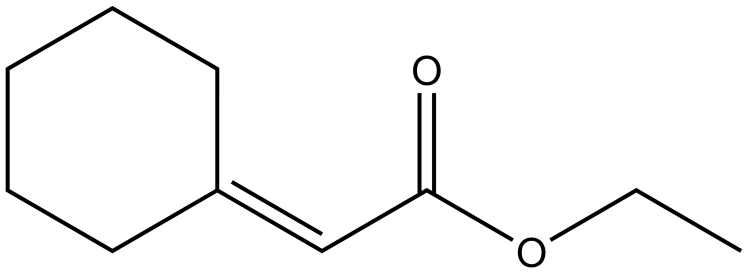
ETH zürich

Laboratory Course OACP I
Spring Semester 2024

The Synthesis of Ethyl 2-Cyclohexylidene Acetate by the Wittig (HWE) Reaction



Loganathan Visva vloganathan@student.ethz.ch
Interdisciplinary Sciences Biochemical-Physical Major

Teaching Assistant
Neves Vieira Adriana adriana.vieira@org.chem.ethz.ch

Abstract The objective of this experiment is to synthesize Ethyl 2-cyclohexylideneacetate and analyze the yield of the product. The synthesis is performed through the Wittig (HWE) reaction of Triethyl phosphonoacetate with cyclohexanone and the usage of magnesium chloride and triethylamine as catalyst. The substance was purified by flash column chromatography. The success of the experiment is determined by thin layer chromatography and infrared spectrum measurements. The product was a pale yellowish clear liquid with a weight of 1.04 g, what would be a yield of about 61%.

Zürich, April 5, 2024

V. Loganathan
.....
Loganathan Visva

Adriana

Introduction

Ethyl 2-cyclohexylideneacetate is an organic compound characterized by the presence of a cyclohexylidene group bonded to an ester functional group. At room temperature, it appears as a colorless to pale yellow liquid. It has a characteristic odor that smells fruity or like floral scents [1]. This compound's structure features a cyclohexene ring conjugated with an ester, which contributes to its chemical reactivity and solubility characteristics. It is not typically soluble in water but shows good solubility in organic solvents like dichlormethane and ethylacetate [2].

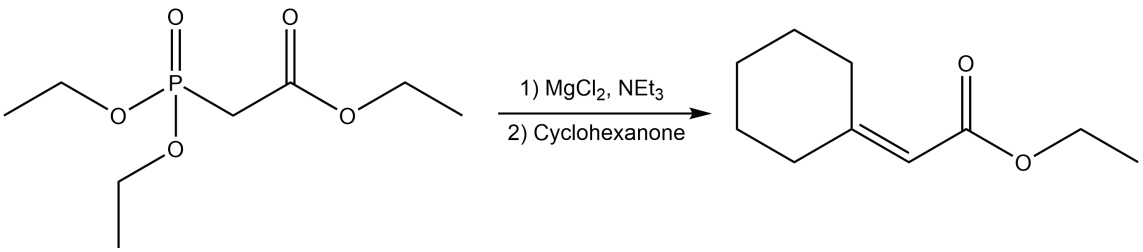
In industrial applications, Ethyl 2-cyclohexylideneacetate is primarily utilized as an intermediate in pharmaceutical manufacturing [3]. Its chemical properties make it suitable for this role, where it is involved in the synthesis of various medicinal products. Its unique odor profile could also be utilized in the formulation of fragrances, scents and perfumes.

Material Properties

In the described experiment, Ethyl 2-cyclohexylideneacetate is produced through the Horner-Wadsworth-Emmons (HWE) reaction.

This is a modified version of the Wittig reaction, that utilized phosphonate-stabilized carbanions [4]. These carbanions, in comparison to ylides in the Wittig reaction, exhibit greater nucleophilicity and reduced basicity, allowing for effective alkylation. Ylides are a class of organic compounds that contain a positively charged atom (usually phosphorus, sulfur, or nitrogen) directly bonded to a negatively charged carbon atom. This results in a neutral molecule that has both positive and negative charges, which are separated by a covalent bond. Additionally, the byproduct formed in this reaction, a dialkylphosphate salt, is readily separable from the desired product via simple aqueous extraction.

This synthesis involves reacting triethylphosphonoacetate with cyclohexanone, utilizing triethylamine as a base to facilitate the reaction.



Substance	GHS Hazard Pictogram	H and P Phrases
Cyclohexanone [5]		H226, H302, H332, H312, H315, H318, H335-P210, P280, P301, P312, P303, P361, P353, P304, P340, P305, P351, P338
Triethyl phosphonoacetate [6]		H319, H411-P264, P273, P280, P305, P351, P338, P337, P313, P391
Triethylamine [7]		H225, H302, H331, H311, H314, H318, H335-P210, P280, P301, P312, P303, P361, P353, P304, P340, P310, P305, P351, P338
Tetrahydrofuran [8]		H225, H302, H319, H351, H336, H335-P202, P210, P233, P301, P312, P305, P351, P338, P308, P313
Hydrochloric acid [9]		H225, H290, H315, H319, H350, H335, H314, H318-P202, P210, P233, P303, P361, P353, P305, P351, P338, P308, P313
Ethyl ether [10]		H224, H302, H336-P210, P233, P240, P241, P301, P312, P403
Ethylcyclohexylideneacetate [1]		H302, H315, H319, H335, P261, P264, P264+P265, P270, P271, P280, P301+P317, P302+P352, P304+P340, P305+P351+P338, P319, P321, P330, P332+P317, P337+P317, P362+P364, P403+P233, P405, P501
Magnesium chloride [11]	-	-
Sodium chloride [12]	-	-
Magnesium sulfate [13]	-	-

Substance	Molar Mass [g/mol]	Density [g/mL]	Melting Point [°C]	Boiling Point [°C]
Cyclohexanone [5]	98.15	0.9478	-47	155.65
Triethyl phosphonoacetate [6]	224.19	1.0917	92-93	261
Triethylamine [7]	101.19	0.7255	-114.7	88.8
Tetrahydrofuran [8]	72.107	0.8876	-108.4	66
Hydrochloric acid [9]	36.46	1.09620	-114	-85
Ethyl ether [10]	74.12	0.713	-116.3	34.6
Ethylcyclohexylideneacetate [1]	168.23	1.0	6	137
Magnesium chloride [11]	95.211	2.32	714	1,412
Sodium chloride [12]	58.443	2.17	800.7	1,465
Magnesium sulfate [13]	120.366 (anhydrous)	2.66 (anhydrous)	1,124 (anhydrous, decomposes)	1,124 (anhydrous, decomposes)
	246.47 (heptahydrate)	1.68 (heptahydrate)	150 (heptahydrate, decomposes)	150 (heptahydrate, decomposes)

Safety Assessment

In the lab, each chemical demands specific safety measures. Cyclohexanone and triethylamine are flammable, with triethylamine also being corrosive. Tetrahydrofuran is not only flammable but forms hazardous peroxides. Hydrochloric acid can cause severe burns, and both it and ethyl ether are harmful when inhaled. Magnesium chloride and sodium chloride are less hazardous but can irritate the eyes.

Also the synthesis should be conducted in a fume hood, because of the harmful vapors. Triethylamine should be added slowly to the mixture because it warms up and that shouldn't be to drastic. Stir the mixture for at least 10 minutes before adding the cyclohexanone drop wise.

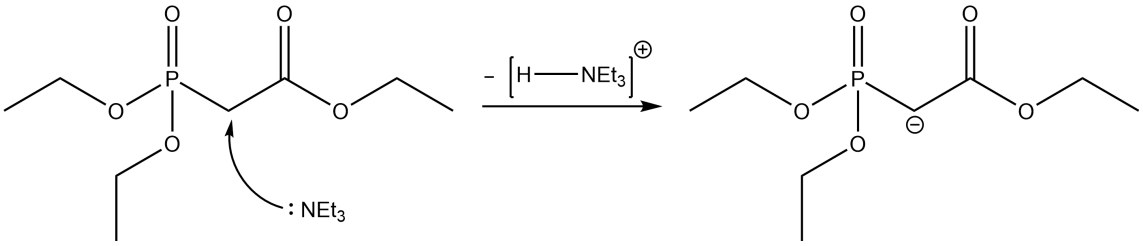
The handling of each requires appropriate personal protective equipment, cautious storage, and clear protocols for exposure response and spill management. Always adhere to detailed guidelines as provided by their respective Safety Data Sheets for safe laboratory practices.
For avoiding any health issues 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

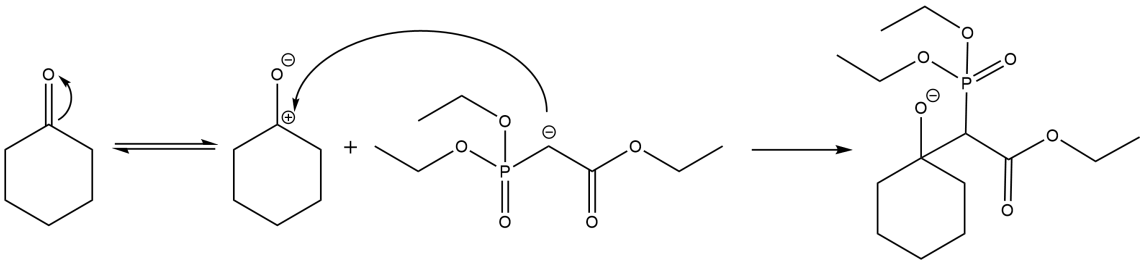
Hydrochloric acid should be disposed into the aqueous acidic waste. Cyclohexanone, ethylether, tetrahydrofuran and triethylamine are organic solvents and should be disposed in the solvent waste. Magnesium chloride and Sodium chloride should be either disposed in the solid waste or dissolved and put in the chlorinated solvent waste. Magnesium sulfate should also be put in the solid waste or dissolved and put in the solvent waste.

Reaction Mechanism

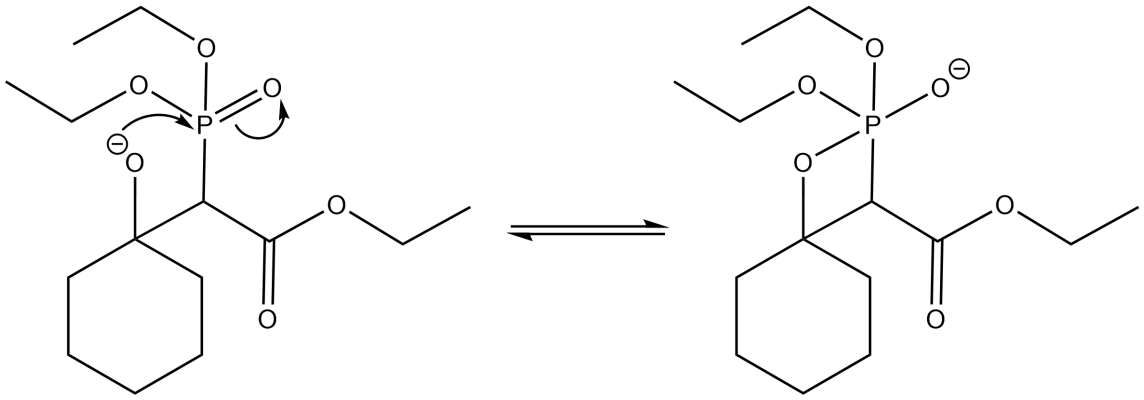
In this synthesis, the HWE reaction, which is an modification of the Wittig reaction, is utilised. Initially, the triethylamine acts as a base to deprotonate triethylphosphonoacetate. This action results in a carbanion that is negatively charged.



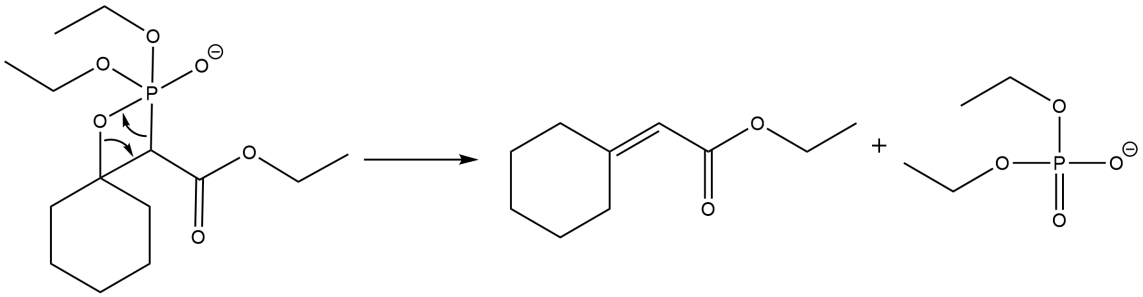
The carbanion is a nucleophile that attacks the electrophilic carbonyl group from the cyclohexanone. That results in the connection of both molecules.



The double bond's electrons between the phosphorus and oxygen atoms shift towards the oxygen, leading to a positive charge on the phosphorus. This allows a nucleophilic attack by the negatively charged oxygen atom of the cyclohexanone molecule.



The Bayer ring strain is really high on the quadratic ring with the phosphorus (P), the oxygen (O) and the two carbon (C) atoms. That leads to the redistribution of the electrons from the C-P and the C-O bonds. That enables the formation of a C-C double bond and a P-O double bond. This rearrangement results in the release of the end product ethyl 2-cyclohexylideneacetate and the byproduct diethylphosphate.



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 52%. This reaction is not really atom efficient.

$$\mathrm{AE} = \frac{M_{\mathrm{Product}}}{M_{\mathrm{Reagents}}} = \frac{168.23\ \mathrm{g/mol}}{224.19\ \mathrm{g/mol} + 98.15\ \mathrm{g/mol}} \approx 52.19\%$$

Experimental

Procedure

Ethyl 2-cyclohexylideneacetate is produced through the Horner-Wadsworth-Emmons (HWE) reaction. The synthesis process utilizes triethylphosphonoacetate and cyclohexanone as reactants, with triethylamine is utilized as a basic catalyst. The product was a pale yellowish clear liquid (1.04 g, 6.182 mmol) with a yield of about 61%.

Observation

For the synthesis of the ethyl 2-cyclohexylideneacetate, a magnetic stir bar was placed in a 100 mL three-neck flask filled with anhydrous magnesium chloride (10 mmol, 0.952g, 1.00 equiv.). A nitrogen supply was connected to one neck of the flask, a dropping funnel was installed on an other neck and on the third neck a septum was put on. With a nitrogen atmosphere was created inside the three-necked flask. That was necessary because in this synthesis tetrahydrofuran was utilized. THF should be kept under a nitrogen atmosphere to prevent peroxide formation. THF can autoxidize upon exposure to air, especially oxygen, leading to the accumulation of dangerous peroxides, which can be explosive. A syringe was utilized to insert the tetrahydrofuran (10 ml) and triethyl phosphonoacetate (10 mmol, 2.1 mL, 1.00 equiv.) through the septum. The mixture was stirred for 5 minutes at room temperature. Next, triethylamine (10 mmol, 1.4 mL, 1.00 equiv.) was slowly added with a syringe through the septum. After an additional 10 minutes of stirring at room temperature, cyclohexanone (10 mmol, 1.0 mL, 1.00 equiv.) was dripped slowly into the mixture in the same way. The mixture became a thick, viscous, milky-white liquid after some time. That happened because the nitrogen flow was too high, what caused the evaporation of the THF, so additional 10 mL of THF were added. That caused the milky-white solution to become thin-liquidy again. After that the nitrogen flow was stopped and the whole system was sealed, all potential escape points for the nitrogen were securely closed up. The resultant mixture was then stirred continuously at room temperature for a duration of 20 hours under the nitrogen atmosphere.

After 20 hours, 10 mL of a 2 M hydrochloric acid were added slowly into the mixture. Then the mixture was transferred into a separation funnel and shaken. A organic phase with lower density accumulated on top, lower down a aqueous phase accumulated. Both phases were separated into flasks. The aqueous phase was transferred into the separation funnel again and 10 mL of ethylacetate were added and the mixture was shaken. That caused again a separate organic phase on top of a aqueous one. The organic phase was collected in the erlenmeyer flask. This process was performed three times. The combined organic layers were washed with a brine solution. The organic phase was subsequently dried over magnesium sulfate and filtered through a 50 mL filter crucible. The residue was disposed, the ethyl 2-cyclohexylidene was inside the mother liquors but still with

solvent. With the help of a rotary evaporator the solvent got removed and a white crystalline solid remained.

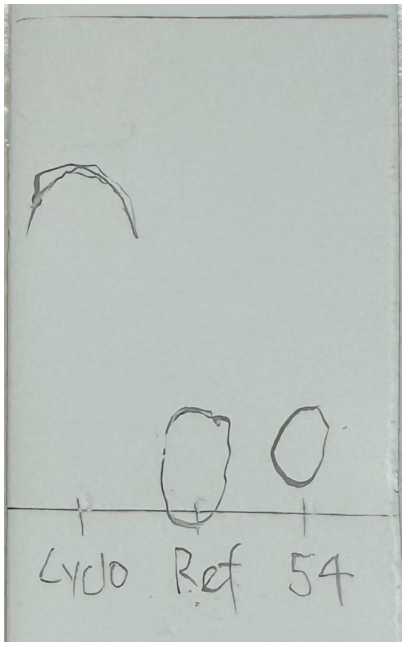
A chromatography column was filled up with a sand layer, a silica layer and a sand layer again. Then the crystalline solid was then put on top of the sand layer. Then on top of the crystalline solid another sand layer was put ontop. A 20 to 1 n-Hexane ethylacetate mixture was produced. This mixture was transferred into the chromatography column. The column was connected to the nitrogen flow, so pressure could be applied to it. Then a flash column chromatography was performed and after that the solution containing the desired product was put in a rotatory evaporator.

Characterisation

TLC and Flash Column Chromatography

A flash column chromatography was conducted to purify the 2-cyclohexylidene acetate. A flash column chromatography is just like a longer reverse thin layer chromatography for the entire mixture. In whole there were 54 flasks that were filled. Some TLC's were conducted, that showed that there was no product inside flask 1 to 11. Flask 1 to 21 had the product. Flask 22-26 had some impurity, maybe a byproduct. Flask 27 to flask 54 again only had the product. So the content of flask 1 to 21 and flask 27 to 54 were put together into a 1 L round bottomed flask. It appeared as a clear liquid.

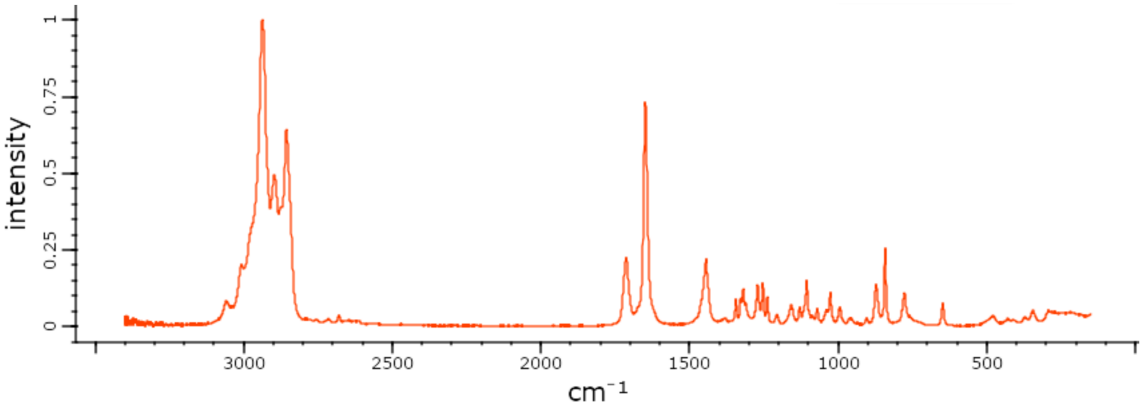
A final TLC was conducted with the liquid in the round bottomed flask. In this TLC, a silicate plate was used and there were three columns. The first column (Cyclo) had only one educt, the cyclohexanone. For the second column (Ref) n-Hexane was put into the flask where the white crystalline substance was originally to take up the residues from the substance. This acted as a reference. The third column (54) only had some drops from flask 54 on it. The solvent consisted of 100% n-Hexane.



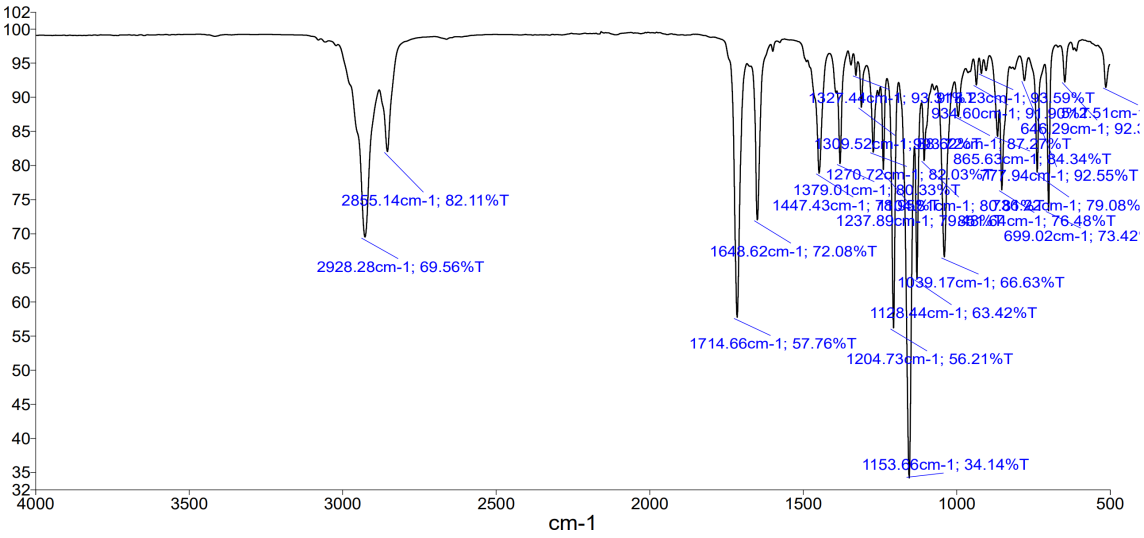
This TLC plate shows that there is no more cyclohexanone in the white crystalline solid and also not in flask 54. The Rf values are 0.66 for cyclohexanone, 0.1 for the white cristalline solid and 0.14 for flask 54.

IR Spectrum

This would be the literature values of the infrared spectrum of ethyl 2-cyclohexylidene acetate [14].



And this would be the measured infrared spectrum of the synthesized ethyl 2-cyclohexylidene acetate from the author.



In the spectral analysis, the observed peaks align with those documented in the literature, although they are weaker in intensity [15]. 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
2855.14	C-H Stretch (Alkane)	82.11
2928.28	C-H Stretch (Alkane)	69.56
1714.66	C=O Stretch (Carbonyl)	57.76
1648.62	C=C Stretch (Alkene)	72.08
1447.43	C-H Bending	78.95
1379.01	C-H Bending	80.33
1327.44	C-H Bending	93.31
1237.89	C-H Bending	79.48
1204.73	C-O Stretch (Ester)	56.21
1153.66	C-O Stretch (Ester/Ether/Alcohol)	34.14
1128.44	C-O Stretch (Aliphatic Ether)	63.42
1039.17	Unknown/Impurity	66.63

References

[1] Ethyl cyclohexylideneacetate. https://pubchem.ncbi.nlm.nih.gov/compound/Ethyl-cyclohexylideneacetate. Accessed: 2024-03-05.

[2] Ethyl cyclohexylideneacetate. https://www.fishersci.ch/shop/products/ethyl-cyclohexylideneacetate-98-thermo-scientific/11476057. Accessed: 2024-03-05.

[3] Ethyl cyclohexylideneacetate, 98 https://www.thermofisher.com/order/catalog/product/B25073.22. Accessed: 2024-03-05.

[4] Dave Farina. Horner-wadsworth-emmons reaction. https://youtu.be/5lIw_MhirBM?si=hNBwibIrDl20q8O6

[5] Cyclohexanone. https://www.matmake.com/s/cyclohexanone. Accessed: 2024-03-04.

[6] Triethyl phosphonoacetate. https://en.wikipedia.org/wiki/Triethyl_phosphonoacetate Accessed: 2024-03-04.

[7] Triethylamine. https://www.chemeurope.com/en/encyclopedia/Triethylamine.html. Accessed: 2024-03-04.

[8] Tetrahydrofuran (thf). https://www.sciencemadness.org/smwiki/index.php/Tetrahydrofuran. Accessed: 2024-03-04.

[9] Hydrochloric acid - the definitive guide. https://www.biologydictionary.net/hydrochloric-acid/ Accessed: 2024-03-04.

[10] Diethyl ether. https://en.wikipedia.org/wiki/Diethyl_ether Accessed: 2024-03-04.

[11] Magnesium chloride. https://en.wikipedia.org/wiki/Magnesium_chloride. Accessed: 2024-03-04.

[12] Sodium chloride. https://en.wikipedia.org/wiki/Sodium_chloride. Accessed: 2024-03-04.

[13] Magnesium sulfate. https://en.wikipedia.org/wiki/Magnesium_sulfate. Accessed: 2024-03-04.

[14] Wiley-VCH Verlag GmbH Co. KGaA. Ir spectrum and properties of ethyl cyclohexylideneacetate. In IR - Industrial Chemicals, Pure Organic Compounds - Wiley, 2024. Accessed on KnowItAllAnywhere on March 31, 2024.

[15] 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.
- H302: Harmful if swallowed.
- H312: Harmful in contact with skin.
- H315: Causes skin irritation.
- H318: Causes serious eye damage.
- H319: Causes serious eye irritation.
- H331: Toxic if inhaled.
- H332: Harmful if inhaled.
- H335: May cause respiratory irritation.
- H336: May cause drowsiness or dizziness.
- H411: Toxic to aquatic life with long lasting effects.
- H290: May be corrosive to metals.
- H314: Causes severe skin burns and eye damage.
- H351: Suspected of causing cancer.
- 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.
- P243: Take precautionary measures against static discharge.
- P264: Wash skin thoroughly after handling.
- P273: Avoid release to the environment.
- P280: Wear protective gloves/protective clothing/eye protection/face protection.
- P301+P312: IF SWALLOWED: Call a POISON CENTER or doctor/physician if you feel unwell.
- P303+P361+P353: IF ON SKIN (or hair): Remove/Take off immediately all contaminated clothing. Rinse skin with water/shower.
- P304+P340: IF INHALED: Remove victim to fresh air and keep at rest in a position 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.
- P337+P313: If eye irritation persists: Get medical advice/attention.
- P403+P233: Store in a well-ventilated place. Keep container tightly closed.
- P405: Store locked up.
- P501: Dispose of contents/container in accordance with local/regional/national/international regulations.

Table from the Experiment Manual

Reactant	MW	Equiv	Moles	Mass g	Volume mL	Purity
Cyclohexanone	98.15	1.00	10 mmol	0.9815	1.0356	-
Triethylphosphonoacetate	224.19	1.00	10 mmol	2.2419	2.0550	-
Triethylamine	101.19	1.00	10 mmol	1.0119	1.3948	-
Magnesium chloride	95.211	1.00	10 mmol	0.95211	-	-
Product	**MW**	**Yield**	**Moles**	**Mass**		
Ethyl-2-Cyclohexylidenacetat	168.23					

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

- 8
BOOK PAGE

TITLE PROJECT

Continued from page

Exp. 3a

Safety

3-Ethylphosphate acetate
Cyclohexanone
Tetrahydrofuran

Don't open the nitrogen valves too quickly + over pressure adapter

Diethylether → Can build peroxides (can happen with light and oxygen!) / highly volatile
→ Don't sniff them!

Triethylamin smells like fish

→ 3-neck flask } -septum -nitrogen supply -dropping funnel

$MgCl_2$ 0,952g
THF 10mL
Triethyl Phosphonoacetate 2,05mL

5 min stirr

Triethylamin 1,4 mL

10 min stirr

Continued to page

SIGNATURE DATE

DISCLOSED TO AND UNDERSTOOD BY DATE PROPRIETARY INFORMATION

- 9
BOOK PAGE

TITLE PROJECT

Continued from page

+10mL THF (the previous THF evaporated, because the nitrogen flow was too high)

Cyclohexanone 1,03 mL

Experimental

Procedure

Really short description + Ausbeute/yield + mass + colour of compound + state (liquid/solid)

Observation

Describe everything and explain everything

Continued to page

SIGNATURE DATE

DISCLOSED TO AND UNDERSTOOD BY DATE

PROPRIETARY INFORMATION

- 10
BOOK PAGE

TITLE PROJECT

Continued from page

After 20h

10mL 2M HCl

~~Ethylacetate~~

Separation funnel

10mL x 3 mal Ethylacetate in aqueous phase

Sodium chloride + organic phase

Dry it over $MgSO_4$ and filter it

100% n-Hexane TLC

Continued to page

SIGNATURE DATE

DISCLOSED TO AND UNDERSTOOD BY DATE PROPRIETARY INFORMATION

- 11

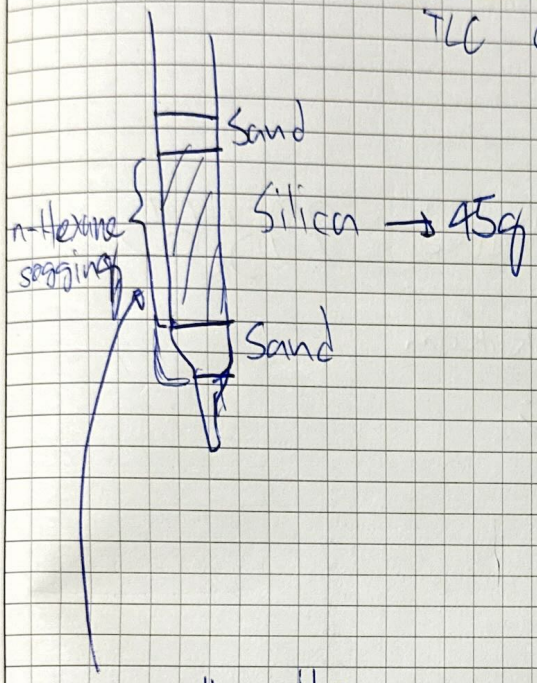
BOOK PAGE

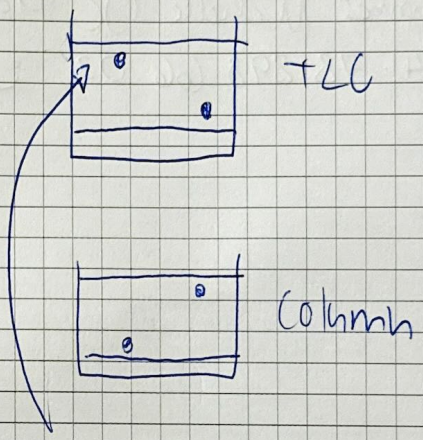
TITLE PROJECT

Continued from page

Column Chromatography

TLC done multiple times





the smaller the silica - „Schicht" the faster the flash column chromatography but also the dirtier the product

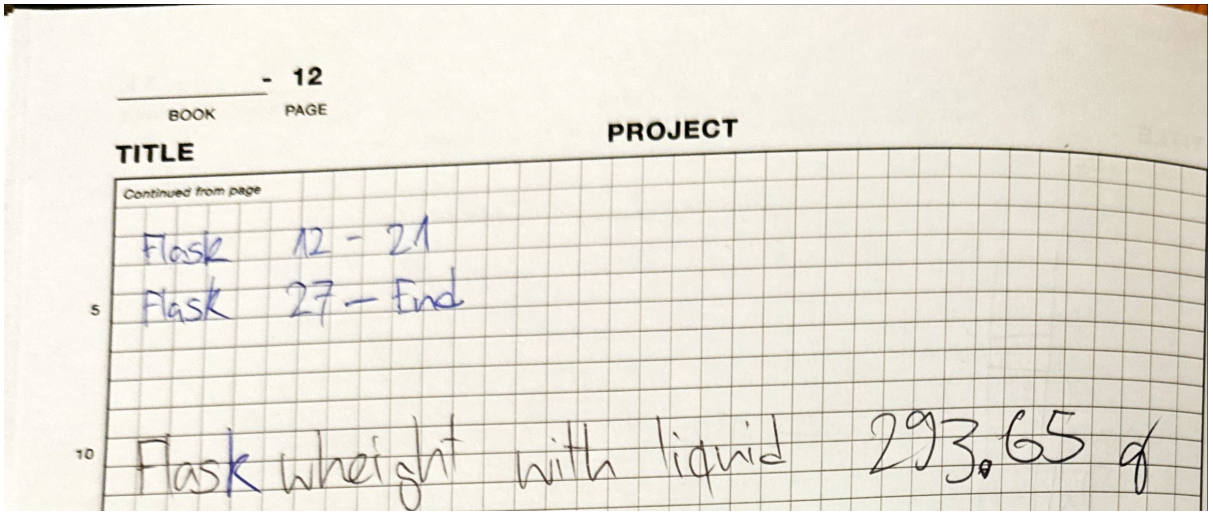
the highest one will come out the first

1L n-Hexane + 50mL Ethylacetat

Continued to page

SIGNATURE DATE

DISCLOSED TO AND UNDERSTOOD BY DATE PROPRIETARY INFORMATION


- 12
BOOK
PAGE
TITLE
PROJECT
Continued from page
Flask 12 - 21
Flask 27 - End
Flask wheight with liquid 293.65 g