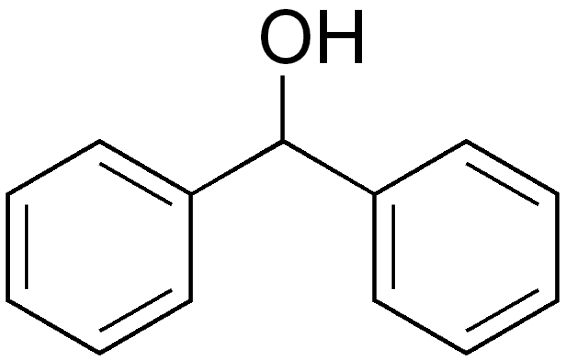
ETH zürich

Laboratory Course OACP I
Spring Semester 2024

The Synthesis of Diphenylmethanol by the Grignard Reaction



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Abstract The objective of this experiment is to synthesize diphenylmethanol and analyze the yield of the product. The synthesis is performed through the Grignard reaction of bromobenzene with magnesium turnings and benzaldehyde. The substance is purified by flash column chromatography. The success of the experiment is determined by thin layer chromatography, a melting point analysis and an infrared spectrum measurement. The product was a white crystalline solid (15.63 g, 88.83 mmol, 2.29 equiv) with a yield of about 229%.

Zürich, April 13, 2024

L. Loganathan

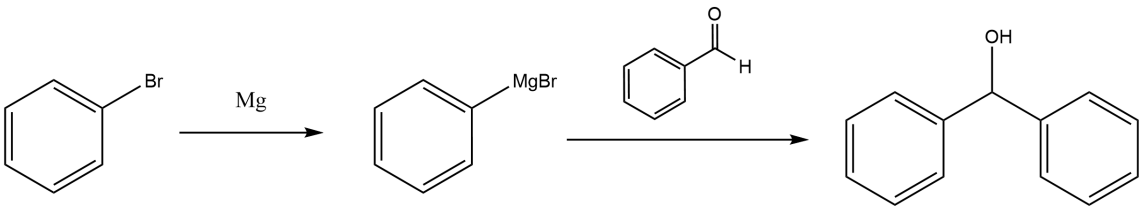
Loganathan Visva

Introduction

Diphenylmethanol, also known by its other name benzhydrol, consists of two phenyl groups joined to a central methanol group. Diphenylmethanol is a crystalline substance with a white hue, emitting a subtle characteristic scent [1]. As a result of its molecular structure, it demonstrates moderate solubility in organic solvents, while its solubility in water remains relatively low. It is used as a raw material in the pharmaceutical industry, particularly for antihistamines, such as diphenhydramine [2].

Material Properties

The synthesis of diphenylmethanol was carried out using the Grignard reaction. Magnesium turnings were combined with bromobenzene to form phenylmagnesium bromide. Upon the addition of a benzaldehyde solution, the following reaction yielded diphenylmethanol.



Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
Magnesium	24.31	1.738	651	1100
Diethyl Ether	74.12	0.7134	-116	34.6
Bromobenzene	157.01	1.5	-30.7	156.2
Iodine	253.81	4.9	113.7	184.4
Benzaldehyde	106.12	1.050	-26	179
Ammonium Chloride	53.49	1.5274	338 (decomposes)	520 (sublimes)
Sodium Bicarbonate	84.01	2.159	decomposes	-
Sodium Chloride	58.44	2.165	801	1413
Sodium Sulfate	142.04	2.671	884	decomposes
Diphenylmethanol	184.24	-	69	298

Substance	GHS Hazard Pictogram	H and P Phrases
Magnesium [3]		H228, H251, H261-P210, P223, P231, P232, P235, P240, P403
Diethyl Ether [4]		H224, H302, H336-P210, P233, P240, P241, P301, P312, P403
Bromobenzene [5]		H226, H315, H411-P210, P233, P240, P241, P273, P303, P361, P353
Iodine [6]		H302, H332, H312, H315, H319, H335, H372, H400-P273, P280, P301, P312, P302, P352, P304, P340, P314
Benzaldehyde [7]		H302, H332, H315, H319, H360, H335, H411-P273, P301, P312, P302, P352, P304, P340, P305, P351, P338, P308, P313
Ammonium chloride [8]		H302, H319-P264, P280, P301, P312, P305, P351, P338, P337, P313, P501
Sodium hydrogen carbonate [9]	-	-
Sodium chloride [10]	-	-
Sodium sulfate [11]	-	-
Diphenylmethanol [12]		H315, H319, H335, P302, P352, P305, P351, P338, P312, P403, P233, P501

Safety Assessment

In the lab, each chemical demands specific safety measures. Diethyl Ether is highly flammable, forms explosive peroxides over time, and vapors can cause dizziness. It must be stored away from light, and any distillation should be conducted under an inert atmosphere with proper monitoring for peroxides. Bromobenzene is less flammable but can cause skin and respiratory irritation. Can cause skin burns, respiratory and eye irritation. It should be handled with caution using personal protective equipment. Benzaldehyde is irritating to the skin and respiratory system and can also be harmful if ingested. Diphenylmethanol can cause skin irritation and respiratory irritation. Ammonium Chloride, Sodium Hydrogen Carbonate (Bicarbonate), Sodium Chloride, and Sodium Sulfate are generally less hazardous but can cause irritation upon prolonged contact.

The entire Grignard reaction should be conducted in a well-ventilated fume hood to avoid exposure to any harmful vapors, particularly from diethyl ether and benzaldehyde. The reaction can be exothermic, so adding reagents slowly to control the reaction rate is important. Moreover, the Grignard reagent should be prepared and handled under anhydrous conditions to prevent any violent reactions with water.

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.

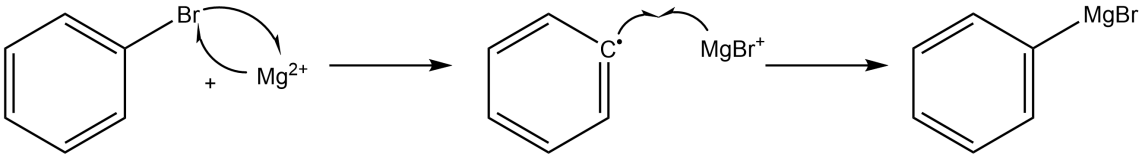
For avoiding any health issues that could be caused by the chemicals, gloves and safety googles should be worn in the laboratory. In a case of an accident the emergency number should be called. With the internal ETH-phones it would be 888, on personal mobile phones the number would be 044 342 11 88. 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

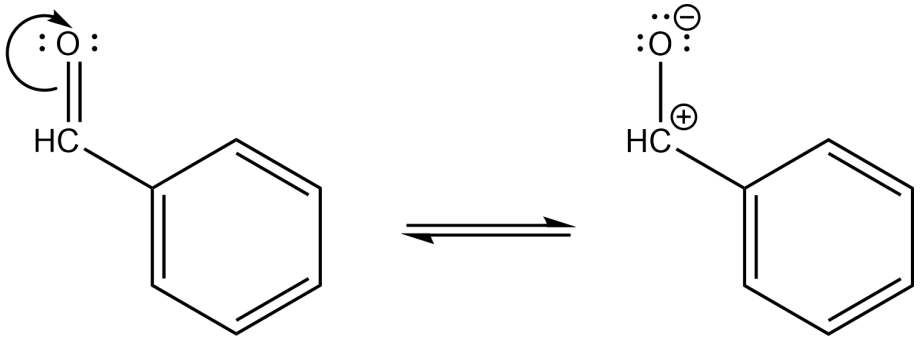
Diethylether, Bromobenzene and benzaldehyde are organic substances and should be disposed into the organic solvent waste. Sodium chloride can just go into the aqueous waste. Diphenylmethanol should go into the chlorinated solvent waste. Ammonium chloride should be disposed into the acidic waste. Sodium hydrogen carbonate should be disposed of into the aqueous basic waste and sodium sulfate into the solid waste. Iodine should be dissolved and put into the organic solvent waste.

Reaction Mechanism

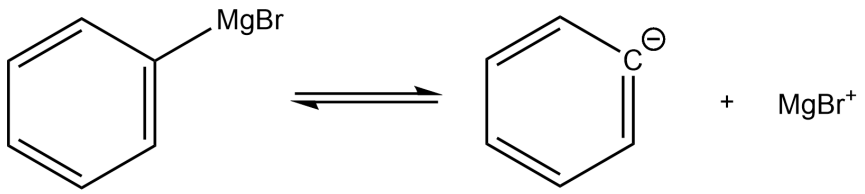
In this synthesis the Grignard reaction gets utilized. Initially, a Grignard reagent is synthesized by reacting bromobenzene with magnesium. In this process, magnesium cation reacts with the bromine atom, splitting the bromine-phenyl bond homolytically and it combines to a magnesium bromide cation. Subsequently, magnesium utilizes its additional electron to form a bond with the phenyl radical.



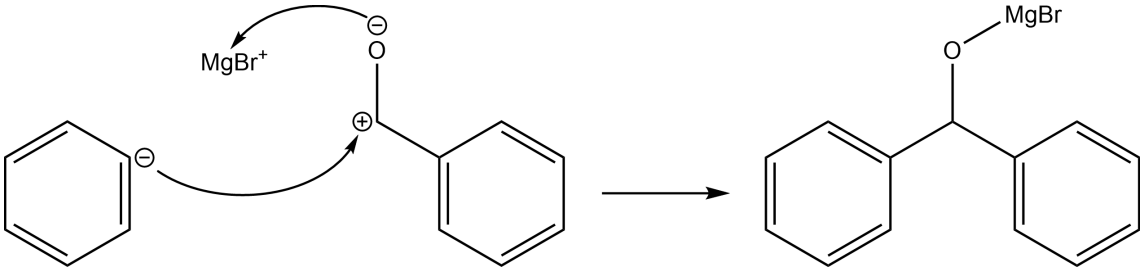
The benzaldehyde changes its resonance structure, which breaks the double bond and makes the oxygen negative and a carbon positive.



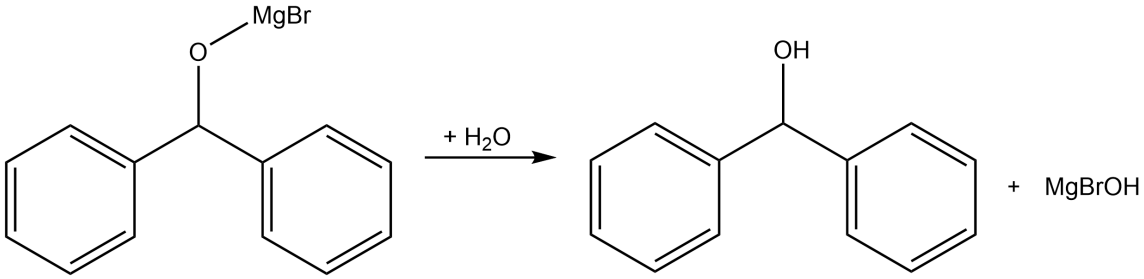
The magnesium bromide splits heterolytically from the benzene ring. This means that one C of the benzene ring is negatively charged and the MgBr is positively charged.



The negatively charged carbon on the benzene performs a nucleophilic attack on the positively charged carbon of the benzaldehyde. The negatively charged oxygen from the benzaldehyde also performs a nucleophilic attack on the magnesium bromide cation. This leads to the formation of magnesium bromide diphenylmethanolate.



During the final stage of the mechanism, the intermediate produced from the second step (magnesium bromide diphenylmethanolate) undergoes hydrolysis, leading to the formation of diphenylmethanol. This signifies the conclusion of the reaction process.



Atom Economy

Atom economy refers to the efficiency of a chemical reaction and it is calculated with the equation below. The atom economy of the reaction is approximately 50%. This reaction is somewhat atom efficient.

$$\text{Atom Economy} = \frac{\sum(M_{\text{Products}} \times \text{Equivalents})}{\sum(M_{\text{Reagents}} \times \text{Equivalents})}$$

$$\text{AE} = \frac{184.24\,\text{g/mol}}{(106.12\,\text{g/mol}) + (157.01\,\text{g/mol} \times 1.35) + (24.31\,\text{g/mol} \times 2.22)} \approx 49.52\%$$

Experimental

Procedure

Diphenylmethanol was synthesized via the Grignard reaction, where magnesium turnings were reacted with bromobenzene to generate phenylmagnesium bromide, which subsequently reacted with benzaldehyde to produce diphenylmethanol. The product was a white crystalline solid (15.63 g, 88.83 mmol, 2.29 equiv) with a yield of about 229%.

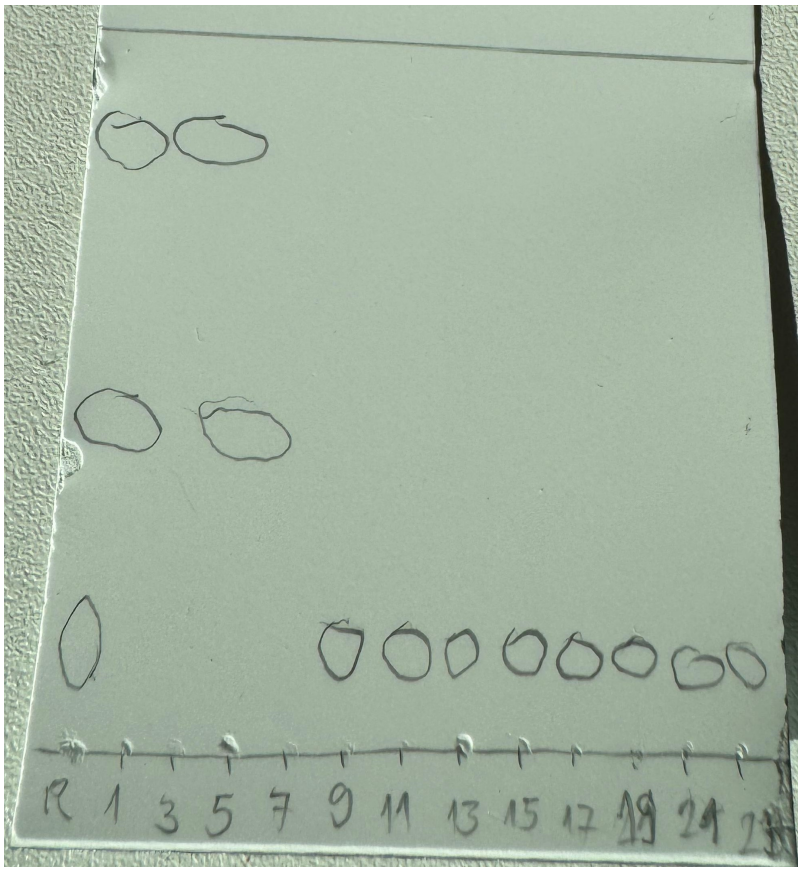
Observation

For the synthesis of the diphenylmethanol, a magnetic stir bar was placed in a 100 mL three-neck flask filled with anhydrous magnesium turnings (82 mmol, 2.00 g, 2.22 equiv.). On the three-neck flask a dropping funnel and a reflux condenser were added. Then 5.3 mL of bromobenzene (7.9 g, 50 mmol, 1.35 equiv.) were placed into the closed dropping funnel. 15 mL Diethylether were added. Then some iodine crystals were added, what turned the mixture into a orange clear liquid. Then the mixture was heated to 50 °C with a oil bath and some drops of bromobenzene were added while stirring. Then the dropping funnel with the bromobenzene was filled up to 20 mL with diethylether. This solution was dripped slowly into the mixture with the dropping funnel. The solution turned milky white. The mixture was heated to 80 °C what caused it to bubble. It became colourless and clear. After that more iodine crystals were added what caused the mixture to turn clear red. But it should turn turbid, that's a sign that the grignard reaction didn't occur. But the mixture was still stirred for one hour at room temperature after which it turned milky green. The mixture was cooled down with an ice bath and inserted in 20 mL of ammonium chloride. With the help of a TLC the teaching assistant figured out that the reaction didn't work out. So the author took about half of the mixture from a lab partner for the flash column chromatography. The eluent for the flash column chromatography was a 15:1 n-hexane ethylacetate mixture. After performing the flash column chromatography, the solution was put into a rotatory evaporator. The leftover white crystalline solid was the product.

Characterisation

TLC and Flash Column Chromatography

A flash column chromatography was conducted to purify the 2-cyclohexylidene acetate. A flash column chromatography is just like a longer reverse thin layer chromatography for the entire mixture. The solvent was 15 parts n-hexane to 1 part ethylacetate. In whole there were 24 flasks that got filled.



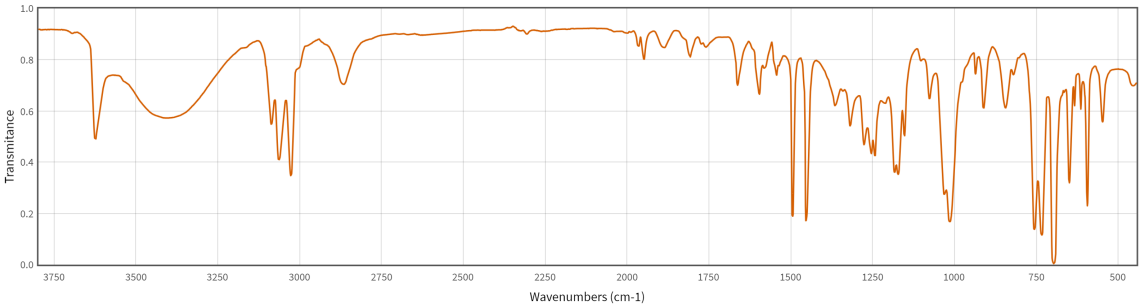
A TLC was conducted with R as the reference and the numbers as the numbers of the flasks from the flash column chromotagraphy. The TLC showed that there was no product inside flask 1 to 7, only impurities. They could stem from bromobenzene and diphenyl, a byproduct from a sidereaction, because they are less polar than diphenylmethanol and therefore rose up higher on the TLC plate. Flask 9 to 24 have diphenylmethanol in them, so the liquid from these flasks was put together into a 500 mL round bottomed flask. It appeared as a clear liquid. After some time on the rotatory evaporator only 15.63 g of a white crystalline solid was left over, what would be a extraordinary high yield of about 229%.

Melting Point Analysis

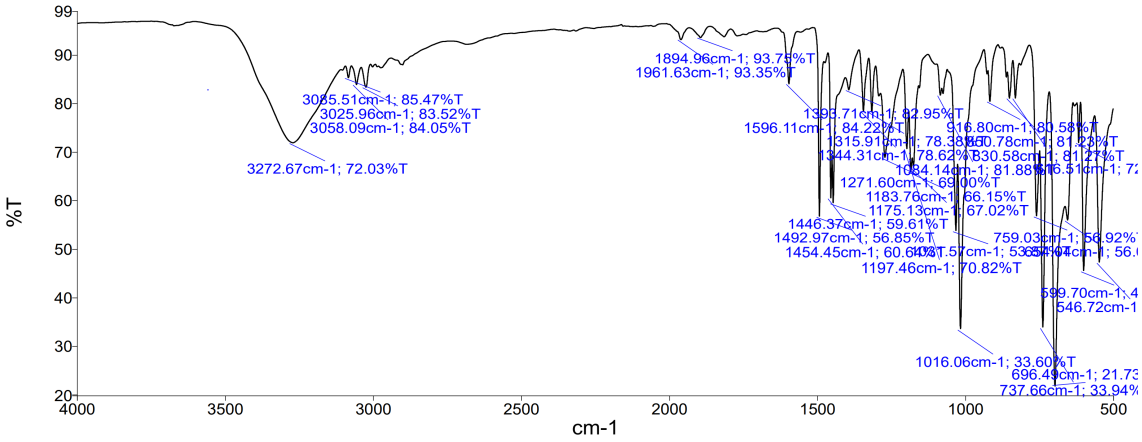
The melting point of the product was analyzed by filling a capillary tube with the white crystalline solid and using a melting point tester. For this the capillary tube was inserted in the melting point tester. At 58°C the crystalline solid started clumping together and at 61 °C the substance completely melted into a clear liquid. The literature value for the melting point of diphenylmethanol is 69 °C, so the measured value from this experiment is somewhat near to it. The slightly lower melting temperate could be explained by the fact that there may are still traces of benzaldehyde with a much lower melting point of - 26°C in the substance.

IR Spectrum

This would be the literature values of the infrared spectrum of diphenylmethanol [13].



And this would be the measured infrared spectrum of the synthesized diphenylmethanol from the author.



Wavenumber (cm^{-1})	Bond	%T
3272.67	O-H stretch (Alcohol)	72.03
3085.51	Aromatic C-H stretch	85.47
3025.96	Aromatic C-H stretch	83.52
3058.09	Aromatic C-H stretch	84.05
1894.96	Unknown/Impurity	93.79
1961.63	Unknown/Impurity	93.35
1596.11	Aromatic C=C stretch	84.22
1446.37	C-H bending (Aromatic)	59.61
1492.97	C-H bending (Aromatic)	56.85
1454.45	C-H bending (Aromatic)	60.69
1175.13	C-H in-plane bend	67.02
1016.06	C-O stretch (Alcohol)	33.60

In the spectral analysis, the observed peaks align with those documented in the literature, although they are weaker in intensity [14]. The wavelengths are accurate, but the absorption rates are lower, indicated by the T%, potentially due to the presence of impurities, variations in sample concentration, or the influence of the instrument's sensitivity and configuration. The impurities could explain the extraordinary high yield.

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Appendix

List of H and P Phrases

- H228: Flammable solid.
- H251: Self-heating; may catch fire.
- H261: In contact with water releases flammable gases.
- P210: Keep away from heat, sparks, open flames, hot surfaces. — No smoking.
- P223: Do not allow contact with water.
- P231: Handle under inert gas.
- P232: Protect from moisture.
- P235: Keep cool.
- P240: Ground/bond container and receiving equipment.
- P403: Store in a well-ventilated place.
- H224: Extremely flammable liquid and vapour.
- H302: Harmful if swallowed.
- H336: May cause drowsiness or dizziness.
- P233: Keep container tightly closed.
- P241: Use explosion-proof electrical/ventilating/lighting equipment.
- P301: IF SWALLOWED: Call a POISON CENTER or doctor/physician if you feel unwell.
- P312: Call a POISON CENTER or doctor/physician if you feel unwell.
- H226: Flammable liquid and vapour.
- H315: Causes skin irritation.
- H411: Toxic to aquatic life with long lasting effects.
- P273: Avoid release to the environment.
- P303: IF ON SKIN (or hair): Wash with plenty of soap and water.
- P361: Remove all contaminated clothes immediately.
- P353: Rinse skin with water/shower.
- H332: Harmful if inhaled.
- H312: Harmful in contact with skin.
- H319: Causes serious eye irritation.
- H335: May cause respiratory irritation.
- H372: Causes damage to organs through prolonged or repeated exposure.
- H400: Very toxic to aquatic life.
- P280: Wear protective gloves/protective clothing/eye protection/face protection.
- P302: IF ON SKIN: Wash with plenty of soap and water.
- P352: Wash with plenty of soap and water.

- P304: IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
- P340: If breathing is difficult, remove victim to fresh air and keep at rest in a position comfortable for breathing.
- P314: Get medical advice/attention if you feel unwell.
- H360: May damage fertility or the unborn child.
- P305: IF IN EYES: Rinse cautiously with water for several minutes.
- P351: Remove contact lenses, if present and easy to do. Continue rinsing.
- P338: Remove contact lenses, if present and easy to do. Continue rinsing.
- P308: IF exposed or if you feel unwell: Call a POISON CENTER or doctor/physician.
- P313: Get medical advice/attention.
- P264: Wash hands thoroughly after handling.
- P501: Dispose of contents/container to an approved waste disposal plant.
- P50: (Note: This appears to be an incomplete code and might need to be corrected or further specified.)

Table from the Experiment Manual

Reactant	MW	Equiv	Moles	Mass	Volume	Purity
Benzaldehyde	106,12	1.00	37 mmol	3.9	3.9	-
Bromobenzene	157,01	1.35	50 mmol	7.9	5.2	-
Magnesium	24,31	2.22	82 mmol	2.00 g	— (solid)	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	
	184,24				69°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	-	C≡N	stretching	nitrile	-
2260-2190	weak	-	C≡C	stretching	alkyne	disubstituted
2175-2140	strong	-	S-C≡N	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	-	C≡C	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	-

Contributors and Attributions

- OChemOnline

Exp. 4a

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Safety Seminar

Brombenzene
Magnesium / Diethylether } highly flammable

Brombenzene / Benzaldehyde → go through gloves!

Magnesium fire → put it out with sand

2g Mg - turnids

5,3 mL Brombenzene in → Dropping funnel

15mL Et_2O

- Adding of 2 Crystals
orangene Flüssigkeit (liquid) → clear

↓

Rührfisch + 50° C heating

some drops Brombenzene

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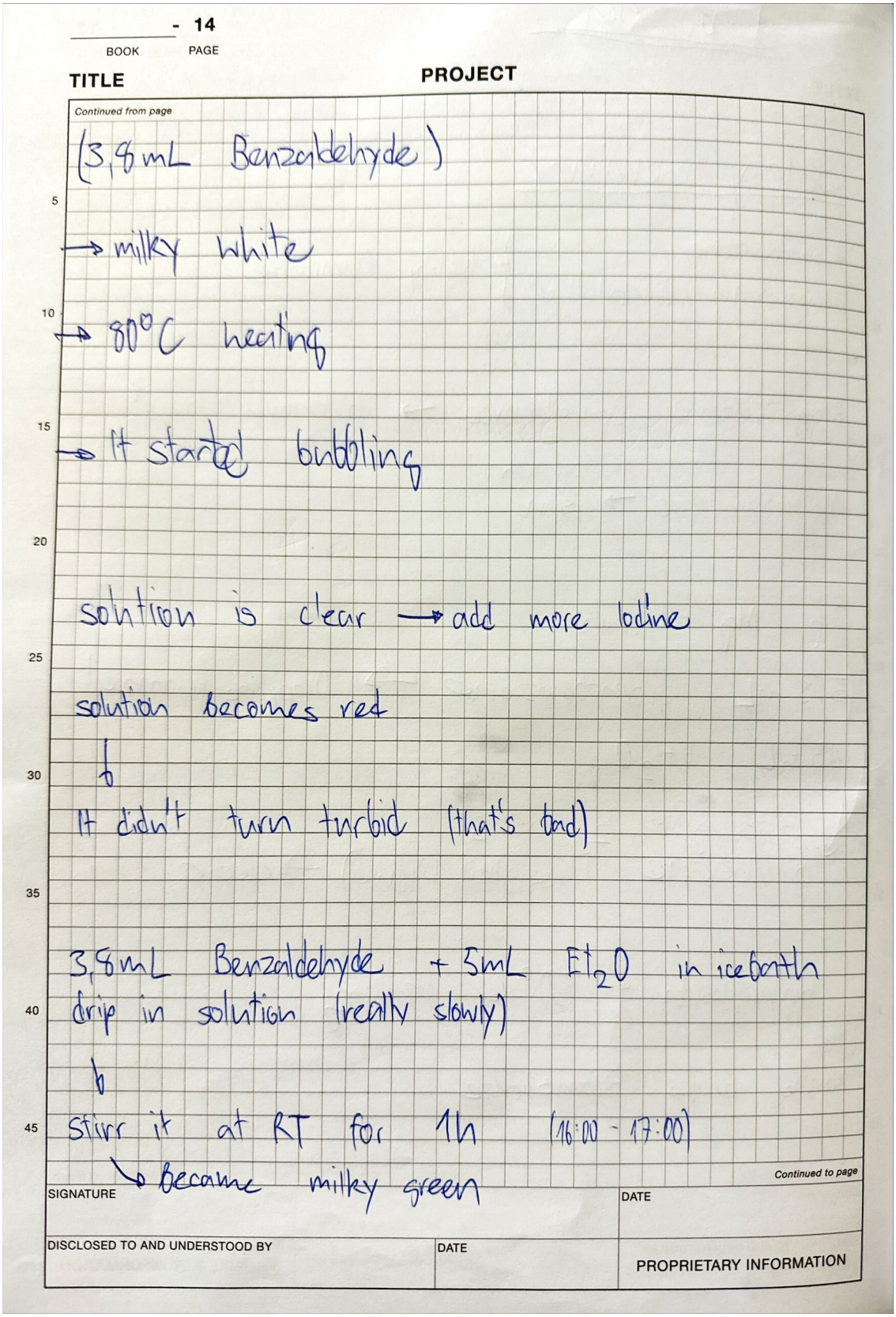

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(3,8 mL Benzaldehyde)

→ milky white

→ 80°C heating

→ it started bubbling

solution is clear → add more Iodine

solution becomes red

↓

it didn't turn turbid (that's bad)

3,8 mL Benzaldehyde + 5mL Et_2O in ice bath
drip in solution (really slowly)

↓

Stirr it at RT for 1h (16:00 - 17:00)

↳ Became milky green

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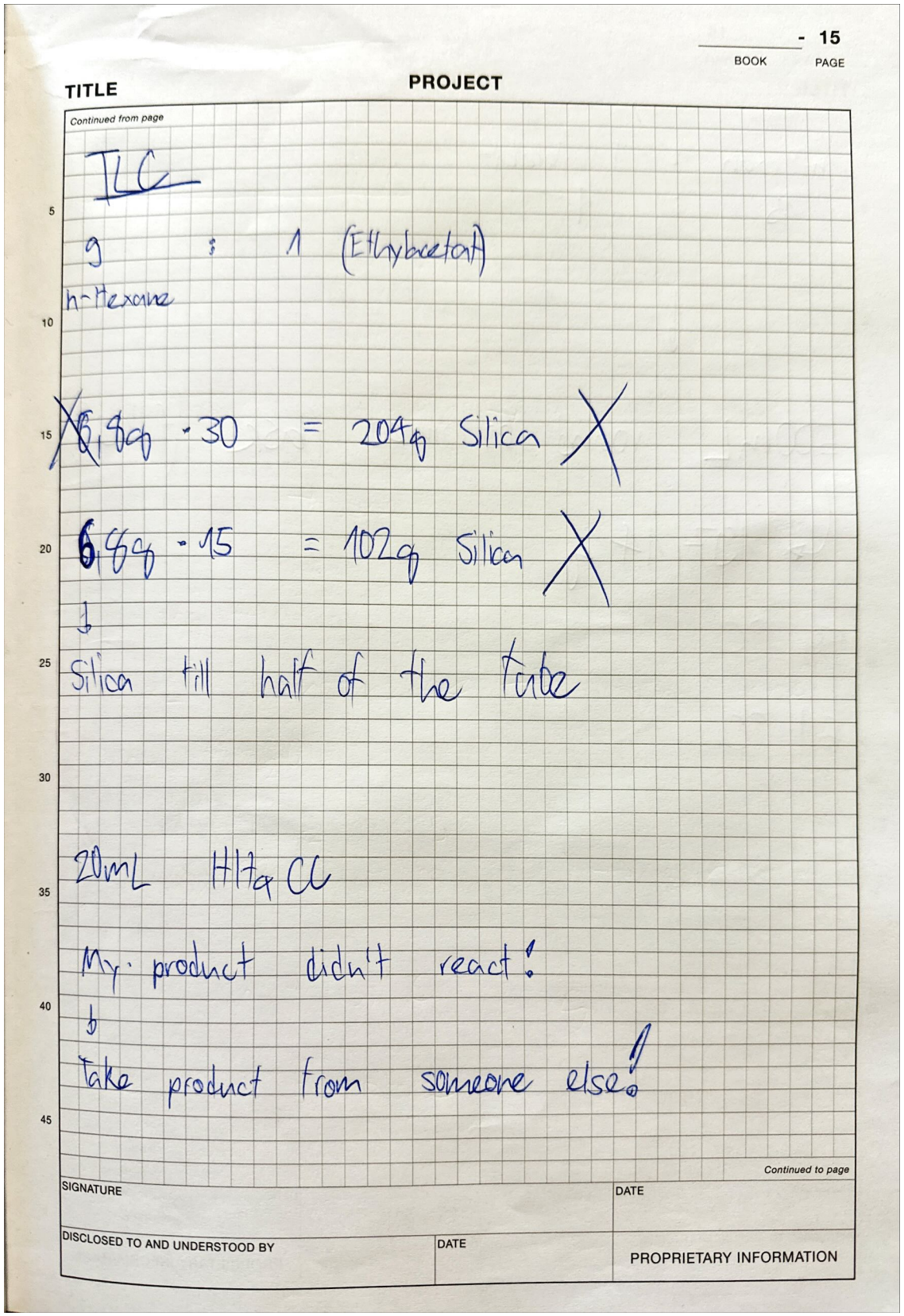

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TLC

9 : 1 (Ethylacetat)

n-Hexane

~~6,8g~~ · 30 = 204g Silica X

6,8g · 15 = 102g Silica X

↓

Silica till half of the tube

20mL CH_2Cl_2

My product didn't react!

↓

Take product from someone else!

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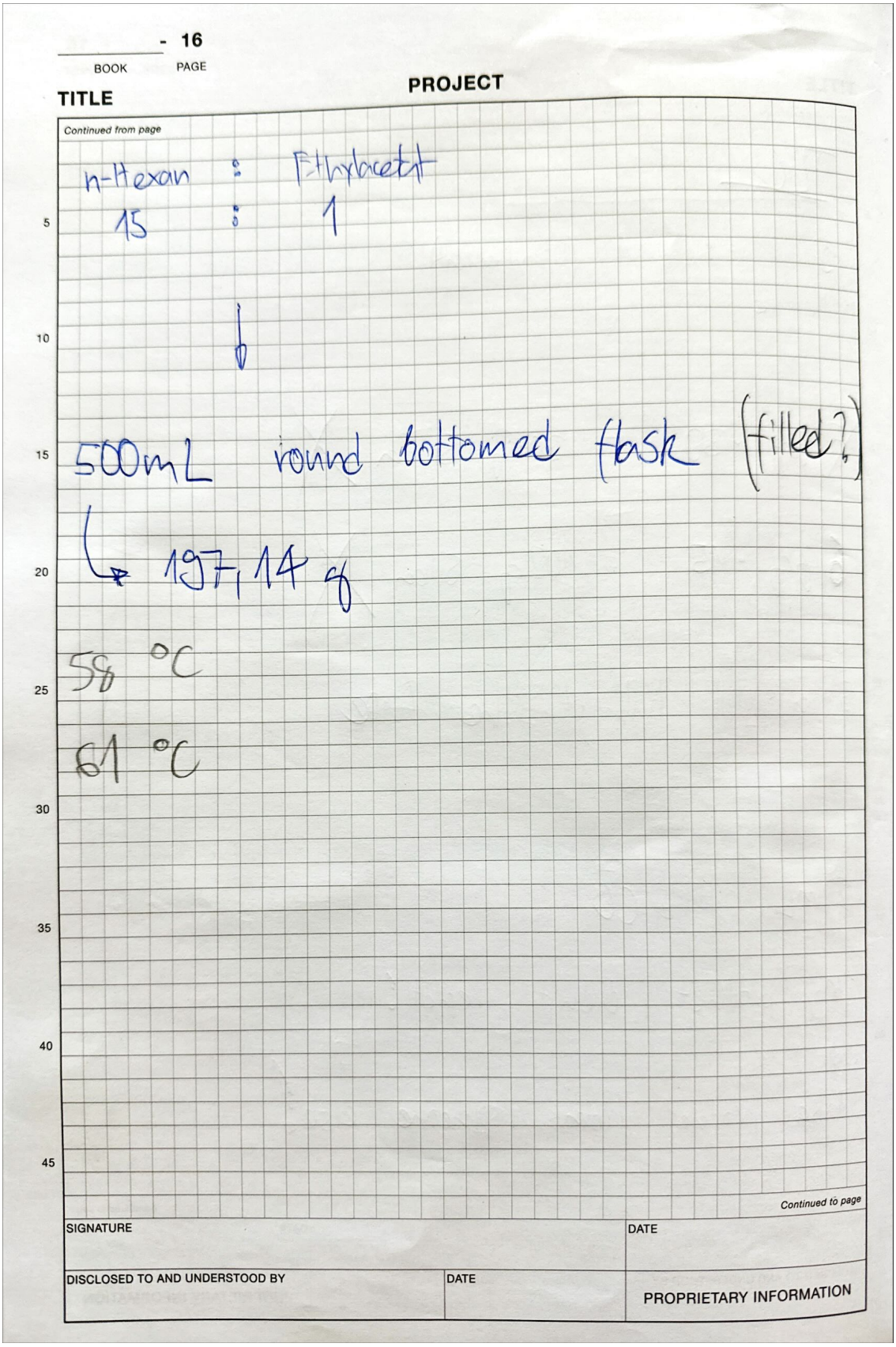
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n-Hexan : Ethylacetat
15 : 1
500mL round bottomed flask (filled?)
↳ 197,14 g
58 °C
61 °C
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