

### **Experiment #8: Synthesis of Novel Aromatic Esters**

When you perform liquid-liquid extraction with  $\text{NaHCO}_3$ , shake the sep funnel GENTLY and vent often, because a lot of carbon dioxide gas will be formed LM

Do not shake the sep funnel right after you have add the sodium Bicarbonate because there is a lot of gas forming. This can cause the lid to blow right off and spill your solution everywhere. This happened to me and it was not pretty. JV

To prevent the top from blowing off, let the funnel sit first, or if you begin shaking right away make sure you use your index and middle finger to hold the stopper in place so it can not pop off. Open the funnel away from you to get rid of gas that is formed. AJ

It is always very important to record all observations that are made during the course of an experiment. Due to the nature of this experiment, however, it is also critical to remember to record any and all changes in the smell of the solution after each step. NL

Not only does this apply to this lab but also any lab involving extraction with a separatory funnel. Be sure to know and clearly identify the layers(i.e. aqueous and organic) and be sure to know which one to save or throw out because if you throw out the wrong layer, you cannot proceed with the rest of the experiment (usually to obtain your product). AG

Make sure that you clean out all of your glassware before beginning this experiment including the distillation equipment. The dirty glassware may thrown off your results and make the containers hard to clean. JS

Do not be worried if your product turns out viscous like a syrup, or even solidifies after you boil off the solvent. There are many different ester synthesis assignments, but there should be one other person in the lab doing the same thing as you. You may want to compare results with them periodically throughout the lab. RR

Be sure to boil off all your solvent, if not you will see the impurities on your GC-FID. KB

Be sure not to sniff your product too hard because it might irritate your nostrils. Also, let your partner know whether it's a strong smell before you let them sniff it!! PO

When you set up your reflux make sure you have the water in and water out connected to the proper connections. Also make sure not to boil you mixture too fast. LC

Be careful during the different separation steps. Make sure you hold up the container to light before beginning to separate because some of the layers look very similar (both were dark green in mine) and you can end up separating the top layer into the bottom. CA

I don't know if this is considered wrong because I did not ask, but I think li set up the reflux in a way that would not work at first. You do not need a seal top for this part and no thermometer if you do not wish. The very top can be open but everything else I think must be sealed. I used the glass arm for the bottom that was open. I figured it was too close to the bottom and that product would be lost. So about 5 minutes into the reflux I had to switch it around and take out that arm and open the top of the funnel. This seemed to work better. Like I said before, it is best to come prepared and know what you are doing with reflux and distillation. NT

A couple of tips for this lab:

-Pre-heat the thermwell at the beginning of the experiment, before you set up the apparatus and collect the reactants.

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-When extracting, your solution might be so dark so that it appears to form only one layer; in fact, there are two layers, look closely and you will be able to distinguish them.

-If evaporating the ether in hot water bath then set up ahead of time so that no time is wasted. BL

When you are boiling off your solvent continue to heat it until the solution becomes syrup-like, otherwise you will have less product and more impurities in your chromatograms. MZ

For experiment 8, use the hot water bath method compared to the force air method to get a purer final product especially if you are using gallic acid. DD

When performing reflux, try to wash the round bottom flask as soon as you can. The boiling makes some of the solution stick to the sides of the flask and if it cools down enough the solution solidifies on the sides and is impossible to remove. This might cause problems for the person who uses the round bottom flask after you.

Also if the mixture from the reflux seems too solid, that you need to use a large amount of MTBE to dissolve it and it doesn't seem completely dissolved, don't worry about it will still form two layers when you add it to the sep. funnel. There might be some solid that remains that does not want to dissolve and you might need to gravity filter it out before you add it to the sep. funnel or it will clog the funnel up and take longer to extract. MZ

Remember to keep track of organic layer during extraction. BMB

When adding the MTBE, please make sure it is conducted under the hood. I really feel that this is a dangerous chemical, and will cause an insane amount of migraines. There might be chunks in your solution, but when pouring the solution in the sep. funnel, leave the chunks behind. MJ

Be sure to check the pH of the aqueous layer, not the organic layer. Add sufficient amounts of  $\text{NaHCO}_3$  until the pH turns basic. Be sure to do a pH paper test after each  $\text{NaHCO}_3$  addition. SF

Many people suggested to fully evaporate your solvent so you will not have additional substances in your IR...well what I did for this lab was that I weighed the product periodically until I got it close to the percent yield. I don't know if this is the right code of ethics, or if this is considered cheating (hopefully not) but I have been getting very good IR and other readings because I am getting a pure product. TM

One of the most fun parts of this lab is smelling all of the different products people came up with. My product smelled too strong to be pleasant, but one of my classmates made a product that smelled remarkably like pineapple. Share your results! JW

When I was draining my lower aqueous layers into the beakers I labeled them, this helps me not to confuse the different layers. When I evaporated my ether in the hot water bath, I did not have my heating source up too high because I did not want to evaporate my ether too fast. LC  
Since the percent yield of the received product is not very high after the first distillation, it would be interesting to see if this lab could be split in two to try another distillation and see how much higher could the percent yield increase of the final product. PO

The student should definitely note the colors in this lab. It should be a requirement to note the color after each step. MJ

I think a great addition/improvement to this lab would be to actually make a product fragrant. For example, use the synthetic aromatic ester to scent a type of soap or lotion. My specific compound was disappointing because the odor was so strong, but I wonder if it was diluted somehow if it would have

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smelled better. Maybe if I would have mixed a tiny amount into a fragrant-free lotion I would have ended up with a nice-smelling product. JW