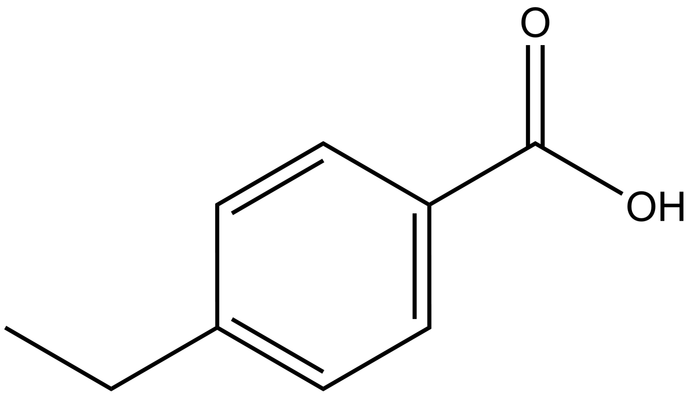
ETH zürich

Laboratory Course OACP I
Spring Semester 2024

The Multi Step Synthesis of 4-Ethylbenzoic Acid



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Abstract The objective of this experiment is to synthesize 4-ethylbenzoic acid and analyze the yield of the product. The synthesis is performed in four steps. In the first step 4-(bromomethyl)benzoic acid is synthesized by a radical reaction with 4-methylbenzoic acid. In the second step a S_N2 reaction transforms it into (4-carboxybenzyl)triphenylphosphonium. In the third step a Wittig reaction produces 4-vinylbenzoic acid and in the final step a palladium on carbon catalyst yields the product 4-ethylbenzoic acid. The success of the experiment is determined by thin layer chromatography and infrared spectrum measurements of each intermediate and the final product.

Zürich, May 15, 2024

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Introduction

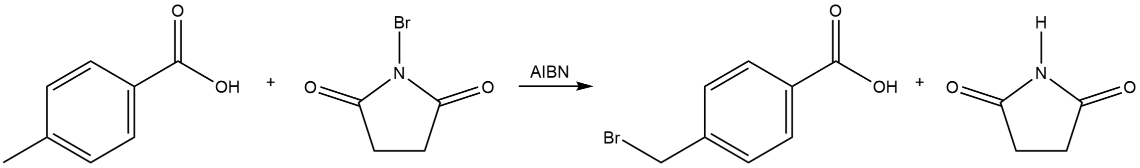
4-Ethylbenzoic acid is a benzene ring substituted with an ethyl group at the para position to a carboxylic acid group at the first position. The carboxylic acid group is a highly polar and reactive functional group due to the potential for hydrogen bonding and its ability to donate a proton (acidic nature). The ethyl group is non-polar, hydrophobic, and generally chemically inert compared to other functional groups. The benzene ring itself is a stable, planar, and aromatic ring of six carbon atoms, with alternating single and double bonds that are delocalized, giving the ring a significant amount of stability and reactivity in electrophilic aromatic substitution reactions.

4-Ethylbenzoic acid has a variety of applications. In organic synthesis it is often used as an intermediate in the preparation of more complex chemical compounds. It has been employed in the synthesis of ethyl 4-vinyl-α-cyano-β-phenylcinnamate, among other compounds. It is also used as an intermediate in the manufacture of pesticides, particularly for insecticides like tebufenozide and methoxyfenozide. It also finds use in the production of liquid crystals and can serve as an initiator in the creation of carbon nanotube materials [1].

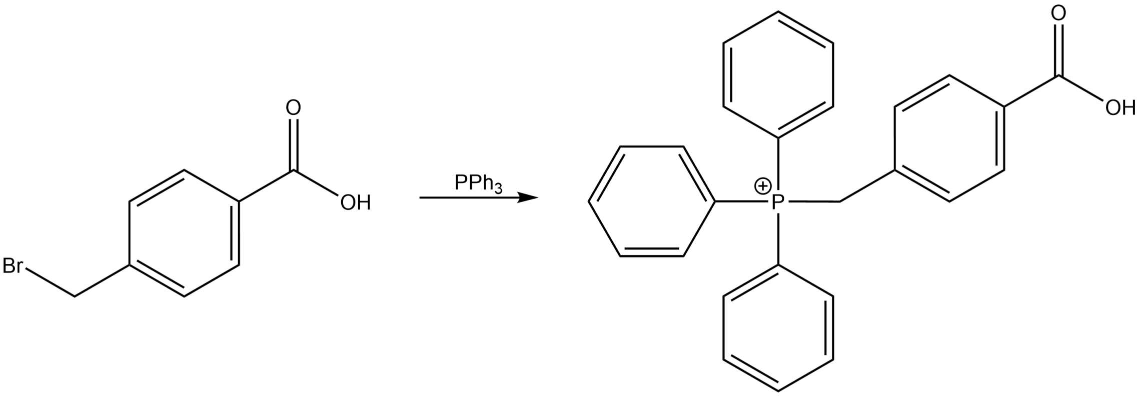
It appears as a powderous solid at room temperature with a white to beige colour [2]. It is soluble in benzene and toluene.

Material Properties

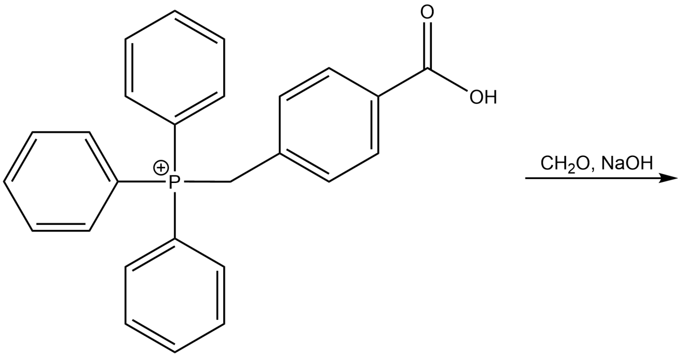
The synthesis of 4-ethylbenzoic acid is carried out in 4 steps. In the first step of the synthesis, 4-methylbenzoic acid undergoes a radical chain reaction in the presence of N-bromosuccinimide (NBS) and azobisisobutyronitrile (AIBN) to yield 4-(bromomethyl)benzoic acid.



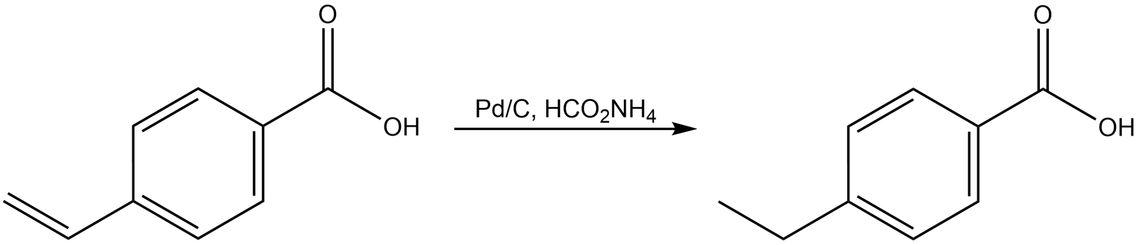
In the second step of the synthesis, 4-(bromomethyl)benzoic acid reacts with triphenylphosphine in an S$_N$2 reaction. This leads to the formation of (4-carboxybenzyl)triphenylphosphonium. Together with bromide anions it forms a salt called (4-carboxybenzyl)triphenylphosphonium bromide, abbreviated as CBTPB.



In the third step of the synthesis, (4-carboxybenzyl)triphenylphosphonium bromide is reacted with formaldehyde and sodium hydroxide in a Wittig reaction to produce 4-vinylbenzoic acid. This step involves the generation of a new carbon-carbon double bond, which is characteristic of the Wittig reaction mechanism.



In the final step, 4-vinylbenzoic acid is combined with ammonium formate and subjected to catalytic hydrogenation to synthesize 4-ethylbenzoic acid. This reaction is catalyzed by a 10 % palladium on carbon catalyst, effectively adding hydrogen atoms to the vinyl group to form the ethyl group in the product.



Substance	GHS Hazard Pictogram	H and P Phrases
CBTPB	-	-
Tert-butyl methyl ether	⚠ 🔥	H225, H315-P210, P233, P240, P241, P242, P303, P361, P353
4-Vinylbenzoic acid	⚠	H315, H319, H335-P261, P264, P271, P280, P302, P352, P305, P351, P338
Palladium on carbon	-	-
Ammonium formate	⚠	H319-P264, P280, P305, P351, P338, P337, P313
Sodium carbonate	⚠	H319-P264, P280, P305, P351, P338, P337, P313
4-Ethylbenzoic acid	⚠	H315, H319, H335, P261, P264, P264+P265, P271, P280, P302+P352, P304+P340, P305+P351+P338, P319, P321, P337+P317, P362+P364, P403+P233, P405

Substance	GHS Hazard Pictogram	H and P Phrases
4-Methylbenzoic acid		H317, P261, P272, P280, P302+P352, P333+P313, P362+P364
N-Bromosuccinimide (NBS)		H272, H290, H302, H314, H315, H319, H335, H400, P210, P220, P234, P260, P261, P264, P264+P265, P270, P271, P273, P280, P301+P317, P301+P330+P331, P302+P352, P302+P361+P354, P304+P340, P305+P351+P338, P305+P354+P338, P316, P319, P321, P330, P332+P317, P337+P317, P362+P364, P363, P370+P378, P390, P391, P403+P233, P405, P406, P501
Azobisisobutyronitrile (AIBN)		H242, H302, H332, H412, P210, P235, P273, P304, P340, P312, P370, P378, P403
4-(Bromomethyl)benzoic acid	-	-
Triphenylphosphine		H302, H317, H318, H372, P260, P280, P301+P312, P302+P352, P305+P351+P338
Formaldehyde		H226, H301, H311, H314, H317, H330, H335, H341, H350, H370, P201, P210, P233, P240, P241, P242, P260, P264, P270, P271, P280, P303+P361+P353, P304+P340+P310, P305+P351+P338
Sodium hydroxide		H290, H314, P234, P260, P280 P303+P361+P353, P304+P340+P310, P305+P351+P338
Hydrochloric acid		H290, H314, H335, P234, P261, P271, P303+P361+P353, P305+P351+P338

Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
4-Methylbenzoic acid [3]	136.15	-	181	274
N-Bromosuccinimide [4]	177.98	2.098	180	221
Azobisisobutyronitrile (AIBN) [5]	164.21	1.1	103	-
4-(Bromomethyl)benzoic acid [6]	215.04	-	226	-
Triphenylphosphine [7]	262.29	1.1	80	377
Tert-butyl methyl ether [8]	88.15	0.7353	-109	55
CBTPB [9]	477.34	-	-	-
Formaldehyde [10]	30.03	1.09	-92	98
Sodium hydroxide [11]	40.00	2.13	318	1390
Hydrochloric acid [12]	36.46	1.639	-114	-85
4-Vinylbenzoic acid [13]	148.16	-	143	282
Palladium on carbon [14]	106.42	12.02	1554	2970
Ammonium formate [15]	63.06	1.28	120	180
Sodium carbonate [16]	105.99	2.54	854	1600
4-Ethylbenzoic acid [17]	150.17	-	112	-

Safety Assessment

In the lab, each chemical demands specific safety measures. 4-Methylbenzoic acid, AIBN, Tert-butyl methyl ether (MTBE), 4-vinylbenzoic acid, ammonium carbonate, sodium carbonate and 4-ethylbenzoic acid can cause skin irritation. NBS, Triphenylphosphine, formaldehyde, sodium hydroxide and hydrochloric acid are strongly corrosive and can cause severe skin burns. NBS, triphenylphosphine and formaldehyde are considered serious health hazards. Formaldehyde and hydrochloric acid are acute toxic. Always avoid direct contact with all the chemicals listed. Also AIBN, formaldehyde and TBME are highly flammable, always keep them away from heat sources.

The entire reaction should be conducted in a well ventilated fume hood to avoid exposure to any harmful vapors, particularly from formaldehyde.

The handling of each requires appropriate personal protective equipment, cautious storage, and clear protocols for exposure response and spill management. Always adhere to detailed guidelines as provided by their respective Safety Data Sheets for safe laboratory practices.

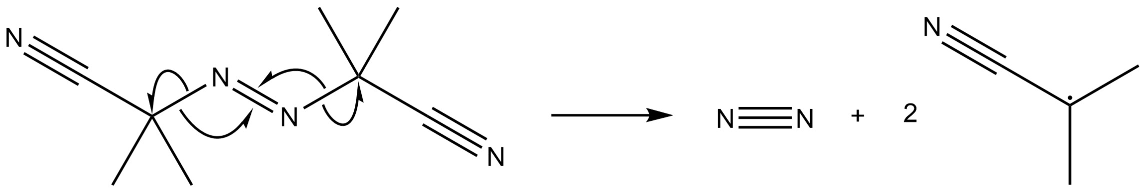
For avoiding any health issues that could be caused by the chemicals, gloves and safety googles should be worn in the laboratory. In a case of an accident the emergency number should be called. With the internal ETH-phones it would be 888, on personal mobile phones the number would be 044 342 11 88. If something get's into the eye, an eye shower has to be performed for at least 15 minutes and then the eye clinic from the university hospital Zurich has to be visited. If sulfuric acid gets spilled on the skin, the clothing over it has to be removed and the contaminated area on the skin has to be rinsed with running water for several minutes. Afterwards a doctor must be seen. All incidents should always be reported.

Waste Disposal

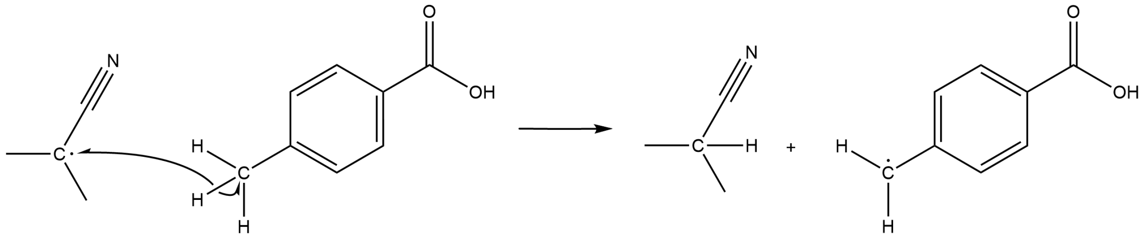
Tert-butyl methyl ether (MTBE) and formaldehyde should be disposed of in the organic solvent waste. NBS is a brominated compound and should be treated as chlorinated waste due to similar disposal requirements for halogenated compounds. 4-Methylbenzoic acid, 4-ethylbenzoic acid, 4-(Bromomethyl)benzoic acid, hydrochloric acid and 4-vinylbenzoic acid should be dissolved and disposed of into the acid waste. Sodium hydroxide and sodium carbonate should be dissolved and disposed of into the basic waste. (4-Carboxybenzyl)triphenylphosphonium bromide, palladium on carbon, ammonium formate, AIBN and triphenylphosphine should go into the solid waste.

Reaction Mechanism Step 1

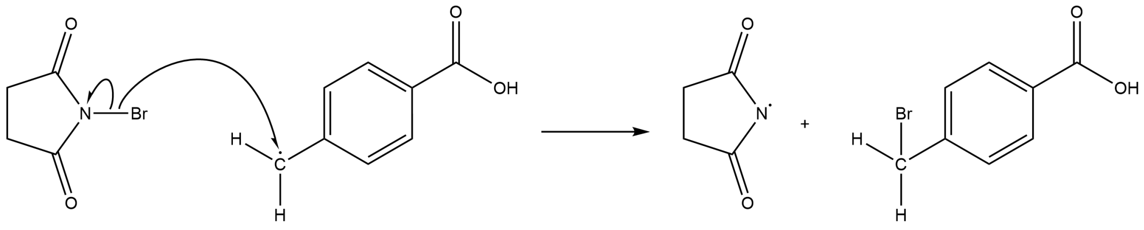
The Azobisisobutyronitrile (AIBN) is a initiator for radical reactions. It decomposes into a nitrogen gas molecule and two 2-cyanoprop-2-yl radicals.



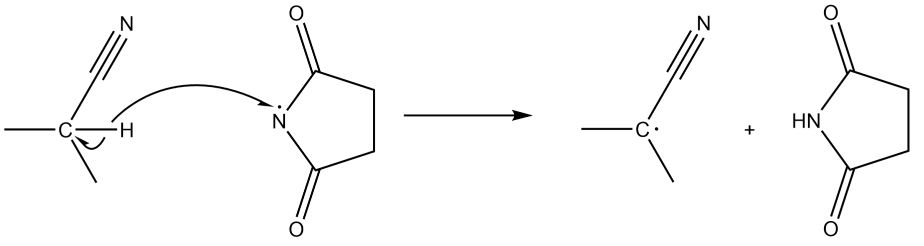
A 2-cyanoprop-2-yl radicals reacts with the 4-methylbenzoic acid and turns it into a radical. In return the 2-cyanoprop-2-yl radical get's a hydrogen atom what turns it into an isobutyronitrile molecule.



The 4-methylbenzoic acid radical gets a bromine atom from the NBS. The NBS turns into a radical.

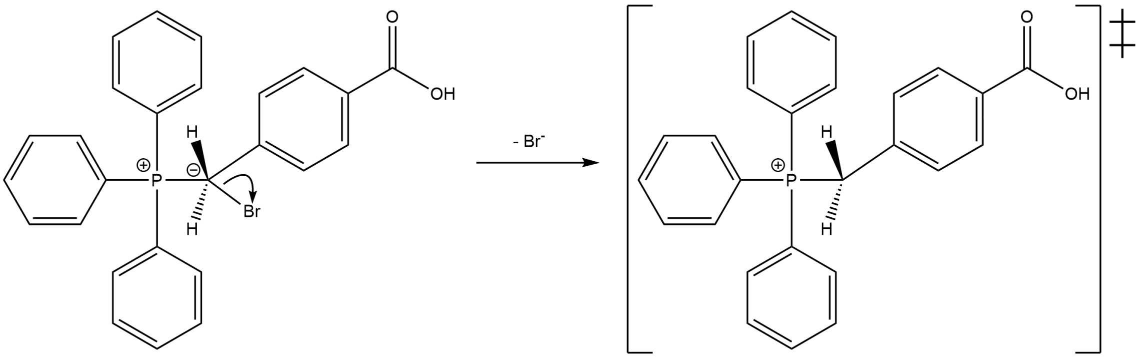


The NBS radical gets a hydrogen atom from the isobutyronitrile molecule and turns into a pyrrolidine-2,5-dione. The isobutyronitrile molecule turns back into a 2-cyanoprop-2-yl radical. That means the AIBN was used as a catalyser in this reaction, the cyanoprop-2-yl radicals can propagate the reaction further.

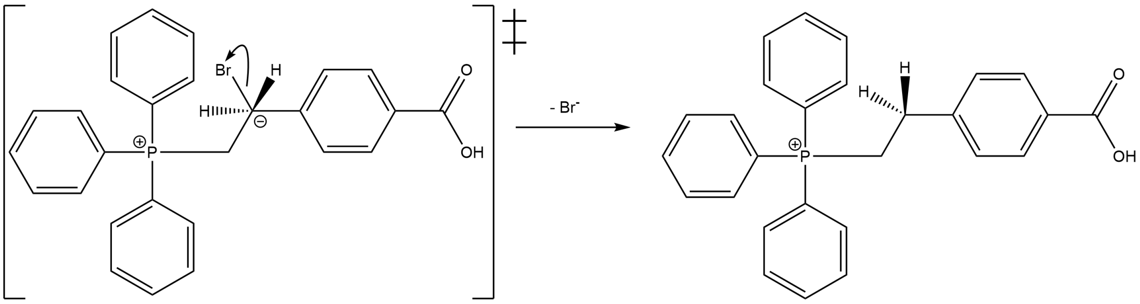


Reaction Mechanism Step 2

The Triphenylphosphine conducts a nucleophilic attack on the 4-(Bromomethyl)benzoic acid from the last reaction. This is a bimolecular nucleophilic substitution (S_N2) reaction. This forms a temporary intermediate.

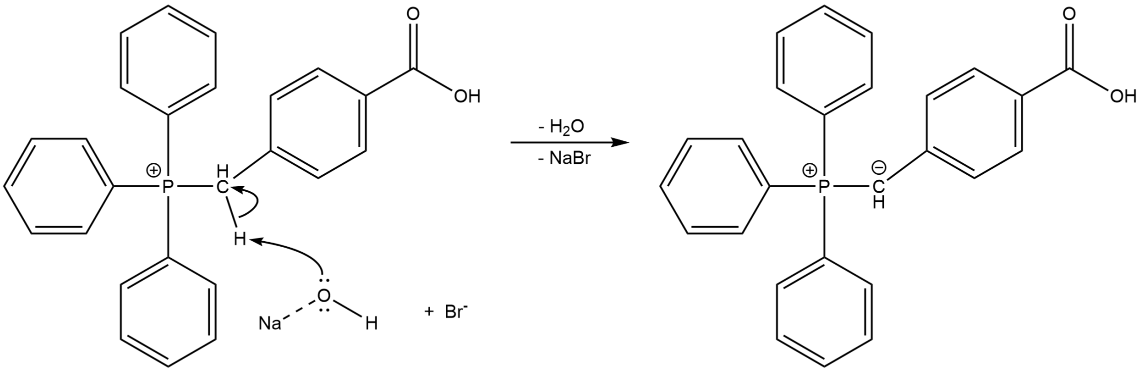


This intermediate is extremely unstable, normally carbon never builds five covalent bonds. The bromine is immediately forced to leave as an anion. This results in the formation of the (4-carboxybenzyl)triphenylphosphonium cation. Together with bromide anions it forms a salt called (4-carboxybenzyl)triphenylphosphonium bromide.

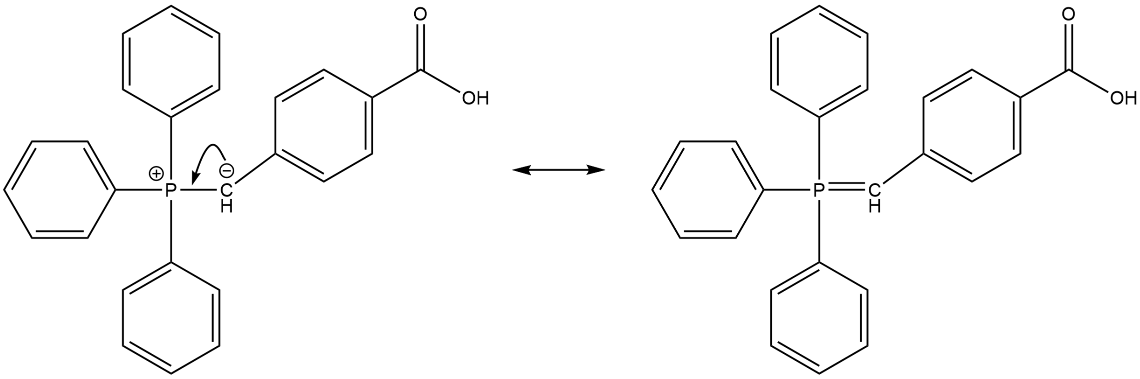


Reaction Mechanism Step 3

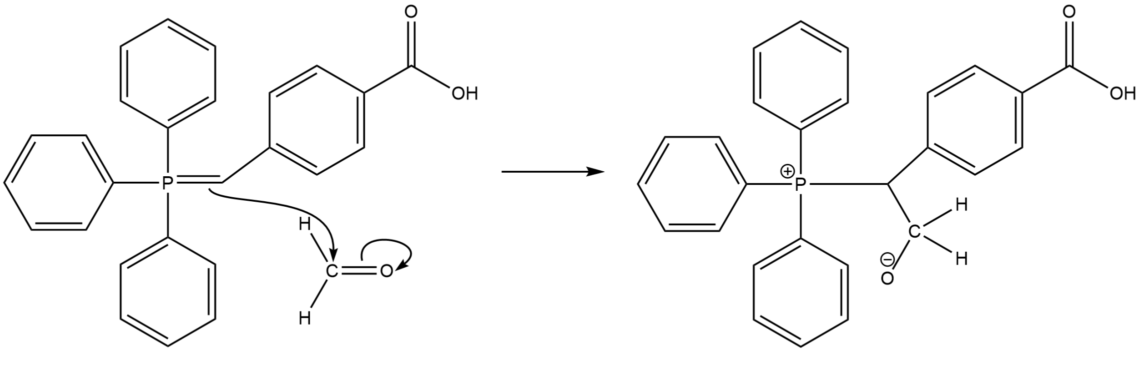
For the next step the Wittig reaction is used. The sodium hydroxide acts as a Brønsted base and deprotonates the (4-Carboxybenzyl)triphenylphosphonium. That results in the formation of a sodiumdihydroxide that immediately breaks up into a sodium cation and water. The sodium cation bonds together with the bromide ion and leaves as sodium bromide together with the water molecule.



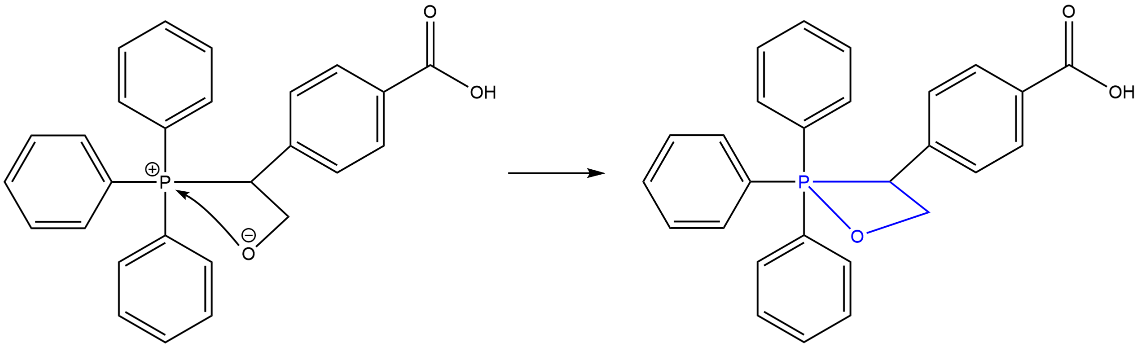
The intermediate has a resonance structure where the charges get distributed through a double bond between the phosphorus and the carbon.



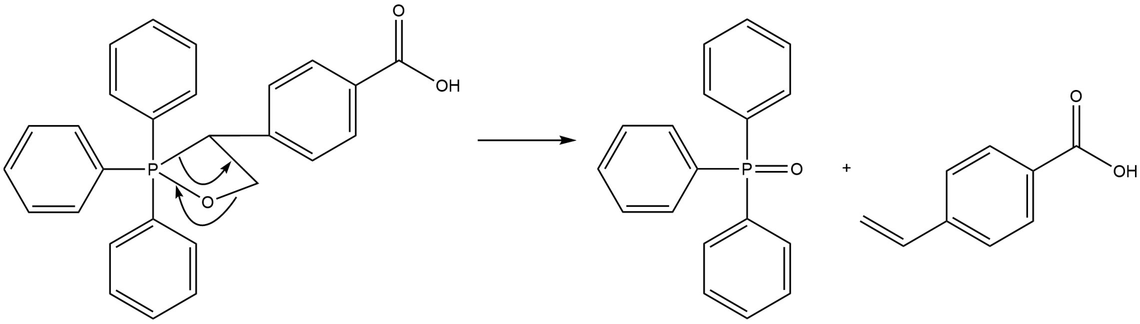
This double bond has a high electron density which conducts a nucleophilic attack on the carbon of the formaldehyde molecule. The oxygen from the formaldehyde takes up a negative charge and a single bond is built to connect both molecules.



The negative charge on the oxygen conducts a nucleophilic attack on the positively charged phosphorus to form a cyclic four ring.



This four ring has very high baeyer ring strain. This leads to the breaking up of the ring into triphenylphosphine oxide and 4-vinylbenzoic acid.

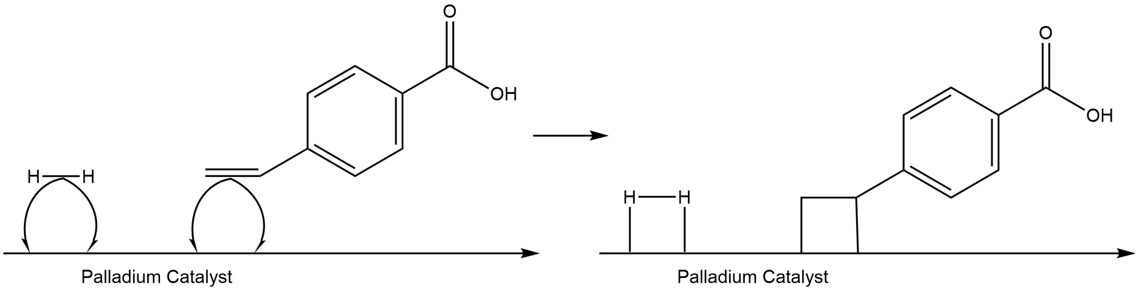


Reaction Mechanism Step 4

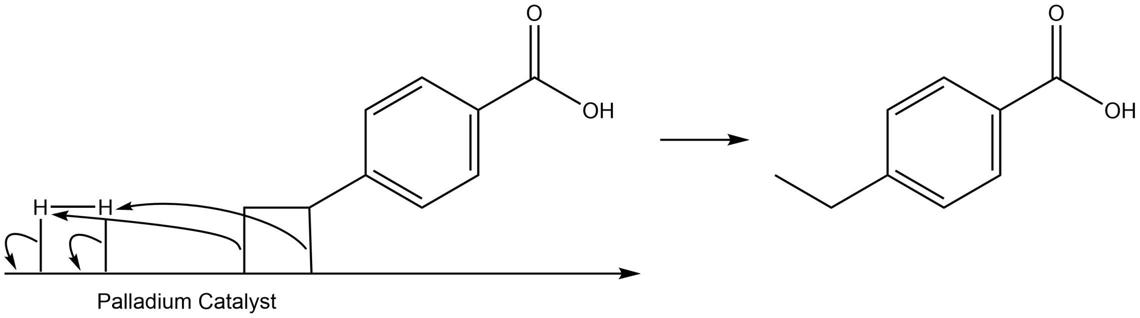
The ammonium formate decomposes upon heating to form hydrogen gas and carbon dioxide. This reaction provides the hydrogen needed for the hydrogenation process.

$$NH_4HCO_2 \xrightarrow{\text{heat}} H_2 + CO_2 + NH_3$$

The vinyl group of 4-vinylbenzoic acid is adsorbed onto the surface of the palladium catalyst, where the palladium atoms provide a surface for the reaction to occur. The pi electrons of the carbon-carbon double bond interact with the palladium metal. This leads to the opening of the double bond of the 4-vinylbenzoic acid and the opening of the hydrogen bond on the catalyst.



The palladium catalyst enables the adding of hydrogen atoms to 4-vinylbenzoic acid. The palladium serves as a facilitator for this addition without altering its own electron count in the process. The electrons from the carbon-carbon double bond in the vinyl group are redistributed as two hydrogen atoms attach to the carbons, what converts the double bond into a single bond and transforms the vinyl group into an ethyl group. As a result, the final product, 4-ethylbenzoic acid, is produced while the palladium catalyst is regained and can be used to catalyze the next hydrogenation cycle.



Atom Economy

Atom economy refers to the efficiency of a chemical reaction and it is calculated with the equation below. The atom economy has to be calculated for every step of the synthesis.

$$\text{AE} = \frac{M_{\text{Product}}}{M_{\text{Reagents}}}$$

The atom economy of the first reaction step is approximately 68%. That makes sense, the AIBN is a catalyst stays the same before and after the reaction. The only thing that really happens in this reaction is that the 4-methylbenzoic acid exchanges a hydrogen with a bromine to the NBS. The NBS is not part of the product so it's not counted in the calculation.

$$\text{AE} = \frac{215.04 \text{ g/mol}}{136.15 \text{ g/mol} + 177.98 \text{ g/mol}} \approx 68.46\%$$

The atom economy of the second reaction step is also 100%. Even though this is a (S_N2) reaction where the bromine leaves, the product is the salt that contains both the carboxybenzyl)triphenylphosphonium cations and the bromide anions.

$$\text{AE} = \frac{477.34 \text{ g/mol}}{215.04 \text{ g/mol} + 262.29 \text{ g/mol}} \approx 100\%$$

The atom economy of the third reaction step is approximately 27%. This can be explained with the fact that the huge triphenylphosphine oxide get's seperated from the product of the reaction 4-vinylbenzoic acid.

$$\text{AE} = \frac{148.16 \text{ g/mol}}{477.34 \text{ g/mol} + 30.03 \text{ g/mol} + 40.00 \text{ g/mol}} \approx 27.07\%$$

The atom economy of the fourth reaction step is approximately 71%. In this reaction the palladium on carbon only functions as a catalyst.

$$\text{AE} = \frac{150.17 \text{ g/mol}}{148.16 \text{ g/mol} + 63.06 \text{ g/mol}} \approx 71.10\%$$

The final atom economy would be calculated by multiplying all the atom economies from each step together. The complete atom economy of the whole reaction would be approximately 13%. That is a very low atom economy, but for a reaction with so many steps this is not bad.

$$\text{AE} = 68.46\% \times 100\% \times 27.07\% \times 71.10\% \times \approx 13.18\%$$

Experimental

Procedure Step 1

Dry cyclohexane, 4-methylbenzoic acid and NBS are mixed together under a nitrogen atmosphere. AIBN is added and after 1 hour the mixture is cooled down to 0°C. After that the resulting precipitate is filtered and washed with cyclohexane. The product 4-(bromomethyl)benzoic acid is a whiteish crystalline and powderous solid.

Observation Step 1

A 250 mL three-necked round bottom flask was equipped with a reflux condenser on the middle neck. A nitrogen supply was connected to one the right neck and a septum was put on the left neck. With that a nitrogen atmosphere could be created inside the three-necked round bottom flask. A magnetic stir bar was also put into the flask. 75 mL of dry cyclohexane were added into the flask. Then 4-methylbenzoic acid (8.169 g, 60.0 mmol, 1.00 equiv) and NBS (10.679 g, 60.0 mmol, 1.00 equiv) were also added into the flask. After that AIBN (49.263 g, 0.3 mmol, 0.005 equiv.) were also added to the flask with a plastic spatula. Then the mixture was slowly heated to reflux. The refluxing was continued for one hour and after that the mixture was cooled down with a ice bath to 0 °C also for one hour. The resulting precipitate was collected by filtration with a frit and a vacuum and washed it was washed with 35 mL of cyclohexane three times. The solid on top of the frit was collected.

The solid is put in a round bottom flask and 150 mL of water were added. The mixture was stirred for 10 minutes. The residue that did not dissolve was collected by filtration with a frit and a vacuum. It was washed with 50 mL of ice-water and dried in a vacuum desiccator over silica.

The product 4-(bromomethyl)benzoic acid was synthesized. It is a whiteish crystalline and powderous solid. For medical reasons, the author was unable to carry out this step of the synthesis by himself. The author had to take excess product from lab colleagues.

Procedure Step 2

Dry acetone, 4-(bromomethyl)benzoic acid and triphenylphosphine are mixed together under a nitrogen atmosphere. The mixture is heated to reflux for 1 hour and then allowed to cool. The reaction mixture is further cooled in an ice bath, and the precipitate is collected via filtration. The filtrate is concentrated and cooled again in an ice bath for 30 minutes. Another batch of the product is collected by filtration. The combined solids are washed with methyl tert-butyl ether and dried. The product (4-carboxybenzyl)triphenylphosphonium bromide is a white clumpy solid (3.756 g, 7.86 mmol, 0.39 equiv.).

Observation Step 2

A 250 mL three-necked round bottom flask was equipped with a reflux condenser on the middle neck. A nitrogen supply was connected to one the right neck. With that a nitrogen atmosphere could be created inside the three-necked round bottom flask. A magnetic stir bar was also put into the flask. The flask was filled with 150 mL dry Acetone. Then 4-(bromomethyl)benzoic acid (5.301 g, 24.65 mmol) and triphenylphosphine (5.246 g, 20.00 mmol, 1.00 equiv.) were added into the round bottom flask. They were heated to reflux for 1 hour, at around 100 °C. The mixture started bubbling really hard.

After the hour, most of the solvent evaporated and a white solid was inside the round bottom flask. This solid was transferred into a 100 mL beaker and the beaker was put into a desiccator. After a night in the desiccator the white clumpy solid was taken out and weighed. The weight of (4-carboxybenzyl)triphenylphosphonium bromide was 3.756 g (7.86 mmol, 0.39 equiv.), that would be a yield of around 39.34 %.

Procedure Step 3

4-carboxybenzyl)triphenylphosphonium bromide is suspended in water. Aqueous formaldehyde solution is added to the suspension. A dropping funnel is filled with a sodium hydroxide solution. The NaOH solution is gradually added over 30 minutes with vigorous stirring at room temperature. The mixture is stirred for an additional hour at room temperature. The mixture is filtered to collect the precipitate and it is washed with water. The liquid filtrate is then acidified to pH 1 by the drop wise addition of half-concentrated HCl. The resulting precipitate is filtered and collected. Then it is washed with ice water and dried in a desiccator over phosphorous pentoxide. The product 4-vinylbenzoic acid is a white powderous solid (1.46 g, 9.85 mmol, 0.99 equiv.).

Observation Step 3

A 100 mL three-necked round bottom flask with a magnetic stir bar was filled with (4-carboxybenzyl) triphenylphosphonium bromide (4.774 g, 10 mmol, 1.00 equiv.). Then 15 mL of water and formaldehyde (25 mL, 335.75 g, 33.58 equiv.) were added in to the flask. The liquid turned milky white. A dropping funnel was installed on the right neck of the three-necked round bottom flask. The dropping funnel was filled with a 5M sodium hydroxide solution (8.00 equiv.). This solution was slowly dripped into the mixture. The mixture was stirred for 1 hour at room temperature. The liquid was put into a frit and a vacuum and it was washed with 25 mL of water for 3 times.

Because of the sodium hydroxide, the (4-carboxybenzyl)triphenylphosphonium derivative is deprotonated and charged. Charged molecules are polar and water is also polar so it is soluble in water. The liquid filtrate contains the (4-carboxybenzyl)triphenylphosphonium derivative. The liquid filtrate was taken out of the vacuum flask and put into a beaker. The pH value was measured with a pH paper. The liquid filtrate was very basic. A 6 M hydrochloric acid solution is dripped into the liquid filtrate until the pH value drops down to a pH of 1. That caused a white fallout to appear. This white fallout is the (4-carboxybenzyl)triphenylphosphonium derivative. It is now protonated again what lowers it's polarity and makes it unsolvable in water. Now the acidified liquid was poured again into a frit under a vacuum and washed with ice water. Now the solid on top of the frit contained the desired product. It was transfered into a 100 mL beaker and was put in to a dessicator to dry over the night. The product 4-vinylbenzoic acid is a white powderous solid (1.46 g, 9.85 mmol, 0.99 equiv.). That would be a yield of around 98.54%.

Procedure Step 4

4-vinylbenzoic acid is dissolved in ethanol. Ammonium formate is added and then the catalyser palladium on carbon is introduced. The mixture is carefully heated to reflux for 1 hour. After the completion of the reaction the mixture is cooled down to room temperature. The cooled mixture is filtered through a small plug of celite and the celite is washed with ethanol. The ethanol is removed under reduced pressure and water is added to the residue. The aqueous phase is extracted with methyl tert-butyl ether. The combined organic extracts are dried over sodium sulfate, filtered. The filtrate is concentrated in vacuo to obtain the final product 4-ethylbenzoic acid. It is a white solid powder (0.28 g, 1.86 mmol, 0.37 equiv.).

Observation Step 4

A 100 mL three-necked round bottom flask was equipped with a reflux condenser and a magnetic stir bar. Then 50 mL of ethanol were transferred into the flask. The 4-vinylbenzoic acid (8g, 5.40 mmol, 1.08 equiv.) was put into the flask and it dissolved in the ethanol. Then ammonium formate (3.153 g, 50 mmol, 10.00 equiv.) was added to the mixture. After this about 74 mg of the catalyst palladium on carbon was added to the mixture. The liquid clear liquid turned black after the adding of the palladium on carbon. The mixture was heated with a oil bath to reflux, that would be about 85 °C. The mixture was stirred for 1 hour at this temperature. After that the mixture was cooled down to room temperature. The bottom of a filter frit was filled with celite and washed with ethanol. The frit was put on top of a vacuum flask. The mixture inside the flask was poured into the frit with the celite. All the palladium on carbon stuck on top of the frit, the liquid inside the vacuum flask was clear again. The clear liquid was put into a 100 mL one-necked round bottom flask and the flask was connected to a rotatory evaporator to evaporate all the ethanol.

After that the round bottom flask was filled with 40 mL of water. Then this mixture was transferred into a 250 mL separation funnel and 30 mL of MTBE was added. The product 4-ethylbenzoic acid was inside the organic phase. MTBE has a density of around 0.74 g/mL and water has a density of around 1.00 g/mL at room temperature. So the organic phase collected on top of the aqueous phase. The aqueous phase was separated and the organic phase was collected inside a beaker. This was repeated 3 times. The organic phase inside the beaker still had traces of water inside. This water could be removed by adding anhydrous magnesium sulfate until it stopped forming clumps. This mixture was filtered with a frit and a vacuum. The liquid filtrate inside the vacuum flask contained the product. This liquid was put inside a one-necked 100 mL round bottom flask and was connected to the rotatory evaporator to evaporate all the MTBE. After the evaporation there was a white solid remaining in the flask.

The white solid still had impurities and so a recrystallization was conducted. For that a little bit of ethanol was added into the round bottom flask to dissolve the white solid. A magnetic stir bar was added to stir the mixture. Then water was slowly dripped inside this mixture like a titration. After some drips a white solid started forming in the mixture, what dissolved quickly again. After dripping more and more water the solid stopped dissolving again. Then more water was added until no more solid appeared further. This mixture was then filtered with a frit and a vacuum. The solid residue on top of the frit contained the product 4-ethylbenzoic acid. The solid residue in the frit was transferred into a 50 mL one-necked round bottom flask. The flask was connected to a vacuum to extract the last traces of the solvents. After that only the final product 4-ethylbenzoic acid was inside the flask. It is a white powderous solid (0.28 g, 1.86 mmol, 0.37 equiv.). That would be a yield of around 34.44%.

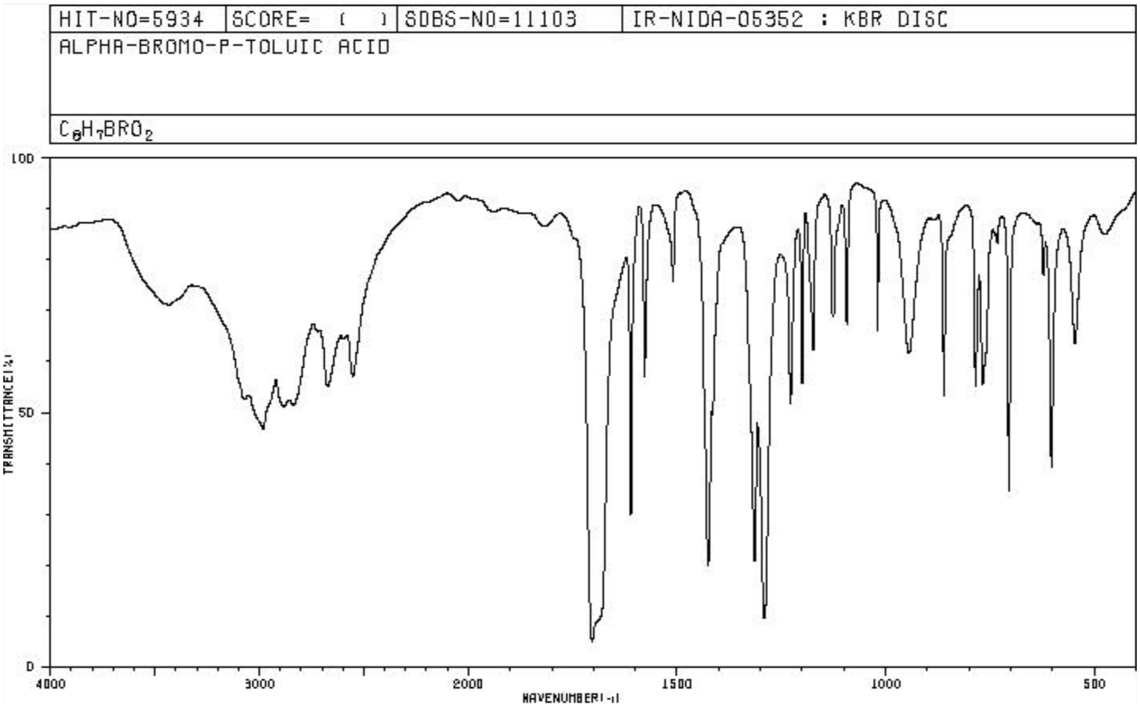
Characterisation

Melting Point Step 1

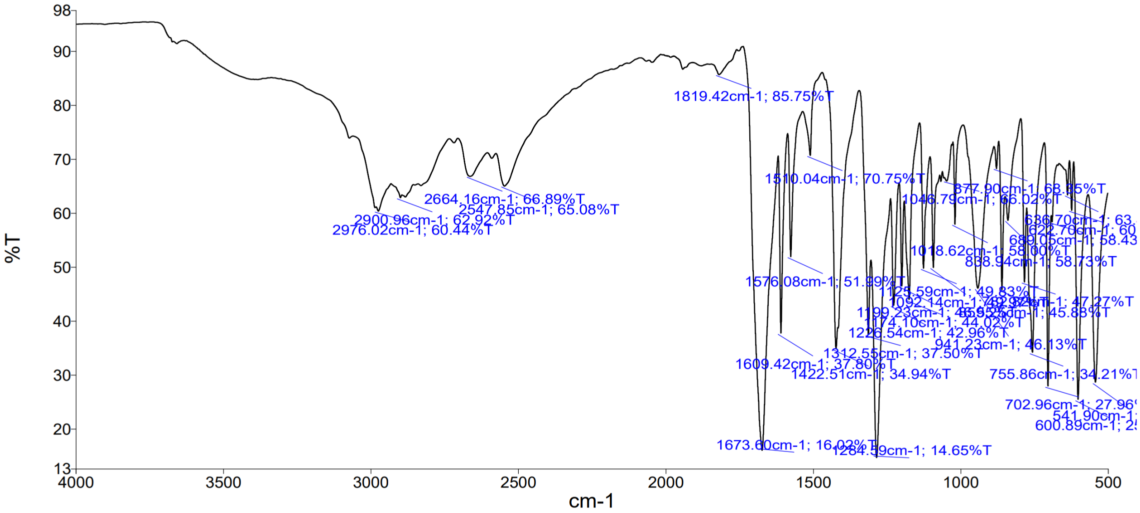
The melting point of the product was analyzed by filling a capillary tube with the white solid and using a melting point tester. For this the capillary tube was inserted in the melting point tester. At around 183 °C the substance melted into a clear liquid. That would be lower than the literature value of 226 °C. Many other colleagues from the lab had similar low values for the melting point.

IR Spectrum Step 1

This would be the literature values of the infrared spectrum of 4-(bromomethyl)benzoic acid [18].



And this would be the measured infrared spectrum of the synthesized 4-(bromomethyl)benzoic acid from the author.

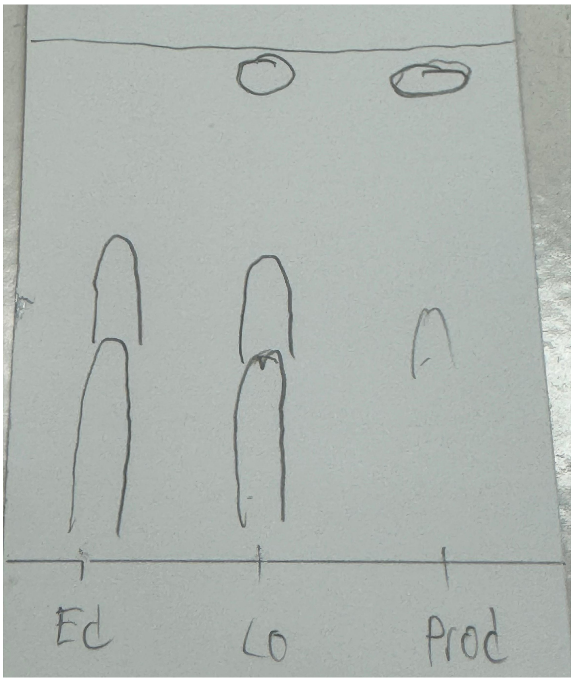


Wavenumber (cm^{-1})	Bond	%T
2976.02	C-H stretch (Aliphatic)	60.44%
2900.96	C-H stretch (Aliphatic)	62.92%
2664.16	C-H stretch (Aliphatic)	66.89%
2547.85	C-H stretch (Aliphatic)	65.08%
1819.42	C=O stretch (Acid anhydride)	85.75%
1673.60	C=O stretch (Carboxylic acid)	16.02%
1609.42	C=C stretch (Aromatic)	37.80%
1576.08	C=C stretch (Aromatic)	51.99%
1510.04	C=C stretch (Aromatic)	70.75%
1422.51	C-H bend (Methyl)	34.94%
1312.55	C-H bending (Methyl)	37.50%
1284.59	C-O stretch (Ester)	14.65%
1226.54	C-O stretch (Ether)	42.96%
1199.23	C-O stretch (Ether)	46.55%
1174.10	C-O stretch (Ether)	44.02%
1125.59	C-O stretch (Ester)	49.83%
1092.14	C-O stretch (Ester)	49.97%
1046.79	C-O stretch (Ether)	66.02%
1018.62	C-O stretch (Ester)	58.00%
941.23	C-H bend (Aromatic)	46.13%
877.90	C-H bend (Aromatic)	68.35%
859.25	C-H out-of-plane bend	45.88%
838.94	C-H out-of-plane bend	58.73%
782.82	C-H out-of-plane bend	47.27%

Wavenumber (cm^{-1})	Bond	%T
755.86	C-H out-of-plane bend	34.21%
702.96	C-H out-of-plane bend	27.96%
689.05	C-H out-of-plane bend	58.43%
636.70	C-H out-of-plane bend	63.43%
622.70	C-H out-of-plane bend	60.58%
600.89	C-H out-of-plane bend	25.49%
541.90	C-H out-of-plane bend	28.67%

TLC Step 2

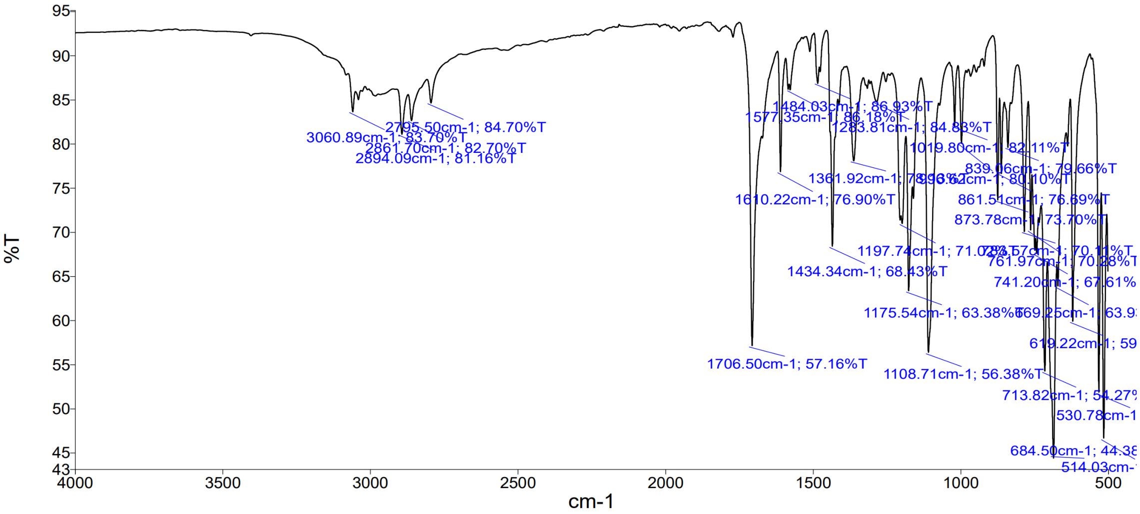
A TLC was conducted to confirm the presence of the product (4-carboxybenzyl)triphenylphosphonium bromide. A silica TLC paper wase used. A little bit of the white clumpy solid product and 4-(bromomethyl)benzoic acid was dissolved in acetone. In this TLC a silicate plate was used and there were three columns. The first column (Ed) had the the solution with the educt 4-(bromomethyl)benzoic acid on it, the third column (Prod) had the solution with the product (4-carboxybenzyl)triphenylphosphonium bromide on it, and the second column (Co) had both solutions on it. The solvent for the TLC was a 3:2 mixture of n-hexane to ethylacetate.



The product (4-carboxybenzyl)triphenylphosphonium bromide rose up higher than the educt 4-(bromomethyl)benzoic acid. The less polar a substance is, the more it rises, because polar substances are strongly attracted by the silica plate and therefore slowed down by it. The product (4-carboxybenzyl)triphenylphosphonium is much less polar than the educt 4-(bromomethyl)benzoic acid because it has three additional benzyl groups, that lower it's polarity significantly. So it makes sense that the product rose up much higher than the educt.

IR Spectrum 2

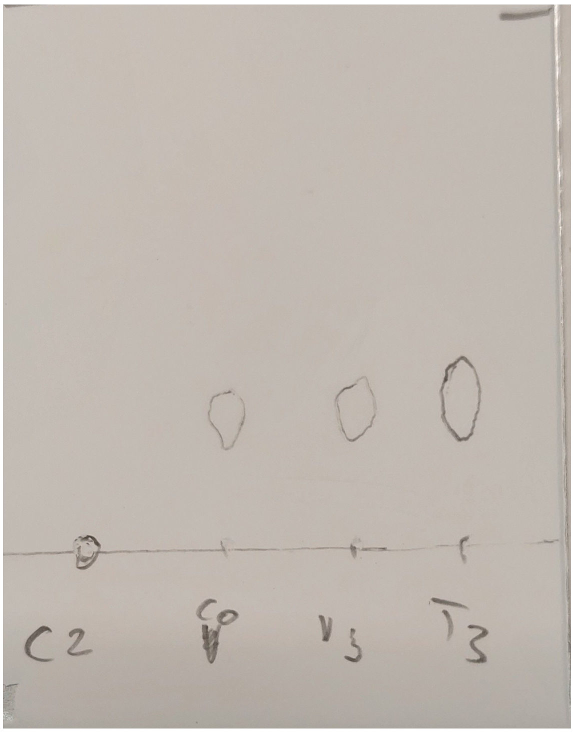
There were no literature values found of the infrared spectrum of (4-carboxybenzyl) triphenylphosphonium bromide. So this would be the measured infrared spectrum of the synthesized (4-carboxybenzyl) triphenylphosphonium bromide from the author.



Wavenumber (cm^{-1})	Bond	%T
3060.89	C-H stretch (Aromatic)	83.70%
2894.09	C-H stretch (Aliphatic)	81.16%
2861.70	C-H stretch (Aliphatic)	82.70%
2795.50	C-H stretch (Aliphatic)	84.70%
1706.50	C=O stretch (Carboxylic acid)	57.16%
1610.22	C=C stretch (Aromatic)	76.90%
1577.35	C=C stretch (Aromatic)	86.18%
1484.03	C=C stretch (Aromatic)	86.93%
1434.34	C-H bending (Methyl)	68.43%
1361.92	C-H bending (Methyl)	78.13%
1283.81	C-O stretch (Ester)	84.83%
1197.74	C-O stretch (Ether)	71.02%
1175.54	C-O stretch (Ether)	63.38%
1108.71	C-O stretch (Ether)	56.38%
1019.80	C-O stretch (Ether)	82.11%
996.62	C-H bending (Aromatic)	80.10%
873.78	C-H out-of-plane bend	73.70%
861.51	C-H out-of-plane bend	76.69%
839.06	C-H out-of-plane bend	79.66%
783.57	C-H out-of-plane bend	70.11%
761.97	C-H out-of-plane bend	70.28%
741.20	C-H out-of-plane bend	67.61%
713.82	C-H out-of-plane bend	54.27%
684.50	C-H out-of-plane bend	44.38%
669.25	C-H out-of-plane bend	63.93%
619.22	C-H out-of-plane bend	59.92%
530.78	C-H out-of-plane bend	51.86%

TLC Step 3

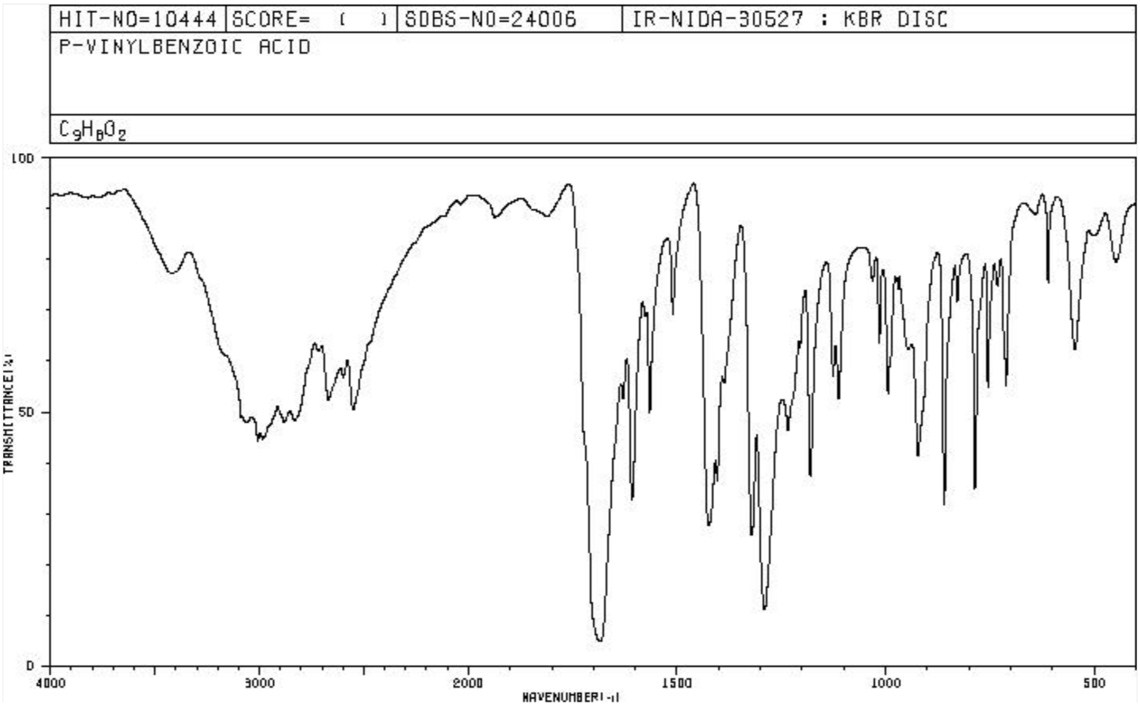
A TLC was conducted to confirm the presence of the product 4-vinylbenzoic acid. A little bit of the white powderous solid product and (4-carboxybenzyl) triphenylphosphonium bromide was dissolved in acetone. In this TLC a silicate plate was used and there were three columns. The first column (C2) had the the solution with the educt (4-carboxybenzyl) triphenylphosphonium bromide acid on it, the third column (V3) had the solution with the product 4-vinylbenzoic acid on it, and the second column (Co) had both solutions on it. The solvent for the TLC was a 3:2 mixture of n-hexane to ethylacetate.



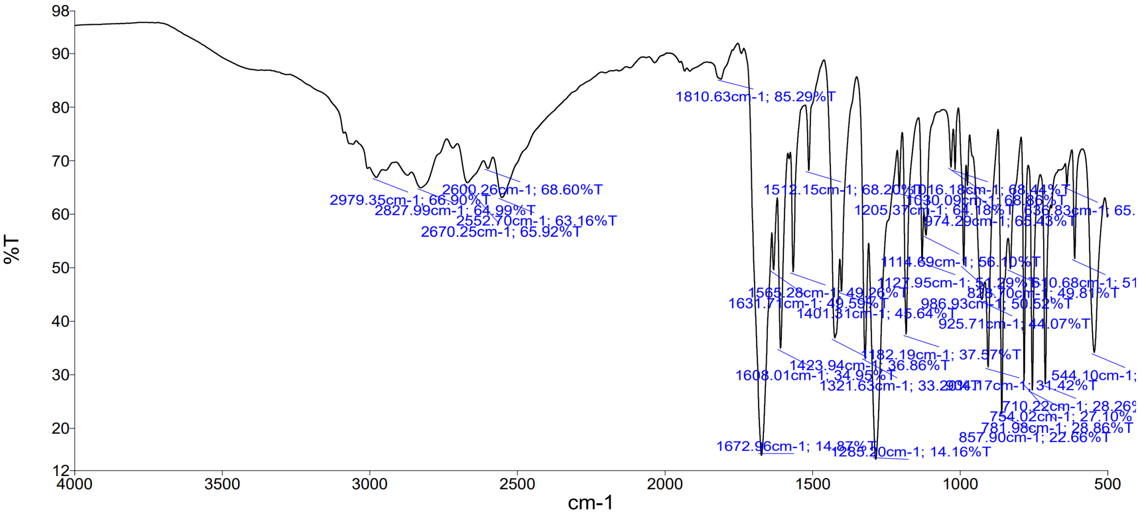
The less polar a substance is the higher it rises on the TLC plate. 4-vinylbenzoic acid is much less polar than (4-carboxybenzyl) triphenylphosphonium bromide. So it makes sense that the 4-vinylbenzoic acid rose up much higher than the (4-carboxybenzyl) triphenylphosphonium bromide.

IR Spectrum 3

This would be the literature values of the infrared spectrum of 4-vinylbenzoic acid [19].



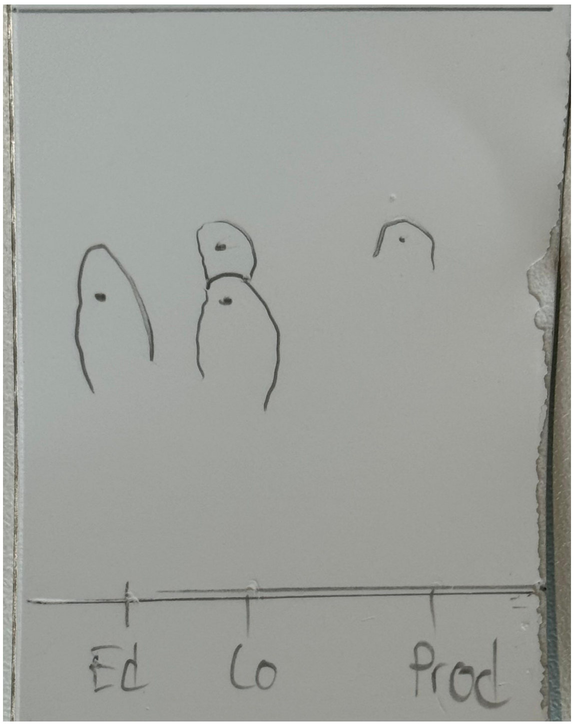
And this would be the measured infrared spectrum of the synthesized 4-vinylbenzoic acid from the author.



Wavenumber (cm^{-1})	Bond	%T
2979.35	C-H stretch (Aliphatic)	66.90%
2827.99	C-H stretch (Aliphatic)	64.99%
2670.25	C-H stretch (Aliphatic)	65.92%
2600.26	C-H stretch (Aliphatic)	68.60%
2552.70	C-H stretch (Aliphatic)	63.16%
1810.63	C=O stretch (Acid anhydride)	85.29%
1672.96	C=O stretch (Carboxylic acid)	14.87%
1631.71	C=C stretch (Aromatic)	49.59%
1608.01	C=C stretch (Aromatic)	34.95%
1565.28	C=C stretch (Aromatic)	49.26%
1512.15	C=C stretch (Aromatic)	68.20%
1423.94	C-H bending (Methyl)	36.86%
1401.31	C-H bending (Methyl)	45.64%
1321.63	C-O stretch (Ester)	33.20%
1285.20	C-O stretch (Ester)	14.16%
1205.37	C-O stretch (Ester)	64.18%
1182.19	C-O stretch (Ether)	37.57%
1127.95	C-O stretch (Ether)	51.29%
1114.69	C-O stretch (Ether)	56.10%
1030.09	C-O stretch (Ester)	68.86%
1016.18	C-O stretch (Ether)	68.44%
986.93	C-H bending (Aromatic)	50.52%
974.29	C-H bending (Aromatic)	65.43%
925.71	C-H out-of-plane bend	44.07%
904.17	C-H out-of-plane bend	31.42%
857.90	C-H out-of-plane bend	22.66%
828.70	C-H out-of-plane bend	49.81%
781.98	C-H out-of-plane bend	28.86%
754.02	C-H out-of-plane bend	27.10%
710.22	C-H out-of-plane bend	28.26%
636.83	C-H out-of-plane bend	65.20%
610.68	C-H out-of-plane bend	51.73%
544.10	C-H out-of-plane bend	34.16%

TLC Step 4

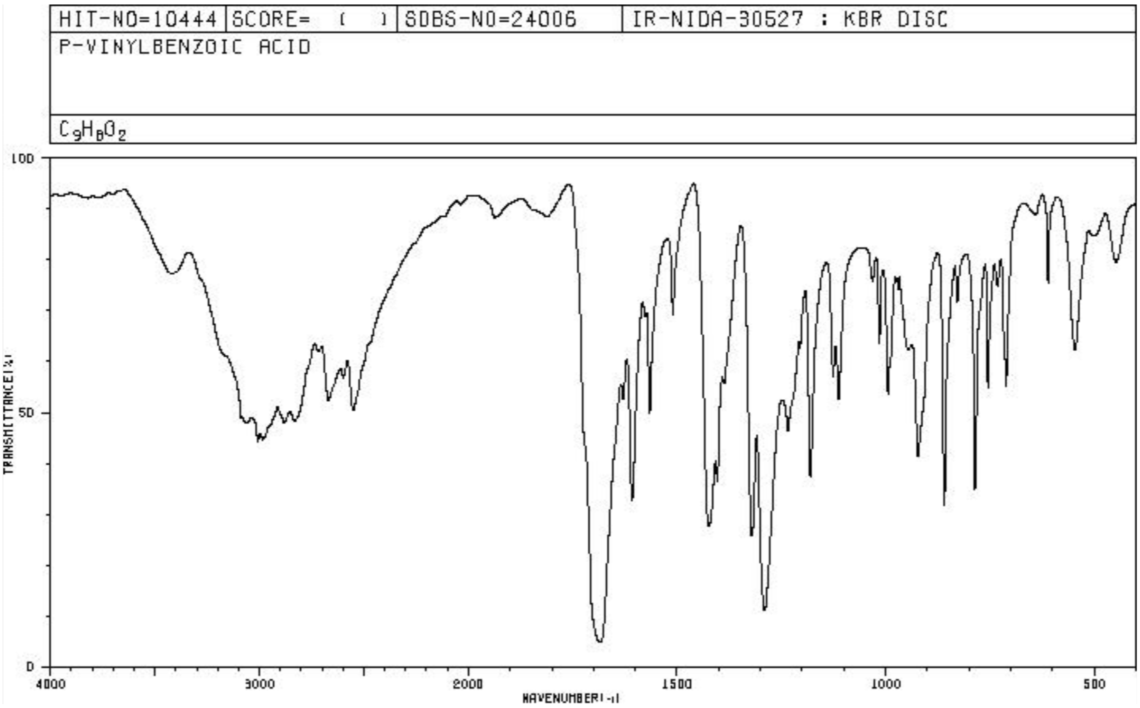
A TLC was conducted to confirm the presence of the product 4-ethylbenzoic acid. A little bit of the white powderous solid product and 4-vinylbenzoic acid was dissolved in acetone. In this TLC a silicate plate was used and there were three columns. The first column (Ed) had the the solution with the educt 4-vinylbenzoic acid on it, the third column (Prod) had the solution with the product 4-ethylbenzoic acid on it, and the second column (Co) had both solutions on it. The solvent for the TLC was a 3:2 mixture of n-hexane to ethylacetate.



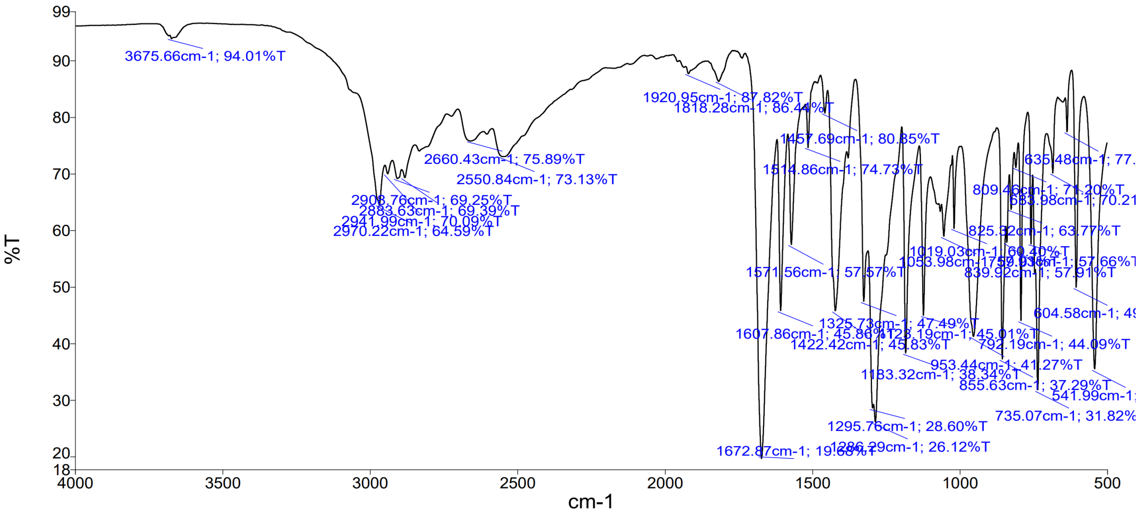
The less polar a substance is the higher it rises on the TLC plate. 4-vinylbenzoic acid and 4-ethylbenzoic acid are really similar, the only difference is that the first has a double bond, a vinyl group and the second has a single bond, a ethyl group. Double bonds affect polarity because the electron density is more concentrated around them compared to single bonds. This concentration results in a more pronounced electron distribution that can create a dipole moment, increasing the overall polarity of the molecule. Furthermore, double bonds in conjugation with aromatic rings, as in 4-vinylbenzoic acid, tend to delocalize electrons, which also can enhance polarity. These little higher polarity results in the little lower rise of the educt 4-vinylbenzoic acid.

IR Spectrum 4

This would be the literature values of the infrared spectrum of 4-ethylbenzoic acid [20].



And this would be the measured infrared spectrum of the synthesized 4-ethylbenzoic acid from the author.



Wavenumber (cm^{-1})	Bond	%T
3675.66	O-H stretch (Alcohol)	94.01%
2970.22	C-H stretch (Aliphatic)	64.59%
2941.99	C-H stretch (Aliphatic)	70.09%
2908.76	C-H stretch (Aliphatic)	69.25%
2883.63	C-H stretch (Aliphatic)	69.39%
2660.43	C-H stretch (Aliphatic)	75.89%
2550.84	C-H stretch (Aliphatic)	73.13%
1920.95	C=O stretch (Acid anhydride)	87.82%
1818.28	C=O stretch (Acid anhydride)	86.44%
1672.87	C=O stretch (Carboxylic acid)	19.68%
1607.86	C=C stretch (Aromatic)	45.86%
1571.56	C=C stretch (Aromatic)	57.57%
1514.86	C=C stretch (Aromatic)	74.73%
1457.69	C-H bending (Methyl)	80.85%
1422.42	C-H bending (Methyl)	45.83%
1325.73	C-O stretch (Ester)	47.49%
1295.76	C-O stretch (Ester)	28.60%
1286.29	C-O stretch (Ester)	26.12%
1183.32	C-O stretch (Ether)	38.34%
1123.19	C-O stretch (Ether)	45.01%
1053.98	C-O stretch (Ether)	59.01%
1019.03	C-O stretch (Ether)	60.40%
953.44	C-H bending (Aromatic)	41.27%
839.92	C-H out-of-plane bend	57.91%
825.32	C-H out-of-plane bend	63.77%
809.46	C-H out-of-plane bend	71.20%
792.19	C-H out-of-plane bend	44.09%
757.93	C-H out-of-plane bend	57.66%
735.07	C-H out-of-plane bend	31.82%
683.98	C-H out-of-plane bend	70.21%
635.48	C-H out-of-plane bend	77.50%
604.58	C-H out-of-plane bend	49.98%
541.99	C-H out-of-plane bend	35.52%

References

[1] Chemical Book. 4-ethylbenzoic acid. https://www.chemicalbook.com/ChemicalProductProperty_EN_CB3153435.htm.

[2] Thermo Fisher Scientific. 4-ethylbenzoic acid. https://www.thermofisher.com/order/catalog/product/184490250.

[3] PubChem. 4-methylbenzoic acid. https://pubchem.ncbi.nlm.nih.gov/compound/4-Methylbenzoic-acid.

[4] PubChem. N-bromosuccinimide. https://pubchem.ncbi.nlm.nih.gov/compound/N-Bromosuccinimide.

[5] PubChem. Azobisisobutyronitrile (aibn). https://pubchem.ncbi.nlm.nih.gov/compound/Azobisisobutyronitrile.

[6] PubChem. 4-(bromomethyl)benzoic acid. https://pubchem.ncbi.nlm.nih.gov/compound/4-_Bromomethyl_benzoic-acid.

[7] PubChem. Triphenylphosphine. https://pubchem.ncbi.nlm.nih.gov/compound/Triphenylphosphine.

[8] PubChem. Tert-butyl methyl ether (mtbe). https://pubchem.ncbi.nlm.nih.gov/compound/Tert-butyl_methyl_ether.

[9] PubChem. (4-carboxybenzyl)triphenylphosphonium bromide. https://pubchem.ncbi.nlm.nih.gov/compound/4-Carboxybenzyl_triphenylphosphonium-bromide.

[10] PubChem. Formaldehyde. https://pubchem.ncbi.nlm.nih.gov/compound/Formaldehyde.

[11] PubChem. Sodium hydroxide. https://pubchem.ncbi.nlm.nih.gov/compound/Sodium_hydroxide.

[12] PubChem. Hydrochloric acid. https://pubchem.ncbi.nlm.nih.gov/compound/Hydrochloric_acid.

[13] PubChem. 4-vinylbenzoic acid. https://pubchem.ncbi.nlm.nih.gov/compound/4-Vinylbenzoic-acid.

[14] PubChem. Palladium on carbon. https://pubchem.ncbi.nlm.nih.gov/compound/Palladium_on_carbon.

[15] PubChem. Ammonium formate. https://pubchem.ncbi.nlm.nih.gov/compound/Ammonium_formate.

[16] PubChem. Sodium carbonate. https://pubchem.ncbi.nlm.nih.gov/compound/Sodium_carbonate.

[17] PubChem. 4-ethylbenzoic acid. https://pubchem.ncbi.nlm.nih.gov/compound/4-Ethylbenzoic-acid.

[18] Chemical Book. 4-bromomethylbenzoic acid(6232-88-8) ir1. https://www.chemicalbook.com/SpectrumEN_6232-88-8_IR1.htm.

[19] Chemical Book. 4-vinylbenzoic acid(1075-49-6) ir1. https://www.chemicalbook.com/SpectrumEN_1075-49-6_IR1.htm.

[20] Chemical Book. 4-ethylbenzoic acid(619-64-7) ir1. https://www.chemicalbook.com/SpectrumEN_619-64-7_IR1.htm.

Appendix

List of H and P Phrases

- **H225**: Highly flammable liquid and vapor.
- **H226**: Flammable liquid and vapor.
- **H242**: Heating may cause a fire.
- **H272**: May intensify fire; oxidizer.
- **H290**: May be corrosive to metals.
- **H302**: Harmful if swallowed.
- **H311**: Toxic in contact with skin.
- **H314**: Causes severe skin burns and eye damage.
- **H315**: Causes skin irritation.
- **H317**: May cause an allergic skin reaction.
- **H318**: Causes serious eye damage.
- **H319**: Causes serious eye irritation.
- **H330**: Fatal if inhaled.
- **H332**: Harmful if inhaled.
- **H335**: May cause respiratory irritation.
- **H341**: Suspected of causing genetic defects.
- **H350**: May cause cancer.
- **H370**: Causes damage to organs.
- **H372**: Causes damage to organs through prolonged or repeated exposure.
- **H400**: Very toxic to aquatic life.
- **H412**: Harmful to aquatic life with long-lasting effects.
- **P201**: Obtain special instructions before use.
- **P202**: Do not handle until all safety precautions have been read and understood.
- **P210**: Keep away from heat, sparks, open flames, hot surfaces. No smoking.
- **P220**: Store away from other materials.
- **P233**: Keep container tightly closed.
- **P234**: Keep only in original container.
- **P240**: Ground/bond container and receiving equipment.
- **P241**: Use explosion-proof electrical/ventilating/lighting/equipment.
- **P242**: Use non-sparking tools.
- **P260**: Do not breathe dust/fume/gas/mist/vapors/spray.
- **P261**: Avoid breathing dust/fume/gas/mist/vapors/spray.
- **P264**: Wash thoroughly after handling.

- **P270**: Do not eat, drink or smoke when using this product.
- **P271**: Use only outdoors or in a well-ventilated area.
- **P273**: Avoid release to the environment.
- **P280**: Wear protective gloves/protective clothing/eye protection/face protection.
- **P301+P312**: IF SWALLOWED: Call a POISON CENTER/doctor if you feel unwell.
- **P301+P330+P331**: IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.
- **P302+P352**: IF ON SKIN: Wash with plenty of water.
- **P303+P361+P353**: IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/shower.
- **P304+P340**: IF INHALED: Remove to fresh air and keep at rest in a position comfortable for breathing.
- **P305+P351+P338**: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
- **P308+P313**: IF exposed or concerned: Get medical advice/attention.
- **P312**: Call a POISON CENTER/doctor if you feel unwell.
- **P321**: Specific treatment (see instructions on this label).
- **P330**: Rinse mouth.
- **P332+P317**: If skin irritation occurs: Get medical help.
- **P337+P313**: If eye irritation persists: Get medical advice/attention.
- **P362**: Take off contaminated clothing and wash before reuse.
- **P363**: Wash contaminated clothing before reuse.
- **P370+P378**: In case of fire: Use appropriate extinguishing media to extinguish.
- **P390**: Absorb spillage to prevent material damage.
- **P391**: Collect spillage.
- **P403+P233**: Store in a well-ventilated place. Keep container tightly closed.
- **P403**: Store in a well-ventilated place.
- **P405**: Store locked up.
- **P406**: Store in a corrosive resistant container with a resistant inner liner.
- **P501**: Dispose of contents/container to an approved waste disposal plant.

Tables from the Experiment Manual

Reactant	MW g/mol	Equiv	Moles mmol	Mass g	Volume	Purity
4-Methylbenzoic acid	136.15	1.00	60 mmol	8.169	-	-
N-Bromosuccinimide	177.98	1.00	60 mmol	10.679	-	-
AIBN	164.21	0.005	0.3 mmol	49.263	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	
4-(Bromomethyl)benzoic acid	215.04				226 °C	

Reactant	MW g/mol	Equiv	Moles mmol	Mass g	Volume	Purity
4-(Bromomethyl)benzoic acid	215.04	1.00	20 mmol	4.301	-	-
Triphenylphospine	262.29	1.00	20 mmol	5.246	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	
CBTPB	477.34				?	

Reactant	MW g/mol	Equiv	Moles mmol	Mass g	Volume	Purity
(4-carboxybenzyl) triphenylphosphonium bromide	477.34	1.00	10 mmol	4.774	-	-
Formaldehyde → 1,09 g/mL	30.03	67.15	671.50	(20.165)	50 mL	37% → ·0,37
NaOH	40.00	8.00	80	3.2	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	
4-Vinylbenzoic acid	148.16				143°C	

Reactant	MW g/mol	Equiv	Moles mmol	Mass	Volume	Purity
4-Vinylbenzoic acid	148.16	1.00	5.0 mmol	0.741 g	-	-
Pd/C 10%	106.42	-	-	74 mg	-	10%
Ammonium formate	63.06	10.0	50	3.153 g	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	
4-Ethylbenzoic acid	150.17				112 °C	

Infrared Spectroscopy Absorption Table

The following table lists **infrared spectroscopy absorptions** by frequency regions.

4000-3000 cm^{-1}

3700-3584	medium	sharp	O-H	stretching	alcohol	free
3550-3200	strong	broad	O-H	stretching	alcohol	intermolecular bonded
3500- 3400	medium	-	N-H	stretching	primary amine	-
3400-3300 3330-3250	medium	-	N-H	stretching	aliphatic primary amine	-
3350-3310	medium	-	N-H	stretching	secondary amine	-
3300-2500	strong	broad	O-H	stretching	carboxylic acid	usually centered on 3000 cm^{-1}
3200-2700	weak	broad	O-H	stretching	alcohol	intramolecular bonded
3000-2800	strong	broad	N-H	stretching	amine salt	-

3000-2500 cm^{-1}

3333-3267	strong	sharp	C-H	stretching	alkyne	-
3100-3000	medium	-	C-H	stretching	alkene	-
3000-2840	medium	-	C-H	stretching	alkane	-
2830-2695	medium	-	C-H	stretching	aldehyde	doublet
2600-2550	weak	-	S-H	stretching	thiol	-

2400-2000 cm^{-1}

2349	strong	-	O=C=O	stretching	carbon dioxide	-
2275-2250	strong	broad	N=C=O	stretching	isocyanate	-
2260-2222	weak	-	CΞN	stretching	nitrile	-
2260-2190	weak	-	CΞC	stretching	alkyne	disubstituted
2175-2140	strong	-	S-CΞN	stretching	thiocyanate	-
2160-2120	strong	-	N=N=N	stretching	azide	-
2150	-	-	C=C=O	stretching	ketene	-
2145-2120	strong	-	N=C=N	stretching	carbodiimide	-
2140-2100	weak	-	CΞC	stretching	alkyne	monosubstituted
2140-1990	strong	-	N=C=S	stretching	isothiocyanate	-
2000-1900	medium	-	C=C=C	stretching	allene	-
2000	-	-	C=C=N	stretching	ketenimine	-

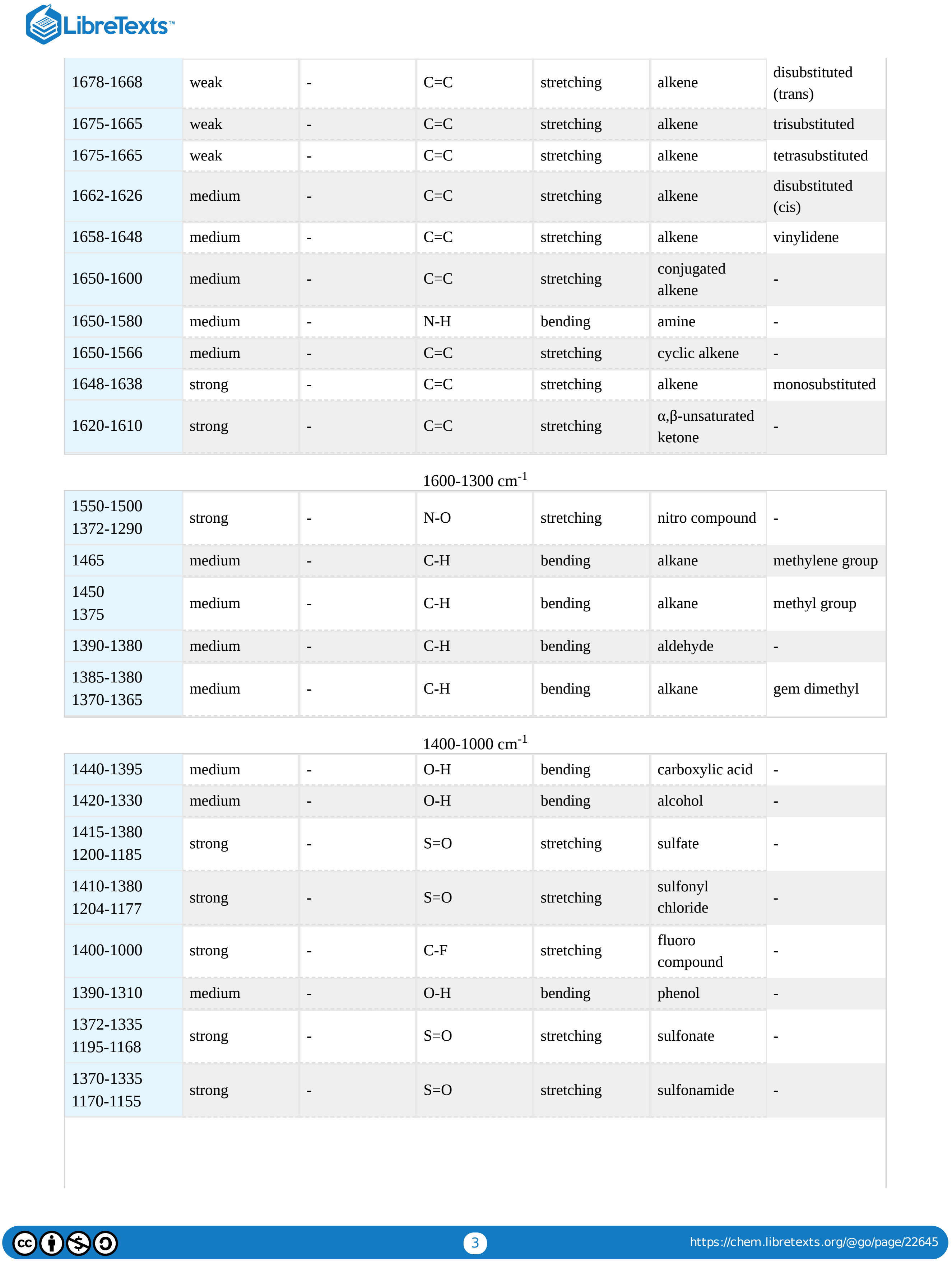
2000-1650 cm^{-1}

2000-1650	weak	-	C-H	bending	aromatic compound	overtone

1870-1540 cm^{-1}

1818 1750	strong	-	C=O	stretching	anhydride	-
1815-1785	strong	-	C=O	stretching	acid halide	-
1800-1770	strong	-	C=O	stretching	conjugated acid halide	-
1775 1720	strong	-	C=O	stretching	conjugated anhydride	-
1770-1780	strong	-	C=O	stretching	vinyl / phenyl ester	-
1760	strong	-	C=O	stretching	carboxylic acid	monomer
1750-1735	strong	-	C=O	stretching	esters	6-membered lactone
1750-1735	strong	-	C=O	stretching	δ-lactone	γ: 1770
1745	strong	-	C=O	stretching	cyclopentanone	-
1740-1720	strong	-	C=O	stretching	aldehyde	-
1730-1715	strong	-	C=O	stretching	α,β-unsaturated ester	or formates
1725-1705	strong	-	C=O	stretching	aliphatic ketone	or cyclohexanone or cyclopentenone
1720-1706	strong	-	C=O	stretching	carboxylic acid	dimer
1710-1680	strong	-	C=O	stretching	conjugated acid	dimer
1710-1685	strong	-	C=O	stretching	conjugated aldehyde	-
1690	strong	-	C=O	stretching	primary amide	free (associated: 1650)
1690-1640	medium	-	C=N	stretching	imine / oxime	-
1685-1666	strong	-	C=O	stretching	conjugated ketone	-
1680	strong	-	C=O	stretching	secondary amide	free (associated: 1640)
1680	strong	-	C=O	stretching	tertiary amide	free (associated: 1630)
1650	strong	-	C=O	stretching	δ-lactam	γ: 1750-1700 β: 1760-1730

1670-1600 cm^{-1}



1678-1668	weak	-	C=C	stretching	alkene	disubstituted (trans)
1675-1665	weak	-	C=C	stretching	alkene	trisubstituted
1675-1665	weak	-	C=C	stretching	alkene	tetrasubstituted
1662-1626	medium	-	C=C	stretching	alkene	disubstituted (cis)
1658-1648	medium	-	C=C	stretching	alkene	vinylidene
1650-1600	medium	-	C=C	stretching	conjugated alkene	-
1650-1580	medium	-	N-H	bending	amine	-
1650-1566	medium	-	C=C	stretching	cyclic alkene	-
1648-1638	strong	-	C=C	stretching	alkene	monosubstituted
1620-1610	strong	-	C=C	stretching	α,β-unsaturated ketone	-

1600-1300 cm^{-1}

1550-1500 1372-1290	strong	-	N-O	stretching	nitro compound	-
1465	medium	-	C-H	bending	alkane	methylene group
1450 1375	medium	-	C-H	bending	alkane	methyl group
1390-1380	medium	-	C-H	bending	aldehyde	-
1385-1380 1370-1365	medium	-	C-H	bending	alkane	gem dimethyl

1400-1000 cm^{-1}

1440-1395	medium	-	O-H	bending	carboxylic acid	-
1420-1330	medium	-	O-H	bending	alcohol	-
1415-1380 1200-1185	strong	-	S=O	stretching	sulfate	-
1410-1380 1204-1177	strong	-	S=O	stretching	sulfonyl chloride	-
1400-1000	strong	-	C-F	stretching	fluoro compound	-
1390-1310	medium	-	O-H	bending	phenol	-
1372-1335 1195-1168	strong	-	S=O	stretching	sulfonate	-
1370-1335 1170-1155	strong	-	S=O	stretching	sulfonamide	-

1350-1342 1165-1150	strong	-	S=O	stretching	sulfonic acid	anhydrous hydrate: 1230-1120
1350-1300 1160-1120	strong	-	S=O	stretching	sulfone	-
1342-1266	strong	-	C-N	stretching	aromatic amine	-
1310-1250	strong	-	C-O	stretching	aromatic ester	-
1275-1200 1075-1020	strong	-	C-O	stretching	alkyl aryl ether	-
1250-1020	medium	-	C-N	stretching	amine	-
1225-1200 1075-1020	strong	-	C-O	stretching	vinyl ether	-
1210-1163	strong	-	C-O	stretching	ester	-
1205-1124	strong	-	C-O	stretching	tertiary alcohol	-
1150-1085	strong	-	C-O	stretching	aliphatic ether	-
1124-1087	strong	-	C-O	stretching	secondary alcohol	-
1085-1050	strong	-	C-O	stretching	primary alcohol	-
1070-1030	strong	-	S=O	stretching	sulfoxide	-
1050-1040	strong	broad	CO-O-CO	stretching	anhydride	-

1000-650 cm^{-1}

995-985 915-905	strong	-	C=C	bending	alkene	monosubstituted
980-960	strong	-	C=C	bending	alkene	disubstituted (trans)
895-885	strong	-	C=C	bending	alkene	vinylidene
850-550	strong	-	C-Cl	stretching	halo compound	-
840-790	medium	-	C=C	bending	alkene	trisubstituted
730-665	strong	-	C=C	bending	alkene	disubstituted (cis)
690-515	strong	-	C-Br	stretching	halo compound	-
600-500	strong	-	C-I	stretching	halo compound	-

900-700 cm^{-1}

880 ± 20 810 ± 20	strong	-	C-H	bending	1,2,4-trisubstituted	-
880 ± 20 780 ± 20 (700 ± 20)	strong	-	C-H	bending	1,3-disubstituted	-

810 ± 20	strong	-	C-H	bending	1,4-disubstituted or 1,2,3,4-tetrasubstituted	-
780 ± 20 (700 ± 20)	strong	-	C-H	bending	1,2,3-trisubstituted	-
755 ± 20	strong	-	C-H	bending	1,2-disubstituted	-
750 ± 20 700 ± 20	strong	-	C-H	bending	monosubstituted benzene derivative	-

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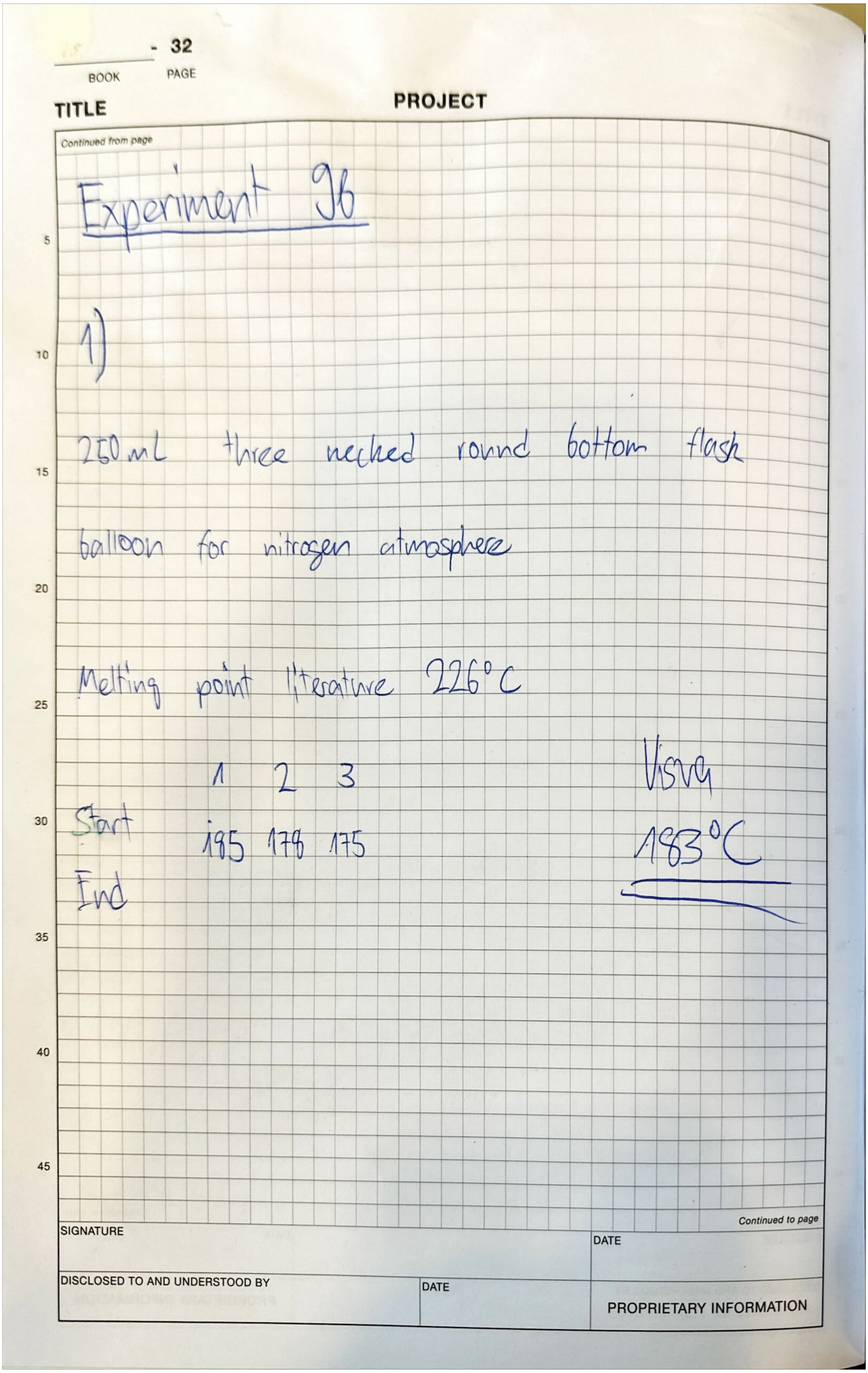

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Experiment 9b

1)

250 mL three necked round bottom flash

balloon for nitrogen atmosphere

Melting point literature 226°C

	1	2	3
Start	185	178	175
End			

Visua

183°C

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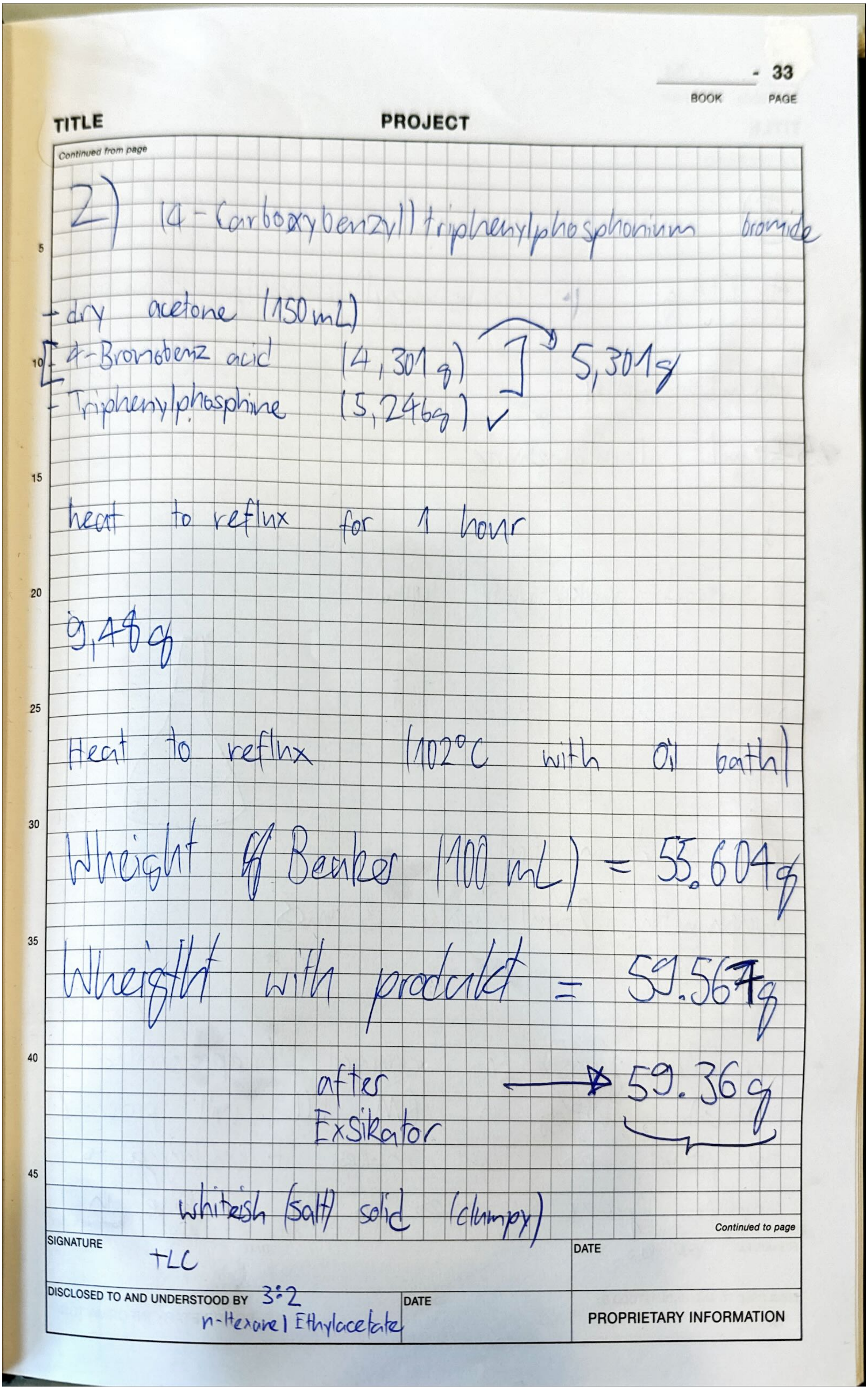

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2) (4-Carboxybenzyl)triphenylphosphonium bromide

- dry acetone (150 mL)
- 4-Bromobenz acid (4,301 g)] → 5,301g
- Triphenylphosphine (5,246g) ✓

heat to reflux for 1 hour

9,48g

Heat to reflux (102°C with oil bath)

Wheight of Beaker (100 mL) = 55.604g

Wheigtht with prodakt = 59.567g

after Exsikator → 59.36 g

whiteish (salt) solid (clumpy)

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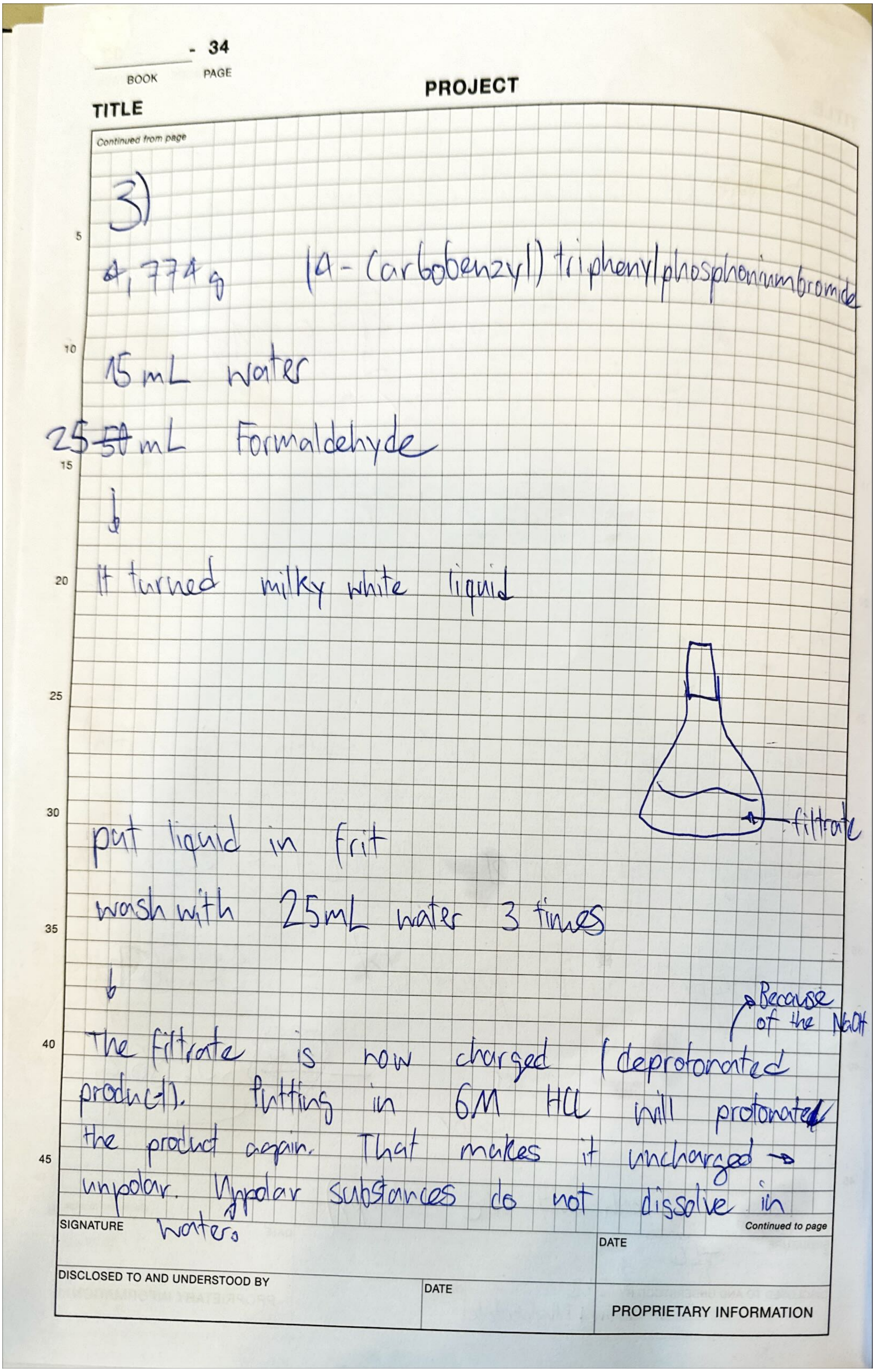

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3)

4,774 g (4-Carbobenzyl)triphenylphosphoniumbromide

15 mL water

25 ~~50~~ mL Formaldehyde

↓

It turned milky white liquid

filtrate

put liquid in frit

wash with 25mL water 3 times

↓

The filtrate is now charged (deprotonated product). Putting in 6M HCL will protonate the product again. That makes it uncharged → unpolar. Unpolar substances do not dissolve in water.

→ Because of the NaOH

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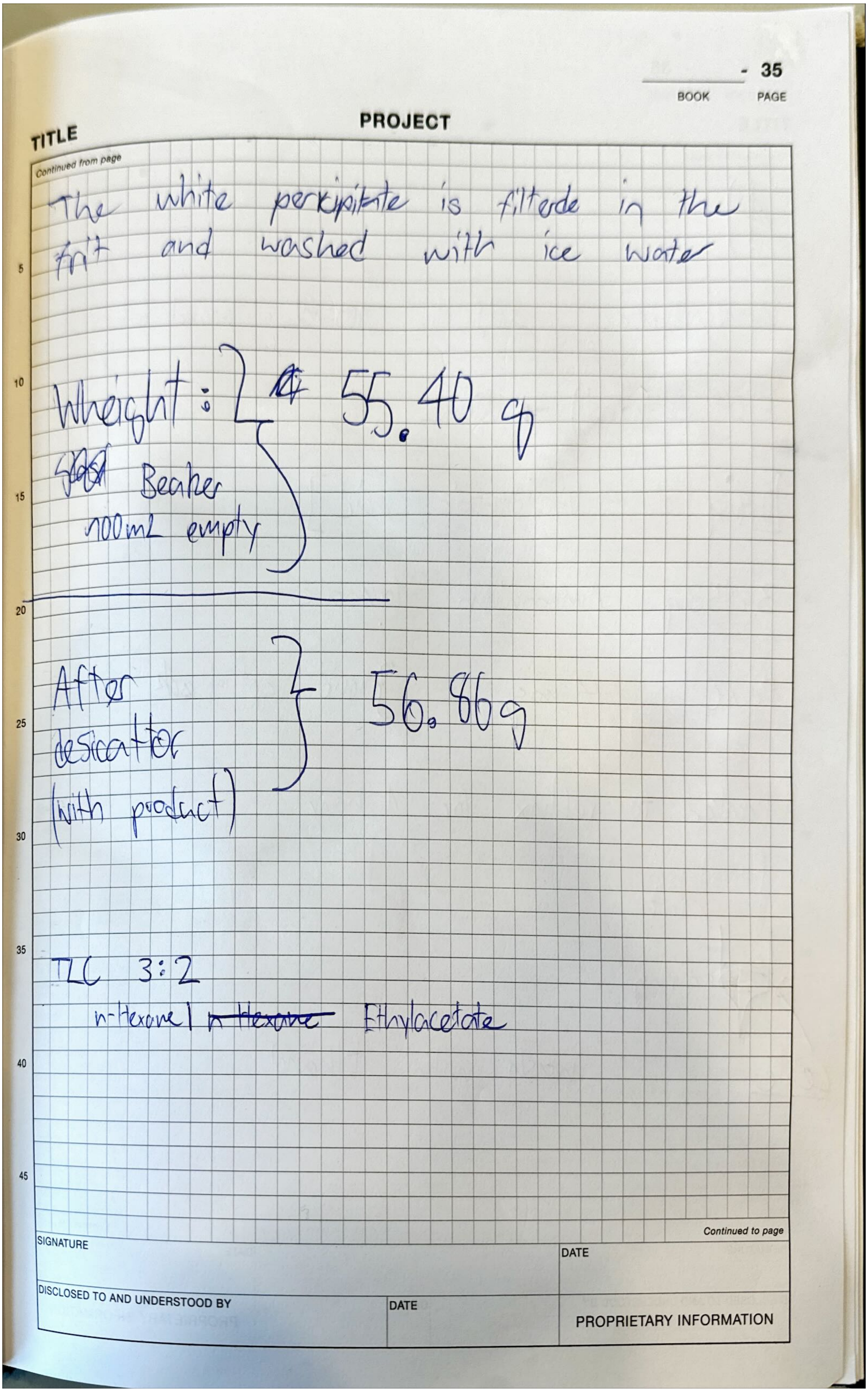

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The white perkipitate is filterde in the frit and washed with ice water

Wheight: } 55.40 g
Beaker
100mL empty

After
desicattor } 56.86g
(with product)

TLC 3:2
n-Hexane | ~~n-Hexane~~ Ethylacetate

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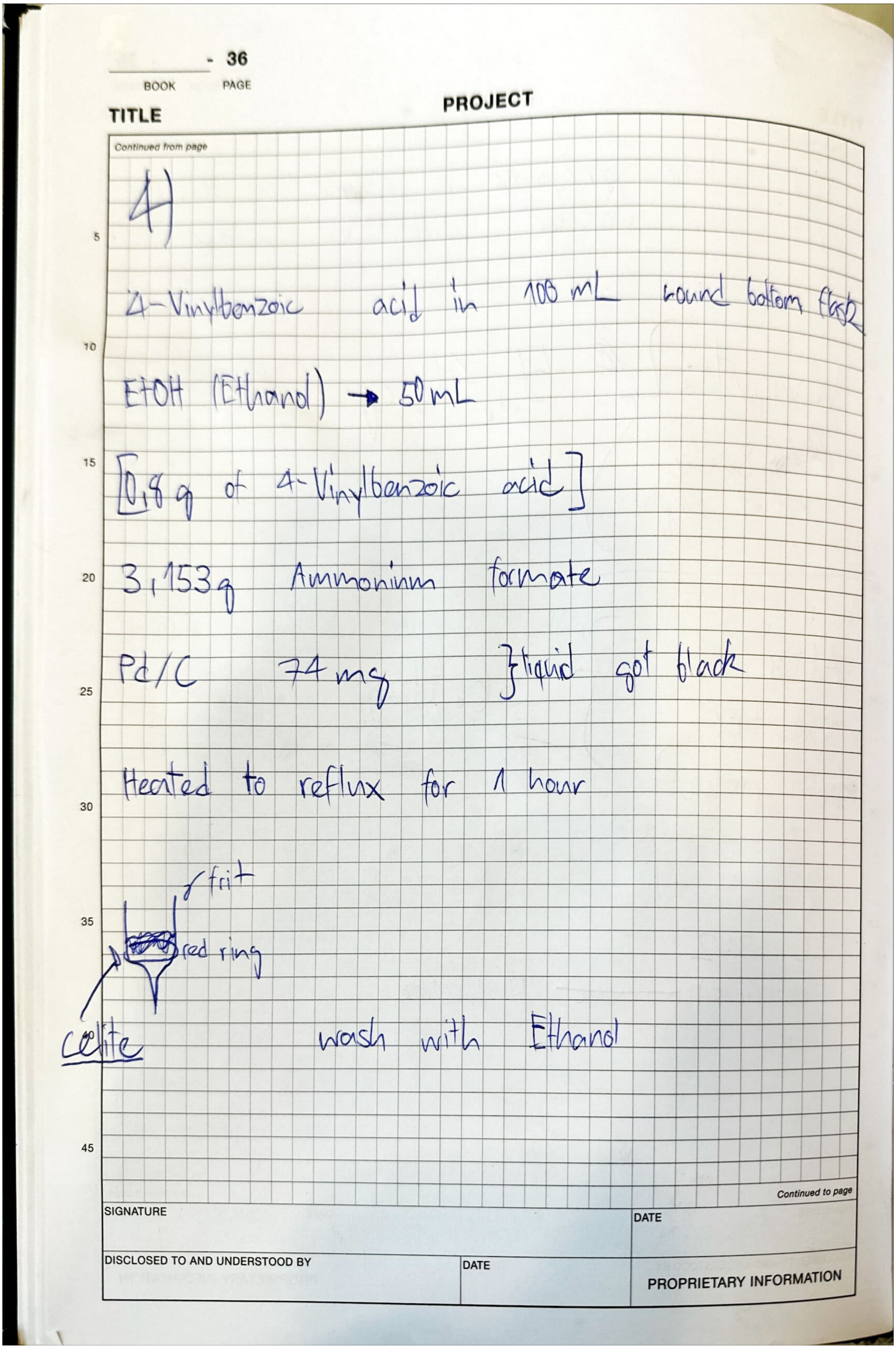

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4)

4-Vinylbenzoic acid in 100 mL round bottom flask

EtOH (Ethanol) → 50 mL

[0,8 g of 4-Vinylbenzoic acid]

3,153 g Ammonium formate

Pd/C 74 mg } liquid got black

Heated to reflux for 1 hour

wash with Ethanol

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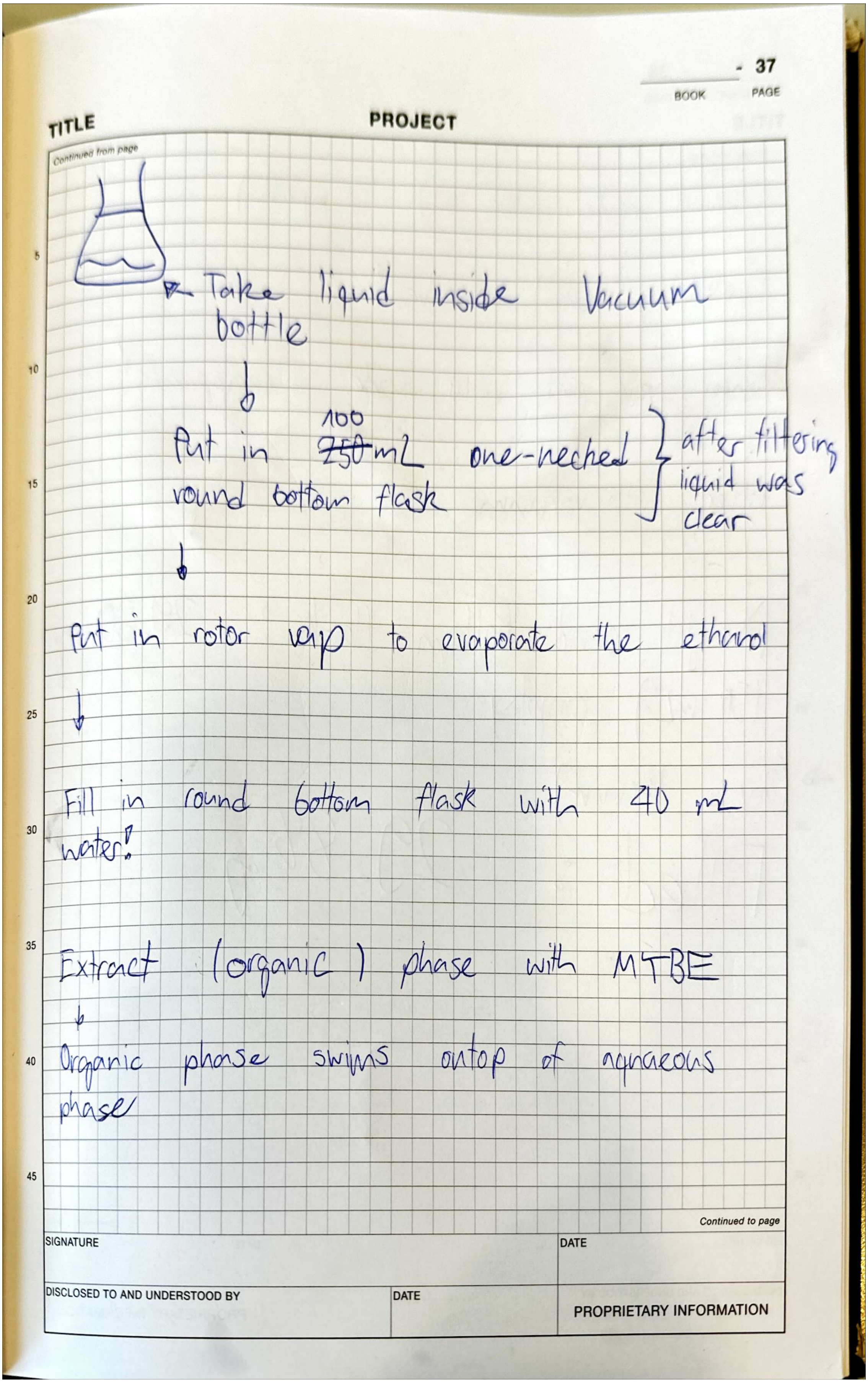

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Take liquid inside Vacuum bottle

↓

Put in ~~250~~ 100 mL one-necked round bottom flask } after filtering liquid was clear

↓

Put in rotor vap to evaporate the ethanol

↓

Fill in round bottom flask with 40 mL water!

Extract (organic) phase with MTBE

↓

Organic phase swims ontop of aquaeous phase

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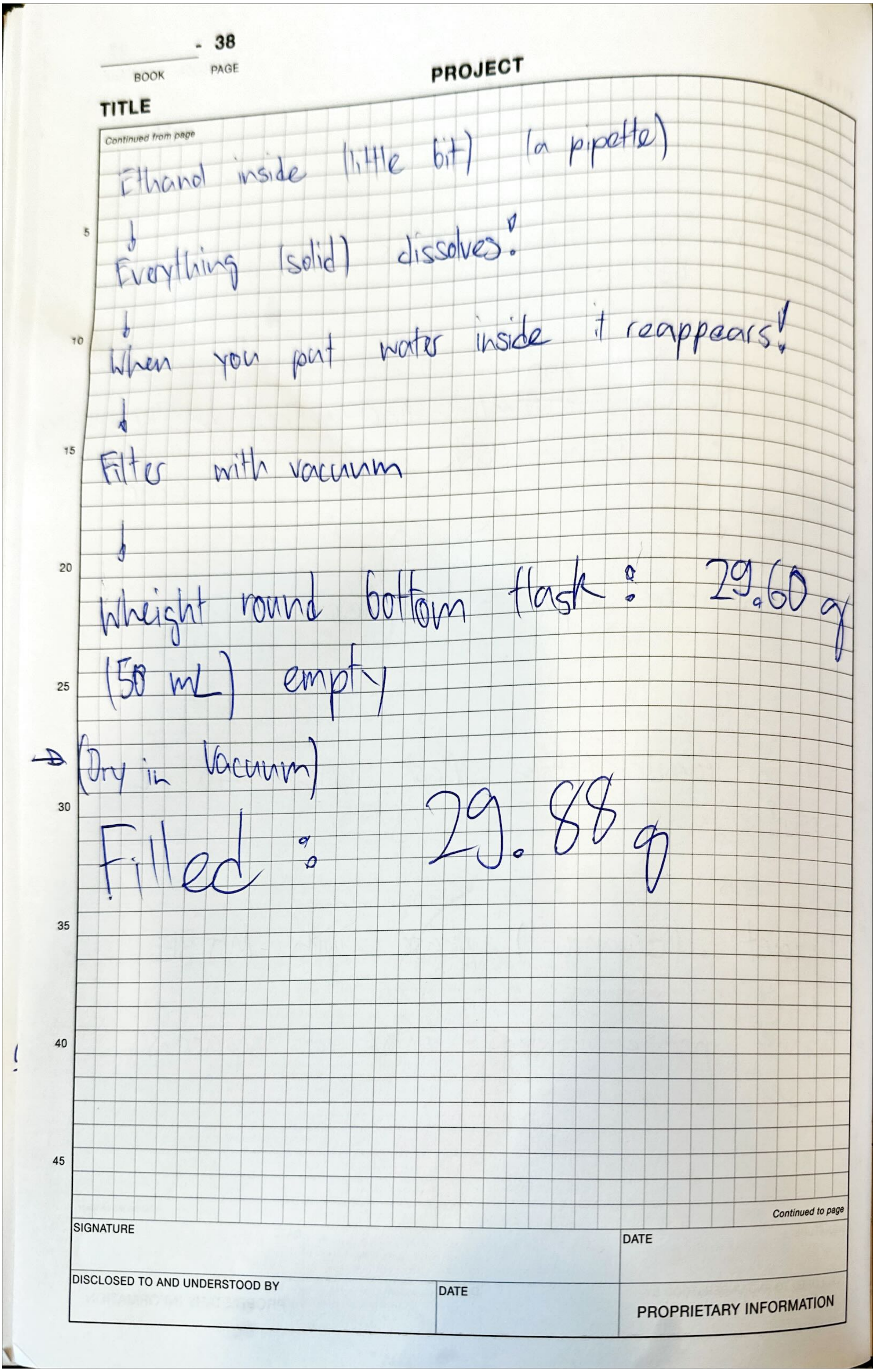

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BOOK PAGE

PROJECT

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Ethanol inside (little bit) (a pipette)

↓

Everything (solid) dissolves!

↓

When you put water inside it reappears!

↓

Filter with vacuum

↓

Wheight round bottom flask : 29.60 g
(50 mL) empty

→ (Dry in Vacuum)

Filled : 29.88 g

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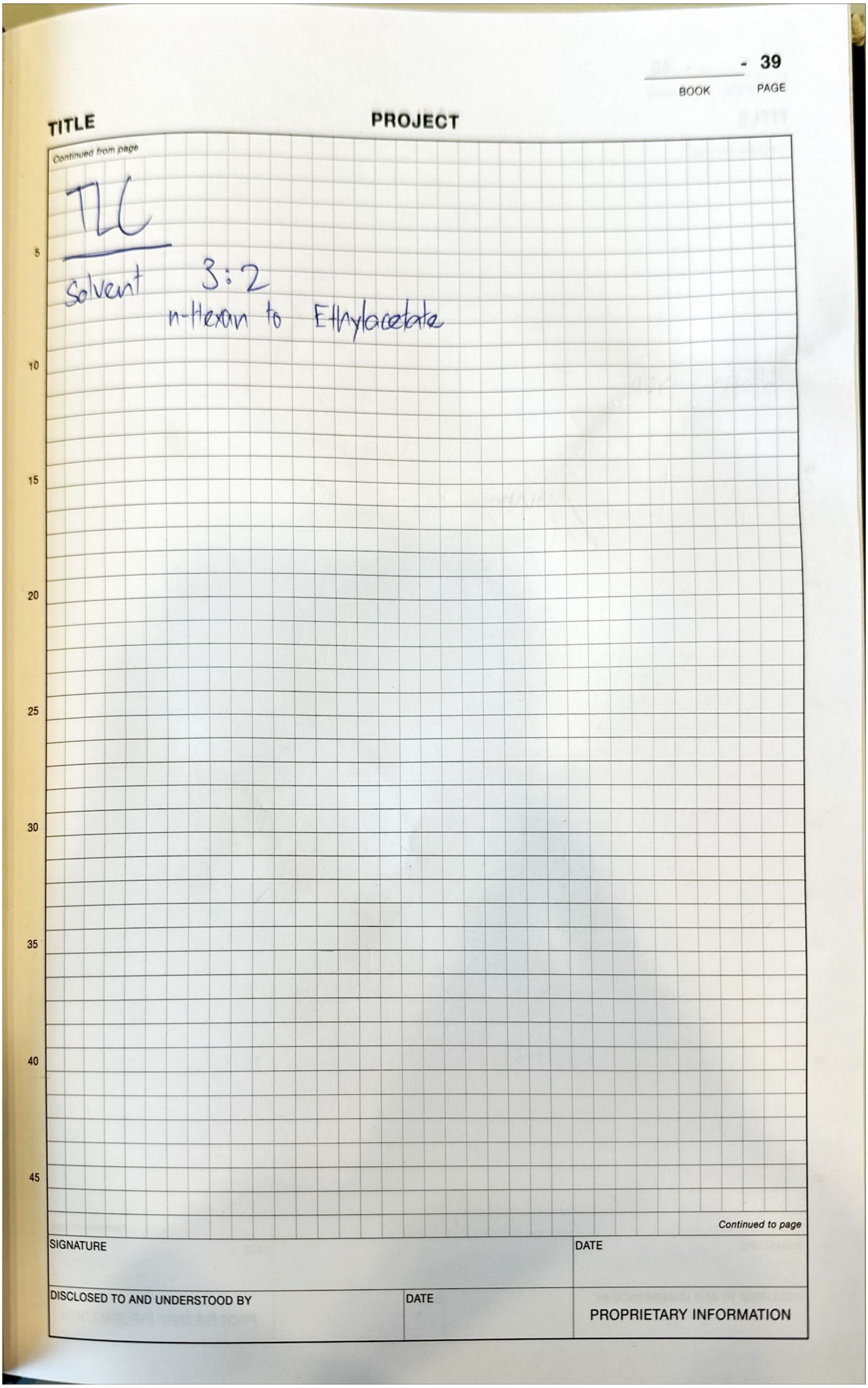

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TLC

Solvent 3:2

n-Hexan to Ethylacetate

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