

# Organic Chemistry, Lab 4: Synthesis, and purification by distillation of Esters

*Keelan Krinsky*

## Submission Information

Name: *Keelan Krinsky*

Student number: *1634953*

Locker number: *365* Group: *7*

Experiment Number: *04*

Demonstrator:

Date of Experiment: *02/08/2018*

Title: *Synthesis, and purification by distillation of Esters*

## Reaction Scheme

## Reactants

Table 1: Limiting Reagent Calculations

Reactants Used	Molecular Weight( $g \cdot mol^{-1}$ )	Mass( $g$ )	Number of Moles(mol)
1-butanol	72.12	$0.81g \cdot ml^{-1} \cdot 8.0ml = 6.5g$	$\left(\frac{6.48g}{72.12g \cdot mol^{-1}}\right) = 9.0 \cdot 10^{-2}$
Glacial acetic Acid	60.05	$1.049 \cdot ml^{-1} \cdot 6.0ml = 6.3g$	$\left(\frac{6.3g}{60.05g \cdot mol^{-1}}\right) = 1.0 \cdot 10^{-1}$

Limiting Reagent: *1-butanol*

## Yield Calculations

Molecular Weight of Product:  $116.16g \cdot mol^{-1}$

Mass of Product:  $14.8929g - 9.3075g = 5.5853g$

Theoretical Yield:  $1.0 \cdot 10^{-1}mol \cdot 116.16g \cdot mol^{-1} = 12g$

Percentage Yield:  $\frac{5.5853g}{12g} \cdot 100\% = 47\%$

## Product analysis and observation

### Physical state of product

Colorless liquid.

### Distillation temperature of product

$115 - 128^{\circ}C$  at 761mm Hg

### Expected:

$27^{\circ}C$

### Found:

$119 - 124^{\circ}C$

## NMR/IR spectra

### 1-butanol

### Structure

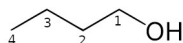


Figure 1: Structure of 1-butanol

Table 2: Interpretation of  $^1H$  NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	3.62	2.53	1.55	1.39	0.93
Ratio of signal	2	1	2	2	3
Multiplicity	triplet	singlet	pentuplet	sextuplet	triplet
No. of H on Adjacent C	2	NA	4	5	2
Assignment	$-CH_2 - CH_2OH$	$-CH_2OH$	$-CH_2 - CH_2OH$	$-CH_2 - CH_2 - CH_2OH$	$CH_3 - CH_2$
Special features ( e.g. Coupling constants)	$J_2 = 7.5Hz$	NA	$J_2 = 5.0Hz, J_3 = 3Hz$	$J_2 = 3.8Hz$	$J_2 = 10Hz$

Table 3: Interpretation of  $^{13}\text{C}$  NMR spectroscopic data

	1-butanol					
	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5	Signal 6
Chemical Shift	171.24	64.38	30.7	21	19.17	13.72
Assignment	$\text{H}_3 - \underline{\text{C}}\text{O}_2 -$	$-\text{CO}_2 - \underline{\text{C}}\text{H}_2 -$	$\underline{\text{C}}\text{H}_3 - \text{CO}_2 -$	$-\text{CO}_2 - \underline{\text{C}}\text{H}_2 - \underline{\text{C}}\text{H}_2 -$	$-\text{CO}_2 - \text{CH}_2 - \underline{\text{C}}\text{H}_2 -$	$-\text{CO}_2 - \text{CH}_2 - \text{CH}_2 - \underline{\text{C}}\text{H}_3$

Table 4: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Position of Signal ( $\text{cm}^{-1}$ )	3350	2970	2950	1070
Intensity	Very strong	strong	strong	strong
Assignment	O-H stretch	C-H stretch (saturated)	C-H stretch(saturated)	C - O, stretch

## Butyl Ethanoate

### Structure

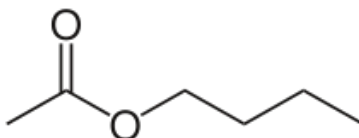


Figure 2: Structure of Butyl Ethanoate

Table 5: Interpretation of  $^1\text{H}$  NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	4.07	2.05	1.55	1.39	0.93
Ratio of signal	2	3	2	2	3
Multiplicity	triplet	singlet	pentuplet	sestuplet	triplet
No. of H on Adjacent C	2	0	4	5	2
Assignment	$\text{O} - \text{CH}_2 - \text{CH}_2 -$	$\text{CH}_3 - \text{CO}_2 -$	$-\text{CH}_2 - \text{CH}_2\text{OH}$	$-\text{CH}_2 - \text{CH}_2 - \text{CH}_2\text{OH}$	$\text{CH}_3 - \text{CH}_2 -$
Special features ( e.g. Coupling costants)	$J_2 = 7.5\text{Hz}$	NA	$J_2 = 4.3\text{Hz}, J_3 = 2.7\text{Hz}$	$J_2 = 5\text{Hz}$	$J_2 = 5\text{Hz}$

Table 6: Interpretation of  $^{13}\text{C}$  NMR spectroscopic data

	Butyl Ethanoate			stuff
	Signal 1	Signal 2	Signal 3	Signal 4
Chemical Shift	62.55	34.85	18.96	13.88
Assignment	$\text{CH}_2 - \underline{\text{C}}\text{H}_2\text{OH}$	$-\text{CH}_2 - \underline{\text{C}}\text{H}_2 - \text{CH}_2\text{OH}$	$-\text{CH}_3 - \underline{\text{C}}\text{H}_2 - \text{CH}_2 - \text{CH}_2\text{OH}$	$\underline{\text{C}}\text{H}_3 - \text{CH}_2 -$

Table 7: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Position of Signal ( $\text{cm}^{-1}$ )	3000	2930	1760	1060
Intensity	strong	strong	very strong	strong
Assignment	C-H stretch(saturated)	C-H stretch (saturated)	C=O stretch	C - O, stretch

## Discussion and Conclusion

The percentage yield of the product was very low, below 50%. The low yield attained may be at least partially related to kinetic considerations. The reaction is relatively slow to reach equilibrium and as such even under reflux the 45 minute reaction period may have been insufficient to allow the reaction to equilibrate. It is also possible that although acetic acid was indeed in excess the excess may not have been sufficient to shift the equilibrium far enough to attain high yields. Finally it is also possible that despite the foil used vapour escaped through the distillation apparatus. The boiling point/ distillation point recorded was also significantly lower than that expected. This relatively low boiling point is most probably related to the low yield as the presence of unreacted reagent both of which have a lower boiling points, and may have begun to boil off first giving the initial reading. This explanation would make sense if the reaction mix had not yet fully equilibrated so that although the excess acetic acid was removed unreacted 1-butanol was still present as a contaminant. In Conclusion, a low yield with an imprecise melting point was attained, indicating an impure product probably contaminated with unreacted reactants, especially 1-butanol.

Date Submitted: 16/08/2018

Signature: \_\_\_\_\_

## Prelab

### Question #1

Esters are usually synthesis by the combination of alcohols and carboxylic acids, although acyl chlorides can also be used in place of the carboxylic acid. These reactants are then heated, normally under reflux, with a strong acid or base catalyst. One of the reactants is normally maintained in excess pushing the equilibrium position to the right, to ensure high yield.

### Question #2

Sodium carbonate reacts with the excess Acetic acid:  $Na_2CO_3 + 2CH_3CO_2H \rightarrow 2CH_3CO_2Na + H_2O + CO_2$ . The sodium salt formed will precipitate out and can be removed during filtration to purify the product.

### Question #3

The acid catalyst provides  $H^+$  ions which attack the C=O bond leading to the formation of a carbocation  $C^+ - OH$ . The carbocation is a stronger electrophile than the delta positive charge on a normal carbonyl group and so the reaction is speed up considerably.