Procedure: 2-Chloro-2-methylbutane

The equipment setup for this lab will require the use of the following glassware assembled as shown:





- Make sure to use the ring stand for the separatory funnel so that it does not tip over and break.
- Set up the separatory funnel such that the bottom rests in the top of the Erlenmeyer flask to avoid spills.
- 1. Place the separatory funnel into a ring stand and make sure the separatory funnel is closed.
- 2. Add 5 mL of 2-methyl-2-butanol to the separatory funnel.
- 3. Add 12.5 mL of concentrated HCl to the separatory funnel and swirl to combine.
- **4.** Put the glass stopper on the separatory funnel, and while holding the stopper one with a finger or two, carefully invert the separatory funnel and vent immediately.
- 5. Close the stopcock and vigorously shake the separatory funnel for 20 seconds or so and then vent once again. Close the stopcock and repeat shaking and venting again.
- **6.** Place the separatory funnel back into the ring stand, remove the glass stopper, and drain the bottom (aqueous) layer into an Erlenmeyer flask.
- 7. Add 10 mL of sodium bicarbonate solution slowly as gas will be produced. Swirl the separatory funnel until the bubbling stops then put the glass stopper on, invert the separatory funnel and immediately vent.
- 8. Close the stopcock and shake the separatory funnel ~ 20 seconds and the vent once again.
- 9. Place the separatory funnel back into the ring stand, remove the glass stopper, and drain the bottom (aqueous) layer into the same Erlenmeyer flask containing the first aqueous extract (bubbling in the Erlenmeyer may occur, so perform this second draining slowly). These aqueous extracts are waste, but do not dispose of them until you have completed the entire experiment.
- 10. Perform a second wash (repeat the last 3 steps) but using sodium chloride solution this time.
- 11. Perform a third wash with 10 mL of distilled water.
- **12.** Drain the organic layer into a clean Erlenmeyer flask, add about 4 spatulas of anhydrous sodium sulfate, and swirl the flask for about 15 seconds.
- 13. Pipet the liquid into a tared beaker and weigh to obtain the mass.
- **14.** Use a drop of the liquid to obtain the IR spectrum.
- 15. Perform the silver nitrate and sodium iodide tests.

Halide Tests for Nucleophilic Substitution

Silver Nitrate

The conditions of the test cause the reaction to proceed via an S_N1 mechanism.

$$AgNO3 + RX \rightarrow AgX(s) + RONO2$$

- 1. Add 2 mL of 0.2 M AgNO₃ in ethanol to a test tube.
- 2. Add one drop of the alkyl halide to the test tube and mix by gently shaking.
- **3.** Record the time it takes for a precipitate to form.
- 4. If no precipitate forms after 5 minutes, place test tube into a beaker of warm water (78 °C).
- 5. If no change occurs after 5 minutes, remove the test tube and clean up.

Note: the color of the precipitate indicates the halide

White AgCl Pale yellow AgBr Dark yellow AgI

Sodium Iodide

The conditions of the test cause the reaction to proceed via an S_N2 mechanism.

$$NaI + RX \rightarrow NaX(s) + RI$$

- 1. Add 1 mL of the sodium iodide solution (in acetone) to a test tube.
- 2. Add two drops of the alkyl halide to the test tube and mix by gently shaking the test tube.
- **3.** Record the time it takes for a precipitate to form.
- **4.** If no precipitate forms after 3 minutes, place test tube into a beaker of warm water (50 °C).
- 5. If no change occurs after 5 minutes, remove the test tube and clean up.