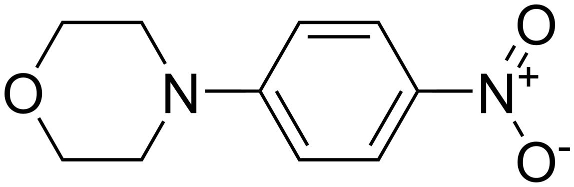
Laboratory Course OACP I
Spring Semester 2024

The Synthesis of 4-(4-Nitrophenyl) morpholine by Nucleophilic Aromatic Substitution



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Abstract The objective of this experiment is to synthesize 4-(4-Nitrophenyl)morpholine and analyze the yield of the product. The synthesis is performed by the nucleophilic aromatic substitution through mixing 4-fluoronitrobenzene and morpholine and addition of sodium carbonate. The substance was purified by recrystallization. The success of the experiment is determined by thin layer chromatography, a melting point analysis and an infrared spectrum measurement. The yield of the synthesis is 10.57%.

Zürich, May 14, 2024

V. Loganathan
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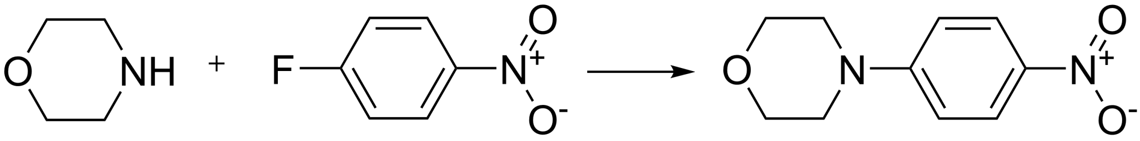
Introduction

4-(4-Nitrophenyl)morpholine features a tetrahydrofuran ring, a five-membered cyclic ether, that serves as a starting point for the compound. This ring is fused to a phenyl group, which is part of the broader family of aromatic hydrocarbons, known for their stability due to their aromaticity. Attached to the phenyl group is a nitro group, a nitrogen atom connected to two oxygen atoms. The nitrogen is connected with a double and a single bond to the oxygen atoms, resulting in a resonance-stabilized structure where the nitrogen bears a positive charge while one oxygen carries a negative charge. This setup is indicative of the nitro group's ability to draw electrons towards itself, classifying it as an electron-withdrawing group. Furthermore, the nitrogen's direct bond to the aromatic ring categorizes it as an anilide linkage, which is a specific type of amine linkage where the nitrogen atom is integrated into the aromatic system. It appears as crystalline solid powder at room temperature with a white to slightly yellowish colour [1].

This compound is involved in synthesizing momelotinib, a treatment for myelofibrosis. Its wide-ranging potential makes it valuable across various sectors and scientific studies. However, detailed information into its extensive uses are not readily available [2].

Material Properties

For the synthesis of 4-(4-Nitrophenyl) morpholine nucleophilic aromatic substitution was used. 4-fluoronitrobenzene and morpholine reacted together to form the product.



Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
4-Fluoronitrobenzene [3]	141.10	1.3300	27	206-207
Morpholine [4]	87.12	1.0007	-4.9	128.9
Sodium carbonate [5]	105.98	2.53	851	-
4-(4-Nitrophenyl)morpholine [6]	208.21	1.265±0.06 (predicted)	152	386.2±37.0 (Predicted)

Substance	GHS Hazard Pictogram	H and P Phrases
4-Fluoronitrobenzene [3]		H301, H302, H312, H317, H319, H331, H373, H412, P260, P261, P264, P270, P271, P272, P273, P280, P301+P316, P301+P317, P302+P352, P304+P340, P305+P351+P338, P316, P319, P321, P330, P333+P317, P337+P317, P362+P364, P403+P233, P405, P501
Morpholine [4]		H226, H302, H331, H311, H314, H318, H361-P210, P280, P301, P312, P303, P361, P353, P304, P340, P310, P305, P351, P338
Sodium carbonate [5]		H319-P264, P280, P305, P351, P338, P337, P313
4-(4-Nitrophenyl)morpholine [7]		H315, H319, H335 P261, P264, P271, P280, P302+P352, P304+P340, P305+P351+P338, P321, P332+P317, P337+P313, P362+P364, P403+P233, P405, P501

Safety Assessment

In the lab, each chemical demands specific safety measures. 4-Fluoronitrobenzene can cause serious health risks. It is highly toxic if inhaled, swallowed, or absorbed through the skin. It can cause respiratory tract irritation, skin burns, and eye damage. Morpholine can cause skin and respiratory tract irritation. Prolonged exposure might lead to more serious health issues. Sodium carbonate is generally considered safe in small quantities however, it can cause eye and skin irritation. 4-(4-Nitrophenyl)morpholine like the other nitro compound, can be hazardous. It can be acutely toxic and cause skin, eye, and respiratory tract irritation. This chemical should be handled with extreme care.

The entire reaction should be conducted in a well ventilated fume hood to avoid exposure to any harmful vapors, particularly from morpholine. The reaction can be exothermic, so adding the morpholine slowly to control the reaction rate is important. The internal temperature should be around 0°C.

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.

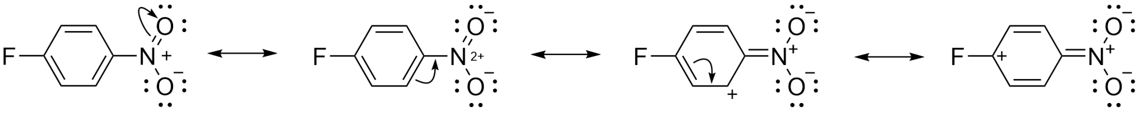
For avoiding any health issues that could be caused by the chemicals, gloves and safety googles should be worn in the laboratory. In a case of an accident the emergency number should be called. With the internal ETH-phones it would be 888, on personal mobile phones the number would be 044 342 11 88.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

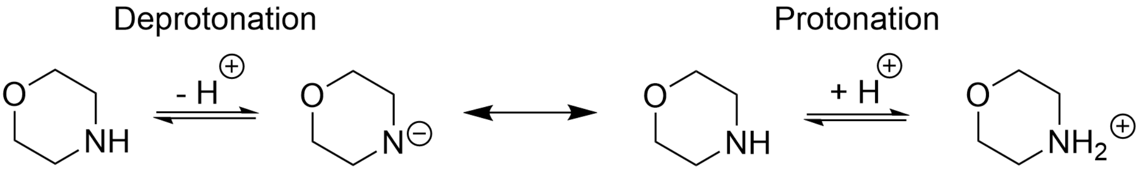
The 4-Fluoronitrobenzene, Morpholine and 4-(4-Nitrophenyl)morpholine should be disposed of into the non chlorinated organic waste. The Sodium carbonate should be disposed of in the solid waste.

Reaction Mechanism

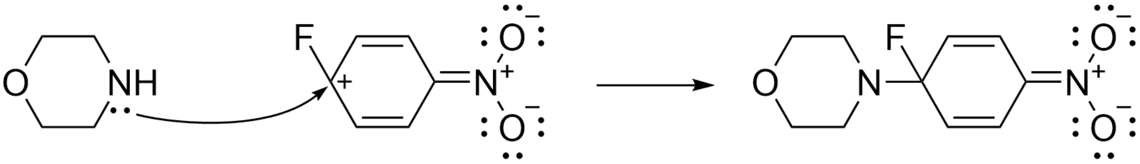
4-Fluoronitrobenzene is always in a superposition of these four resonance structures. They contribute to the stability of 4-Fluoronitrobenzene.



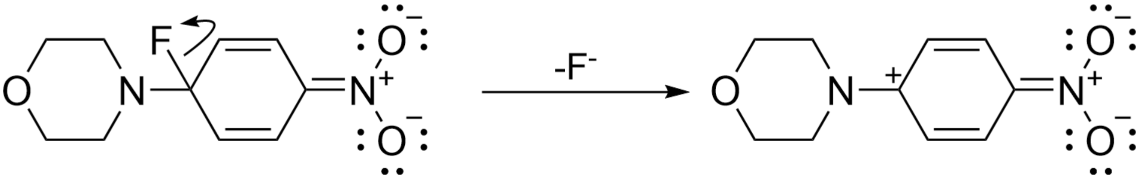
In morpholine's equilibrium it exists in two forms. The equilibrium maintains a balance between these forms: one with a positive charge, known as the protonated form, and the other with a negative charge, the deprotonated form, also called cyclopentadienyl anion.



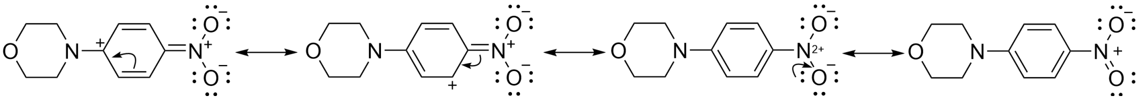
The nucleophile morpholine attacks the electrophilic carbon next to the fluorine of the 4-fluoronitrobenzene. That leads to a bond between the morpholine and the 4-fluoronitrobenzene.



The fluorine leaves as a fluoride anion. This results in the formation of the product 4-(4-Nitrophenyl) morpholine.



4-(4-Nitrophenyl)morpholine has four resonance structures. The last resonance structure is the most stable one because the aromacity of the benzene ring is restored and charge separation is minimized.



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 94%. This reaction is extremely atom efficient. The molar mass of the product is 208.21 g/mol and the cumulative molar masses of the educts are 228.22 g/mol. So a substance with 20 g/mol leaves this reaction. This could be the fluorine and the hydrogen that leave.

$$\mathrm{AE} = \frac{M_{\mathrm{Product}}}{M_{\mathrm{Reagents}}} = \frac{208.21\ \mathrm{g/mol}}{141.10\ \mathrm{g/mol} + 87.12\ \mathrm{g/mol}} \approx 93.70\%$$

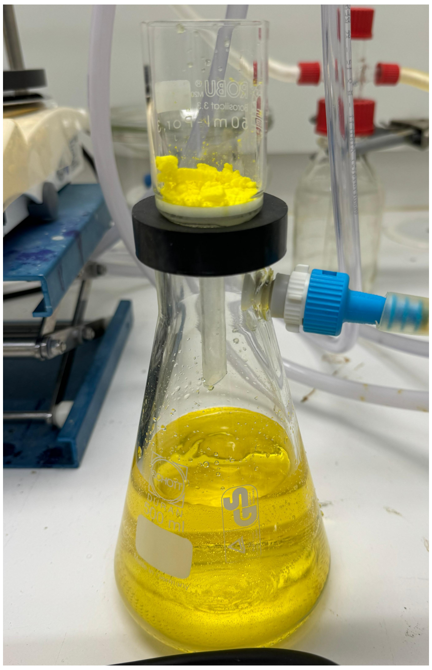
Experimental

Procedure

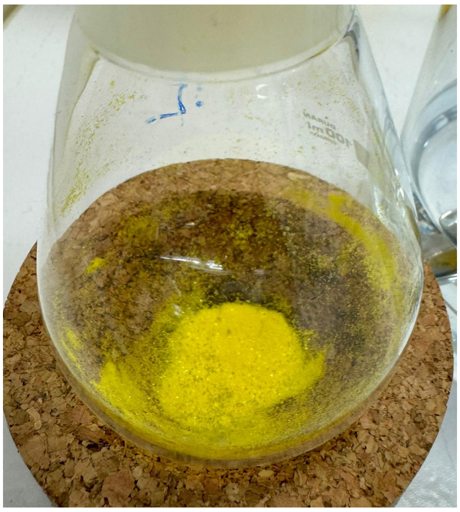
The synthesis is performed by the nucleophilic aromatic substitution. 4-fluoronitrobenzene and morpholine are reacted at 0 °C. Then sodium carbonate is washed and the resulting yellow solid is filtered, washed with water and dried using a frit and a vacuum. The product was a yellow lumpy powderous solid (0.11 g, 0.53 mmol, 0.11 equiv.).

Observation

4-Fluoronitrobenzene (7.1 g, 5 mmol, 1.00 equiv.) was filled into a 100 mL three necked round bottom flask with a thermometer and a magnetic stirr bar. The flask was cooled with an icebath to around 0°C. Then morpholine (2.16 mL, 25 mmol, 5.00 equiv.) was added slowly with a 5 mL syringe. The mixture turned into a clear yellow liquid. The flask was removed from the ice bath and the mixture was stirred for 20 minutes at room temperature. As the temperature slowly went from around 0°C to room temperature, the liquid turned from clear yellow to milky yellow. Then 10 mL of a saturated sodium carbonate solution were added into the mixture. The mixture was filtered with a vacuum and a frit and washed with water. On the frit there was a yellow solid remaining that got put into a round bottom flask.



This solid was then dissolved in boiling hot ethanol for the recrystallization. The yellow solid dissolved and then the round bottom flask was left to cool down to room temperature. After that it was put into a ice bath to cool down further. The contents of the round bottom flask were put into a frit with a vacuum again and on the frit there remained a yellow solid, lumpy powder, this is the product 4-(4-nitrophenyl)morpholine.

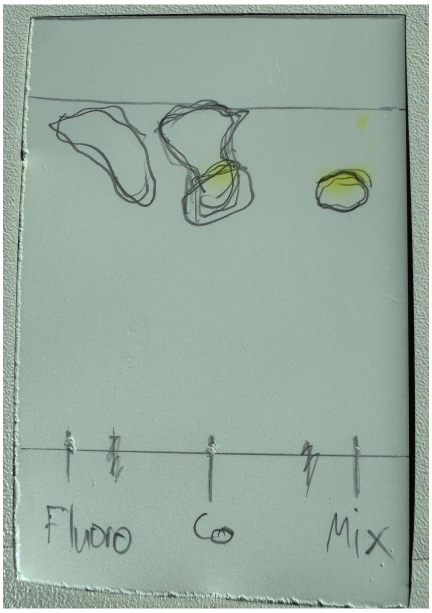


The weight of the product is 0.32 g. But the product still had some ethanol in it, that bumped up the weight. So the round bottom flask with the product was put into a rotarotary evaporator to remove the ethanol. After 20 minutes in the rotarotary evaporator the round bottom flask was removed and the weight of the product was measured again. The weight was 0.11 g (0.53 mmol, 0.11 equiv.), that would be a yield of around 10.57%.

Characterisation

Thin Layer Chromatography

A little bit of the solid yellow product and 4-fluoronitrobenzene was dissolved in acetone. In this TLC a silicate plate was used and there were three columns. The first column (Fluoro) had the the solution with the educt 4-fluoronitrobenzene on it, the third column (Mix) had the solution with the product 4-(4-nitrophenyl) morpholine on it, and the second column (Co) had both solutions on it. The solvent for the TLC was pure ethylacetate.



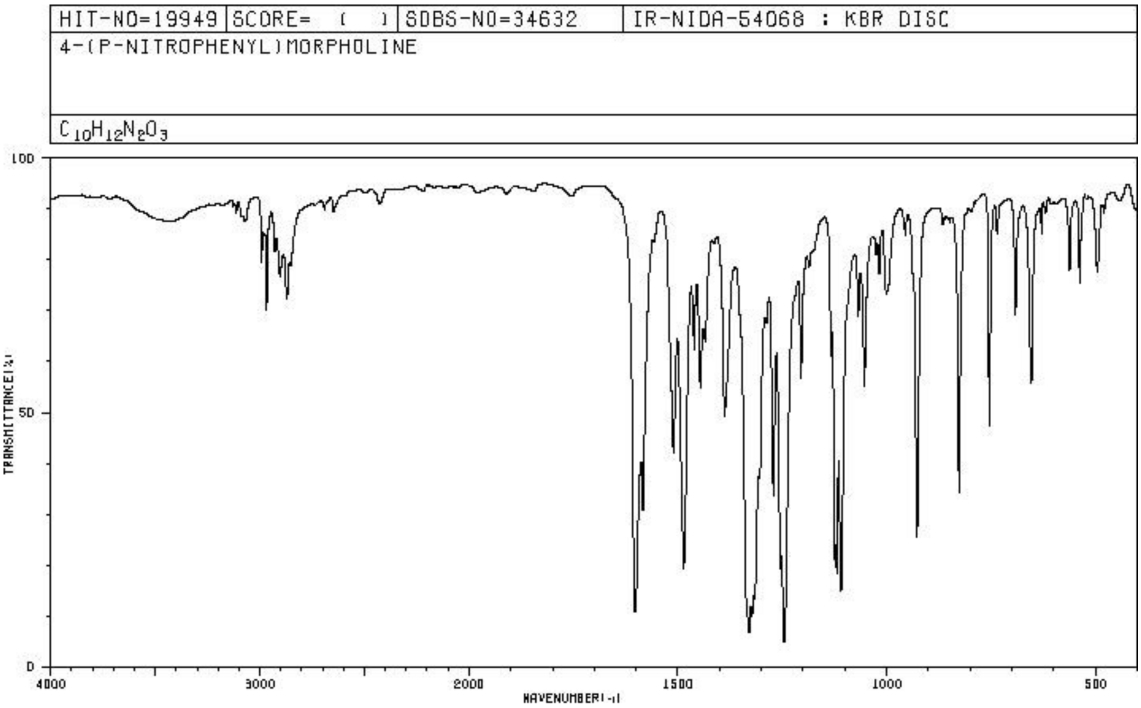
The educt 4-fluoronitrobenzene rose up higher than the product 4-(4-nitrophenyl)morpholine. The polarity of 4-fluoronitrobenzene may be higher than of the product, but there are also other factors in the rise of a TLC plate.

Melting Point Analysis

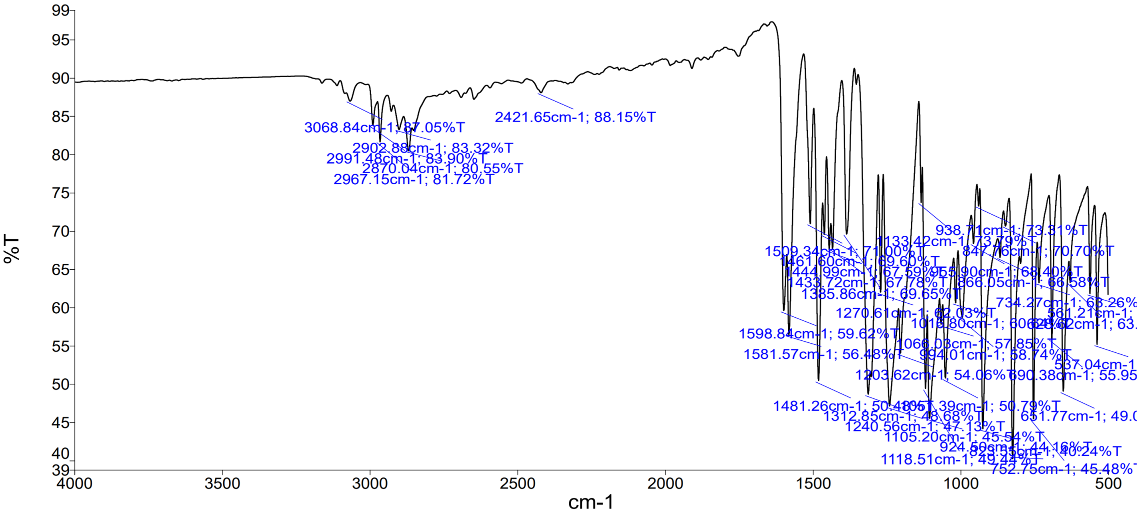
The melting point of the product was analyzed by filling a capillary tube with the yellow solid and using a melting point tester. For this the capillary tube was inserted in the melting point tester. At around 150 °C the substance melted into a clear liquid. That would be only slightly lower than the literature value of 152 °C. This could maybe be explained with the fact that there may be traces of the educt morpholine left, that has a melting point of -4.9 °C, what could have lowered the melting point of the product a little bit.

IR Spectrum

This would be the literature values of the infrared spectrum of 4-(4-nitrophenyl) morpholine.



And this would be the measured infrared spectrum of the synthesized 4-(4-nitrophenyl) morpholine from the author.



In the spectral analysis, the observed peaks mostly align with those documented in the literature, although they are weaker in intensity [8]. 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
3068.84	Aromatic C-H stretch	87.05%
2991.48	Aliphatic C-H stretch	83.90%
2967.15	Aliphatic C-H stretch	81.72%
2902.88	Aliphatic C-H stretch	83.32%
2870.04	Aliphatic C-H stretch	80.55%
1598.84	C=C stretch (Aromatic) or NO_2 asymmetric stretch	59.62%
1581.57	C=C stretch (Aromatic)	56.48%
1509.34	N-O asymmetric stretch (Nitro compound)	71.00%
1481.26	C-H bending (Aliphatic)	50.48%
1461.60	C-H bending (Aliphatic)	69.60%
1444.99	C-H bending (Aliphatic)	67.59%
1433.72	C-H bending (Aliphatic)	67.78%
1385.86	C-H bending (Aliphatic)	69.65%
1312.85	C-N stretching (Amines)	48.68%
1270.61	C-N stretching (Amines)	62.03%
1240.56	C-O or C-N stretching (Ethers, alcohols, amines)	47.13%
1203.62	C-N stretching (Amines)	54.06%
1133.42	C-N stretching (Amines)	73.79%
1118.51	C-N stretching (Amines)	49.44%
1105.20	C-N stretching (Amines)	45.54%
1066.03	C-N stretching (Amines)	57.85%
1016.80	C-O or C-N stretching (Ethers, alcohols, amines)	60.64%
994.01	C-O or C-N stretching (Ethers, alcohols, amines)	58.74%
866.05	C-H out-of-plane bend (Aromatic)	66.58%
847.76	C-H out-of-plane bend (Aromatic)	70.70%
823.55	C-H out-of-plane bend (Aromatic)	40.24%
752.75	C-H out-of-plane bend (Aromatic)	45.48%
690.38	C-H out-of-plane bend (Aromatic)	55.95%
651.77	C-H out-of-plane bend (Aromatic)	49.08%
628.62	C-H out-of-plane bend (Aromatic)	63.32%

References

[1] ChemSynthesis. 4-(4-nitrophenyl)morpholine. `https://www.chemsynthesis.com/base/chemical-structure-29057.html`.

[2] ChemicalBook. 4-(4-nitrophenyl)morpholine - properties and chemical information. `https://www.chemicalbook.com/ChemicalProductProperty_EN_CB7667570.htm`, 2023. CAS No. 10389-51-2, Accessed April 09, 2024.

[3] National Center for Biotechnology Information. Pubchem compound summary for cid 9590, 1-fluoro-4-nitrobenzene. `https://pubchem.ncbi.nlm.nih.gov/compound/1-Fluoro-4-nitrobenzene`, 2024. Retrieved April 10, 2024.

[4] Sigma-Aldrich. Safety data sheet for morpholine, catalog number 252360. `https://www.sigmaaldrich.com/CH/en/sds/SIAL/252360?userType=anonymous`, 2024. Accessed April 10, 2024.

[5] Sigma-Aldrich. Safety data sheet for sodium carbonate, catalog number 223530. `https://www.sigmaaldrich.com/CH/en/sds/SIGALD/223530?userType=anonymous`, 2024. Accessed April 10, 2024.

[6] ChemicalBook. 4-(4-nitrophenyl)morpholine - properties and chemical information. `https://www.chemicalbook.com/ChemicalProductProperty_EN_CB7667570.htm`, 2023. CAS No. 10389-51-2, Accessed April 23, 2023.

[7] National Center for Biotechnology Information. Pubchem compound summary for cid 25222, 4-(4-nitrophenyl)morpholine. `https://pubchem.ncbi.nlm.nih.gov/compound/4-_4-Nitrophenyl_morpholine`, 2024. Retrieved April 10, 2024.

[8] 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

- **H225**: Highly flammable liquid and vapor.
- **H314**: Causes severe skin burns and eye damage.
- **H315**: Causes skin irritation.
- **H318**: Causes serious eye damage.
- **H319**: Causes serious eye irritation.
- **H335**: May cause respiratory irritation.
- **H336**: May cause drowsiness or dizziness.
- **H351**: Suspected of causing cancer.
- **H290**: May be corrosive to metals.
- **P210**: Keep away from heat, sparks, open flames, hot surfaces. — No smoking.
- **P233**: Keep container tightly closed.
- **P234**: Keep only in original container.
- **P240**: Ground/bond container and receiving equipment.
- **P280**: Wear protective gloves, protective clothing, eye protection, face protection.
- **P303+P361+P353**: IF ON SKIN (or hair): Remove/Take off immediately all contaminated clothing. Rinse skin with water/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 if you feel unwell: Get medical advice/attention.
- **P202**: Do not handle until all safety precautions have been read and understood.
- **P261**: Avoid breathing dust/fume/gas/mist/vapors/spray.
- **P264**: Wash hands thoroughly after handling.
- **P302+P352**: IF ON SKIN: Wash with plenty of water.
- **P304+P340**: IF INHALED: Remove person to fresh air and keep 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.
- **P363**: Wash contaminated clothing before reuse.

Table from the Experiment Manual

Reactant	**MW** (g/mol)	**Equiv**	**Moles** (mmol)	**Mass** (g)	**Volume** (mL)	**Purity**
4-Fluoronitrobenzene	141,10	1.00	5 mmol	0,71	-	-
Morpholine	87,12	5.00	25	2,18	2,16	-
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	
4-(4-Nitrophenyl)morpholine	208,21				386 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	-

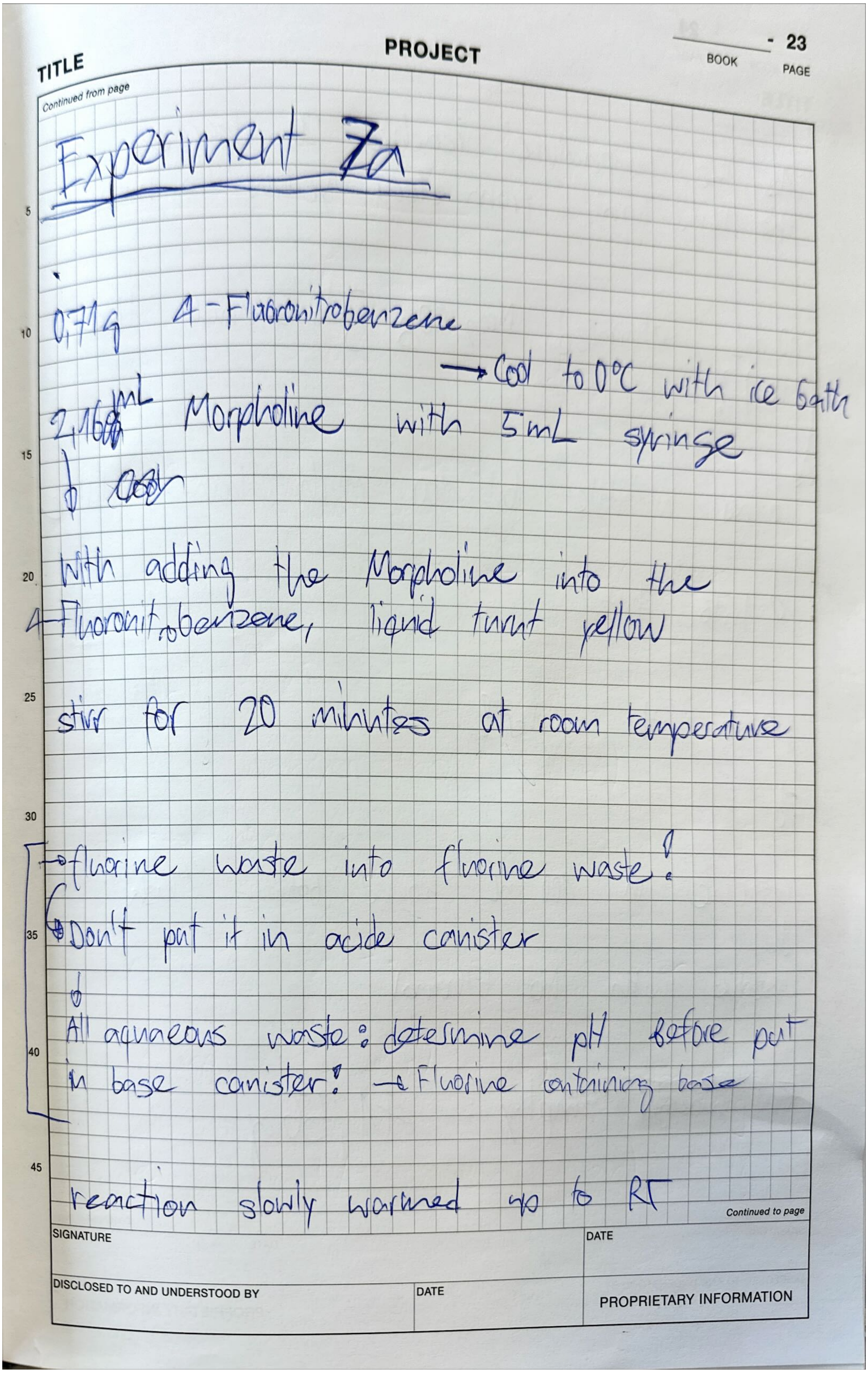
900-700 cm^{-1}

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	-

Contributors and Attributions

- OChemOnline



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Experiment 7a

0,71g 4-Fluoronitrobenzene

→ Cool to 0°C with ice bath

2,16 mL Morpholine with 5mL syringe

↓ ~~cool~~

With adding the Morpholine into the 4-Fluoronitrobenzene, liquid turnt yellow

stir for 20 minutes at room temperature

→ fluorine waste into fluorine waste!

→ Don't put it in acide canister

↓

All aqueous waste: determine pH before put in base canister! → Fluorine containing base

reaction slowly warmed up to RT

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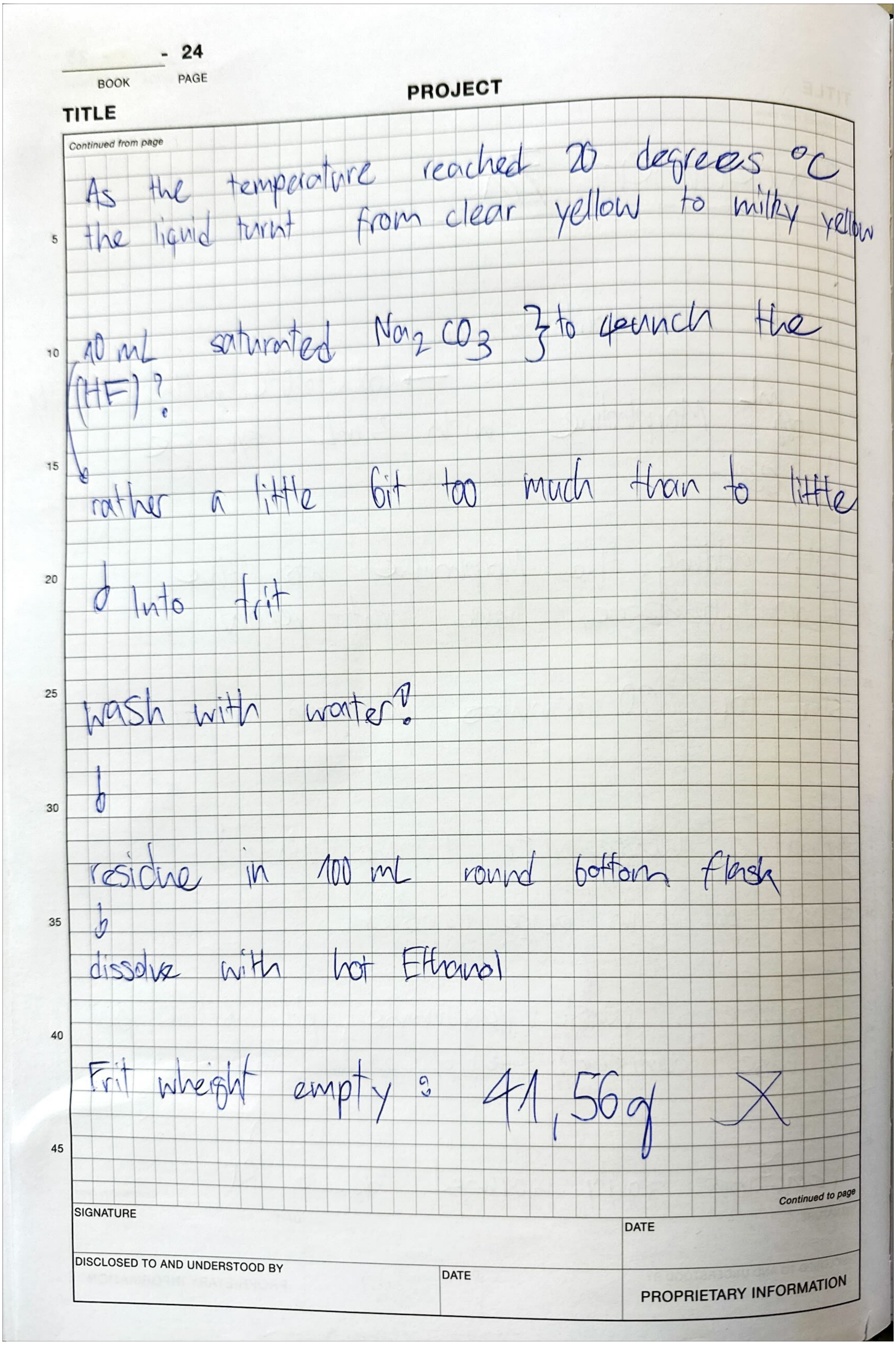

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As the temperature reached 20 degrees °C the liquid turnt from clear yellow to milky yellow

10 mL saturated Na_2CO_3 } to qeunch the (HF)?

rather a little bit too much than to little

↓ Into frit

wash with water?

↓

residue in 100 mL round bottom flask

↓

dissolve with hot Ethanol

Frit wheight empty : 41,56g X

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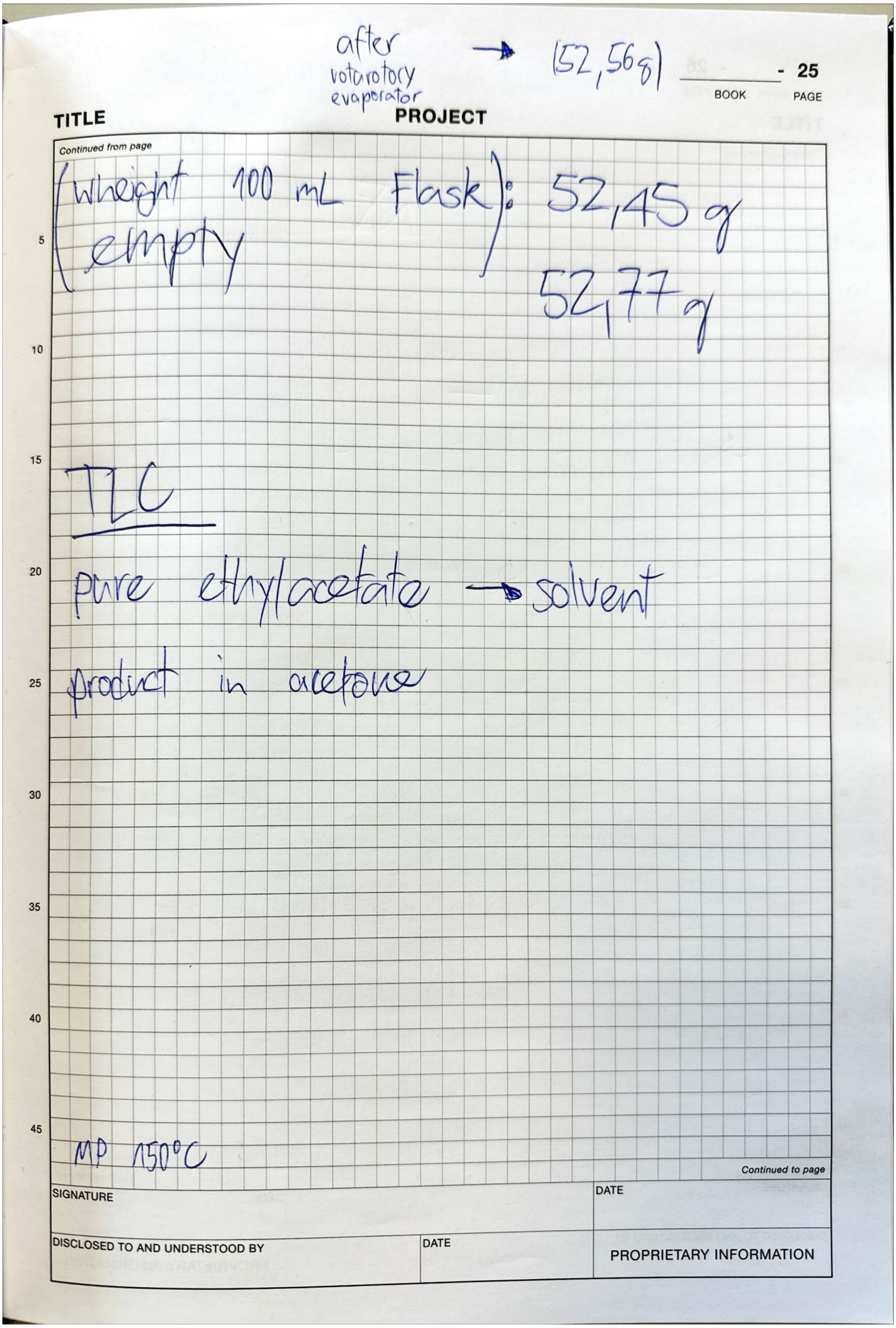

after votarotory evaporator → (52,56g)

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(wheight 100 mL Flask empty): 52,45 g

52,77 g

TLC

pure ethylacetate → solvent

product in acetone

MP 150°C

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