Assigning H's on NMR Spectra

Introduction

In this lab, you will be analyzing a set of $^1\text{H}$ NMR spectra by assigning the hydrogens in a compound to sets of peaks in the spectrum.

Please read "Analyzing NMR Spectra" before coming to lab. Also, review the lecture notes on NMR spectroscopy (you may wish to bring them to lab for reference as well). You do not need to record this lab in your lab notebook.

The spectra that you will see in this lab were taken under actual experimental conditions, so they are not as simple as the ones we will deal with in class. There are some impurities, including solvent peaks and water, in some of the compounds. We will discuss how to deal with these.

When you come to lab, you will receive a packet of 15 proton NMR spectra. I have written the structure of each compound on its spectrum. You will match up the peaks with the hydrogens on the compound, following the procedure below.

Procedure

For each NMR spectrum:

• Draw the H's on each compound and divide them into sets of equivalent H's. Label each set with a letter.

• Make a table, listing the letter, integration, splitting, and expected chemical shifts for each set of H's.

• Identify the TMS peak, solvent peak, and possible water peaks on the spectrum.

• Measure the integration of each set of peaks that belong to the compound with a ruler. Write the value in mm, then write the number of H's represented by that peak ("2H" etc).

• Match up the letters in your table with the sets of peaks in the spectrum using all of the data (chemical shift, integration, and splitting). You may need to combine some of your letters if some of the peaks overlap. Kindly circle the letters so that I can find them easily!

When you have finished, put your name on the NMR packet and turn it in, along with the questions. You may write the answers on the lab and turn it in.
Questions

1. What H’s are likely to overlap in the following compound?

\[
\begin{array}{c}
\text{O} \\
\text{H} \\
\text{O} \\
\end{array}
\]

2. At what chemical shift do the following appear?
   a) CHCl₃
   b) TMS
   c) H₂O in CDCl₃
   d) DMSO-d₆
   e) H₂O in DMSO-d₆

3. How can you tell at a glance whether an NMR spectrum represents an aromatic compound?

4. How can you tell if 3 peaks close together are a triplet or a double and a singlet close together?

5. Based on this lab, at what chemical shift do OH’s on aromatic rings appear? How does this compare to regular OH’s?

6. Put the following in order, from farthest upfield to farthest downfield.

\[
\begin{array}{c}
\text{H} \quad \text{H} \\
\text{O} \\
\text{H} \quad \text{H} \\
\end{array}
\quad
\begin{array}{c}
\text{H} \quad \text{H} \\
\text{O} \\
\text{H} \quad \text{H} \\
\text{H} \\
\end{array}
\quad
\begin{array}{c}
\text{H} \quad \text{H} \\
\text{H} \\
\text{H} \\
\end{array}
\quad
\begin{array}{c}
\text{H} \quad \text{H} \\
\text{H} \\
\text{H} \\
\end{array}
\quad
\begin{array}{c}
\text{H}_2\text{N} \\
\text{H} \\
\text{H} \\
\text{H} \\
\end{array}
\]

7. What is the most likely integration ratio for the following measurements?
   a) 15mm, 30mm, 30mm, 45mm
   b) 6mm, 38mm, 20mm
   c) 7mm, 10mm, 13mm