CHM 254

Organic Chemistry Laboratory

October, 2013

Identification of an Unknown Organic Compound

Introduction

Before the development of modern spectroscopic techniques such as IR and NMR, the structure of an organic compound was determined by physical properties and chemical tests and by conversion to a solid derivative with known melting point. Today IR spectroscopy makes it easy to identify the functional groups present in a molecule. NMR methods can establish the carbon-hydrogen framework of the molecule. Mass spectroscopy provides molecular weight and whether halogen atoms such as chlorine or bromine are present in a molecule. A combination of these three spectroscopic methods provides a powerful tool for the identification of the structure of an unknown organic compound. However, qualitative organic analysis -the process of identifying a compound by chemical tests-still plays a role in organic chemistry laboratories.

Goals

Each group will identify an unknown sample of an organic compound using a combination of qualitative organic analysis and spectroscopic methods. Collect four spectra (IR, ^{1}H and ^{13}C NMR, and mass) for the unknown compound. Deduce the structure and submit your report with a set of spectra and your deduction logic. If the unknown is sufficiently complex and warrant additional information, your TA will help you to obtain DEPT ^{13}C NMR and 2D NMR spectra.

Procedure

In this unknown-identification lab, the following data should be collected. To make most efficient usage of the instruments, the order of collecting spectroscopic data should be staggered among student groups, i.e., one group is using IR while another group is using the NMR. Each group of four students will be assigned one of the ~15 unknown compounds.

- Determine whether your unknown is an acid, a phenol, a base (amine), or a neutral compound. (refer to lab manual pp. 293-303 on how to do this)

- Collect a good quality IR spectrum of your unknown, and save as a .png file with its #, e.g., unknown_13_IR.png (preferably in 905 x 495 px) and use a flash drive to transfer the file to your own computer.
- Collect a good quality ^{1}H NMR spectrum of your unknown, and save directly as a .png file in the Topspin software, e.g., unknown HNMR_45.png and email to yourself as an attachment.
- Collect a good quality ^{13}C NMR spectrum of your unknown, and save as a .png file, e.g., unknown CNMR_45.png.
- Submit your sample (~2 mg, label the vial with your sample #) to Dr. Fei Yang for a Mass Spectrum. Dr. Fei Yang will email you your mass spectrum to you so please put your email address on the form to be filled when you submit your sample.

After you have collected all of the required data, carefully deduce the structure of your unknown. Be careful with your sample because there is only limited amount of unknowns. Don't use any more than you need for one experiment. In your lab report, give detailed analysis for each set of data you collected and their relationship to the structure you have proposed. Merely give a final structure is not acceptable. The deduced structure should be labeled with the unknown number. Finally submit all of your graphic spectra images in .png file format to Dr. Gung by email.

How to save screen plots in .png file format

1. NMR

After collecting your spectrum, save directly to .png file in Topspin.

2. IR

When your spectrum is on the computer screen, go to menu under Edit, click Copy, this puts the spectrum on the clipboard. Go to Start→Accessories→Paint. In the Paint program, go to menu under Edit, click Paste. Now your spectrum should show up in Paint. Using Save as to save in .png file format.