Organic Chemistry, Lab 6: Microscale sythesis: oxidation and dehydration

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Submission Information

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Experiment Number: *O4*
Demonstrator: *Thabo*
Date of Experiment: *02/08/2018*
Title: *Synthesis and purification, of carboxylic acid, by purification.*

Reaction Scheme

Yield Calculations.

Part A

Table 1: Limiting Reagent Calculations

Reactants Used	**Molecular Weight**($g \cdot mol^{-1}$)	**Mass**(g)	**Number of Moles(mol)**
Cyclohexanone	98.15	$0.9478g \cdot ml^{-1} \cdot 1.0ml = 0.9478g$	$\left(\frac{0.95g}{98.15g \cdot mol^{-1}}\right) = 9.7 \cdot 10^{-3}$
$NaBH_4$	37.83	0.2056	$\left(\frac{0.2056g}{37.83g \cdot mol^{-1}}\right) = 5.435 \cdot 10^{-3}$

Limiting Reagent: *Cyclohexanone* (as $NaBH_4$ and cyclohexanone react 2:1 and moles of $NaBH_4 >$ 2x number of moles of cyclohexanone)

Molecular Weight of Product: $100.158g \cdot mol^{-1}$
Mass of Product: $8.9162g - 8.3010g = 0.5152g$
Theoretical Yield: $9.7 \cdot 10^{-3} mol \cdot 100.158g \cdot mol^{-1} = 0.9672g$
Percentage Yield: $\frac{0.5152g}{0.97g} \cdot 100\% = 53\%$

Part B

Table 2: Limiting Reagent Calculations

Reactants Used	**Molecular Weight**$(g \cdot mol^{-1})$	**Mass**(g)	**Number of Moles(mol)**
Cyclohexanol	100.158	$0.9624g \cdot ml^{-1} \cdot 2.00ml = 1.93g$	$\left(\frac{1.925g}{100.158g \cdot mol^{-1}}\right) = 1.93 \cdot 10^{-2}$

Limiting Reagent: *Cyclohexanol*
Molecular Weight of Product: $82.143 \cdot mol^{-1}$
Mass of Product: $11.1998g - 10.2041g = 0.9957g$
Theoretical Yield: $1.93 \cdot 10^{-2} mol \cdot 82.143g \cdot mol^{-1} = 1.59g$
Percentage Yield: $\frac{0.9957g}{1.59g} \cdot 100\% = 62.6\%$

Product analysis and observation

Part A

Physical state of product

Colorless liquid

NMR/IR spectra

Cyclohexanone

Structure

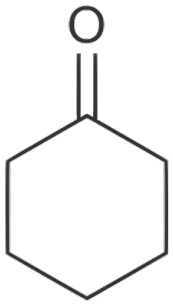

Figure 1: Cyclohexanone

Table 3: Interpretation of ^1H NMR spectroscopic data

	Signal 1	Signal 2	Signal 3
Chemical Shift	2.34	1.88	1.73
Ratio of signal	2	2	1
Multiplicity	triplet	pentuplet	pentuplet
No. of H on Adjacent C	2	4	4
Assignment	$CH_2 - C\underline{H_2}(C(=O) - C$	$-C(=O) - CH_2 - CH_2 - C\underline{H_2}-$	$-C\underline{H}(-CH_2)(-CH) - CH$
Special features (e.g. Coupling costants)	$J_2 = 12.5Hz$	$J_2 = 3.75Hz$	$J_2 = 4Hz$

Table 4: Interpretation of ^{13}C NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Chemical Shift	212.08	41.99	27.06	25.03
Assignment	$\underline{C} = O$	$-CH_2 - \underline{C}H_2 - C(=O)-$	$-CH_2 - \underline{C}H_2 - CH_2 - C(=O)-$	$-\underline{C}H_2 - CH_2 - CH_2 - C(=O)-$

Table 5: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3
Position of Signal (cm^{-1})	2970	2860	1710
Intensity	strong	strong	very strong
Assignment	=C-H stretch	C-H stretch	C=O stretch

Cyclohexanol

Structure

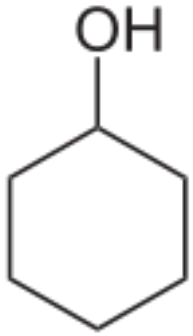

Figure 2: Cyclohexanol

Table 6: Limiting Reagent Calculations

Reactants Used	**Molecular Weight**($g \cdot mol^{-1}$)	**Mass**(g)	**Number of Moles(mol)**
Cyclohexanol	100.158	$0.9624g \cdot ml^{-1} \cdot 2.00ml = 1.93g$	$\left(\frac{1.925g}{100.158g \cdot mol^{-1}}\right) = 1.93 \cdot 10^{-2}$

Table 7: Interpretation of ^{13}C NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Chemical Shift	70.13	35.41	25.39	24.08
Assignment	$\underline{C}-OH$	$-CH_2-\underline{C}H_2-C(OH)-$	$-CH_2-\underline{C}H_2-CH_2-C(OH)-$	$-\underline{C}H_2-CH_2-CH_2-C(OH)-$

Table 8: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Position of Signal (cm^{-1})	3400	2950	2860	1050
Intensity	strong	strong	strong	strong
Assignment	=O-H stretch	C-H stretch	C-H stretch	$C-O$, stretch

Part B

Physical state of product

Colorless liquid

Boiling point

Expected:

$82°C$ at $760mmHg \rightarrow 73°C$ at $740mmHg$

Found:

$70-75°C$ at $740mmHg$

NMR/IR spectra

Cyclohexene

Structure

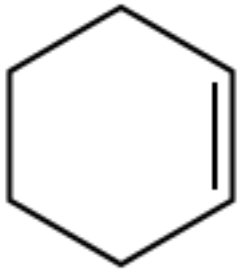

Figure 3: Cyclohexene

Table 9: Interpretation of ^1H NMR spectroscopic data

	Signal 1	Signal 2	Signal 3
Chemical Shift	5.67	1.99	1.61
Ratio of signal	1	2	2
Multiplicity	singlet	doublet	triplet
No. of H on Adjacent C	0	1	2
Assignment	$-C\underline{H} = C$	$CH_2 - C\underline{H_2} - CH = CH-$	$C\underline{H_2} - CH_2 - CH = CH-$
Special features (e.g. Coupling costants)		$J_2 = 2Hz, J_3 = 1.25Hz$	$J_2 = 2.5Hz$

Table 10: Interpretation of ^{13}C NMR spectroscopic data

	Signal 1	Signal 2	Signal 3
Chemical Shift	127.27	25.18	134.79
Assignment	$\underline{C}H = CH-$	$\underline{C}H_2 - CH = CH$	$\underline{C}H_2 - CH_2 - CH = CH$

Table 11: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Position of Signal (cm^{-1})	3020	2920	2870	1680
Intensity	medium	strong	strong	weak
Assignment	=C-H stretch	C-H stretch	C-H stretch	$C = C$, stretch

Discussion and Conclusion

In both steps there was a significant loss of product, more especially the fist step in which cyclohexanone was reduced. This loss was most probably due to the loss of vapour during relfux as the relfux condensers were sealed only with aluminium foil, and not air tight seals. There may also have been a loss of vapour through the flow out pipe if the vapour was not cooled rapidly enough to condense within the holding beaker. This explanation coincides with the lower yield of the first step, as the reflux temperature was higher and hence more vapour was lost from the system. The purity of the final cyclohexene product however appeared relatively high. The observed boiling point range fell closely around the range anticipated for the given atmospheric pressure, and from the TLC it can been seen that the product ran as a single dot, closely in line with the cyclohexene sample run, and well below the cyclohexanone and cyclohexanol samples. In conclusion, low yields were achieved due to vapour loss, however the final product was of high purity, demonstration very similar physical and chemical properties to pure standard samples.

Date Submitted: 16/08/2018 Signature:_____