneously catches on fire, without having a spark or anything, it just starts burning.

{hot plates}

A device that's got a heating coil in it that heats the plate that you can set stuff on. It's also got a magnet buried in it. When that magnet turns, if you had another magnet that was inside your beaker, the larger magnet causes the one in your beaker to spin, so you can end up with a consistent stirring rate without you ever having to physically touch it. Remember to retrieve these magnets when you are done; because they are so small, they are one of the most easily lost pieces of equipment. Because the surface can be awfully hot on contact, it's better to diffuse that heat, a sand bath. The sand bath takes a while to heat up, but it also dissipates and retains that heat. You bury that piece of glassware in the sand; the further you push it in the better heat contact you're going to get.

{hot plate locations}

If you're using a beaker or something with a flat surface, I recommend a stir bar, something more geometrically compatible.
Water, you have to put a lot of energy in to remove water, but you could do it another way, which still requires energy. Boiling point is the point at which vapor pressure reaches atmospheric pressure. At atmospheric pressure, ether's got a boiling point of 34.5° C. But if you drop the pressure, vapor pressure doesn't change, it keeps on plugging along. If you lower the surrounding atmospheric pressure, you're lowering the boiling point. Ether you could remove just by putting it under vacuum.

{description of reduced-pressure evaporation}

You need the flow control valve because if you take a solution and suddenly expose it to vacuum, especially if it happens to be a good vacuum, you may get this instantaneous form of boiling called bumping, where the whole solution just bubbles up at one, which means it could get dragged into the vacuum system without having evaporated, which means you're going to lose your product as well. Use the flow control value to gently introduce the system to vacuum. For ether, you wouldn't even need to heat it, but you would still have to stir it to prevent that bumping from occurring. The more agitation that you have, the more it's going to evaporate.

The vapor pressure of a liquid only depends on temperature, not on any external factors. If you lower the surrounding atmospheric pressure, you drop the boiling point, since not as much of it has to be turned into vapor in order to boil.

{Part B}

{Presentation identical to lab 3A}

When carbonate or bicarbonate react with acid, you make carbonic acid, which is not thermodynamically stable at room temperature and pressure. When you do your separations, vent a lot more with the carbonate than you did the sodium hydroxide.

{Emphasis of secondary containment on acid/base cart}

Since the compound that doen't want to give up the proton is trying to react with the base that doesn't want to get the proton, this reaction does not happen. That's the point of this part of the lab: if we have three compounds that are mutually-soluble, we could use a weak base to target the stronger acid, leaving the weaker acid alone.

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Aqueous (NaOH) layer – acidify until a precipitate forms and the sol'n becomes acidic.

Benzoic acid has low – but non-zero – solubility in cold water. Small quantities of ice-cold water can be used to transfer or wash benzoic acid.

It is advisable to tare (measure mass) the filter paper before using so that an accurate mass of the product can be obtained.

drying agent – usually a dehydrate hydrate, such as $MgSO_4$, $NaSO_4$, $CaSO_4$, $ZnSO_4$. used to remove water (not just any liquid) from an organic solution.
– usually, a powdered drying agent will clump together in organic solution when exposed to water. It, after swirling the sol'n for a few minutes, clumps have formed and there is still powdered drying agent remaining, it means there had been enough drying agent to remove water. If only clumps are visible, more drying agent should be added to ensure complete dryness.