## Lab 8A • 10/24/11

{lab report due dates}
{lab report format}
If that Rf value for an individual spot matched a spot that's in your compound, that means that that reference compound is in your unknown compound.
{forms of data}
The solvent front distance for each plate, if you developed multiple plates. Then, we have the compound distance for the different samples. If you end up with multiple spots for you unknown, that doesn't mean something went wrong, it just means that you have more than one compound that's in your unknown. Solvent front distance means from the starting line, how far did the solvent travel through up to the top of your TLC plate. You don't measure from the bottom of the plate, you measure from the starting line, cause that's where the solvent first interacted with your compounds. Wherever the solvent stopped, that's the solvent front. So you measure the distance between those two. The solvent front distance is independent form the known or unknown.

It's a perfectly valid piece of evidence to say that you see that glowing dot in your unknown, that's a good piece of evidence that that compound was in your unknown. You can use the colors of the dots themselves. If you have something that glowed unusually under the UV lamp, that just happens to mean that compound's UV reactive. If you happen to see that same bright spot again in your unknown, logical deduction to say: since that's the only one of my reference compounds that glowed like that, if it glows like that for the unknown and you have the same Rf value, two pieces of evidence that that might really be the compound that's in your unknown.
{calculations}
Rf value is the distance the compound travels versus the solvent. The Rf better be less that 1, because nothing can travel faster than the solvent. Nothing should have traveled the same distance as the solvent.
{errors in measurement}
For example, if you had a TLC plate and your dot was this big relative to your place, and if the starting line was here, the bottom of the dot might have a value of 0.5, but the top might have a value of 0.9, so anywhere in there could theoretically be what the Rf value should have been.

Compound distance would be how far does one of your reference compounds travel from the starting line up to wherever it ended. Whatever crap you might have had in your development chambers may have ridden up with the solvent and made a haze across the top of your TLC plate along the solvent front. Normally, you measure right in the middle of the dot, as much of the middle that you can determine. If you have a dot that's shaped like this, you might end up with some really strange-shaped dots. This might be one of two things: you either have a compound that's partially stuck to plate; another possibility is that you've got some by-product. If you have a long trail, then you have to do the best that you can to figure out where the center is.

Check the bottom of your plate. Maybe your starting line was tilted, or maybe the TLC plate itself was tilted. You still gotta measure from the line to the solvent. If you had a situation like this, if you drew your starting line straight but your solvent line was crooked, then you'd have to look at the bottom of you TLC plate. If the bottom of your TLC plate is also crooked, then go ahead and measure just from your starting line, straight up parallel to the side of the plate.

{handling chemicals for lab}

A seal is only a seal if you make a seal.

An Sn1 mechanism. If you had a tertiary alkyl halide, then that halogen could come off (with enough thermal energy). If you had water as a solvent, water could now be the thing that comes and attacks in, and then we've got a deprotonation step, ending up with an alcohol. We're about to do exactly the opposite. We're going to take this starting alcohol – 2-methypentan-2-ol, also sometimes called t-pentyl alcohol – and react it with concentrated hydrochloric acid, HCl. It's going to make t-pentyl chloride. Does that mean we can cause this reaction to go either direction? Yes. Soon, you're going to need to know that sometimes a reaction step is reversible, meaning that there's an equilibrium that can happen for an individual reaction step. In this reaction of concentrated HCl with an alcohol, the first step of that reaction will be the alcohol attacking an H+. That's not necessarily an energetically-favorable process, since you're going to be making an oxygen that is plus charged. As soon as that hydrogen hops on, it can fall bak off again. This is our first use of a reversibel reaction arrow. For clarity's sake, let me contrast this arrow with another arrow that also points in two directions. Notice that the arrow I used in this reaction, the mechanism, has an arrow pointing one way up top and the other direction on the bottom; they're two separate lines to show those two different directions. The other arrow is only one line and has arrowheads at both sides. That's used for resonance structures. It's not allowable to swap those arrows. When talking about a reversible reaction step, make sure to use this double arrow, whereas for resonance structures we use a single but double-headed arrow.

–OH group attacks plus charge. Not necessarily favorable, so the oxygen tries to become neutral again, which means the hydrogen could fall back off and we go back to the start, or, every once in a while, maybe, instead of the hydrogen coming off, the carbon-oxygen bond will break. If so, that's going to leave us with a carbocation. If there's a lot of water around in solution, it could be possible for water to come back and attack. But because water is a separate layer in this reaction, it may not be the thing that predominates. Because we have concentrated hydrochloric acid, there's going to be a lot of chloride around, which means it's possible for chloride to attack instead, which is how we end up with our product.

We're doing this reaction neat, which means not using a solvent. You'd have two different layers that are going to form during this reaction. Your aqueous layer, and then the product, which is not going to be polar enough to be water-soluble. In all of these cases where we form multiple layers, which one's going to be on top? Your organic layer, which is your product, or the aqueous layer? How do you know it's the organic layer? Usually, organics are less dense than water, so normally you'd be right, and today you'd be right as well. The product has a density of 0.866 g/mL, less dense than water, it should be your top layer.

{reaction procedure}

A wash is a treatment of the organic layer where you care about the organic layer, not the wash itself. Let's say we did this reaction, but let's say there's some non-zero trace of the hydrochloric acid that stays in the organic layer. You might want to get rid of that before you try to get rid of your compound, just in case that extra acid has some kind of negative effect on your compound. You can shake the organic layer in a mixture of sodium bicarbonate; the bicarbonate would react with the acid and leave you with water and carbon dioxide, which would then combine with the aqueous layer that the bicarbonate was provided in. It's effectively going to remove the excess acid from the organic layer. But you don't care about the aqueous layer; that's just what you had the bicarbonate dissolved in, and that's what's taking some of the impurities away. Once you've mixed it all together, you're going to drain that aqueous layer out and throw it away and only keep the organic. That's why it's called a wash. You start with the organic, you add something else, you mix it up, then you drain and keep only the organic. Then you do it with the next wash, separately – add it in, shake it, drain it out. The next wash, the same thing, each time draining out the aqueous layer, so the aqueous layers do not get combined with each other.

Your going to start with the organic in your separatory funnel. You're going to add the wash; the organic layer, since it's less dense than water, should float on top, the aqueous layer should be on the bottom. You add the wash in, put the stopper on, shake, vent, shake, vent, shake, vent, just like we did for extraction. This is like extraction except you're not extracting anything except byproducts. After you've added it and then shake and vent, then you drain and dispose of the aqueous layer. The first one you're using is saturated sodium chloride. Saturated – the term means you've got the maximum amount of sodium chloride in that solution possible. Why are we doing the wash with sodium chloride? Because salt makes the aqueous layer more polar, so this helps to pull out any really polar impurities from your organic mixture. What if you do the reaction and the layers won't separate? That might happen if you've got something in that aqueous layer that might be slightly organic-soluble, so maybe the compound is bridging the organic and aqueous layers. Or, it could be that the solvent you're using is not completely hydrophobic. If you need help separating the layers, sodium chloride is there to increase the polarity of the aqueous phase, to make it even less organic like, then the layers will more easily separate. [Also used to flood environment with chloride and ensure removal of product].

What's the second wash, the bicarbonate? Why do you think we're using the bicarbonate? To remove excess acid. Your third was is a water layer. You're using just pure water to have one more chance to remove aqueous-insoluble impurities.

{work-up description}

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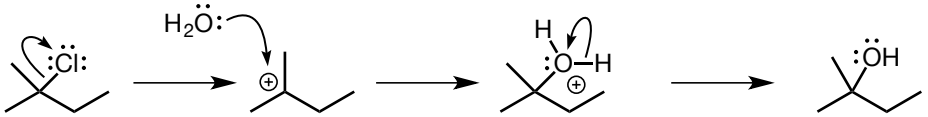
neat – without solvent

1) saturated NaCl – used to increase polarity of water layer
2) $NaHCO_3$ – used to remove excess acid
3) $H_2O$ – one last chance to remove aqueous impurities

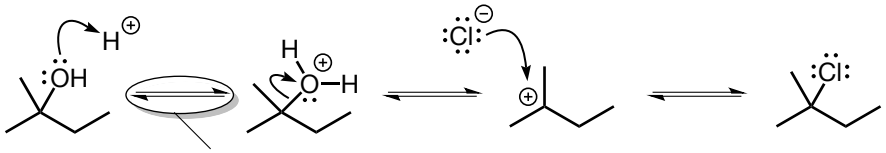
**Structures**

***10/24/11 lab • 1***




***10/24/11 lab • 2***

*reaction of 3° alcohols w/ HCl (conc)*




*reversible reaction*

***10/24/11 lab • 3***



*used for resonance structures*