Organic Chemistry, Lab 3b:

Keelan Krinsky

Submission Information

Name: *Keelan Krinksy*
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Locker number: *365* Group: *7*
Experiment Number: *O3(b)*
Demonstrator:
Date of Experiment: *02/08/2018*
Title: *Synthesis and purification, of carboxylic acid, by purification.*

Table 1: Limiting Reagent Calculations

Reactants Used	**Molecular Weight**$(g \cdot mol^{-1})$	**Mass**(g)	**Number of Moles(mol)**
Benzyl Alcohol	108.14	$1.044g \cdot ml^{-1} \cdot 0.51ml = 0.53$	$\left(\frac{0.53g}{108.14g \cdot mol^{-1}}\right) = 4.8 \cdot 10^{-3}$
Potassium Permanganate	158.034	$6.04g/100ml \cdot 25.0ml = 1.51$	$\left(\frac{1.51g}{158.034g \cdot mol^{-1}}\right) = 9.55 \cdot 10^{-3}$
Sodium Carbonate	105.99	0.5009	$\left(\frac{0.5009g}{105.99g \cdot mol^{-1}}\right) = 4.726 \cdot 10^{-3}$
HCl	36.46	Excess	Excess

Limiting Reagent: *Benzyl Alcohol*

Yield Calculations

Molecular Weight of Product: $122.12g \cdot mol^{-1}$
Mass of Product: $0.4783g$
Theoretical Yield: $4.8 \cdot 10^{-3} mol \cdot 122.12g \cdot mol^{-1} = 0.59g$
Percentage Yield: $\frac{0.4783g}{0.59g} \cdot 100\% = 81\%$

Product analysis and observation

Physical state of product

Solid (White needle like crystals)

Melting/Boiling Point of product

Expected:

$122°C$

Found:

$119 - 124\,°C$

NMR/IR spectra

Benzyl Alcohol

Structure

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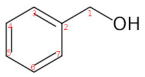

Figure 1: 2D Formula of Benzyl Alcohol

Table 2: Interpretation of ^{1}H NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	7.29	7.25	7.22	4.48	2.5
Ratio of signal	2	2	1	2	1
Multiplicity	triplet	doublet	triplet	singlet	singlet
No. of H on Adjacent C	2	1	2	0	0
Assignment	$Ar - C_{4,6}\underline{H}$	$Ar - C_5\underline{H}$	$Ar - C_{3,7}\underline{H}$	$C_6H_5 - C\underline{H}_2$	$Ar - C_{3,7}\underline{H}$
Special features (e.g. Coupling costants)	$J_{\text{ortho}} = 8Hz$	$J_{\text{orthp}} = 7.8Hz$	$J = 8.1Hz$		

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

Benzyl Alcohol					
	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	140.91	128.38	127.39	126.95	64.7
Assignment	$C_6H_5\underline{C}H_2OH$	$Ar - C_2$	$Ar - C_{3,7}$	$Ar - C_{4,6}$	$Ar - C_5$

Table 4: Interpretation of IR spectroscopic data

Benzyl Alcohol						
	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5	Signal 6
Position of Signal (cm^{-1})	3225	3032	2874	1602	1501	1454
Intensity	Very strong	strong	strong	strong	strong	Very strong
Assignment	O-H stretch	C-H stretch (saturated)	C-H stretch(unsaturated)	$C = C$, Aromatic	$C = C$, Aromatic	$C = C$, Aromatic

Benzoic Acid

Structure

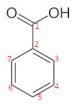

Figure 2: 2D Formula of Benzoic Acid

Table 5: Interpretation of ^1H NMR spectroscopic data

	Signal 1	Signal 2	Signal 3	Signal 4
Chemical Shift	12.09	8.12	7.62	7.45
Ratio of signal	1	2	2	2
Multiplicity	singlet	doublet of doublets	doublet of doublets	triplet
Number of Carbon on Adjacent Hydrogen	0	1	1	2
Assignment	$COO\underline{H}$	$Ar - C_{3,7}\underline{H}$	$Ar - C_{4,6}\underline{H}$	$Ar - C_5\underline{H}$
Special features(e.g. Coupling costants)		$J_{\text{ortho}} = 7.98$	$J_{\text{ortho}} = 4.77, J_{\text{meta}} = 1.0$	$J_{\text{ortho}} = 8.77, J_{\text{meta}} = 4.86$

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

Benzoic Acid					
	Signal 1	Signal 2	Signal 3	Signal 4	Signal 5
Chemical Shift	172.77	133.83	130.28	129.44	128.49
Assignment	$C_6H_5\underline{C}H_2O_2H$	$Ar - C_5$	$Ar - C_{3,7}$	$Ar - C_2$	$Ar - C_{4,6}$

Table 7: Interpretation of IR spectroscopic data

Benzoic Acid				
	Signal 1	Signal 2	Signal 3	Signal 4
Position of Signal (cm^{-1})	3073	1690	1340	950
Intensity	strong	very strong	strong	moderate
Assignment	$O - H$ stretching	$C = O$, stretching	$O - H$ bending	$C - C/C = C$ of aromatic ring

TLC

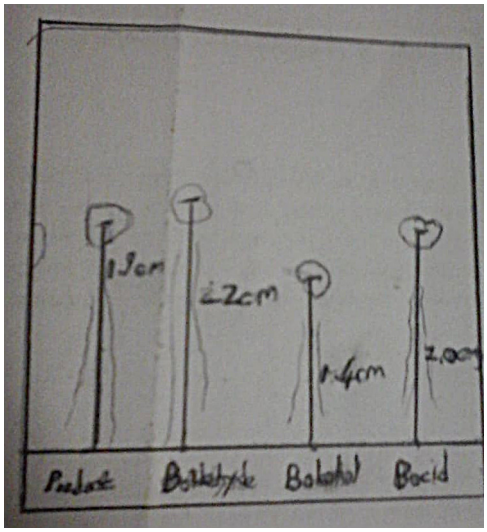

Figure 3: TLC of Product and reactants, with Ethanol solvent

Calculated Rf values.

1. Product $=\frac{1.9cm}{6.0cm} = 0.32$
2. Benzaldehyde $=\frac{2.2cm}{6.0cm} = 0.37$
3. Benzyl; alcohol$=\frac{1.4cm}{6.0cm} = 0.23$
4. Benzoic acid $\frac{2.0cm}{6.0cm} = 3.3$

Discussion and Conclusion

A relatively high percentage yield was achieved in the experiment], above 80%. This high yield may be attributable the addition of sodium carbonate, which reacted with the benzoic acid as it was formed to form sodium salts, shifting the equilibrium associated with alcohol oxidation to the right, increasing product formation. Moreover the conditions of reflux raising the reaction rate to considerable above that normally achieved in the volatile solvents required help to ensure that the reaction ran to completion, with no unreacted alcohol remaining. From the TLC it seems more likely, that the slight difference in theoretical and actual yield resulted from some alcohol stopping its oxidation at the aldehyde stage rather then fully oxidizing to form benzoic acid, as the product RF value was very close to that of benzoic acid, and could conceivable have contained a fraction of benzaldehyde, (the separation is not sufficient to clearly distinguish between acid and aldehyde) but appears to contain little or no alcohol. The Melting point observed is consistent with this explanation, as the small fraction of aldehyde (which cannot H bond) dropped the bottom limit for the observed melting point range slightly lower than the expected melting point. In conclusion, high product yield was achieved with the observed melting point, and TLC analysis indicating that aldehyde, and not alcohol, as the main contaminant.

Date Submitted: 08/08/2018 Signature:_____