**CHEMISTRY 3514 ORGANIC CHEMISTRY 2 Oct 2014**

**LABORATORY #6: IDENTIFICATION OF AN ORGANIC MIXTURE VIA GAS CHROMATOGRAPHY**

 **AND INFRARED SPECTROSCOPY (Due Thursday Oct 9, 2014)**

**6.1. Introduction**

In the several labs you have performed thus far, the emphasis has been on separating and then characterizing substantially pure compounds.

Because you’ve ultimately obtained `pure’ compounds, melting point or boiling point as well as qualitative features like color ( bone white, snazzy red, snot green) sufficed to positively identify the compound.

Often, however, a mixture of compounds is obtained for which it would be inconvenient or unnecessary to perform a separation. In such case, organic chemists resort to several basic instrumental methods.

In this lab you will gain a working knowledge about two of them: gas chromatography (GC) and Infrared Spectroscopy (IR).

**6.2. Purpose of the Lab**

Instrumental identification of the components in an unknown mixture of organic compounds prepared from the following list:

**methanol isopropanol butanol n-hexane n-heptane n-octane**

**CH3OH (CH3)2CHOH C4H9OH CH3(CH2)4CH3 CH3(CH2 )5CH3 CH3(CH2)6 CH3**

**5.3. Procedure**

***5.3.1. Characterization of Reference Compounds by IR and GC***

After the instructor demonstrates how to set up and use the IR and GC instruments (a PE Spectrum 1 FTIR and the HP 6890 GC with FID detectors):

**Part 1: IR survey bands of unknown**

a) Run your unknown using one of the two ATR FTIR and decide whether you have an alkane mixture, or, an alcohol mixture.

 Record on the IR itself, the conditions of the scan.

b) Tabulate in a Table in your lab note book under `**Observations**’ relevant band positions (cm-1, strengths e.g. vs, s, ms, m, w, vw and shapes, e.g. broad, doublet, multiplet ) you think best characterize each. If you think you know the molecular motion say so in **comments.** Compare you results to the reference FTIR spectra available near the FTIR instruments to ensure that you know what class of compounds you are dealing with.

“ ***Table 1: Characteristic Mid-IR bands for Unknown Mixture \_\_\_\_\_\_\_”***

 **Observed cm-1**  **strength** **shape** **comments**

 2950 vs doublet C-H symmetric stretch

 2850 vs multiplet C-H asymmetric stretch

 1470 m multiplet CH2 bend

 1375 m multiplet CH3 bend

**Part II: GC chromatogram of unknown mixtures**

c) Based on your survey scan above, pick which GC to use. Record in your lab book under **Observations** in a Table labeled `**GC conditions’ t**he particular GC you are using (alkane instrument or alcohol instrument) inlet temperature, gas flow, split ratio, thermal ramp conditions, injection method (wet needle) and name of the GC file you are going to store. Make sure to clearly label the table.

 Example: ***Table 2: GC conditions for surve of unknown mixture \_\_\_\_\_\_***

 GC used: alkane instrument

 ` inlet T 400 C

 He flow 0.3 mL/min

 Split 100:1

 Ramp 50🡪 150 at 10 degrees/ min

 Injection wet needle

 File xxxx

Run the instrument as indicated by the instructor. Record and print your chromatogram, making sure to transfer table 2 directly onto the hard copy of the chromatogram .

**Tape the the raw data (IR spectrum for unknown and GC for unknown) in your lab book in the Observations section, making sure to include the unknown ID.**

**Part III: Analysis**

Use the reference chromatograms to help you decide which of the various component alcohols or alkanes you have. These reference chromatograms can be directly taped into your lab books along with your unknown chromatograms.

**6.4. What and How to Report**

In Results summarize in table form

**1) the class of compounds you think you have (alcohols or alkanes ?) and why, based on FTIR bands:**

Example:

**Compound class of unknown \_**\_\_\_: ***Alkanes:***

 ***observed typical C-H stretches near 2950-2850 and both CH2 and CH3 bends in the 1470-1350 cm-1 regions.***

***There are no indications of broad, OH bands near 3400 cm-1, eliminating ROH as a class.***

 **2) the retention times obtained for both your unknown and versus reference mix in a table, e.g.**

 **Table 3: Proposed unknown identification based on GC**

 **Compound observed tr(min) reference tr**

 **Heptane 2.13 2.16**

 **Octane` 3.34 3.38**

 TURN IN THE ENTIRE LAB NOTEBOOK WITH THE FOREGOING ALL ATTACHED.

 ( NO LOOSE PAGES OR CHARTS !!!)

REQUIRED SOLVENTS FOR IR/GC LAB

**methanol acetone isopropanol n-hexane n-heptane n-octane**

**CH3OH (CH3)2CO (CH3)2CHOH CH3(CH2)4CH3 CH3(CH2 )5CH3 CH3(CH2)6 CH3**

**UNKNOWN PREPARATIONS**

**SAMPLE COMPONENT A (5 ml) COMPONENT B (5 ml) TEAM**

#1 METHANOL ACETONE Dara/Christy

#2 ISOPROPANOL METHANOL Vicky/Ellen

#3 HEXANE OCTANE Becky/Nancy

#4 ACETONE OCTANE Wayne/Brandon

#5 METHANOL HEPTANE Julie/Michal

#6 ACETONE ISOPROPANOL Thien/Phuong

#7 OCTANE METHANOL Chris/April/Crystal

**1:1:1 REFERENCE MIX PREPARATIONS (5 mL:5mL:5mL)**

Reference mix #1 hexane:heptane:octane

Reference mix #2 methanol:isopropanol: aceton