Name of the project:	**Digitization of chemistry experiments to improve the quality and support chemistry teaching in secondary schools**
Acronym:	**ChemIQSoc**
Project number:	**2021-1-SK01-KA220-VET-000027995**

Co-funded by the Erasmus+ Programme of the European Union

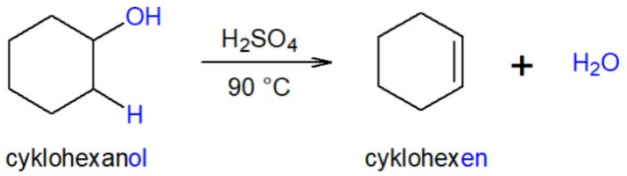
Tittle: Preparation of cyclohexene

Work instructions

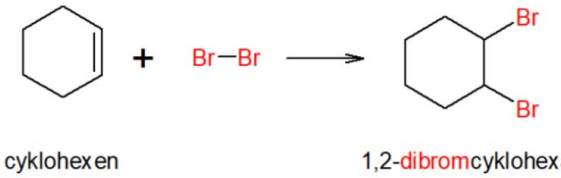
Task: Prepare cyclohexene by dehydration of cyclohexanol, calculate the relative yield of the reaction, measure the refractive index of the product, perform proof of multiple bonding.

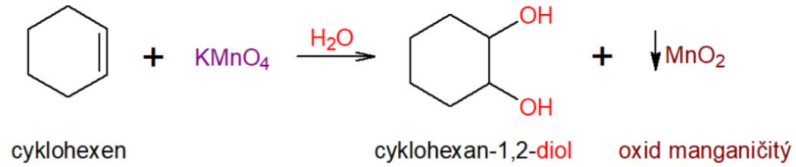
Theory

The preparation of cyclohexene is an example of an elimination reaction. It involves dehydration i.e. the splitting off a water molecule. The reaction follows the equation:



Proof of the double bond is made either by reaction with bromine water (in this case it will be necessary to replace the bromine with iodine), which is accompanied by a colour change from orange to clear solution, or with potassium permanganate in a neutral environment, when a brown precipitate is formed.





Extraction is a method of separating mixtures of substances based on the different solubility of the substances in a pair of mutually immiscible solvents. In this work, extraction can be easily used to purify the product from unreacted starting substances. Water is distilled along with the product, while the starting cyclohexanol and sulfuric acid are partially entrained by the vapours. Cyclohexanol is water soluble and can be removed by extraction with water. Residual acids are removed from organic solutions by extraction with $NaHCO_3$ or Na_2CO_3, when the acid is converted to a salt that is readily soluble in water. Extraction with acid or alkali must be followed by extraction with water or brine. The main reason for this is to remove residual salts

after the previous extraction. This is followed by drying of the organic matter with an inorganic drying agent (most commonly used: $CaCl_2$, Na_2SO_4, $MgSO_4$) and filtration.

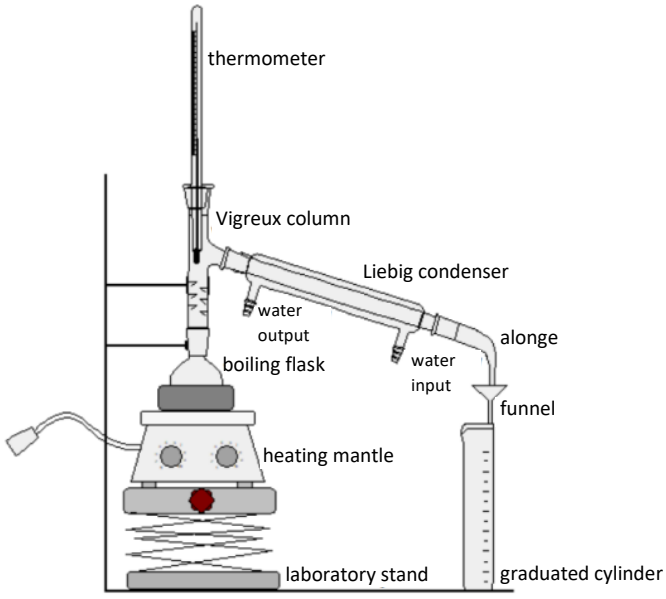
Equipment: heating mantle, Vigreoux column, laboratory stand, boiling flask (250 ml), pipette, pipetting balloon, condenser, distillation adapter (allonge), thermometer, separating funnel, filter paper, funnel (2 pcs.), graduated cylinder, refractometer

Chemicals: cyclohexanol, sulfuric acid, potassium permanganate, sodium chloride, sodium carbonate, calcium chloride

Procedures:

1. We build a distillation apparatus consisting of a 250 ml flask, a Vigreux column, a condenser and a thermometer.
2. Introduce cyclohexanol (0.3 mol) and 0.9 ml of 96% concentrated sulfuric acid into the boiling flask.
3. Heat the flask on the heating mantle and draw the distillate to 90°C. Stop the distillation early to avoid overheating the distillation residue and bursting the flask.
4. Pour the still hot distillation residue into a waste chemical bottle.
5. Transfer the obtained distillate to a separating funnel and extract 15 ml of 5% $NaHCO_3$ solution and then 20 ml of brine.
6. The purified cyclohexene is then dried with $CaCl_2$ and filtered through a small, DRY! funnel.
7. Measure the refractive index and perform proof reactions for the presence of the double bond with potassium permanganate.

Apparatus:



Management of chemical substances

Chemicals	Form	H-statements	P-statements
Cyclohexanol	Liquid	H302 + H332, H315, H335	P261
H_2SO_4	Liquid, 98%	H290, H314	P260, P280, P303 + P361 + P353, P280, P304 + P340 + P310, P305 + P351 + P338
$KMnO_4$	3% (w/w) solution	H272, H302, H314, H410	P210, P220, P260, P280, P305 + P351 + P338, P370 + P378
NaCl	Solid	---	---
Na_2SO_4	Solid	H315, H302, H319, H335	P301 + P312 + P330, P305 + P351 + P338
$CaCl_2$	Solid	H319	P305 + P351 + P338
Cyclohexene (product)	Liquid	H225, H302, H304, H311	P210, P280, P301 + P310, P312, P331

Sources of risk and assessment of risk severity

There is no risk when following all the principles for working with chemicals and using personal protective equipment (gloves, goggles, lab coat).

Waste management method

These substances and their packaging must be disposed of as hazardous waste. Pour the waste into a labelled container. Do not return unconsumed residues to the storage bottles. Dispose of broken glass in a marked container.

This combustible material can be incinerated in a chemical waste incinerator equipped with an afterburner and gas scrubber. Pass residual quantities and non-recoverable solutions to a certified disposal company. Dispose of contaminated packaging as unconsumed product.

Risk reduction measures

Wear a face shield or safety glasses. Wear protective gloves. Gloves must be inspected before use. Use proper glove removal technique without touching the outer surface of the gloves to prevent skin contact with this product. Do not eat, drink, or smoke while working. Wash hands with soap and water after work or when work is interrupted or treat with a protective cream. In case of an accident or if you feel unwell, inform the teacher immediately. Prevent further leakage or spillage unless there is a risk involved. Do not allow to enter drains. Prevent discharge into the surrounding environment.

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Worksheet

Calculation

1. Calculate the volume of 0.3 mol of cyclohexanol needed to prepare cyclohexene. $M(C_6H_{10})$ = ... $g.mol^{-1}$, $\rho(C_6H_{10}, 20°C)$ = ... $g.cm^{-3}$.

2. Calculate the mass of $NaHCO_3$ required for the preparation of 15 ml of a 5 % solution $NaHCO_3$. $M(NaHCO_3)$ = ... $g.mol^{-1}$, $\rho(NaHCO_3, 20°C)$ = ... $g.cm^{-3}$.

3. Calculate the theoretical yield of cyclohexene.

4. Calculate the practical yield of cyclohexene in %.

Observation

1. Describe the appearance and odor of cyclohexanol and cyclohexene.

Feature	Cyclohexanol	Cyclohexene
Appearance		
Odour		

2. Search for the basic physicochemical properties of cyclohexanol and cyclohexene.

Feature	**Cyclohexanol**	**Cyclohexene**
Solubility in water		
Solubility in other solvents		
Boiling point		
Density		
Refractive index		

3. Record the refractive index of the prepared cyclohexene and compare it with the declared value in the tables.

   $n(C_6H_{10})$ = ...

Yield

1. Record the volume of cyclohexene prepared.

   $V(C_6H_{10})$ = ... ml

Questions

1. Write the equation for the preparation of cyclohexene from cyclohexanol.

2. Explain the principle of the elimination reaction using the example of the preparation of cyclohexene. What molecules are cleaved off in this reaction?

3. Describe the uses of cyclohexene.

4. Outline the apparatus you used in the preparation of cyclohexene. You can also draw the apparatus using specialist software, e.g. ChemSketch.

5. Explain the importance of the following operations in the preparation of cyclohexene:

   a. Extraction:

   b. Addition of calcium chloride:

   c. Distillation:

   d. Measurement of refractive index:

6. Describe the purpose of using a Vigreux column in a distillation apparatus in the preparation of cyclohexene.

7. Draw the aqueous and organic layers in the separatory funnel in the correct order.

8. Suggest what proof reactions can be used to confirm the presence of a double bond in cyclohexene? Describe their progress and visual result.

Conclusion

Briefly summarize the objective of the experiment, the main results and compare them with the expected values.