**Laboratory #6: Gas Chromatography-Mass Spectroscopy (GC-MS)**

**as a Tool to Determine Organic Compositions**

**Due Friday (before break) 10 March (25 pts)**

**Introduction:**

It is common in organic synthesis to come to the end of a long train of individual preparative steps and wonder if more than one product has been. This is particularly so when carrying out new syntheses, but also arises after running purportedly well-known preparations.

So far, we have not had the tools to deal with a true mixture of unknowns. While GC analysis can answer the question of how many components, IR alone will not be sufficient help to identify specific compounds . If a mixture is being analyzed, the spectra will only reveal that certain functional groups are present, but not which group goes with which molecular species in the mixture.

Using a combination of GC and a `mass selective detector’ (MSD), however, can provide definitive answers to both how many and what components are in the product mix. This is accomplished because the GC peaks, which contain pure and separated components, are separately `cracked’ in the MSD into characteristic molecular fragments whose masses and intensities are recorded, and whose specific `cracking pattern’ provides a molecular fingerprint for the compound being cracked. The observed pattern can then be compared against reference patterns from NIST (National Institute for Standards and Technology) using standard mass spectral search programs

**Objectives of this Laboratory:**

Student teams will be given unknown organic mixtures and with the help of the instructor will run a GC-MS analysis with ASC’s 5890N GC-MS to ascertain the number and identity of the components in their unknown mixture using the resident HP Chem Station MSD software.

Additionally, students will be required to assign the observed masses in each component’s cracking spectrum to specific mass fragments. This is done to confirm the validity of the software’s prediction for the unknown identities.

What to Report (in your note books)

 0) Industry standard followed (all pages dated/signed/numbered) **2 pts**

1. **Purpose ( full sentences) 2 pts**
2. **Procedure 3 pts**

GC-MS run conditions and instrument settings

ID of instrument (make/model), thermal ramp schedule, injection volume (wet needle)

File name of stored data; unknown number, inlet temperature, split ratio, He flow rate

1. **Observations 4 pts**

MSD spectra and associated Table of main m/e and counts –title, labels should be clear

1. **Results 14 pts**

Chem station prediction of component identities with % confidence

Tabulation of major, diagnostic m/e fragments for each identified component vs fragment ID