Organic Chemistry, Lab 5: carbon-carbon bond formation in aldol and diels-Alder Reactions

Keelan Krinsky

Submission Information

Name: *Keelan Krinksy*
Student number: *1634953*
Locker number: *365* Group: *7*
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)**
Maleic Anhydride	98.06	$0.201g$	$\left(\frac{0.201g}{98.06g \cdot mol^{-1}}\right) = 2.05 \cdot 10^{-3}$
Cyclopentadiene	66.10	$0.786g \cdot ml^{-1} \cdot 0.20ml = 0.16g$	$\left(\frac{0.16g}{66.10g \cdot mol^{-1}}\right) = 2.4 \cdot 10^{-1}$

Limiting Reagent: *Maleic Acid*
Molecular Weight of Product: $164.16g \cdot mol^{-1}$

Mass of Product: $8.0149g - 7.864g = 0.1509g$
Theoretical Yield: $2.05 \cdot 10^{-3} mol \cdot 164.16g \cdot mol^{-1} = 0.337g$
Percentage Yield: $\frac{1.509g}{0.337g} \cdot 100\% = 44.8\%$

Part B

Table 2: Limiting Reagent Calculations

Reactants Used	**Molecular Weight**$(g \cdot mol^{-1})$	**Mass**(g)	**Number of Moles(mol)**
Benzyldehyde	106.12	$1.044g \cdot ml^{-1} \cdot 0.60ml = 0.63g$	$\left(\frac{0.63g}{106.12g \cdot mol^{-1}}\right) = 5.9 \cdot 10^{-3}$
Acetone	58.08	$0.7845g \cdot ml^{-1} \cdot 0.22ml = 0.17g$	$\left(\frac{0.17g}{58.08g \cdot mol^{-1}}\right) = 2.9 \cdot 10^{-3}$

Limiting Reagent: *Acetone*
Molecular Weight of Product: $234.29 \cdot mol^{-1}$
Mass of Product: $8.5849g - 8.0090g = 0.1509g$
Theoretical Yield: $2.9 \cdot 10^{-3} mol \cdot 234.29g \cdot mol^{-1} = 0.68g$
Percentage Yield: $\frac{0.5759g}{0.68g} \cdot 100\% = 85\%$

Product analysis and observation

Part A

Physical state of product

white (crystalline) solid

Melting point

Expected:

$165°C - 167°C$

Found:

$162°C - 168°C$

NMR/IR spectra

cis-5-Norbornene-endo-5,6-dicarboxylic anhydride

Structure

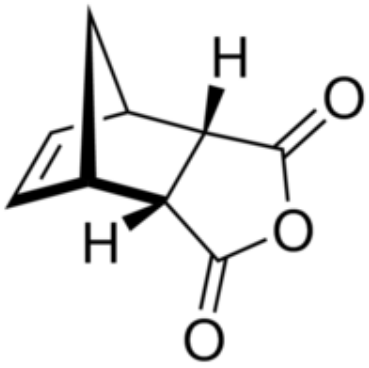

Figure 1: cis-5-Norbornene-endo-5,6-dicarboxylic anhydride

Table 3: Limiting Reagent Calculations

Reactants Used	**Molecular Weight**$(g \cdot mol^{-1})$	**Mass**(g)	**Number of Moles(mol)**
Benzyldehyde	106.12	$1.044g \cdot ml^{-1} \cdot 0.60ml = 0.63g$	$\left(\frac{0.63g}{106.12g \cdot mol^{-1}}\right) = 5.9 \cdot 10^{-3}$
Acetone	58.08	$0.7845g \cdot ml^{-1} \cdot 0.22ml = 0.17g$	$\left(\frac{0.17g}{58.08g \cdot mol^{-1}}\right) = 2.9 \cdot 10^{-3}$

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

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	172.88	135.97	52.89	47.55	45.81
Assignment	$CH - \underline{C}O_2-$	$-CH = \underline{C}H-$	$-CH\underline{C}H - CO_2$	$CH - \underline{C}H(-CH_2) - CH$	$CH - \underline{C}H_2 - CH$

Table 5: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Position of Signal (cm^{-1})	3010	2980	1780	1690	1050
Intensity	weak	weak	intermediate	strong	strong
Assignment	=C-H stretch	C-H stretch	C=O stretch	$C = C$, stretch	$C - O$, stretch

Part B

Physical state of product

light yellow (crystalline) solid

Melting point

Expected:

$110°C - 111°C$

Found:

$109°C - 111°C$

NMR/IR spectra

Dibenzylideneacetone

Structure

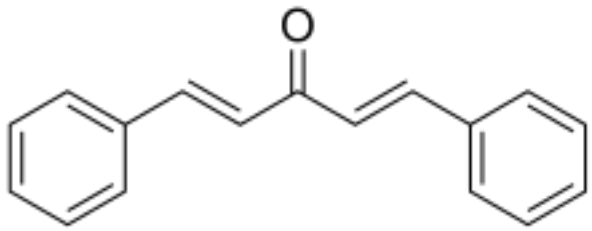

Figure 2: cis-5-Norbornene-endo-5,6-dicarboxylic anhydride

Table 6: Interpretation of ^1H NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	7.745	7.605	7.4	7.395	7.075
Ratio of signal	2	4	4	2	2
Multiplicity	doublet	multiplet	doublet	doublet	doublet
No. of H on Adjacent C	2(1 unique)	2	2(1 unique)	1	1
Assignment	$Ar - C_4C\underline{H}$	$Ar - C_{3,5}C\underline{H}$	$Ar - C_{2,6}C\underline{H}$	$CH = C\underline{H} - C = O-$	$Ar - C\underline{H} + Ch - C(= O)-$
Special features (e.g. Coupling costants)	$J_2 = 15Hz$	$J_2 = 2.5Hz, J_3 = 1.25Hz$	$J_2 = 1Hz$	$J_2 = 5.0Hz$	$J_2 = 1Hz$

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

cis-5-Norbornene-endo-5,6-dicarboxylic anhydride							
	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5	Signal 6	NA
Chemical Shift	188.85	143.27	134.79	130.47	128.94	128.37	125.42
Assignment	$\underline{C} = O$	$Ar - \underline{C}H = CH$	$Ar - CH = \underline{C}H - CO_2$	$Ar - \underline{C_4}$	$Ar - \underline{C_{3,5}}$	$Ar - \underline{C_{2,6}}$	$Ar - \underline{C_1}(-CH)$

Table 8: Interpretation of IR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Position of Signal (cm^{-1})	3020	3000	1700	1640	1590
Intensity	weak	weak	very strong	intermediate	strong
Assignment	=C-H stretch	C-H stretch	C=O stretch	$C = C$, stretch	$C = C$, conjugated

Discussion and Conclusion

Date Submitted: 16/08/2018 Signature:_____