Lab 6A • 10/12/11

{lab report dates}
Lab reports must be typed or computer formatted, generated. {lab report formats; deciding to require PDF} I prefer e-mailed lab reports; it cuts down on the number of trees we're wasting. {describing how to make PDFs on Macs and Windows; PDF4Free} Please do not both e-mail me and give me a printed copy. If you're going to e-mail me a lab report, please don't print one. There might come a time where you might need to do a little of both, because at some point we're going to start getting print-outs of spectra. Since not everybody has a scanner, I don't make you scan spectra. If you wanted to, that'd be great; then you could have an all-electronic report. But if you didn't, that would be fine to do part of you write-up electronic, but turn in just the spectra in paper form. [should we set up e-mailing from the instruments?]

If you e-mail a lab report, I am paranoid about grades. There is this thing called FERPA, which, to boil it down, says I can't realize grade information to anyone except you with your permission. E-mail is an insecure form of communication, meaning I can e-mail you something, but then if your brother, sister, girlfriend, boyfriend, father, mother, somebody gets on your computer without my knowledge, they could potentially see that grade. If you send me a lab report, you need to send me something that says, effectively, I agree to receive lab grades by e-mail. What you will be agreeing to by that, if you send me that sentence, if someone breaks into your e-mail account, you're not going to hold me responsible if they see your grade information. This may sound a little paranoid, but we are sue-happy in the US, and I don't feel like being sued for something that trivial, a ten-point assignment. But it is still a grade, and it is still your private information, so if you want to have e-mailed grades, you need to tell me that. Otherwise, I can easily give you the grades and the feedback here in lab, so that no e-mail is ever generated.

To the format of the lab report itself. This is going to be a somewhat reduced form of what would normally be generated in research. A real presentation of research appears in what is known as a peer-reviewed journal article. Peer-reviewed means your peers, your contemporaries, the other people working in the field, they have a chance to read whatever publication you're going to make before it gets published. So, you do some research, you think that you invented something cool, you send it to Journal of the American Chemical Society (JACS); Angewante Chemie, very popular journal from Germany; a number of chemical societies around the world publish journals that contain research. So you send it to one of these journals, they send it out to different reviewers. They comment on it; they say, "is this research valid? are the procedures sound? was something worthwhile actually discovered, or is it just filling up paper needlessly? They go back and forth and examine it and they send it back to you with comments. Very rarely does something get accepted the very first time; they might send even just minor comments; they might as you to rewrite part; they might as you to do more research first. Eventually, once you've got a document that they're happy with, then it gets published in some form of journal, a publication. That collective body of all of those journals is called the chemical literature. That's where most discoveries end up getting published – unless it's some super-secret governmental or business thing in which case they won't publish the results because there's some dollars or national secrets involved.

The structure of these reports usually works like this: you start with a one-paragraph abstract that just summarized the whole thing, so if people are flipping through just to see what new's happened, they can read the one-paragraph synopsis and get what they need from that. The more complete article itself would first present the history of the research – who had done research in this field previously, what was important about that research, how does this project that you just did add to that knowledge, and why is it valuable, therefore. You're not only speaking to the chemistry of it, but to be realistic, or maybe pessimistic, you can't do research with out money. So at the same time you're publishing your results, you're also letting the grant writers, the people who are giving you the money, know why was their money used for something good. After that, we then have a full, exhaustively detailed procedure. You then list all of your various forms of data and any calculations that you made. You draw whatever conclusions from that data that you can, and then you'll often discuss what further research what you just discovered will enable. That sets you up to be able to do more research and set you up to possibly to get more money to do the research.

What you did in here with the extraction, none of us are going to make any money off of. And, we're not going for a peer-reviewed journal, so we don't need quite all the trappings of a traditional full report. Here's what you're going to have: a purpose statement, so that you can tell me in a very short way what was the main point of the lab. You'll present your forms of data, and you'll have some calculations. {advisement that lab format is simplified} After the data and calculations, you'll have a conclusion statement, which is effectively a parallel with your purpose statement. In speech classes you might have heard something like this: if you're going to present, tell 'em what you're going to tell them, tell 'em, and then tell 'em what you told them. Purpose is: here's what I'm going to tell you; data and calculations: here's my experiment; conclusions: here's what the point of the experiment is. After that, then there's the chance for discussion. I do not have you discuss theory on the lab report; that's what the lab quiz is for. On the lab quiz, you will be addressing those kinds of issues; but, keep it out of the lab report. That lets the lab report be a bit shorter, you have a little bit less work to do all the way along[, and it allows you to devote more time to the lecture material].

Let me go over each of those sections again in a bit more detail. The point of this lab – from a chemical perspective – has nothing to do with learning extraction. The way the lab text is written, you might have gotten the impression that it's all about learning extractions. Yes, as students, you're learning how to do extractions, but when you're writing any kind of chemical report, the person on the other end probably doesn't give a damn whether you learned something or not. They're expecting that you already know it and know how to do chemistry well; they're wanting to look at what is the research that you did. Because that's the way reports are generally written, we're going to match that in here – which means, any reference to learning, or anything that you learned how to do in a technique, leave that out. That goes along philosophically with how lab reports or formal reports are generally (although not always) written. They're usually written entirely from a third-person perspective, meaning there's no "I", "me", "our", "we", any of those first-person words; there's no "you", "yours", "y'all"; none of that, it's all "it". [This might contradict what you might have learned in English class: namely, to avoid passive constructions, such as "the book was read"; [instead, say things like] "he read the book", to put the focus on the person. That's exactly what you don't do in a research paper. You do do it in a passive way: "the mass was measured", "the results were obtained". There's no person involved with that, because the philosophy behind that is it is focusing on the science. That science is something that's logical and is above whatever human emotion we might put into it. But some people don't like that philosophy. Some people think: no, we as humans do the work, we should be honored for that work, and we should be mentioned in whatever publications that we make. Most chemistry publications follow the impersonal form – no mention of the person, so no first or second person. Tied with that, that's why you as students, although you're learning techniques, don't talk about the leaning process in your learning statement. {commentary on evaluation of purpose statement in Chem 1A}

The purpose statement is [instead] a concise statement of the qualitative or quantitative goals of the experiment. Qualitative means without numbers, so you're looking for a precipitate, or you're looking to synthesize something, or to see if bubbles form, or something like that – that's what qualitative means. Quantitative means your goal is to get the melting point, the boiling point. {purpose statement for preset lab; the measurements may not in themselves be the goal, but instead may just be pieces of data used in obtaining the primary goal].

Yes, in any real report, we would write down a fully detailed procedure. But, you already did the procedure for your pre-lab. Why would I make you type it up again? If I was really being proper, I should say put some biblographic reference to your text in the lab report, but I'm not going to make you do that. You know the procedure, I know the procedure, it's from the book, I told you which pages from the book, so why do we have to do this little dance where you tell me what I already know. So, don't even mention procedure.

Your next section is going to be data. The sentence: "the data indicates that a good result was obtained"; what's wrong with that sentence? The data indicate a good result was obtained, because data is a plural word. If you say the data is, that's bad grammar. The data are. Data comes from datum, a Latin term; datum is the singular. So, this datum is good, these data are good. {commentary on proper use of data} What are data? How is it distinct from calculations? It's whatever you directly observe, whatever direct qualitative or quantitative observations are made, without any form of mathematical manipulation. As soon as you start mixing numbers, adding and subtracting, those are not data, those are calculations. Data are direct qualitative or quantitative observations that require no manipulation. Organizing data into a table – that's not a calculation, it's just organization. Printing a graph our from a set of data – that's just another representation of data, so that would still classify as data as well. The reason I make such a distinction between data – wich are the observations – and calculations – which are your manipulations, is that there's two types of mistakes you can make: you might have an experiment that went really well, a titration where you got perfect results, but you accidentally take the wrong numbers and put them in the wrong places in whatever equation that you're using. If you separate out and first show me an equation and then show me how you substitute it and have that all separate from your data, then I can [more easily] tell where a mistake was made. {reduced amount of calculations in o-chem}

{description of data in experiment}

Most of the time in this class, even if you have a bad lab experience, but you write it up correctly, you'll get full credit. Do you know how to write about what happened; that's what's important to me. If you make a mistake, if you spilled some compound and your data look bad, I "won't care" as long as you carefully notate what happened.

{masses}{recommendation for constructing a table}
Since all you're taking is masses, it wouldn't look as good on paper to just say, mass of mixture A, mass of this compound, mass of that compound, cause then you've just got a grocery list; visually not very attractive, makes it a little bit slower to interpret those data. But it into a table and organize the information, much more compactly presented, much more logically presented. If I pur"grams" at the top like that, I don't need to write "grams" every single place I write a number, again it makes it a little bit more visually appealing.

You always, always, always have to put in units, either at the top of the table or each number. You can never presume that someone knows what units you're using, ever.
{proper writing of formulas}{need to be able to make subscripts}{H2O, not H20}

These are two perfectly valid English words. When we have [multiple] combinations that can be valid [homonyms], that means your spellchecks won't catch it. So I get all the time people talking about trail one and trail two , which means they had a good outdoor adventure. It's trial 1 and trial 2. {more description of lab data}

Let's move on to calculations. Calculations are any mathematical manipulation of data. What is a piece of data and what is a calculation. If you take a watchglass and get it's mass, then you put a compound on it and you get the combined mass. The mass of the compound itself is a calculation, because you did not observe it. You observe the watchglass; you observed the combined mass, and by different you got the mass of the compound. But that means, the mass of the compound is not a piece of data. I will let you omit calculations for mass by difference.

% composition – mass of the compound divided by the total mass, time 100%. % yield – the mass measured divided by the theoretical mass; how much did you get versus how much should you have gotten; multiply that by 100%. % yield we'll use in reaction, and we'll calculate the theoretical mass by a molar calculation. % error – experimental value minus the theoretical value, all divided by the theoretical value, times 100%. Some people express this equation with an absolute value, but if you put the absolute sign, you're always have to explain what the value is, since the sign cannot be determined from the number itself. If you get a positive error, you know your value's too big, and if you get a negative error, you know you're value's too small.

Here's the way you need to format calculations. First, write out the equation in word or symbols. If you put variable naes in that's fine, but otherwise write it out in words, just like this. This is so that I know that you know what the formula is supposed to be. If all I see is numbers with no explanation and you do it wrong, it's hard for me to grade because I don't know which part you don't know. So, write the equation out first. Take a set of data, put it in that equation, write out and solve the equation, show the substitution, but only do it once. If you do the same kind of calculation multiple times, after the tenth time you've shown me the substituted equation, am I really learning anything new. Only do a substituted equation once, then make a table for all of your results. Sometimes we can blur the line between data and calculations. If we're already go a nice table set up with the samples labeled, I've got the masses, what if I threw another column in. So I could present everything as one compact block, I could include the results of the calculations in the data table. You can bend the distinction between data and calculations if you do it in a logical way.

Any time you measure a value in here that's known, that's in the literature, you've got to compare your result to it. {discussion of melting point in lab} For melting point, percent error must always be done in Kelvin. Why do we have to use Kelvin? There's physical problem and a mathematical problem. What's the normal freezing point of water? 0° C. So if I measured it as 1° C, what would the percent error be? You can't calculated it because you'd be dividing by zero. But you know it's a perfectly valid observation, but you can't use Celsius, you can't use Fahrenheit, you can't use relative scales; you have to use an absolute scale, which is what Kelvin is. Always do your errors in Kelvin. That way, also, if you do state a percent error, someone else, who knows the value is really supposeed to be in Kelvin, will know what the magnitude of the error really means. If you did end up with a melting point reagent, the normal process is to average the extremes of the dane, and make that average your melting point. If you did multiple trials, average those to do your percent error.

Conclusion(s) – This should be a summary of the results of an experiment that addresses the goals as presented in the purpose statement. In other words, if in the purpose said that the goals was the percent composition, then in your conclusion, state the percent composition. {examples}

The last section would be discussion. Let me talk about what the discussion section and is not. If you want to talk about theory, you can, but be careful, because when you start talking about theory, I have to mark you off if you say things that are incorrect. It is not true that the heavier the lab book, the better. Source of error. Of course, if we were in a physics class, we'd sit down and figure out what is systematic error, what's random error. Of course everywhere we do a mass measurement, the balances could be miscalibrated. But I don't want you to tell me, because did you sit down and calibrate the balance? Did you check if the balance was calibrated or not? This is not physics class, so we presume our equipment works correctly. Human error. I'll see sentences like this: the % recovery might be artificially low because of human error. Then fire the human and get a better one! That's what your boss would do, so be careful what you're writing there. What do you put in the discussion section? If you did have some literature result that you're comparing to, this would be the place to mention it. If you have something that definitely went wrong, then you could mention it, but don't throw things in there just to make a discussion section. If you're lab ran beautifully, perfectly, nothing went wrong, you got 0% on your melting point, maybe you won't even have a discussion section.

Chromatography

Did any of you as a kid do the type of experiment where you took a paper towel and you put a big ink blot on the paper towel and then put the paper towel in water to see what happens? You did? My one person – yay! What happens when you do that? The water crawls up the paper, and the ink moves with it. But does it stay in one spot? Depending on the type of reaction, you might see the one dot turn into several. That is exactly an example of chromatography.

Chromatography is a technique by which a mixture of compounds can be separated due to differences in their polarity, size, or charge. To simplify our discussion, I'm going to stick to just polarity today.

Let me show you an example of a type of chromatography. This example will actually be for gas chromatography. Imagine that we have some kind of tube, and that the inside of that tube (not the outside) is coated with some form of material. For the sake of argument, let's say this is some type of hydrophilic material, something that's maybe very polar in nature. Imagine now that we allow vapor to pass through it. That vapor might contain a mixture of compounds. If the compounds themselves are polar, they're going to end up interacting with this polar coating. As the progress through, they might get slowed down moving through, because some of the time they're getting attracted to the coating. A non-polar compound, in this example, if it passes through, it will do just that – it will tend to pass through, without as much interaction. Because of this difference in polarities, because of the difference of the interaction with those compounds with that coating, you can therefore separate the compounds out. The different forms of chromatography all have the same kind of idea: you have a stationary phase that contains some kind of separatory material that can separate the compounds on the basis of polarity. You'll then have a mobile phase. In this gas example, you have nitrogen, which is a non-reactive gas. You may make that to carry the volatilized compounds through the system. Different kinds of chromatography use different kinds of mobile and stationary phases, but it still some kind of action of the [mobile] phase passing through and separation can occur.

So the stationary phase is an immobile phase that contains a material that will separate compounds that are passed through it. Support – means a platform or a structure or a piece of glass or metal tube or something that provides the foundation upon with this compound that does the separating is applied. It's an immobile support that contains a material that contains a compound that can separate on the basis of polarity. Think of it like hydrogen bonding. You're going to have a material that has a bunch of –OH bonds on the surface of the material. If I pass an alcohol that also has the potential to form hydrogen bonds over that, the alcohol will get tangled up, you could say, distracted by that separatory material. You've got some solvent that's pushing it through also, so it won't get permanently stuck, but it will get slowed down, which is the whole idea behind how compound end up being separated out. An immobile support that contains a compound that separate the components of a mixture passed through it. The mobile phase is therefore a non-reactive gas or solvent that carries the compounds through the stationary phase.

Let me review this example as an example of gas chromatography. In gas chromatography, it's exactly this situation. There's a long tube, usually called a column. That column may not be like what you're thinking about, a structural building column. A column means long tube – so long, in fact, that it's wrapped up in a loop. In that column, you pass a gas, either nitrogen, argon, helium, some kind of gas that's non-reactive, and, at the point where you put your sample in, there's a heating element that vaporizes your sample. So it turns in to a gas. The carrier gas, it's called, pushes that through the column. As the different compounds interact differently with the surface of the column, they will come out of the column at different times. A sample is volatilized, which means evaporated, and passed through a column, a tube, that is coated on the inside with some form of separatory material – that is the stationary phase. The sample is pushed through the column by some form of inert gas called the carrier gas – that carrier gas is the mobile phase.

Column chromatography – works like this. We have some kind of tube, might look much like a pipette. Might be the same size as a pipette on really small-scale applications. On larger scale, it might be a few centimeters across, half a meter tall; a substantially larger column, still same general principle. At an industrial scale, it might be several meters tall and a meter wide, a huge kind of separatory device. In all of these columns, it works this way: you pack the bottom of it with sand or rock or something non-reactive that provides a nice flat surface. Usually, you finish off with sand so the bottom of the column is perfectly flat, because you're trying to separate these compound out by how far they travel through some kind of material. Generally, for these columns we use silica gel, which is essentially just pulverized glass. That pulverized glass has a somewhat polar surface. The difference between polar and non-polar compounds passing through this, that's what's going to separate the compounds out. When you prepare a column, you put all of this material in there, and then you load the sample. You put your compound mixture at the very top of the silica gel and make a layer. On top of that, you force through a solvent. You'll fill it up with solvent, and then you'll often add air pressure to push the solvent through the gel. The solvent first mixes with your sample, dissolves it up. It starts carrying it through the silica gel. The different compounds will flow through it at different rates because some of them will become entangled or interactive with the silica gel; others won't. As the solvent comes through, you'll use a series of beakers or test tubes to collect the eluent (the stuff that comes through). From that, you can separate your compounds.

In this case, you don't have a coating. This entire thing is filled with powdery substance. To load the column, you put that silica gel into some sort of solvent. It won't dissolve, but it'll make kind of a slurry. You pour that into the column, tapping the column as that stuff settles down; you get a nice layer of material. If any of you have ever used diatomaceous earth in pool filters, same kind of principle.

They're not trapped, because otherwise you'll never get your compounds out. But they'll interact somewhat more aggressively than others. It's just electrostatic interactions. Think of it this way. Static electricity; not the same phenomenon, but close enough parallel.

You've get something that's got static electricity, two things will stick together, but then they'll come back apart again. That's what's going on in this case. Two really polar materials will associate with each other for a brief period of time, since they're both polar, but if you have a solvent that keeps on pushing through, eventually that solvent will make the compound move further on. There's different kinds of materials for different applications. Silica gel is one of the most common ones.

TLC. It is not tender loving care; it is thin layer chromatography.

Why is it called thin layer chromatography? Because it looks just like this. Usually have some kind of plate that has got either a plastic or glass backing to it. That plate is covered with some kind of material. This white material on here is silica gel, is some of this moderately polar material that I keep referring to. If you think of the paper towel example, if I put a spot of something down on the bottom of this plate, place it in the solvent, allow the solvent to crawl up the plate by capillary action, then that sample will brought along the place, but maybe some components in that sample will stick more to the plate and travel more slowly, and other components won't interact with the plate so much, and will move through the plate more quickly. That is what happens with thin layer chromatography.

In thin layer chromatography, you're going to put some compounds onto a plate. You have them on a starting point on that plate. You then allow solvent to pass through. Based on how much the compounds interact with the plate versus the solvent, compounds will travel different distances up the plate. When the solvent has reached the top of the plate, then you stop the development process. Why? Once it has reached the top of the plate, different dots will have travelled different distances. What we're going to calculate is difference between how far the solvent travels, versus how far each compound travels. From that, we're going to calculate a Rf value, which stands for retention factor. It is equal to the compound distance (c) divided by the solvent distant (s). If you have the same compound developed in the same solvent on the same kind of material TLC plate, Rf values are reproducible. What's so great about that? Here's a scenario. Let's say that we had a pain-killer tablet, an analgesic tablet. Analgesics tend to contain a limited number of compounds. Some of them are aspirin based, some of them will have acetaminophen in it, or acetyl salicylic acid, or a decomposition product of that, salicylic acid, or maybe you'll have other compounds. In with that, some of them have caffeine.

What if we wanted to analyze on of these tablets? We could arrange a selection of reference compounds: get some pure acetylsalicylic acid, ibuprofen, caffeine. We put each one of those reference compounds on a TLC and run it, get those Rf values. Then, we take out tablet, crush it up and dissolve it, filter it, take the mixture from that, put that on a TLC plate and develop it. If you end up with a plate where one of your target spots is at the same level as one of your reference compounds, that let's you identify what your pot is in the mixture. That means that we'll be able to identify, simply by separation, which of these compounds are in your analgesic. The reference compounds, you run those to establish Rf values for each of the individual compounds. You run the mixture, and you figure out the different Rf values for the multiple spots that you might get out of it. Where you get a match in Rf values, you've identified one of the components in that mixture.

Preparing, developing, and visualizing the plate

Preparing TLC plates. I need a starting point line on this plate. Why? Because if I'm measuring different distances, I need a common reference point. To do this correctly, you would hope that the botom of the TLC plate is perfectly flat, and we need to draw a flat line across it. You've got to be careful with a TLC plate if you are the type of person that write with a lot of pressure, because you could just scrape the silica gel off the silica plate. So if you write too hard, you scrape it off; if you scrape off enough of it, the solvent won't pass through, because it's like you've made a barrier, so you've ruined your TLC plate. So, don't write too hard. Also, you must use pencil. Why? If you put pen, and you're going to pass an organic solvent through it, you're going to dissolve that ink and it's going to crawl up the plate as well. Your compounds are going to end up being transparant; the ink will therefore mask the presence of these compounds. You must use pencil, and don't use too much pressure. To prepare the plate, you draw a line in pencil 0.5 to 1.0 cm from the bottom.

At that starting line, you're then going to use micropipettes to make little spots on your TLC plate. We cannot use normal pipettes because the spots that they would make would be gigantic, very likely would cover from one side to the other of the plate. If the whole point of this technique is that we're trying to separate compounds and measure the distances that they travel, if you've got a blob that's huge, where do you measure the distance traveled from? The top of the blob, the bottom of the blob, the middle of the blob? So if you have a small, compact dot, that makes it much easier to analyze a compound. Depending on how good your technique is, depending on how large the plate is, you could squeeze in maybe three, four, five different compounds on one TLC plate. If you can't them all on one, it doesn't matter, because if you're using the same kind of plate and the same kind of solvent, you're going to get reproducible Rf values, so it doesn't matter if they all fit on one plate. The plate is then spotted with one or more compounds.

Development. We have different TLC solvents, which are some of the same solvents you might use in extraction. This chromatography technique is based on polarity. Let's say that you have a mixture of carboxylic acids that needs separating from each other. Carboxylic acids are some of the more polar functional groups that you can run into. Particularly, the can experience a lot of hydrogen bonding.

If you have silica gel which has a lot of –OH groups on the surface of that gel, and if you're passing a carboxylic acid over it, it may be stuck (not permanently) on that plate. If you don't use the right kind of solvent, like a very non-polar solvent, more polar functional groups would be more attracted to the plate than to a non-polar solvent. So when a non-polar solvent passes through the plate, maybe the dots won't move. In that kind of situation, you need a more polar solvent. The more polar solvent would be able to push those dots along more effectively and cause more separation. But let's say we've got a solvent that was too polar. It if was too polar, it would just carry the dots as far as the solvent travels. You would lose the separation. So if it's not polar enough, or if it's too polar, you don't get good separation. A solvent or solvent mixture is chosen to give the optimal separation of the compounds in the mixture being analyzed.

The most common solvent set I used was hexanes and ethyl acetate. Hexanes, much more non-polar; ethyl acetate, somewhat polar. If you need a more non-polar environment, heavy on the hexanes, just a smidge of ethyl acetate. If you need to push polar groups through, you might bulk up on the ethyl acetate. TLC is not just a technique for identifying components of a mixture or separating compounds; it can also be used to check the progress of a reaction. Imagine that on a TLC plate I put my starting material, and let's say I already know what my product would be so I put that on the TLC plate as well. If have my starting material, my product, and then my reaction mixture, all on the same plate, once the plate's developed, I can see: is there only product present; is there only reactant present. It's a quick way to check the progress of a reaction, which if a reaction takes three hours, you kinda want to know if it's working or not. In lab, we had a series of reagent bottles on our lab bench where I had maybe 5% ethyl acetate, 15%, 25%, different levels of polarity. Any time I was doing a reaction that I had not tested before, I'd make TLC plates and try them in the solvents, to see which one gave me the better separation. If you need something more polar still, then sometimes a chloroform/acetic acid mixture is used. Chloroform is more polar than hexanes, and acetic acid is significantly more polar than ethyl acetate (it is, in fact, water-soluble, it's so polar). If you were separating a mixture of carboxylic acids, this system might work better. As a side point, if you're doing column chromatography, some kinds of silica gel go transparent/translucent in chloroform. If you're separating a mixture of colored compounds, you could watch those colors crawl down the column.

We have a development chamber. Look what's in it: a piece of filter paper. Why do you think we have this piece of filter paper in here? {admonition to not delay studying the theory). It is to maintain solvent equilibrium. You have this solvent that is crawling up the TLC plate. Solvents can evaporate, so what if it evaporates off of the plate? That means the solvent never gets all the way up the plate, the dots don't travel very well. You want to make sure this is entirely saturated with the solvent to prevent solvent from evaporating off of the plate. That's the purpose of the filter paper. As soon as you put solvent in there, solvent crawls up the filter paper, evaporates off the filter paper, saturates the environment, and makes it more likely that your TLC plate will develop correctly. Notice that the filter paper does not go all the way around. It's not because of visualization, although of course you want to be able to see your plate. Because if the sides or the top of the plate touch the solvent [on the filter paper], the solvent's going to flow this way, and you only want to flow one direction on your TLC plate. When you're using the TLC chamber, make sure the lid is on it, cause if the lid's open, everything evaporates. Filter paper – use to ensure that the interior of the chamber is fully saturated with the solvent to prevent solvent from evaporating off of the plate. You'll have your TLC plate in there, leaned up against the wall, but the TLC plate must not touch the filter paper; otherwise, solvent may flow across – side-to-side – the TLC plate.

Here's a flat view of the chamber with my TLC, with its starting line. I had better make sure that the solvent level is below the starting line. Why? What would happen if the starting level is above the line? It would go away. I wouldn't travel up the plate, because if they were already in the solvent, the solvent is just going to dissolve the dots, then everything goes up the plate together. You have to allow the solvent to pass through the dots from below; you cannot allow the dots to be dissolved beforehand.

When do we stop? Once it gets up to the top. Do we want it to touch the very top? No, because we need to make sure that we have a clear viewing of where that solvent progresses. The way that you can tell that is to look at your chamber, and you can see how far the solvent has crawled up the place, and when it gets to about the same distance from the top as your start line is from the bottom, that's when you pull your plate out of the mixture. As soon as you pull it out of the development chamber, the solvent's going to evaporate. Since you need to measure how far the solvent went, because that's part of your Rf calculation, as soon as you pull it out, immediately mark a line where the solvent had gotten to. Once the solvent reaches near the top of the plate, remove the plate and mark the position of the solvent front.

Visualization – All of the compounds that you are going to be clear or colorless, or if there is some color, it's likely to be just a light yellow – so light that you won't be able to see it at all on your TLC plates. Fortunately, the silica gels in these TLC plates has a UV-reactive dye in it. So wherever UV light hits the plate, it's going to glow green. If your compounds interact with UV light, that means it's going to absorb the UV light. If your compound absorbs UV light, then that means the UV light doesn't make it to the plate, which means that point where the compound is will look like a dark spot on your plate. Once you take this out of the solvent chamber, let it dry out; shine a UV light on the TLC, then circle all of the spots that you see on the plate. The only drawback to that is – what if your compound doesn't react to UV light? Then it will pass right through and you won't know it [the compound] is there.

But it's a good first step – it's a non-destructive visualization, since all you're doing is shining light on the TLC plate. Some forms of silica gel contain a dye that's responsive to UV light. If a spot contains a compound that also is active to UV, that compound will absorb UV light, and it will therefore appear as a dark spot against the TLC plate.

Because that's not always good enough, there are other visualization techniques. One approach is to put your TLC plate in an iodine chamber – a chamber that contains a small portion of iodine crystals. Iodine (for what ever reason) will stick to some compounds more than others. It will stick to organic compounds much more than it would to silica gel. When your spots are, in general, iodine will accumulate, and you'll get these dark brownish, orangish, yellowish spots that appear. There's an even better technique, which is to use a chemical stain. In that technique, the TLC plate is dipped into a compound that will react in some way to the spots on the plate. There are different stains used for different types of compounds, different classes of chemicals. The most-common stain I used was based off of this compound, p-anisaldehyde, which is related to the plant anise. It happens to turn a variety of colors depending on the functional group – pink for alcohols, purple for carboxylic acids, organge for ketones. This is a destructive technique, because you're causing a chemical reaction. Once you have your dots, you measure the distance the dots travel, versus the solvent front. That way, you can calculate your Rf values.

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I. Purpose – a concise statement of the qualitative or qualitative goals in an experiment.
II. Data – Direct qualitative or quantitative observations that require no manipulation.
III. Calculations – Any mathematical manipulation of data.
% composition = mass of compound / mass of mixture * 100%
% yield = mass obtained / theoretical mass * 100%
% error = experimental value – theoretical value / theoretical value * 100%
% error for temperature must always be calculated in Kelvin
IV. Conclusions – A summary of the results of an experiment that addresses the goals as presented in the purpose statement.
V. Discussion –

Chromatography – A technique by which a mixture of compounds can be separated on the basis of their polarity.
stationary phase – an immobile support that contains a compound that can separate the components of a mixture passed through it.
mobile phase – a gas or liquid that carries the mixture to be separated through the stationary phase.

Gas chromatography (GC) – A sample is volatilized (evaporated) and passed through a column (a tube) that is coated on the inside with some form of separatory material (stationary phase). The sample is pushed through the column by an inert gas (such as N2 or Ar) called the carrier gas (mobile phase).

TLC – thin-layer chromatography
Rf – retention factor = compound distance (c) / solvent distance (s)

Preparing TLC plates – TLC plates must always be marked in pencil, since the ink of a pen would travel with the development solvent.
*Do no press too hard with a pencil on a TLC plate since the silica gel can be scraped off.
– Draw a line across the bottom of the plate parallel to the bottom roughly 1 cm from the bottom.
– The plate is then spotted with one or more compounds.

Development –TLC solvents – A solvent or solvent mixture is chosen so that it most effectively separates the components of the sample being separated.
– hexanes / ethyl acetate (the more polar component)
– chloroform / acetic acid

Development chamber – Filter paper – used to ensure that the interior of the chamber is fully saturated with solvent to prevent solvent evaporating off the plate.
*The TLC plate must not touch the filter paper, otherwise solvent could flow sideways across the plate.

The level of solvent in the chamber must be below the start line.
– Once the solvent nears the top of the plate, remove the plate from the chamber and immediately mark the position of the solvent front.

VIsualization
– Some forms of silica gel contain a dye that's responsive to UV light. If a spot contains a compound that absorbs UV light, the spot will appear dark, since UC light would not reach the dye. (non-destructive)
– chemical stain – the TLC plate is dipped in a compound that will react with the spots on the plate. (destructive)

Structures

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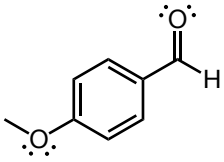

p-anisaldehyde