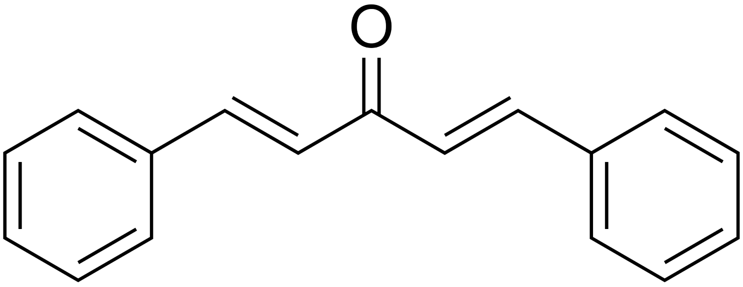
ETH zürich

Laboratory Course OACP I
Spring Semester 2024

The Synthesis of (E,E)-Dibenzylideneacetone by Aldol Condensation and Recrystallization



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Abstract The objective of this experiment is to synthesize (E,E)-dibenzylideneacetone and analyze the yield of the product. The synthesis is performed through an aldol condensation reaction with NaOH, followed by a recrystallisation process for purification. The success of the experiment is determined by the melting point, thin layer chromatography and an IR spectrum measurement. 1.373 grams of dibenzylideneacetone were synthesized, equating to a yield of around 51%.

Zürich, May 9, 2024

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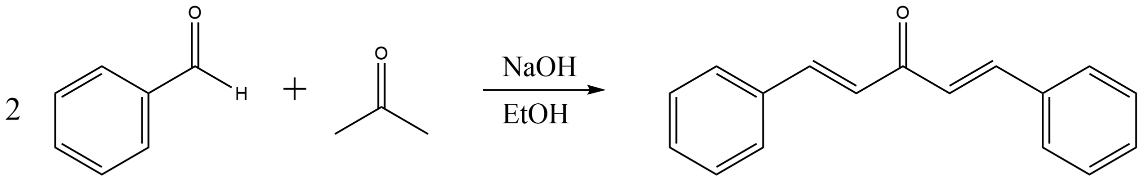
Introduction

(E,E)-Dibenzylideneacetone, also known as dibenzalacetone, is a yellow, crystalline solid. It is insoluble in water but soluble in ethanol [1]. It is is a Ketone group connected to two ethenylbenzene groups. It is commonly used as a UV blocker, due to its ability to absorb ultraviolet radiation. It is used as a ligand for palladium in organometallic chemistry [2]. This compound is synthesized through an aldol condensation reaction, that forms carbon-carbon bonds.

Material Properties

In this experiment dibenzalacetone is synthesized by an aldol condensation reaction. Benzaldehyde is mixed with acetone. Then sodium hydroxide is added and works as a catalyst. Ethanol is used as the solvent.



Substance	GHS Hazard Pictogram	H and P Phrases
Ethanol [3]		H225, H319-P210, P233, P240, P241, P242, P305, P351, P338
Acetone [4]		H225, H319, H336-P210, P233, P240, P241, P242, P305, P351, P338
Benzaldehyde [5]		H302, H332, H315, H319, H360, H335, H411-P273, P301, P312, P302, P352, P304, P340, P305, P351, P338, P308, P313
Sodium hydroxide [6]		H290, H314, H318, H315, H319-P234, P260, P280, P303, P361, P353, P304, P340, P310, P305, P351, P338
Dibenzalacetone [1]		H317, H400, H410, P261, P272, P273, P280, P302+P352, P321, P333+P317, P362+P364, P391, P501

Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
Ethanol [3]	46.07	0.789	-114.1	78.37
Acetone [4]	58.08	0.791	-94.7	56.05
Benzaldehyde [5]	106.12	1.05	-26	179
Sodium hydroxide [6]	40.00	-	318	1388
Dibenzalacetone [1]	234.29	1.0477	111	130

Safety Assessment

In this experiment several chemical hazards must be considered. Ethanol is highly flammable, and its vapors can irritate the eyes. Acetone is similarly flammable and can irritate the eyes, skin, and respiratory system. Benzaldehyde is harmful if inhaled and can cause skin and eye irritation and is suspected of causing genetic defects. Sodium hydroxide is corrosive, causing severe skin burns and eye damage, and should be handled with caution.

The NaOH solution should be added carefully drop by drop, slowly over a duration of 30 minutes. The reaction requires chilling to 0 °C, which means handling cold materials, where there is a risk of cold burns, so proper insulation or protective measures should be taken. Benzaldehyde and acetone should also be added carefully drop by drop, slowly over a duration of 15 minutes.

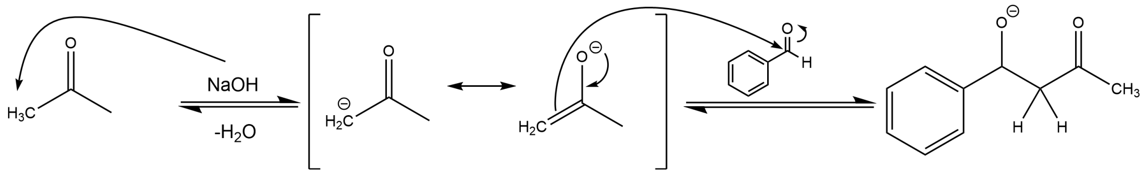
For avoiding any health issues that could be caused by the chemicals, gloves and safety googles should be worn in the laboratory. Also the synthesis should be conducted in a fume hood, because of the harmful vapors. In a case of an accident the emergency number should be called. With the internal ETH-phones 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

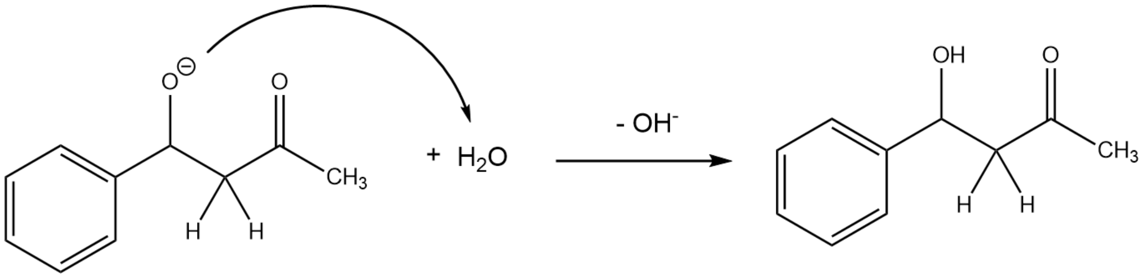
The ethanol, acetone and benzaldehyde are liquid organic solvents without chlorine so they should be disposed in the liquid solvent waste. Even though sodium hydroxide and the dibenzalacetone are solid at room temperature so they should be disposed in the basic waste, because they could react with the vapor in the air. Benzaldehyde and dibenzalacetone are especially harmful to aquatic life.

Reaction Mechanism

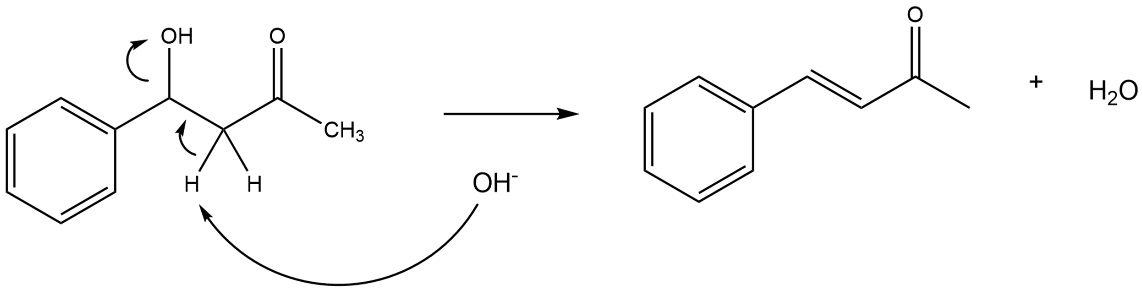
In this synthesis an aldol condensation is used. The OH from the NaOH deprotonates the beta position of the acetone. That leaves behind a pair of electrons and that creates a negative charge on the carbon, what creates an enolate. An enolate is an anion formed when an alpha hydrogen in the carbonyl compound (like ketones or aldehydes) is removed by a base. This negative charge makes the carbon nucleophilic and the negative charge can move between two carbons and makes a resonance structure. That leads to the formation of a double bond on the acetone molecule, that's a clear indication that a elimination reaction happened. This would be the E1cb mechanism. The "E" stands for elimination, "1" signifies that the rate-determining step is unimolecular, and "cb" stands for "conjugate base." In the context of aldol condensation, an E1cb mechanism involves the formation of an enolate anion from the deprotonation of an aldehyde or ketone. This enolate anion then acts as a nucleophile.



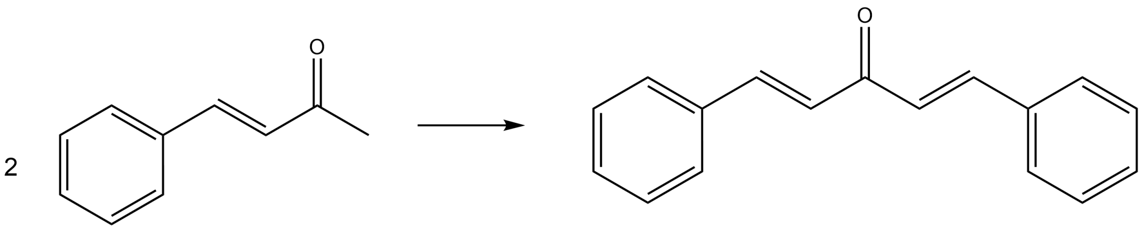
The resulting negatively charged oxygen atom gets a proton from a water molecule resulting in the separation of a hydroxy group.



During the aldol condensation, the oxygen atom that was initially protonated releases as a hydroxide ion. The carbon atom, which was involved in forming the new carbon-carbon bond, loses a proton. This loss of a proton leads to the formation of a double bond. That step completes the aldol condensation reaction.



This reaction repeats on the other side of the molecule. A complete (E,E)-dibenzylideneaceton molecule is created.



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 76%. This reaction is somewhat atom efficient.

$$\mathrm{AE} = \frac{M_{\mathrm{Product}}}{M_{\mathrm{Reagents}}} = \frac{234.29\ \mathrm{g/mol}}{2 \times 106.12\ \mathrm{g/mol} + 58.08\ \mathrm{g/mol} + 40.00\ \mathrm{g/mol}} \approx 75.50\%$$

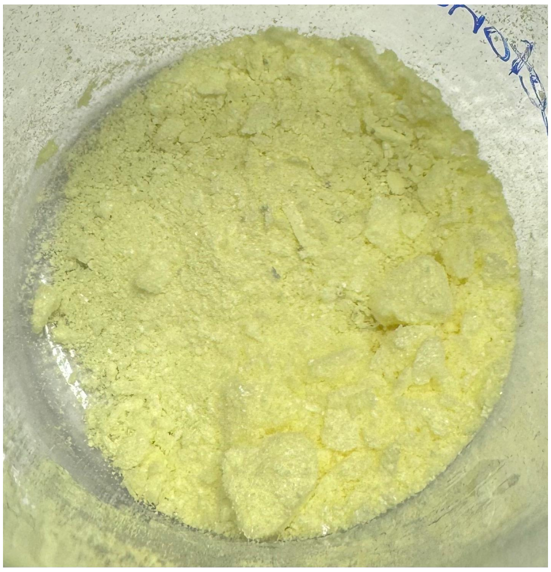
Experimental

Procedure

Dibenzalacetone was synthesized through an aldol condensation. The process involves mixing benzaldehyde with acetone and introducing sodium hydroxide, which acts as a catalyst. Ethanol serves as the solvent for the reaction. The product was a yellow crystalline solid (1.373 g, 5.86 mmol, 0.51 equiv.) with a yield of about 51%.

Observation

For the synthesis of the dibenzalacetone, a magnetic stir bar was placed in a 100 mL single-neck flask, which was then filled with a sodium hydroxide solution (2.30g NaOH, 57.32 mmol, 5 equiv.) into 25 mL of water. After the addition of 17 mL of ethanol to the flask, a thermometer was put into the mixture and it was cooled down to near 0°C with an ice bath. A slow addition of benzaldehyde (2.39 mL, 23.5 mmol, 2.05 equiv.) and acetone (0.85 mL, 11.46 mmol, 1 equiv.) to the cooled solution was performed over 15 minutes. Then the mixture was stirred at room temperature for about an hour. After that the milky yellow liquid was tested with a TLC.

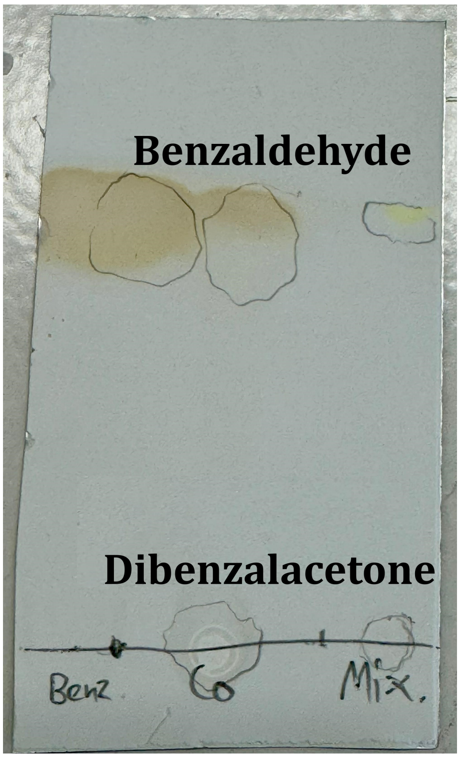


The resultant solid was filtered and washed with water. Recrystallization from ethanol yielded a yellow crystalline solid of (E,E)-Dibenzylideneacetone (1.373 g, 5.86 mmol, 0.51 equiv.) with a yield of about 51%.

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 [7]. 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 (Benz) had only one educt, the benzaldehyde. The second column (Co) had benzaldehyde and the yellow liquid mixture with the product dibenzalacetone on it. The third column (Mix) only had the yellow liquid mixture on it. The solvent consisted of 1 part n-Hexane and 1 part ethylacetate.

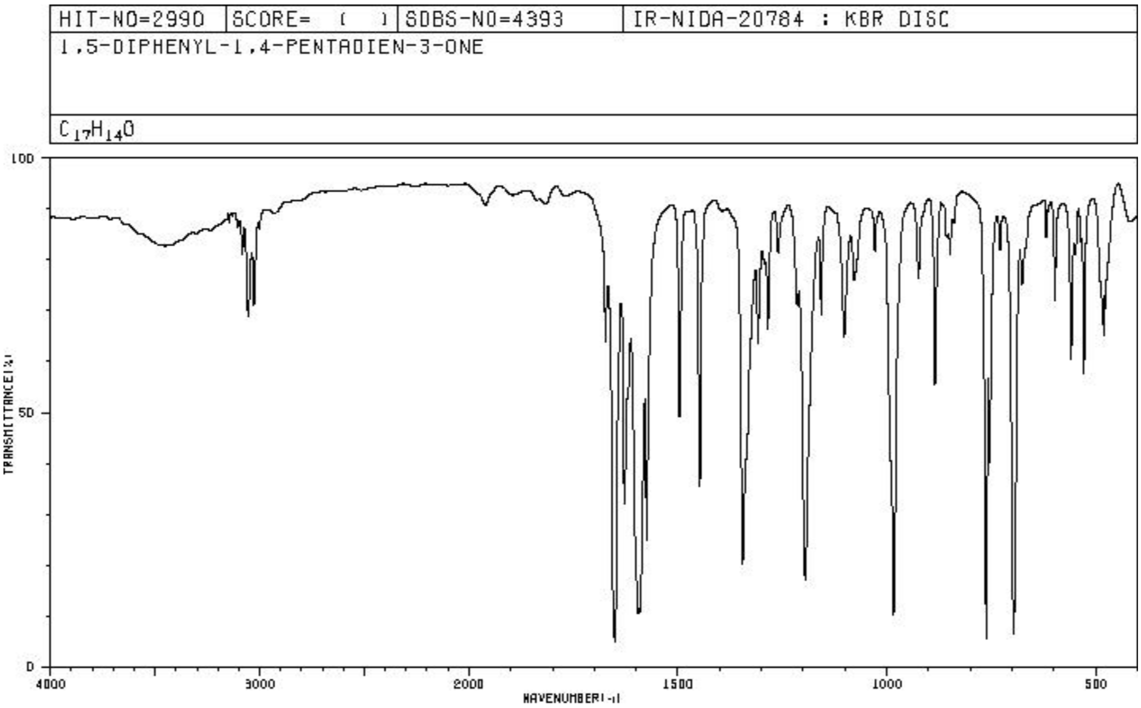
The less polar the substance is, the more it rises, because the polar substances are strongly attracted by the silica plate and therefore slowed down by it. But the benzaldehyde rose higher up than the dibenzalacetone even though it is more polar. The Rf values are 0.80 for benzaldehyde and 0.08 for dibenzalacetone. The third column (Mix) still has two spots, what indicates that there still are traces of the educt inside the mixture, which didn't react into the product.

Melting Point

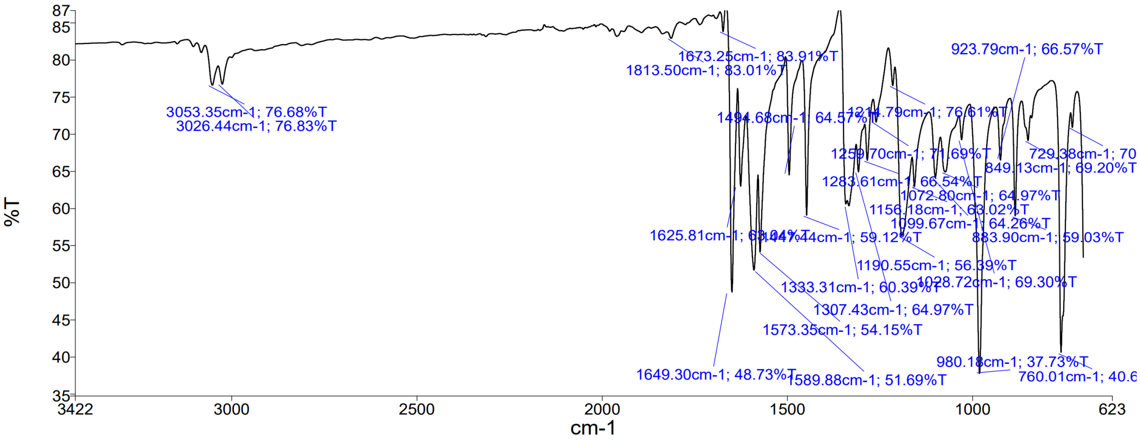
The melting point of the product was analyzed by filling a one ended melting point capillary tube with the yellow crystalline solid and using a melting point tester. For this the capillary tube was inserted in the melting point tester and the start temperature was set to 105 °C with 0.5°C/min increment. At 107.5 °C the crystalline solid started clumping together and at 109.0 °C the substance completely melted into a clear liquid. The literature value for the melting point of dibenzalacetone is 111 °C, so the measured value from this experiment is really near to it. The slightly lower melting temperate could be explained by the fact that there may are still traces of benzaldehyde with a much lower melting point of - 26°C in the substance.

Infrared Spectrum

This would be the literature values of the infrared spectrum of dibenzalacetone [8].



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



In the spectral analysis, the observed peaks 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
3053.35	Aromatic C-H stretch	76.68
3026.44	Aromatic C-H stretch	76.83
1673.25	C=O stretch (Conjugated enone)	83.91
1649.30	C=C stretch (Aromatic rings)	48.73
1625.81	C=C stretch (Conjugated system)	68.44
1589.88	C=C stretch (Conjugated system)	51.69
1573.35	Aromatic C=C stretch	54.15
1494.68	Aromatic C=C stretch	64.57
1190.55	Aromatic C-H stretch	56.39
980.18	Out of plane =C-H bending	37.73

Discussion

What is the purpose of NaOH in the reaction? The OH from the NaOH deprotonates the beta position of the acetone. That leaves behind a pair of electrons and that creates a negative charge on the carbon, what creates an enolate.

What is the difference between the aldol condensation and aldol reaction? The aldol reaction refers to the initial addition of the enolate ion of a ketone or aldehyde to a carbonyl group, forming a β-hydroxy ketone or aldehyde (aldol). The aldol condensation is a further reaction where the β-hydroxy product undergoes dehydration to form an α, β-unsaturated carbonyl compound. In summary, the aldol reaction is the addition step, and the aldol condensation includes this addition followed by the elimination of water.

Why is specifically benzaldehyde used? Are there side products in expected in the reactions? Benzaldehyde is commonly used in aldol condensations because it lacks α-hydrogens and therefore cannot form an enolate to self-condense. This reduces the likelihood of side reactions and simplifies the product mixture. However, side reactions such as polymerization or multiple aldol additions can still occur, potentially leading to complex mixtures of products.

Enolate was formed as an intermediate in the reactions. Those enolates can either attack the starting ketone (self-condensation) or the aldehyde (cross-condensation). Why is cross-condensation prefferably observed leading to the desired product? Cross-condensation is often the preferred pathway in reactions where the aldehyde, such as benzaldehyde, lacks α-hydrogens and is, therefore, unable to form an enolate. While the ketone could potentially react with another molecule of itself in a self-condensation reaction, the aldehyde is typically more electrophilic. This higher electrophilicity of the aldehyde makes it a more favorable target for the nucleophilic attack by the enolate generated from the ketone. The difference in electrophilicity drives the reaction towards the formation of a cross-condensation product, as the enolate from the ketone is more likely to attack the more electrophilic aldehyde, rather than another molecule of the less reactive ketone.

Is the aldol product or elimination product obtained and what parameters influence this selectivity? Whether the aldol addition product or the elimination product (α, β,-unsaturated compound) is obtained can depend on several factors like the type of catalyst used (acidic or basic conditions), the temperature of the reaction, the time allowed for a reaction to proceed and the removal of water from the reaction mixture (which can drive the reaction towards the elimination product.

How would the concern of possibly obtaining side products be addressed and how were the presence of possible side products experimentally checked? Concerns about side products are addressed through careful control of reaction conditions and purification steps. The presence of side products can be checked by techniques such as Thin Layer Chromatography (TLC) and IR spectroscopy.

References

[1] National Center for Biotechnology Information (2024). Pubchem compound summary for cid 640180, dibenzylideneacetone. `https://pubchem.ncbi.nlm.nih.gov/compound/Dibenzylideneacetone`

[2] Horst Meyer, Steffen Voss, Richard Walter. Palladium(0)-dibenzylidenaceton-komplexe. `https://patents.google.com/patent/DE102007018703`.

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[7] Dave Farina. Thin layer chromatography. `https://youtu.be/I4u_1ST7Ezk?si=6WwgzVh7VbJFOLkl`

[8] ChemicalBook. Infrared spectrum for compound with cas 538-58-9 (dibenzylideneacetone) ir1. `https://www.chemicalbook.com/SpectrumEN_538-58-9_IR1.htm`, 2024. Accessed 10, March 2024.

[9] Infrared spectroscopy absorption table. `https://chem.libretexts.org/Ancillary_Materials/Reference/Reference_Tables/Spectroscopic_Reference_Tables/Infrared_Spectroscopy_Absorption_Table`. Accessed on 2024-02-.

Appendix

List of H and P Phrases

- **H225**: Highly flammable liquid and vapor.
- **H319**: Causes serious eye irritation.
- **H336**: May cause drowsiness or dizziness.
- **H360**: May damage fertility or the unborn child.
- **H411**: Toxic to aquatic life with long lasting effects.
- **P210**: Keep away from heat/sparks/open flames/hot surfaces - No smoking.
- **P233**: Keep container tightly closed.
- **P240**: Ground/bond container and receiving equipment.
- **P241**: Use explosion-proof electrical/ventilating/lighting equipment.
- **P242**: Use only non-sparking tools.
- **P305+P351+P338**: If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.
- **P302+P352**: IF ON SKIN: Wash with plenty of water.
- **P304+P340**: IF INHALED: Remove person to fresh air and keep comfortable for breathing.
- **P312**: Call a POISON CENTER/doctor if you feel unwell.

Table from the Experiment Manual

Reactant	**MW**	**Equiv**	**Moles**	**Mass** g	**Volume** mL	**Purity** %
Acetone	58.08	1.00	11.46 mmol	0.666	0,011	
Benzaldehyde	106.12	2.05	23.5 mmol	2.494	2,375	
Sodium hydroxide	40.00	5.00	57,31 mmol	2.292	1,076	
Product	**MW**	**Yield**	**Moles**	**Mass**	**MP**	

Infrared Spectroscopy Absorption Table

The following table lists **infrared spectroscopy absorptions** by frequency regions.

4000-3000 cm^{-1}

3700-3584	medium	sharp	O-H	stretching	alcohol	free
3550-3200	strong	broad	O-H	stretching	alcohol	intermolecular bonded
3500- 3400	medium	-	N-H	stretching	primary amine	-
3400-3300 3330-3250	medium	-	N-H	stretching	aliphatic primary amine	-
3350-3310	medium	-	N-H	stretching	secondary amine	-
3300-2500	strong	broad	O-H	stretching	carboxylic acid	usually centered on 3000 cm^{-1}
3200-2700	weak	broad	O-H	stretching	alcohol	intramolecular bonded
3000-2800	strong	broad	N-H	stretching	amine salt	-

3000-2500 cm^{-1}

3333-3267	strong	sharp	C-H	stretching	alkyne	-
3100-3000	medium	-	C-H	stretching	alkene	-
3000-2840	medium	-	C-H	stretching	alkane	-
2830-2695	medium	-	C-H	stretching	aldehyde	doublet
2600-2550	weak	-	S-H	stretching	thiol	-

2400-2000 cm^{-1}

2349	strong	-	O=C=O	stretching	carbon dioxide	-
2275-2250	strong	broad	N=C=O	stretching	isocyanate	-
2260-2222	weak	-	C≡N	stretching	nitrile	-
2260-2190	weak	-	C≡C	stretching	alkyne	disubstituted
2175-2140	strong	-	S-C≡N	stretching	thiocyanate	-
2160-2120	strong	-	N=N=N	stretching	azide	-
2150	-	-	C=C=O	stretching	ketene	-
2145-2120	strong	-	N=C=N	stretching	carbodiimide	-
2140-2100	weak	-	C≡C	stretching	alkyne	monosubstituted
2140-1990	strong	-	N=C=S	stretching	isothiocyanate	-
2000-1900	medium	-	C=C=C	stretching	allene	-
2000	-	-	C=C=N	stretching	ketenimine	-

2000-1650 cm^{-1}

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

1870-1540 cm^{-1}

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

1670-1600 cm^{-1}

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

1600-1300 cm^{-1}

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

1400-1000 cm^{-1}

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

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

1000-650 cm^{-1}

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

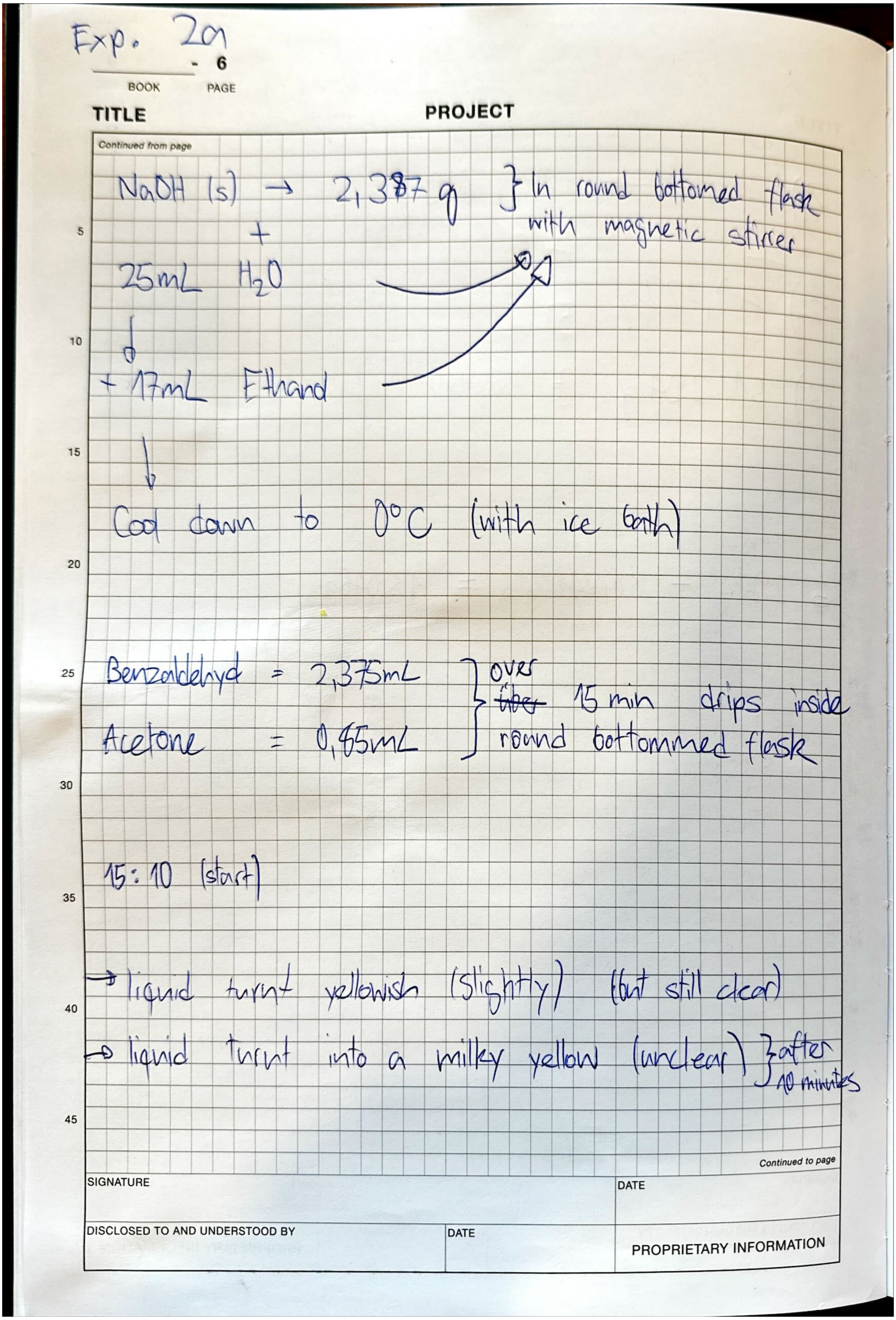
900-700 cm^{-1}

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

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

Contributors and Attributions

- OChemOnline



Exp. 2a - 6

BOOK PAGE

TITLE **PROJECT**

Continued from page

NaOH (s) → 2,387 g } In round bottomed flask with magnetic stirrer

\+

25mL H_2O

↓

\+ 17mL Ethanol

↓

Cool down to 0°C (with ice bath)

Benzaldehyd = 2,375mL
Acetone = 0,85mL
} over ~~über~~ 15 min drips inside round bottommed flask

15:10 (start)

→ liquid turnt yellowish (slightly) (but still clear)

→ liquid turnt into a milky yellow (unclear) } after 10 minutes

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SIGNATURE	DATE

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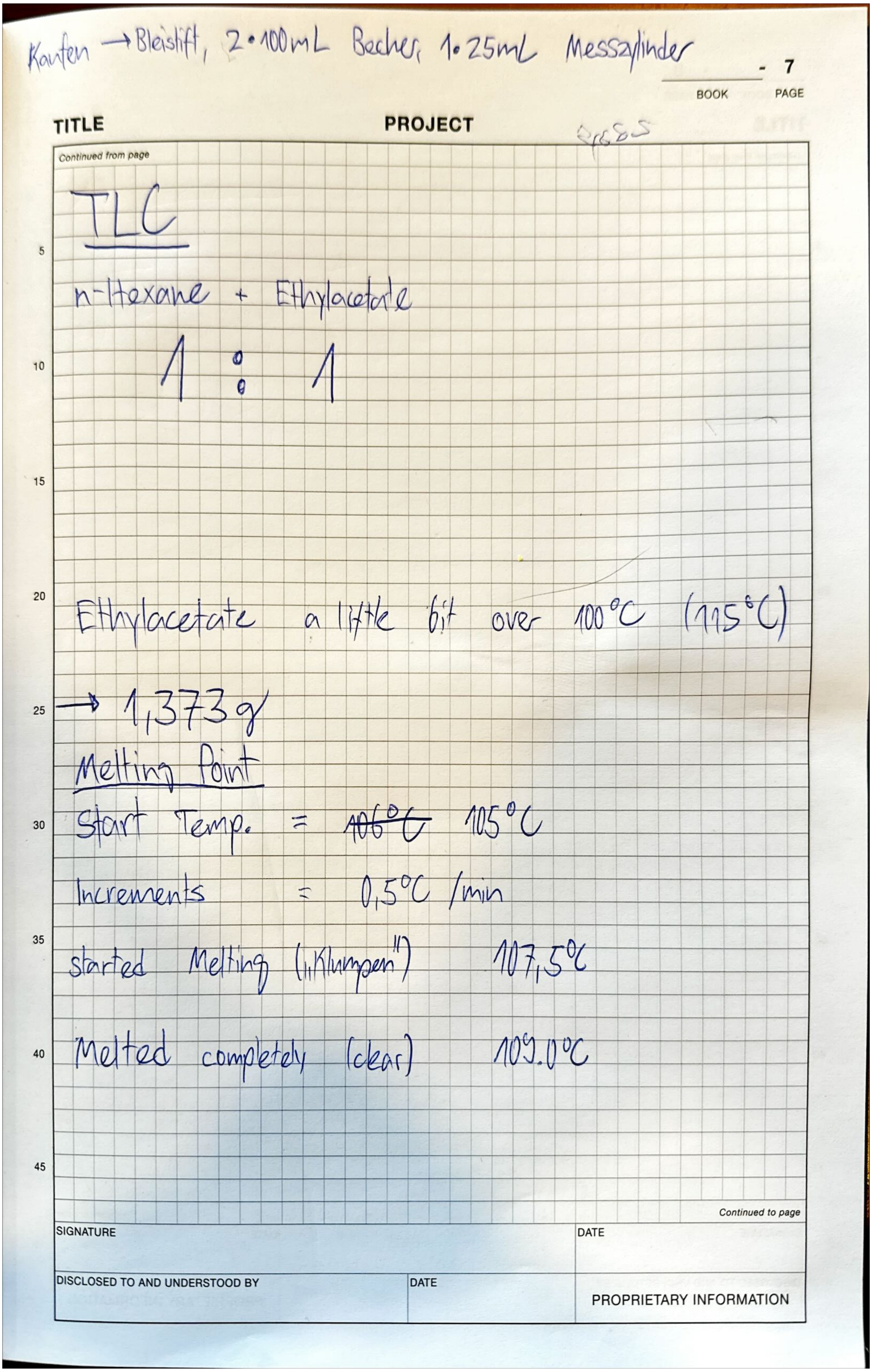

Kaufen → Bleistift, 2•100mL Becher, 1•25mL Messzylinder

- 7

BOOK PAGE

TITLE **PROJECT** 97,85

Continued from page

TLC

n-Hexane + Ethylacetate

1 : 1

Ethylacetate a little bit over 100°C (115°C)

→ 1,373 g

Melting Point

Start Temp. = ~~106°C~~ 105°C

Increments = 0,5°C /min

started Melting („Klumpen") 107,5°C

Melted completely (clear) 109.0°C

Continued to page

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