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CHEM 345
9/18/2019

Natural Products Isolation

Reference: Handout; Chemistry Lessons: phase changes, chirality, natural product extractions, solubility; Green lessons: alternative solvents (liquid CO2)

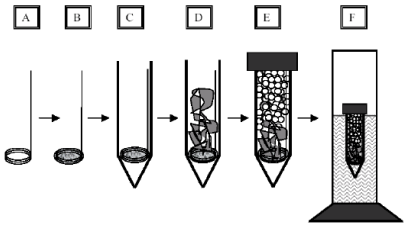
Purpose: To isolate and purify organic compounds from their natural source

Table of Reagents:

Reagents	Amount	MW	Mmol	BP (°C)	MP (°C)	Density
Orange Rind	~4 mL	-	-	-	-	-
Dry Ice	~1 g	142.04 g		1429 °C	884°C	2.66 g/cm^3
Water (H_2O)	Varied	18.02 g		100 °C	0 °C	0.997 gm/cm^3

Safety:

- Risk of vessel rupture and/or flying projectiles due to high pressure
- Shattering of glass may occur – DO NOT USE ANY GLASS
- Material could get into your eyes – wear your safety googles
- Contact with dry ice can damage skin → wear safety gloves
- Tubes may become brittle → Do not liquify CO_2 > 5 times in same tube

Experimental Procedures	Data & Observations
1. Grate Rind → Grate colored part of peel ($\frac{1}{4}-\frac{1}{2}$ orange). Make sure to have at least 2.5 g of the orange	*Orange Rind:* 2.6834 g
2. Extraction Vessel a. Record mass of 15 mL centrifuge tube (weigh in small beaker) b. Make solid trap with 20 cm of copper wire and 1.5 cm filter paper c. Wrap 3 coils of wire so it stops at the beginning of the taper. Use rest of wire as handle painting up d. Cut off extra wire as all wire should be in the tube e. Place one piece of filter paper between the coils. Fold up edges around wire to make base f. Reweigh the trap; Add ~2.5 g of orange rind until tube half-full g. Record weight and calculate mass of orange peel in tube	*15 mL Centrifuge Tube:* 5.4203 g (w/o cap) and 6.6032 g (w/ cap) *Trap:* 5.765 g (w/o cap) and 6.9321 g (w/ cap) *Trap with Orange:* 47.4797 g (w/o cap) and 48.6637 g (w/ cap) *Observations:*  *Figure 1. Extraction Vessel Procedure*

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3. Extraction Environment a. Fill plastic cylinder bottle with $\frac{1}{2}-\frac{2}{3}$ of warm tap water. ****Do not heat or add hot water**** b. Top off the centrifuge tube with crushed dry ice. Tap tip of tube and add until full. Seal cap and if broken, replace the cap. c. Drop centrifuge tube into the cylinder. Do not place anything on top of the cylinder as the glass might break or the cap might shoot off. d. Observe from the side.	<u>*Observations:*</u> • Started making a 'hissing' noise; had to redo the experiment (Loose cap)
4. Liquid CO_2 should appear ~ 15-30 sec. • If no liquid appears after 1 min, then seal is broke; Replace cap and retry ****Liquid should boil, and the gas should escape for 2 min and 45 seconds****	<u>*Observations:*</u> • Liquid CO_2 started to appear inside the tube; gas escaping on outside while boiling on inside
5. To prevent freezing the tube on one side, rotate the cylinder on its base ****NEVER remove the tube from plastic cylinder when the CO_2 is still liquid!!!****	
6. CO_2 should pass through the orange peel and move towards the bottom of the tube • If not able to reach bottom of tube → oil will start to gather in region containing the orange peel. This will not allow for isolation of the product	<u>*Observations:*</u> • Oil starting to form at the bottom of the tube (yellow in color)
7. Remove tube with tweezers when gas is no longer escaping, and the liquid has evaporated. Slowly open up the cap and let the air escape first.	
8. Rearrange the orange peels before the 2nd extraction. Make path to the bottom of the tube by breaking up the solid mass. Repeat extraction.	

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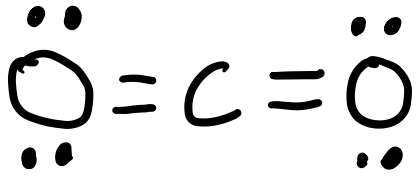
9. Extraction Completed a. Product should be at the tip of the tube (~0.1 mL of pale, yellow oil) b. Pull the wire handle to remove the solid and the trap c. Avoid solid remains in the tube to minimize product loss	• D-Limonene oil gathered at the bottom
10. Dry outside of the tube. Weigh tube and determine the mass of the product. Calculate % recovery based on the product gather and the grind used.	*Recovered Sample:* 0.2635 g $\% \, Recovery = \frac{0.2635}{2.6838} \times 100 = 9.81\%$

Post-lab Questions:

1. $\% \text{ Recovery} = \frac{mass \text{ recovered } material}{mass \, of \, starting \, mixture} \times 100 = \frac{0.2635}{2.6838} \times 100 = 9.81\% \, of \, limonene$

→ We recovered 9.81 % of limonene from the orange rind

2. **a)** CO_2 is non-polar due to it being symmetrical. There is no unequal share of electrons, thus there is no unequal pull to one side as well as each oxygen is pulling 180° from each other.



b) Limonene would be able to dissolve in hexane and liquid CO_2, because with limonene being non-polar it would mix well with a non-polar solvent. This is because in chemistry, "like dissolves like" which means a non-polar solute will dissolve in a non-polar solvent, however, a polar solute will not dissolve in a non-polar solvent (water does not dissolve oil).

c) Coffee is stated to be a polar compound, which means that it would not work in our experiment, since we are using a non-polar solvent, liquid CO_2.

d) (+)-limonene is in the R configuration.

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3. In our experiment we manipulated the conditions to observe liquid CO_2 by sealing the cap to build up pressure within the tube. This resulted in the optimal conditions, increase in pressure and being at the optimal temperature inside the tube, to make the change from a solid phase to a liquid phase of CO_2.

4. In this experiment we created an E-Factor of 0. This is because all the chemicals that we used was recycled back to the environment such as the CO_2 (reverted back to a gas in the air) and the orange rind (biodegradable).

$$E\ Factor = \frac{waste}{product} = \frac{0\ g}{0.2635\ g} = 0$$

5. Simply looking at the steam distillation process, we could already conclude that the Liquid CO_2 extraction method is much greener. Starting off with the ether in the steam distillation, we would be creating chemical waste that is not necessary in the Liquid CO_2 extraction, in which we did not create any waste at all. This creation of waste corresponds to the first principle of Green Chemistry in which to create a chemical synthesis that produces no waste for cleanup. Furthermore, combining principles three and ten of the Green Chemistry, the Liquid CO_2 extraction method uses no toxic chemicals (ether) as well as uses a substance that goes back to the environment after use (CO_2). Overall, the Liquid CO_2 extraction method is much greener process that the steam distillation process.