16. SN1 Reaction: Synthesis of 2-Chloro-2-methylbutane

This is the first synthesis that you will carry out. Amyl alcohol (2-methyl-2-butanol) will be reacted with hydrochloric acid and 2-chloro-2-methylbutane will be formed. This proceeds via a SN1 mechanism. The product will separate due to decreased water solubility. The product will be analyzed by a silver nitrate test and via IR. The percent yield of the reaction will be calculated.

**PRE-EXPERIMENT ASSIGNMENT**

Study this chapter of the manual and the lecture notes on the Chemistry Department web site. Read sections 7.3, 7.4, 7.5, 7.6 of Klein. Do the first seven parts of your notebook writeup.

**A student who has prepared for the SN1 Reaction experiment should be able to:**

1. Give a balanced equation for the synthesis of 2-chloro-2-methylbutane from hydrochloric acid and amyl alcohol.
2. Write the mechanism of the reaction.
3. Give the name if given the structure, and write the structure if given the name of all chemicals involved in this reaction.
4. Explain why the product separates from the starting material.
5. Explain why the product floats on top of the aqueous layer instead of settling to the bottom.
6. Explain the proper use and all the components of a separatory funnel.
7. Understand the expected IR absorbance of both the organic starting material and product.
8. Identify and explain safety considerations for this experiment.
9. Perform the day's experiments safely and successfully.

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

10. Calculate the theoretical maximum amount of product that may form if given a reaction and all starting amounts.
11. Calculate the percent yield of a reaction if given amount of product produced and theoretical maximum amounts.
12. Give the balanced equation for the reaction of silver nitrate with alkyl halides.
13. Be able to predict the results of silver nitrate analyses with various compounds.
Safety Concerns

Wear goggles and gloves the entire time. Hydrochloric acid is very corrosive and will burn skin and eyes upon contact. In case of accidental contact wash affected area with copious amount of soap and water. Neutralize any acid spills with sodium bicarbonate (baking soda) and wipe up immediately.

Gasses will be released when sodium bicarbonate is added to your separatory funnel. Do not stopper funnel after the addition of sodium bicarbonate solution. Vent separatory funnel frequently while shaking. Do not vent separatory funnel toward another person or yourself. Liquids may spray out while venting.

Chlorinated hydrocarbons tend to be toxic materials. The target organs are the liver and kidneys. Be careful not to breathe product or get on skin.

EXPERIMENT

Measure approximately 2 mL of 2-methyl-2-butanol using your 10 mL graduated cylinder and place it in your 10 mL Erlenmeyer flask. (Remember do not waste time obtaining exactly 2.00 mL but it is necessary to read the graduated cylinder to 2 decimal places and write this value directly in your lab notebook. Instructors will deduct points if exact target volumes are obtained.) Pour approximately 5 mL of hydrochloric acid into a graduated cylinder. Again do not waste time obtaining exactly 5.00 mL; it is not important. It is important however to read acid volume accurately and precisely to 2 decimal places and record this volume directly in notebook. Be careful when handling concentrated acid. In the hood, with constant swirling, slowly add the concentrated hydrochloric acid to the Erlenmeyer flask. Swirl the flask constantly for 5 minutes. Observe and note the changes which occur. Write these observations in your notebook.

Transfer the reaction mixture to your separatory funnel. Drain off the aqueous layer into a beaker. (If you are unsure which layer this is, perform a check.) Then add approximately 2 mL saturated sodium bicarbonate solution. (This volume can be anywhere from 1-4mL. The exact volume is not important. Record approximate volume used in lab book. Do not read this volume to 2 decimal places.) CAUTION! Gas will be released during this step. Swirl the funnel to mix well. Do not stopper. Allow the layers to separate. Drain off the aqueous layer into the same beaker used above.

Obtain about 4 mL of saturated sodium chloride solution (brine). Add about half of this amount or about 2 mL of brine. Swirl the contents to mix. Allow the layers to separate, drain off the aqueous layer. Add the rest of the brine. Swirl to mix. Allow the layers to separate, drain off the aqueous layer into same beaker.
Drain the organic layer into a clean dry 10 mL Erlenmeyer flask. Add anhydrous sodium sulfate just until some material remains free flowing like sand. Let material sit and dry for approximately 5 minutes. Weigh a clean empty shell vial, then start cleaning up.

Using a pipette, transfer the dry organic material to the preweighed clean glass shell vial. Be sure to leave the solid sodium sulfate behind. Weigh the vial. Calculate the mass of product synthesized. Calculate the percent yield.

**Product Analysis (Instrumental and Qualitative)**

Add approximately one milliliter of 0.1M aqueous silver nitrate to two test tubes. It is not necessary to measure out exactly 1.0 mL of the reagent. Simply put liquid into the test tube to the approximate depth of a pinky finger width or a little less. Add four drops of 2-methyl-2-butanol material to one test tube. Using a clean pipette add four drops of product to the other test tube. Note observations.

Analyze the product and the starting alcohol using IR. Review the proper IR procedure and spectra interpretation from chapter 8. Compare the product IR with the IR of the starting material to determine if the anticipated product was formed.

**CLEANUP**

Discard used pipettes in broken glass container. Dispose of all aqueous solutions and any surplus 2-methyl-2-butanol down sink. Place organic product in the HALOGENATED organic liquid waste container. Wipe up work area with damp sponge.

**POST-EXPERIMENT**

Complete data sheet. Turn in data sheet and white notebook pages to instructor. Prepare for the upcoming quiz on SN1 Reaction: Synthesis of 2-chloro-2-methylbutane.

**REFERENCES**


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