**CHEMISTRY 3514 ORGANIC CHEMISTRY**

**LABORATORY #3: Identification of an Organic Mixture via Gas Chromatography**

**and Infrared spectroscopy (Due Monday 18 September)**

**3.1. Introduction**

You must first read the lab module:

**Infrared Spectroscopy and Gas Chromatography:**

**How Chemists Know `Who Be What?’**

**3.2. Purpose of the Lab**

Identification of the components in an unknown mixture of organic compounds prepared from the list below using FTIR and GC :

**ethanol 1-butanol cyclohexanol**







**hexane cyclohexane octane**







**propanone (acetone) 2-butanone (methyl ethyl ketone) cyclohexanone**







**3.3. Procedure**

***3.3.1. Identifying Compounds by IR and GC***

Although the instructor will demonstrate how to set up and use the IR and GC instruments (PE Spectrum FTIR and the HP 6890 GC with FID detectors), make sure to read **Practical Notes on IR** (section 3 of GC/IR lab module), and **Practical Notes on GC** (section 5 of GC/IR lab module.)

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

a) Run your unknown using one of the three ATR FTIR and decide what functional group(s) are present in your unknown based on

position and shape of the observed bands. Record on the IR spectrum itself, the conditions of the scan.

b) Tabulate in a Table (see Table 1 example below) 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. You must use `Bart’ for alcohols and ‘Lisa’ for alkanes. Ketones can be observed on either. Record in your lab book under **Observations** in a Table labeled `**GC conditions’ t**he particular GC you are using (Bart or Lisa) 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 survey of unknown mixture \_\_\_\_\_\_***

GC used: Lisa

` inlet T 300 C

He flow 0.3 mL/min

Split 100:1

Ramp 50🡪 150 oC at 10 degrees/ min; 30 second hold at 150 oC

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

**3.4. What and How to Report**

In **Results** summarize in table form:

**1) the class(es) of compounds you think you have (just alcohols, just alkanes ? alcohols + ketones) and your Rationale for**

**why, based on FTIR bands:**

Example:

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

***Rationale:***

*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. There is no strong, sharp singlet at or near 1700 cm-1 eliminating ketones as a class.*

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

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

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

**Cyclohexane 0.79 0.79 Lisa**

**Octane` 1.34 1.35 Lisa**

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

( NO LOOSE PAGES OR CHARTS !!!)