

Properties of Hydrocarbons

Period 5 – June 15, 2011

From Bell, Clark and Taber, Chapter 16, pages 183 – 184 (Expts 16A,B,D,E,F)

Conduct this experiment in a hood! You will examine some chemical and physical properties of selected saturated, unsaturated and aromatic hydrocarbons. Several reagents used in this experiment must be handled with care. **Sulfuric acid and bromine** can cause burns and must be washed immediately with water if spilled on the skin. Exposure to vapors of **bromine and methylene chloride (dichloromethane)** should be avoided, so be sure to perform all work in the hood.

- Using clean disposable pipettes, place ~1.5 mL of the following compounds in separate small labeled test tubes. (**HINT:** The height of the liquid in the pipette required to obtain ~1.5 mL of sample is marked on the standard pipette taped on the side of the community hoods)
 - In three separate test tubes, place approximate 1.5 mL samples of the three known compounds: **cyclohexane, cyclohexene, toluene**
 - In two separate test tubes, place approximate 1.5 mL samples of two unknowns (assigned by your TAs). Each work group should obtain one of the following unknown pairs to work with:
 - Unknown A and Unknown B or
 - Unknown C and Unknown D or
 - Unknown A and Unknown D or
 - Unknown B and Unknown C
 - You will be using these test tubes containing knowns and unknowns to obtain samples for testing in the following experiments A, B, C, D and E below.
- Also have ready in your hood:
 - A warm water bath (use a 400 mL beaker half filled with water warmed on a hot plate) with the temperature adjusted between 50 - 60° C (no warmer than 60° C!).
 - 20 - 13 x 100-mm (medium sized) test tubes; separate the test tubes into 4 groups consisting of 5 test tubes each.
 - Two test tube racks.
- For each group of test tubes, number the tubes 1 thru 5 in order to track the knowns and unknowns which will be placed in the test tubes.

A. Test Water Solubility

- For the first group of 5 test tubes:
- Place ~2 mL of water in each test tube. **HINT:** Fill a 10 mL graduated cylinder with water and use a disposable pipette to remove 2 mL portions of water for addition to the test tubes.
- Using a disposable pipette, add 8 drops of the cyclohexane to the first test tube and swirl vigorously to determine if it is soluble in water. Do you observe a

second layer and/or globules suspended in the water or floating on top of the water or do you obtain a homogeneous solution? Look carefully since it is

sometimes difficult to distinguish the two phases. Save this and the mixtures from the following step 4. for density determinations.

4. Repeat the above procedure for each of your known and unknown compounds and record your results. Be sure that you have a disposable pipette dedicated to each of your knowns/unknowns. Cross contamination of samples will invalidate your results!

B. Test for Relative Density

Reexamine the above mixtures prepared above and observe whether the hydrocarbon is more dense (sinks to test tube bottom) or less dense (floats on top of water) than water.

C. Bromine Addition

1. In the community hood, use the solvent dispenser to dispense 1 mL of **methylene chloride (dichloromethane)** in each of the next group of 5 clean test tubes.
2. At your hood, add 5 drops of cyclohexane to the first test tube containing methylene chloride and then add 5 drops of your other knowns and unknowns to the remaining labeled test tubes containing methylene chloride.
3. Take these test tubes to the community hood and add in a dropwise fashion 3 drops of a 2% solution of bromine in methylene chloride to each tube with gentle swirling. As you add each drop of the bromine solution, note whether the bromine color remains or disappears after the addition. Color disappearance (but not color dilution!) indicates the compound is unsaturated.
4. Record your observations for each known and unknown.

D. Potassium Permanganate Oxidation

1. In the community hood, use the solvent dispenser to dispense 1 mL of acetone in each of the next group of 5 clean test tubes.
2. At your hood, add 5 drops of cyclohexane to the first test tube containing acetone and then add 5 drops of your remaining knowns and unknowns to the remaining test tubes containing acetone.
3. Take these test tubes to the community hood and add in a dropwise fashion 3 drops of a 1% aqueous solution of potassium permanganate to each tube with gentle swirling. As you add the permanganate, note whether the purple permanganate color remains, disappears or changes color after the addition. Do you see formation of a precipitate? After the addition, be sure to allow the tube to sit for 5 – 10 minutes to make your final observation since the permanganate oxidation may not be instantaneous. Again, disappearance of the purple color indicates the presence of an unsaturated compound.
4. When the purple permanganate color disappears, what else do you observe in the test tube? Explain this observation.
5. Record your observations for each known and unknown.

E. Reaction With Sulfuric Acid

1. In the community hood, use the solvent dispenser to dispense 1 mL of **concentrated sulfuric acid (USE CAUTION; SULFURIC ACID CAUSES**

BURNS IF SPILLED ON SKIN AND CLOTHING) in each of the next group of 5 clean test tubes.

2. Add 6 drops of cyclohexane to the first test tube containing sulfuric acid. Reaction of a compound with sulfuric acid is indicated by dissolution of the sample, a color change, production of heat, and/or formation of an insoluble material. If the cyclohexane does not initially dissolve or otherwise react, carefully warm the test tube in a warm (50 – 60°C, no higher than 60° C!) water bath for 3 - 5 minutes (with constant swirling), making sure that the hydrocarbon does not evaporate. Observe whether the cyclohexane dissolves/reacts after warming. While **saturated aliphatic hydrocarbons** do not react with sulfuric acid, **unsaturated aliphatic hydrocarbons** do. Simple **aromatic hydrocarbons** react with warm (but not room temperature) sulfuric acid to form an aromatic sulfonic acid which dissolves to give a solution.
3. Repeat this procedure for each of the remaining knowns and unknowns and record your results.

F. Interpretation of Results

Interpret your observations for the known and unknown compounds. You should answer the following questions:

1. What are your conclusions about the solubility of saturated, unsaturated and aromatic hydrocarbons in water?
2. Are hydrocarbons more or less dense than water?
3. Which of your known and unknown compounds reacted with bromine? Do your observations correlate with what you expected regarding the reactivity of hydrocarbons with bromine?
4. Which of your known and unknown compounds reacted with potassium permanganate? Do your observations correlate with what you expected regarding the reactivity of hydrocarbons with potassium permanganate?
5. Which of your knowns and unknowns reacted with concentrated sulfuric acid? Do your observations correlate with what you expected regarding the reactivity of unsaturated compounds with sulfuric acid?
6. Based on the results of your tests, which of your unknowns are saturated and unsaturated hydrocarbons?
7. By using both your lecture (Carey & Giuliano) and lab (Bell, Clark and Taber) texts, write equations illustrating what happens to **cyclohexene** during the reactions with bromine, potassium permanganate and sulfuric acid. Where it is pertinent, indicate the proper stereochemistry of the products and indicate why that stereochemistry resulted.