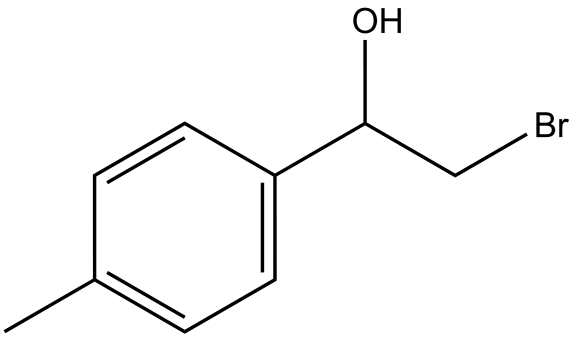
ETH zürich

Laboratory Course OACP I
Spring Semester 2024

The Synthesis of 2-Bromo-1-p-tolylethanol by Electrophilic Alkene Addition



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Abstract The objective of this experiment is to synthesize 2-bromo-1-p-tolylethanol and analyze the yield of the product. The synthesis is performed through the electrophilic alkene addition of 4-methylstyrene with NBS and water. The substance was purified by flash column chromatography. The success of the experiment is determined by thin layer chromatography, melting point analysis and infrared spectrum measurements.

Zürich, May 3, 2024

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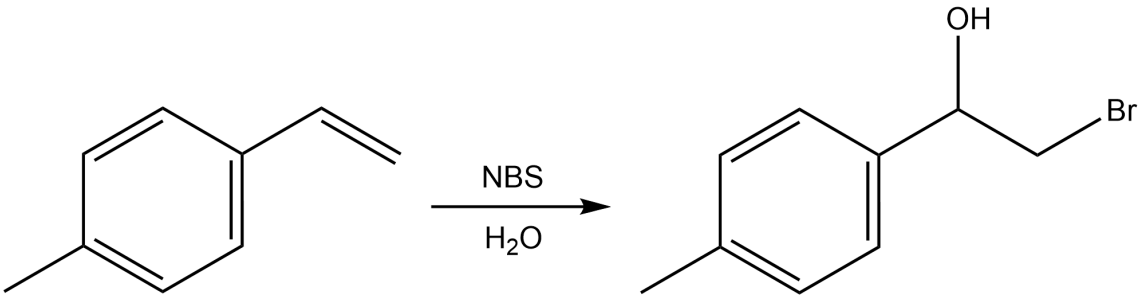
Introduction

2-Bromo-1-p-tolylethanol consists of a p-tolyl group, that means the phenyl group has a methyl group at the para position, which is directly opposite the attachment point of the phenyl group to the rest of the molecule. The other side of the phenyl ring is connected to a ethyl structure with a alcohol group on the first carbon and a bromide at the second carbon.

2-Bromo-1-p-tolylethanol appears to be a colorless to pale yellow liquid at room temperature. This compound is used in the synthesis of various chemicals and can be involved in pharmaceutical research, particularly as a building block for producing more complex molecules. However, detailed information on specific applications or industrial uses might not be readily available [1].

Material Properties

For the synthesis of 2-bromo-1-p-tolylethanol electrophilic additions to Alkenes was used. To a solution of 4-methylstyrene in THF, N-Bromosuccinimide (NBS) and water were mixed together to react and form the product 2-bromo-1-p-tolylethanol.



Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
4-Methylstyrene [2]	118.18	0.9173	-37.8	172
Tetrahydrofuran [3]	72.11	0.8892	-108.3	65
N-Bromosuccinimide [4]	177.98	2.098	174	338.9
Sodium thiosulfate pentahydrate [5]	248.19	1.667	48	decomposes
Ethylacetate [6]	88.11	0.902	-83	77
Sodium chloride [7]	58.443	2.17	800.7	1'465
Sodium sulfate [8]	142.04	2.671	888	-
2-Bromo-1-p-tolylethanol [1]	215.09	1.4	-	282

Substance	GHS Hazard Pictogram	H and P Phrases
4-Methylstyrene		H226, H304, H315, H319, H332, H335, H411, H412, P210, P233, P240, P241, P242, P243, P261, P264, P264+P265, P271, P273, P280, P301+P316, P302+P352, P303+P361+P353, P304+P340, P305+P351+P338, P317, P319, P321, P331, P332+P317, P337+P317, P362+P364, P370+P378, P391, P403+P233, P403+P235, P405, P501
Tetrahydrofuran		H225, H302, H319, H351, H336, H335-P202, P210, P233, P301, P312, P305, P351, P338, P308, P313
N-Bromosuccinimide		H272, H290, H315, H319, H317, H341, H400-P210, P273, P280, P302, P352, P305, P351, P338, P308, P313
Sodium thiosulfate pentahydrate	-	-
Ethylacetate		H225, H319, H336-P210, P233, P240, P241, P242, P305, P351, P338
Sodium chloride	-	-
Sodium sulfate		-
2-Bromo-1-p-tolylethanol	-	-

Safety Assessment

In the lab, each chemical demands specific safety measures. 4-Methylstyrene produces highly flammable liquid and vapor and can cause skin irritation and allergic reactions is suspected of causing genetic defects. Always keep it away from heat. Tetrahydrofuran is extremely flammable and can cause serious eye irritation, drowsiness or dizziness. Always keep the substance away from heat, and avoiding inhalation of vapors. N-Bromosuccinimide is a strong oxidizer that enhances the risk of fire. It can cause severe skin burns, eye damage and respiratory irritation. Ethyl Acetate also produces highly flammable liquid and vapor. It can cause serious eye irritation and should be kept away from heat. Sodium thiosulfate pentahydrate, sodium chloride and sodium sulfate are generally considered non-hazardous. However, standard laboratory precautions should be taken when handling any chemical, especially contact with the eyes or the skin should always be avoided.

The entire reaction should be conducted in a well ventilated fume hood to avoid exposure to any harmful vapors. Many substances in this experiment produce harmful or flammable vapors particularly 4-methylstyrene, tetrahydrofuran, n-bromosuccinimide and ethyl acetate.

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.

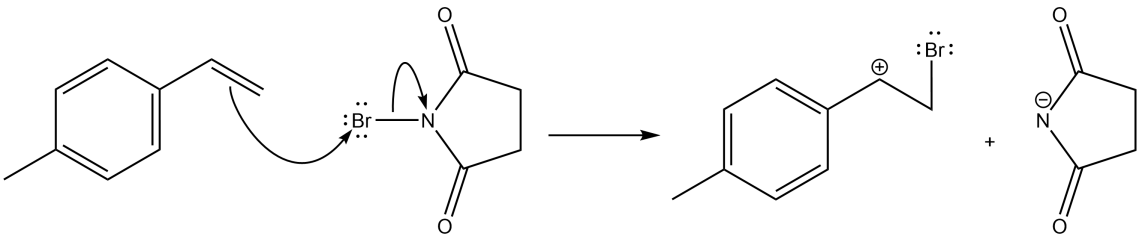
For avoiding any health issues that could be caused by the chemicals, gloves and safety googles should be worn in the laboratory. In a case of an accident the emergency number should be called. With the internal ETH-phones it would be 888, on personal mobile phones the number would be 044 342 11 88.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

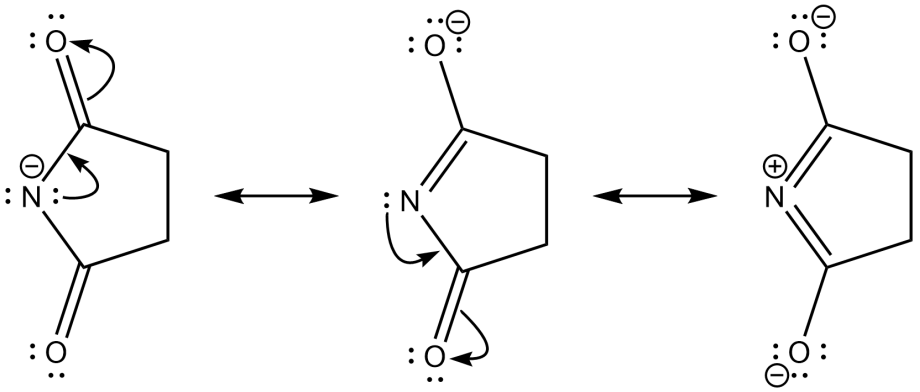
4-Methylstyrene, tetrahydrofuran, ethylacetate and 2-bromo-1-p-tolylethanol should be disposed into the organic solvent waste. Sodium sulfate, sodium chloride, sodium thiosulfate and NBS are solid at room temperature and should be disposed of into the solid waste.

Reaction Mechanism

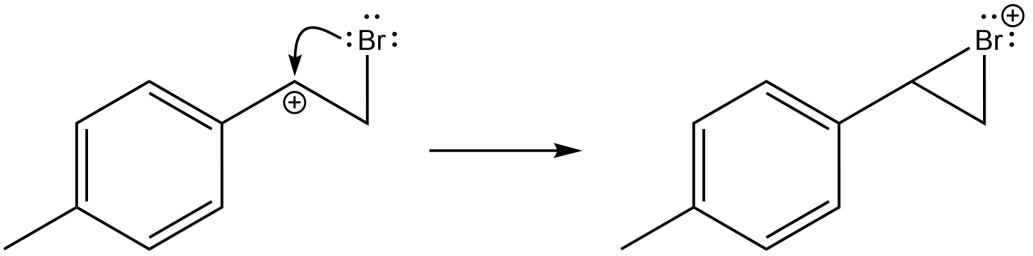
During the initial stage of the reaction mechanism, the nucleophilic Π bond in the 4-methylstyrene attacks the electrophilic bromine atom present in the N-bromosuccinimide. This interaction leads to the formation of a bromonium cation intermediate called 2-(p-tolyl)bromiran-1-ium and a N-bromosuccinimide anion called 2,5-dioxopyrrolidin-1-ide.



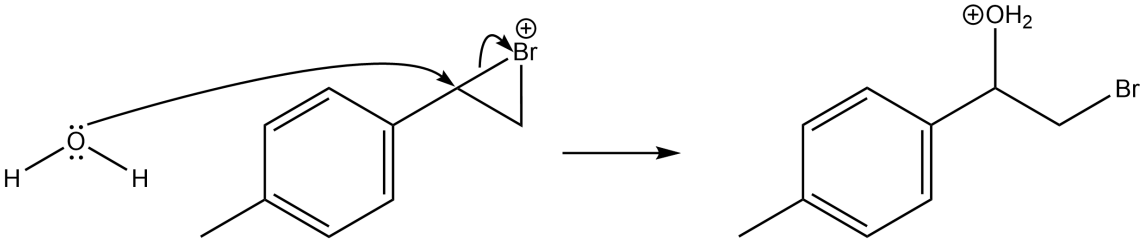
2,5-dioxopyrrolidin-1-ide has three resonance structures that add to it's resonance. The structure with a neutral nitrogen, a negative oxygen, and a neutral oxygen is the most stable. This form avoids charge separation and places the negative charge on the more electronegative atom. Less stable is the structure with a negatively charged nitrogen and two neutral oxygen. There is no charge separation, but the negative charge is not on the most electronegative atom. And the least stable is the structure with a positively charged nitrogen and two negative oxygen. Despite placing negative charges on oxygen atoms, the introduction of a positive charge on the less electronegative nitrogen makes this the least stable due to significant charge separation.



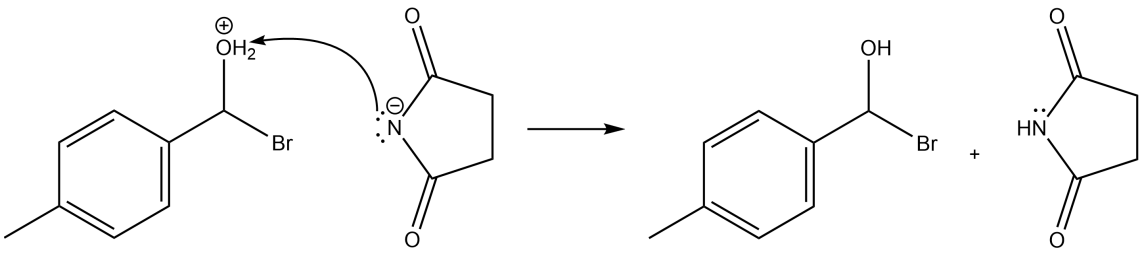
Intramolecular reactions happen much faster than intermolecular reactions. One of the lone pairs of the bromine conducts a nucleophilic attack on the positively charged carbon. That leads to the formation of a covalent bond between the carbon and the bromine.



The water conducts a nucleophilic attack on the carbon atom what causes the formation of a covalent bond between the oxygen of the water molecule and the carbon.It also causes the covalent bond between the carbon and the bromine to break and the electron pair to go back to the bromine and become a lone pair again. what leads to the formation of (bromo(p-tolyl)methyl)oxonium.



The (bromo(p-tolyl)methyl)oxonium now can act as a Brønsted–Lowry acid and the 2,5-dioxopyrrolidin-1-ide can act as a lewis base. That results in the formation of the product 2-Bromo-1-p-tolylethanol and pyrrolidine-2,5-dione.



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 68.46%. This reaction is somewhat atom efficient.

$$\mathrm{AE} = \frac{M_{\mathrm{Product}}}{M_{\mathrm{Reagents}}} = \frac{215.09\ \mathrm{g/mol}}{118.18\ \mathrm{g/mol} + 18.02\ \mathrm{g/mol} + 177.98\ \mathrm{g/mol}} \approx 68.46\%$$

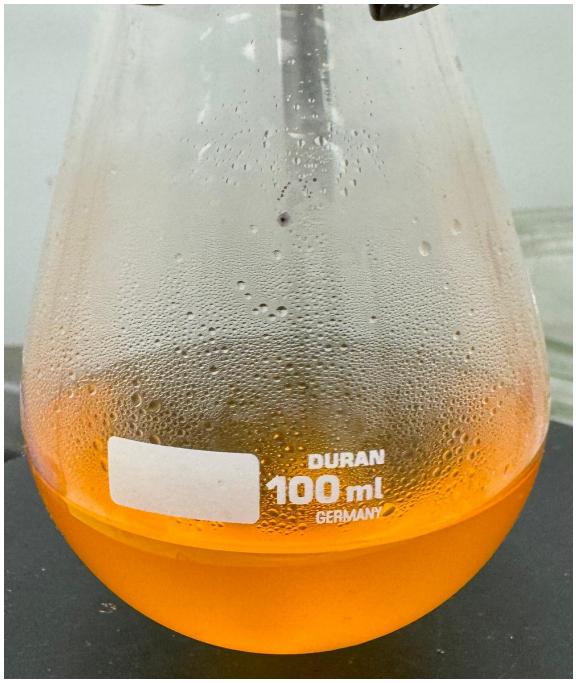
Experimental

Procedure

The synthesis was performed by electrophilic alkene addition. In this procedure, 4-methylstyrene is mixed with THF and water, to which NBS is introduced. The reaction is monitored using TLC until it reaches completion. Post-reaction, a solution of sodium thiosulfate pentahydrate is added. The solvent THF is then removed by evaporation. The resulting product is then extracted multiple times using ethyl acetate. The organic extracts are then cleansed with a saline solution, dried over anhydrous sodium sulfate, filtered, and the solvent is removed with a frit and a vacuum to obtain the desired product. The product 2-bromo- 1-p-tolylethanol was a clear liquid (2.26 g, 10.51 mmol, 0.70 equiv.) with a yield of about 70.05%.

Observation

In a 100 mL one-necked round bottom flask a magnetic stir bar was positioned. Then 30 mL THF, 6 mL water and NBS (4.005 g, 22.5 mmol, 1.50 equiv.) were added into the flask. It appeared as a yellow liquid mixture. After putting in the 4-methylstyrene (1.93 mL, 15.0 mmol, 1.00 equiv.) and mixing the solution, it turned into a milky white liquid. The mixture was stirred for 15 minutes at room temperature. Then a TLC was done, that still showed traces of the educt in the mixture, so the solution was stirred for 45 minutes again. In this time it slowly turned from milky white to milky yellow and later to milky orange. Then a second TLC was done that showed the mixture only with product and no educt.



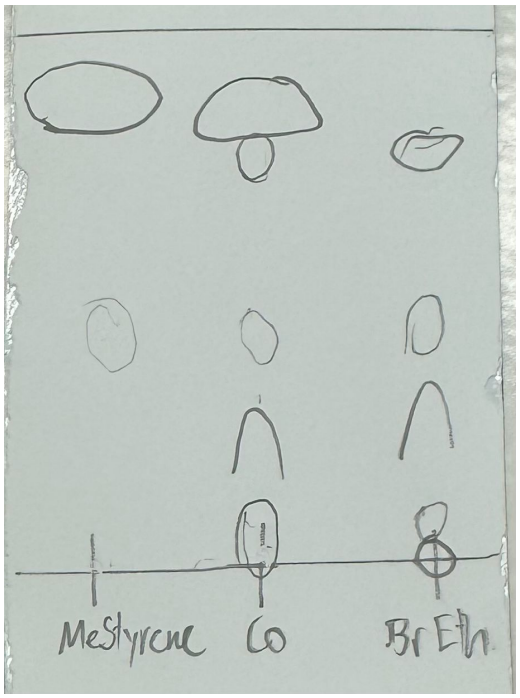
Then 3.8 g sodium thiosulfate pentahydrate was dissolved in 10 mL of water. This solution was put into the orange mixture, what caused it to turn milky white. The mixture was then put into a rotarotary evaporator to evaporate the THF. There remained a clear liquid. Then with the clear liquid was put into a 100 mL separation funnel with ethylacetate. The organic layer swam on top of the aqueous layer and was separated. The organic phase was washed with a saturated sodium chloride solution.Then so much magnesium sulfate was put inside this mixture until it stopped clumping. Then this mixture was put into a frit with a vacuum. The white solid on top of the frit does not contain the product. The product is contained in the liquid inside the vacuum flask.

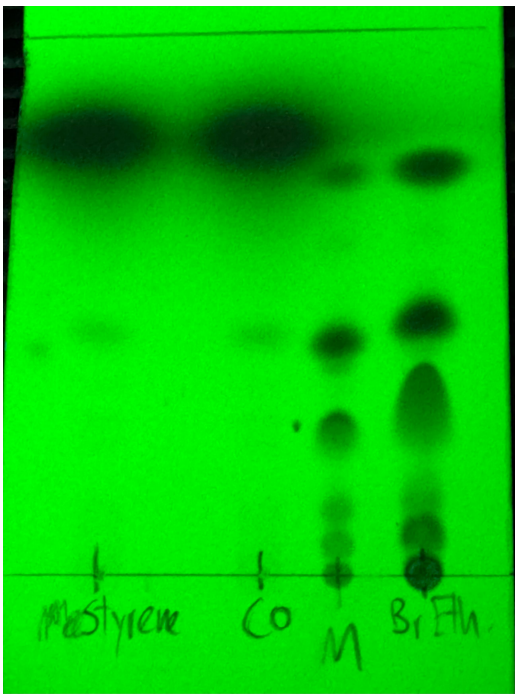
A chromatography column was filled up with a silica layer until the middle of he column and on top with a sand layer. Then the chromatography column was filled up to the sand layer with the eluent mixture 9:1 n-hexane to ethylacetate. Then the liquid was put on top of the sand layer. The liquid was absorbed into the sand layer. Then another sand layer was put on top. Then the column was filled with the eluent mixture and was connected to the nitrogen flow so pressure could be applied to it. Then a flash column chromatography was performed. The liquid in the test tubes with the product was poured into a pear flask.This solution containing the desired product was put in a rotatory evaporator to evaporate the eluent. After that only the product 2-Bromo1-p-tolylethanol was left over. It was a clear liquid (2.26 g, 10.51 mmol, 0.70 equiv.) with a yield of about 70.05%.

Characterisation

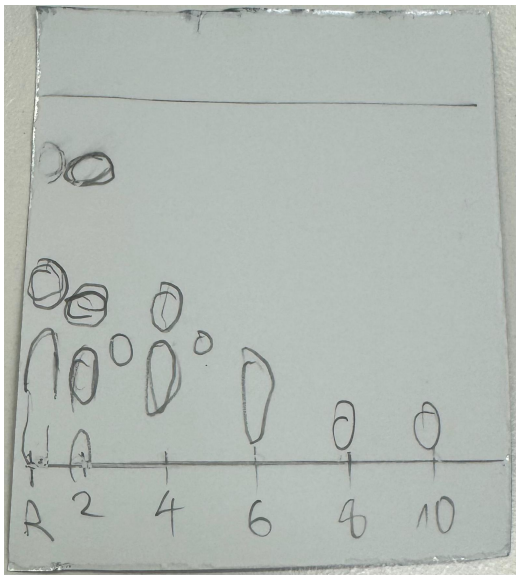
TLC and Flash Column Chromatography

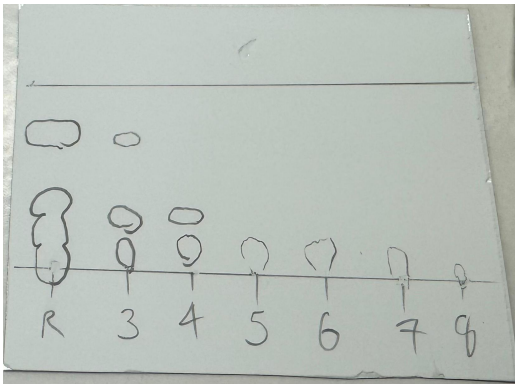
The first TLC after 15 minutes of stirring showed that there were still traces of the educt in the mixture. That means not everything reacted, so the stirring was continued. The second TLC after one hour showed that no more educt was in the mixture. The reaction could be continued. The eluent was a 9:1 n-hexane to ethylacetate mixture. The first column (MeStyrene) had the educt methylstyrene dissolved in acetone on it. The third column (BrEth) had the mixture dissolved in acetone on it. The second column (Co) had both on it.





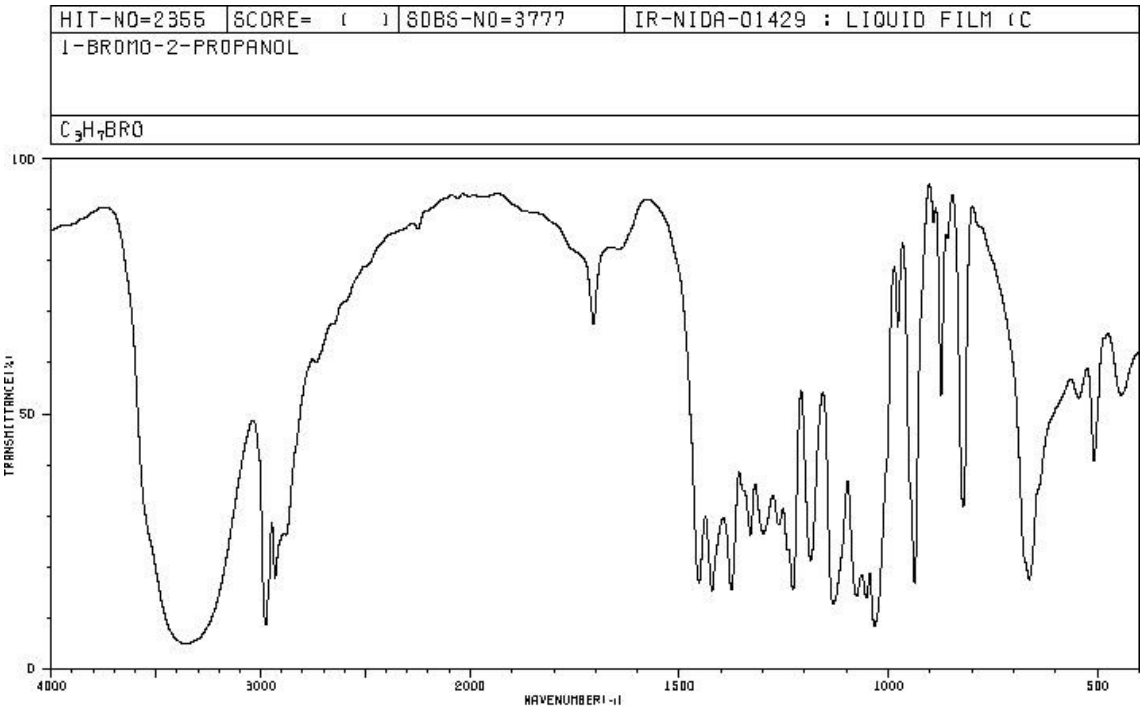
Later a flash column chromatography was performed with the same eluent mixture. There was a TLC done after 10 flasks to check if there is still the desired product inside. R is the reference from the origanl mixture. The numbers stand for the flask from which a sample was put on top with a capillary tube. The first TLC shows that there is only product in the flasks 4 to 8. The second TLC analyzed the flask 3 to 8. The second TLC showed that there is only product in flask 3 to 7. So the contents of flask 3 to 7 were put together into a round bottom flask for the rotarotary evaporation.



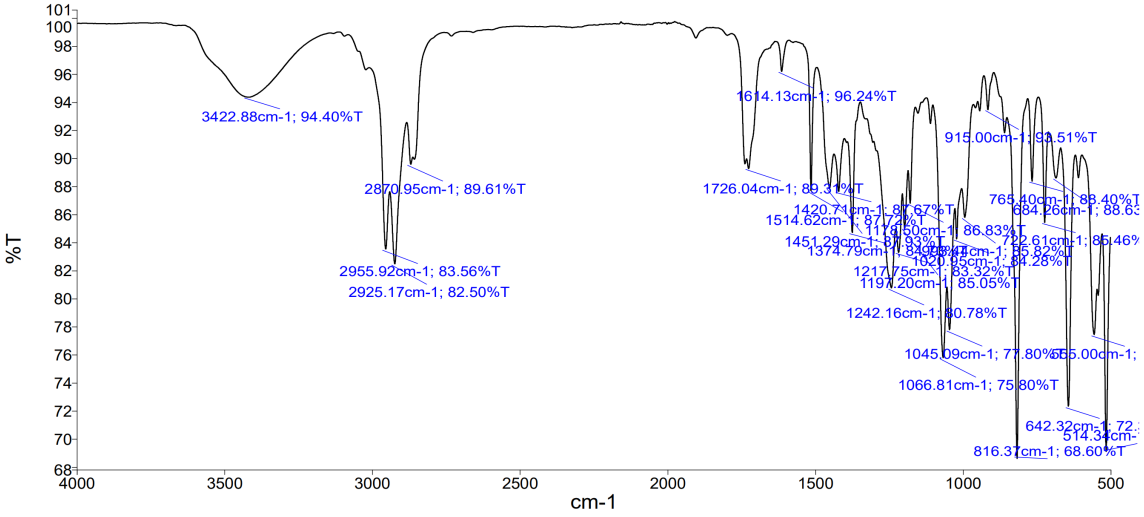


IR Spectrum

There were no literature IR values found for 2-bromo-1-p-tolylethanol. Instead there were literature values taken from 1-bromo-2-propanol, a structural similar compound that lacks the benzene ring.



And this would be the measured infrared spectrum of the synthesized 2-bromo-1-p-tolylethanol from the author.



In the spectral analysis, the observed peaks mostly align with those documented in the literature, although they are weaker in intensity [9]. 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.

Wavenumber (cm^{-1})	Bond	%T
3422.88	O-H stretch (broad)	94.40%
2955.92	C-H stretch (Aliphatic)	83.56%
2925.17	C-H stretch (Aliphatic)	82.50%
2870.95	C-H stretch (Aliphatic)	89.61%
1726.04	C=O stretch (Ester)	89.31%
1614.13	C=C stretch (Aromatic)	96.24%
1514.62	C=C stretch (Aromatic)	87.72%
1451.29	C-H bending (Methyl)	87.93%
1420.71	C-H bending (Methyl)	87.67%
1374.79	C-H bending (Methyl)	84.73%
1242.16	C-N stretch (Amine)	80.78%
1217.75	C-O stretch (Alcohol)	83.32%
1197.20	C-O stretch (Alcohol)	85.05%
1178.50	C-O stretch (Alcohol)	86.83%
1066.81	C-O stretch (Ether)	75.80%
1045.09	C-O stretch (Ether)	77.80%
1020.95	C-O stretch (Ether)	84.28%
993.44	C-O stretch (Ether)	85.82%
915.00	C-H bend (Aromatic)	93.51%
816.37	C-H out-of-plane bend	68.60%
765.40	C-H out-of-plane bend	88.40%
722.61	C-H out-of-plane bend	85.46%
684.26	C-H out-of-plane bend	88.63%
642.32	C-H out-of-plane bend	72.33%
555.00	C-H out-of-plane bend	77.45%
514.34	C-H out-of-plane bend	69.11%

References

[1] Chemspider. (1s)-2-bromo-1-(4-methylphenyl)ethanol. https://www.chemspider.com/Chemical-Structure.9519865.html.

[2] PubChem. 4-methylstyrene. https://pubchem.ncbi.nlm.nih.gov/compound/4-Methylstyrene

[3] PubChem. Tetrahydrofuran. https://pubchem.ncbi.nlm.nih.gov/compound/Tetrahydrofuran.

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

[5] PubChem. Thiosulfuric acid, disodium salt, pentahydrate. https://pubchem.ncbi.nlm.nih.gov/compound/Thiosulfuric-acid_-disodium-salt_-pentahydrate

[6] PubChem. Ethyl acetate. https://pubchem.ncbi.nlm.nih.gov/compound/Ethylacetate.

[7] PubChem. Sodium chloride. https://pubchem.ncbi.nlm.nih.gov/compound/Sodium-Chloride

[8] PubChem. Sodium sulfate. https://pubchem.ncbi.nlm.nih.gov/compound/Sodium-Sulfate.

[9] Infrared spectroscopy absorption table. https://chem.libretexts.org/Ancillary_Materials/Reference/Reference_Tables/Spectroscopic_Reference_Tables/Infrared_Spectroscopy_Absorption_Table. Accessed on 02.03.2024.

Appendix

List of H and P Phrases

- **H226**: Highly flammable liquid and vapor.
- **H304**: May be fatal if swallowed and enters airways.
- **H315**: Causes skin irritation.
- **H319**: Causes serious eye irritation.
- **H332**: Harmful if inhaled.
- **H335**: May cause respiratory irritation.
- **H351**: Suspected of causing cancer.
- **H360**: May damage fertility or the unborn child.
- **H372**: Causes damage to organs through prolonged or repeated exposure.
- **H290**: May be corrosive to metals.
- **H302**: Harmful if swallowed.
- **H312**: Harmful in contact with skin.
- **H317**: May cause an allergic skin reaction.
- **H318**: Causes serious eye damage.
- **H336**: May cause drowsiness or dizziness.
- **H361**: Suspected of damaging fertility or the unborn child.
- **H373**: May cause damage to organs through prolonged or repeated exposure.
- **H400**: Very toxic to aquatic life.
- **H410**: Very toxic to aquatic life with long lasting effects.
- **H411**: Toxic to aquatic life with long lasting effects.
- **P210**: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.
- **P233**: Keep container tightly closed.
- **P240**: Ground and bond container and receiving equipment.
- **P241**: Use explosion-proof equipment.
- **P242**: Use non-sparking tools.
- **P243**: Take action to prevent static discharges.
- **P261**: Avoid breathing dust/fume/gas/mist/vapors/spray.
- **P264**: Wash skin 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, and eye protection.
- **P301+P310**: IF SWALLOWED: Immediately call a POISON CENTER or doctor.

- **P303+P361+P353**: IF ON SKIN (or hair): Remove immediately all contaminated clothing. Rinse skin with water or shower.
- **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.
- **P332+P313**: If skin irritation occurs: Get medical advice/attention.
- **P362+P364**: Take off contaminated clothing and wash it before reuse.
- **P370+P378**: In case of fire: Use appropriate media to extinguish.
- **P403+P233**: Store in a well-ventilated place. Keep container tightly closed.
- **P405**: Store locked up.
- **P501**: Dispose of contents/container to an appropriate waste disposal plant.

Table from the Experiment Manual

Reactant	MW	Equiv	Moles (mmol)	Mass (g)	Volume(mL)	Purity
4-Methylstyrene	118.18	1.00	15.0 mmol	1,773	1,93	-
N-Bromosuccinimid	177.98	1.50	22.5	4,005	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**		
2-Bromo-1-p-tolylethanol	215.09					

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	-

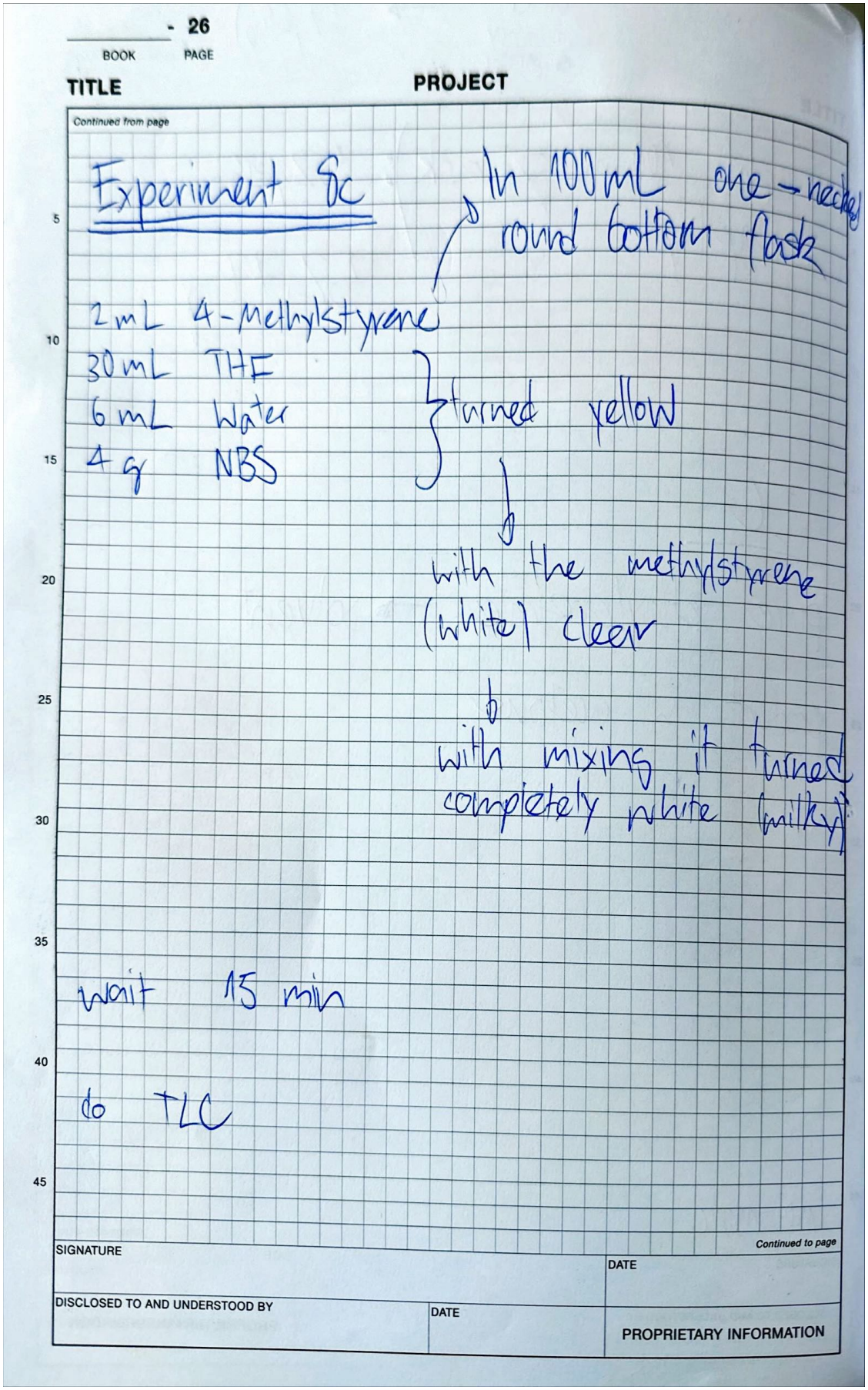
900-700 cm^{-1}

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



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TITLE PROJECT

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Experiment 8c

In 100 mL one-necked round bottom flask

2 mL 4-Methylstyrene
30 mL THF
6 mL Water
4 g NBS

} turned yellow

with the methylstyrene (white) clear

with mixing it turned completely white (milky)

wait 15 min

do TLC

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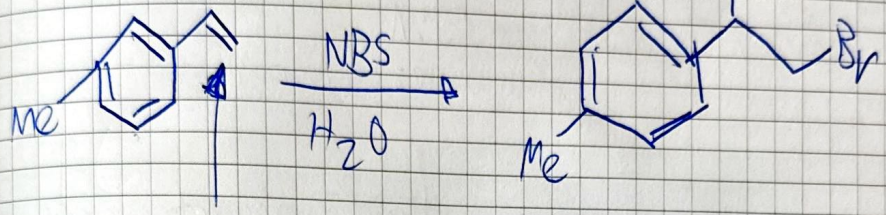
3,99 g sodium thiosulfate pentahydrate in 10 mL water

↓

4-Methylstyrene



double bond into single bond

TLC

9:1

n-Hexane : Ethylacetate

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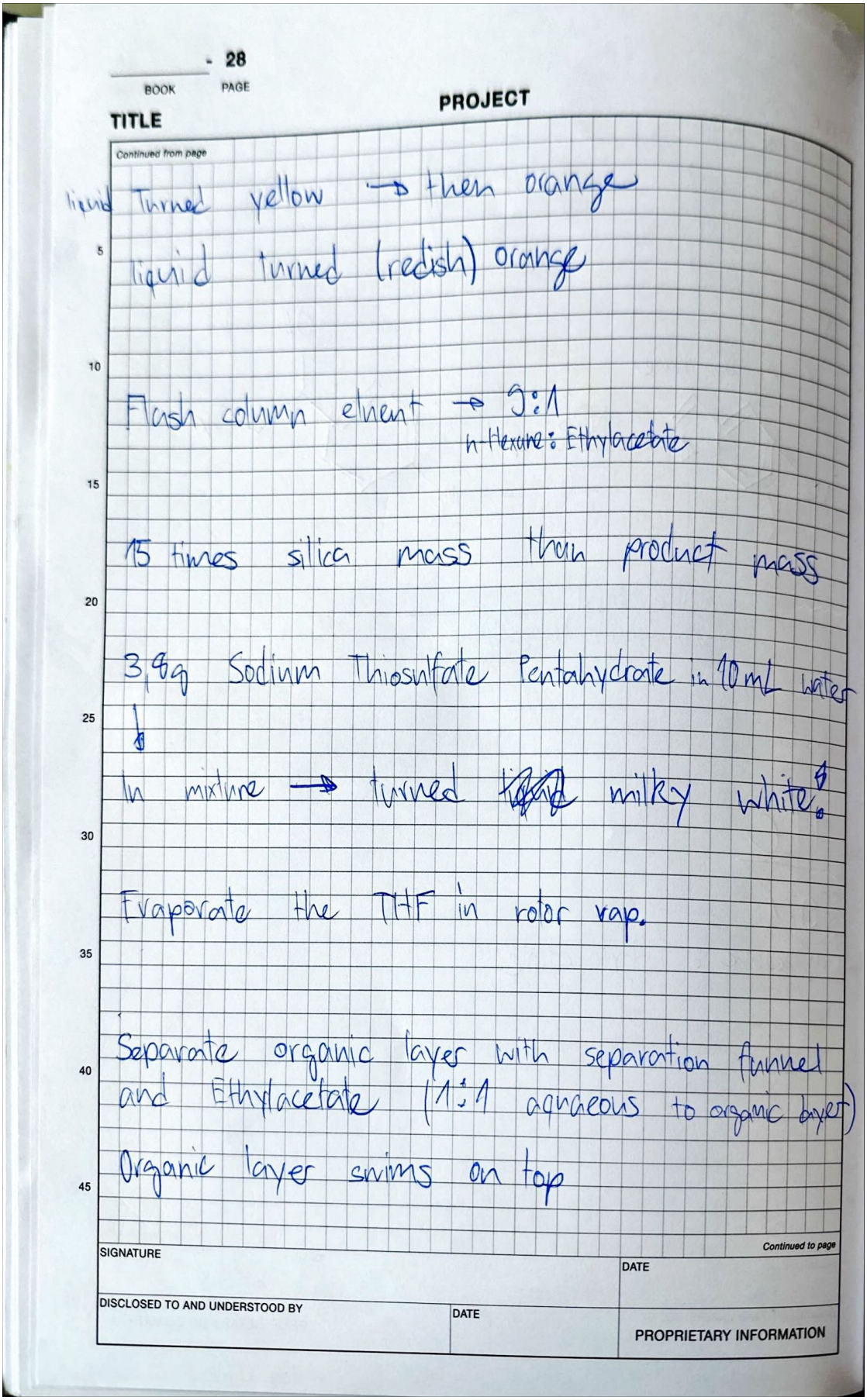

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TITLE PROJECT

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liquid Turned yellow → then orange

liquid turned (redish) orange

Flash column eluent → 9:1
n-Hexane : Ethylacetate

15 times silica mass than product mass

3,8g Sodium Thiosulfate Pentahydrate in 10mL water

↓

In mixture → turned ~~[illegible]~~ milky white.

Evaporate the THF in rotor vap.

Separate organic layer with separation funnel and Ethylacetate (1:1 aqueous to organic layer)

Organic layer swims on top

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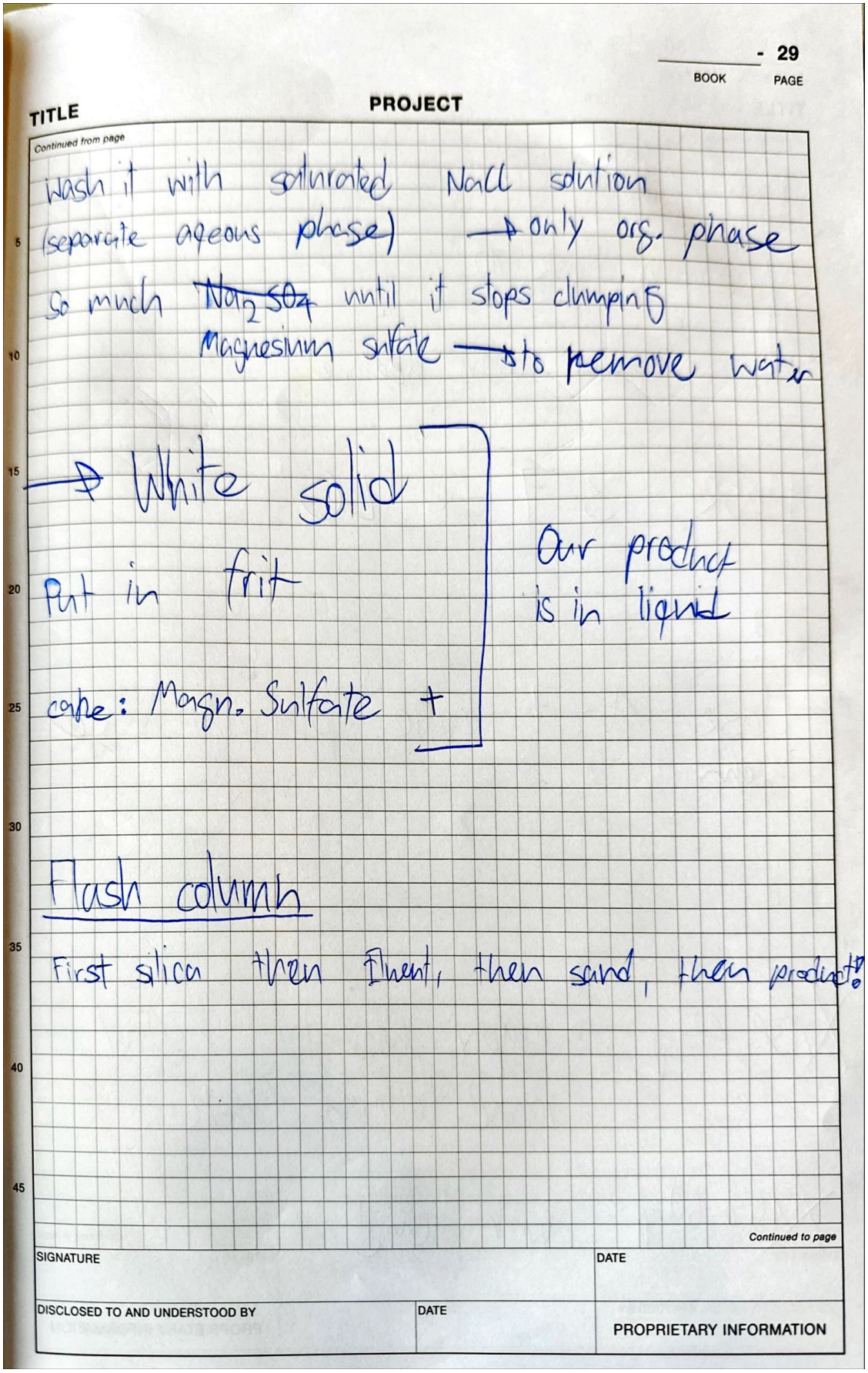

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BOOK PAGE

TITLE PROJECT

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Wash it with saturated NaCl solution (separate aqeous phase) → only org. phase

So much Na_2SO_4 until it stops clumping

Magnesium sulfate → to remove water

→ White solid

Put in frit

Our product is in liquid

cake: Magn. Sulfate +

Flash column

First silica then Ehent, then sand, then product

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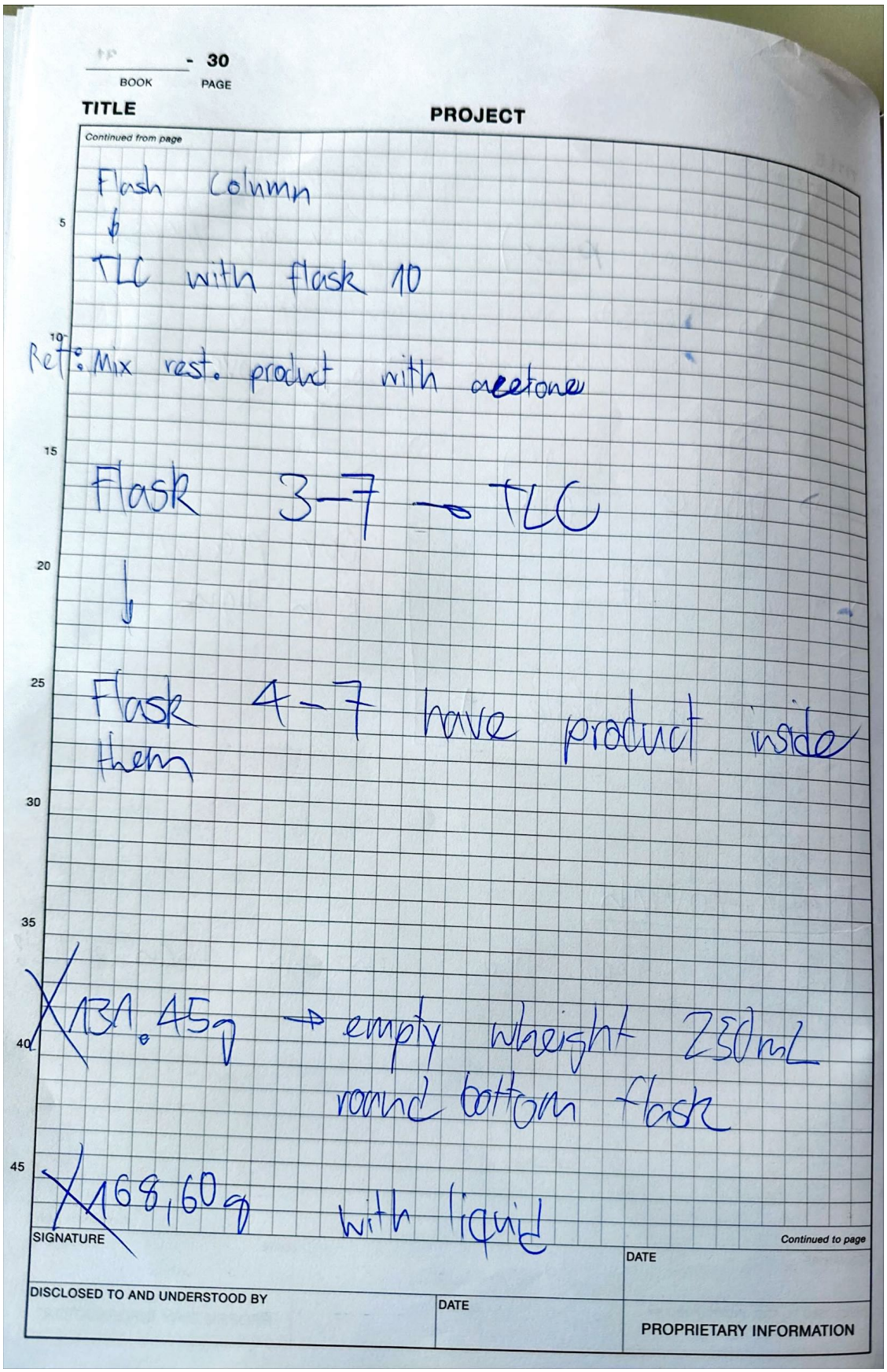

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Flash Column

↓

TLC with flask 10

Ref.: Mix rest. product with acetone

Flask 3-7 → TLC

↓

Flask 4-7 have product inside them

~~13A.45g~~ → empty wheight 250mL round bottom flask

~~168,60g~~ with liquid

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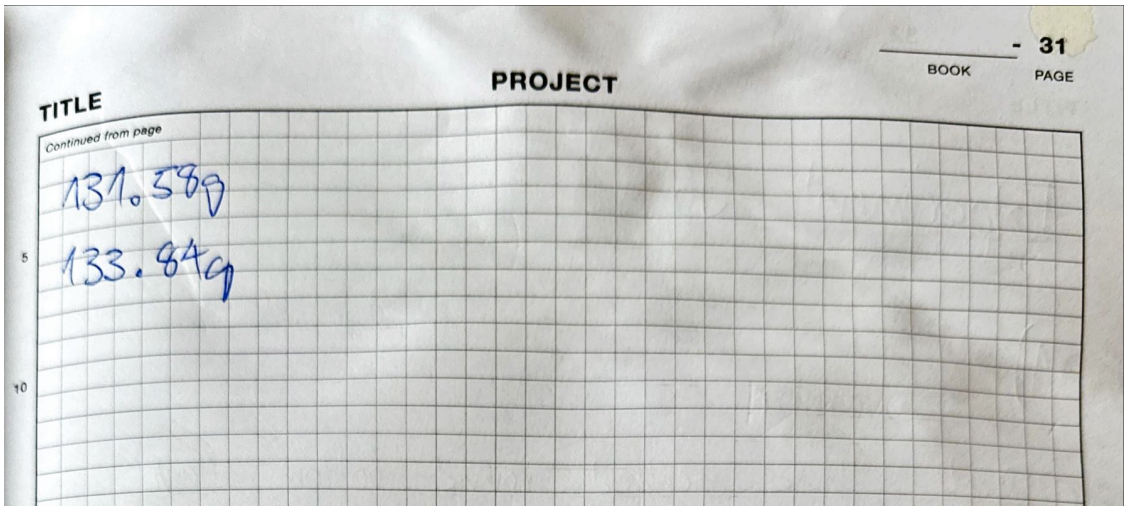
PROPRIETARY INFORMATION


PROJECT
BOOK
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131.58g
133.84g
5
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