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Driving Equilibria: Dean-Stark Trap

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Overview

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A Dean-Stark trap is a special piece of glassware, which allows the collection of water during a reaction through an azeotropic distillation. The desire to collect water from a reaction can have various reasons. It can drive the equilibria in reactions, where water is formed as a byproduct. According to Le Chatelier's principle, a change in temperature, pressure, concentration, or volume will cause a readjustment of a reversible reaction to establish a new equilibrium. An acetal formation is a reversible reaction, where water is formed as a byproduct. In such cases, achieving good yields is possible by driving the equilibrium towards the product side via the removal of water. The Dean-Stark trap also allows the determination of water content or can be used to remove water from a solvent mixture through an azeotropic distillation.

Principles

A reaction equilibrium can be influenced with an excess of reagent or removal of a formed product in order to drive the equilibrium to the product side. Equilibria can be also influenced by temperature or pressure. This underlying principle is called Le Chatelier's principle and states that a change in temperature, pressure, concentration, or volume will cause a readjustment of the reaction to establish a new equilibrium. By adding an excess of reagent, the concentration changes and a new equilibrium establishes, favoring the product side. For instance, driving the equilibrium of a hydrolysis can be easily achieved by adding an excess of water.

Influencing the equilibrium of a reaction where water is formed as a byproduct, like an esterification, is not straightforward and requires special glassware. This special piece of glassware is called a Dean-Stark trap and helps to remove the formed water from the reaction medium (**Figure 1**). Solvents that form an azeotrope with water, like toluene, are commonly employed. An azeotrope is a point in a distillation where the composition of the liquid phase is equal to the composition of the gas phase. A further separation through a simple distillation past the azeotropic point is not possible. This is an advantage when using the Dean-Stark trap to influence equilibria, because it will ensure the continuous removal of water. Upon heating the reaction mixture, the formed toluene/water azeotrope will distill over, condense in the condenser, and flow into the Dean-Stark trap. Toluene and water will form two separate layers with toluene as the top layer and water as the bottom layer. While toluene can flow back into the reaction flask, water gets trapped as the bottom layer and ultimately removed from the reaction equilibrium thus driving the reaction towards the product side.

Figure 1. Dean-Stark apparatus

Procedure

1. Preparation

1. Take a 250 mL round bottom flask equipped with a magnetic stir bar.
2. Place an oil bath under the round bottom flask on a magnetic stirrer.
3. Fill the round-bottom flask with 7.5 g (0.05 mol) *m*-Nitrobenzaldehyde and add 75 mL of toluene.
4. Add 3.1 mL (3.45 g, 0.055 mol) ethylene glycol.

5. Attach the Dean-Stark trap to the round bottom flask.
6. Attach a reflux condenser on top of the Dean-Stark trap.

2. Running the Reaction

1. Set the oil bath temperature to 170 °C and heat the reaction mixture to reflux.
2. Monitor the reaction by measuring the water amount in the Dean-Stark trap.
3. The reaction is done when no further water becomes trapped in the side arm of the Dean-Stark trap.
4. After approximately 2 h, the total amount of collected water is approximately 0.8 mL.

3. Workup

1. Release the water and remove the combined organic solvent from the reaction mixture under reduced pressure in a rotary evaporator.
2. Dissolve the yellow residue in 8 mL ethanol under reflux.
3. Cool down the solution.
4. The desired acetal will crystallize.
5. Filter the solid and dry it under reduced pressure.

Results

Water will form and becomes trapped over the course of the reaction. The theoretical amount of formed water upon complete conversion can be calculated and compared with the measured amount of the trapped water to determine the reaction progress.

Applications and Summary

This experiment demonstrates vividly Le Chatelier's principle and how it can drive an equilibrium.

Dean-Stark traps are commonly used to remove water from a solvent mixture under various circumstances. For example, the removal of water through a simple distillation when water does not form an azeotrope with the other solvent, is possible with a Dean-Stark trap based on its design. In the case of an azeotropic distillation, the addition of an entrainer is necessary. An entrainer is an organic solvent, which will form an azeotrope with water but does not mix with water in the liquid phase. The addition of an entrainer ensures the continuous removal of water, which becomes trapped in the side arm of the Dean-Stark trap. Unlike the Dean-Stark trap, a normal distillation apparatus requires the continuous addition of an entrainer since the distilled entrainer cannot flow back to the solvent mixture.

The Dean-Stark trap can also be used to drive the equilibria of reactions, where water forms as a byproduct, like in an ester or acetal formation. Through an azeotropic distillation where the solvent is also the entrainer, water is removed from the reaction and therefore from the equilibrium.

Finally, an azeotropic distillation with a Dean-Stark trap can also be used to determine the water content of solvents or solvent mixtures. Not only can water be removed with a Dean-Stark trap, but also volatile alcohols by placing 5-Å molecular sieves in the trap.