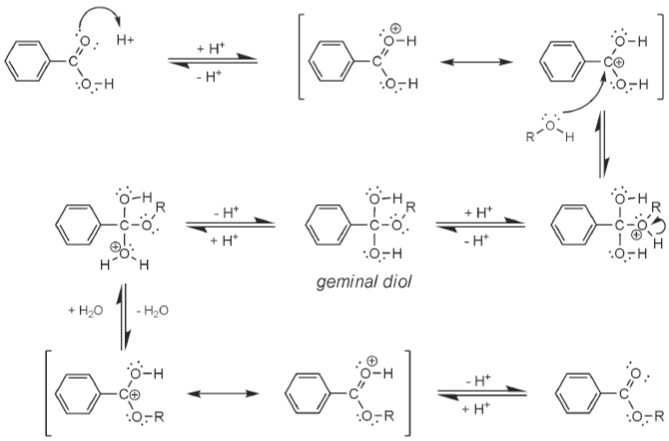
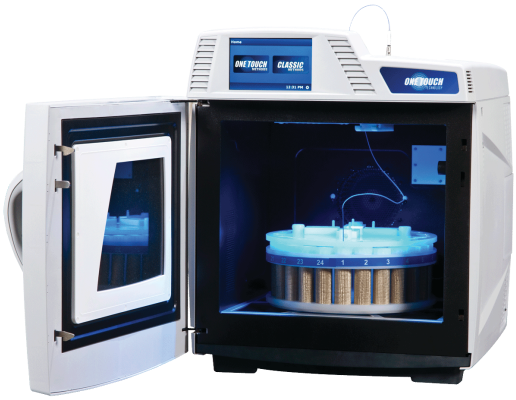
Esterification Experiment

Introduction

Esters are organic compounds derived from carboxylic acids in which the acidic hydrogen has been replaced with an organic group. They are known for their pleasant odors and comprise the major flavor components of a number of fruits. In addition, they play a significant sole in primary metabolism and other biochemical processes.

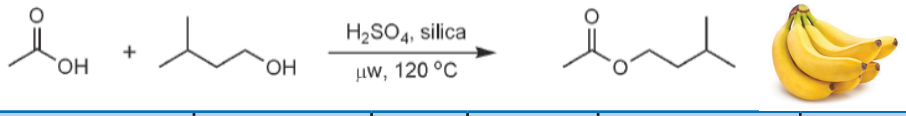
Esters can be synthesized in a number of different ways. One common method is the acid catalyzed condensation of a carboxylic acid and an alcohol. The mechanism involves an initial protonation of the carboxylic acid to form a resonance-stabilized cation. The addition of the alcohol to the cationic carbon and loss of one of the protons on the oxygen of the alcohol form a species called a geminal diol. Protonation of one of the hydroxyl groups in this diol, followed by loss of water, yields the ester product. To obtain significant yields of the product, the equilibrium must be shifted by either added excess of one of the starting materials or by removing water and/or the ester product.





In this experiment, 3-methyl-1-butanol is used in the esterification of ethanoic (acetic) acid to give a banana-smelling ester. Using conventional heating, the esterification reaction reaches equilibrium after a few hours of reflux. In this experiment, microwave heating in the CEM **MARS 6 Synthesis**™ microwave system is used to significantly accelerate the reaction. Both excess acetic acid and the removal of the water are utilized to drive the reaction to completion.

Materials and Methods



Reagent	MW (g/mol)	mmol	Mass (g)	Density (g/mL)	Volume (mL)
Glacial acetic acid	60	70	4.2	1.049	4.0
3-methyl-1-butanol	88	23	2.0	0.809	2.5
Sulfuric Acid					10 drops
Silica beads			0.20		

Procedure for 3-methylbutyl ethanoate (isoamylacetate)

Glacial acetic acid (4.0 mL, 70 mmol), 3-methyl-1-butanol (2.5 mL, 23 mmol), concentrated sulfuric acid (10 drops), and silica beads (0.20 g) are placed in a **MARS 6**™ **GlassChem** reaction vessel containing a stir bar. Place the vent plug in

the top of the vessel. Finger-tighten the vessel top and then use the preset torque wrench to tighten it until a single click is heard. Insert protective sleeves into the turntable receptacles and place the vessel fully into one of the sleeves. Note the position of the vessel in the turntable. Place the reaction control vessels into protective sleeves in the turntable and securely fasten the turntable shield. After the turntable and shield are properly installed in the microwave cavity, take the fiber optic probe that is connected to the cavity outside and insert the end into the control vessel thermowell. **Program a Ramp to Temperature Method to heat the reaction mixture to 120 ºC over a 3 minute period and hold for 5 minutes**. After the reaction had completed and the vessel has cooled to below 50 ºC, remove the vessel from the microwave.

Sodium bicarbonate ($NaHCO_3$) (10 mL of a 10% solution) is placed in a separatory funnel. The reaction mixture is transfered from the microwave vessel into the funnel with a pipette. This will lead to a very effervescent reaction as the excess acetic acid is neutralized. Water is added at this point until any visible solid is dissolved. Diethyl ether (5 mL) is added to the organic layer and the solution swirled. The aqueous layer is then removed. An additional 5 mL of sodium bicarbonate is added and the solution swirled again. After the layers have separated, the organic layer is removed to a small Erlenmeyer flask. The aqueous layer is extracted two more times with 5 mL of diethyl ether, each time. Remove the organic layer after each extraction and combine it with the first ether layer. The combined organic layers are returned to the empty separatory funnel and are washed with saturated NaCl solution (5 mL). The organic layer is placed in a clean Erlenmeyer flask and then dried over sodium sulfate (Na2So4). The dried organics are transferred into a pre-weighed flask. The diethyl ether is removed on a rotary evaporator or under a stream of nitrogen. The flask plus product is weighed and the yield calculated for the reaction. The refractive index is determined and compared to that in the literature. The chemical structure index is determined and compared to that in the literature. The chemical structure of the product is confirmed by IR, ^{1}H-NMR and/or ^{13}C-NMR spectroscopy.

“Clean, Fast Organic Chemistry: Microwave-assisted Laboratory Experiments.” Nicholas Leadbeater, PhD. and Cynthia McGowan, Ph.D.

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