

Free micro-scale synthesis of alkenes report example

[Health & Medicine](#), [Alcoholism](#)



Abstract

This experiment involved alkene preparation by alcohol dehydration (water elimination) in the presence of an acid catalyst. Elimination, combination, rearrangement and substitution are the basic types of chemical reactions known in organic chemistry. Alcohol dehydration is an important transformation that leads to the yield of alkene and it is an example of an elimination reaction. Dehydration is a process that involves the elimination reaction of an alcohol. There is a loss of H from one carbon and OH from adjacent carbon, hence, resulting in a water molecule removal that results in the formation of alkyne or alkene. When an alcohol loses its functional group (hydroxide ion) and a hydrogen ion, water loss occurs. The hydroxide ion is a poor leaving group. Therefore, it has to be protonated first to become a good leaving group H₂O. In many cases, the alcohol dehydration requires heat and an acid catalyst. Sulphuric acid and phosphoric acid are the most commonly used but in this experiment, phosphoric acid was used as a catalyst to help increase the rate of reaction. In the presence of an acid catalyst, alkenes can be hydrated which would be the reverse of acid catalysed dehydration of alcohol. Therefore, an alkene is removed by distillation immediately as it is formed to prevent it from forming back to alcohol through hydration.

Objective

Synthesis of Alkenes via the acid catalysed dehydration of 3-methyl-3-pentanol

Procedure

1. 25 ml of 3-methyl-3-pentanol was placed in a 5-mL conical vial that contained a spin vane or a boiling chip.

We added 0. 25 ml of 85% phosphoric acid to the alcohol

We fitted the reaction vial with a Hickman still and then placed a water-jacketed condenser for cooling on the top.

The vial was placed in a sand bath/heat block, and water was turned on to the condenser.

The temperature was adjusted to between 100 and 105°C

The reaction mixture was heated until the collar of the Hickman was filled with the liquid alkene mixture.

The liquid product was transferred to a vial from the collar of the still and dried over CaCl_2

During the drying process, the vial was capped.

A filter was used to transfer the dried liquid into a clean, tared vial and weighed.

The product distribution was then determined by using gas chromatography.

Results and Discussion

The weight of the vial = 6. 5112 g

The total weight of the vial and the liquid = 6. 8551 g

Therefore, the weight of the liquid is given by;

The total weight of the vial and the liquid - the weight of the vial

$6. 8551 - 6. 5112 = 0. 3439 \text{ g}$

In alcohol dehydration, the OH and H are lost from adjacent carbons and an acid catalyst is required. Alcohol undergoes dehydration only if its OH

functional group is converted to a better leaving group. Protonation converts $-OH$ into a better leaving group $-OH_2^+$ and then, water departs leaving behind a carbocation. The final distillation was done very carefully using clean, dry equipment to obtain pure products. From the experimental analysis, the predominant alkene expected was obtained. Gas chromatographic analysis was then used to determine purity and product distribution of the alkene collected.

Post Lab Questions

Question: Explain the purpose of adding anhydrous $CaCl_2$ to the distillate in the experiment?

Answer: The anhydrous calcium chloride acts as an inorganic drying agent, and it was, therefore, added to the distillate to remove any remaining water. It binds with water strongly and hence removes any water traces from the solution. Anhydrous $CaCl_2$ also absorbs ethanol, depending on the amount used and ethanol may be removed as well.

Question: Why is elimination favoured over substitution at high temperatures?

Answer: Elimination reactions consist greater free energies of activation as compared to substitution reactions since more bonding changes occur during elimination. On using higher temperatures, the rate of both elimination and substitution increases. However, since the energy barrier for elimination is higher than in substitution, the molecule proportion that can cross them is significantly higher. The molecule proportion that can surmount energy of activation barrier for elimination increases more, compared to molecule

proportions that can undergo substitution. Elimination is more efficient at lower temperatures than in higher temperature since it has no energy to surpass the activation energy at high temperatures. Elimination and substitution reactions compete because it is a question of nucleophile or basic properties. Furthermore, elimination is favoured over substitution when a stronger base is used, and when the entropy increases (temperature increases).

Conclusion

Works Cited

John, McMurry. Fundamentals of Organic Chemistry. 3rd Ed. Pacific Grove CA: Brooks/ Cole Publishing Company: Cornell University, 1994.