Friedel-Crafts Alkylation (Lab X-C)

Background Reading

From the chapter 16 class notes, read the following sections:
Mechanism, Friedel-Crafts Alkylation, and Substituent Category 1.

Keywords

Lewis Acid, Alkylation, Electrophilic Aromatic Substitution, Hydride Shift, and e^{-1} Donating Induction

Compound, Reaction, and Yield Data

- Compound information should include masses, moles, BP's and densities.
- Mechanism should include creation of carbocation, hydride shift, and the electrophilic aromatic substitution. Include all appropriate curved arrows.
- Determine theoretic and actual yields based on the limiting reagent,which will be either toluene or the alkyl halide. Why not $AlCl_3$?
- Report the temperature range at which you collected the toluene and the product from the distillation in your Observations section.
- Include the product distribution (%'s) from your gas chromatogram report.

Procedure

1. Do steps 1 – 6 entirely in a hood.
   Add a dry stir bar to a dry 25-ml round-bottom flask.
   Then carefully weigh 0.5 g $AlCl_{3(s)}$.
   Place $AlCl_{3(s)}$ in the flask before removing it from the hood with the balance.
   Use a spatula to ensure that no solid remains in or on the neck of the flask.

   Caution – $AlCl_{3(s)}$ reacts violently with water, releasing $HCl_{(g)}$ vapors and heat.
   HCl solutions and vapors are corrosive and cause acid burns.
   Use gloves and avoid all contact with skin, eyes, and nose.

2. Clamp flask to stand directly above a stir plate. Attach a Claisen adapter to the flask.
   Then, attach a 125-ml separatory funnel to the adapter, directly above the flask.
   Attach the small-bore reflux condenser to the adapter. Clamp the condenser.
   Attach water hoses so that water will flow from bottom to top.
   Ensure stopcock on funnel is closed. Then, add 3.5 ml each of 1-chlorobutane and toluene to funnel. Do not put a stopper on the funnel. Turn on water.

3. Begin stirring while slowly and carefully adding the liquid solution.
   Be sure to close stopcock completely as soon as addition is complete.
   Note that both $HCl_{(g)}$ and heat are evolved.

   Caution – $HCl_{(g)}$ is corrosive and causes acid burns.
   Use gloves and avoid all contact with skin, eyes, and nose.

4. Continue stirring for total of 30 minutes.

5. Put the 125-ml separatory funnel on a ring clamped to a stand.
   Add 30 ml of ice-water. Then, slowly add 1 ml of concentrated HCl.
   Decant the reaction mixture from the 25-ml round-bottom flask into the separatory funnel.
   Dispose of the solids in the appropriately-labeled waste jar.

   Caution – Concentrated HCl is corrosive and causes acid burns.
   Use gloves and avoid all contact with skin, eyes, and nose.

6. Put stopper on funnel. Shake and vent at least three times.
   Then, allow layers to separate, and remove lower layer.
   The lower layer should be aqueous, but test with water after removing it.
   Place the aqueous layer in the appropriately-labeled waste jar.

   Caution - Pressure typically builds inside separatory funnel when shaken!
   Be sure to ***vent*** stopcock frequently while shaking!
   Do not vent towards yourself or any other individual!

7. Place organic layer in a 25-ml Erlenmeyer flask. Add up to 2 g of anhydrous $CaCl_{2(s)}$.
   Put a #0 stopper on the flask and allow organic layer to dry for 10 minutes.

8. Decant organic layer into a 25-ml round-bottom flask, and add a boiling stone.
   Set up for vacuum distillation using a 50-ml heating mantle, small bore condenser,
   and vacuum adapter. Attach a small round-bottom flask as the receiver.

9. Apply vacuum and heat. Maintain a very low water flow rate.
   Discard all distillate up to 115 ^{o}C (toluene) in the appropriately-labeled waste jar.
   Attach a clean, weighed, 25-ml round-bottom flask as the receiver.
   Collect product above 140 ^{o}C (typically between 165 ^{o}C and 175 ^{o}C).

   Caution – Apparatus will be very hot. Allow to cool before handling.

10. Weigh product and analyze with gas chromatograph and infrared spectrophotometer.
    Dispose of product in the appropriately-labeled waste jar.

Post-Lab Questions

1. Why are the first six steps performed in a hood? See step 3 notes.

2. This experiment will not work if the apparatus is wet.
   Show the balanced reaction that occurs between water and $AlCl_{3(s)}$.
   The two products are a solid and a gas.

3. What alkyl chloride could be used instead of 1-chlorobutane
   to obtain the same product?

4. Why is an excess of toluene used?
   Refer to class notes on Friedel-Crafts Alkylation.

5. Why does the hydride shift occur?
   Consider the relative stability of the carbocations.