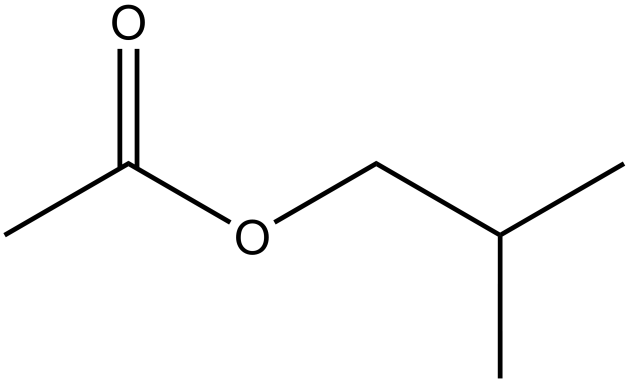
The Synthesis of Isobutyl Acetate by Esterification and Distillation



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Abstract The objective of this experiment is to synthesize isobutyl acetate and analyze the yield of the product. The synthesis is performed through the Fischer-Speier esterification of acetic acid with 2-methyl-1-propanol and the usage of sulfuric acid as catalyst. The substance was purified by distillation. The success of the experiment is determined by thin layer chromatography, infrared spectrum measurements and the characteristic cherry odor of isobutyl acetate.

Zürich, March 18, 2024

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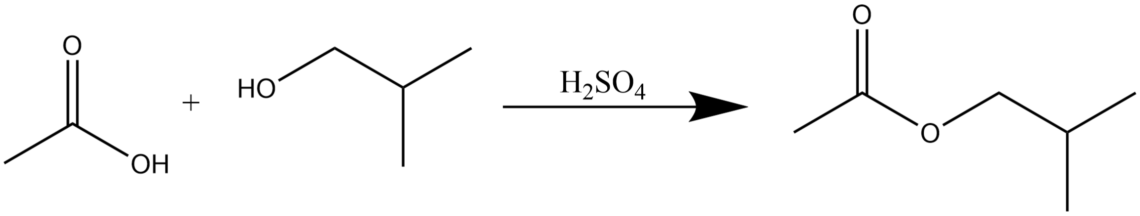
Introduction

Isobutyl acetate consists of a butyl group connected to a carboxylic ester group. At room temperature it is a clear, colourless liquid with a fruity odor and a flavour like cherry, raspberry and strawberry [1]. Higher concentrations of this substance are irritating to the skin and the respiratory system. Isobutyl acetate naturally occurs in many different fruits. It serves as a solvent for paints, lacquers, varnishes and it can be used in the production of coatings [2]. Also it can be used as flavor and fragrance agent and it is found in cosmetic products like nail polishers.

Material Properties

For the synthesis of isobutyl acetate the Fischer-Speier esterification was used. 2-Methyl-1-propanol (isobutanol) was mixed with acetic acid and sulfuric acid was utilized as a catalyst.



Substance	GHS Hazard Pictogram	H and P Phrases
2-Methyl-1-propanol [3]		H226, H315, H318, H336, H335-P210, P233, P240, P280, P303, P361, P353, P305, P351, P338
Acetic acid [4]		H226, H314, H318, H315, H319-P210, P280, P303, P361, P353, P305, P351, P338, P310
Sulfuric acid[5]		H290, H314, H318, H315, H319-P234, P280, P303, P361, P353, P304, P340, P310, P305, P351, P338, P363
Dichloromethane [6]		H315, H319, H351, H336-P202, P261, P264, P302, P352, P305, P351, P338, P308, P313
Sodium hydrogencarbonate [7]		-
Magnesium sulfate [8]		-
Isobutyl acetate [9]		H225, H336-P210

Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
2-Methyl-1-propanol [3]	74.12	0.803	-108	108
Acetic acid [4]	60.05	1.049	16.7	118.1
Sulfuric acid [5]	98.08	1.84	10	337
Dichloromethane [6]	84.93	1.325	-96.7	39.6
Sodium hydrogencarbonate [7]	84.006	2.20	50	-
Magnesium sulfate [8]	120.37	2.66	1’124	-
Isobutyl acetate [9]	116.16	0.872	-99	118

Safety Assessment

In this experiment several chemical hazards must be considered. Isobutanol is a flammable liquid and vapor that can cause serious eye irritation. Acetic acid can cause severe skin burns and eye damage, as well as respiratory irritation. Sulfuric acid can cause severe skin burns and eye damage. Dichloromethane is a carcinogen cause and can cause drowsiness or dizziness. Sodium hydrogen carbonate is generally less hazardous, but it can release carbon dioxide gas when reacting with acids, which can increase pressure in closed systems.

Also the synthesis should be conducted in a fume hood, because of the harmful vapors. Sulfuric acid must be added dropwise to the reaction mixture to control the exothermic reaction and avoid splashing. A reflux condenser is used to prevent the escape of flammable vapors. The reaction mixture should be heated carefully to prevent bumping and over-boiling. Once the reaction is complete, the mixture should be allowed to cool to room temperature before further handling. The organic layer’s washing with sodium hydrogen carbonate requires regular venting of the separatory funnel to avoid the build-up of pressure due to CO_2 evolution.

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.

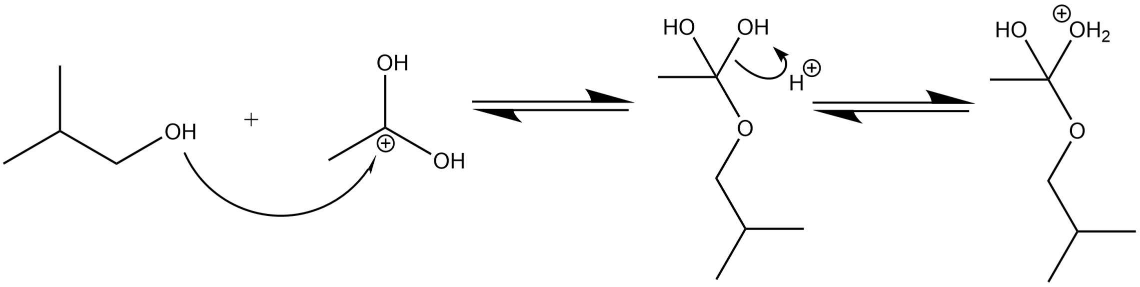
Waste Disposal

Isobutanol, Dichloromethane and isobutyl acetate are organic molecules. After the synthesis, the product liquid doesn’t entirely consist of isobutyl acetate, there may be traces of the educts in there. So the liquid should be seen as an solvent that contains chlorine, because of the dichloromethane that was used in the purification. The acid in the aqueous phase got neutralized when it was combined with sodium hydrogencarbonate, so it should be disposed in the aqueous acidic or basic waste, depending on the pH. Sulfuric acid is especially harmful to aquatic life, so it should be disposed in the acid waste. Even though the sodium hydrogencarbonate is solid at room temperature, it is still able to react with the vapor in the air, so it should be disposed in the basic waste. Magnesium sulfate should be disposed in the solid waste.

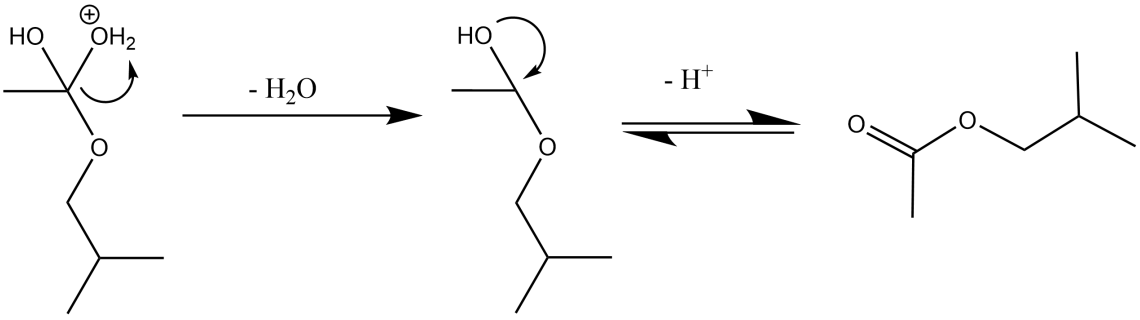
Reaction Mechanism

The production of isobutyl acetate is enhanced through an acid-catalyzed esterification of acetic acid. In this method, sulfuric acid acts as a catalyst and initiates the reaction by protonating the carboxyl group of acetic acid. This protonation leads to the formation of a more reactive intermediate, the carbocation.

The carbocation carries a positive charge, which makes it an electrophile that likes to react with nucleophiles. In this reaction, isobutanol serves as the nucleophile. Its hydroxyl group attacks the positively charged carbon atom of the carbocation. That leads to the formation of a new covalent bond between the oxygen of the alcohol group and the carbon of the carbocation. This forms a protonated ester that is a tetrahedral intermediate.



At last a molecule of water and a proton get removed. The water molecule is the leaving group. This is what creates the isobutyl acetate.



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 87%. This reaction is really atom efficient.

$$\mathrm{AE} = \frac{M_{\text{Product}}}{M_{\text{Reagents}}} = \frac{116.16\ \text{g/mol}}{74.12\ \text{g/mol} + 60.05\ \text{g/mol}} \approx 86.58\%$$

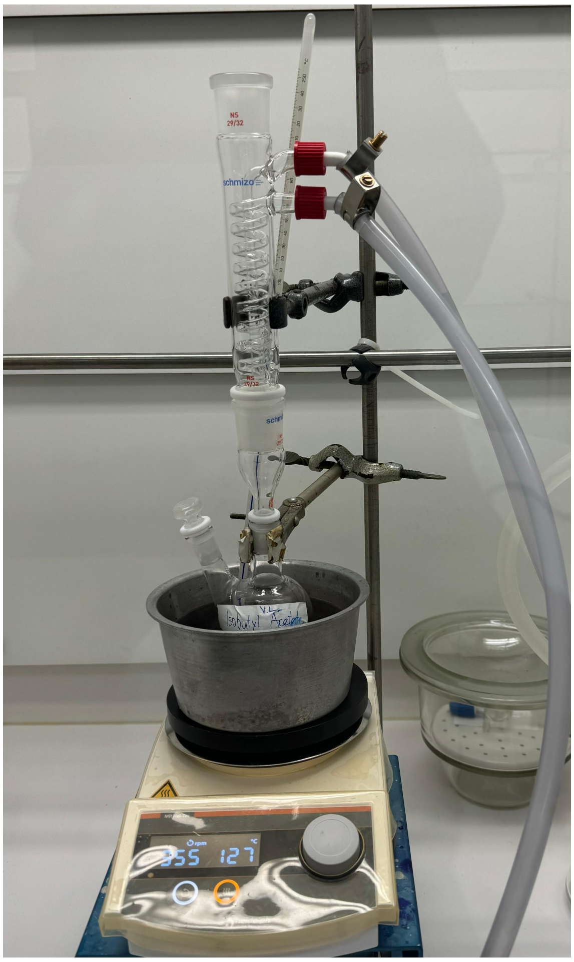
Experimental

Procedure

The production of isobutyl acetate was achieved using the Fischer-Speier esterification process. In this procedure, 2-methyl-1-propanol (isobutanol) was combined with acetic acid, with sulfuric acid serving as the catalyst to facilitate the reaction. The product was a colorless liquid (13.046 g, 112.31 mmol, 1.12 equiv.). That would be 112% of the theoretical yield, which is not possible.

Observation

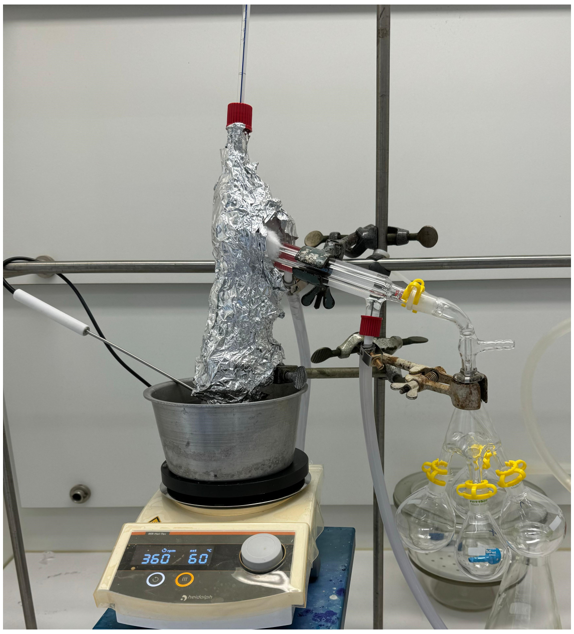
For the synthesis of isobutyl acetate, isobutanol (9.2 mL, 100 mmol, 1.0 equiv.) was mixed with acetic acid (13.7 mL, 240 mmol, 2.4 equiv.) in a 100 mL double neck round bottom flask with a magnetic stirring bar. Then sulfuric acid (2.0 mL, 37.52 mmol, 0.375 equiv.) was added slowly. A reflux condenser was attached to the flask and the cooling water was switched on. With the help of an oil bath, the mixture was heated to around 130 °C for about 2 hours. This resulted in a red liquid that was analyzed by TLC.



After that, the reaction mixture was transferred into a separatory funnel, ensuring complete transfer by rinsing the original flask with a small quantity of water and adding it into the funnel. Water was then added to the funnel to double the volume of the aqueous phase relative to the organic phase. The isobutyl acetate was inside the organic phase, so it had to be collected in a 100 mL erlenmeyer flask. Both phases were allowed to settle and separate for some minutes. The organic phase first had a lower density, so it was the upper phase. The lower aqueous phase was collected in an erlenmeyer flask. The upper organic phase was collected in another erlenmeyer flask. The aqueous phase underwent an extraction with an equivalent volume of dichloromethane. Thanks to the dichloromethane, now the organic residues in the aqueous phase had a higher density now and they sank down. The separatory funnel was used again for the removal of the aqueous phase and the organic phase was collected again into the erlenmeyer flask.

A laboratory partner made a saturated sodium hydrogen carbonate solution. The solution was transferred into the seperatory funnel with the combined organic extracts. The solution was shaken and the funnel opened to release CO_2 that was created by the reaction. This process was repeated many times until no more CO_2 was created, which meant that the reaction was complete. This was visible by the absence of CO_2 bubbles in the solution and no sound of escaping gas when opening the funnel. The organic phase was subsequently dried over magnesium sulfate and filtered through a 50 mL filter crucible. The residue was disposed, the isobutyl acetate was inside the mother liquors. With the use of a rotary evaporator the solvent got removed. The liquid turned yellow.

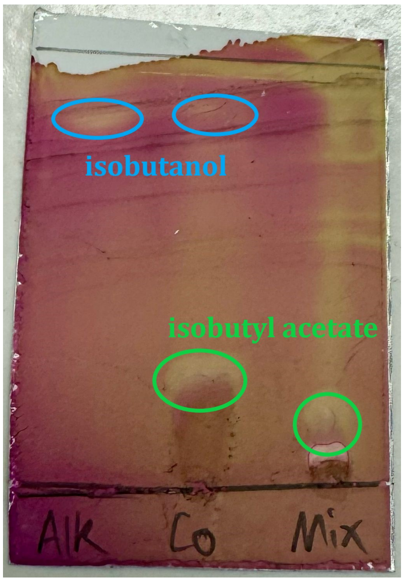
This liquid still had many other components in it. Through distillation, pure isobutyl acetate should be achieved. With the oil bath and a condenser, the mixture was heated up. First to 100 °C into flask number 1, then to 130 °C into flask number 2 and to 170 °C to flask number 3. After tared weighing, the colorless liquid in flask 3 that could be isobutyl acetate weighed 13.046 g (112.31 mmol, 1.12 equiv.). That would be 112% of the theoretical yield, which is not possible. The IR spectrum shows no peaks that could have come from isobutanol or dichloromethane. So flask 3 doesn't have much impurities, what could have bumped the weight up. So probably the tarring of the scale was conducted wrongly.



Characterisation

Thin Layer Chromatography

Thin layer chromatography (TLC) is an analytical technique that separates organic compounds based on their affinity for two phases: a stationary phase and a mobile phase [10]. In TLC, a thin layer of adsorbent, typically silica gel or alumina, is applied to a plate. The mixture that has to be analyzed is then spotted near the base of the plate. The plate is placed in a solvent system, and the solvent (mobile phase) travels up the plate by capillary action, carrying the compounds with it. The different compounds move at different rates based on their solubility in the solvent and their interaction with the stationary phase, resulting in separation. After the solvent has traveled a desired distance, the plate is removed, dried, and visualized to observe the separated compounds. The visualisation can happen with putting the silica plate into a potassium permanganate stainer or looking at the plate under UV light. The retention factor (Rf value) of a substance can be determined by dividing the distance traveled by the substance with the distance traveled by the solvent front.



In this TLC, a silicate plate was used and there were three columns. The first column (Alk) had only one educt, the isobutanol. The second column (Co) had isobutanol and the red liquid mixture with the product isobutyl acetate on it. The third column (Mix) only had the red liquid mixture on it. The solvent consisted of 1 part n-Hexane and 7 parts ethylacetate.

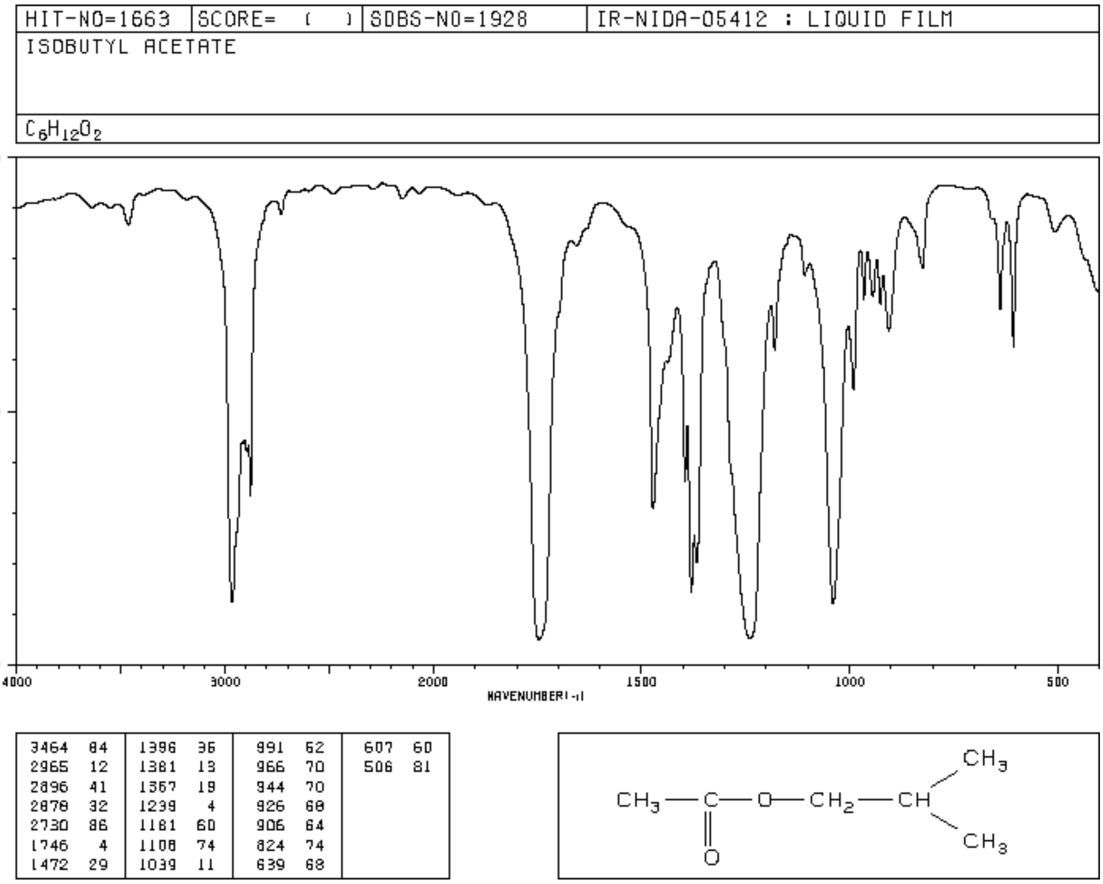
The less polar the substance is, the more it rises, because the polar substances are strongly attracted by the silica plate and therefore slowed down by it. The isobutanol rose higher up than the isobutyl acetate because it is less polar. The Rf values are 0.88 for isobutanol and 0.15 for isobutyl acetate.

Characteristic Odor

For the author the odor of the isobutyl acetate was like cherry. But that is a really subjective measurement, that could also be influenced by psychological factors like that the author expected it to smell like cherry. That would be an example of conformation bias or placebo effect.

Infrared Spectrum

This would be the literature values of the infrared spectrum of isobutyl acetate [11].



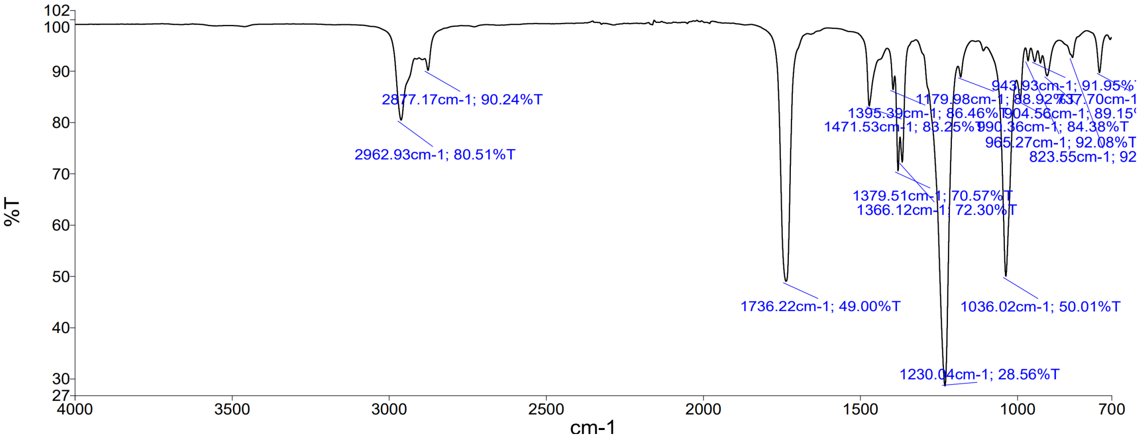
HIT-NO=1663 | SCORE= ( ) | SDBS-NO=1928 | IR-NIDA-05412 : LIQUID FILM

ISOBUTYL ACETATE

$C_6H_{12}O_2$

3464	84	1396	36	991	62	607	60
2965	12	1381	13	966	70	506	81
2896	41	1367	19	944	70		
2878	32	1239	4	926	68		
2730	86	1161	60	906	64		
1746	4	1108	74	824	74		
1472	29	1039	11	639	68		

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



In the spectral analysis, the observed peaks align with those documented in the literature, although they are weaker in intensity [12]. 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
1736.22	C=O stretch (Esters)	49.00
2962.93	C-H stretch (Methyl, Methylene)	80.51
2877.17	C-H stretch (Methyl, Methylene)	90.24
1379.51	C-H bending (Methyl)	70.57
1366.12	C-H bending (Methyl)	72.30
1230.04	C-O stretch (Esters)	28.56
1036.02	C-O and O-H vibrations	50.01

Discussion

Why is the acidic proton from the starting acid not deprotonated?
Because of the acidic environment of the reaction. The sulfuric acid is a much stronger acid than the acetic acid. The acetic acid has a pKa of 4.76 and the sulfuric acid has a pKa of -2.8. The acetic acid never has the chance to deprotonate because the sulfuric acid deprotonates instantaneously.

What could happen to the alcohol moiety if it gets protonated as a first step in the reaction mechanism?
In the Fischer esterification mechanism, if the alcohol moiety is protonated first, it does not act as the attacker in the reaction sequence [13]. Instead, the protonated alcohol becomes a better leaving group, and in this electrophilic state, it is the carboxylic acid that undergoes a nucleophilic attack by the alcohol. The negative charge on the nucleophile is attracted to the positive charge of the protonated alcohol, leading to the formation of water and the ester product. But if the alcohol deprotonates and becomes a better leaving group, that's detrimental for the reaction, because the water can leave and there will be no nucleophilic attack on the acetic acid anymore. The reaction is reversible, so the side product will just go back to be the alcohol.

What happens to the released proton after the ester product formation?
After the ester product is formed, the proton is transferred to a water molecule, which is a byproduct of the reaction. This helps to regenerate the acid catalyst, which can then participate in further esterification reactions. Therefore the acid catalyst remains in the reaction mixture and is not consumed.

If the final ester is protonated, it becomes more reactive than your initial acid. How is it that mostly the ester is obtained?
The final ester can indeed become protonated but the molecules that result from the protonation are charged positively and therefore are typically short-lived and would quickly react to form a more stable structure.

What could be done to stop the catalyst from reacting in the end?
To stop the catalyst from reacting towards the end of a Fischer esterification, the acid catalyst could be neutralized [14]. This is typically done by adding a base, such as sodium bicarbonate, to the reaction mixture after the esterification is complete. The base will react with the acid to form a salt and water, effectively removing the acid catalyst from the reaction mixture and halting the catalysis.

Are there any other ways to control the equilibrium of this reaction?
The principle of Le Chatelier can be applied for this. Continuously removing the byproduct water and adding the educts, alcohol and carboxylic acid, can improve the formation of ester. The water can also be removed by an Dean-Stark apparatus. The reaction is also exothermic, that means it generates heat. So cooling it down would also improve esterification.

How would the concern be addressed that the equilibrium of the reaction may not have fully shifted towards the desired product and that the final product may be contaminated with the starting alcohol?
The steps that were already mentioned in the last question should be followed. In addition the duration of the reaction time should be increased. A 100 % pure product without any educts because they all reacted, is not really possible. But with farther separation steps like distillation, the purity of the product could be increased greatly.

References

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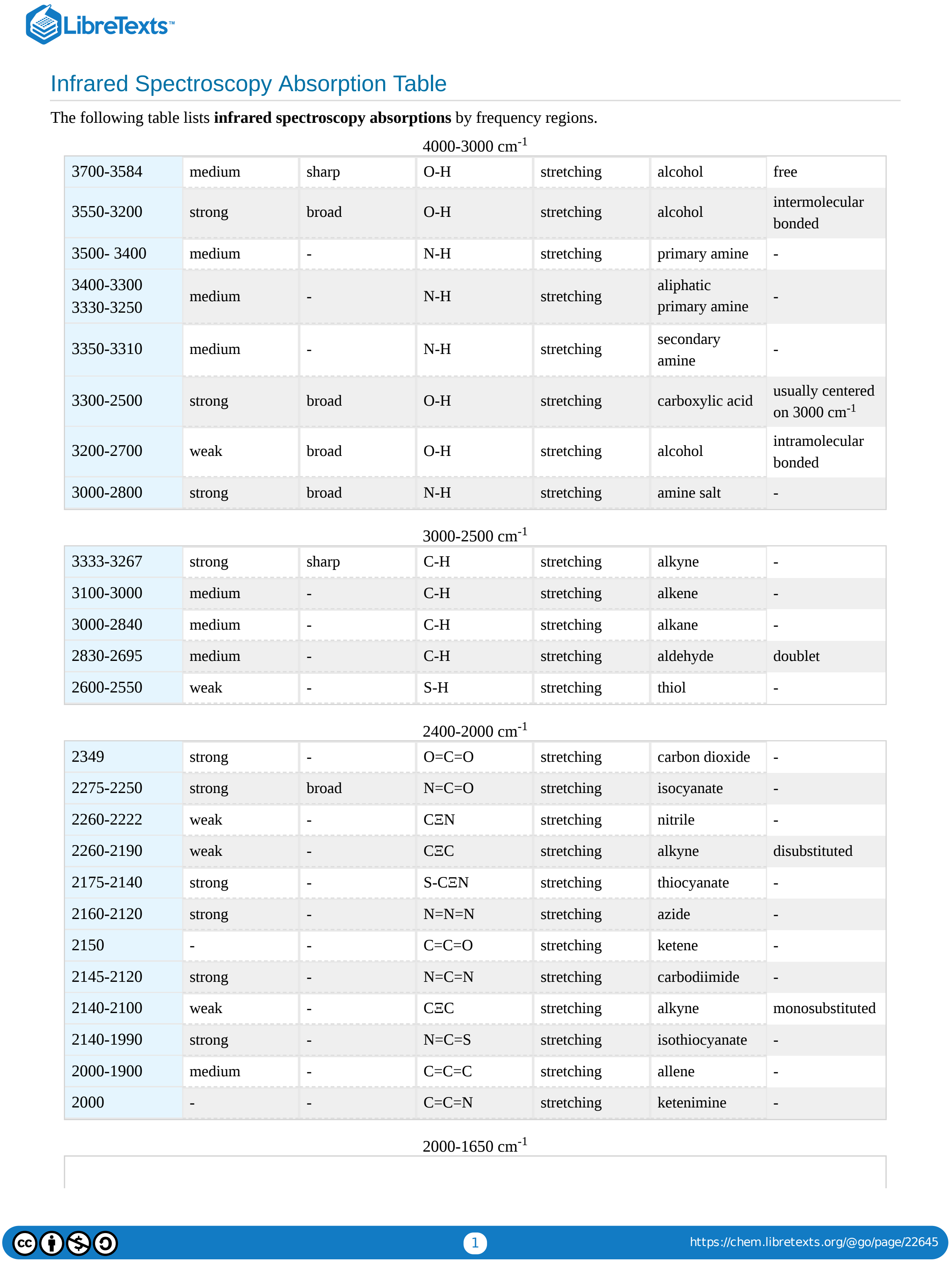
Appendix

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	Mass g	Volume mL	Purity
2-Methylpropan-1-ol	74,12	1.00	100 mmol	7,412	9,230	99,5%
Acetic acid	60,05	2.40	240 mmol	14,412	13,739	≤ 99,7%
Sulfuric acid	98,08	0.375	37,52 mmol	3,68	2.0 ml	95% - 98%
Product	**MW**	**Yield**	**Moles**	**Mass**	**BP**	
Isobutyl acetate	116,16	112% ???	112,31 mmol	13,046	115-117 °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}

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

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

1870-1540 cm^{-1}

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

1670-1600 cm^{-1}

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

1600-1300 cm^{-1}

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

1400-1000 cm^{-1}

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

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

1000-650 cm^{-1}

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

900-700 cm^{-1}

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

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

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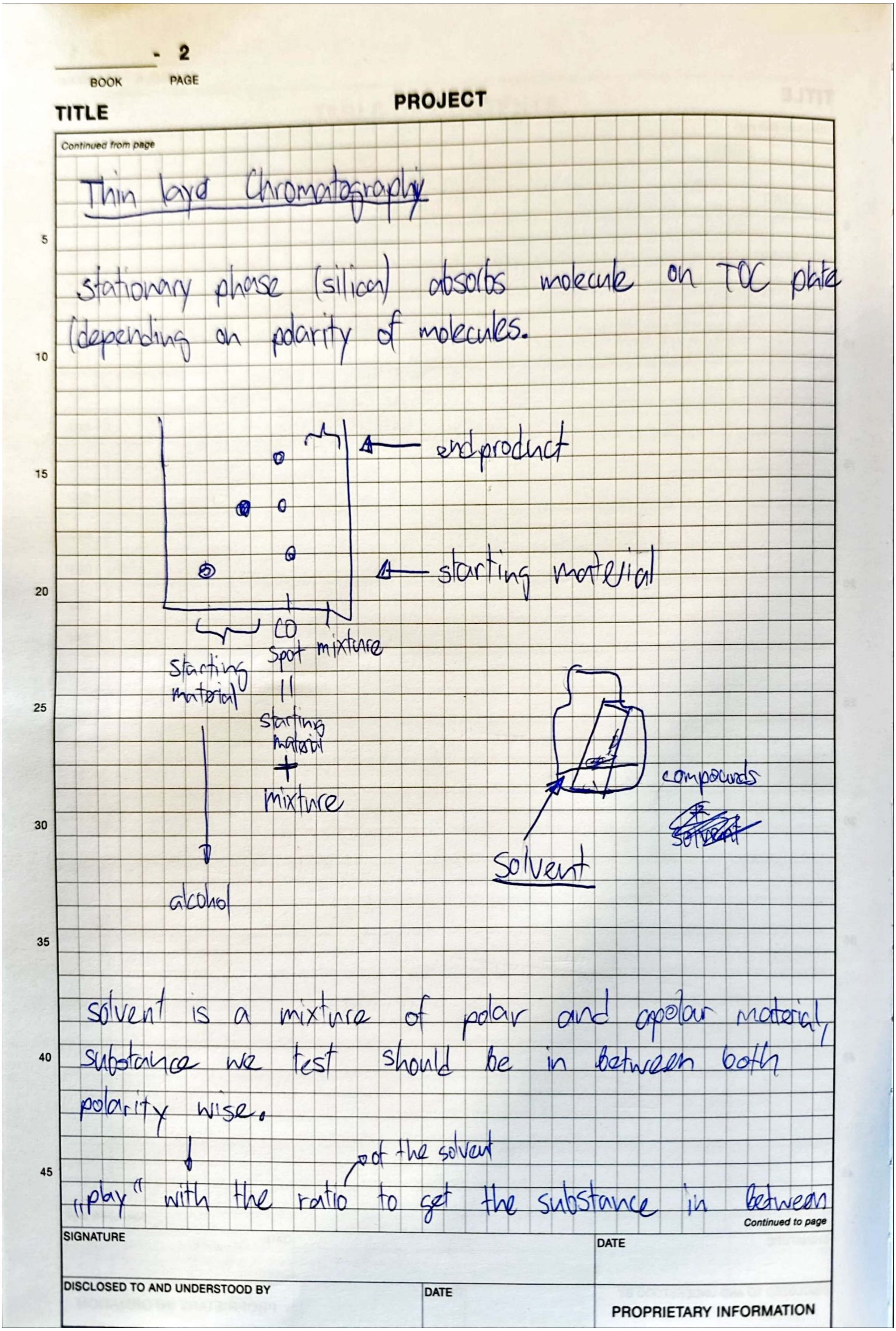
- OChemOnline



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TITLE PROJECT

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Thin layer Chromatography

stationary phase (silicon) absorbs molecule on TLC plate (depending on polarity of molecules.

solvent is a mixture of polar and apolar material, substance we test should be in between both polarity wise.

↓

„play" with the ratio of the solvent to get the substance in between

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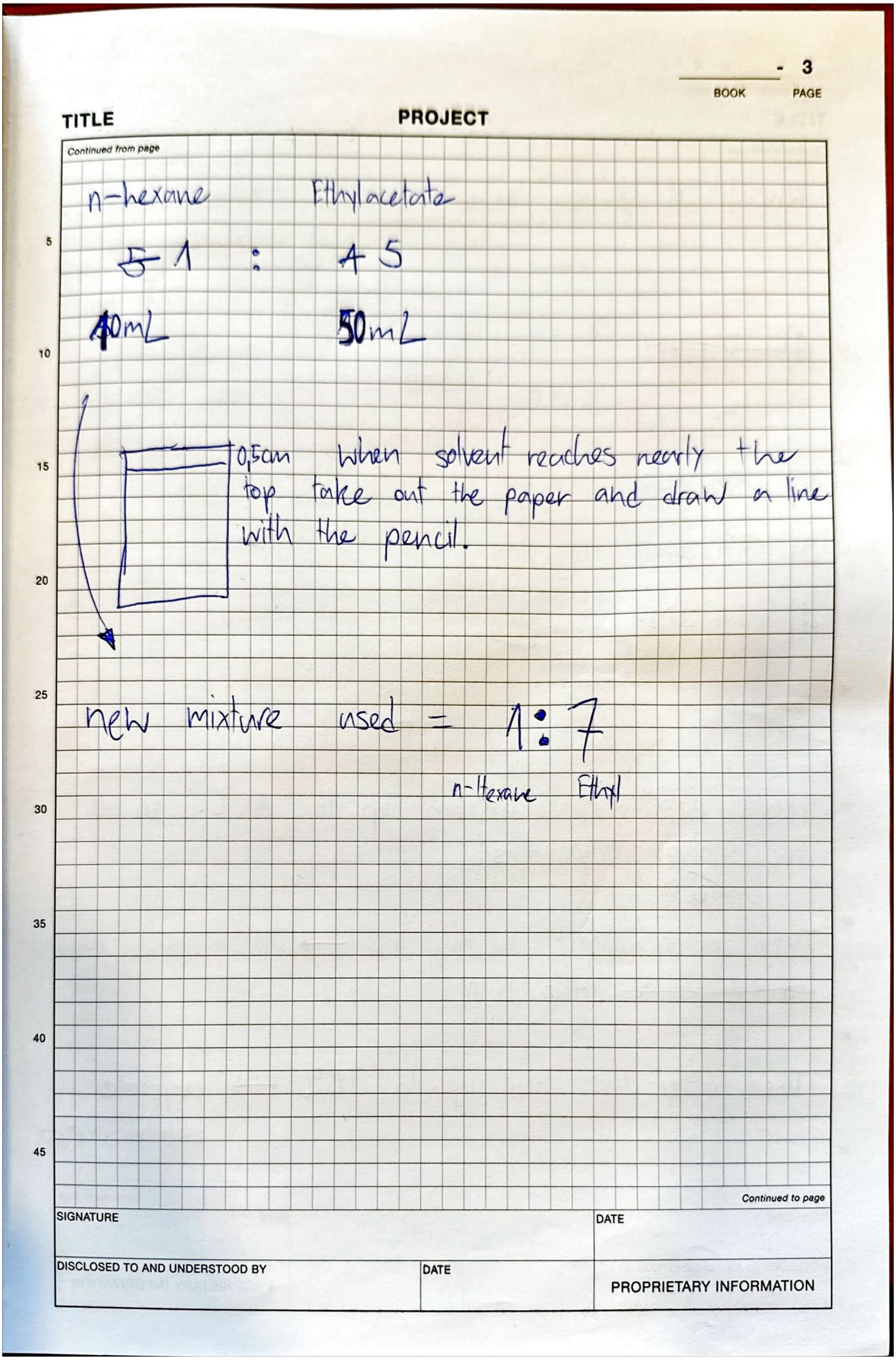

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TITLE PROJECT

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n-hexane Ethylacetata

~~5~~ 1 : 4 5

10mL 50mL

0,5cm when solvent reaches nearly the top take out the paper and draw a line with the pencil.

new mixture used = 1 : 7

n-Hexane Ethyl

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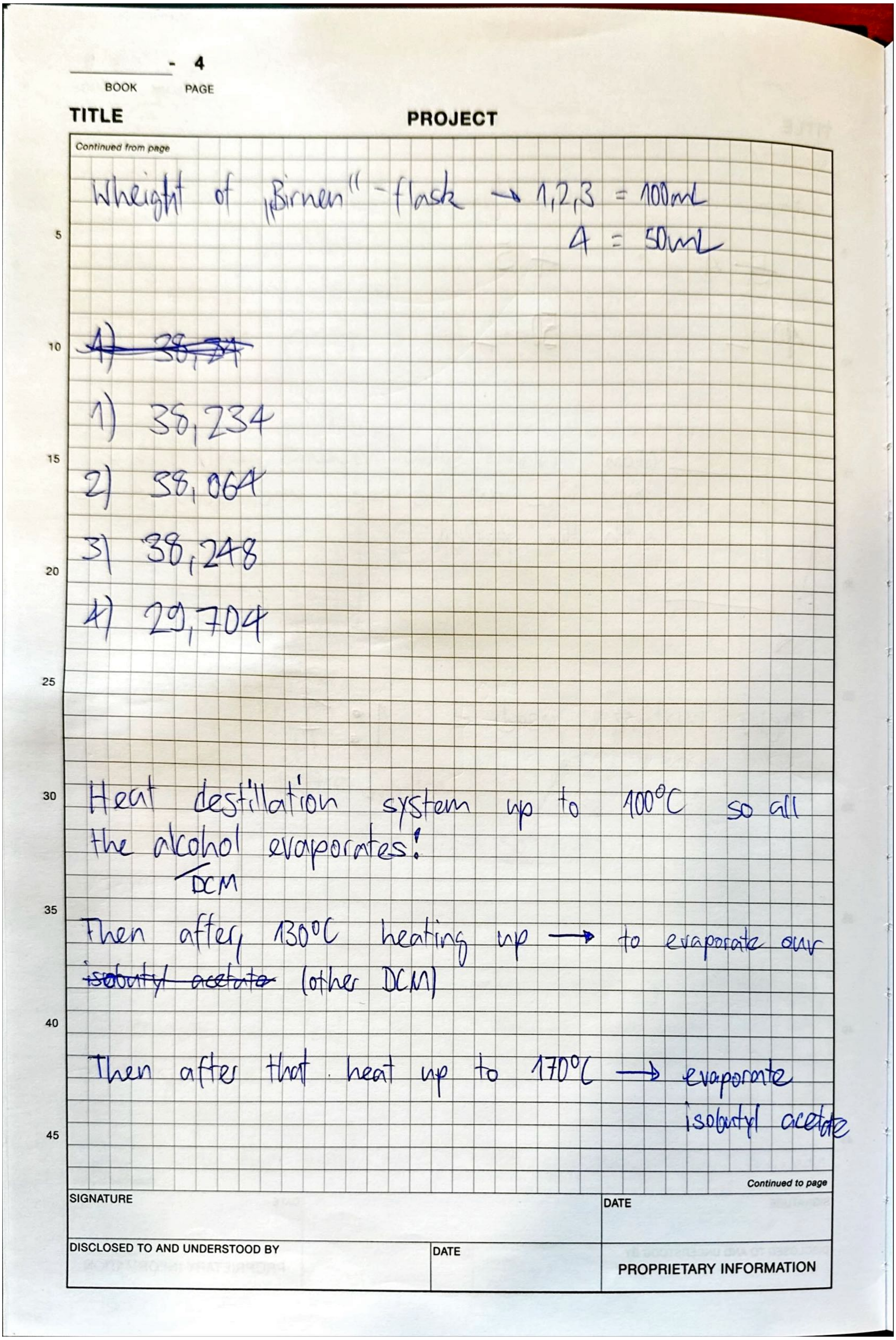

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BOOK PAGE

TITLE PROJECT

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Wheight of „Birnen"-flask → 1,2,3 = 100mL
4 = 50mL

~~1) 38,24~~

1) 38,234

2) 38,064

3) 38,248

4) 29,704

Heat destillation system up to 100°C so all the alcohol evaporates!
DCM

Then after, 130°C heating up → to evaporate our ~~isobutyl acetate~~ (other DCM)

Then after that heat up to 170°C → evaporate isobutyl acetate

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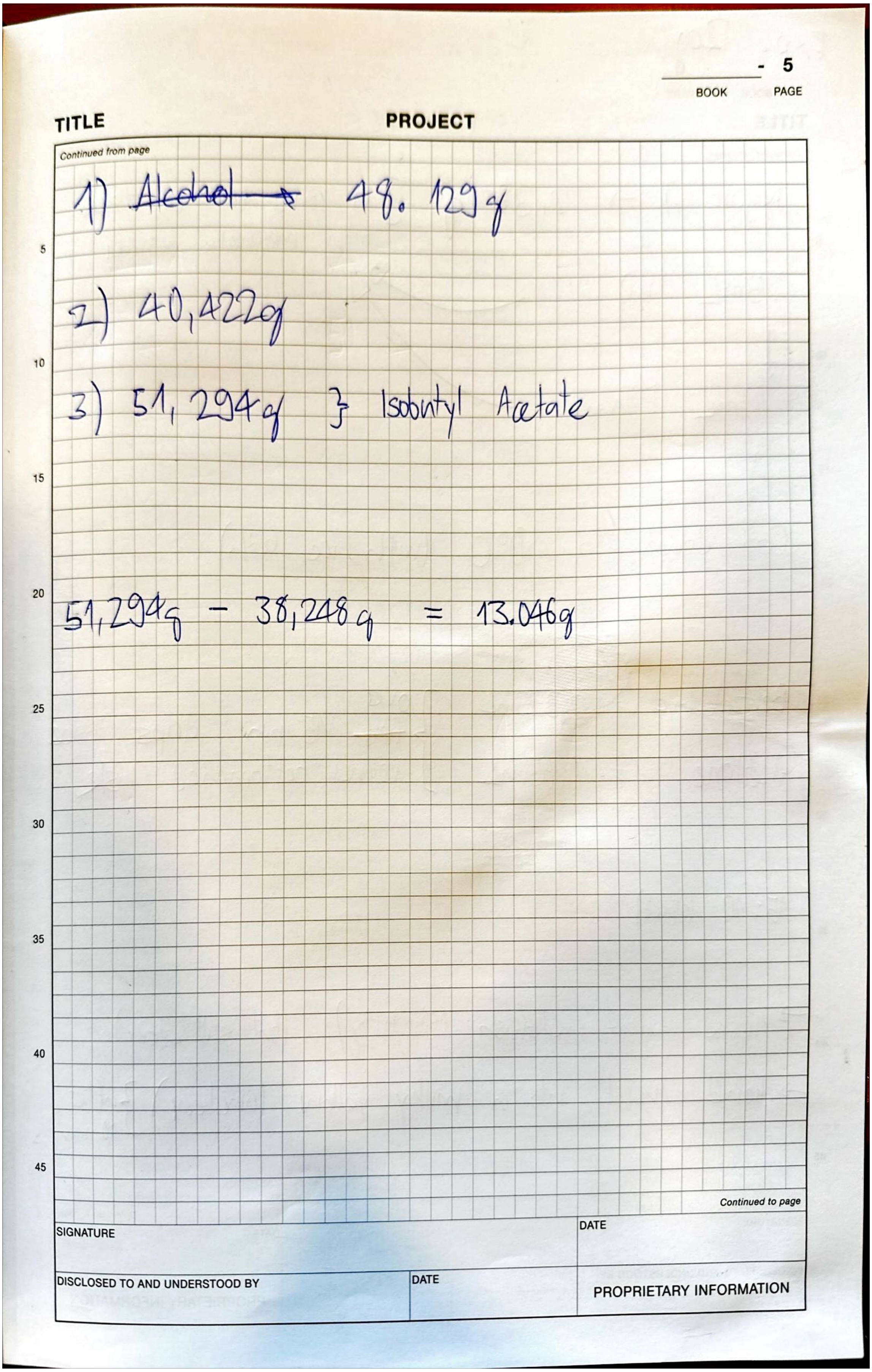

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TITLE PROJECT

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1) ~~Alcohol →~~ 48.129g

2) 40,422g

3) 51,294g } Isobutyl Acetate

51,294g − 38,248g = 13.046g

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