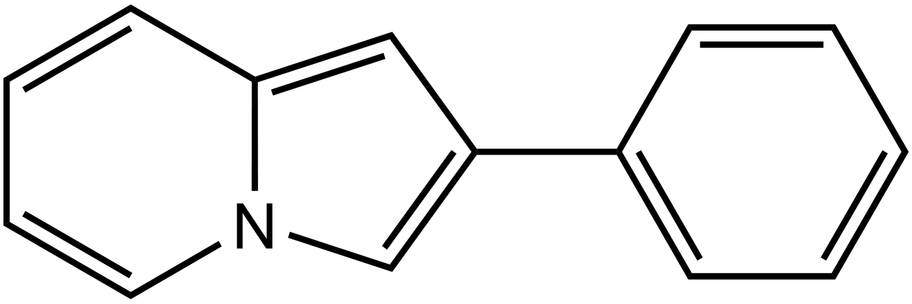
Laboratory Course OACP I
Spring Semester 2024

The Synthesis of 2-Phenylindolizine by Heterocycle Chemistry



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Abstract The objective of this experiment is to first synthesize pyridinium salt and then synthesize 2-phenylindolizine. Then the yield of the product should be determined. The synthesis is performed through the reaction of 2-methylpyridine with 2-bromoacetophenone what yields pyridinium salt. Then the pyridinium salt and sodium hydrogencarbonate react to form 2-phenylindolizine. The substance was purified by recrystallization . The success of the experiment is determined by thin layer chromatography, a melting point analysis and a infrared spectrum measurement, for both substances. The product was a beige solid (0.9771 g, 5.06 mmol) with a yield of 38.48%.

Zürich, April 13, 2024

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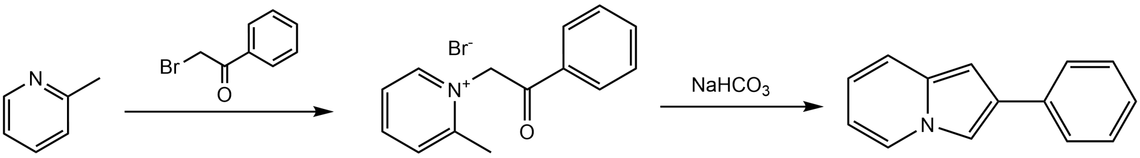
Introduction

2-Phenylindolizine consists of a indolizine ring in the center that has a bicyclic structure with a six-membered benzene ring fused to a five-membered pyridine ring. Attached to this bicyclic system is a benzene ring. Both rings are aromatic, the compound is really stable. At room temperature it appears as a beige solid powder [1].

2-Phenylindolizine is a compound with a molecular framework that has shown a wide range of biological activities [2]. It's part of the indolizine class of compounds which are recognized for their ability to interact with various biological targets, such as phospholipase A2, histamine receptors, and calcium channels. These interactions make them significant in the drug discovery process for various therapeutic areas. Notably, derivatives of indolizine have been explored for their anti-inflammatory and potential anti-cancer effects.

Material Properties

For the synthesis of 2-phenylindolizine pyridinium salt had to be synthesized first. This was achieved by mixing 2-methylpyridine and 2-bromoacetophenone and using toluene as a solvent. Then the pyridinium salt was mixed with sodium hydrogencarbonate and water was used as a solvent. That resulted in the creation of 2-phenylindolizine.



Substance	GHS Hazard Pictogram	H and P Phrases
2-Methylpyridine [3]		H226, H302, H332, H311, H314, H318, H335-P210, P280, P301, P312, P303, P361, P353, P304, P340, P310, P305, P351, P338
Toluene [4]		H225, H315, H361, H336, H373, H304, H412-P202, P210, P273, P301, P310, P303, P361, P353, P331
2-Bromoacetophenone [5]		H314-P260, P280, P303, P361, P353, P304, P340, P310, P305, P351, P338, P363
Sodium bicarbonate [6]	-	-
Pyridinium salt[7]	-	-
2-Phenylindolizine [8]	-	-

Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
2-Methylpyridine	93.13	0.9443	-70	128-129
Toluene	92.14	0.8636	-94.9	110.6
2-Bromoacetophenone	199.03	-	50.5	135
Sodium Bicarbonate	84.01	2.159	-	-
Pyridinium salt	292.17	-	214	-
2-Phenylindolizine	193.24	-	215	-

Safety Assessment

In this experiment several chemical hazards must be considered. Toluene is highly flammable, and its vapors can irritate the eyes. 2-Methylpyridine presents multiple risks, including flammability and health hazards like skin and eye damage. Toluene is also highly flammable and can cause organ damage upon prolonged or repeated exposure. 2-Bromoacetophenone carries risks of severe skin burns and eye damage. Be careful when handling 2-Phenylindolizine due to its potential biological hazards.In general all chemicals should be handled with caution.

For avoiding any health issues that could be caused by the chemicals, gloves and safety googles should be worn in the laboratory. Also the synthesis should be conducted in a fume hood, because of the harmful vapors. 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 sodium hydroxide 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.

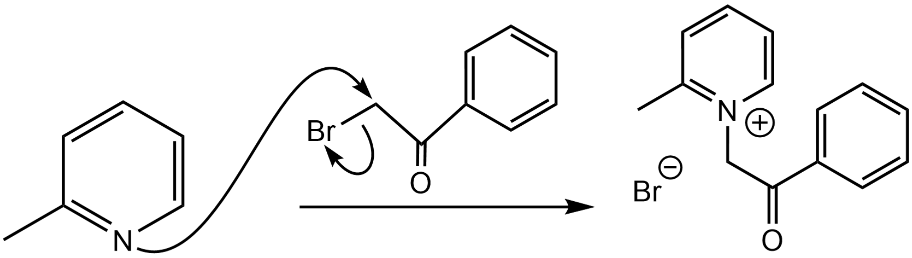
Waste Disposal

Waste that contains sodium bicarbonate should be disposed in the aqueous basic waste. 2-methylpyridine, toluene and 2-bromoacetophenone are organic molecules and should be disposed of in the non chlorinated solvent waste. Pyridinium salt should be dissolved in an organic solvent and also put into the non chlorinated solvent waste.

Reaction Mechanism

Synthesis of the Pyridinium Salt

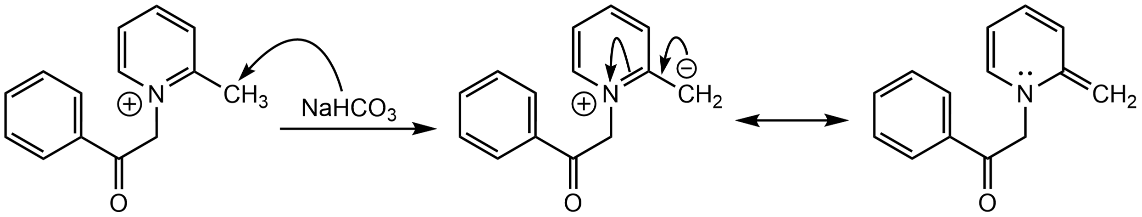
The carbonyl group and bromine atom on the 2-bromoacetophenone molecule enhances the electrophilic character of the alpha carbon. The carbonyl's double bond oxygen draws electron density through polarization, while the bromine, being electronegative, further amplifies the positive charge on the carbon atom next to it. 2-methylpyridine is so nucleophilic because of the electron-rich nitrogen with a lone pair. This lone pair from the nitrogen attacks the electron deficient carbon connected to the bromine. When the nitrogen's lone pair bonds with this carbon, the oxygen of the carbonyl group gains a negative charge, transforming temporarily into an alkoxide anion.



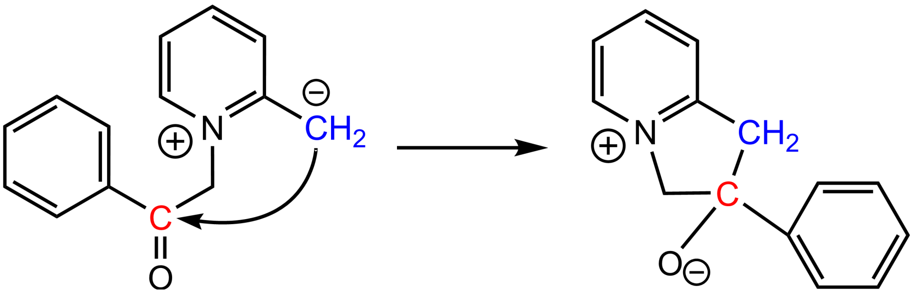
The attack on the electrophilic carbon forms a complex intermediate where the bromine atom remains connected to the carbon and the carbonyl's oxygen has an extra electron what results in a negative charge. After that the bromide ion leaves, which allows the rebuilding of the carbonyl's characteristic double bond. This marks the synthesis of the pyridinium salt.

Synthesis of the 2-Phenylindolizine

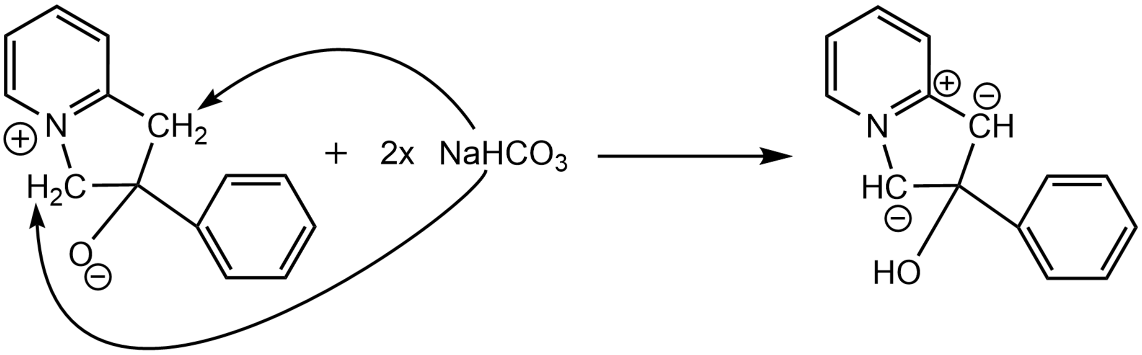
Sodium bicarbonate deprotonates the most acidic proton from the pyridine's methyl group, what results in a carbon with a negative charge making it nucleophilic. The acetophenones carbonyl group still retains its electrophilic character.



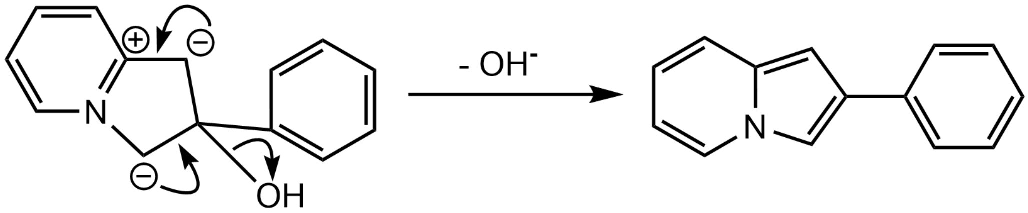
The negatively charged carbon (blue), created by the removal of a proton, initiates an attack on the carbonyl carbon (red), what creates a cyclic structure, in the shape of a cyclopentane. This leads to the formation of an intermediate that features an alkoxide group.



The ring structure that only consists of single bonds gets deprotonated from sodium hydroxide at two sites with relatively acidic hydrogens. That leads to the formation of two carbanions within the ring. Simultaneously, the alkoxide ion receives a proton, converting it back to an alcohol.



The molecule undergoes a sequence of intramolecular electron exchanges, what leads to the regaining of the nitrogen's lone pair and the releasing of an OH^- anion. These adjustments happen stepwise and lead to the regaining of the ring's aromatic character and the production of the final compound 2-phenylindolizine.



Atom Economy

Atom economy refers to the efficiency of a chemical reaction and it is calculated with the equation below. The total atom economy of the reaction is approximately 30%. This reaction is not really atom efficient.

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

$$\text{AE (Pyridinium salt)} = \frac{212.27\,\text{g/mol}}{93.13\,\text{g/mol} + 199.03\,\text{g/mol}} \approx 72.65\%$$

$$\text{AE (Phenylindolizine)} = \frac{193.24\,\text{g/mol}}{212.27\,\text{g/mol} + 3 \times 84.01\,\text{g/mol}} \approx 41.62\%$$

$$\text{AE (total)} = 72.65\% \times 41.62\% \approx 30.23\%$$

Experimental

Procedure

First pyridinium salt gets synthesized by reacting 2-methylpyridine and bromoacetophenone in toluene and extracting the pyridinium salt. 3.8406 g (13.15 mmol) of the white powderlike solid pyridinium salt could be extracted, what is a pretty low yield, normally the yield should be above 7 g.

Then the pyridinium salt is mixed with sodium hydrogencarbonate and water to form the product 2-phenylindolizine. The product was a beige solid (0.9771 g, 5.06 mmol) with a yield of 38.48% if the yield would be calculated with 3.8406 g of pyridinium salt. If the yield would be calculated with 7.01 g pyridinium salt it would be about 21.06%.

Observation

Synthesis of the Pyridinium Salt

The synthesis of pyridinium salt synthesis begins by mixing toluene (7 mL) and 2-methylpyridine (3.6 g, 3.76 mL, 38.65 mmol, 1.00 equiv.) together in a three necked round bottom flask with a reflux condenser and heating them up until boiling is reached. After that 2-bromoacetophenone (7.76 g, 39.03 mmol, 1.01 equiv.) is added. The reaction mixture crystallizes shortly after combination. The mixture is maintained under reflux for an hour to ensure completion of the reaction. After returning to room temperature, the resulting colorless solid is isolated through filtration, rinsed with toluene to remove impurities, and dried with a vacuum and a frit. This results in pyridinium salt (3.8406 g, 13.15 mmol).

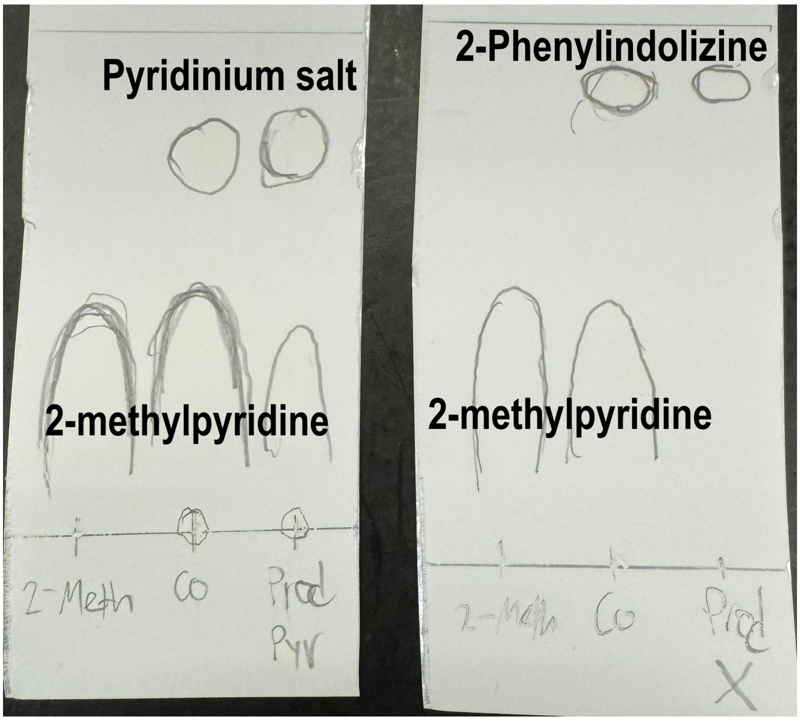
Synthesis of the 2-Phenylindolizine

Water is added to the pyridinium salt and the mixture gets heated. Sodium bicarbonate (7.38 g, 87.48 mmol, 3.66 equiv.) is incrementally introduced to the solution, resulting in a color change to intense yellow. Shortly thereafter, the formation of a yellowish precipitate occurs. The mixture is then stirred at 80 °C to drive the reaction to completion. Once the reaction cools down to room temperature, the product is collected by filtration, washed with water to ensure purity, and dried with a vacuum and a frit. That resulted in the product, 2-phenylindolizine.

Characterisation

Thin Layer Chromatography

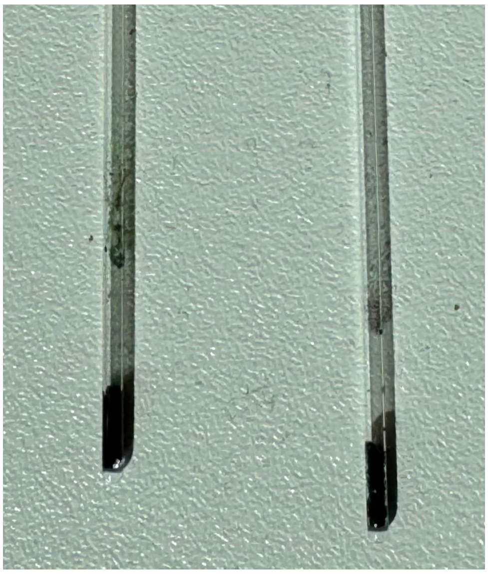
A TLC was conducted for the pyridinium salt and the 2-phenylindolizine. In this TLC, a silicate plate was used and there were three columns. The TLC paper on the left analyzed the pyridinium salt and the TLC paper on the right analyzed the product 2-phenylindolizine. A little bit of the pyridinium salt and the 2-phenylindolizine were dissolved in a little bit of acetone for the TLC. In both TLC papers the first column (2-Meth) had only one educt, the 2-methylpyridine on it. On the left TLC paper, the third column (Prod Pyr) had only pyridinium solution on it. The second column (Co) had both the pyridinium solution and the 2-methylpiridine on it. On the right TLC paper, the third column (Prod X) had only 2-phenylindolizine solution on it. The second column (Co) had both the 2-phenylindolizine solution and the 2-methylpiridine on it. The solvent for both TLC papers consisted of 3 parts n-pentane and 5 parts ethylacetate.



The TLC plate on the left shows that in all three columns there still is the educt 2-methylpiridine. That means the first reaction was not complete, that would explain the low yield of pyridinium salt. The pyridinium salt is charged, what means it is highly polar and stays on the base line. The TLC plate on the right shows that in the third column (Prod X) there is only one spot, that means there is only the product 2-phenylindolizine left. Maybe the washing with toluene for the purification of the pyridinium salt does not remove the 2-methylpiridine but the washing with water for the purification of 2-phenylindolizine does.

Melting Point Analysis

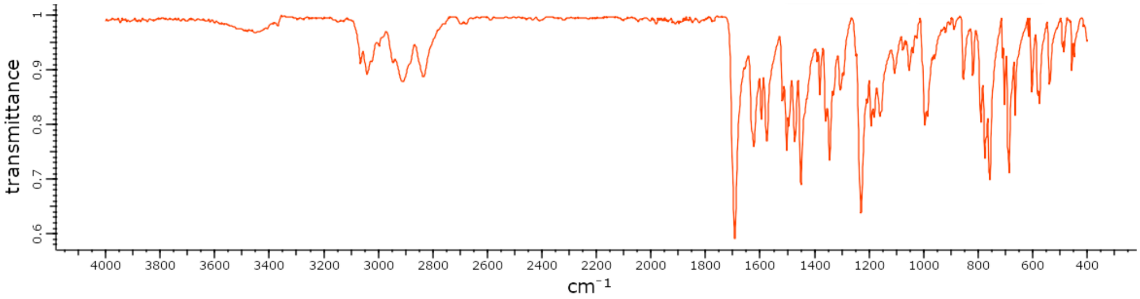
The melting point of the product was analyzed by filling two capillary tubes with the pyridinium salt and the 2-phenylindolizine and using a melting point tester. For this the capillary tubes were inserted in the melting point tester. Both substances didn't melt, they decomposed. That means they turned black instead of melting into a clear liquid. The pyridinium salt completely decomposed at around 214 °C and the 2-phenylindolizine at around 207 °C.



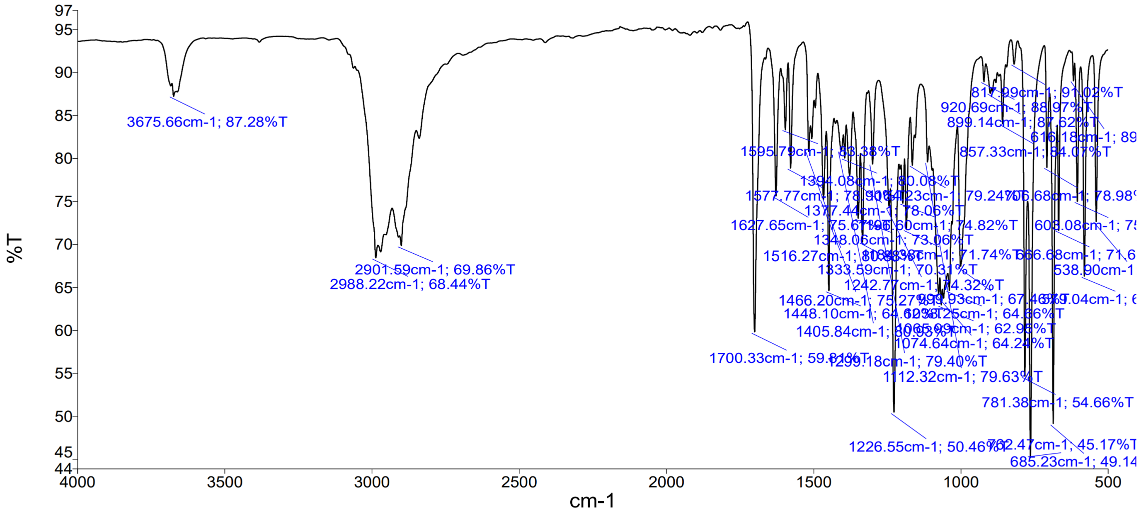
IR Spectrum

IR Spectrum of Pyridinium Salt

This would be the literature values of the infrared spectrum of the pyridinium salt [9].



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



In the spectral analysis, the observed peaks mostly align with those documented in the literature, although they are weaker in intensity [10]. 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 peak at 3675.66 cm^{-1} is a O-H stretch, it does not appear in the IR spectrum from the literature. This could maybe result from the absorption of moisture from the air, pyridinium salts are known for their hygroscopy [11].

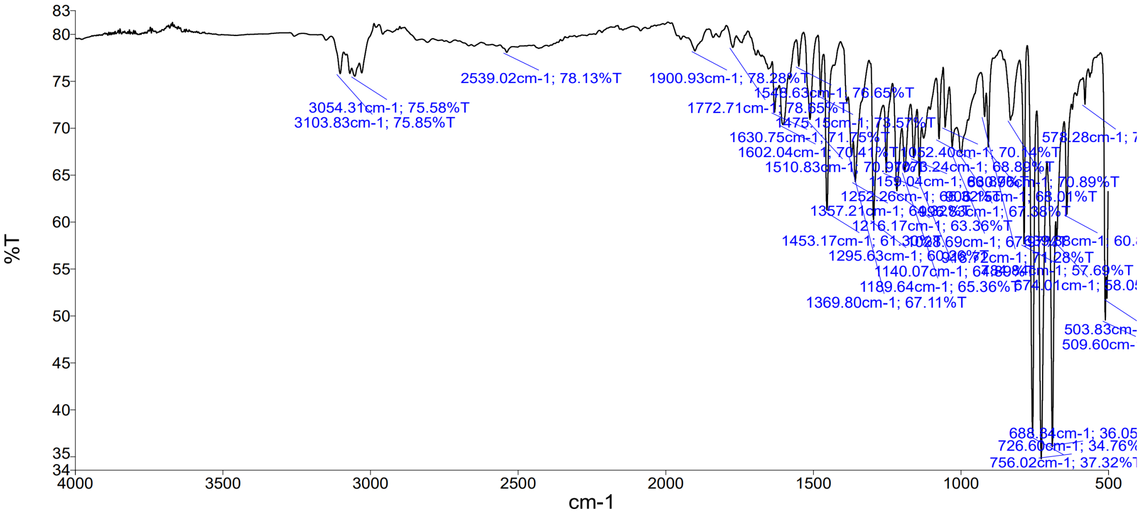
Wavenumber (cm^{-1})	Bond	%T
3675.66	O-H stretch (free hydroxyl group or moisture)	87.28
2988.22	Symmetric CH_2 stretch	68.44
2901.59	Asymmetric CH_2 stretch	69.86
1700.33	Carbonyl C=O stretch	59.81
1595.79	Aromatic C=C stretch	38.38
1577.77	Aromatic C=C stretch	29.30
1458.93	C-H bending (Aromatic)	61.64
1374.08	C-H bending (Alkenes)	73.60
1242.70	C-N stretch	14.32
1226.55	C-N stretch (nitrile) or C-O stretch (ester/ether)	50.46
1176.20	C-H in-plane bend	27.99
899.14	Out-of-plane C-H bend	62.90

IR Spectrum of 2-Phenylindolizine

The available data for 2-phenylindolizine was limited to its wavenumber values for the bond types and stretches, since a picture of its infrared spectrum was not available.

Wavenumber (cm^{-1})	Bond
3101	=C-H Stretching (Aromatic)
3072	=C-H Stretching (Aromatic)
3028	=C-H Stretching (Alkenes)
1514	C=C Stretching (Aromatic)
1454	C-H Bending (CH_2, CH_3)

And this would be the measured infrared spectrum of the synthesized 2-phenylindolizine from the author.



Wavenumber (cm^{-1})	Bond	%T
3103.83	Aromatic C-H Stretch	75.85
3054.31	Aromatic C-H Stretch	75.58
1630.75	C=C Stretch (Aromatic)	71.75
1500-1450	C-H Bending (Aromatic)	Various
756.02	Out-of-Plane C-H Bends	37.32

In the spectral analysis, the observed peaks align more or less with the literature values. The literature for that compound is not that numerous, maybe this could explain the deviations.

References

[1] Fisher Scientific . Safety data sheet: 2-phenylindolizine. `https://www.fishersci.com/store/msds?partNumber=AC276530010&productDescription=2-PHENYLINDOLIZINE%2C+99%2B%25+1GR&vendorId=VN00032119&countryCode=US&language=en`. 2024. Accessed March 22, 2024.

[2] KN, Al-Attraqchi OHA, Tratrat C, Nayak SK, Morsy MA, Aldhubiab BE, Attimarad M, Nair AB, Sreeharsha N, Venugopala R, et al. Novel series of methyl 3-(substituted benzoyl)-7-substituted-2-phenylindolizine-1-carboxylates as promising anti-inflammatory agents: Molecular modeling studies. biomolecules. `https://doi.org/10.3390/biom9110661`, 2019. Accessed March 22, 2024.

[3] Sigma-Aldrich. Safety data sheet for 2-methylpyridine, catalog number 109835. `https://www.sigmaaldrich.com/CH/en/sds/ALDRICH/109835?userType=anonymous`, 2024. Accessed March 22, 2024.

[4] Sigma-Aldrich. Safety data sheet for toluene, catalog number 244511. `https://www.sigmaaldrich.com/CH/en/sds/SIAL/244511?userType=anonymous`, 2024. Accessed March 22, 2024.

[5] Sigma-Aldrich. Safety data sheet for 2-bromoacetophenone, catalog number 115835. `https://www.sigmaaldrich.com/CH/en/sds/ALDRICH/115835?userType=anonymous`, 2024. Accessed March 22, 2024.

[6] Sigma-Aldrich. Safety data sheet for sodium bicarbonate, catalog number 792519. `https://www.sigmaaldrich.com/CH/en/sds/SIGALD/792519?userType=anonymous`. 2024. Accessed March 22, 2024.

[7] National Center for Biotechnology Information. Pubchem compound summary for cid 12316824, 2-methyl-1-(2-oxo-2-phenylethyl)pyridinium bromide. `https://pubchem.ncbi.nlm.nih.gov/compound/2-Methyl-1-_2-oxo-2-phenylethyl_pyridinium-bromide`, 2024. Accessed March 22, 2024.

[8] American Chemical Society. Substance detail for indolizine 2-phenyl-. SciFinder®, 2024. Accessed March 22, 2024.

[9] Wiley-VCH Verlag GmbH Co. KGaA. Ir spectrum and properties of 2-methyl-n-phenacylpyridinium bromide. In IR - Industrial Chemicals, Pure Organic Compounds - Wiley, 2024. Accessed on KnowItAllAnywhere on March 19, 2024.

[10] 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.

[11] Subbiah Sowmiah, José M. S. S. Esperança, Luís P. N. Rebelo, and Carlos A. M. Afonso. Pyridinium salts: from synthesis to reactivity and applications. *Organic Chemistry Frontiers*, 2018(5):453–493, 2018.

Appendix

List of H and P Phrases

- **H226**: Flammable liquid and vapor.
- **H302**: Harmful if swallowed.
- **H332**: Harmful if inhaled.
- **H311**: Toxic in contact with skin.
- **H314**: Causes severe skin burns and eye damage.
- **H318**: Causes serious eye damage.
- **H335**: May cause respiratory irritation.
- **P210**: Keep away from heat, sparks, open flames, hot surfaces. — No smoking.
- **P280**: Wear protective gloves, protective clothing, eye protection, face protection.
- **P301**: IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.
- **P312**: Call a POISON CENTER or doctor/physician if you feel unwell.
- **P303**: IF ON SKIN (or hair): Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower.
- **P361**: Remove/Take off immediately all contaminated clothing.
- **P353**: Rinse skin with water/shower.
- **P304**: IF INHALED: Remove to fresh air and keep at rest in a position comfortable for breathing.
- **P340**: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
- **P310**: Immediately call a POISON CENTER or doctor/physician.
- **P305**: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
- **P351**: Rinse continuously with water for several minutes.
- **P338**: Remove contact lenses, if present and easy to do. Continue rinsing.
- **P331**: Do NOT induce vomiting.
- **P260**: Do not breathe dust/fume/gas/mist/vapors/spray.
- **P363**: Wash contaminated clothing before reuse.

Table from the Experiment Manual

Reactant	**MW**	**Equiv**	**Moles** mmol	**Mass** g	**Volume** mL	**Purity**
2-Methylpyridine	93.15	1.00	38.65	3.60 g	3.76	-
Bromoacetophenone	199.03	1.01	39.03	7.76	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP** °C	
Pyridinium salt	292.17				214	

Note: 2-Methylpyridine = 2-Picoline

Reactant	**MW**	**Equiv**	**Moles** mmol	**Mass** g	**Volume**	**Purity**
Pyridinium salt	292.17	1.00	24 mmol	7.01	-	-
Sodium bicarbonate	84.01	3.66	87.48	7.38	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP** °C	
2-Phenylindolizine	193.24				215	

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	-

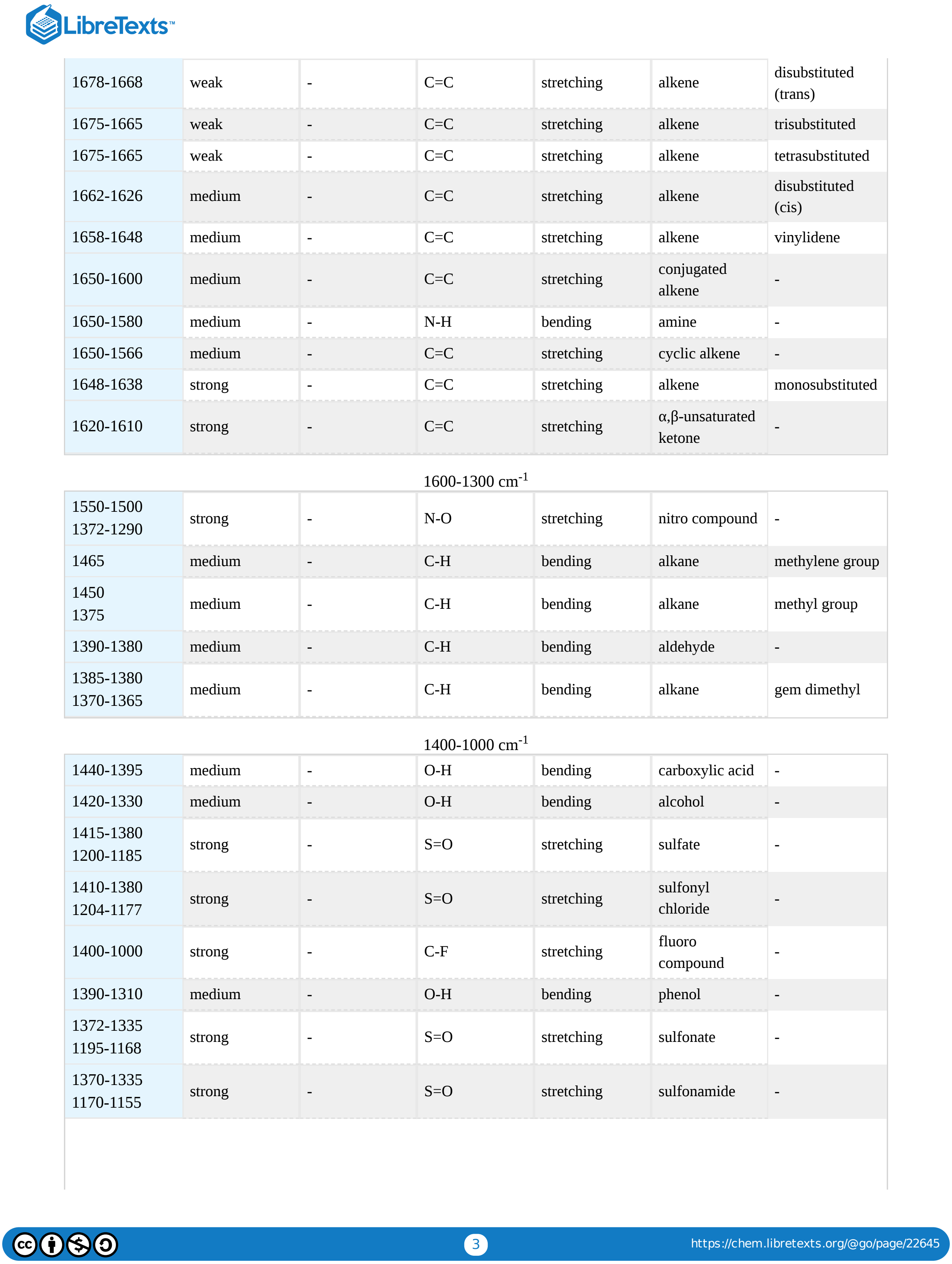
2000-1650 cm^{-1}

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	-

3 https://chem.libretexts.org/@go/page/22645

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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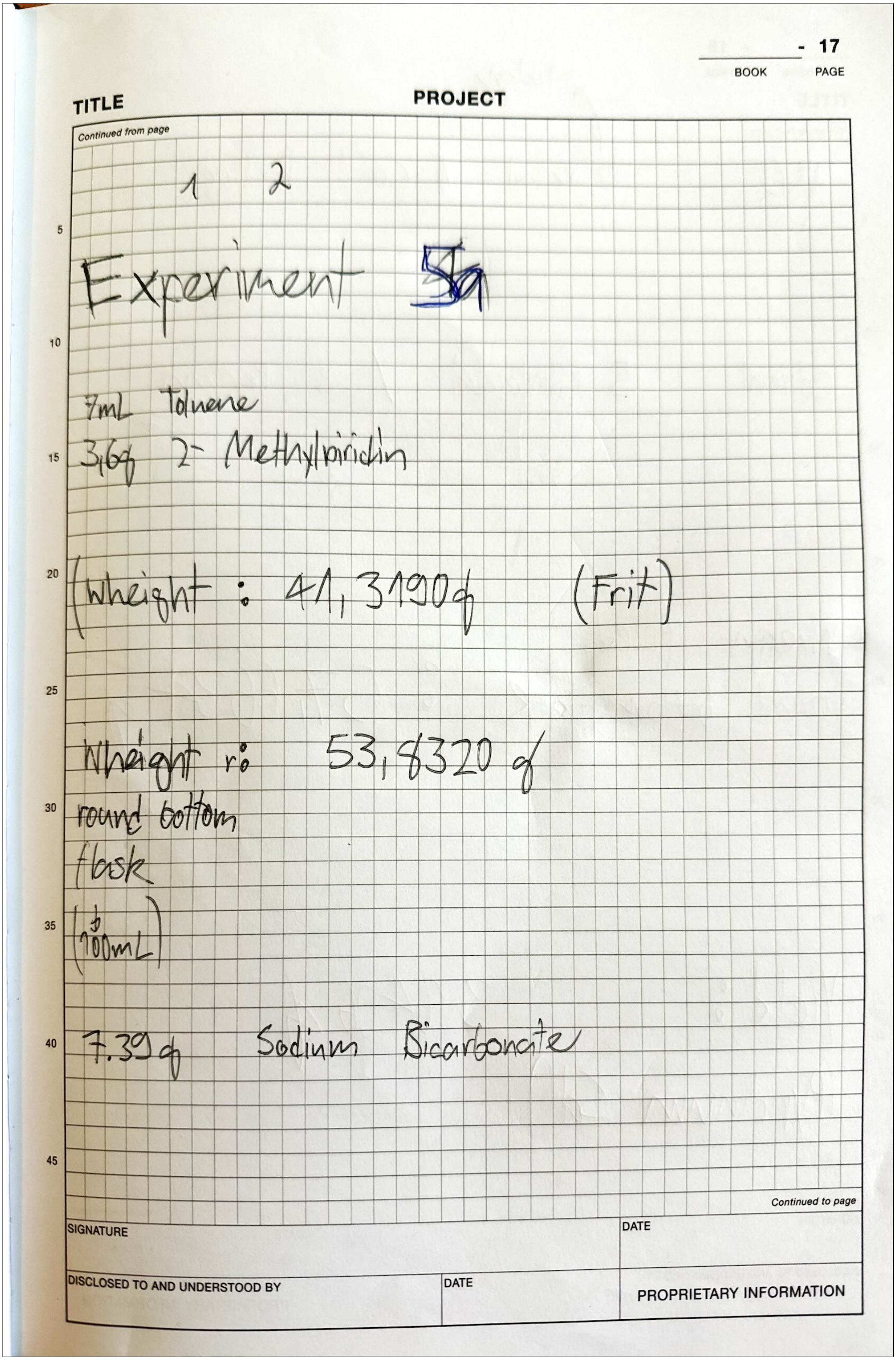

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1 2

Experiment 5a

7mL Toluene
3,6g 2- Methylpiridin

(Wheight : 41,3190g (Frit)

Wheight r: 53,8320 g
round bottom
flask
($\overset{5}{1}$00mL)

7.39g Sodium Bicarbonate

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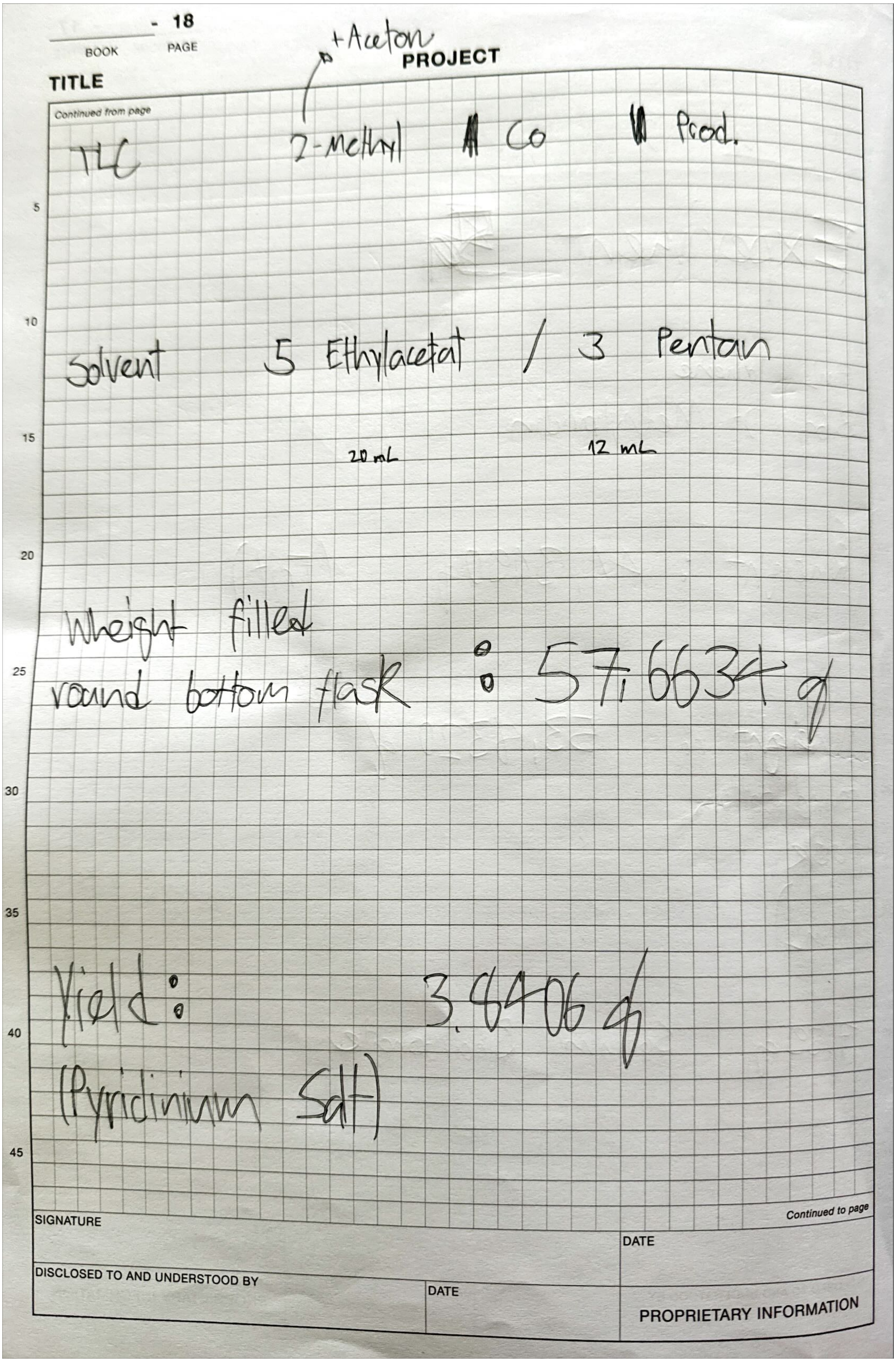

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TLC

+Aceton

2-Methyl Co Prod.

Solvent 5 Ethylacetat / 3 Pentan

20 mL 12 mL

Wheight filled
round bottom flask : 57,6634 g

Yield : 3.4406 g
(Pyridinium Salt)

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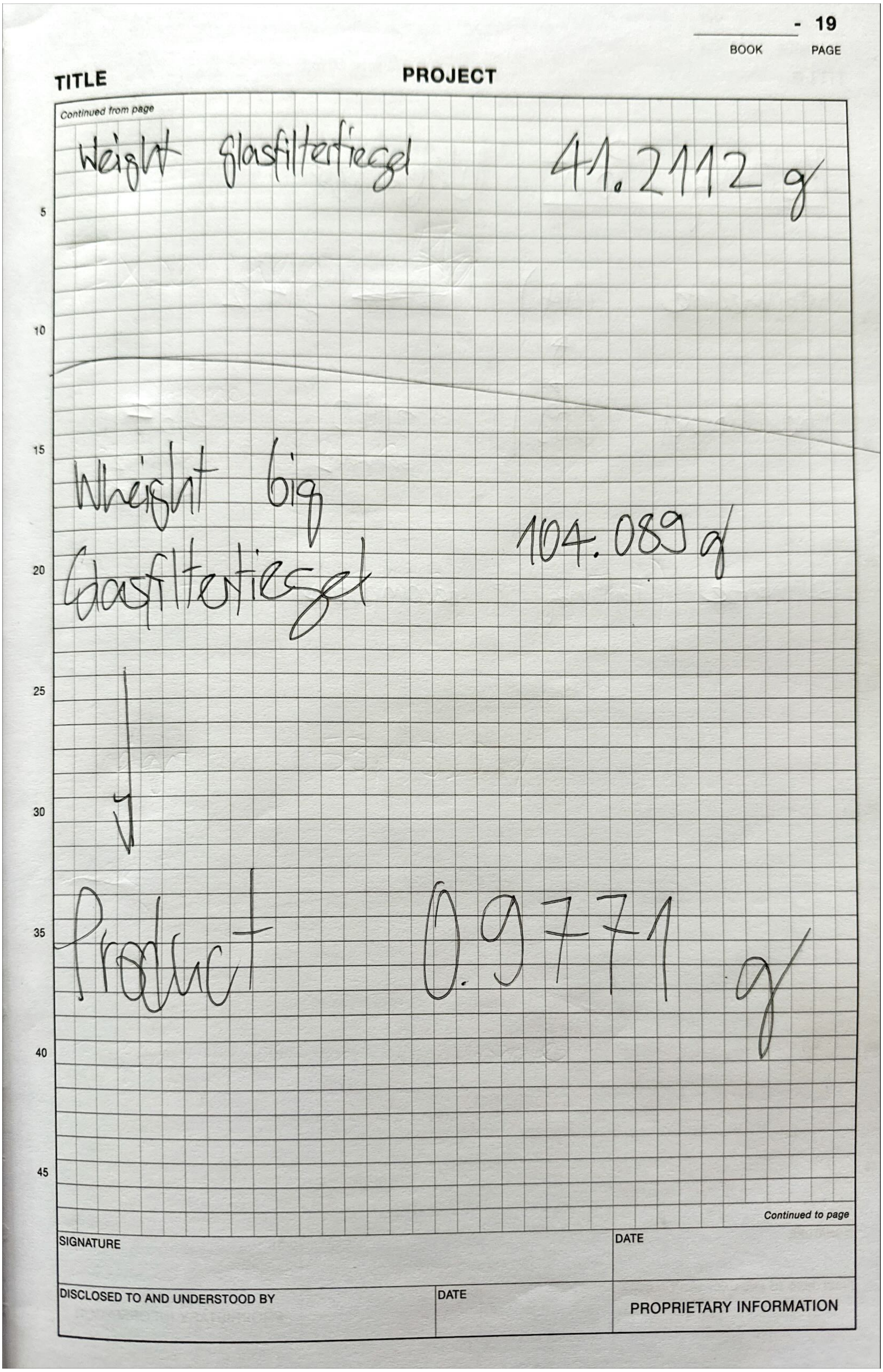

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Weight glasfiltertiegel 41.2112 g

Wheight big Glasfiltertiegel 104.089 g

↓

Product 0.9771 g

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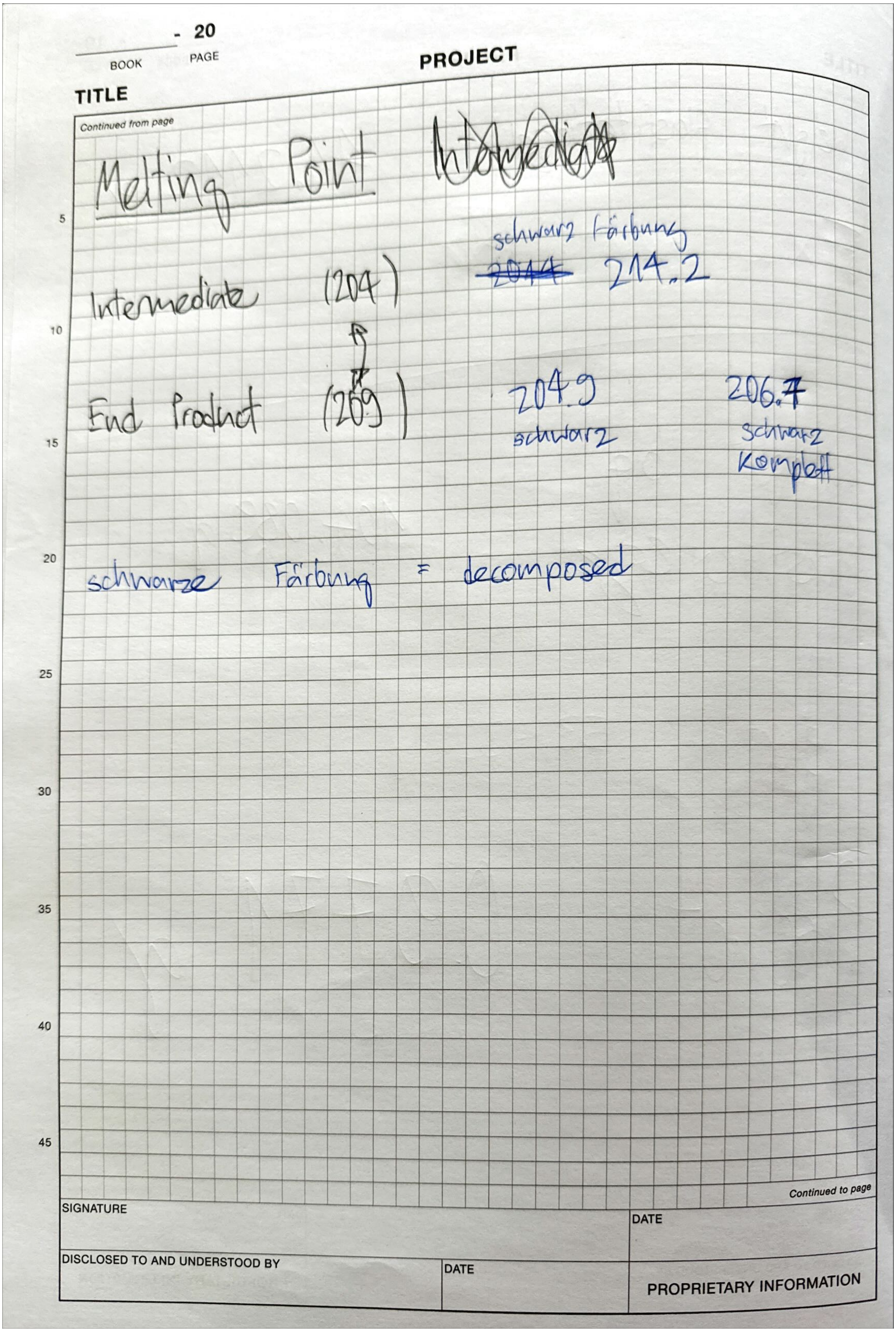

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Melting Point ~~Intermediate~~

Intermediate (204) schwarz Färbung ~~204.4~~ 214.2

End Product (209) 204.9 schwarz 206.7 schwarz komplett

schwarze Färbung = decomposed

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