

Procedure:

1. Set up a hot water bath with an immersion heater in a 400 mL beaker
2. Place 2 mL (2g) of cyclohexanol in a reaction vial using a Pasteur Pipet. Add 1.5 mL of conc. Phosphoric acid (14.8M) to the reaction vial.
3. Seal the vial with a Teflon-coated cap. Be sure it is tight. Shake the reaction vial well before placing into heating bath. Place the vial into the hot bath
4. After 20 minutes, use tongs to remove the vial from the boiling water and place on the counter for a minute to cool.
5. Fill a test tube with about 8-10 mL of 10% sodium carbonate solution.
6. Place the reaction vial in a cold-water bath for another 2-3 minutes

Isolation of Product

7. Carefully open the vial. Go very slow as the materials are under pressure and may squirt out. Slowly pour the reaction mixture into the test tube containing the sodium carbonate solution. The excess phosphoric acid will be neutralized by the sodium carbonate and can be seen by the generation of carbon dioxide bubbles. Gently stir the mixture a few times until the bubbles stop.
8. Transfer the upper organic layer from the test tube to a clean reaction vial using a Pasteur pipet. The organic layer will be a cloudy, viscous, colorless liquid. Wash the organic layer twice with water. Try to remove as much water as possible each time.

Analysis

9. Cyclohexene is a clear, colorless liquid with a mildly piercing odor and a boiling point of 83 °C. The product mixture contains some unreacted cyclohexanol, water and cyclohexene. Do not bother to dry the product.
10. Place 4-5 drops of the cyclohexene mixture in a clean reaction vial. Perform the bromine addition test by adding a few drops of bromine water. Seal with a Teflon-coated cap and swirl the mixture. If the brown color of the bromine water disappears, the mixture contains an alkene. Add a few more drops of bromine water and repeat the procedure. The more drops of bromine water that are required to produce a clear solution, the greater the amount of cyclohexene in the sample.
11. Place 4-5 drops of cyclohexene in another clean reaction vial. Perform the Baeyer test by adding one drop of sulfuric acid solution followed by 1% potassium permanganate solution, drop by drop. Seal the vial and mix the solution gently by swirling. The disappearance of the purple permanganate color from the reaction mixture confirms the presence of the alkene.

Disposal.

The aqueous solutions can be all disposed of by flushing down the sink with excess water. The cyclohexene product and solutions for the Baeyer and permanganate tests should be turned into your instructor for disposal.

Questions:

1. Draw out the mechanism for the dehydration of cyclohexanol via an E1 process using o-phosphoric acid as the catalyst.
2. Of what use are double bonds in an organic compound?
3. What differences in physical properties are associated with lipids (fats) that are saturated from those that are unsaturated?
4. What are the products from the dehydration of 2-methyl-2-butanol? Which is the major product and why?