Cosmetic chemistry

Synthesis of dibezalacetone

Table 1 Amount of reactants and products

Chemical	n (mol)	ν	ξ (mol)	Reactant	M ($g.mol^{-1}$)	m (g)	ρ(20°C) ($g.cm^{-3}$)	V (cm^3)
Sodium hydroxide	0,0250				39,999	1,00		
Bezaldehyde	0,0567	2	0,0284	excess	106,120	6,02	1,0440	5,8
Acetone	0,0270	1	0,0270	limiting	58,080	1,57	0,7899	2,0
Dibenzalacetone	0,0270	1			234,290	6,33		

Table 2 percentage yield of dibenzalacetone

Theoretical yield	(g)	6,33
Practical yield	(g)	
Percentage yield	(%)	0,0

ρ(H_2O, 20°C)	**($g.cm^{-3}$)**	0,9982

Legend

n	amount of a substance
ν	stoichiometric coefficient
ξ	extent of reaction
M	molar mass
m	mass
ρ(20°C)	density
V	volume

Laboratory manual

Preparation of dibenzalacetone (1,5-Diphenyl-1,4-pentadien-3-one)

Introduction

The aim of the work is the preparation of dibenzalacetone (1,5-Diphenyl-1,4-pentadien-3-one).

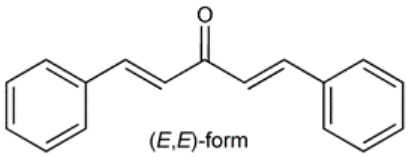
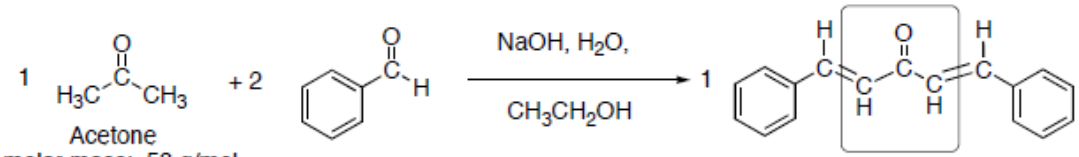


Fig.1.:*Dibenzalacetone*[2]

The compound dibenzalacetone (DBA) is used as an **active UV-blocking ingredient in sunscreens**.[1] It is also used as an anti-inflammatory agent, and to relieve pain and swelling. In addition, DBA is also useful in the production of organometallic ligands, and derivatives of DBA may find use in polymer reactions[4,5].
It has two double bonds in the aliphatic chain and exists in three geometric isomers: cis-cis, cis-trans, and trans-trans. It can be prepared by condensation of benzaldehyde and acetone in water or in ethanol in the presence of sodium hydroxide, yielding exclusively the trans-trans isomer.[6] The melting points are 110 to 111 °C for the (trans-trans isomer) and 60 °C for the (cis-trans isomer). It is insoluble in water, dissolving in ethanol.

The preparation consists of the base-catalysed aldol condensation of benzaldehyde with acetone (the so-called Claisen-Schmidt reaction)[3]:



Acetone
molar mass: 58 g/mol
density: 0.79g/mL
limiting reagent

Benzaldehyde
molar mass: 106 g/mol
density: 1.04 g/mL
an excess will be used

original acetone carbons

Dibenzalacetone
molar mass: 234 g/mol
melting point: somewhere between 80 and 120°C

Assignment

1. Familiarize yourself with the dangerous properties of the substances you will be working with.

2. Prepare all tools and chemicals for the job.
3. Prepare solutions and mixtures as indicated in the instructions (re-calculate substance quantity in ***mol*** *on* ***grams*** *or on* ***mililitre***).
4. Determine the melting point of the product.
5. Calculate the theoretical and relative yield of the product.
6. Complete the worksheet.
7. Answer the questions in the worksheet.

Materials

Analytical balance, weighting boat, chemical spoon, beakers (150 ml, 50 ml), stirring rod, pipette (5 ml), Büchner funnel, suction flask, ice, Petri dish or hourglass, test tube, glass marker pen, glassware labels

Chemicals

1. sodium hydroxide
2. benzaldehyde
3. acetone
4. 80% ethanol
5. distilled water

Preparing of solutions

1. Calculate the mass in ***g*** of **sodium hydroxide** in case we need to use **0.025 mol of sodium hydroxide** to prepare the product.
2. Calculate the volume of **benzaldehyde** in ***ml*** if, to prepare the product, we need to add **0.0567 mol of benzaldehyde** dissolved in 40 ml of ethanol to the reaction.
3. Calculate the volume of **acetone** in ***ml*** if we need to add **0.027 mol of acetone** to the reaction to prepare the product.

Procedure

Preparation of dibenzalacetone (1,5-Diphenyl-1,4-pentadien-3-one)

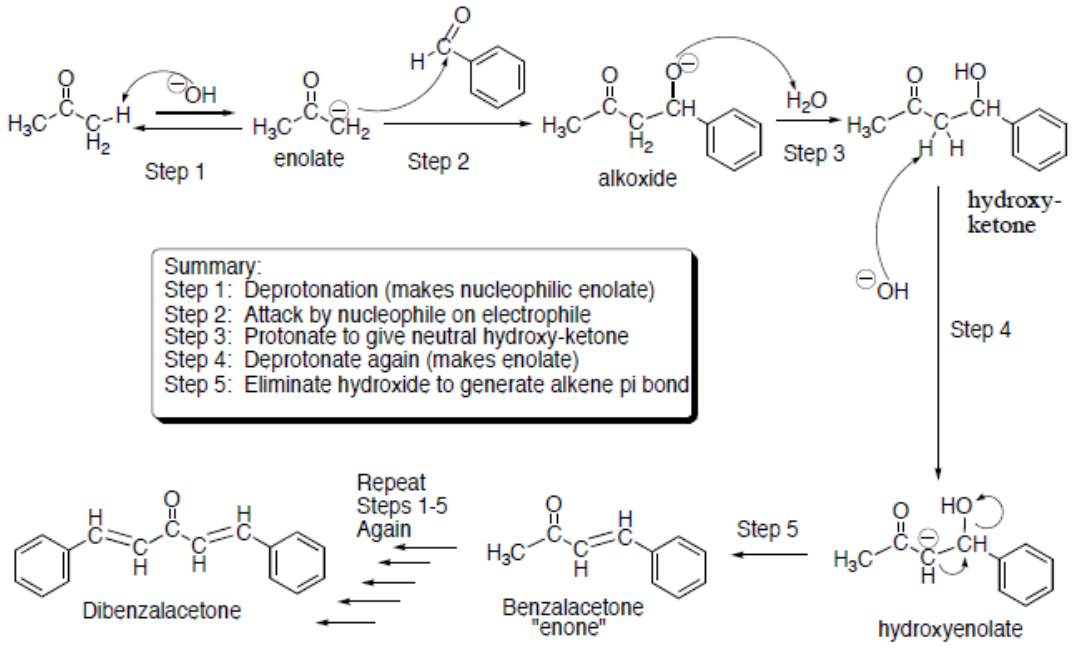
1. Dissolve **0.025 mol** of sodium hydroxide in 20 ml of distilled water in a beaker (150 ml) and cool the mixture to room temperature.
2. With constant stirring add **0.0567 mol** of benzaldehyde dissolved in 40 ml of ethanol.
3. Then **slowly** add **0.027 mol** of acetone with a pipette while constantly stirring. **First, a yellow solution is formed,** and a few minutes after adding the entire amount of acetone, a thick **yellow precipitate** falls out.
4. **Stir the reaction mixture for another 20 minutes.**
5. Then add 40 ml of distilled water. Mix the suspension.
6. Aspirate the product on a Büchner funnel and wash it with 50 ml of distilled water (to remove excess sodium hydroxide).

7. **<u>The crude product</u> is purified by crystallization from boiling ethanol** - so that the **substance is dissolved in boiling ethanol (volume of ethanol is 25 ml).** (Boiling point of ethanol is 78.3 °C (1013 hPa)).
8. Add 4 ml of distilled water during boiling to the solution and then cool the mixture first with cold water and then with an ice bath.
9. The crystals of the product pour on a Büchner funnel and washed with 80% ethanol, which we cool beforehand in an ice bath.
10. Allow the **filter cake with the product** to air dry on a Büchner funnel and then transfer it to a Petri dish or hourglass.
11. The product is a bright yellow crystalline substance. Determine the melting point of the product.
12. Calculate the theoretical and relative yield of the product.

Duration of analysis:
Preparing solutions and mixtures as indicated in the instructions and re-calculating substance quantity in ***<u>mol</u>*** *on* ***grams*** *or on* ***mililitre*** takes about 20 minutes.
Preparation of substance takes about 50 minutes, cleaning of the product by re-crystallization takes 20 minutes and drying of product takes about 20 minutes.

Accuracy:
Verification of the success of the work is carried out by means of determine the melting point of the product.

Use of method:
Method is suitable for understanding the preparation of compound by means of Claisen-Schmidt reaction - **<u>The base-catalysed aldol condensation of benzaldehyde with acetone</u>**[3], which mechanism is shown in the picture:



Safety Information

<table>
<tr><th>Chemical</th><th>Safety Information</th></tr>
<tr><td></td><td>Web: https://www.merckmillipore.com/SK/sk/product/Sodium-hydroxide,MDA_CHEM-106462</td></tr>
<tr><td></td><td>
<table>
<tr><th>CAS #</th><th>EC Number</th><th>Hill Formula</th><th>Chemical Formula</th><th>Molar Mass</th></tr>
<tr><td>1310-73-2</td><td>215-185-5</td><td>HNaO</td><td>NaOH</td><td>40 g/mol</td></tr>
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</td></tr>
<tr><td>sodium hydroxide</td><td>Hazard Statement(s)
H290: May be corrosive to metals.
H314: Causes severe skin burns and eye damage.
Precautionary Statement(s)
P234: Keep only in the original packaging.
P260: Do not breathe dust.
P280: Wear protective gloves/ protective clothing/ eye protection/ face protection/ hearing protection.
P303 + P361 + P353: IN CONTACT WITH SKIN (or hair): Remove all contaminated articles of clothing. Rinse skin immediately with water.
P304 + P340 + P310: AFTER INHALATION: Move the person to fresh air and allow him/her to breathe comfortably. Call the TOXICOLOGICAL INFORMATION CENTER/physician immediately.
P305 + P351 + P338: AFTER COVERING EYES: Gently flush with water for several minutes. If you use contact lenses and it is possible, remove them. Continue rinsing.</td></tr>
<tr><td></td><td>
<table>
<tr><th>CAS #</th><th>EC Number</th><th>Hill Formula</th><th>Chemical Formula</th><th>Molar Mass</th></tr>
<tr><td>100-52-7</td><td>202-860-4</td><td>C_7H_6O</td><td>C_6H_5CHO</td><td>106,12 g/mol</td></tr>
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</td></tr>
<tr><td>benzaldehyde</td><td>Hazard Statement(s)
H302 + H332: Harmful if swallowed or inhaled.
H315: Irritating to skin.
H319: Causes serious eye irritation.
H335: May cause respiratory irritation.
H360D: May cause harm to the unborn child.
H411: Toxic to aquatic life, with long-lasting effects.
Precautionary Statement(s)
P273: Avoid release into the environment.
P301 + P312: AFTER INGESTION: In case of health problems, call the NATIONAL TOXICOLOGICAL INFORMATION CENTRE/physician.
P302 + P352: IN CONTACT WITH SKIN: Wash with plenty of water.
P304 + P340 + P312: AFTER INHALATION: Move the person to fresh air and allow him/her to breathe comfortably. In case of health problems, call the NATIONAL TOXICOLOGICAL INFORMATION CENTER/physician.
P305 + P351 + P338: AFTER COVERING EYES: Gently flush with water for several minutes. If you use contact lenses and it is possible, remove them. Continue rinsing.
P308 + P313: After exposure or suspected exposure: Seek medical attention/care
Web: https://www.merckmillipore.com/SK/sk/product/Benzaldehyde,MDA_CHEM-801756</td></tr>
<tr><td></td><td>
<table>
<tr><th>CAS #</th><th>EC Number</th><th>Hill Formula</th><th>Chemical Formula</th><th>Molar Mass</th></tr>
<tr><td>67-64-1</td><td>200-662-2</td><td>C_3H_6O</td><td>CH_3COCH_3</td><td>58.08 g/mol</td></tr>
</table>
</td></tr>
<tr><td>acetone</td><td>Hazard Statement(s)
H225: Very flammable liquid and vapour.
H319: Causes severe eye irritation.
H336: May cause drowsiness or dizziness.
EUH066: Repeated exposure may cause drying or cracking of the skin.</td></tr>
</table>

	Precautionary Statement(s) P210: Keep away from heat, hot surfaces, sparks, open flames and other sources of ignition. Do not smoke. P233: Keep container tightly closed. P240: Ground and secure the container and filling device. P241: Use electrical/ ventilation/ lighting equipment in explosive atmospheres. P242: Use non-sparking apparatus. P305 + P351 + P338: AFTER COVERING EYES: Gently flush them with water for a few minutes. If you use contact lenses and it is possible, remove them. Continue rinsing. P403 + P233: Store in a well-ventilated place. Keep the container tightly closed. Web: https://www.merckmillipore.com/SK/sk/product/Acetone,MDA_CHEM-100012
	CAS # / **EC Number** / **Hill Formula** / **Chemical Formula** / **Molar Mass** **64-17-5** / 200-578-6 / C_2H_6O / C_2H_5OH / 46.07 g/mol
ethanol	**Hazard Statement(s)** H225: Very flammable liquid and vapour. H319: Causes severe eye irritation. **Precautionary Statement(s)** P210: Keep away from heat, hot surfaces, sparks, open flames and other sources of ignition. Do not smoke. P233: Keep container tightly closed. P240: Ground and secure the container and filling device. P241: Use electrical/ ventilation/ lighting equipment in explosive atmospheres. P242: Use non-sparking apparatus. P305 + P351 + P338: AFTER COVERING EYES: Gently flush them with water for a few minutes. If you use contact lenses and it is possible, remove them. Continue rinsing. P403 + P233: Store in a well-ventilated place. Keep the container tightly closed. Web: https://www.merckmillipore.com/SK/sk/product/Ethanol,MDA_CHEM-818760

References

1. R. Periasamy, R. Rajamohan, S. Kothainayaki, K. Sivakumar, *Spectral investigation and structural characterization of Dibenzalacetone: β-Cyclodextrin inclusion complex*, **Journal of Molecular Structure**, Vol. 1068, 25 June 2014, Pages 155-163
2. *https://www.sigmaaldrich.com/SK/en/product/aldrich/246425*
3. *https://web.mnstate.edu/jasperse/chem365/Aldol%20Reaction.pdf*
4. *https://medium.com/@delpippo/dibenzalacetone-31d2843b3f43*
5. O. Houshia, A. Walwil, H. Jumaa, H. Qrareya, *Assessment of the Ratio of Geometric Isomers of Dibenzalacetone Spectroscopically*, Journal of Pharmaceutical Research International 31(4): 1-9, 2019; Article no.JPRI.52538 ISSN: 2456-9119
6. *https://www.vedantu.com/question-answer/are-the-three-geometric-isomers-of-dibenzalac-class-11-chemistry-cbse-6080e562dfee7e00e2057c3e*
7. *Merck Millipore. 2017. Reagents, Chemicals and Labware*

Laboratory manual

Isolation of Chamazulene from Chamomile flowers

Introduction

Chamazulene

Already in 1561, the German scholar Joachimus Camerarius knew the bluish essential oil of chamomile and recommended it for the treatment of several diseases. The well-known English chemist and perfumer George William Septimus Piesse distilled it in 1865 and Alfred Edmund Sherdanal isolated the component chamazulene from the essential oil in 1915, which causes its bluish to dark blue colour.[1]

Chamazulene ($C_{14}H_{16}$) is a bicyclic sesquiterpenoid polyalkene and is an important compound used as an anti-inflammatory agent in herbal medicines and cosmetic products. Chamazulene is not a natural product, but is formed by the decomposition of its precursor, matricin, during steam distillation of plants. Chamazulene gives colour to the essential oil in blue tones depending on its concentration. In the essential oil, it creates a light blue colour at low concentration, and a dark blue colour at high concentration. The plants that supply chamazulene in the world are limited.[2]

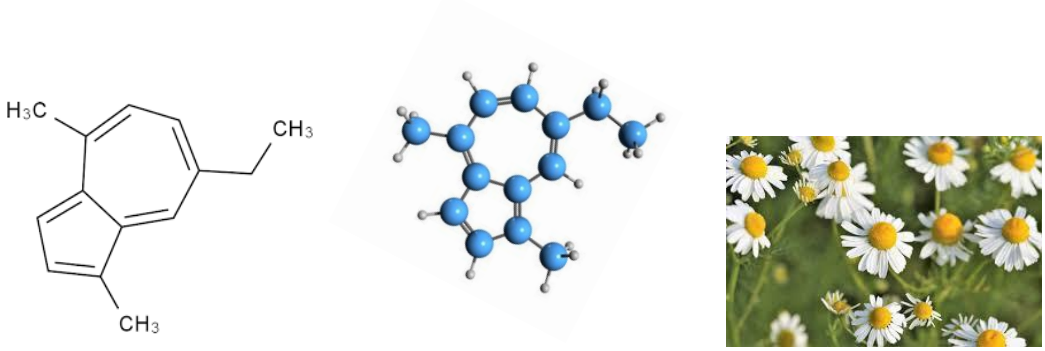


Fig.1.: 2D and 3D structure of chamazulene in chamomile flowers

Germany, Hungary, Slovakia, Argentina, Egypt, Chile, Russia, Poland, Czech Republic, Belgium, Brazil, France, Spain and Greece are among the countries with the highest production in the world.[2]

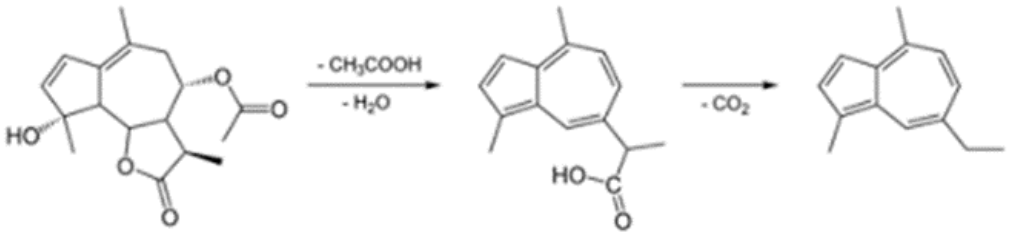
Separation of bioactive compounds from medicinal plants is scientifically and industrial important, because of the highly medicinal activity of pure compounds (antispasmodic, anti-inflammatory and antimicrobial). The bioactive compounds can be extracted from medicinal plants with different techniques such as classical solvent extraction procedures, ultrasound-assisted extraction, microwave-assisted extraction, extraction with ionic liquids, accelerated (pressurized) solvent extraction, supercritical fluid extraction, solid-phase extraction and distillation methods.[3]

Chamomile essential oil contains between 10 and 25 % chamazulene. Sometimes there is very little (about 5 %) or no chamazulene at all. However, its precursor, the sesquiterpene lactone, matricin, which is colourless, is present in the floral debris. The conversion of matricin to chamazulene takes place during distillation.[1]
The boiling point of chamazulene is 161 °C.

Isolation of Chamazulene from Chamomile flowers

The principle of the work is the formation (by conversion of matricin to chamazulene occurs during reflux) and isolation of CHAMAZULENE from chamomile essential oil by steam distillation.[3]
Then qualitative identification by thin-layer chromatography (if a standard is available) or determination of the refractive index by refractometry ($n_{Chamazulene}$ = 1,585).

Reaction scheme:



Matricin (proazulene) ***chamazulene***

Assignment

1. Familiarize yourself with the dangerous properties of the substances you will be working with.
2. Prepare all tools and chemicals for the job.
3. Construct the apparatus.
4. After work calculate the percentage of essential oil containing chamazulene in a given amounth plant.
5. Determine the refractive index of chamazulene by refractometry.
6. Draw the apparatus.
7. Complete the worksheet.
8. Answer the questions in the worksheet.

Materials

Analytical balance, round-bottomed brewing flask (500 ml), boiling stones, heating nest (sand bath), extraction attachment, reflux condenser, Erlenmeyer flask, separating funnel, filtration funnel, round flask (small), rotary vacuum evaporator, beakers, thin-layer chromatography plate (TLC plate).

Chemicals

10 g dried chamomile flowers, n-hexane, distilled water

Apparatus

Fig.2.: Apparatus for the preparation and isolation of chamazulene from chamomile essential oil

Procedure

1. Transfer 10 g of dried chamomile flowers into a 500 ml round-bottomed brewing flask and add 250 ml of water, add the boiling stones and place the flask in a heating nest.
2. Place the extraction attachment **on the flask** to catch the essential oil.
3. Pour approximately 25 ml of water and 10 ml of n-hexane **into the extractor through the side tube** so that the **level of the organic phase** in the collection tube **reaches a level equal to the bending height of the connecting thin tube.**
4. **Close the side opening** of the extension **with a glass stopper** and **attach a reflux condenser** to the opening above the collection tube.
5. Heat the contents of the flask to reflux for 3 hours.
6. **The distillate**, which is condensed in the condenser drips **into the collection vessel below**. The excess separated water (bottom clear phase) **is continuously returned to the brew flask**.
7. **The required essential oil is trapped in the upper - organic layer (n-hexane). Chamazulene**, which is formed during reflux, **gradually colors the organic layer as blue.**
8. After three hours, we stop heating the mixture.
9. Then carefully pour the contents of the extraction attachment **through the side opening into the separating funnel.**
10. Pour out the **aqueous layer (bottom layer) from the separating funnel**.
11. Pour out the **upper - organic blue layer containing the camomile essential oil into a smaller, dry and pre-weighed round flask**.
12. **Then using a rotary vacuum evaporator (RVE) evaporate the solvent** from the round flask and the **isolated camomile silica remains in the flask.**
13. After weighing, **calculate the percentage of essential oil containing chamazulene** in a given amounth plant.

Work duration:
Preparation of the apparatus takes 20 minutes, isolation of chamazulene 3 hours, cleaning of the product using a vacuum rotary evaporator 20 minutes.

Accuracy:
Verification of the success of the work is carried out by means of TLC or by measuring the refractive index of the isolated compound.

Use of method:

Method is suitable for isolation of Chamazulene from Chamomile flowers. Other methods, which can be used for bioactive compounds are:, ultrasound-assisted extraction, microwave-assisted extraction, extraction with ionic liquids, accelerated (pressurized) solvent extraction, supercritical fluid extraction , solid-phase extraction and other distillation methods.

Safety Information

Chemical	Safety Information
n-hexane	Web: https://www.merckmillipore.com/SK/sk/product/n-Hexane,MDA_CHEM-104374
	CAS # / **EC Number** / **Hill Formula** / **Molar Mass** **110-54-3** / 203-777-6 / C_6H_{14} / 86.18 g/mol
	Hazard Statement(s) H225: Highly flammable liquid and vapour. H304: May be fatal if swallowed and enters airways. H315: Irritating to skin. H336: May cause drowsiness or dizziness. H361f: Suspected of damaging fertility. H373: May cause damage to organs through prolonged or repeated exposure by inhalation. H411: Toxic to aquatic organisms, with long-term effects. **Precautionary Statement(s)** P202: Do not use until all safety precautions have been read and understood. P210: Keep away from heat, hot surfaces, sparks, open flames and other sources of ignition. Don't smoke. P273: Avoid release to the environment. P301 + P310: IF SWALLOWED: Immediately call a POISON CENTER/doctor. P303 + P361 + P353: IF ON SKIN (or hair): Take off all contaminated clothing. Rinse the skin immediately with water. P331: Do not induce vomiting. P403 + P233: Keep in a well-ventilated place. Keep the container tightly closed.

References

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Laboratory manual

Preparation of 2-Ethoxynaphthalene or Ethyl 2-naphthyl ether (Nerolin-synthetic perfume)

Introduction

The aim of this work is the preparation of 2-Ethoxynaphthalene or Ethyl 2-naphthyl ether (Nerolin-synthetic perfume).

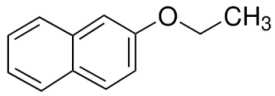
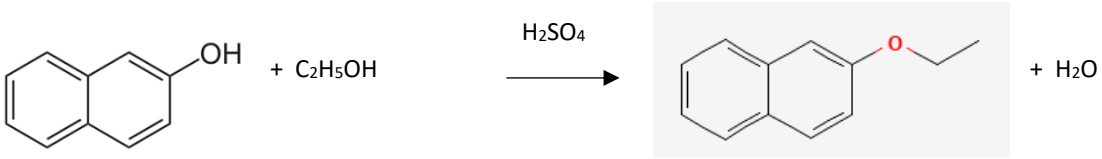


Fig. 1.: *2-Ethoxynaphthalene or Ethyl 2-naphthyl ether (Nerolin)[1].*

Synonym(s): *Ethyl 2-naphthyl ether, 2-naphthol ethyl ether, Neroline, beta-naphthol ethyl ether, beta-naphthyl ethyl ether, bromelia, ethyl beta-naphtholate, ethyl beta-naphthyl ether, nerolin bromelia[2]*

The compound 2-Ethoxynaphthalene or Ethyl 2-naphthyl ether has an odor reminiscent of orange blossom with a faint fruity base. It has a corresponding sweet taste reminiscent of strawberries (only in extreme dilution). Synthesized Neroline is used in citrus and floral perfumes or colognes.[3]

The preparation of Ethyl 2-naphthyl ether consists of alkylation on oxygen with ethanol under the catalytic action of sulphuric acid[5]:



Ethyl 2-naphthyl ether produces white crystals (relative density 1. 0640, melting point 37.5 °C, boiling point 282 °C, refractive index 1. 5932) with a delicate, lingering orange blossom odour. This compound is insoluble in water, soluble in alcohol, ether, chloroform, carbon disulfide, petroleum ether, toluene and other organic solvents. It is also referred to as Neroline Bromelia, or scented Neroline. **Synthesized Neroline is used in citrus, floral perfumes or colognes.**[3,4]

*Note: **Natural Neroli essential oil is obtained by distillation from the "Neroli flowers" of the bitter orange (Citrus aurantium amara).** The essential oil is light, sweetish and volatile. The oil is one of the most expensive essential oils in the world. The bitter orange tree grows up to 10 m in height, but its fruits are not consumed and are smaller than those of the sweet orange tree. Its oil was named in the 17th century in honour of the Italian Princess of Neroli, who loved the scent of this oil, scented her gloves with it and added it to her bath.*

Assignment

1. Familiarize yourself with the dangerous properties of the substances you will be working with.
2. Prepare all tools and chemicals for the job.
3. Prepare solutions and reagents: ***0,38 mol*** of ethanol recalculate to volume in ***ml***! and 5 % (percentage by weight) solution of sodium hydroxide (100 ml).
4. Construct the apparatus with reflux condenser.
5. After work, weigh the final product and calculate the relative yield in percentage.
6. Determine the melting point of prepared compound.
7. Draw the apparatus to the worksheet.
8. Complete the worksheet.
9. Answer the questions.

Materials

Analytical balance, round-bottomed flask (100 ml), boiling stones, thermometer, reflux condenser, beakers, Büchner funnel, extraction flask, oil bath, or apparatus for vacuum distillation or steam distillation.

Chemicals

naphth-2-ol (14,4 g),
ethanol (0,38 mol) - **recalculate** to volume in ***ml***!
concentrated sulphuric acid (16 ml),
5 % (percentage by weight) solution of sodium hydroxide (100 ml)

Procedure

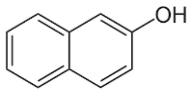
1. Prepare **0,38 mol of ethanol** (re-calculate to volume on ***ml***) in a round-bottomed flask.
2. Carefully add 16 ml of concentrated sulphuric acid in small batches, so that the temperature does not exceed 50 °C (or cool with a water bath).
3. Then add 14,4 g of naphth-2-ol and heat under reflux in a bath heated to 120 °C **for half an hour.**
4. Cool the reaction mixture and pour into 250 ml of water.
5. Aspirate the solids on a Büchner funnel, then transfer to a beaker and dissolve in 100 ml of warm solution of 5 % of sodium hydroxide.
6. After cooling the mixture (to at least 20 °C), then the undissolved product again aspirate on Büchner funnel.
7. Dissolve the **crude product** in 100 ml of hot water and, after cooling, drain on a Büchner funnel and dry on air.
8. The **crude product** may be further **purified by steam distillation or vacuum distillation** (b.v. 140 °C/2kPa) or by **crystallisation from ethanol**.
9. Determine the melting point of prepared compound.

Duration of analysis:
Preparation of solutions (5 % solution (percentage by weight) of sodium hydroxide (100 ml), also mixtures as indicated in the instructions and re-calculating substance quantity in ***mol*** *on* ***mililitre*** takes about 20 minutes. Contruction of the apparatus and preparation of substance takes about 160 minutes, cleaning of the crude product by crystallization from ethanol takes about 20 minutes.

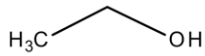
Accuracy:
Verification of the success of the work is carried out by means of determine the melting point of the product.

Use of method:
Method is suitable for understanding the preparation of compound by means of alkylation on oxygen with ethanol under the catalytic action of sulphuric acid and for practicing methods of purification of the prepared crude product by recrystallisation and by vacuum distillation or steam distillation.

Safety Information

Chemical	Safety Information
naphth-2-ol 	**Hazard Statement(s)** H302 + H332: Harmful if swallowed or inhaled. H317: May cause allergic skin reaction. H318: Causes serious eye damage. H400: Very toxic to aquatic life. **Precautionary Statement(s)** P273: Avoid release into the environment. P280: Wear protective gloves/goggles/face protection. P301 + P312: AFTER INGESTION: In case of health problems, call the NATIONAL TOXICOLOGICAL INFORMATION CENTER/physician. P302 + P352: IN CONTACT WITH SKIN: Wash with plenty of water. P304 + P340 + P312: AFTER INHALATION: Move the person to fresh air and allow him/her to breathe comfortably. In case of health problems, call the NATIONAL TOXICOLOGICAL INFORMATION CENTER/physician. P305 + P351 + P338: AFTER COVERING EYES: Gently flush with water for several minutes. If you use contact lenses and it is possible, remove them. Continue rinsing. Web: https://www.merckmillipore.com/SK/sk/product/2-Naphthol,MDA_CHEM-822290

CAS #	EC Number	Hill Formula	Molar Mass
35-19-3	205-182-7	$C_{10}H_8O$	144.17 g/mol

Ethanol 	**Hazard Statement(s)** H225: Very flammable liquid and vapour. H319: Causes severe eye irritation. **Precautionary Statement(s)** P210: Keep away from heat, hot surfaces, sparks, open flames and other sources of ignition. Do not smoke. P233: Keep container tightly closed. P240: Ground and secure the container and filling device. P241: Use electrical/ ventilation/ lighting equipment in explosive atmospheres. P242: Use non-sparking apparatus. P305 + P351 + P338: AFTER COVERING EYES: Gently flush them with water for a few minutes. If you use contact lenses and it is possible, remove them. Continue rinsing. P403 + P233: Store in a well-ventilated place. Keep the container tightly closed. Web: https://www.merckmillipore.com/SK/sk/product/Ethanol,MDA_CHEM-818760

CAS #	EC Number	Hill Formula	Chemical Formula	Molar Mass

	64-17-5	200-578-6	C_2H_6O	C_2H_5OH	46.07 g/mol

sulphuric acid

Hazard Statement(s)
H290: May be corrosive to metals.
H314: Causes severe skin burns and eye damage.
Precautionary Statement(s)
P234: Keep only in the original packaging.
P280: Wear protective gloves/protective clothing/eye protection/face protection/ear protection.
P303 + P361 + P353: IN CONTACT WITH SKIN (or hair): remove all contaminated articles of clothing. Rinse skin immediately with water.
P304 + P340 + P310: AFTER INHALATION: Move the person to fresh air and allow him/her to breathe comfortably. Call the TOXICOLOGICAL INFORMATION CENTER/physician immediately.
P305 + P351 + P338: AFTER COVERING EYES: Gently flush with water for several minutes. If you use contact lenses and it is possible, remove them. Continue rinsing.
P363: Wash contaminated clothing before further use.
Web: https://www.merckmillipore.com/SK/sk/product/Sulfuric-acid-980-0,MDA_CHEM-112080

CAS #	EC Number	Hill Formula	Chemical Formula	Molar Mass
7664-93-9	231-639-5	H_2O_4S	H_2SO_4	98.08 g/mol

Sodium hydroxide

Hazard Statement(s)
H290: May be corrosive to metals.
H314: Causes severe skin burns and eye damage.
Precautionary Statement(s)
P234: Keep only in the original packaging.
P260: Do not breathe dust.
P280: Wear protective gloves/ protective clothing/ eye protection/ face protection/ hearing protection.
P303 + P361 + P353: IN CONTACT WITH SKIN (or hair): Remove all contaminated articles of clothing. Rinse skin immediately with water.
P304 + P340 + P310: AFTER INHALATION: Move the person to fresh air and allow him/her to breathe comfortably. Call the TOXICOLOGICAL INFORMATION CENTER/physician immediately.
P305 + P351 + P338: AFTER COVERING EYES: Gently flush with water for several minutes. If you use contact lenses and it is possible, remove them. Continue rinsing.
Web: https://www.merckmillipore.com/SK/sk/product/Sodium-hydroxide,MDA_CHEM-106462

CAS #	EC Number	Hill Formula	Chemical Formula	Molar Mass
1310-73-2	215-185-5	HNaO	NaOH	40.00 g/mol

References

1. *https://www.indiamart.com/proddetail/nerolin-bromelia-25677772412.html*
2. *https://www.sigmaaldrich.com/SK/en/substance/nerolinbromelia1722293185*
3. *https://sk.odowell.com/nerolin-bromelia.html*
4. *https://www.chembk.com/en/chem/Nerolin%20bromelia*
5. J.Šauliová, *Preparativní organická chemie a Laboratoře z organické chemie I*, Přírodovědecká fakulta, Univerzita Jana Evangelisty Purkyně, Ústí nad Labem, 2007
6. Merck Millipore. 2017. *Reagents, Chemicals and Labware.* [ONLINE] Available at: http://www.merckmillipore.com/GB/en.

Worksheet

Preparation of 2-Ethoxynaphthalene or Ethyl 2-naphthyl ether (Nerolin-synthetic perfume)

Chemical calculations

1. **Consider what amounts of solutions you need for an individual for individual work and for all group members for group work. From an environmental point of view, only prepare volumes of solutions that you will use, including an adequate reserve.**

TABLE 1 SOLUTIONS AND REAGENTS

Solution	Alternative Work in groups			
	Amount of substance in moles	Mass	Volume	Notes
Napht-2-ol	X	14,4 g	X	
Ethanol	0,38 mol	X	……	Recalculate to volume in mililiters.
Sulphuric acid (concentrated)	X	X	16 ml	
5 % solution of Sodium hydroxide (NaOH)	X	……	100 ml of solution NaOH	It is needed to prepare 100 ml of 5 % solution of NaOH. Calculate amounth of NaOH in grams and dissolve in 50 ml water in beaker. Then solution pour into a volumetric flask (100 ml) and add water to 100 ml.

1. **Calculate volume of ethanol required for the reaction, if you need 0.38 mol of ethanol. Molar mass, M(Ethanol, C_2H_5OH) = 46,068 g.mol^{-1} , density, ρ (Ethanol, C_2H_5OH) = 0,785 g.cm^{-3}.**

$$n(Ethanol) = \frac{m\ (Ethanol)}{M\ (Ethanol)}$$

$$m(Ethanol) = n(Ethanol)\ .M(Ethanol)$$

m (Ethanol) = ... g

Recalcutate the mass of ethanol to volume of ethanol:

$$\rho = \frac{m}{V}$$

$$V(Ethanol) = \frac{m\ (\text{Ethanol})}{\rho\ (\text{Ethanol})}$$

Record the volume of ethanol you have to measure by graduated cylinder:

V (Ethanol) = …… g

2. **Calculate the mass of sodium hydroxide, you have to weight for preparing 100 ml of 5 % solution.** ***This mass then dissolve in 50 ml water, in beaker. Then solution pour into a volumetric flask (100 ml) and add water to 100 ml.***
5 % solution, w(NaOH) = 0,05
ρ (5 % NaOH) = 1,0539 g.cm^{-3} – (*note: from chemical tables)*

$$w\ (NaOH) = \frac{m(NaOH)}{m(solution)}$$

$$m(NaOH) = w(NaOH)\ .\ \boldsymbol{m(solution)}$$

100 ml of solution

$$\rho = \frac{m}{V}$$

$$m(solutiom) = \boldsymbol{\rho(solution)}\ .\ \boldsymbol{V(solution)}$$

$$m(NaOH) = w(NaOH)\ .\ \boldsymbol{\rho(solution)}\ .\ \boldsymbol{V(solution)}$$

Record the mass of NaOH you have to weight:

V (Ethanol) = …… g

This mass then dissolve in 50 ml water, in beaker. Then solution pour into a volumetric flask (100 ml) and add water to 100 ml.

Principle, properties and apparatus

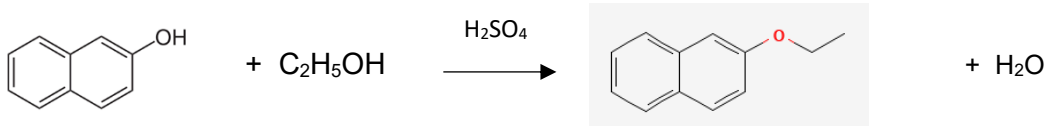
3. Record the information about principle of preparation of the 2-ethoxynaphtalene together with reaction

4. Record information about properties of this substance.
5. Draw the apparatus and named all parts of it.

Results

Calculation the percentage yield of the product (2-ethoxynaphtalene)

To calculate the percentage yield, it is important to take into account the course of the chemical reaction and calculate the theoretical yield of the product and record the weight of the prepared product = practical yield in grams:



a) Calculation of theoretical yield of the product (2-ethoxynaphtalene):
M (naphth-2-ol) = 144,17 g.mol^{-1}
M (2-etoxynaphtalene) = 172,23 g.mol^{-1}
m (naphth-2-ol) = 14,4 g.mol^{-1} (the mass weighted for reaction)

From the reaction:

$$\frac{n\,(2-\text{ethoxynaphtalene})}{n\,(\text{naphth}-2-\text{ol})} = \frac{1}{1}$$

$$n(2-\text{ethoxynaphtalene}) = n(\text{naphth}-2-\text{ol}))$$

$$n(2-\text{ethoxynaphtalene}) = \frac{m(\text{naphth}-2-\text{ol})}{M(\text{naphth}-2-\text{ol})}$$

n(2-ethoxynaphatalene) = …….. mol

For mass of 2-ethoxynaphtalene:

$$m = n.M$$

$$m(2-\text{ethoxynaphtalene}) = n(2-\text{ethoxynaphtalene})\,.M\,(2-\text{ethoxynaphtalene})$$

Theoretical yield of the product (2-ethoxynaphtalene):

m (2-ethoxynaphtalene, theoretical) =…… g

b) Record the weight of prepared product = practical yield in grams:

m (2-ethoxynaphtalene, practical yield) = ……. g

c) Relative yield (percentage yield) of product (2-ethoxynaphtalene):
Calculating is based on the theoretical yield, m (2-ethoxynaphtalene, theoretical) = …… g (calculated earlier in step a)) and practical yield after weighting of the prepared product (step b)):

$$Percentage\ yield\ (2-\text{ethoxynaphtalene}) = \frac{m\ (2-\text{ethoxynaphtalene, practical yield})}{m\ (2-ethoxynaphtalene, theoretical\ yield)} . 100$$

Conclusion

Evaluate the success of your synthesis.

……

……

……

Questions

Chemistry, Organic chemistry, Cosmetic chemistry

1. What is a chemical formula of 2-ethoxynaphtalene?
2. Explain the role of the sulfuric acid in the preparation of 2-ethoxynaphtalene.
3. Why is ethanol used in the synthesis of 2-ethoxynaphtalene?
4. Describe the apparatus, which is used for preparation od 2-ethoxynaphtalene.
5. The 2-ethoxynaphtalene is insoluble in water, but soluble in……. .
6. Compare the natural Neroli oil with the synthetic Neroline in terms of origin.
7. Why is important to determine the melting point of 2-ethoxynaphtalene after preparation?
8. How would you confirm identity of 2-ethoxynaphtalene after synthesis?
9. Which safety precaution should be taken when hadling sulfuric acid in the lab.?