

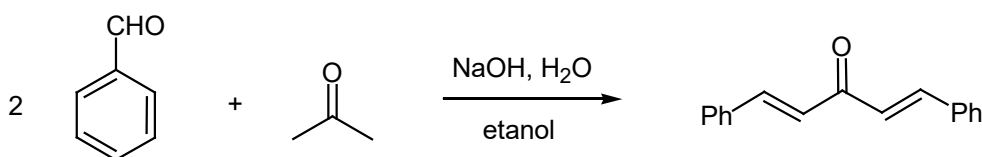
Title: Preparation of dibenzalacetone

Work instructions

Task: React benzaldehyde and acetone to prepare 1,5-diphenyl-1,4-pentadien-3-one (dibenzalacetone).

Theory

The cross-aldol condensation of benzaldehyde with acetone is an addition-elimination reaction of an aromatic electrophile with an enolizable ketone in a basic medium to form dibenzalacetone according to Eq:



Equipment: Erlenmayer flask (100 ml), magnetic stirrer with stir bar, graduated cylinder, laboratory stand, Büchner funnel, filter paper, rubber adapter, suction flask, water jet pump, glass rod, pH paper, reflux condenser, cooling water inlet and outlet hoses

Chemicals: benzaldehyde, acetone, ethanol, sodium hydroxide

Procedures:

1. All work is carried out with safety goggles!

Preparation of dibenzalacetone

1. In a 100 ml Erlenmayer flask with a stir bar, mix 25 ml of cold 10% aqueous NaOH with 20 ml of ethanol. Gradually add a mixture of 2.5 ml of pre-distilled benzaldehyde and 1 ml of acetone while stirring. Keep the reaction mixture at laboratory temperature for 15 min while stirring.
2. Cool the reaction mixture, filter the excluded product through a Büchner funnel and wash with cold water until the filtrate reacts neutrally. Purify the crude product by crystallisation from ethanol. After cooling in a water bath, filter the excluded product through a Büchner funnel. Yellow crystals are obtained with melting point ~ 112°C.

Management of chemical substances

Chemicals	Form	H-statements	P-statements
Benzaldehyde	Liquid, 99%	H302+312, H315	P280, P301+312+330
Acetone	Liquid, 98%	H225, H319, H336	P210, P261, P305+351+338
Ethanol	Liquid, 97%	H225, H319	P210, P233, P280, P303+P361 + P353, P337+P313, P370+P378
NaOH	Solid	H314, H290	P280, P305 + P351 + P338, P310

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

Dispose of waste materials in a marked container. Do not return unconsumed residues to storage bottles. Dispose of broken glass in a marked container.

Risk reduction measures

Avoid direct exposure, use protective equipment. Do not expose to prolonged or repeated exposure. In the event of an accident or if you feel unwell, inform the teacher immediately. These substances or their containers must be disposed of as hazardous waste. Do not eat, drink, smoke. Wash your hands with warm water and soap or treat them with a reparative cream after work or when interrupting work.

Worksheet

Calculations

- Calculate the mass of NaOH required to prepare 25 mL of 10 % NaOH solution.
 $M(\text{NaOH}) = \dots\dots\dots \text{g.mol}^{-1}$, $\rho(\text{NaOH}, 20^\circ\text{C}) = \dots\dots\dots \text{g.cm}^{-3}$.

- Calculate the theoretical yield of dibenzalacetone.

- Calculate the practical yield of dibenzalacetone v %.

Observation

- Describe the appearance and odor of dibenzalacetone.
- Search for the basic physicochemical properties of dibenzalacetone.

Feature	Dibenzalacetone
Solubility in water	
Solubility in other solvents	
Melting temperature	

Yield

1. Record the mass of the prepared dibenzalacetone.

$m(\text{C}_{17}\text{H}_{14}\text{O}) = \dots\dots\dots \text{g}$

Questions

1. Write the equation for the preparation of dibenzalacetone from benzaldehyde and acetone.
2. Write the IUPAC systematic name of dibenzalacetone.
3. Evaluate the importance of using a reflux condenser. Write what consequences its omission could have on the progress and outcome of the synthesis?
4. Write the meaning of product recrystallization. List what factors influence the choice of solvent for recrystallization?
5. Explain why dibenzalacetone is an important compound in industry. How its structure contributes to its use?
6. Write and explain what cross aldol condensation is and what is its importance in the preparation of dibenzalacetone?

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7. Write why ethanol is used to recrystallize dibenzalacetone.

8. Write why it is important to maintain the reaction mixture at laboratory temperature during mixing. What would happen if the temperature rose too much?

Conclusion

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

Disclaimer

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