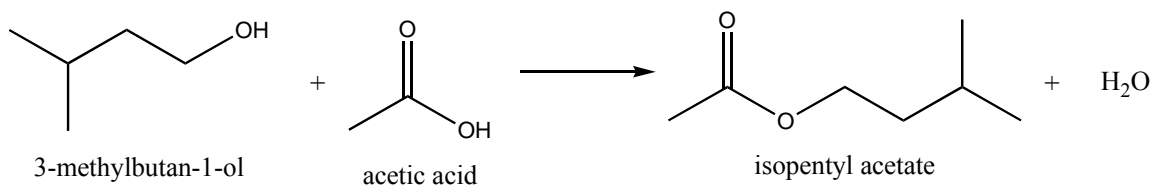


Ester Synthesis (Synthesis of Banana Oil)

Equation:



Procedure:

Synthesis:

Assemble a **reflux apparatus** using a 50 or a 100 mL round-bottom flask and an air condenser. In the flask place 3.0 mL of isopentyl alcohol (AKA: isoamyl alcohol and 3-methyl-1-butanol), 4.0 mL of glacial acetic acid, and 0.8 mL of concentrated sulfuric acid. Since you will be heating the mixture to reflux, place one piece of boiling stone as well. Heat the mixture using a heating mantle to a gentle boil and let the mixture reflux for one hour. After the one-hour period is complete, discontinue the heating and allow the mixture to cool to room temperature.

Work-up:

Transfer the mixture into a 30 mL separatory funnel. To the reaction flask add 10 mL of water for rinsing, and then transfer the rinse into the separatory funnel. Gently shake the contents of the separatory funnel, and then separate and discard the lower aqueous layer. What is left in the funnel at this point is the crude ester product, which contains some acetic acid. The acetic acid could now be neutralized and removed by addition of 5 mL of 5% aqueous sodium bicarbonate solution. Carefully add the bicarbonate solution and shake the resulting mixture until no carbon dioxide gas is evolved. Separate and discard the lower aqueous layer. Repeat the extraction of acetic acid with two more 5 mL portion of sodium bicarbonate.

What is the equation for the reaction between sodium bicarbonate and the residual acetic acid?

Add 2 mL of saturated sodium chloride solution to the crude product and stir the resulting mixture gently. *This process is called **salting out**. It is essentially a washing, which enhances the difference between the polarities of the two organic and aqueous layers by making the aqueous layer more polar. This forces any residual ionic (or polar) substance out of the organic layer, thus increasing its purity.* Discard the aqueous layer.

To remove any trace of water, transfer the organic layer into an Erlenmeyer flask, and dry it using magnesium sulfate. After the drying is complete, filter the solids out (using the pipet plug technique). At this point, the product mixture should be free of water, acid, and any ionic substance.

Final Purification by Distillation:

Assemble a simple distillation apparatus using clean and dry glassware. Choose the distillation flask based on the volume of the partly purified banana oil you have.

Normally, the volume of the mixture should be between one half-to-two thirds of the volume of the flask. Start heating and distilling. The product of interest (isopentyl acetate) will be collected between 134 and 143 °C. Monitor the temperature closely. You may collect the distillates at lower temperatures in a graduated cylinder, but you should collect the product of interest in a clean and dry pear-shaped flask with a side arm.

For your report:

Weigh the product (the distillate) and determine the % Yield.

Obtain an IR spectrum of the product and compare it to the literature IR given to you by your instructor.