Esterification

Ilana Berlin

03/27/2025

Purpose: React butyl alcohol with acetic acid in presence of Dowex catalyst to produce butyl acetate. Remove water with makeshift Dean-Stark trap to shift equilibrium towards product. Categorize the product and look for evidence of starting material with IR and H-NMR.

Reagent Table and Calculations:

Reactants & Products	FW (mg/mmol)	bp (°C)	density (g/mL)	Equiv.	mmol	mass (mg)	volume (µL)
acetic acid, 17.4 M	60.05	118	1.049	1.0	10.0	NA	575
butyl alcohol	74.12	117.7	0.810	1.0	10.0	741	915
butyl acetate	116.16	124-126	0.88	1.0	7.15 (10.0)	831 (1162)	NA
isobutyl alcohol	74.12	108	0.803	1.0	10.0	741	923
isobutyl acetate	116.16	115-117	0.867	1.0	10.0 (theoretical)	1162 (theoretical)	NA
isoamyl alcohol	88.15	130	0.809	1.0	10.0	882	1090
isoamyl acetate	130.19	142	0.867	1.0	10.0 (theoretical)	1302 (theoretical)	NA
1-pentanol	88.15	118-119	0.812	1.0	10.0	882	1086

pentyl acetate	130.18	142-149	0.876	1.0	10.0 (theoretical)	1302 (theoretical)	NA

() - Theoretical

Acetic Acid: 575µL(1.049mg/1µL) = 603mg (1mmol/60.05mg) = 10.0mmol

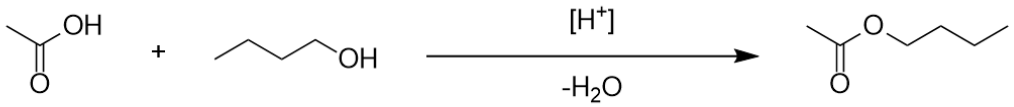
Butyl Alcohol: 915µL (0.810mg/1µL) = 741mg (1mmol/74.12mg) = 10.0mmol

(Butyl Acetate: 10.0mmol (116.16mg/1mmol) = 1162mg

Butyl Acetate: 831mg(1mmol/116.16g) = 7.15mmol

Percent Yield: 7.15/10.0 = 71.5%

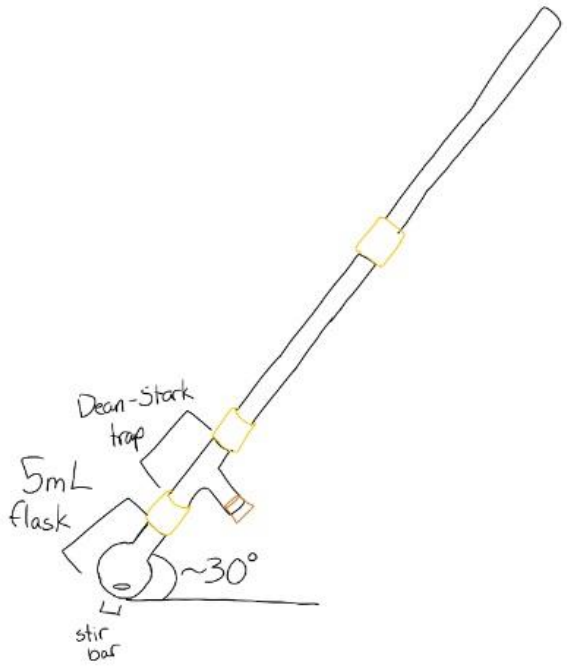
Net Reaction Equation: (Use Chemdraw)



References:

(1) Kateley, L. J., *Guide for Organic Chemistry Laboratory*, Seventeenth edition, Lake Forest College, 2011

Apparatus (Label parts):

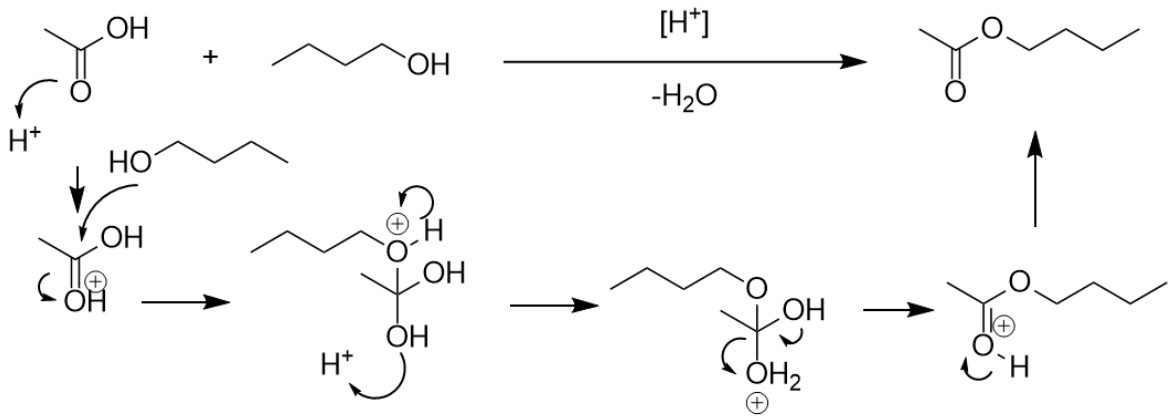


Experimental: (be concise and use abbreviations h, min, and soln)

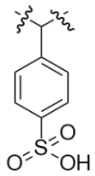
Weight without Dowex: 7.394g. A small quantity of washed Dowex 50X2-100 ion exchange resin was added to flask. Weight of flask with resin catalyst and stir bar was 7.470g. The total mass of the catalyst was 0.076g. 575 µL of acetic acid and 915µL of butyl alcohol was added to the flask.

Flask was heated by hot plate. Soln collected on the cork in the side arm. After around 20min the top layer of the side arm was tipped backed into the flask. Tapping on the side arm ensured that water settled to the bottom. After another 40min, the water layer stopped growing which indicated the reaction was complete. The flask was tipped back to ensure all soln in the side arm returned to the flask. Soln was cooled to room temp. Flask was weighed for a total weight of 8.301g which indicated 0.831g of product was produced.

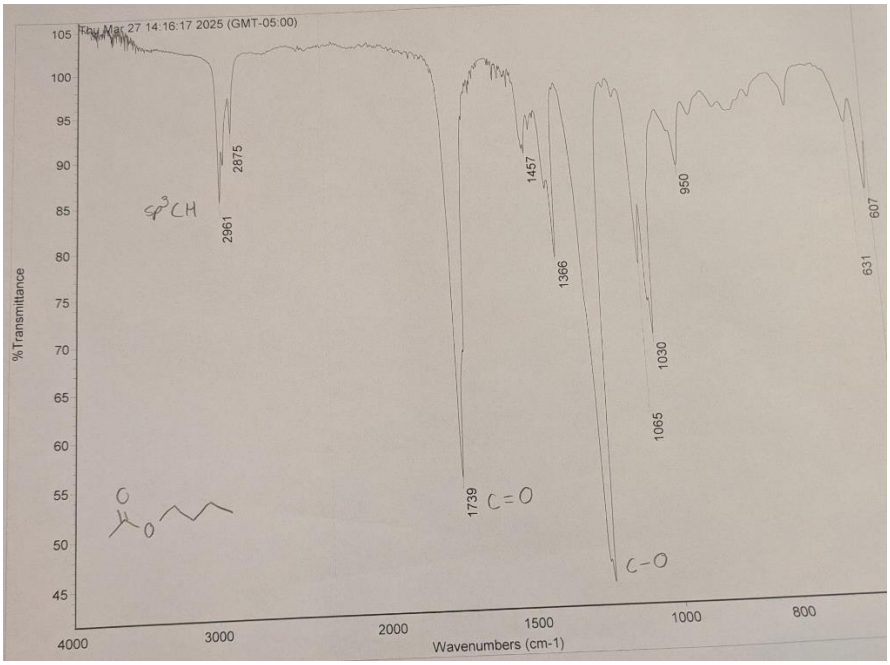
Mechanism: (Use chemdraw and net reaction equasion)



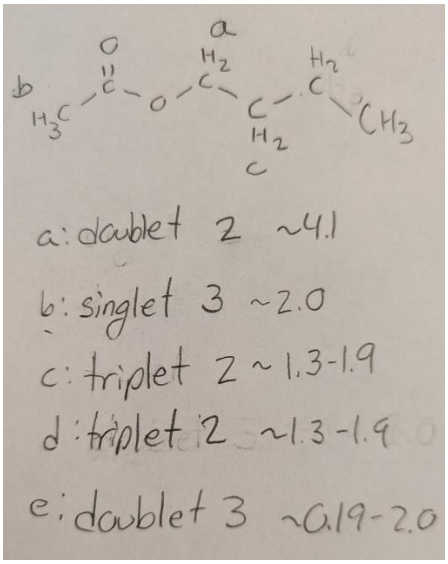
Dowex catalyst:

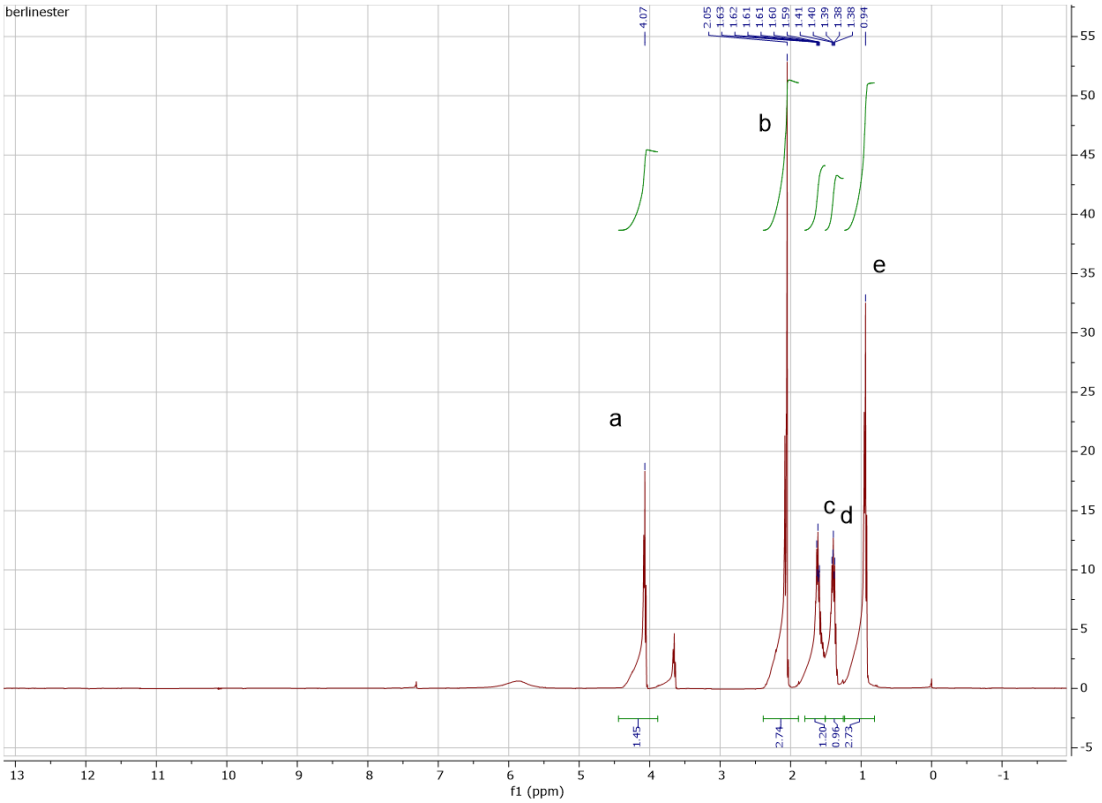


IR:



H-NMR:


a: doublet 2 ~4.1
b: singlet 3 ~2.0
c: triplet 2 ~1.3-1.9
d: triplet 2 ~1.3-1.9
e: doublet 3 ~0.19-2.0


berlinester
a
b
c
d
e
f1 (ppm)

Questions:

1. **Why is it important to remove water in this reaction?**

This reaction is easily reversable, so water is collected to prevent the reaction from happening in reverse. It shifts the equilibrium toward the desired product.

2. **Briefly describe an azeotrope and how it is used to remove water in this reaction**

An azeotrope is a mixture of solutions that is not separable by distillation since it reaches a point where the vapor has a constant concentration of each compound in the solution. Since both materials vaporized at a similar temperature but are immiscible, so we can separate them with a makeshift Dean-Stark trap.

Conclusion: (key findings, yield, and improvements)

Esterification is a reversable reaction so removing water during the reaction ensures that the reactions favors the products. Water and butyl acetate form an azeotropic solution so they cannot be separated using typical distillation.

The final percent yield was 71.5%. Some product was left in the side arm of the makeshift Dean-Stark trap to ensure that water was not accidentally added back into the solution. If the experiment were to be repeated I would love to start with a different acid to see all the scents and I would start the hot plate preheating earlier to speed up the reaction. The ideal temperature seemed to be when the hotplate was set to ~ 180°.