**Oxidation of Alcohols: Preparation of Cyclohexanone**

**Introduction**

In this particular experiment, cyclohexanone would be formed through the mixture of bleach and acetic acid from cyclohexanol through the oxidation process called: Chapman-Stevens oxidation.   
**Mechanism**



**Theoretical Yield**

**MSDS**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Compound** | **Hazards** | **Boiling Point (C)** | **Melting Point (C)** | **Solubility (g/mL)** | **Molecular weight (g/mol)** | **Density (g/cm^3)** |
| **Hypochlorous acid** | Strong oxidizer |  |  | In water | 52.46 | 1.4 |
| **Acetic Acid (glacial)** | Acidic, Corrosive | 119 | 17 | Miscible | 60.05 | 1.05 |
| **Sodium hypochlorite** | Corrosive | 101 | 18 | 0.293 | 74.42 | 1.11 |
| **Sodium bisulfate** | Corrosive | 315 | 58.5 | 0.5 | 120.06 | 2.74 |
| **Sodium chloride** | Irritant | 1413 | 801 | 3.59 | 58.44 | 2.17 |
| **Cyclohexanol** | Flammable, Irritant | 161.84 | 25.93 | 3.60 | 100.16 | 0.96 |
| **Cyclohexanone** | Flammable, Irritant | 155.6 | -47 | 0.086 | 98.15 | 0.95 |
| **Sodium Hydroxide** | Corrosive | 1388 | 318 | 1.11 | 40.00 | 2.13 |
| **Methylene Chloride** | Harmful | 39.6 | -96.7 | 0.013 | 84.93 | 1.33 |

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

***Safety Precautions***

*Concentrated Acetic acid, sodium hypochlorite, and sodium bisulfite are extremely corrosive to the human skin. If by any chance, these reagents come into contact with you, immediately wash the affected area with water. Methylene chloride is a minor health hazard, thus precaution should be taken while handling this reagent. Gloves, eye ware, and protective clothing are a must. Sodium hydroxide, cyclohexanone are irritants thus, avoid skin contact.*

***Wastes***

*Aqueous Waste: The Aqueous layer remaining after methylene chloride extraction*

*Organic Waste: Reaction product and acetone rinses of glassware.*

*Solid Chemical Waste: Used drying agents.*

**Procedure**

1. 10mmoles of cyclohexanol in the Erlenmeyer flask along with the magnetic stir bar.
   1. Have the flask placed on the stirring motor and start stirring
2. While stirring, add 2.5 mL of glacial acetic acid.
3. Place 15mL of bleach into the separatory funnel; place the funnel above the Erlenmeyer flask.
4. Add the bleach into the Erlenmeyer flask, drop by drop through the separatory funnel to the cyclohexanol/acetic acid mixture.
5. The addition of bleach should take 10-15 minutes.
6. The solution after the addition of bleach should be pale yellow to yellow-green in color.
7. Test for excess hypochlorous acid by using the KI starch paper.
8. Stir it for 15 minutes at room temperature.
9. Add 10-20 drops of saturated sodium bisulfite solution to get rid of the hypochlorous acid.
10. Test for remaining hypocholorous acid using the KI starch paper.
11. Add 2 drops of thymol blue indicator.
12. Add 6M NaOH until the solution turns light blue.
13. Add solid NaCl until the solution is saturated, meaning, excess NaCl will stop dissolving.
14. Decant the liquid into the separatory funnel.
15. Extract with 5mL of methylene chloride and save the organic layer
16. Extract the aqueous layer again with the addition of 5mL of methylene chloride
    1. Make sure to vent the separatory funnel due to the pressure.
17. Combine both of the organic layers.
18. Dry the layer with anhydrous sodium sulfate.
19. Decant to remove the drying agent.
20. Remove the methyl chloride from the solution by doing a vacuum distillation.
    1. Perform this while the side-arm flask in a tub of warm water.
21. Weigh the product to determine the yield.
22. Run an IR spectrum. Determining the purity of the compound.