

### Experiment #3: Hydrodistillation of Essential Oils

Cumin may not be the best option to choose in this lab. While it smells nice, it reduces to so little a product that you cannot see it. Use oregano instead. MS

Include a description in the lab description why the oil vapors boil off with the water vapors. Is it a direct partial pressure relationship? Why doesn't the oil boil off first, since it has the lower boiling point? MS

When you are distilling just crank up the heat and make sure to watch the flask. Your solution may bubble up but as long as it stays in the flask keep the heat as high as it will go. Of course if it starts to bubble out of the flask turn the heat down. CA

Having the students further pulverize the spice they decide to use before hydrodistillation could give some better percent yields. CA

Between all my friends, I felt that cloves produced the greatest amount of product so it might be advantages to use cloves, since in the lab, the amount of yield is veryyyyyy smaaaalllll TM

If possible, use a large thermwell that completely covers the round bottom. This will speed up the distillation process (which can be lengthy). LM

Be sure that whenever you are distilling to use a thermwell that is properly working and you have plugged into the correct electrical socket because distillation takes forever and malfunctioning lab materials can make the process even longer. AG

Also make sure that the thermwell is actually touching the round bottom flask. This will help with the heating and speed up the process. AJ

Make sure you pay close attention to the directions on part II (extraction of essential oils) especially number 2. You will only be working with the lower layer each time that you extract with the NaCl. If you try to speed up the lab after the lengthy distillation you may get inconclusive results and may have to start over or have a huge blunder. JS

Remember when you write your lab report that your end product is not PURE essential oil, but that the oil is a major component of the product. This will be evident when you perform your thin layer chromatography analysis and see more than one spot where you tested your product. RR

When you are trying to remove the solvent do not over heat the solution at the end of the experiment , this can cause you to lose some of your product. JV

To be more efficient and precise, instead of measuring your sample into a weight boat then transferring it to a distilling flask, tar the flask and measure your sample directly into it. In this way there will not be any sample loss. Also, make sure not to spill too much liquid in between extractions and transfers, so that way your percent yield will be higher. NT

Couple of tips for this experiment:

-At the beginning of lab, plug in the thermwell so that it heats up while you set up and grab all the reactants.

-Use a spice that is already in powder form, otherwise you will need to grind it first.

-When extracting, do not shake the separatory funnel, but gently turn it upside down and then right-side-up (releasing gas in between); this will prevent emulsion.

## Student Comments Spring 2009 Dominican University

-Set up the hot water bath during the extraction so that you do not have to wait for the water to reach boiling temperature when you evaporate the solvent. BL

If you use oregano, at the end it seems like there is so much of it in the round bottom flask that it will never come out and when you try to wash it out it clogs up the opening. The best way to clean the flask is by completely filling the round bottom with water. This makes the oregano watery and easier to pour out. ZM

If you can not obtain the powder, during the grinding process try to make it into the finest powder as possible. The finer the powder the better the results. DD

Make sure when conducting the extraction of dichloromethane with NaCl, that the aq. upper layer does not leak into the lower layer. If this does occur it will show up on the NMR and IR. To insure that this doesn't happen, when draining make sure to stop once the liquid starts to reach the tip of the sep. funnel. MJ

I received a big shock when at the end of the lab (the evaporation step) I turned around for a second and upon checking the beaker found that there was apparently nothing in it. Three hours of lab and my beaker had nothing to show for it. This is completely fine however. You can smell the oil in the beaker and when you weigh it you will find that there is something there. Just don't be as surprised as I was. DF

I choose oregano for my hydrodistillation but was disappointed at the end of lab when we didn't have an essential oil standard to compare it to. If you're like me and appreciate a comparative analysis, make sure we have the standard for comparison before you begin lab. JW

When doing liquid-liquid extraction double check to make sure which is the organic layer. You don't want to accidentally extract and discard the layer with your product. GL

When boiling out your solvent in the bath, don't worry if all the liquid in the jar boils out b/c your essential oil will still be inside the jar as a film adhered to the insides of the jar. For analysis, addition of solvent is all that is needed to get a sample for IR. Don't be afraid that you lost your sample. SF

In the ethanol lab when the distillation was done slowly, the percent yield of ethanol was higher. This may also apply to this lab. When you see that the distillate is starting to be collected, turn the heat down a little bit to make the collecting process occur more slowly. This may result in a more pure product, which will be reflected in the analyses of the product. RR

Another thing i noticed is that the procedure called for use of 500ml round bottom flasks and thermwells. However the thermwells were not large enough to hold the round bottom flasks so instead of sitting neatly in the thermwells they did not make full contact with its surface, at the same time there were a few thermwells that were large enough and these worked well and improved the amount of time needed for the lab, maybe these could be provided instead for this lab to help with time. KB

Since essential oils can be obtained from a number of spices, but also wood, nuts, seeds and even flowers it might be really nice to encourage students to use other materials and use other methods suitable for their material used. AG

For step 2 in part II. It makes more sense to obtain 55 mL NaCl solution to extract the dichloromethane layer and pipette out the 15 mL of each extraction. Rather than going back and forth to obtain 15 mL JM

I ran into some research indicating that it is more efficient to use microwaves when extracting essential oils. It does not affect the purity of the product but it has been found to increase the yield. It would be interesting to test this out. YM