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Organic Lab 309:03

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Experiment 5: Preparation of Synthetic Banana Oil

February 18, 2019

Purpose:

The purpose of this experiment is to prepare synthetic banana oil.

Equations:

Mechanisms:

Amounts and Properties:

Table 1: Important properties of chemicals

Chemicals	**Mol Weight**	**BP**	**Density**	**Solubility**	**Amount**
Acetic Acid	60.1	118	1.049	miscible	17 mL
Isopentyl Alcohol	88.1	130	.815	2.7	150 mmol
Isopentyl Acetate	130.2	142	.876	.25	
Sulfuric Acid	98.1	290	1.84	miscible	1 mL

Hazards and Safety:

Acetic acid causes chemical burns that can seriously damage skin and eyes; its vapors are highly irritating to eyes and respiratory tract. Wear gloves, dispense under a hood, avoid contact and don't breathe vapors. Sulfuric acid causes chemical burns that can seriously damage skin and eyes. Wear gloves and avoid contact. Isopentyl alcohol and isopentyl acetate can irritate skin, eyes, and respiratory tract. To dispose: Dispose within the marked containers under the hood unless otherwise specified.

Procedure:

Reaction:

1. Weight 150 mmol of isopentyl alcohol into a round bottom flask of appropriate size and add a stirring bar.
2. Under hood, add 17 mL of glacial acetic acid then carefully mix in 1 mL of concentrated sulfuric acid while stirring.
3. Connect condenser to reaction flask, turn on water for condenser, start the stirrer and heat for reflux for one hour after boiling begins.

Separation:

1. Allow mixture to cool to room temperature slowly.
2. Turn off cooling water and remove reflux condenser.
3. Transfer mix to separatory funnel, leaving stirring bar behind, and wash with 50 mL of water.
4. Drain aqueous layer and leave organic layer in funnel.
5. Carefully wash organic layer with 2 successive portions of 5% aqueous sodium bicarbonate, draining aqueous layer each washing.
    a. During first washing shir layers until gas evolution subsides before stoperring funnel and vent it frequently.
6. Dry isopentyl acetate with anhydrous sodium sulfate or magnesium sulfate.

Purification and Analysis:

1. Assemble distillery apparatus.
2. Make sure thermometer bulb is positioned and checked.
3. Distill crude product collecting any liquid that distills between 136 degrees C and 143 degrees C.
4. Record actual boiling range observed and wait until the entire thermometer bulb is moist with condensing vapors, liquid is distilling into receiver and temperature is stable before you record initial temperature reading.
5. Stop distillation when only a drop of water remains in flask or when temperature reaches 143 degrees C.
6. If distillate is cloudy or contains water droplets, dry it.
7. Weight distillate.
8. Assuming the first is isopentyl alcohol, the second is isopentyl acetate, and the third is acetic acid.

Observations:

When the sodium bicarbonate was added in the separatory funnel, bubbles began to form due to the carbon dioxide being produced as a by product in the washing. The organic layer was clearer than the aqueous layer and the aqueous layer was a little foggy.

Measurements:

Table 2: Numbers gathered during experiment

Isopentyl Alcohol used:	16.00 mL
25 mL Flask before: 27.274g	25 mL Flask after: 29.084g
Start First Plateau: 122 degrees C	End First Plateau: 123 degrees C
Start Banana Oil Plateau: 122 degrees C	Start Banana Oil Plateau: 123 degrees C

Data and Calculations:

Mass of Banana Oil acquired: 29.084g - 27.274g = 1.81g

Theoretical yield:

$$16.00 \frac{\frac{mL * .815\,g}{1\,mL} * 1\,mol}{\frac{88.1\,g}{1\,mol} * 1\,mol} * 130.2\,g = 19.27\,g$$

Percent yield: 1.81g / 19.27g * 100 = 9.392%

Discussion:

During the distillation process, there was an error that lead to the distillation occuring for over an hour. While this distillation took over an hour, the distillate that came out was also not being read at the right temperatures because of a loose thermometer bulb or the thermometer being placed a little too high at the ideal position. This lead to two different flasks filled with banana oil and the first flask potentially having an impure sample. The second flask theoretically has all pure sample due to a plateau being seen and changing the first flask halfway after an error was spotted.

Conclusions:

After conducting the experiment, the yield was 1.81g which was 9.392% of the theoretical yield. If there was more time, another distillation could've been done with the two samples again with a better positioning of the thermometer to get better results. Having the thermometer or thermometer bulb fully functioning can lead to a larger yield, but due to them being placed incorrectly or malfunctioning, the temperature plateaus were the same which led to not being able to find the correct separation between the compounds at their respective boiling points. Based on the C-NMR that was given there was a ketone signal given at around the 172 ppm mark. This makes sense with an integral part of the banana oil structure. Along with this, from the H-NMR the Hydrogens that show a signal at the 2 ppm range is near the ketone group. There is also a signal at the 4 ppm range which hides two Hydrogens which relates to the ether group with one side of it being the ketone and the other side being the rest of ether. The ones at

the .75 ppm has a splitting of a doublet which relates to another H with a splitting of a very faint septet which yields the isopropyl group at the end of the compound.

Exercises:

1. Using 150 mmol, the amount to use is .15 mol. The equilibrium constant is: $K=\frac{[Isopentyl\ Acetate]}{[Acetic\ Acid][Isopentyl\ Alcohol]}$. Ice table for this is:

	Acetic Acid	Isopentyl Alcohol	Isopentyl Acetate
Initial	.15	.15	0
Change	.15-.15a	.15-.15a	.15a

Now to replace into the constant equation with its values and get:

$4.2 = \frac{.15a}{(.15-.15a)\square^2}$. From here solve for a to get either 2.82 or .305. Since 2.82 is too large, it can be tossed out as an arbitrary value. A) Using .305, Isopentyl Acetate = .15(.305) = .046 mole. B1) Due to an incomplete reaction: .15 - .046 = .104 mol * $\frac{130.2\,g}{1\,mol}=13.54\,g\,lost$.

B2) Since the amount during the experiment was about 50 mL, using the solubility of .25g per 100 mL, the mass lost would be half of the solubility which is .125g lost. B3) Each wash with sodium bicarbonate was with 20 mL so this would be the solubility multiplied by 40 mL to yield: .1 g lost. To compare the amount that was lost was greater than the calculations that were shown here. This can be because of not washing long enough and due to the distillery process with bad equipment placement. The total loss was about 17.46g from the theoretical calculation.

2. The gas that escaped during the washing process is Carbon Dioxide formed by both acetic acid and sulfuric acid reacting with sodium bicarbonate.
   a. $CH\square_3COOH+NaHCO\square_3\rightarrow CH\square_3COONa+CO\square_2+H\square_2O$
   b. $H\square_2SO\square_4+2NaHCO\square_3\rightarrow Na\square_2SO\square_4+¿2CO\square_2+2H\square_2O$
3. A) Atom economy for this is: $\frac{130.2}{60.1+88.1}=.879$, Reaction Efficiency: Atom eff * % yield Reaction efficiency: .879 * 9.392% = 8.256% B) Choosing reactants with almost the same total molar mass as the target atom to get an atom economy of almost one can net a higher yield, but having a higher % yield requires doing the experiment very carefully. C) A green effect is the high atom economy for this experiment and an anti-green effect is the isopentyl alcohol and isopentyl acetate is harmful that can hurt environments.
4. A) The water would be contained within the distillate of the first sample and ruin the actual yield. B) Forgetting the sulfuric acid leads to a lowered yield of isopentyl acetate since sulfuric acid would speed the reaction and increase the yield acting as a catalyst. C) Using twice the amount of acetic acid wouldn't affect the final yield if the washing was done correctly. D) Leaving out the washing step, the yield would be affected by adding

more acetic acid to the potential amount of distillate. E) If the thermometer was placed higher than it was, there would be an inaccurate reading and that could create a lowered yield of each substance and that each substance could have been collecting other samples that came after each run.\

5.

	Acetic Acid	Isopentyl Alcohol	Isopentyl Acetate
Initial	1M	1M	0
Change	1 - a	1- a	a

Max is 67%, K = 4.2

$4.2 = \frac{a}{(1-a)\square^2} \rightarrow 4.2(a¿¿2-2a+1)=a¿\rightarrow$ a = 1.621 and .0617. 1.621 is arbitrary so the value is .0617. This is around 61.7% of the yield which is close to the max of 67%.