

NMR Short Course 2010

Problem Set III

Use provided spectra to determine the structure of a certain natural product with molecular structure $C_{15}H_{22}O_5$ exhibits 16 proton signals, all of which are 1H multiplets except for 3H doublets at 0.99 and 1.21 ppm, a 3H singlet at 1.44, and a 1H singlet at 5.82 ppm (It is not shown on the proton spectrum).

The ^{13}C spectrum of this compound consists of 15 signals, the 11 signals shown at the HETCOR spectrum, plus signals at 80, 95, 106 and 172 ppm (They are not shown on the carbon spectrum).

The COSY and NOESY spectra for this compound are attached.

(1). From the data in the figure, prepare a table showing which hydrogens, as designated by their signal number, are attached to each carbon. From this, determine the multiplicity (CH₃, CH₂, CH, or C) of each carbon.

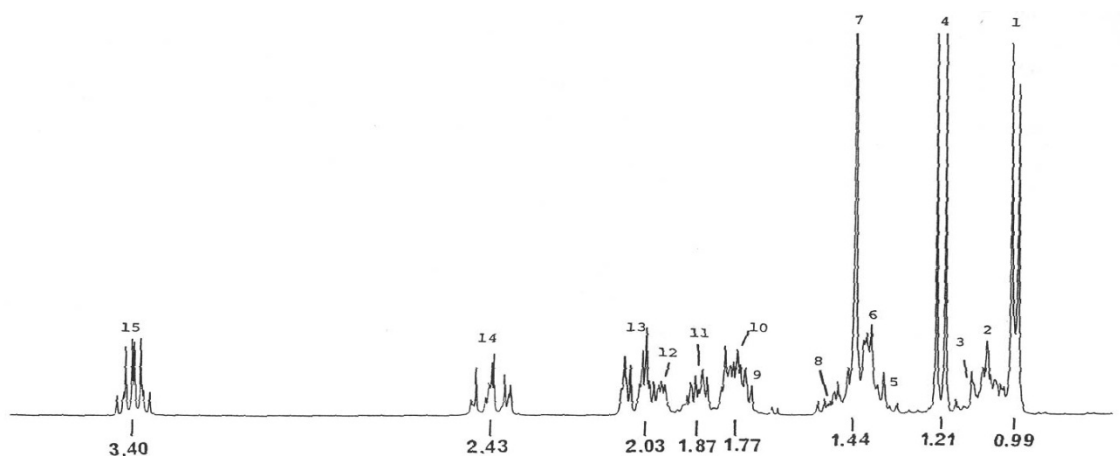
(2). From the COSY spectrum, prepare a table showing which protons are coupled to each other.

(3). From a comparison of COSY and NOESY, determine which protons, though not directly coupled through bonds, are nonetheless close in space.

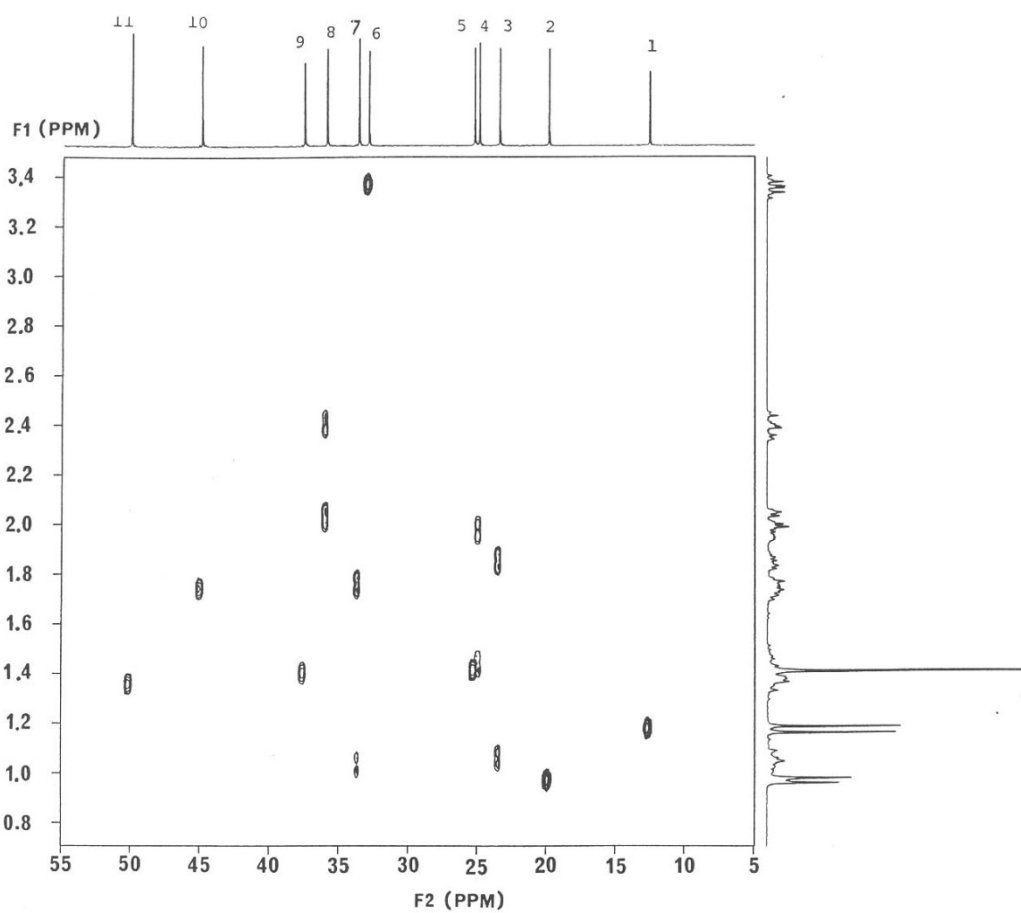
(4). Using the following 2D INADEQUATE spectrum data of the compound, as well as above information to complete a possible structure of the compound.

Carbon-Carbon Correlation:

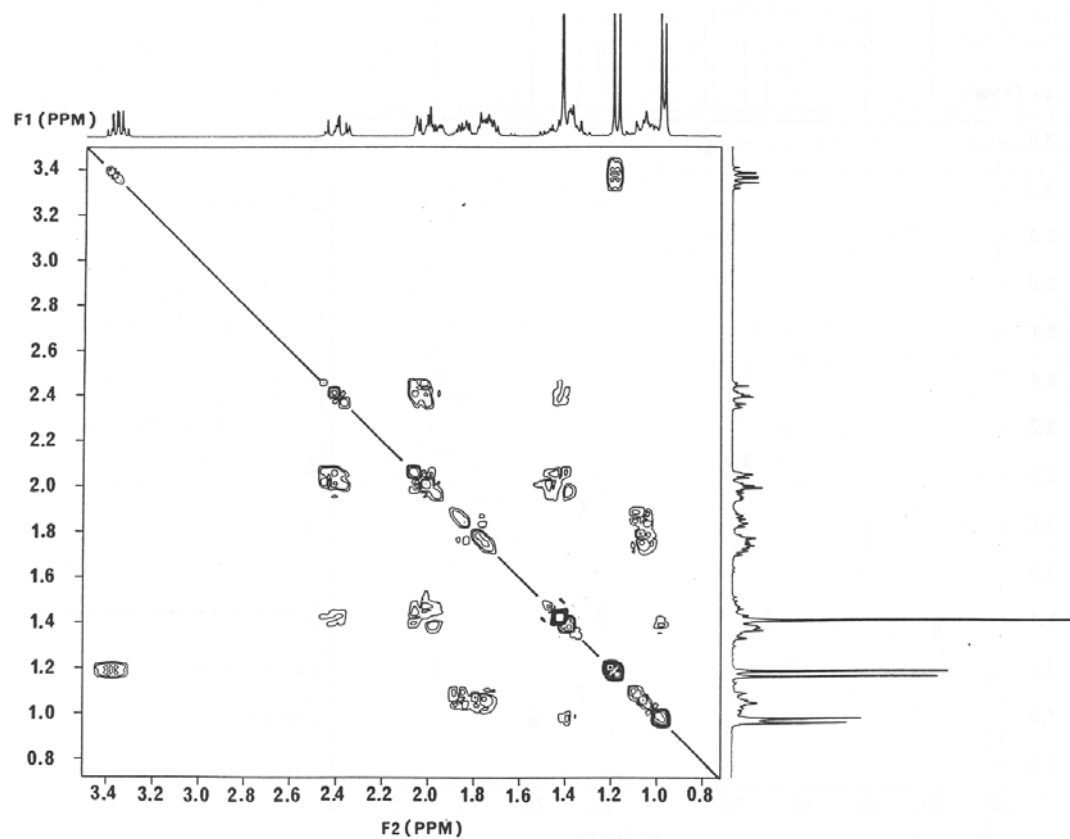
1 ----6	9 ----2,7,11
2 ----9	10 ----3,6,12
3 ----7, 10	11 ----4,9,12
4 ----8,11	12 ----10,11,13
5 ----14	13 ----12
6 ----1,10,15	14 ----5,8
7 ----3,9	15 ----6
8 ----4,14	



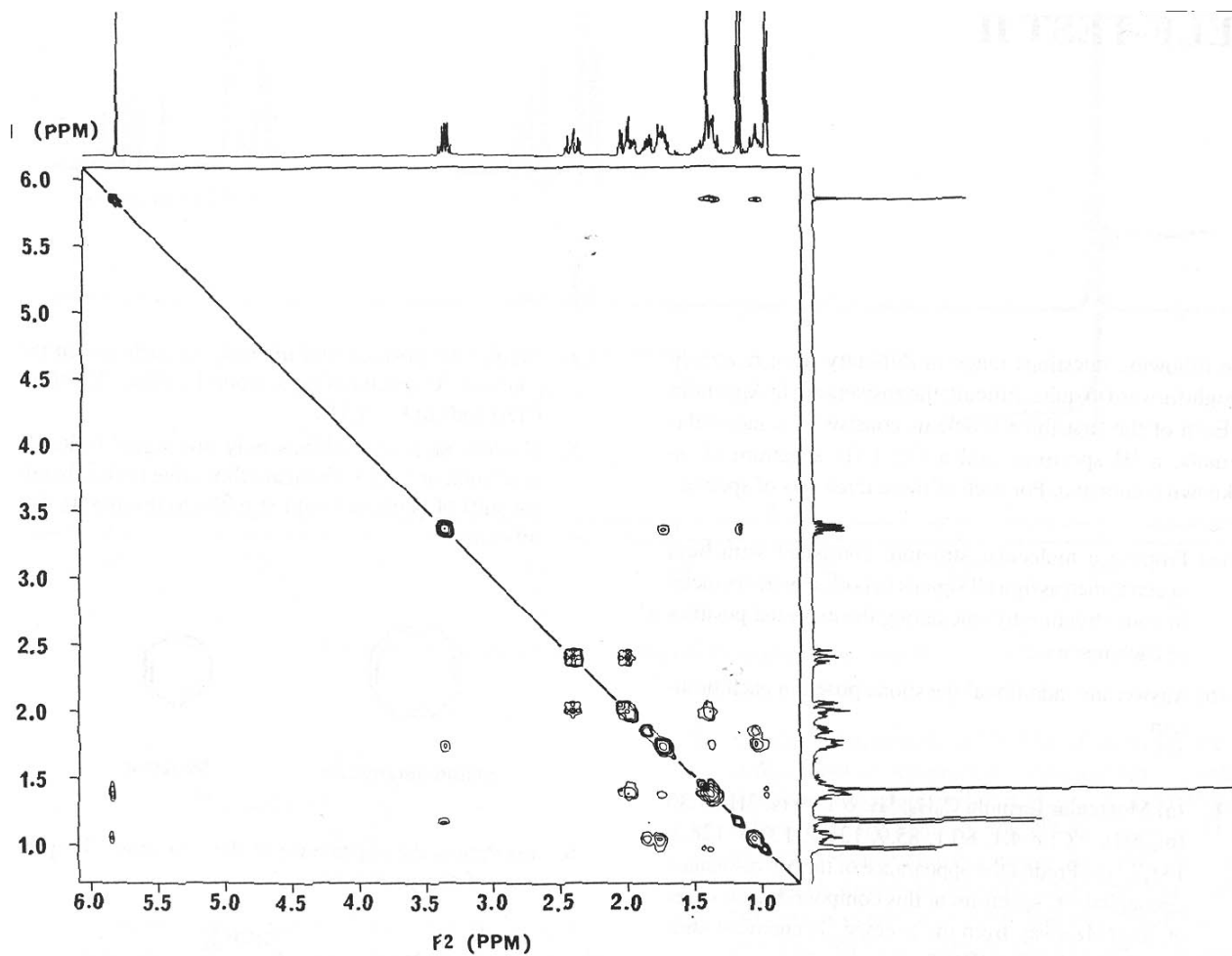
Partial ^1H spectrum of $\text{C}_{15}\text{H}_{22}\text{O}_5$. Note that the ^1H singlet at 5.82 ppm (signal 16) is not shown.



Partial 2D HETCOR spectrum of $\text{C}_{15}\text{H}_{22}\text{O}_5$. Note that the correlation between carbon signal 13 and hydrogen signal 16 is not shown.



The COSY spectrum of $C_{15}H_{22}O_5$.



The NOESY spectrum of $C_{15}H_{22}O_5$.