EXPERIMENT 2

Properties of Alkanes, Alkenes, and Alcohols - Melting Point, Boiling Point, Solubility, and Reactions

Materials Needed

Compounds to be tested: cyclohexane, cyclohexene, octadecane, *tert*-butyl alcohol, ethanol, glycerol
Glassware: at least 6 small test tubes, Pasteur pipets and bulb, mp capillary tube
Reagents: distilled water, hexane, 3 M HCl(aq), 3 M NaOH(aq), conc. H_2SO_4(aq), Br_2/CCl_4, CrO_3/H_2SO_4, acetone
Apparatus: MelTemp melting point apparatus

Additional Reading Assignment

Denniston, Chapters 11.2, 11.5, 12.1, 12.5, 13.1, 13.4, 13.5.

Introduction

A main focus of much of this course is on how the structure of organic molecules determines their properties, both physical and chemical. Physical properties include such things as melting point (mp), boiling point (bp), refractive index (n_D), density (d), and optical rotation (α_D). Solubilities in various solvents and the types of reactions a compound undergoes are considered chemical properties. In this lab you will determine some of the properties of a small group of alkanes, alkenes, and alcohols. You will test the water solubility and hexane solubility of each compound. The reactivity of the compounds with certain reactants (3 M HCl(aq), 3 M NaOH(aq), conc. H_2SO_4(aq), Br_2/CCl_4 solution, CrO_3/H_2SO_4 solution) will be examined. You will also observe the mp of a solid alkane and the bp of a liquid alkane and look up literature values for the mps and bps of the other compounds being tested.

A hallmark of scientific experimentation is ***careful observation*** and ***noting of <u>all</u> experimental details***. To cultivate the habit of close observation, you should make a note of as many characteristics of the substances to be tested as possible. Always note the color and physical form of each compound you work with in the lab. As you perform the different tests, try to observe and note any subtle differences between the behaviors of different compounds. Such things as color changes, cloudiness, warming of the solution ("heat evolution"), fizzing ("gas evolution"), should all be carefully noted.

Background

Water Solubility Test. The general rule for solubility is "like dissolves like". Hence water, a very polar solvent, dissolves only the most polar organic compounds such as simple alcohols and amines. Non-polar compounds do not dissolve in water.

Hexane Solubility Test. Only non-polar compounds will dissolve in this very non-polar alkane solvent.

Reactivity with 3 M HCl(aq). This dilute form of hydrochloric acid only reacts with organic bases such as amines. Signs of reaction include heat evolution and dissolution. (If a compound does not dissolve in water but does dissolve in HCl(aq) then it must be reacting with the HCl.)

Reactivity with 3 M NaOH(aq). This base reacts with acidic organic compounds such as carboxylic acids and phenols. Signs of reaction include heat evolution and dissolution. (If a compound does not dissolve in water but does dissolve in NaOH(aq) then it must be reacting with the NaOH.)

Reactivity with Concentrated Sulfuric Acid. Concentrated sulfuric acid is a very strong acid that is capable of protonating compounds that have available electron pairs. The unshared pairs of electrons in oxygen and nitrogen containing compounds are readily protonated as are the extra electrons in a double or triple bond. Signs of reaction include strong evolution of heat or gas fumes and a marked color change.

Reactivity with Bromine. (See pp 340-342 in Denniston). Br_2 readily adds to the double bond of an alkene to form a dibromoalkane. (See p 429). A reaction is indicated by the disappearance of the red color of the bromine.

Reactivity with CrO_3. (See pp 377-379 in Denniston). The chromium(VI) is an oxidizing agent that can oxidize alcohols. A reaction is indicated by a change in color from orange to green:

$$\underset{\text{orange}}{Cr^{6+}} + 3e^- \longrightarrow \underset{\text{green}}{Cr^{3+}}$$

Boiling Point. The bp of a compound depends on the strength of the intermolecular forces between individual molecules of the compound. Compounds that can form hydrogen bonds between their molecules have higher bps than those that cannot. Non-polar compounds, such as alkanes and alkenes, have relatively low bps because the only intermolecular forces are weak London dispersion forces. Also, boiling points generally increase with increasing molecular weight because the overall strength of the London dispersion forces increases with increasing molecular size. The best way to measure the bp of a compound is to distill it.

Melting Point. Like the boiling point, the mp of a compound depends on the strength of the intermolecular forces between individual molecules of the compound. However, mp is more complex because it also depends on the ability of the molecules to pack tightly in a crystal lattice. All other things being equal, more symmetrical molecules pack better and, thus, have higher mps. Melting points are usually measured using a small sample at the bottom of a capillary tube. The tube is placed in a heating medium and the temperature is then slowly increased. The sample is observed and the temperatures at which melting starts and is finished are noted. Always report both the start and end temperature of the melting point range.

LABORATORY PROCEDURE

SAFETY PRECAUTIONS

- **WEAR YOUR SAFETY GOGGLES AT ALL TIMES.**
- IT IS RECOMMENDED THAT YOU WEAR DISPOSABLE GLOVES WHEN CARRYING OUT THE SOLUBILITY AND REACTIVITY TESTS.
- IF YOU GET A SOLUTION ON YOUR SKIN, BE SURE TO FLUSH THE AREA PROMPTLY WITH LOTS OF WATER.
- **TAKE SPECIAL CARE WITH CONC. H_2SO_4. IF YOU GET CONCENTRATED H_2SO_4 ON YOUR SKIN OR CLOTHING, RINSE IT WITH WATER IMMEDIATELY. DOING SO WILL AVOID SERIOUS INJURY.**
- IF YOU ACCIDENTALLY SPILL CONC. H_2SO_4, Br_2, or CrO_3 SOLUTIONS ANYWHERE IN THE LAB NOTIFY THE INSTRUCTOR OR T.A. IMMEDIATELY.
- CARRY OUT ALL TESTS WHILE WORKING IN A FUME HOOD.
- DO NOT DUMP THE HEXANE, BROMINE, or CrO_3TEST SOLUTIONS DOWN THE DRAIN; USE THE WASTE CONTAINERS LOCATED IN THE FRONT FUME HOOD.

Proper lab procedures: note taking and sample labeling

Make sure to promptly and neatly record all observations directly on the provided data sheet. Label all test tubes! It is very easy to get them mixed up if you don't.

General procedure for solubility tests

Place approx. 0.5 mL (10 drops) of the liquid or 0.1 – 0.2 g (1 small spatula tip) of the solid to be tested, in a test tube and add a total of approx. 3-4 mL of the solvent (either distilled water or hexane) in several portions. Cover the tube with a cork or rubber sleeve stopper and shake it vigorously so as to mix the contents thoroughly after each addition of solvent. If the substance dissolves completely, record it as soluble. Continue shaking for at least five minutes if the substance does not dissolve at first in order to make sure you have allowed adequate time for dissolution to take place.

Sometimes, especially for liquids, it is difficult to decide whether or not a substance has dissolved. After thorough mixing, allow the sample to stand motionless for 2 minutes. If the original two liquid layers reappear, you should record the substance as insoluble. Likewise, if the solution becomes cloudy and does not clear upon standing, you can consider the substance insoluble. When testing liquid samples, be sure to look carefully for a layer of undissolved liquid, which may be either lying on top or at the very bottom of the test tube.

Solubility in H_2O. Test all six samples using water as the solvent. (Use the general procedure given above.) For insoluble liquids make sure to observe whether the substance floats on top or lies at the bottom of the tube.

Solubility in hexane. Test the solubility of all six samples using hexane as the solvent. Again use the general procedure given above. Save the resulting cyclohexane, cyclohexene, octadecane solutions for use in the bromine test. (Make sure the test tubes are labeled so that you remember what each one contains.) Discard the other test solutions in the hexane waste container in the hood.

Reactivity with 3 M NaOH(aq). Use the general procedure for solubility tests *to* ***test only those compounds that did not dissolve in water***. Use 3 M NaOH(aq) as the solvent. Watch carefully for any sign of reaction as discussed in the background.

Reactivity with 3 M HCl(aq). Use the general procedure for solubility tests to ***test only those compounds that did not dissolve in water***. Use 3 M HCl(aq) as the solvent. Watch carefully for any sign of reaction as discussed in the background.

Reactivity with Concentrated H_2SO_4. Make sure to use a ***dry*** test tube and carefully add ***only 5-6 drops*** (not 3 mL as in the other tests) of concentrated H_2SO_4. Observe the results carefully and note exactly what happens on the data table. If the substance reacts, heat will be evolved, and a dark color will appear.

Reactivity with Bromine. To the solutions left over from the hexane solubility tests add the Br_2/CCl_4 solution dropwise observing the solution after each drop. Add at least ten drops to each solution and note the results carefully on your observations table.

Reactivity with CrO_3. ***Test the three alcohols only.*** Place ***1-2 drops*** of the alcohol in a test tube and add a total of approx. 3 mL of acetone. The alcohol should easily dissolve. Now add ***5-6 drops*** of the CrO_3/H_2SO_4 solution. Make sure to observe carefully the color of the CrO_3/H_2SO_4 solution before adding it and the color of the test solution after adding the CrO_3/H_2SO_4.

Boiling Point. Observe the boiling point of cyclohexane by reading the thermometer of the ongoing distillation set up by the instructor in the hood.

Melting Point. Use one of the Meltemp apparatuses available in the lab to measure the mp range of octadecane. The instructor will demonstrate the procedure.

PRE-LABORATORY QUESTIONS

EXPERIMENT 2

Properties of Alkanes, Alkenes, and Alcohols - Melting Point, Boiling Point, Solubility, and Reactions

Name _____ Date _____

1. Give the structures of all six compounds being tested in this lab.

2. Which compound would be expected to have the highest boiling point, cyclohexene, *tert*-butyl alcohol, or glycerol? Explain your answer completely by identifying the types of intermolecular forces involved.

3.Use a chemistry textbook, the Internet, or other reference to find a description of what a distillation is. Below, either use a labeled sketch or describe in words how a distillation apparatus works.

IN-LAB OBSERVATIONS/DATA

EXPERIMENT 2: Properties of Alkanes, Alkenes, and Alcohols - Melting Point, Boiling Point, Solubility, and Reactions

Names _____Date _____

Cyclohexane:

general observations _____
_____

solubility in water _____
_____

solubility in hexane _____
_____

reactivity with 3 M NaOH _____
_____

reactivity with 3 M HCl _____
_____

reactivity with conc H_2SO_4 _____
_____

reactivity with Br_2 _____
_____

boiling point _____barometric pressure_____

Cyclohexene:

general observations _____
_____

solubility in water _____
_____

solubility in hexane _____
_____

reactivity with 3 M NaOH _____
_____

reactivity with 3 M HCl _____
_____

reactivity with conc H_2SO_4 _____
_____

reactivity with Br_2_____
_____

EXPT #2- IN-LAB OBSERVATIONS/DATA (continued)

Names _____Date _____

Octadecane:

general observations _____
_____

solubility in water _____
_____

solubility in hexane _____
_____

reactivity with 3 M NaOH _____
_____

reactivity with 3 M HCl _____
_____

reactivity with conc H_2SO_4 _____
_____

reactivity with Br_2 _____
_____

melting point range found _____ literature mp and ref_____

***tert*-Butyl alcohol:**

general observations _____
_____

solubility in water _____
_____

solubility in hexane _____
_____

reactivity with 3 M NaOH _____
_____

reactivity with 3 M HCl _____
_____

reactivity with conc H_2SO_4 _____
_____
_____

reactivity with CrO_3/H_2SO_4 _____
_____
_____

EXPT #2- IN-LAB OBSERVATIONS/DATA (continued)

Names _____Date _____

Ethanol:

general observations _____
_____

solubility in water _____
_____

solubility in hexane _____
_____

reactivity with 3 M NaOH _____
_____

reactivity with 3 M HCl _____
_____

reactivity with conc H_2SO_4 _____
_____
_____

reactivity with CrO_3/H_2SO_4 _____
_____
_____

Glycerol:

general observations _____
_____

solubility in water _____
_____

solubility in hexane _____
_____

reactivity with 3 M NaOH _____
_____

reactivity with 3 M HCl _____
_____

reactivity with conc H_2SO_4 _____
_____
_____

reactivity with CrO_3/H_2SO_4 _____
_____
_____

REPORT - EXPERIMENT 2

Results Summary Table **EXPT. 2 Properties of Alkanes, Alkenes, and Alcohols**

Name _____Date _____

Compound Name **and Structure**	Appearance and Physical State	Solubility Tests			Reactivity Tests					bp (°C)		mp (°C)	
		H_2O	density	hexane	NaOH	HCl	H_2SO_4	Br_2	CrO_3	expt'l	lit	expt'l	lit
cyclohexane									na			na	
cyclohexene									na	na		na	
octadecane									na	na			
tert-butyl alcohol								na		na		na	
ethanol								na		na		na	
glycerol								na		na		na	

"na " = not applicable

QUESTIONS (If you find it difficult to fit your answer neatly in the spaces provided then feel free to attach a separate typewritten sheet).

1. Only one of the three compounds tested should have shown any reaction in the bromine test. Which one is it? Explain. Write an equation for the reaction it underwent.

2. Were the water insoluble liquids more or less dense than water? Suggest a reason for this (hint: think in terms of intermolecular forces.).

3. Use reasoning based on intermolecular forces to explain fully the reason why octadecane is a solid while hexane is a liquid.

4. Are alkanes considered to be acids or bases, both, or neither? Look at the results of your NaOH and HCl tests to decide. Explain.

5. Which of the alcohols tested react with oxidizing agents such as CrO_3? One of the tested alcohols does not react. Which one is it and why doesn't it? Refer to chapter 13.5 in Denniston if necessary.