

Synthesis of cyclohexanone oxime

This experiment is the second step in a two step synthesis. The cyclohexanone synthesized last week will be transformed into cyclohexanone oxime by the addition of hydroxyl amine. This synthesis is an important commercial step in the manufacture of Nylon-6.

Cyclohexanone oxime will be synthesized, isolated, and analyzed by melting point. This reaction is one example of the many derivatives which can be formed from carbonyl containing molecules. The mechanism begins with a nucleophilic attack on the carbon of the carbonyl by the nitrogen of the hydroxylamine. This forms a tetrahedral intermediate which then undergoes a series of proton transfers. The sp^2 carbon is reformed and the oxygen eliminated.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter of the manual and the lecture notes on the Organic Chemistry departmental web site. Review lecture notes concerning nucleophilic attack of carbonyls. Complete the first seven sections of your notebook write up.

A student who has prepared for the oxime synthesis experiment should be able to:

1. Draw the reaction including all starting materials and products.
2. Define or explain the role of sodium hydroxide.
3. Draw the mechanism of sodium hydroxide reacting with hydroxyl amine hydrochloride.
4. Explain the value or application of this chemistry.
5. Explain how the identity and purity of the product will be assessed.
6. Identify and explain safety considerations for the day's experiment.
7. Perform the day's experiment safely and successfully.

Quizzes given after the experiment has been performed may also include:

8. Draw the complete mechanism.
9. Explain observations that lead one to believe product was being formed.
10. Predict the impact on percent yield and purity based on changes in procedures such as temperature, reaction time, changing amounts of reactants.
11. Calculate the percent yield for the single step and both steps in the multi-step synthesis.

SAFETY CONCERNS

Sodium hydroxide solution is corrosive and will cause burns. Hydroxylamine is toxic to aquatic organisms. Do not dispose of down drain.

PROCEDURE

Place a 100 mL beaker having approximately 30 mL of water and one boiling stone on a hot plate in the hood. Adjust the setting to 3 or 4 to just get the water to form bubbles. The water should be approximately 90°C at this point.

Determine your starting mass or volume of cyclohexanone. The density of cyclohexanone is 0.948 g/mL. For each 0.1 mL of cyclohexanone you will need to add 0.5 mL each of aqueous hydroxyl amine hydrochloride and sodium hydroxide solution. Take your starting volume of cyclohexanone, divide by 0.1. Multiply this by 0.5 mL. This is your target volume of both aqueous hydroxyl amine (2.5 M) and sodium hydroxide (2.5 M). If you begin with 0.5 mL (0.48 g) of cyclohexanone, you will use 2.5 mL each of aqueous hydroxyl amine (2.5 M) and sodium hydroxide (2.5 M).

If the amount of your cyclohexanone from last week's synthesis is less than 0.3 mL, augment your material with available reagent cyclohexanone to bring the total up to approximately 0.5 mL.

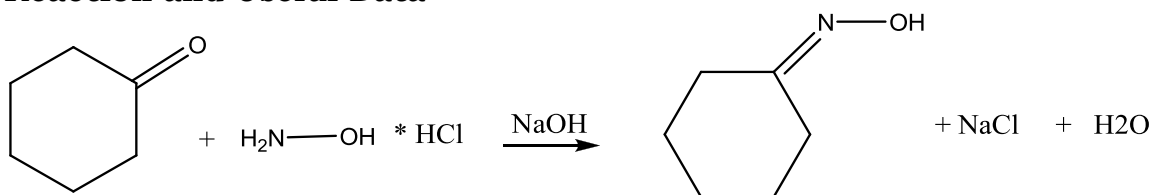
Using 10mL graduated cylinders, measure the 2.5 M aqueous hydroxylamine hydrochloride and the 2.5 mL of 2.5 M aqueous sodium hydroxide solution. Record exact volume used. Add these materials and one boiling stone to a 10mL Erlenmeyer flask.

In the hood, add the cyclohexanone to the E. flask. Swirl the flask and gently place the flask into the beaker of hot water. Be careful not to allow E. flask to tip into the water and flood. Write your observations directly into lab notebook.

Heat for 15 minutes in slowly bubbling water. If water cools, heat for longer time. Remove from water bath. Let cool slowly and undisturbed to room temperature. Note observations. After flask has reached room temperature, immerse in a 100mL beaker approximately half full of ice water. Allow to cool for a minimum of 5 minutes.

Collect crystals using vacuum filtration and a Hirsch funnel. Run the vacuum for a MINIMUM of 5 minutes. Water is difficult to remove completely. Record mass collected. Obtain melting point.

Reaction and Useful Data



	Cyclohexanone	hydroxylamine hydrochloride (Aq)	Sodium Hydroxide (Aq)	Cyclohexanone oxime
M	-	2.5 M	2.5 M	
dens	0.9478 g/ml			
m.wt	98.15 g/mol	69.49 g/mol	40.0g/mol	113.16 g/mol
b.p.	155 °C			206-210°C
m.p.	-16.4°C			91 °C

CLEANUP

Discard used pipettes in broken glass container. Dispose of all aqueous solutions in the aqueous non-halogenated waste container.

Hydroxylamine is toxic to aquatic animals so DO NOT dispose of down drain. Place organic product in the NON-HALOGENATED organic solid waste container. Wipe up work area with damp sponge.

POST-EXPERIMENT

Write lab report and turn in at beginning of next lab period.

REFERENCES

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