

Friedel-Crafts Acylation for Synthesis of a Substituted Acetophenone

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Purpose: Combine an aromatic hydrocarbon, ortho-xylene, acting as a nucleophile with acetyl chloride, acting as an electrophile, aluminum trichloride acting as a catalyst, and dichloromethane as a solvent. Observe electrophilic aromatic substitution, Friedel Crafts' reaction, that forms 3,4 dimethylacetophenone. Isolate product by removing aluminum solids with acid and drying. Categorize the product using IR and H-NMR.

Reagent Table and Calculations:

Reagent/ Product	MW (g/mol)	Density (g/mL)	BP (°C)	MP (°C)	mmol	Volume (μL)	Mass (mg)
Ortho-xylene	106.16	0.8801	144	NA	(5.00)	(603)	(531)
					4.97	600	528
AlCl ₃ Aluminum Trichloride	133.34	NA	NA	NA	(6.00)	NA	(800)
					7.06		941
Acetyl Chloride	78.50	1.104	52	-112	7.00	500	552
3,4 Dimethyl- Acetophenone	148.20	NA	NA	NA	(4.97)	NA	(736)
					2.08		380

() – Theoretical

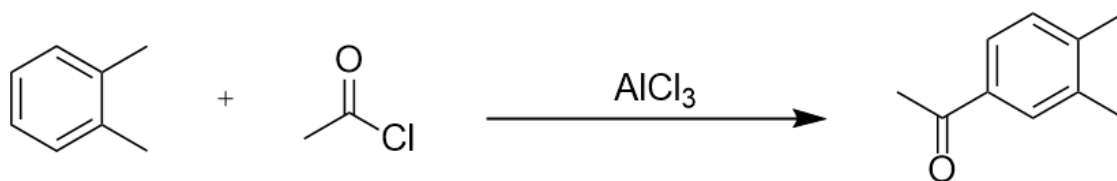
Ortho-xylene: $600\mu\text{L} = 0.6\text{mL}(0.8801\text{g}/1\text{mL}) = 528\text{mg}(1\text{mmol}/106.16\text{mg}) = 4.79\text{mmol}$

AlCl_3 : $800\text{mg} (1\text{mmol}/133.34\text{mmol}) = 6.00\text{mmol}$

3,4 Dimethylacetophenone: $380\text{mg} (1\text{mmol}/148.2\text{mg}) = 2.08\text{mmol}$

Record Calculations

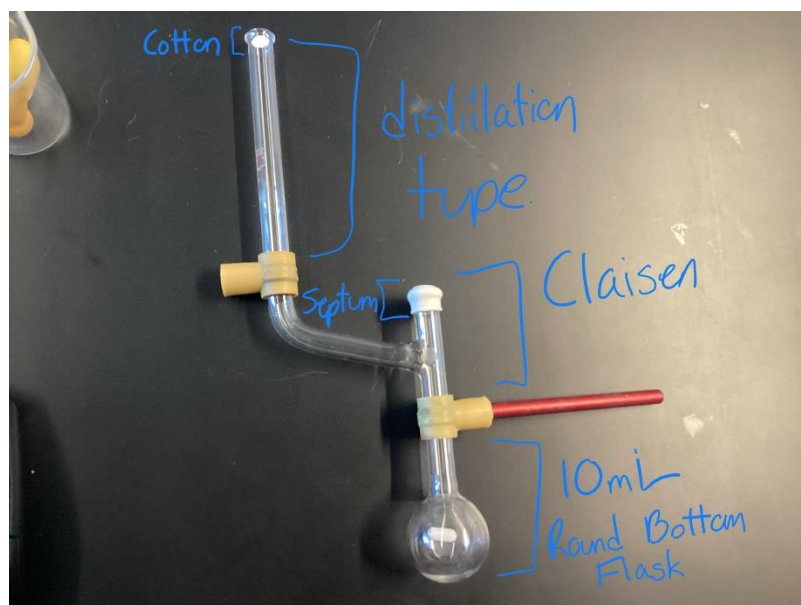
Net Reaction Equation: (Use Chemdraw)



References:

- (1) Kateley, L. J., *Guide for Organic Chemistry Laboratory*, Seventeenth edition, Lake Forest College, 2011
- (2) Sigma Aldrich. 3,4 Dimethylacetophenone. Merck 2025

Apparatus: Label Pieces



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

Apparatus was constructed. A magnetic stir bar and about 1.5mL of methylene chloride was added to the flask. A pre-weighted sample of aluminum trichloride was weighed for a mass of 8.293g. The sample was added to the flask using a funnel and the container was reweighed for a mass of 8.165. Another vial with a full weight of 10.520g and an empty weight of 9.707g was added for a total of 941mg of AlCl_3 .

The stir plate was turned on so the soln was stirred during additions. 0.5mL (7.00mL) of acetyl chloride was slowly dripped into the solution via syringe through the septum over 1-2min. The soln foamed as the acetyl chloride was added. The color of the soln shifted from cloudy white to golden yellow.

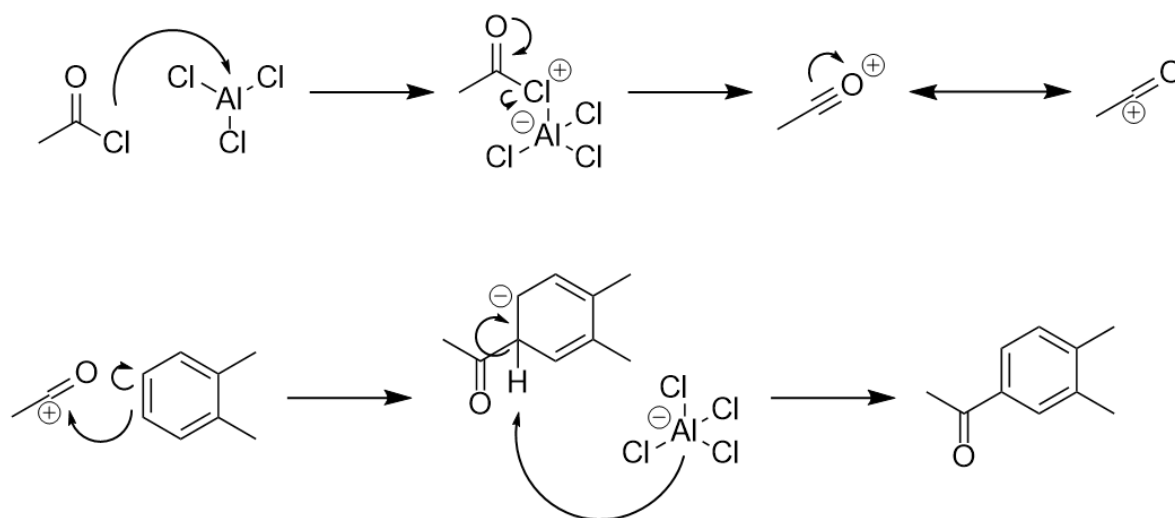
0.6mL (4.97mmol) of ortho-xylene was slowly dripped into the solution via syringe through the septum over 3-5min. The temperature of the soln increased since the reaction was

exothermic. The syringe and tube were washed with 1mL of solvent. Soln was stirred for 5min. The color darkened.

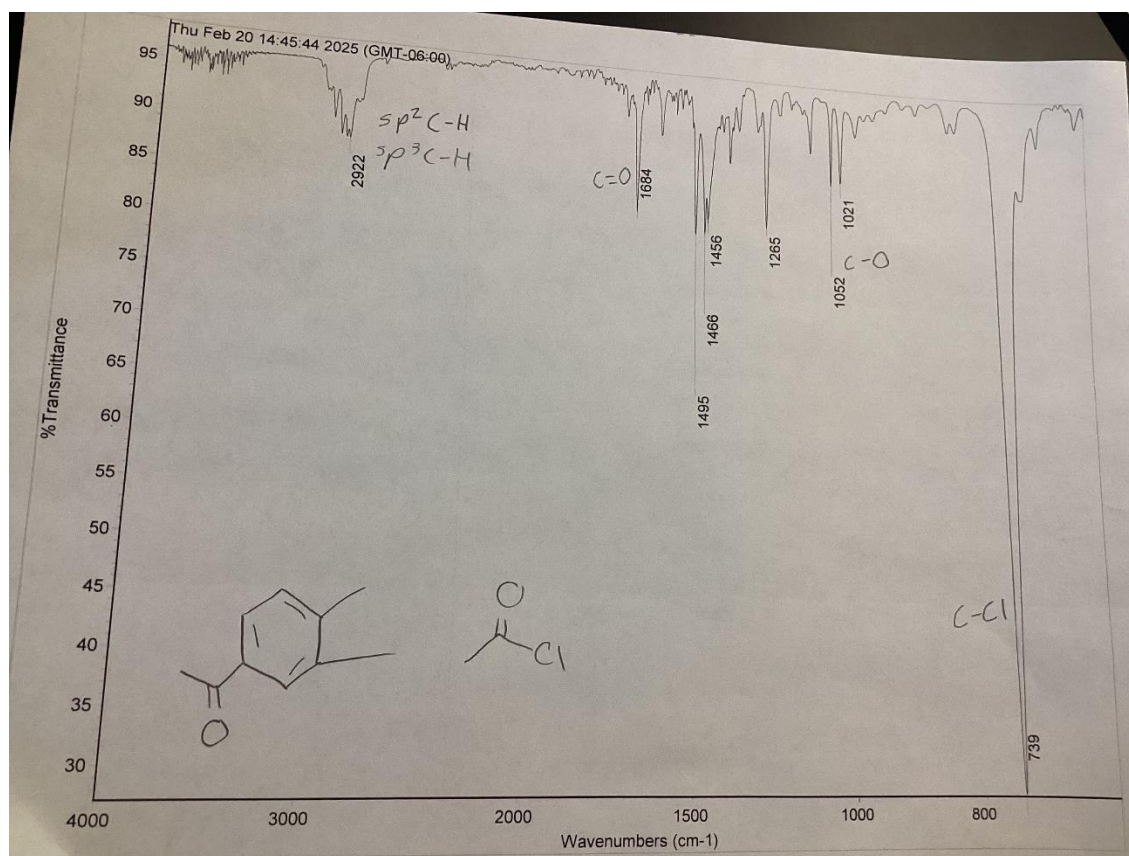
An ice bath was prepared if needed. Approximately 2mL of 6M HCl was pipetted into soln. Soln bubbled yellow, white solid formed, and soln became light yellow and transparent. 4 more mL of HCl were added. More HCl was added until all white solid dissolved. Then soln was transferred to separatory funnel. Small amount of solvent was used to wash as much soln as possible into funnel.

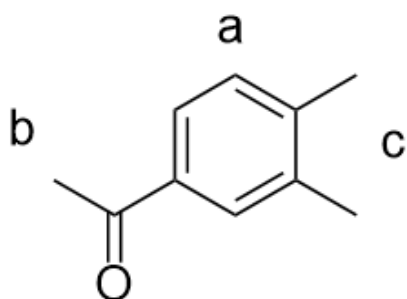
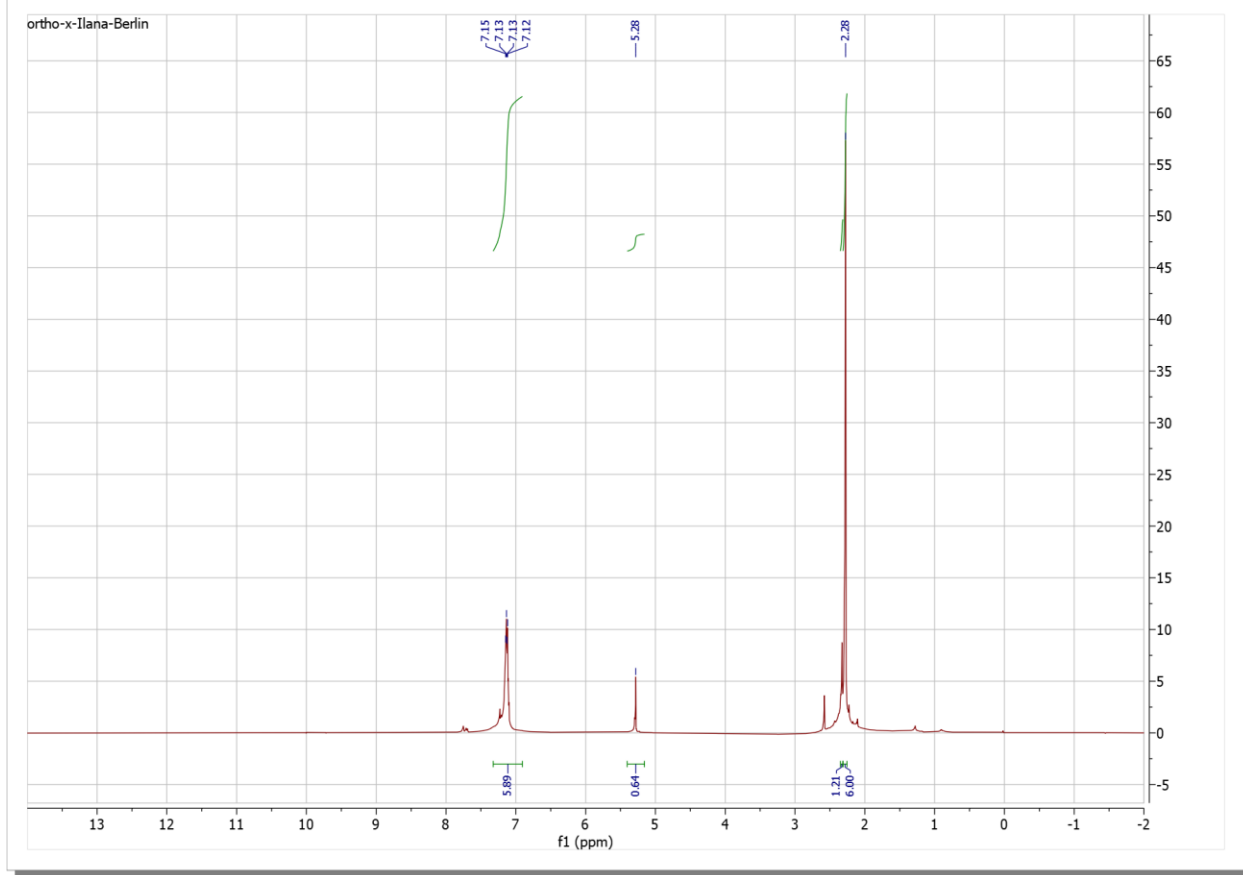
Soln was extracted 3 times with 3mL of solvent. The combined organic layer was washed with 3mL of water. The combined organic layer that was separated was washed with 3mL of sodium bicarb. Soln was dried with anhydrous sodium sulfate for 5min. Soln was decanted into weighed, mass of 12.863g centrifuge tube and dried. Product was weighed, percent yield of 42% was calculated and spectra were run.

Mechanism: (Use chemdraw and net reaction equation)



Spectra:





Peaks shifted due to starting material in solution.

Questions: 1) Explain why conjugation lowers the wavenumber for the carbonyl stretch from the typical 1715 cm^{-1} to about 1650 cm^{-1} ?

The C=O shows up lower in the IR Spectra because it is near the aromatic ring so they are conjugate. It is also a very weak peak because there is starting material remaining .

2) Which layer in the extraction was the organic layer? Explain.

The bottom layer of the separatory funnel was the organic layer because the solvent, dichloromethane, is denser than water despite the thickness of the water due to all the aluminum salt dissolved in it.

3) Why does this reaction stop after a single acetylation

The reaction stops after a single acetylation because the added ketone is an electron withdrawing/deactivating group. This draws electron density away from the aromatic ring, making it a worse nucleophile so the Friedel-Crafts reaction will not happen again.

Conclusion: (key findings, yield, and improvements)

The resulting percent yield was 42%. Product loss may have happened when adding the HCl as adding it too rapidly can burn the product. There was also product loss in the extraction process. To improve results add things to the solution at a slower, more controlled rate. Give the reaction longer to happen by increasing waiting times between steps.

There is still a lot of starting material in the product. This can be seen from the weak carbonyl peak and the strong C-Cl peak in the IR Spectra and the shifted peaks in the H-NMR spectra.

The Friedel-Crafts reaction is thermodynamically favorable because it is an exothermic reaction. The catalyst AlCl_3 helps increase the electrophilicity of acetyl chloride and later acts as a nucleophile, removing a H to reform the aromatic ring.