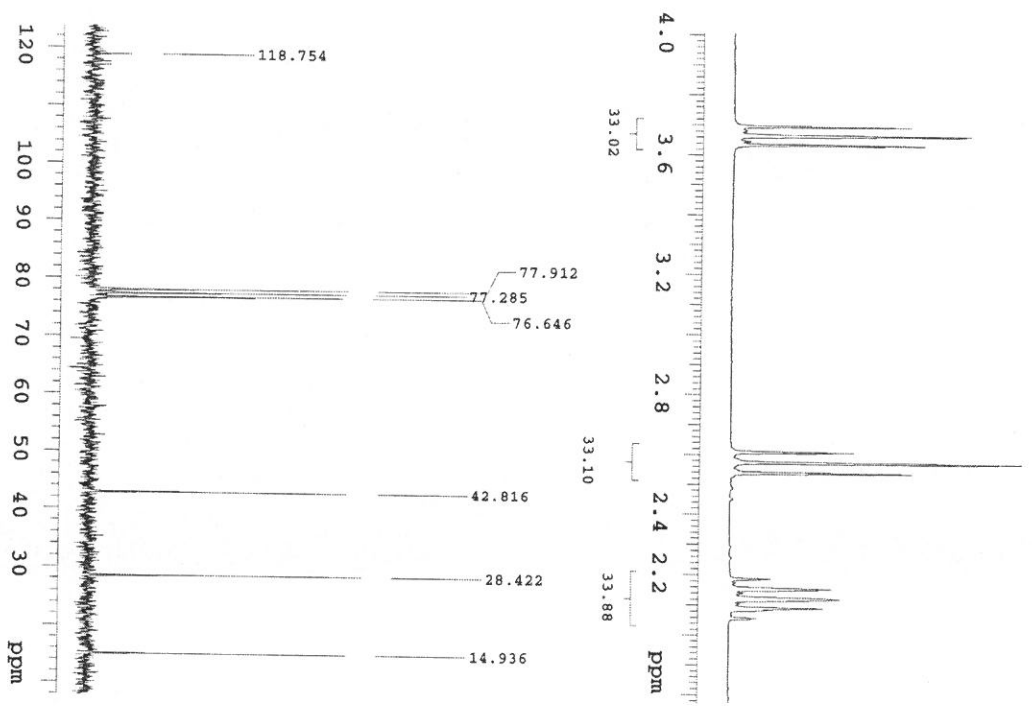
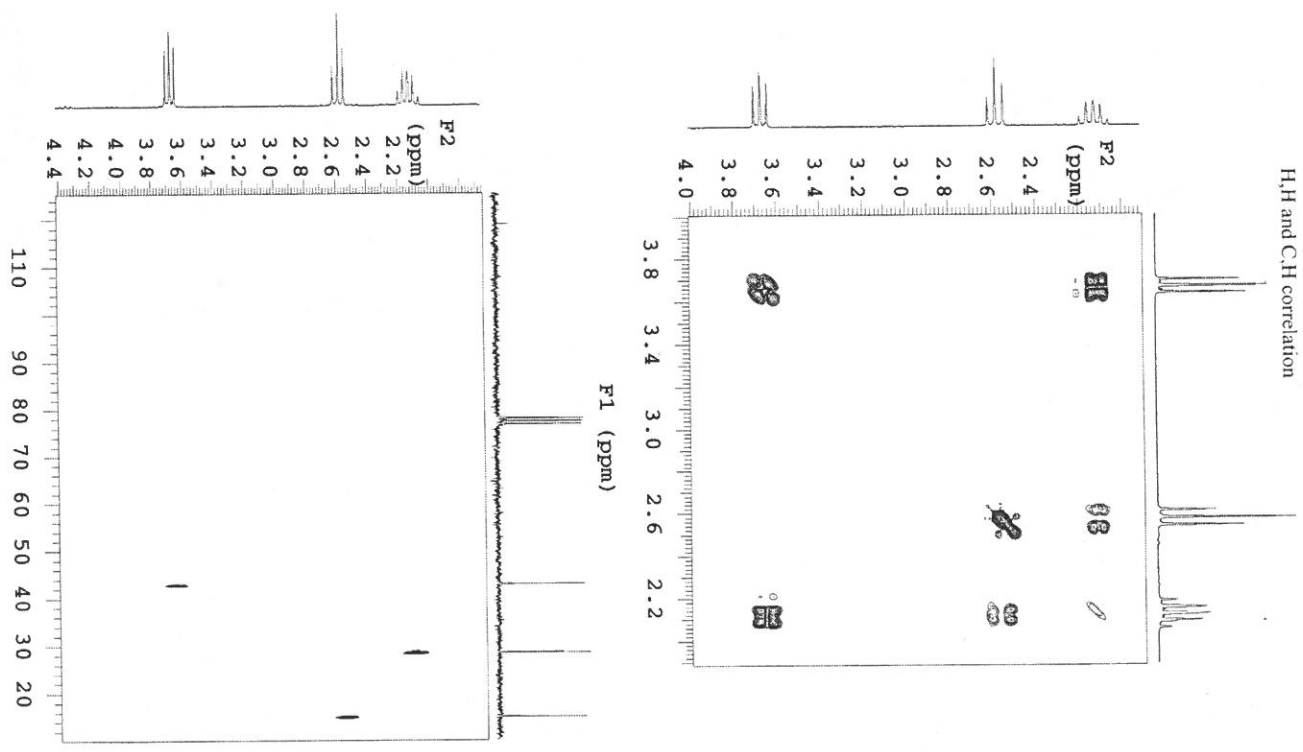


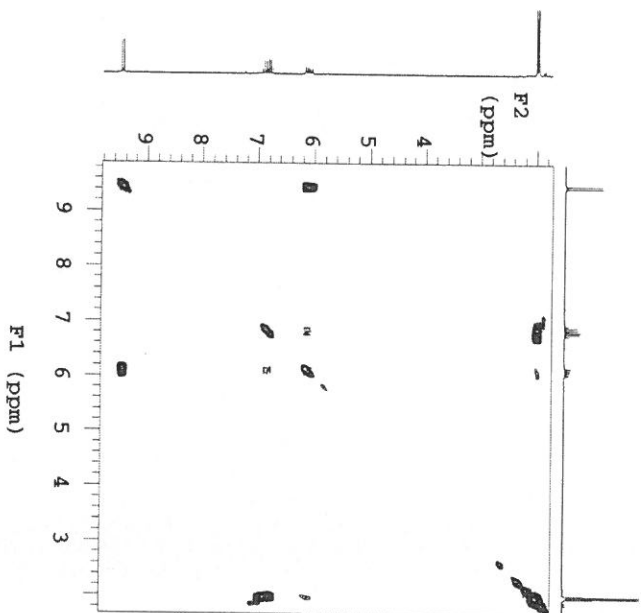
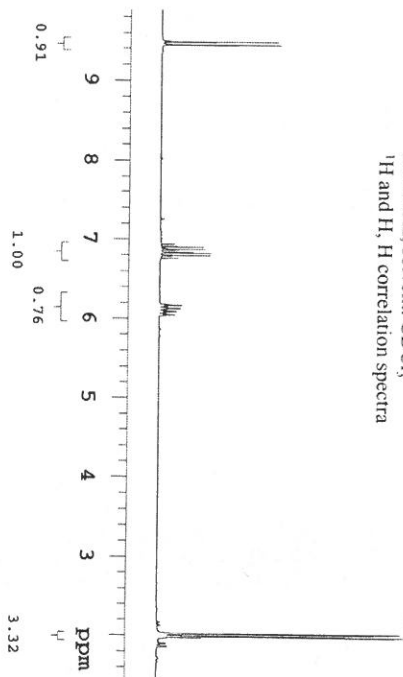
**Problem 1: C<sub>4</sub>H<sub>6</sub>NCl**  
 IR: 2249 cm<sup>-1</sup>  
 200 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H and <sup>13</sup>C spectra



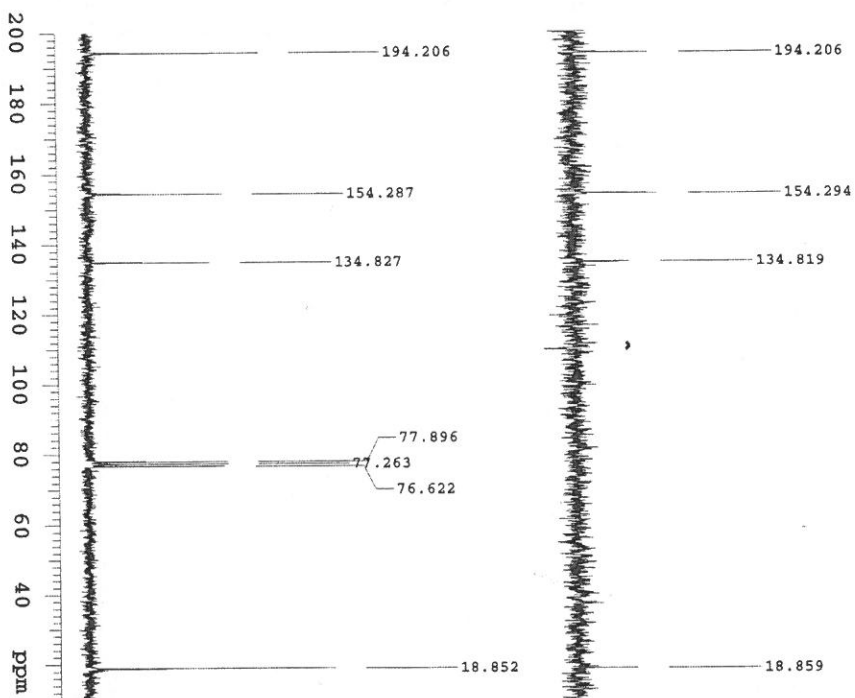
**Problem 1: C<sub>4</sub>H<sub>6</sub>NCl**  
 200 MHz, solvent: CDCl<sub>3</sub>  
 H,H and C,H correlation



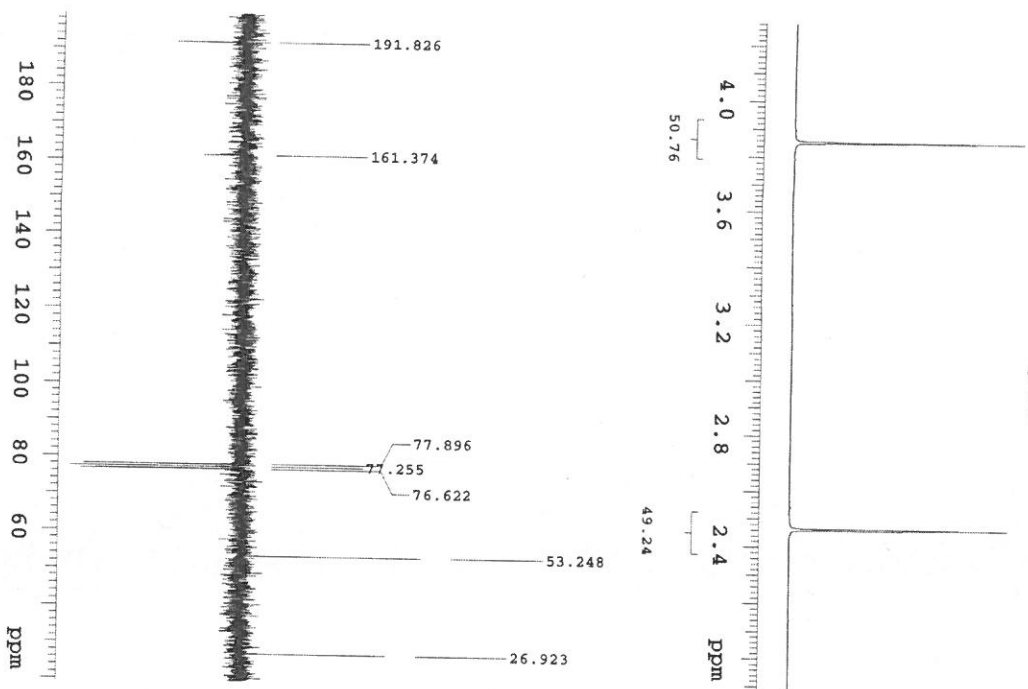
**Problem 2: C<sub>4</sub>H<sub>6</sub>O**  
 IR: 1654, 1691 cm<sup>-1</sup>  
 200 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H and H<sub>1</sub>, H correlation spectra



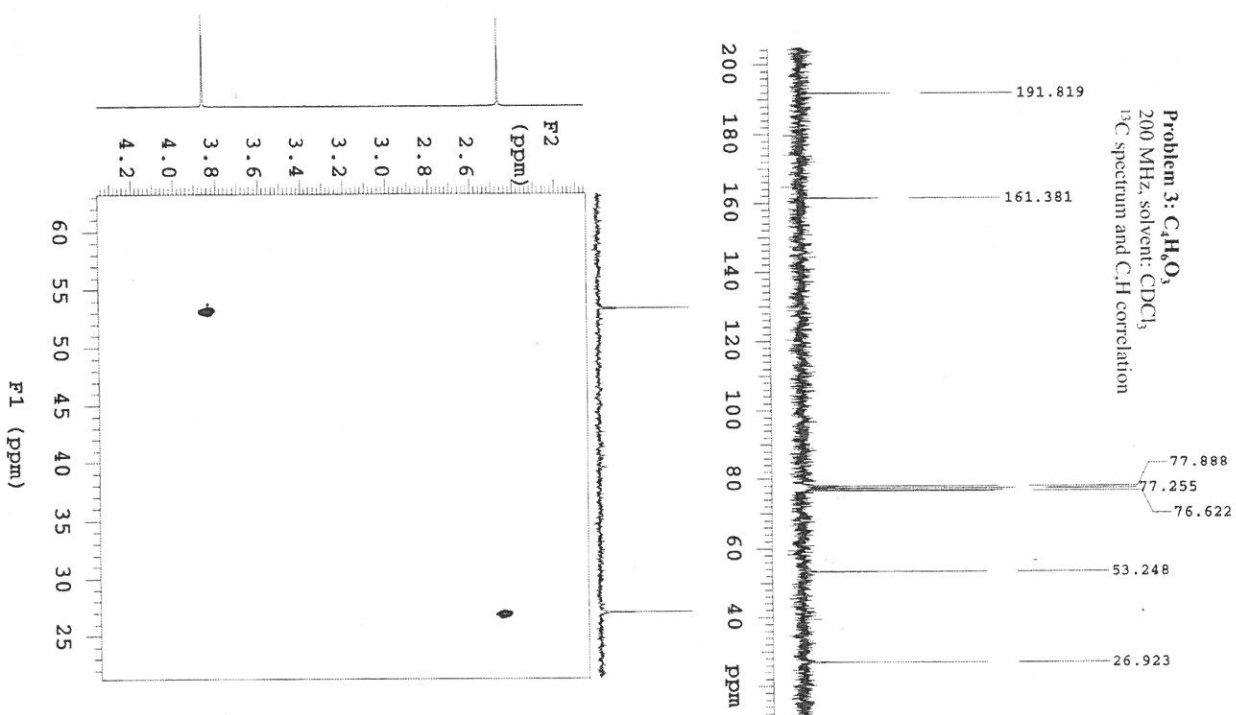
**Problem 2: C<sub>4</sub>H<sub>6</sub>O**  
 200 MHz, solvent: CDCl<sub>3</sub>  
<sup>13</sup>C and DEPT spectra



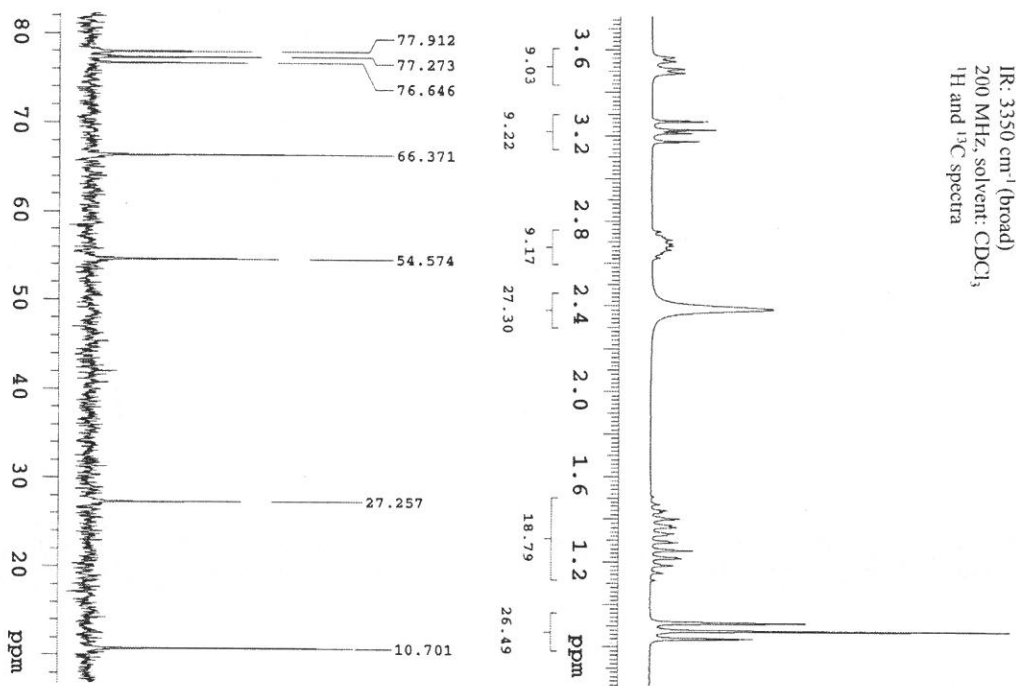
**Problem 3:**  $C_4H_6O_3$   
 IR:  $1730\text{ cm}^{-1}$  (broad)  
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



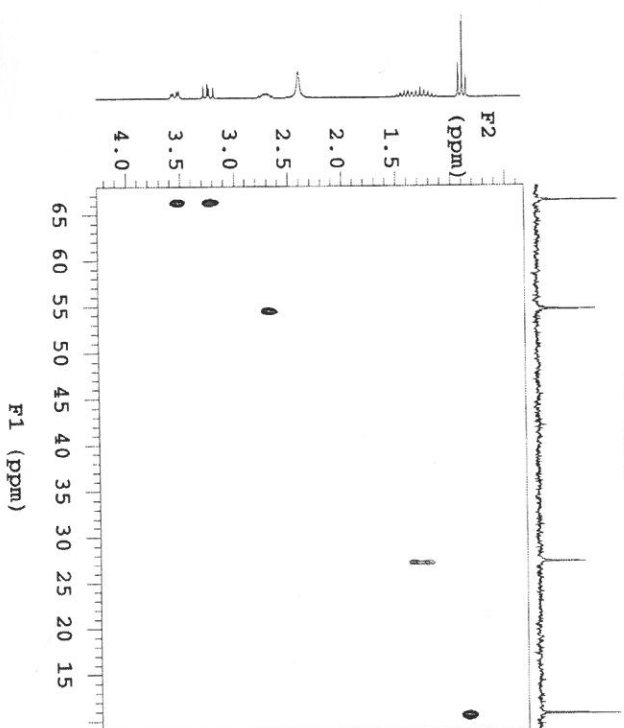
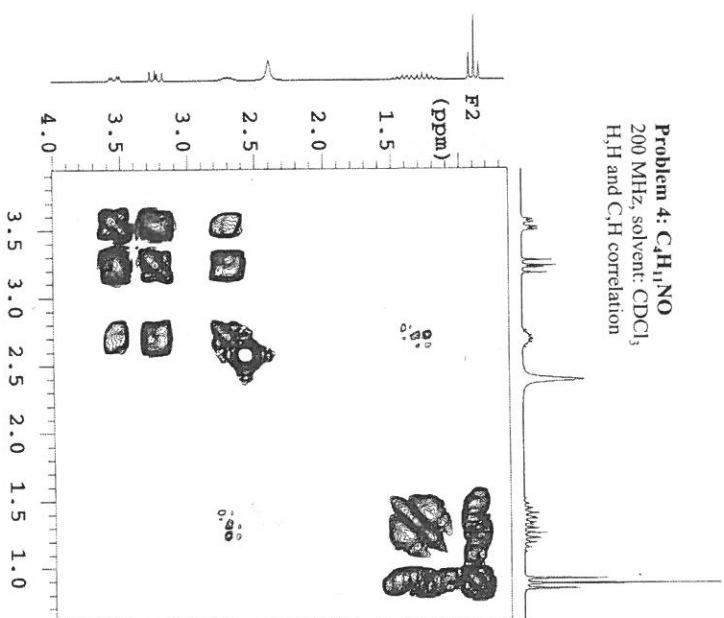
**Problem 3:**  $C_4H_6O_3$   
 200 MHz, solvent:  $CDCl_3$   
 $^{13}C$  spectrum and C,H correlation



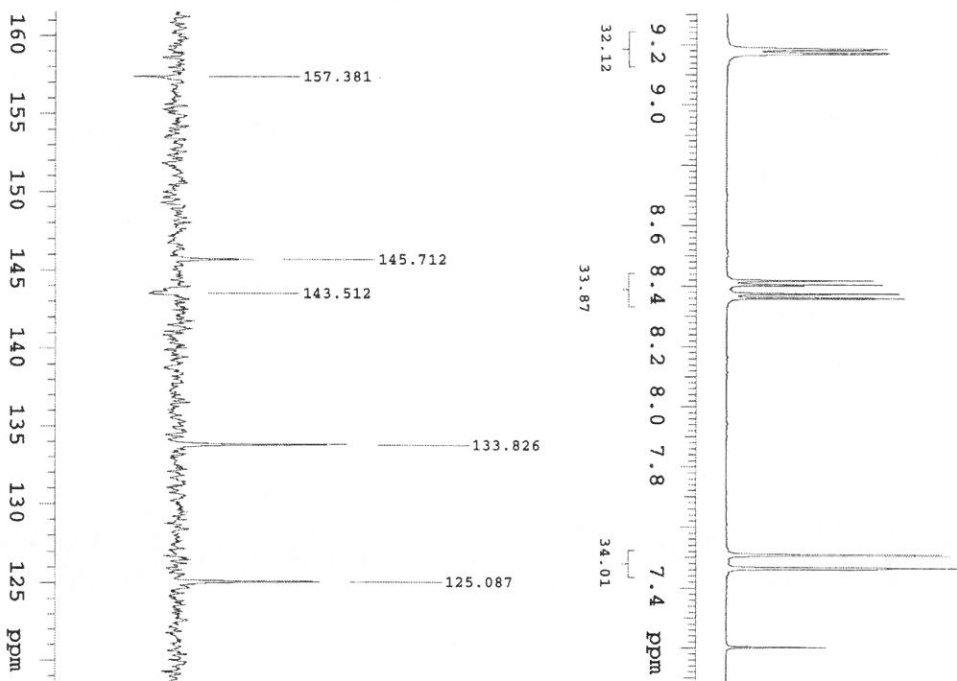
**Problem 4:**  $C_8H_{11}NO$   
 IR:  $3350\text{ cm}^{-1}$  (broad)  
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  spectra



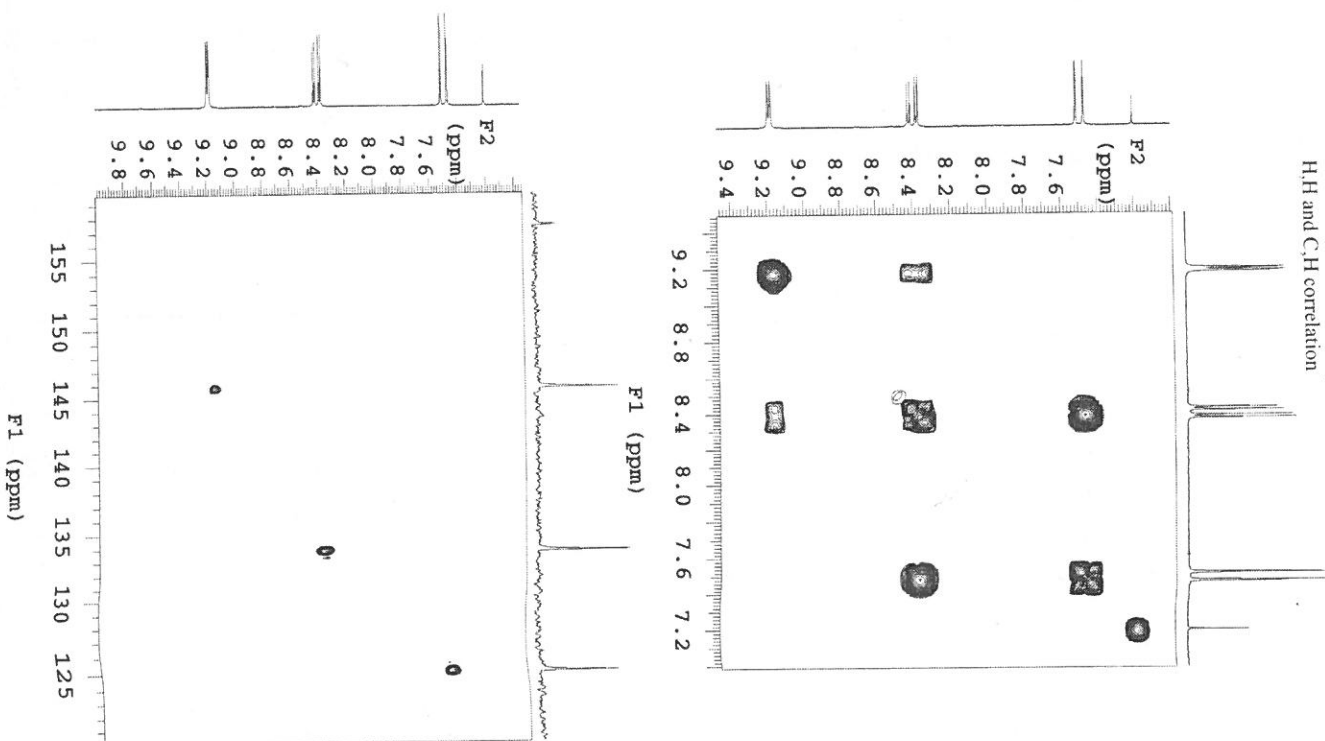
**Problem 4:**  $C_8H_{11}NO$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  correlation



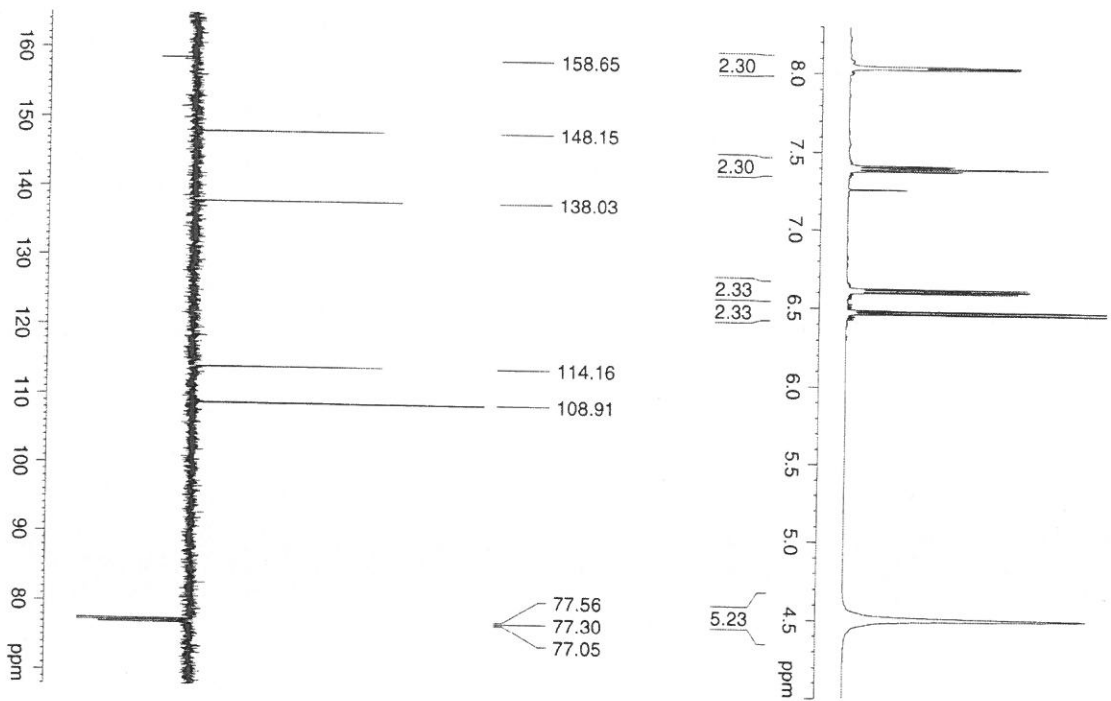
**Problem 5:**  $C_8H_8N_2O_2Cl$   
 IR: 1355, 1562  $cm^{-1}$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



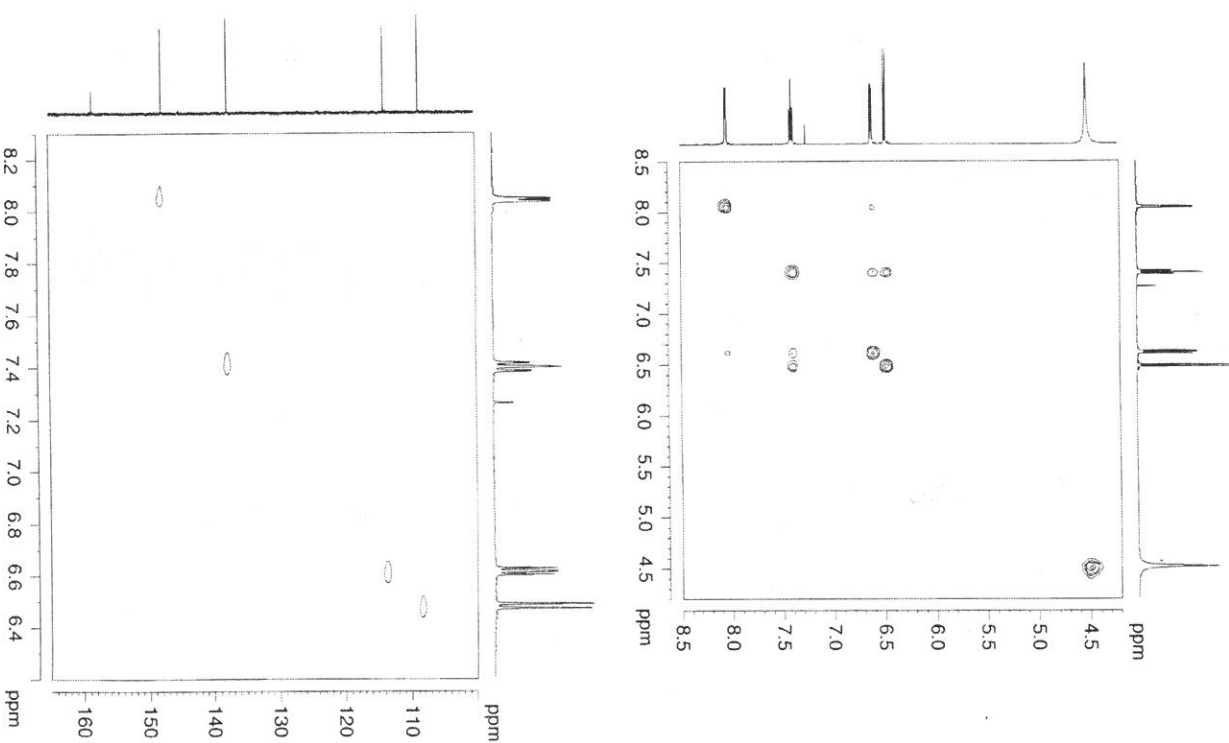
**Problem 5:**  $C_8H_8N_2O_2Cl$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $C/H$  correlation



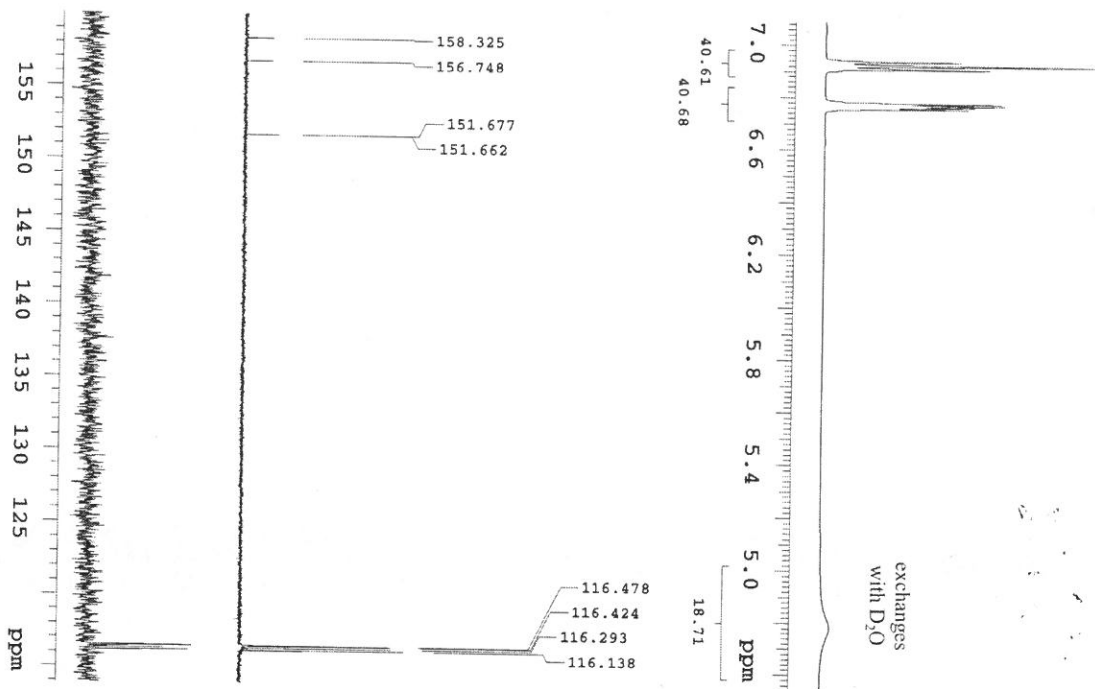
**Problem 6:**  $C_5H_6N_2$   
 IR: 3296, 3360  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



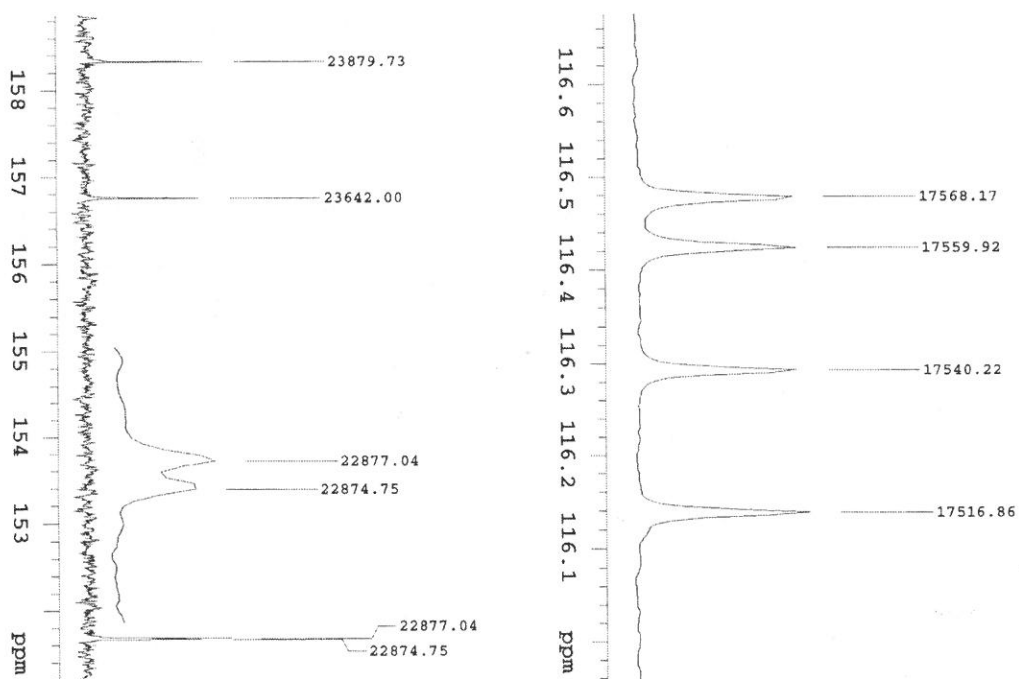
**Problem 6:**  $C_5H_6N_2$   
 500 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



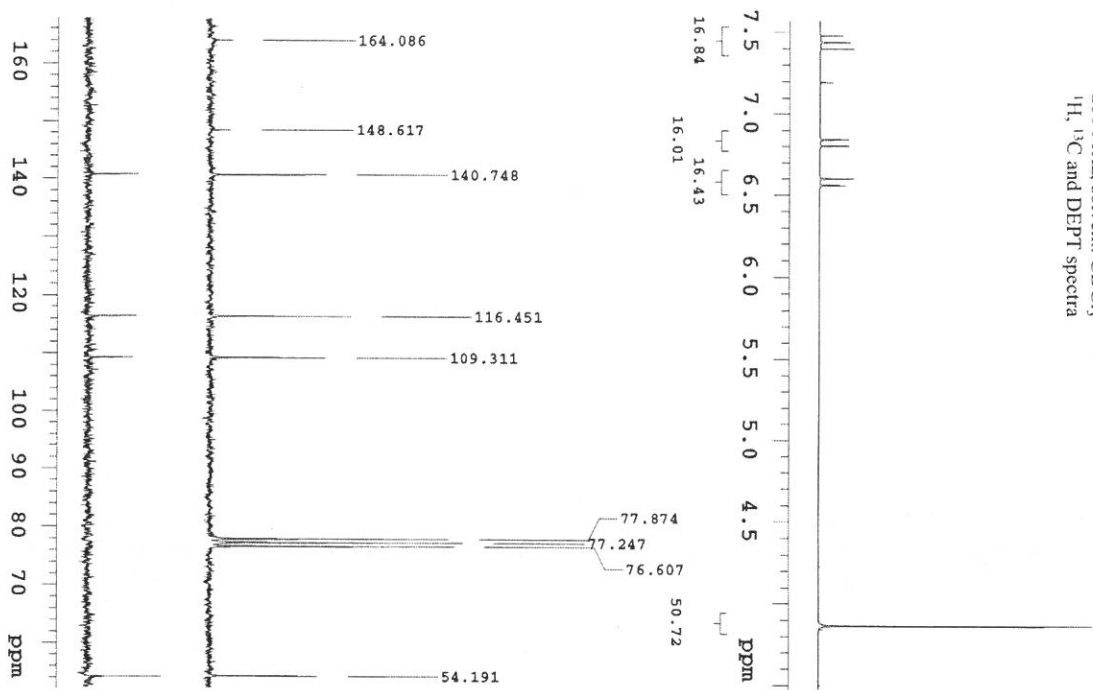
**Problem 7:  $C_6H_5OF$**   
 $^{19}F$  NMR:  $\delta = 125$  ppm  
 IR:  $3215\text{ cm}^{-1}$   
 600 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra



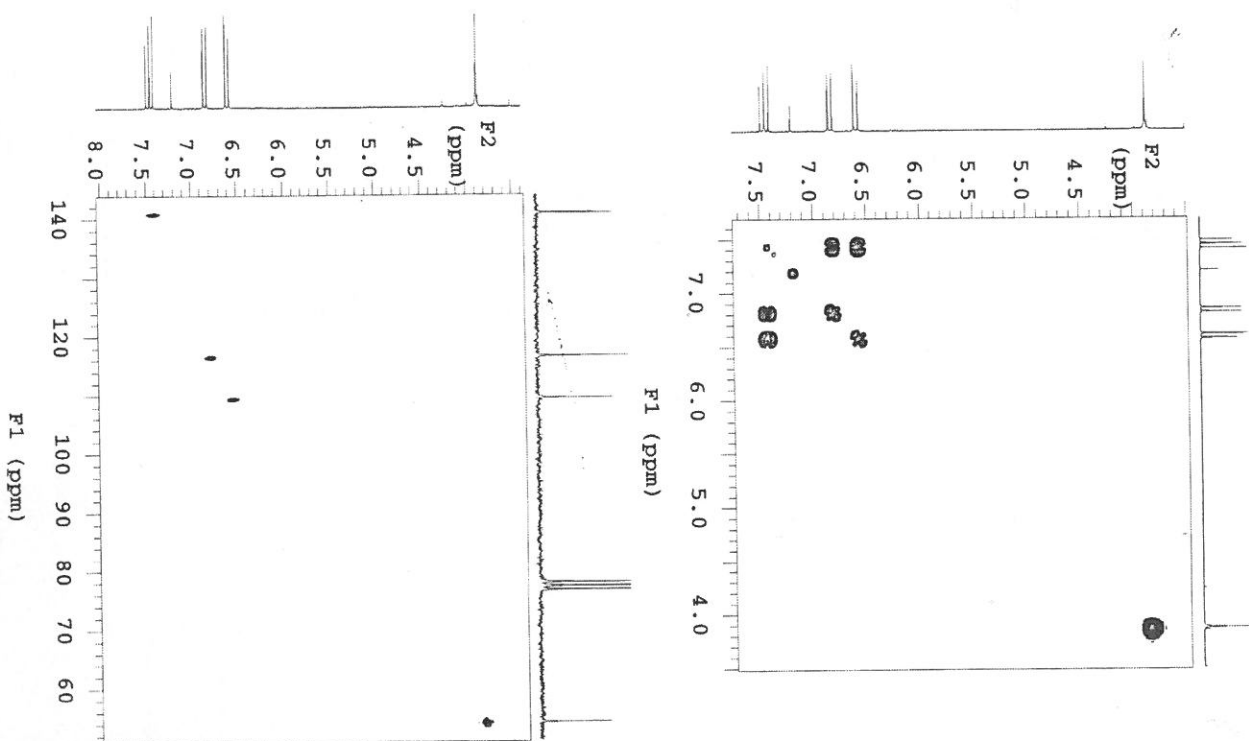
**Problem 7:  $C_6H_5OF$**   
 600 MHz, solvent:  $CDCl_3$   
 $^{13}C$  spectra (expansion)  
 scale in ppm, peak frequency in Hz



**Problem 8:  $C_6H_6NOCl$**   
 IR: no bands characteristic of functional groups  
 200 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra

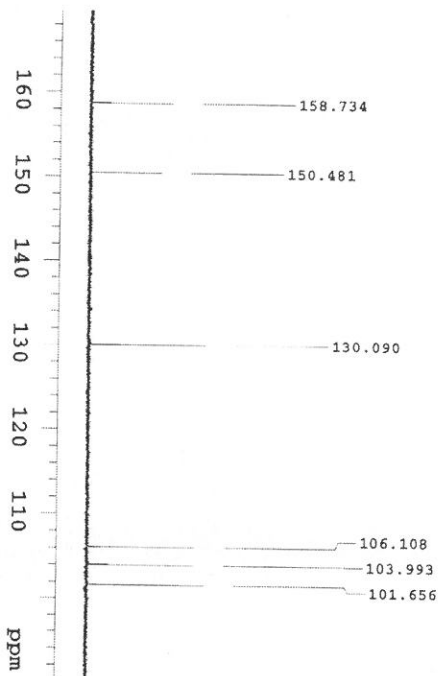
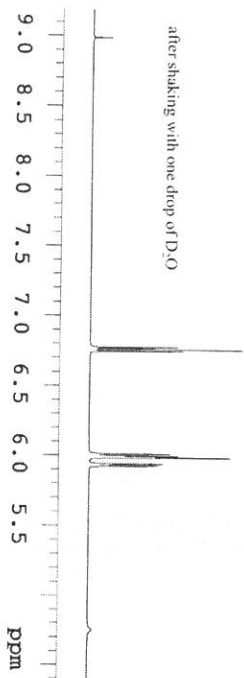


**Problem 8:  $C_6H_6NOCl$**   
 200 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation

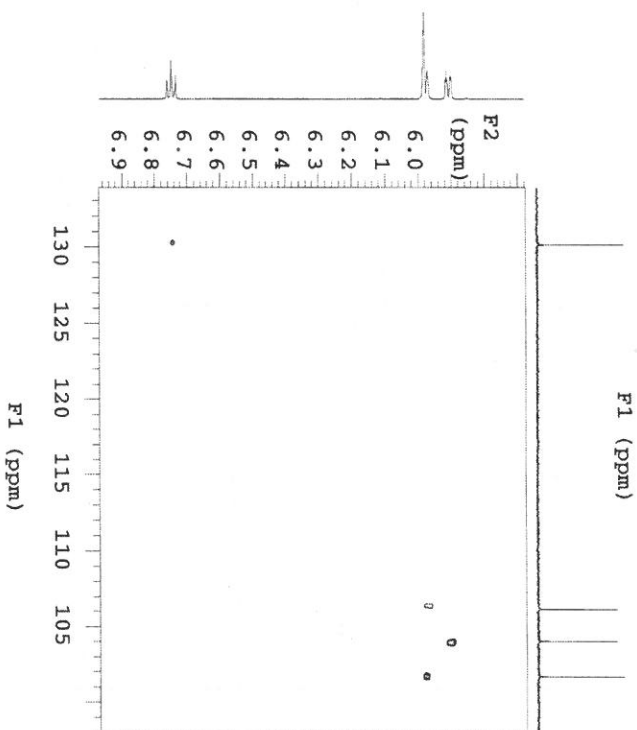
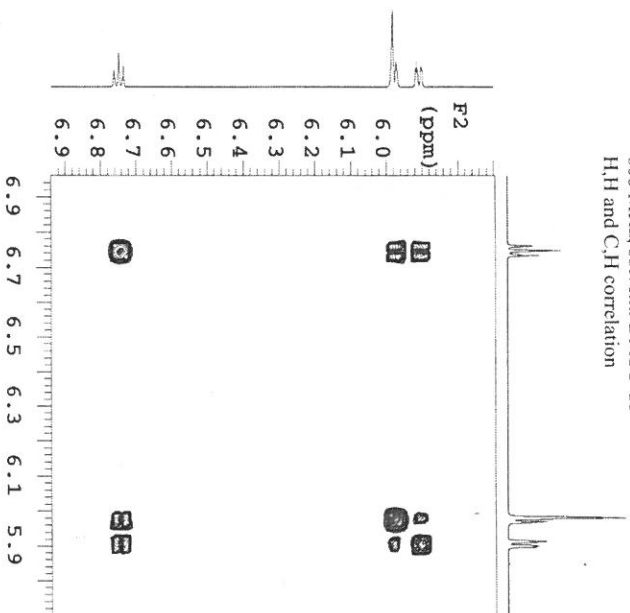




**Problem 9: C<sub>6</sub>H<sub>7</sub>NO**  
 IR: 3166, 3304, 3360 cm<sup>-1</sup>  
 600 MHz, solvent: DMSO-d<sub>6</sub>  
<sup>1</sup>H and <sup>13</sup>C spectra



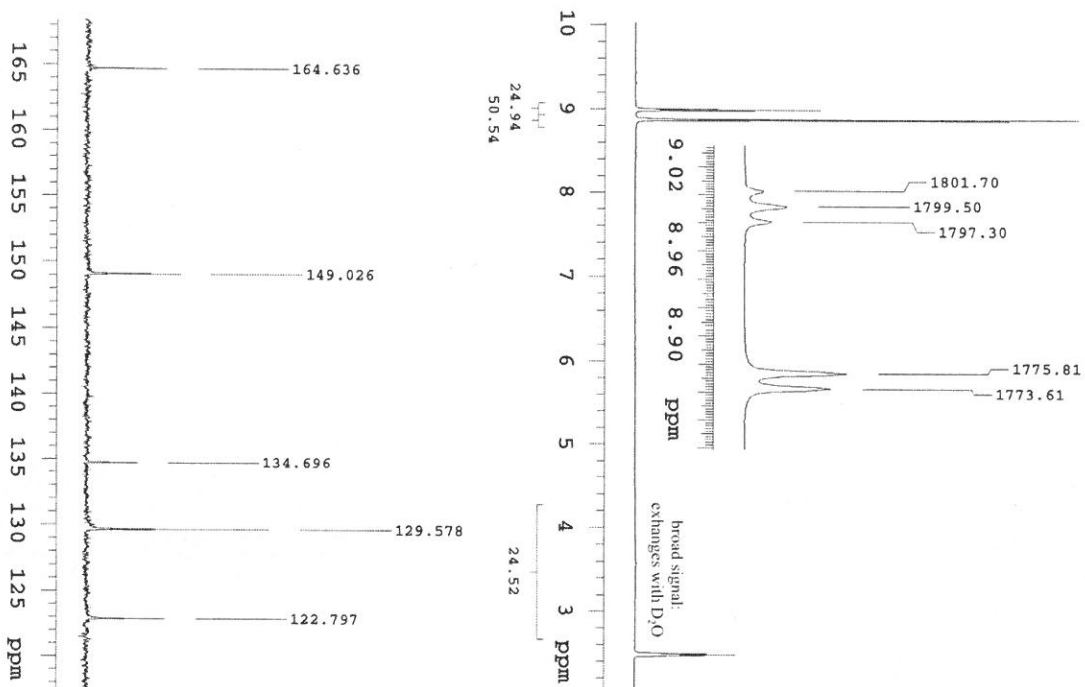
**Problem 9: C<sub>6</sub>H<sub>7</sub>NO**  
 600 MHz, solvent: DMSO-d<sub>6</sub>  
 H,H and C,H correlation



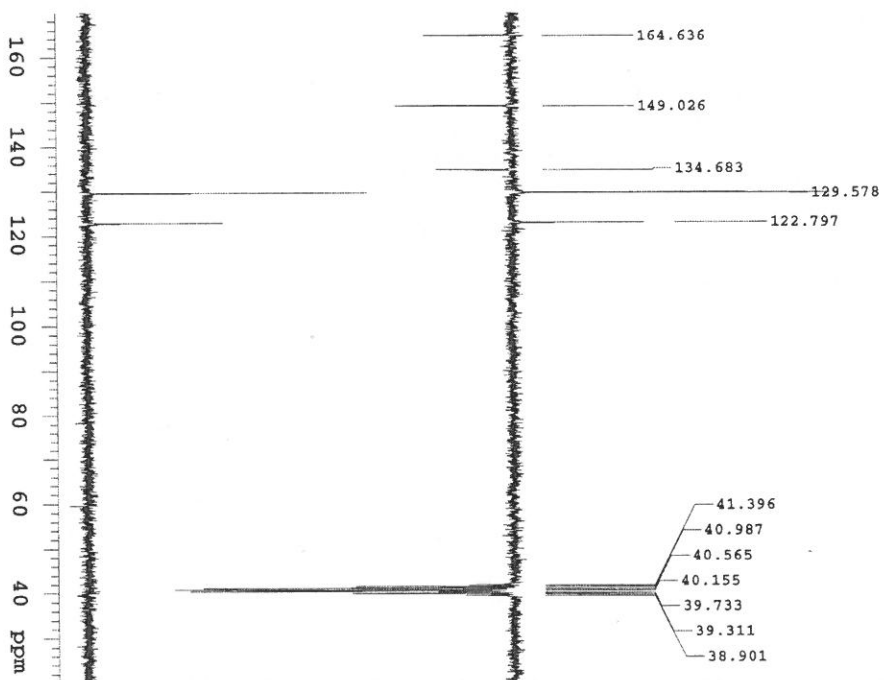
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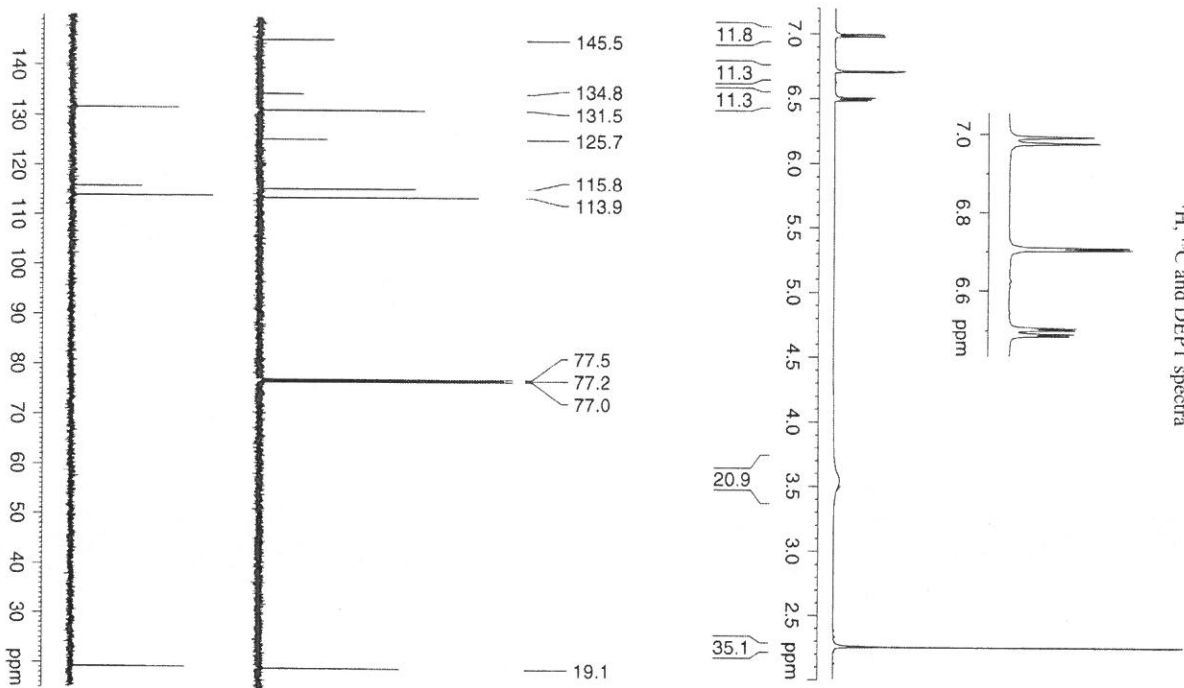
**Problem 10:**  $C_7H_4N_2O_6$   
 IR: 1348, 1545, 1703, 3093  $cm^{-1}$   
 200 MHz, solvent: DMSO- $d_6$   
 $^1H$  and  $^{13}C$  spectra  
 $^1H$  expansion: peak frequency in Hz



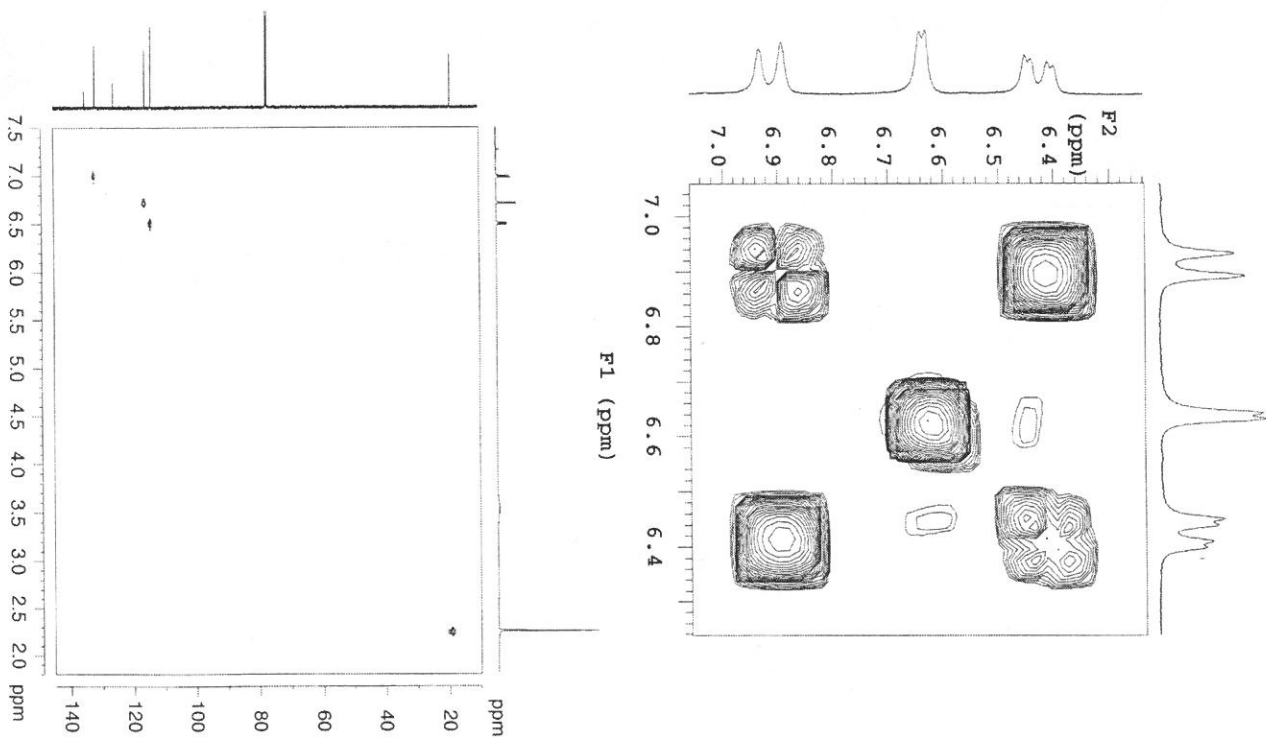
**Problem 10:**  $C_7H_4N_2O_6$   
 200 MHz, solvent: DMSO- $d_6$   
 APT and DEPT spectra



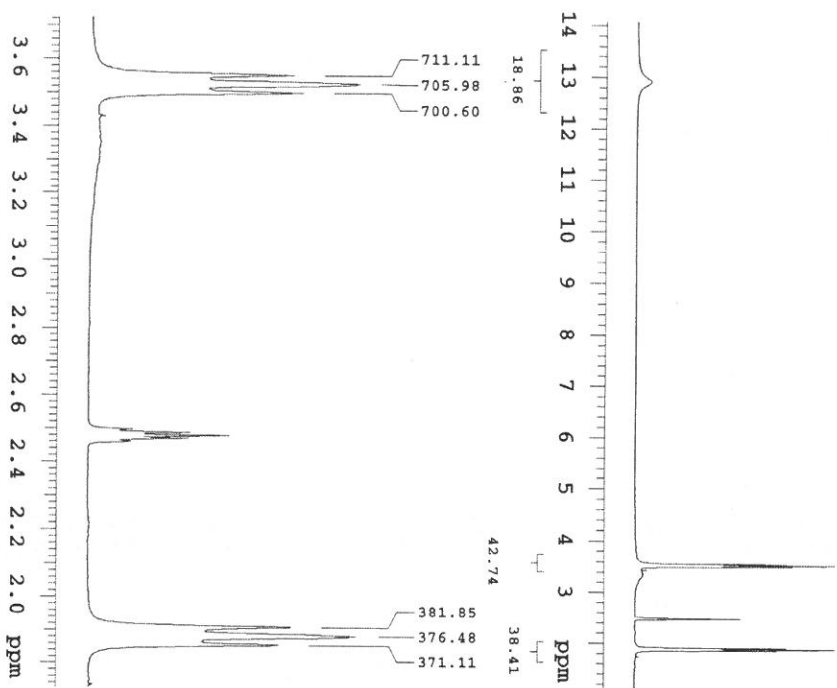
**Problem 11:  $C_7H_8NCl$**   
 IR: 3355, 3430  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra



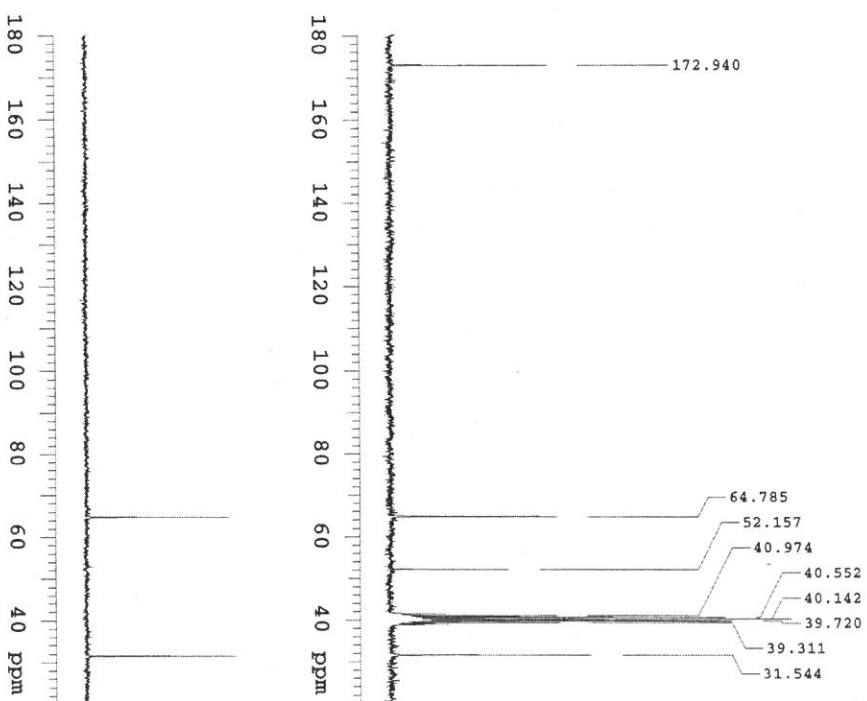
**Problem 11:  $C_7H_8NCl$**   
 solvent:  $CDCl_3$   
 HH correlation, 200 MHz  
 CH correlation, 500 MHz



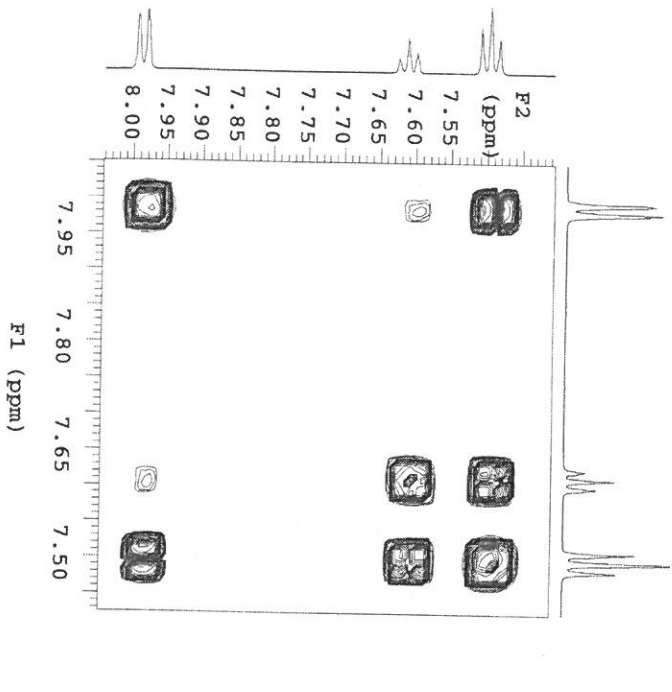
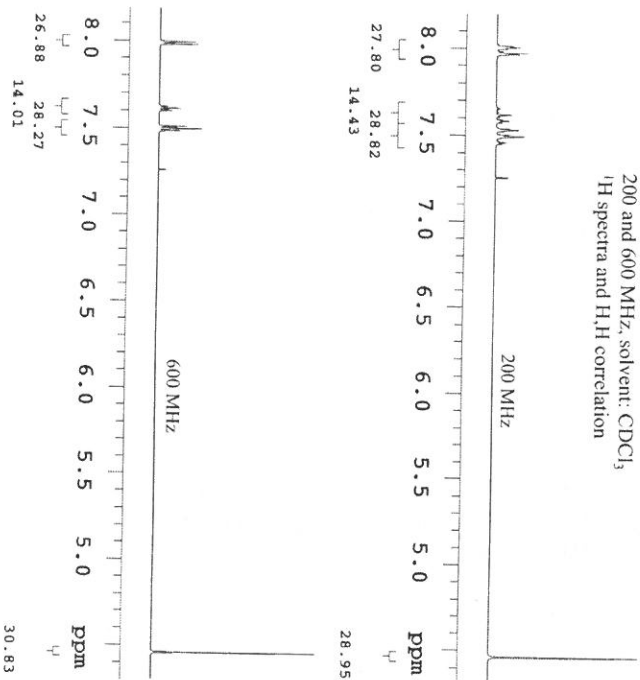
**Problem 12:**  $C_7H_{10}O_5$   
 IR: 1720 (strong), 2930-2970 (broad)  $cm^{-1}$   
 200 MHz, solvent: DMSO- $d_6$  (with small amount of water:  $\delta = 3.4$  ppm broad)  
 $^1H$  spectra with expansion



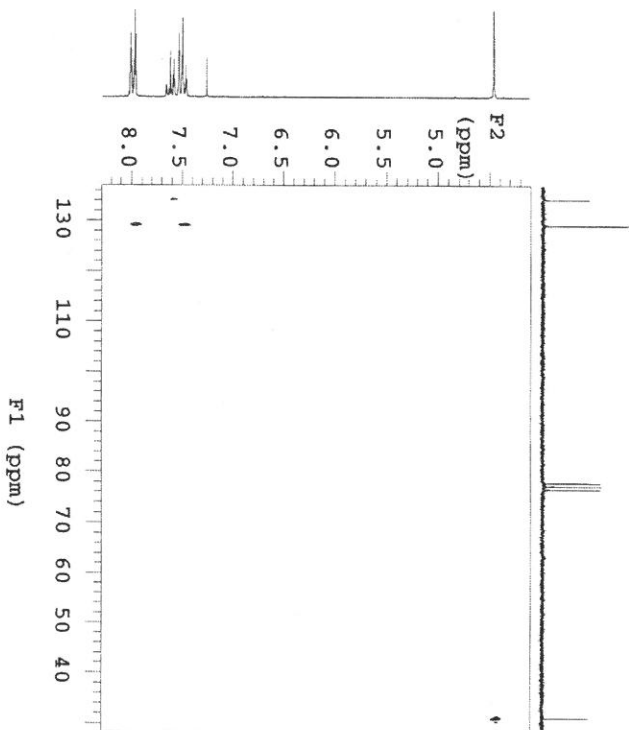
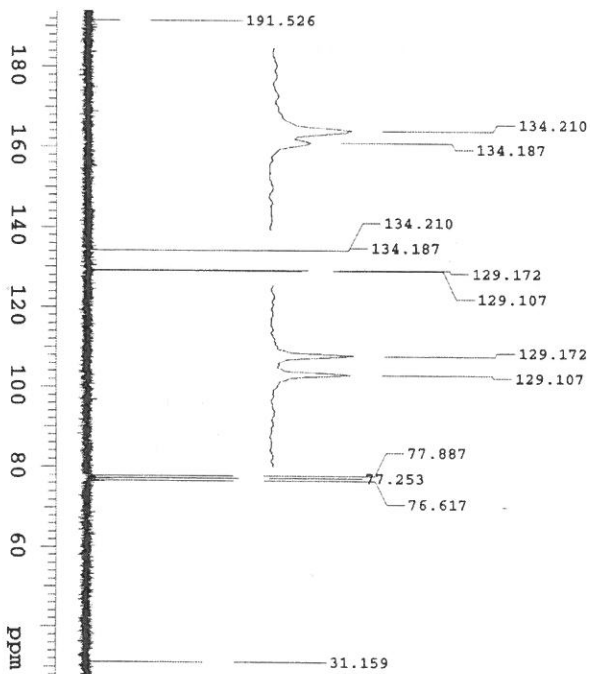
**Problem 12:**  $C_7H_{10}O_5$   
 200 MHz, solvent: DMSO- $d_6$   
 $^{13}C$  and DEPT spectra



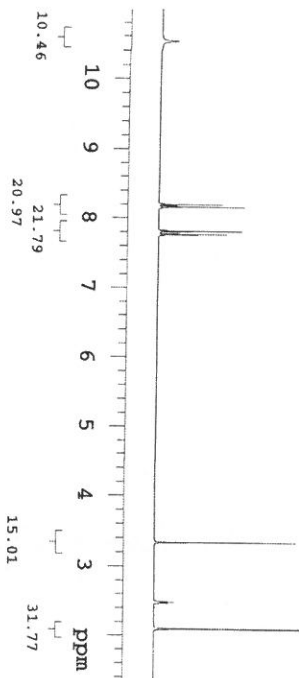
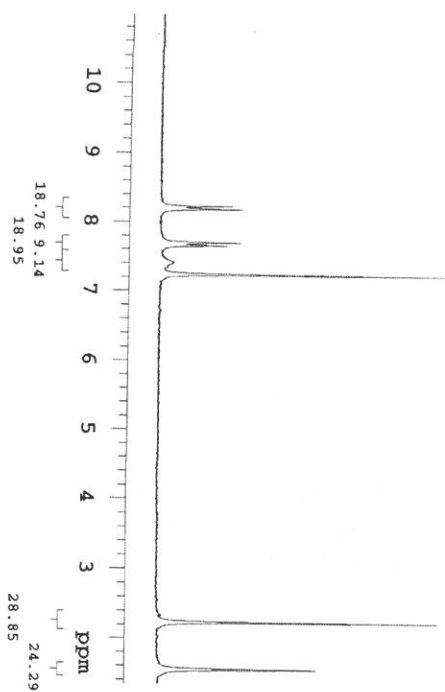
**Problem 13: C<sub>8</sub>H<sub>7</sub>OBr**  
 IR: 1641 cm<sup>-1</sup>  
 200 and 600 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H spectra and H,H correlation



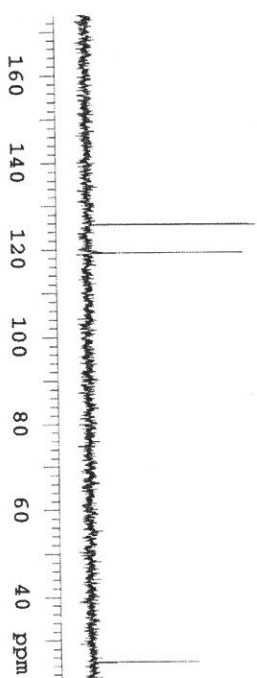
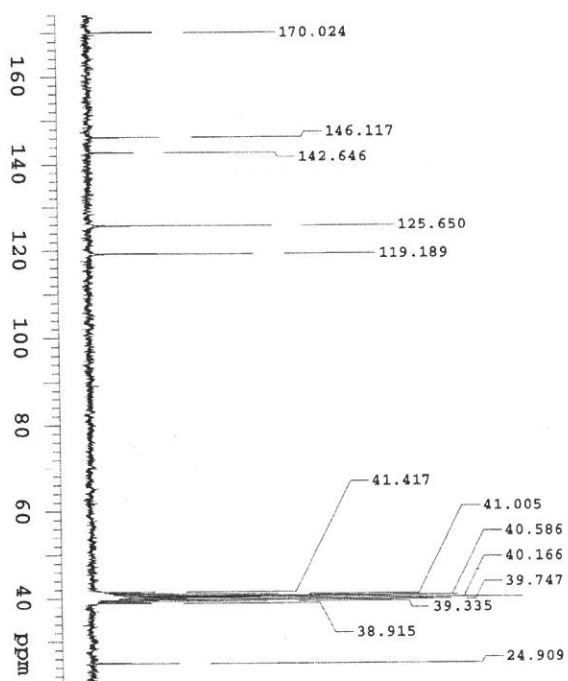
**Problem 13: C<sub>8</sub>H<sub>7</sub>OBr**  
 200 MHz, solvent: CDCl<sub>3</sub>  
<sup>13</sup>C spectra and C,H correlation



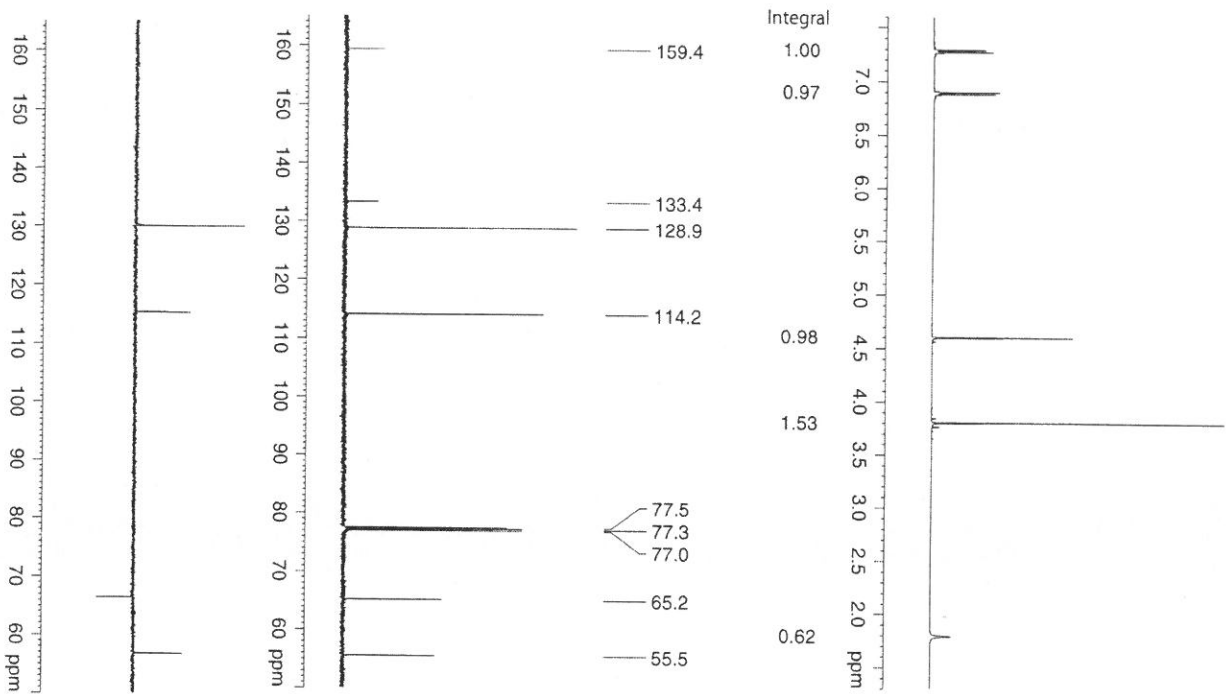
**Problem 14:**  $C_8H_{10}N_2O$   
 IR: 1681, 3093, 3159, 3279  $cm^{-1}$   
 200 MHz, solvent:  $CDCl_3$  (top) and DMSO- $d_6$  (below)  
 $^1H$  spectra



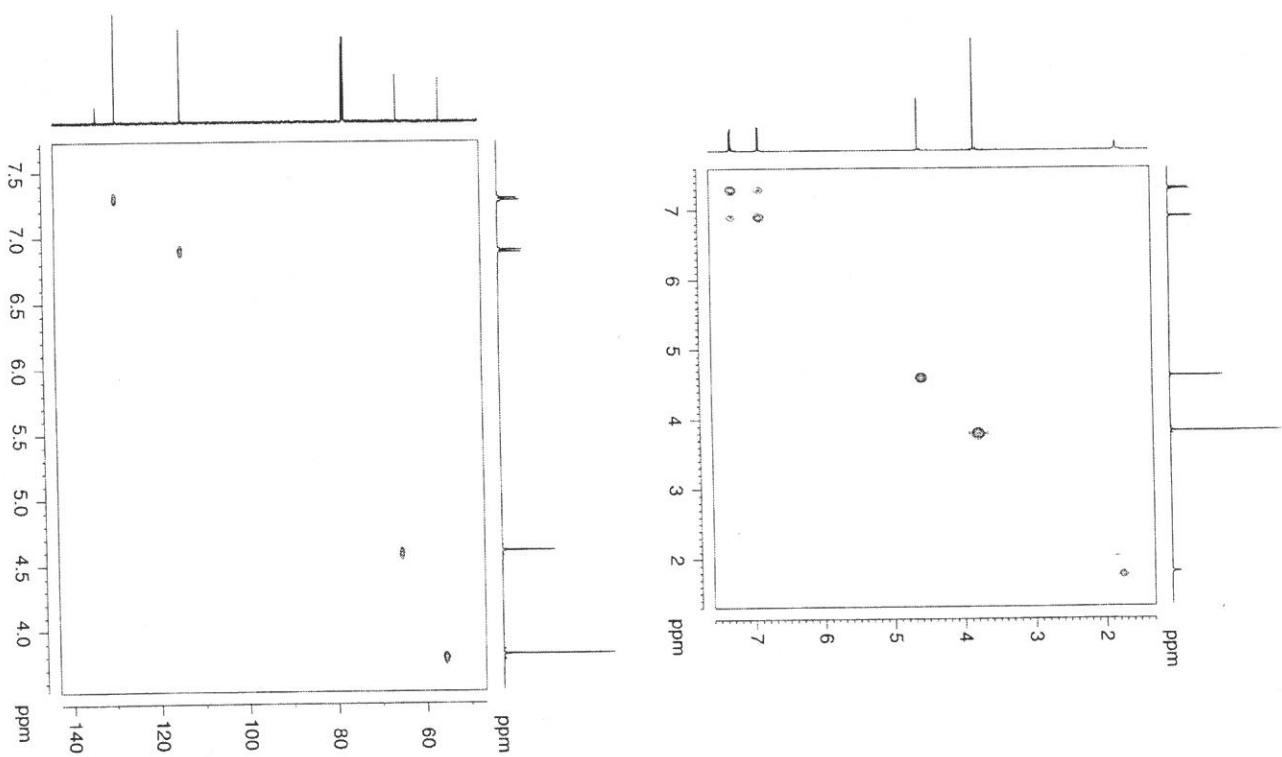
**Problem 14:**  $C_8H_{10}N_2O$   
 200 MHz, solvent: DMSO- $d_6$   
 $^{13}C$  and DEPT spectra



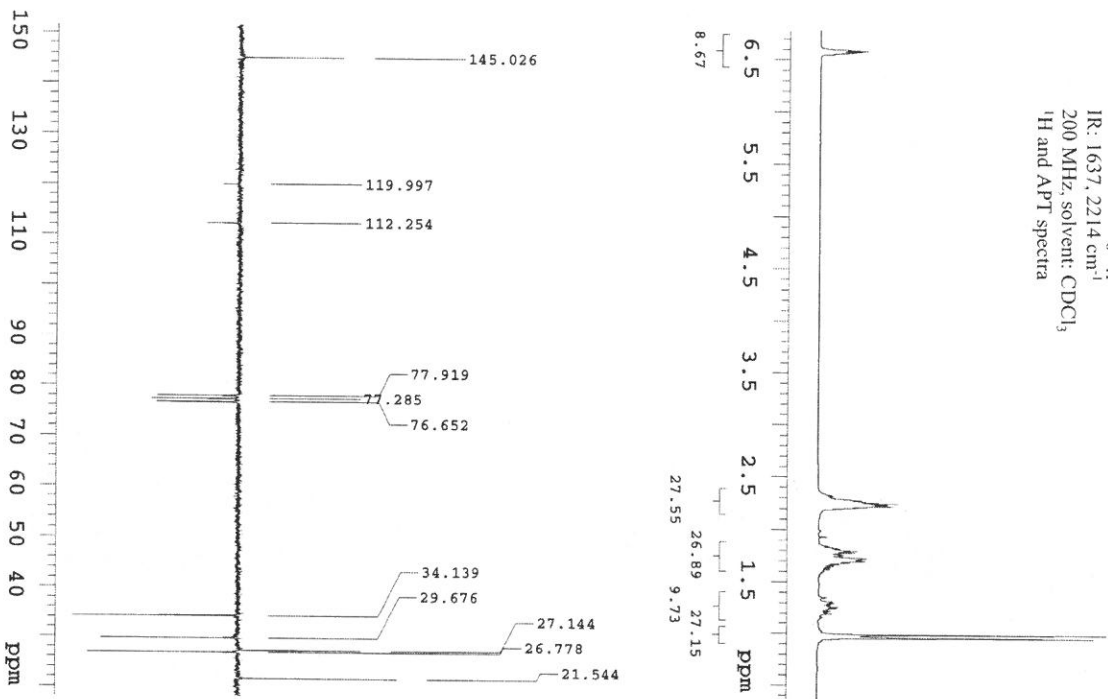
**Problem 15:**  $C_8H_{10}O_2$   
 IR: 3365 (very broad)  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra



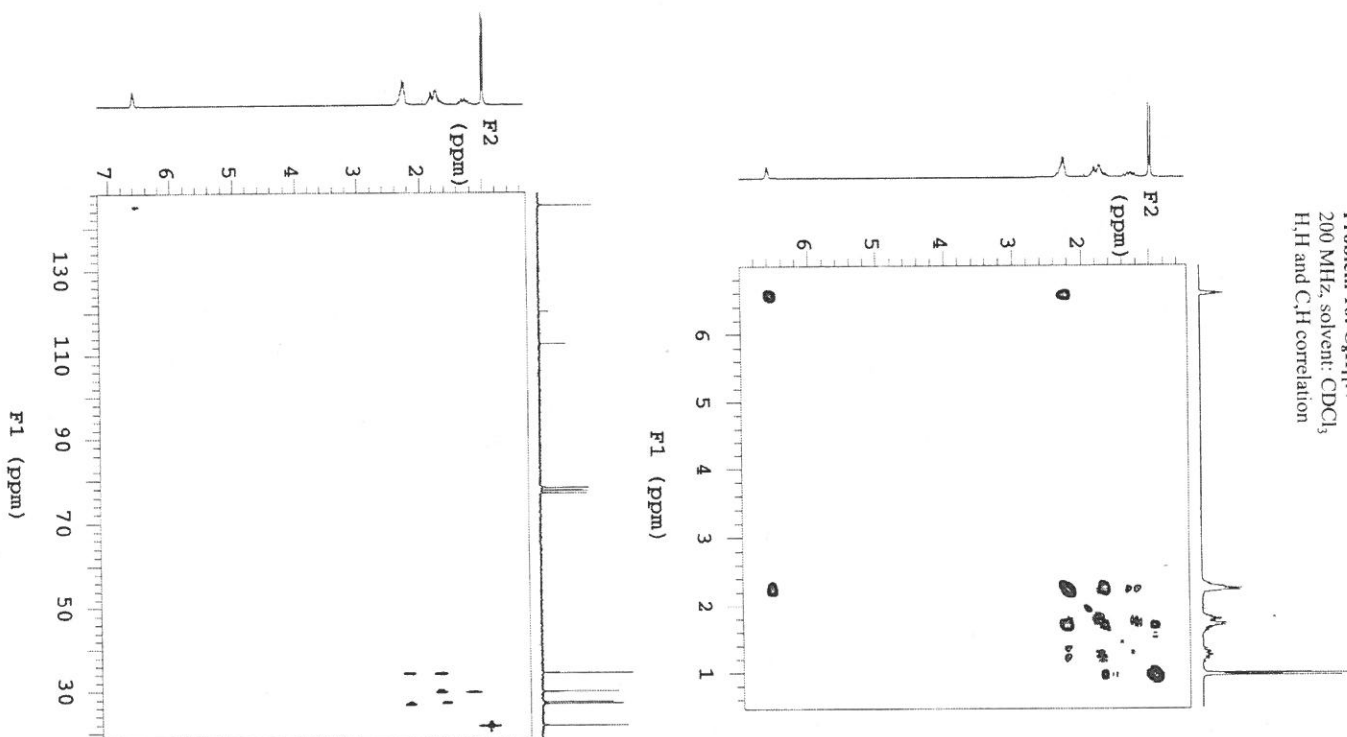
**Problem 16:**  $C_8H_{10}O_2$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $C/H$  correlation



**Problem 16:**  $C_8H_{11}N$   
 IR: 1637, 2214  $cm^{-1}$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra

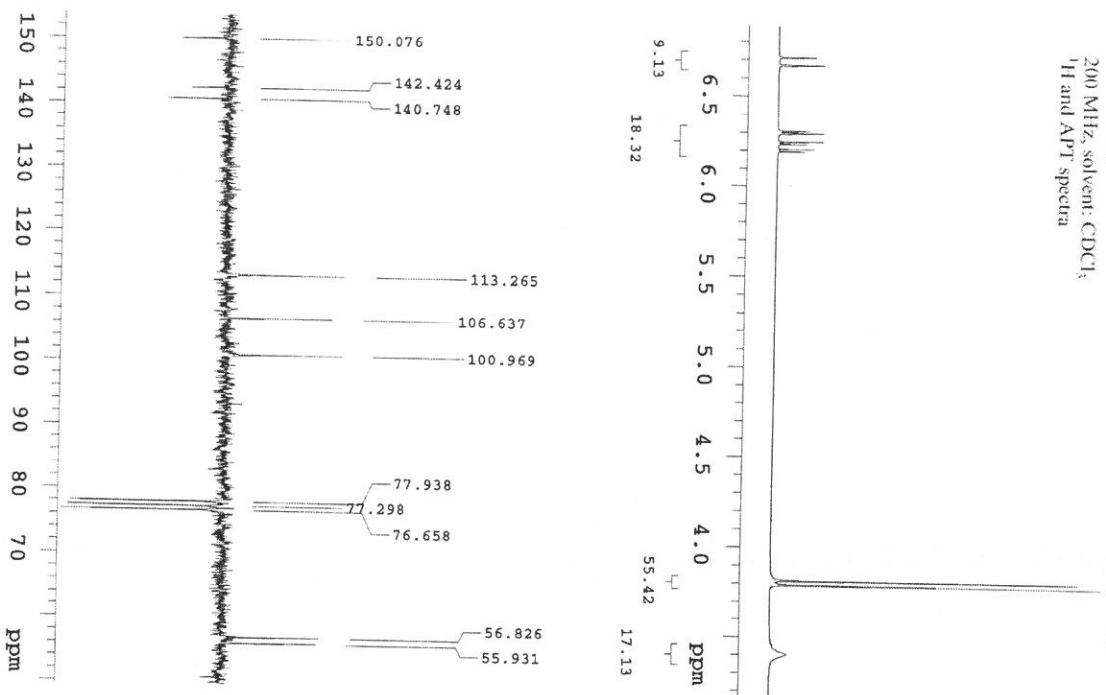


**Problem 16:**  $C_8H_{11}N$   
 200 MHz, solvent:  $CDCl_3$   
 $H_1H$  and  $C_1H$  correlation

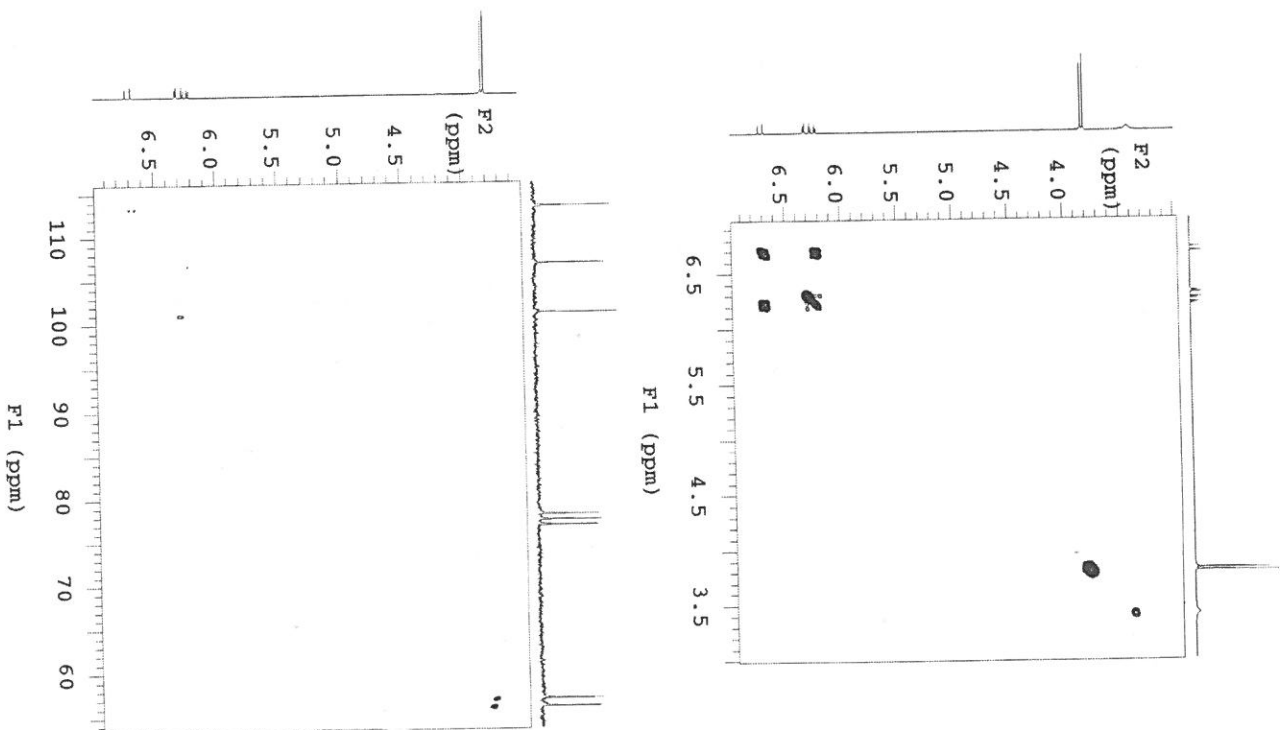




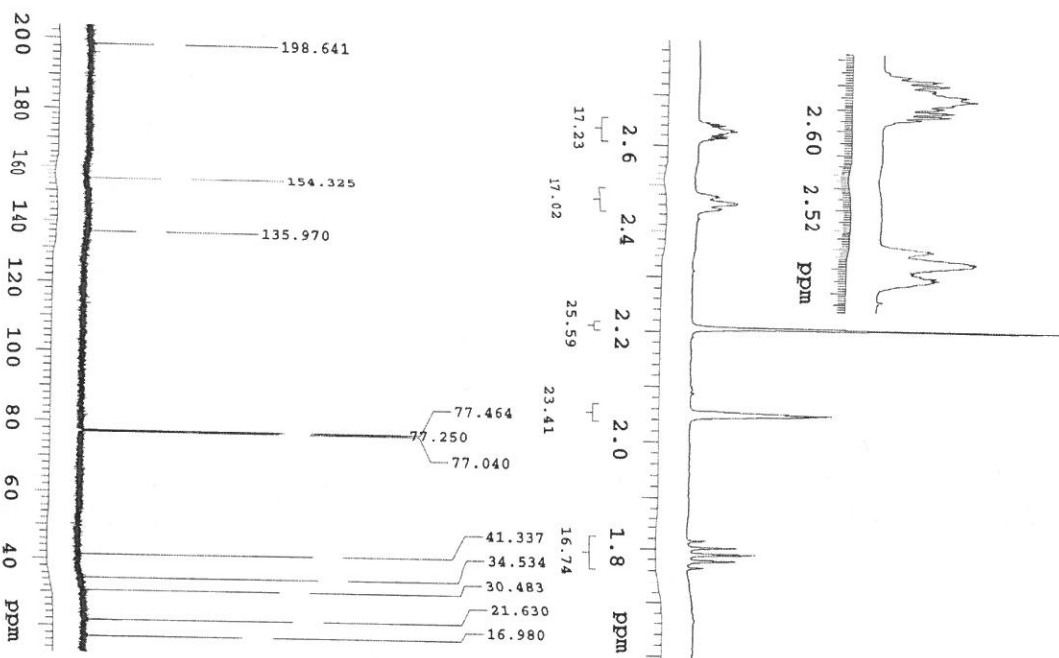
Problem 17:  $C_{11}H_{11}NO_2$   
 IR:  $3379\text{ cm}^{-1}$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



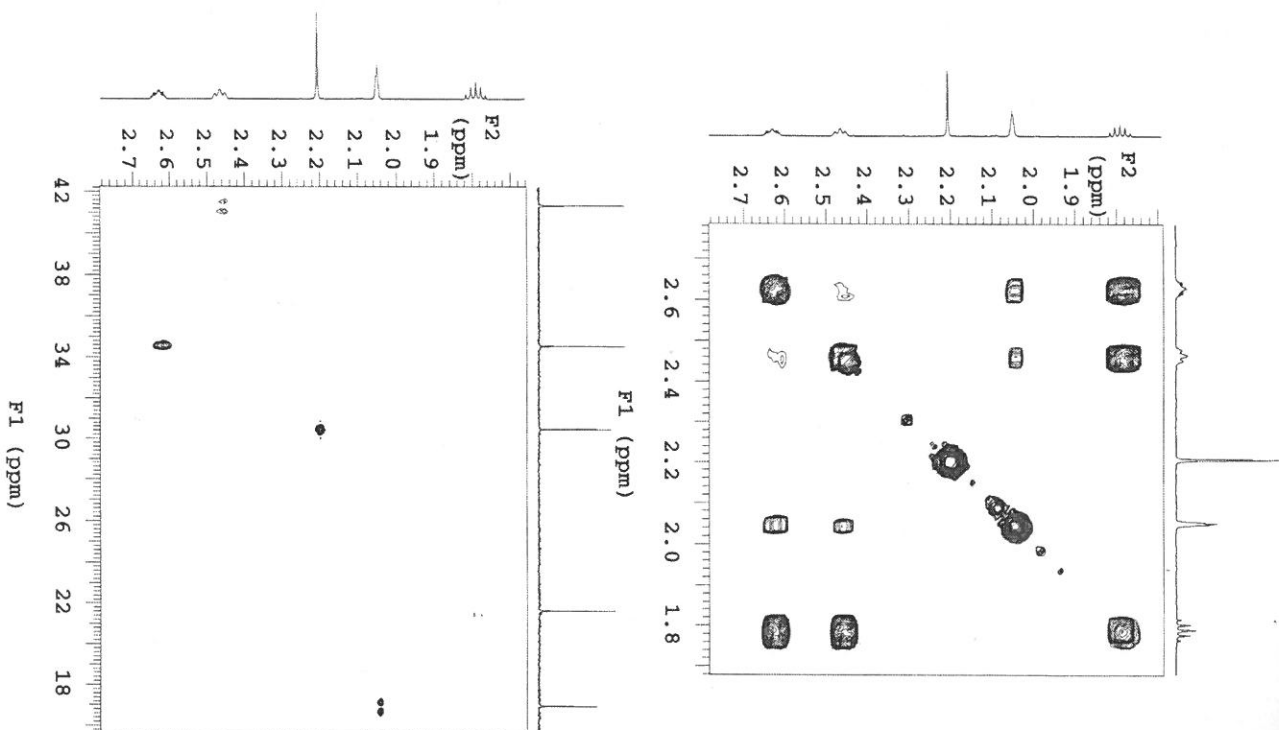
Problem 17:  $C_{11}H_{11}NO_2$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  correlation



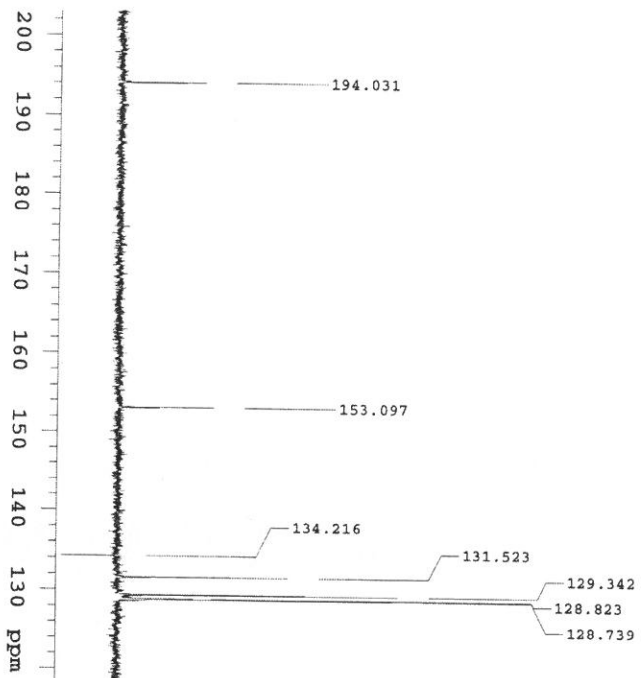
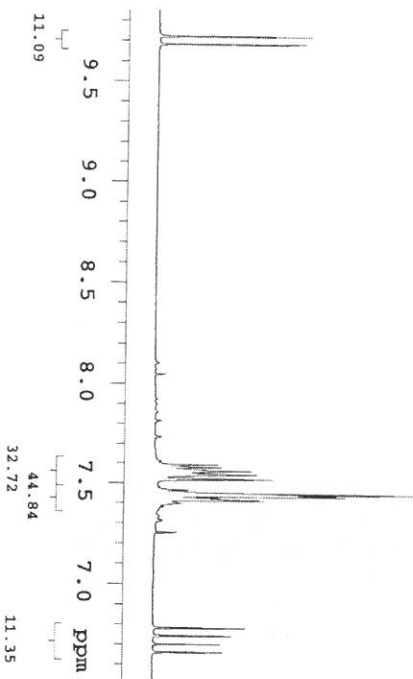
Problem 18:  $C_8H_{12}O$   
 IR:  $1680\text{ cm}^{-1}$   
 600 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  spectra



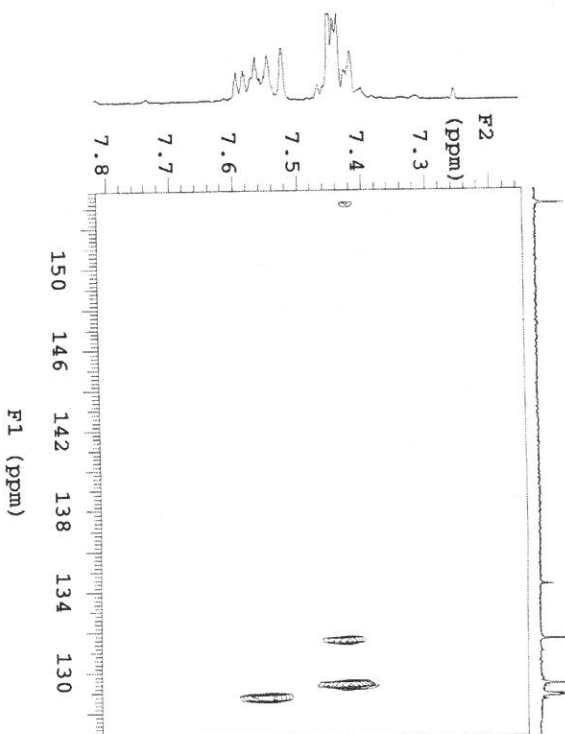
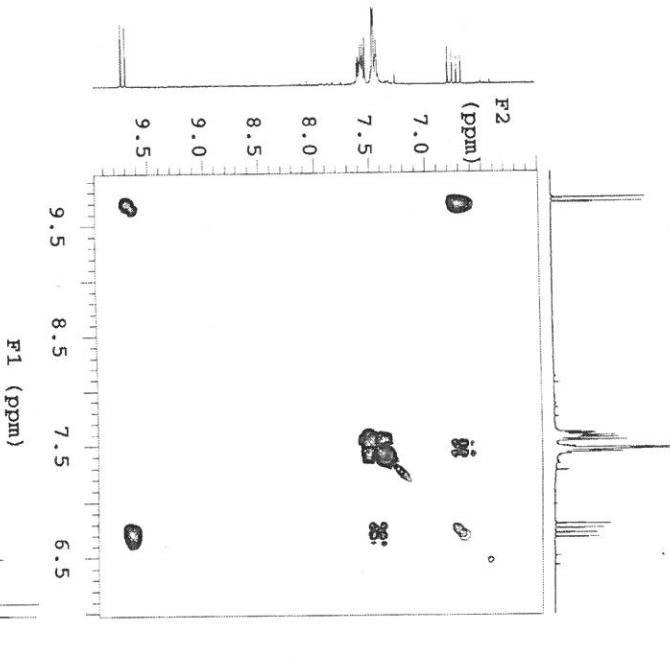
Problem 18:  $C_8H_{12}O$   
 600 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  correlation



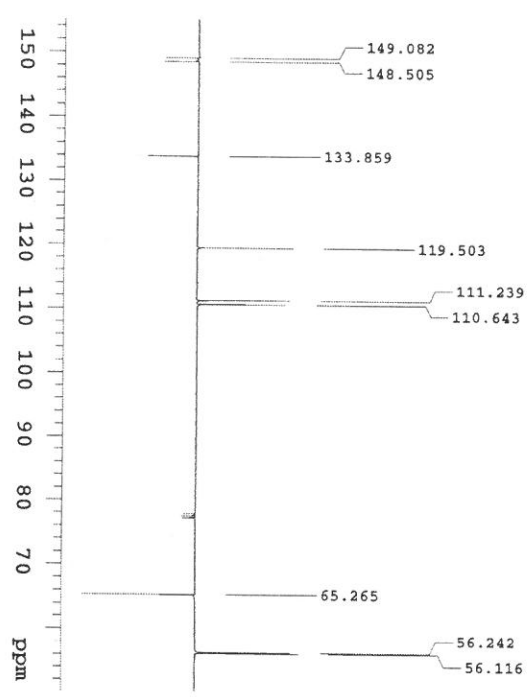
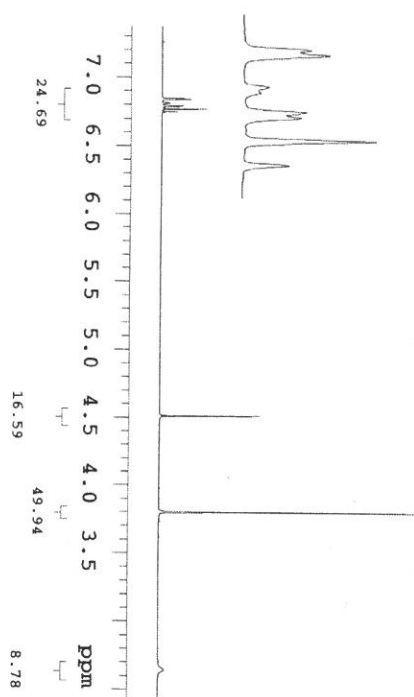
**Problem 19: C<sub>6</sub>H<sub>6</sub>O**  
 IR: 1625, 1681 (strong) cm<sup>-1</sup>  
 200 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H and APT spectra



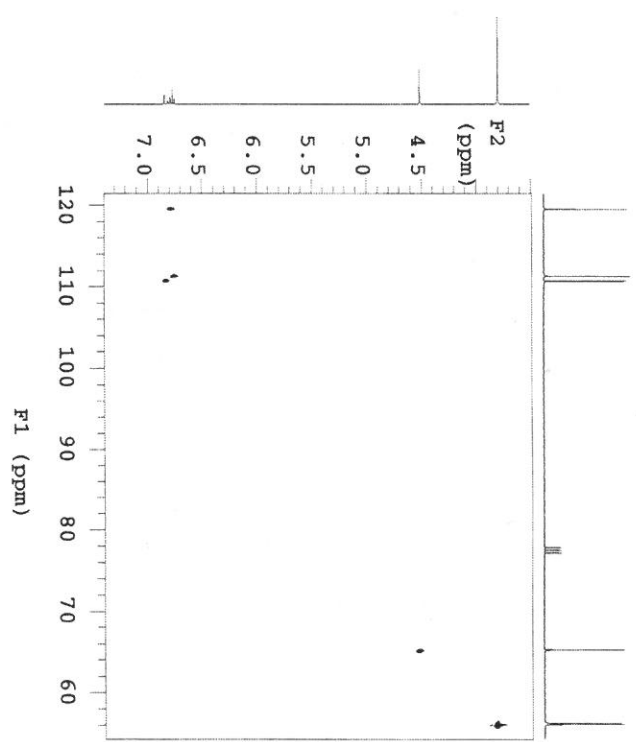
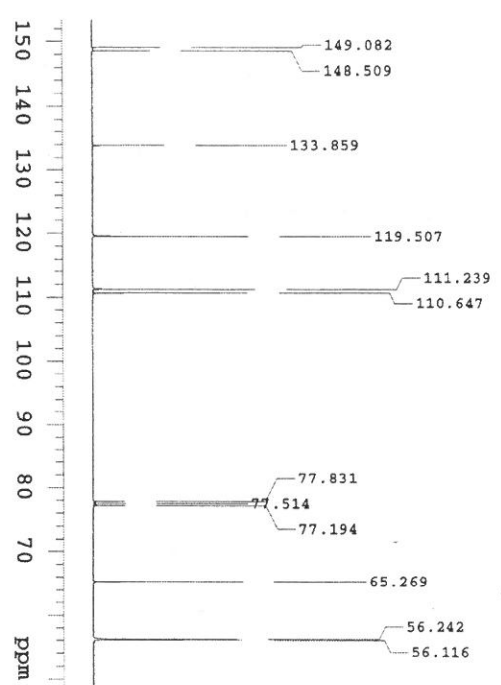
**Problem 19: C<sub>6</sub>H<sub>6</sub>O**  
 200 MHz, solvent: CDCl<sub>3</sub>  
 H,H correlation and a part of C,H correlation



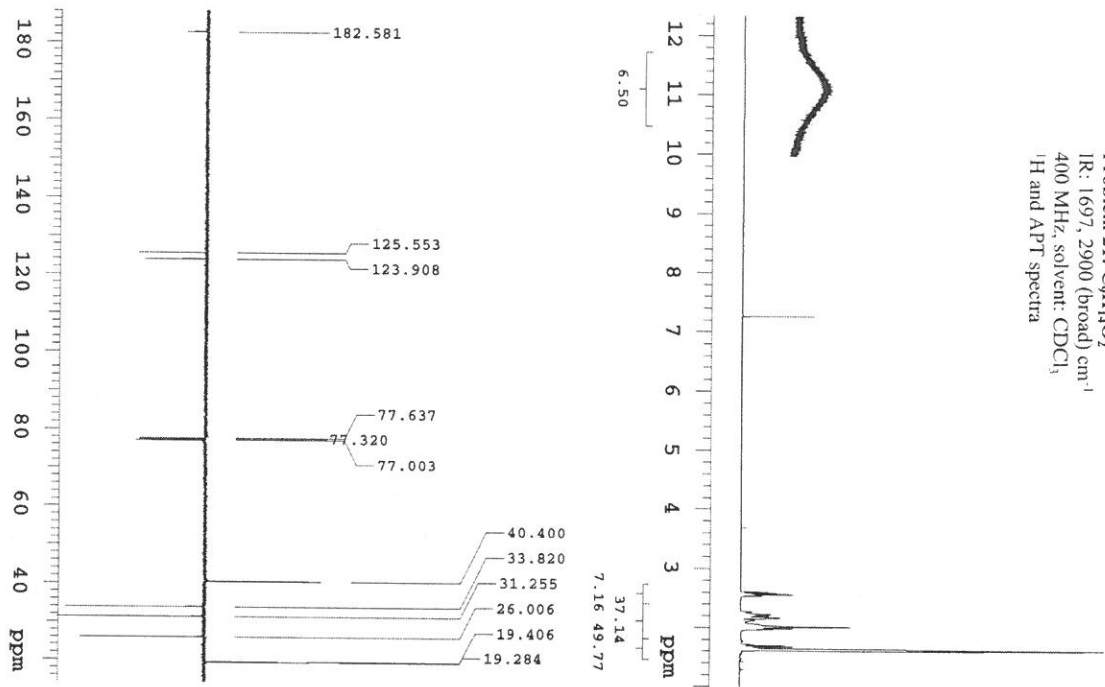
**Problem 20:**  $C_9H_{12}O_3$   
 IR:  $3480\text{ cm}^{-1}$   
 400 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



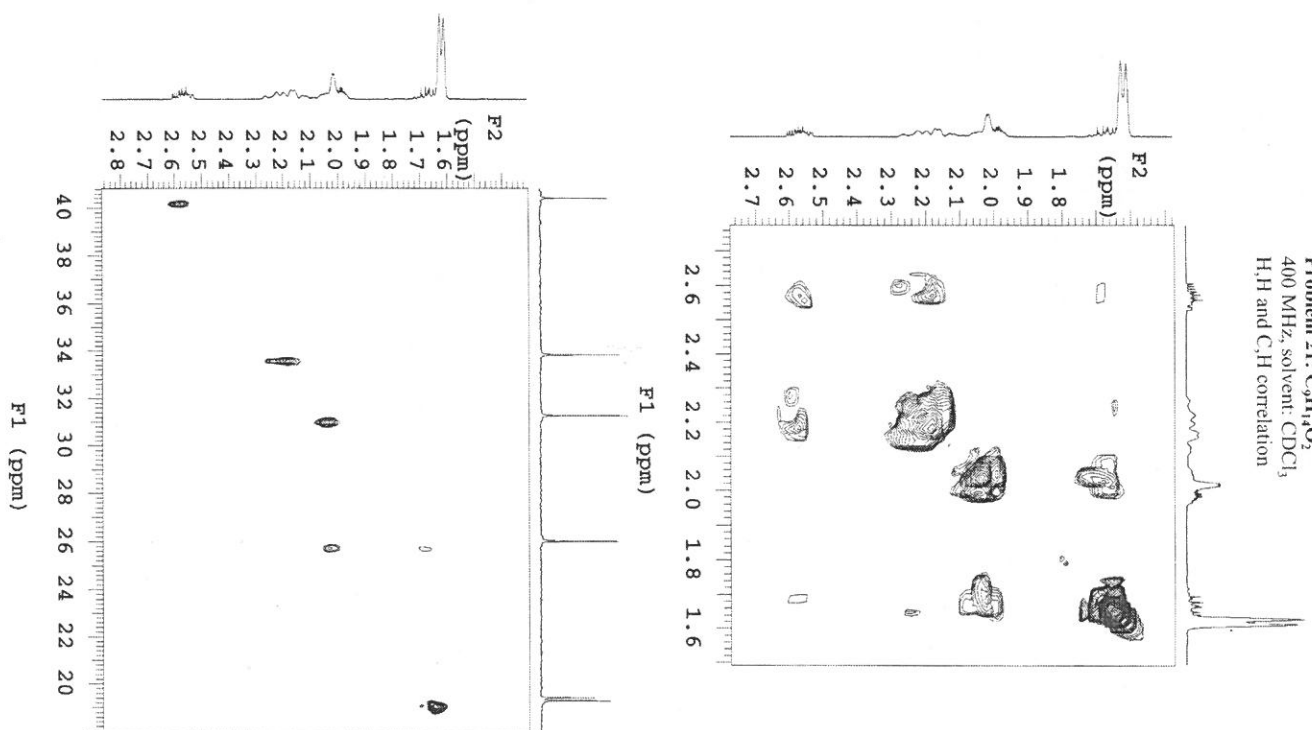
**Problem 20:**  $C_9H_{12}O_3$   
 400 MHz, solvent:  $CDCl_3$   
 $^{13}C$  spectra and  $C,H$  correlation



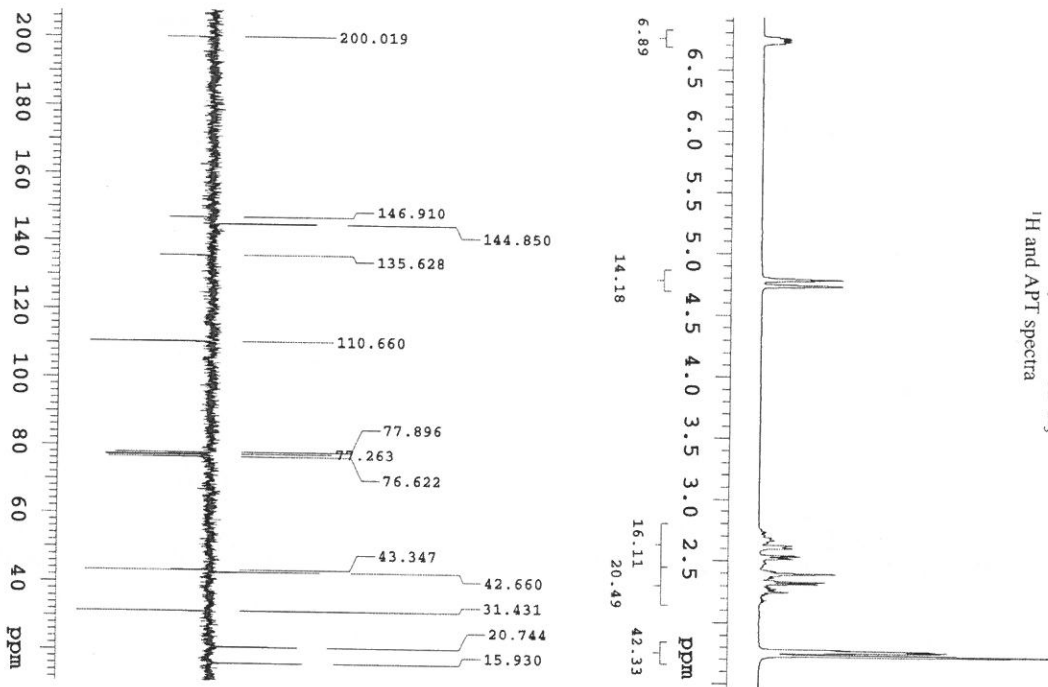
**Problem 21:**  $C_9H_{14}O_2$   
 IR: 1697, 2900 (broad)  $cm^{-1}$   
 400 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



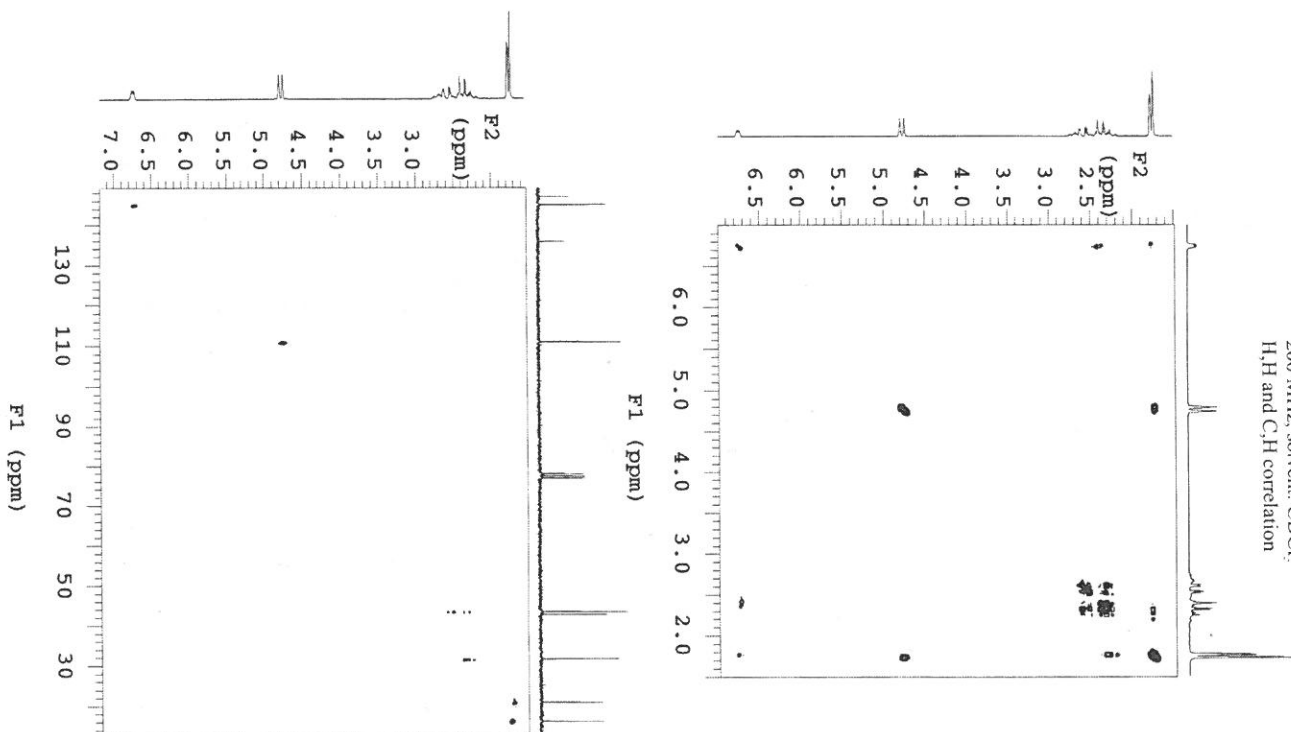
**Problem 21:**  $C_9H_{14}O_2$   
 400 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



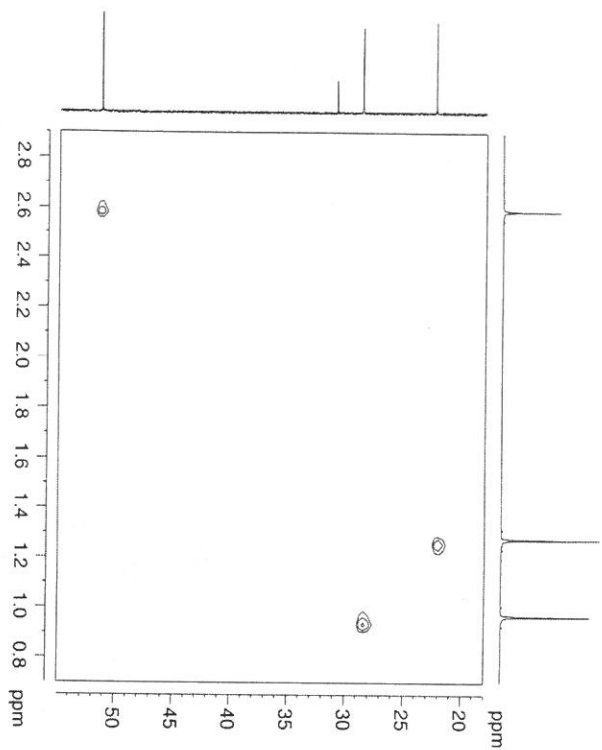
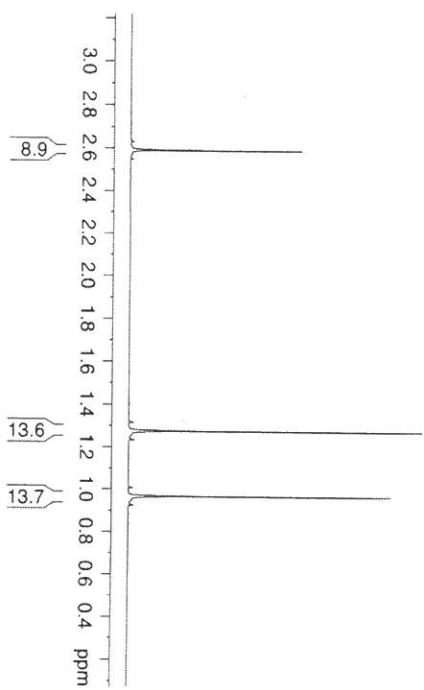
**Problem 22:**  $C_{10}H_{14}O$   
 IR: 1631, 1732  $cm^{-1}$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



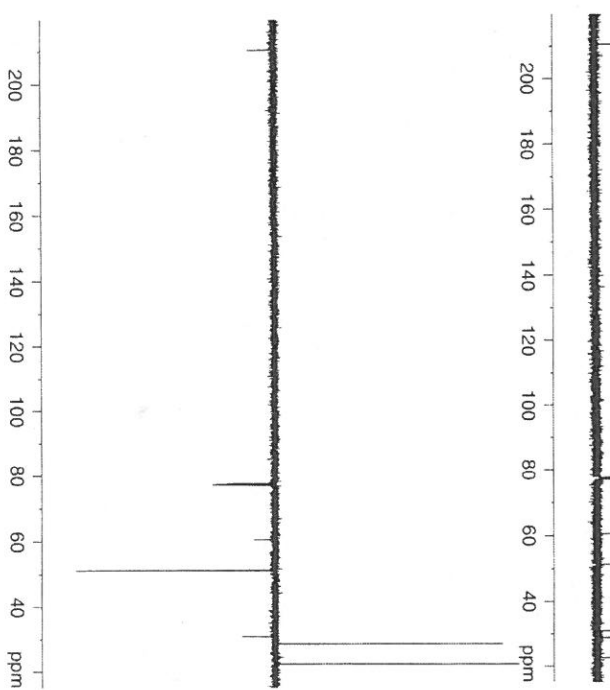
**Problem 22:**  $C_{10}H_{14}O$   
 200 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



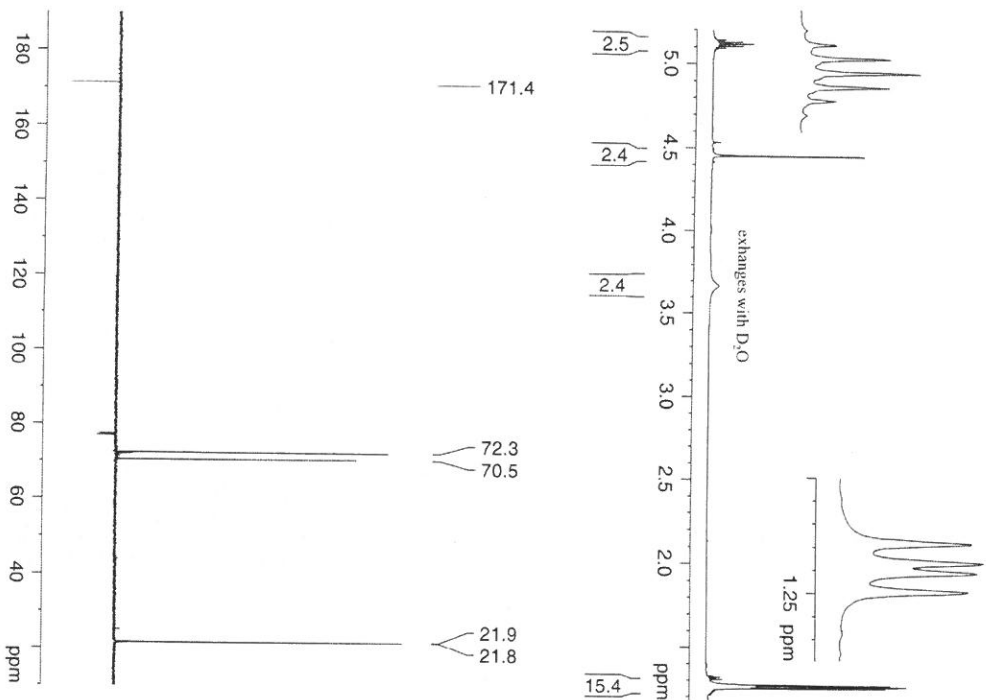
**Problem 23:**  $C_{10}H_{16}O_2$   
 IR:  $1725\text{ cm}^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  spectrum and  $C, H$  correlation



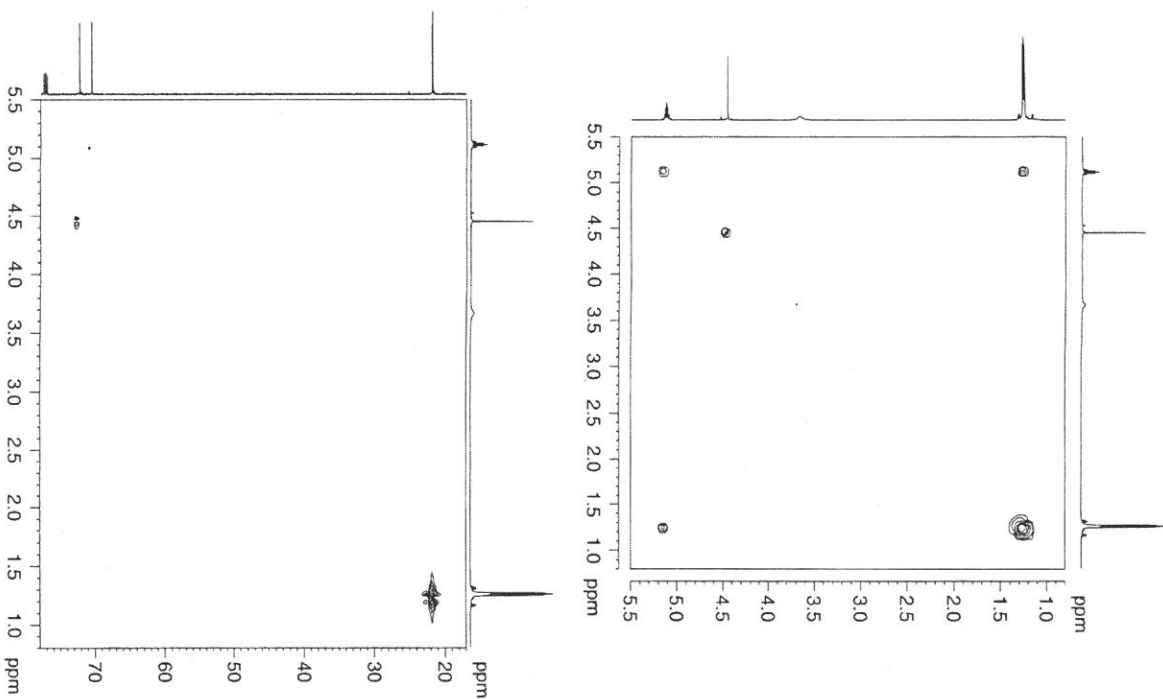
**Problem 23:**  $C_{10}H_{16}O_2$   
 500 MHz, solvent:  $CDCl_3$   
 $^{13}C$  and APT spectra



**Problem 24:**  $C_{10}H_{18}O_6$   
 IR: 1730 (strong), 3340 (broad)  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra

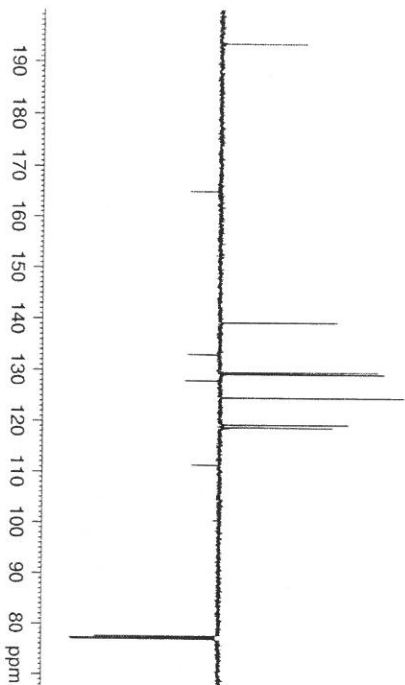
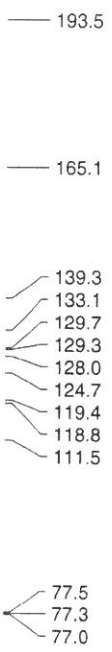
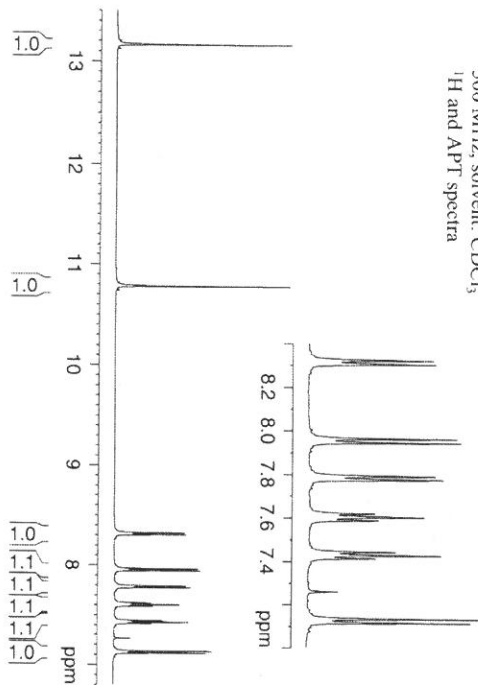


**Problem 24:**  $C_{10}H_{18}O_6$   
 500 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation

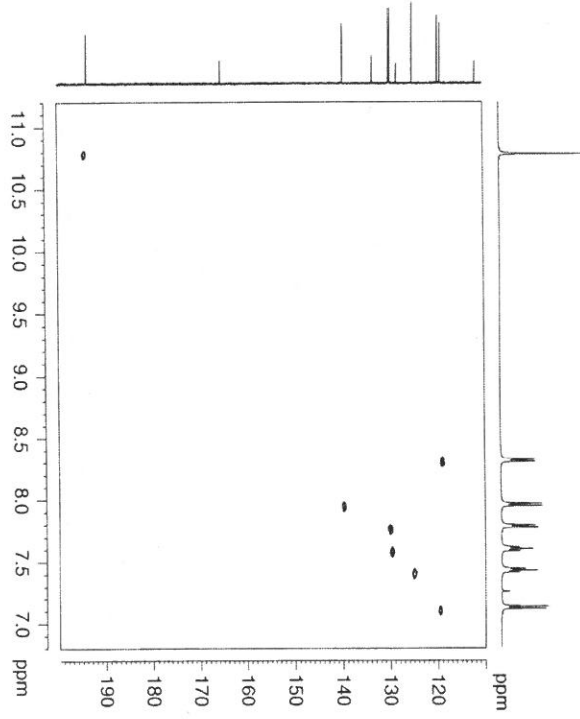
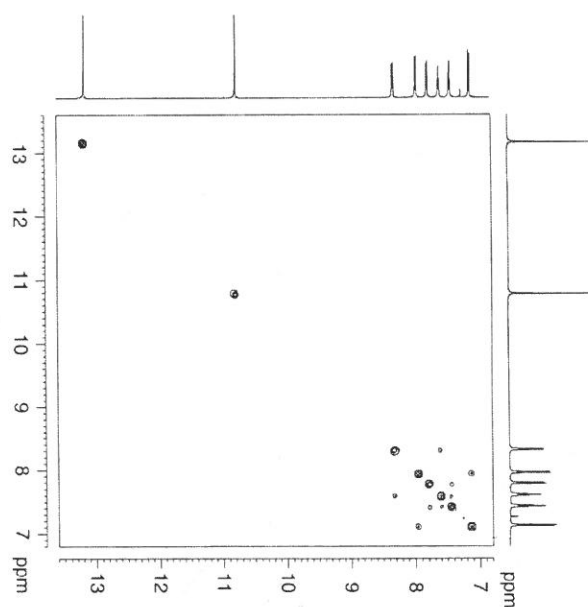




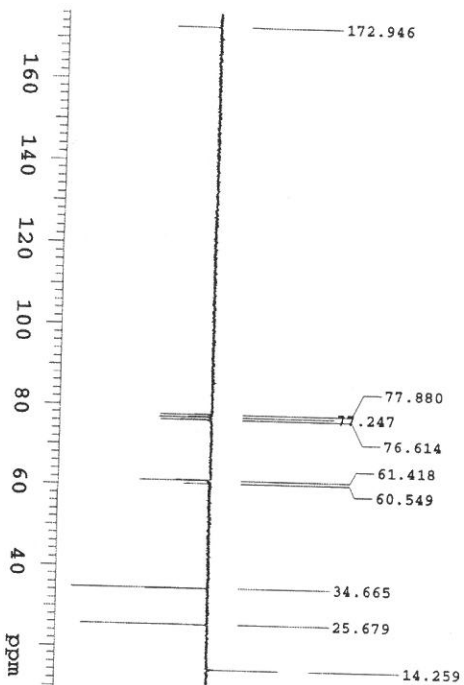
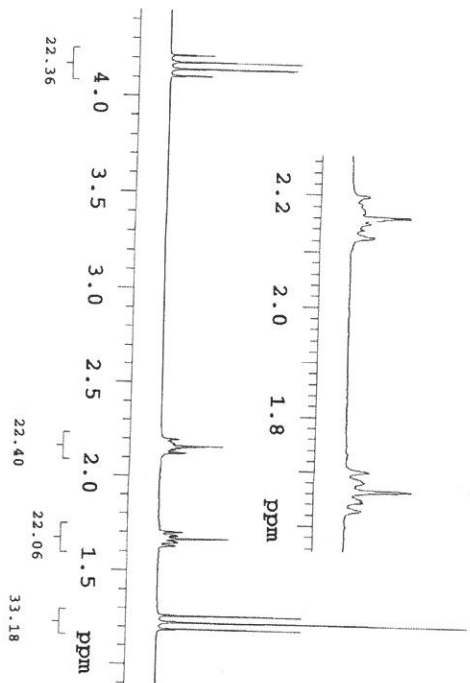
**Problem 25:**  $C_{11}H_8O_2$   
 IR: 1670 (strong), 3360  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  spectra



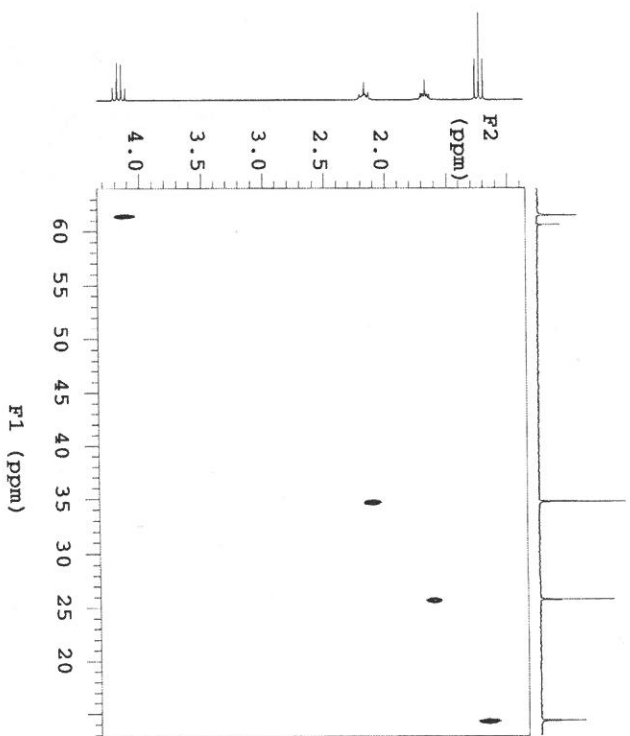
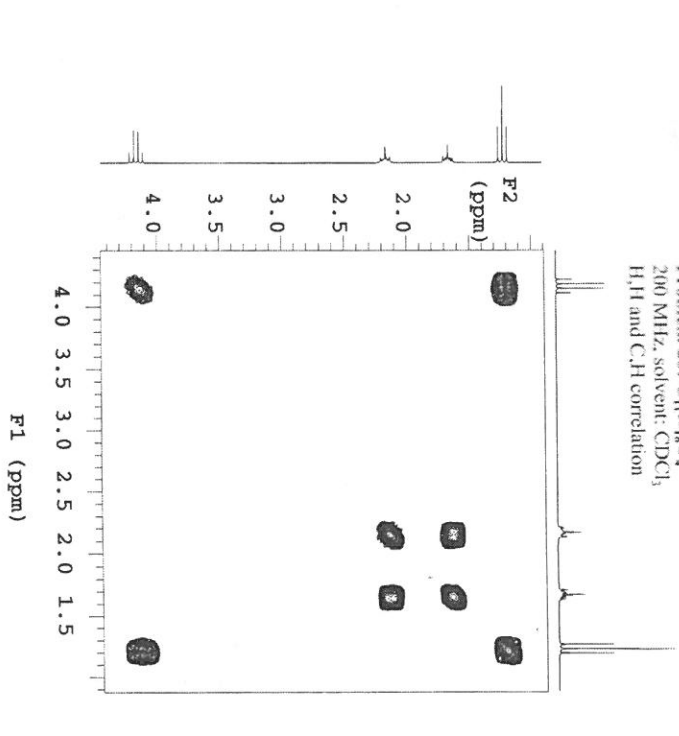
**Problem 25:**  $C_{11}H_8O_2$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  correlation



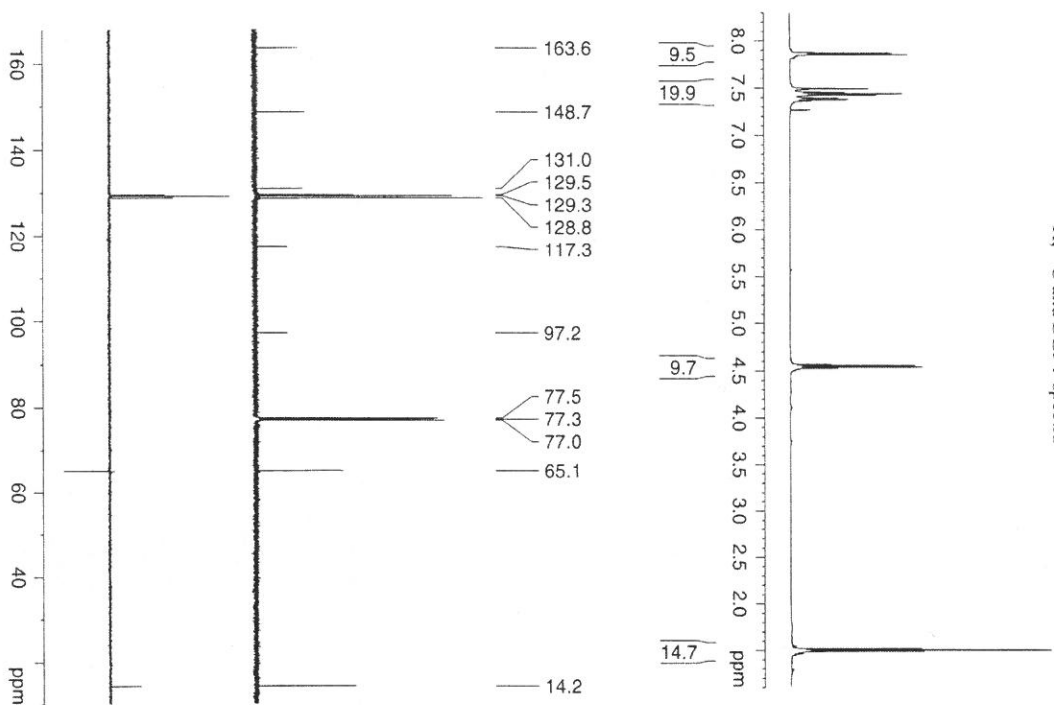
**Problem 26:**  $C_{11}H_{18}O_4$   
 IR:  $1732\text{ cm}^{-1}$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  with expansion and  $APt$  spectra



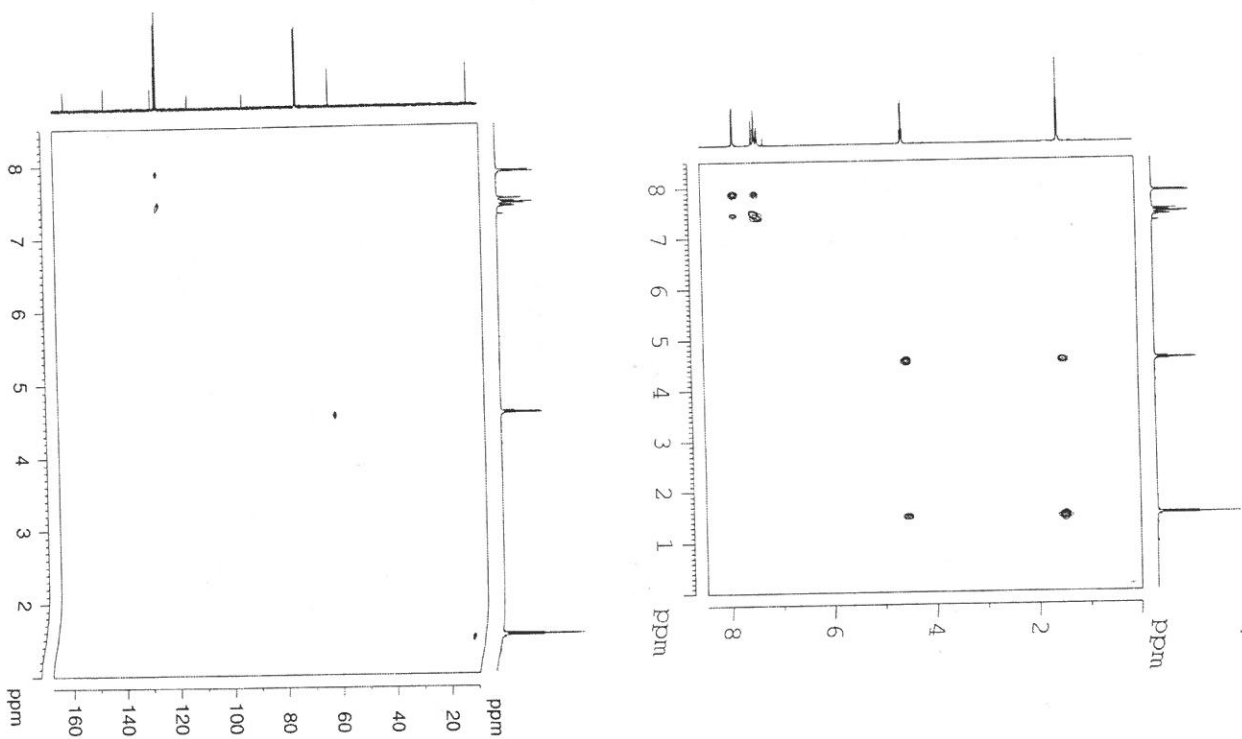
**Problem 26:**  $C_{11}H_{18}O_4$   
 200 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



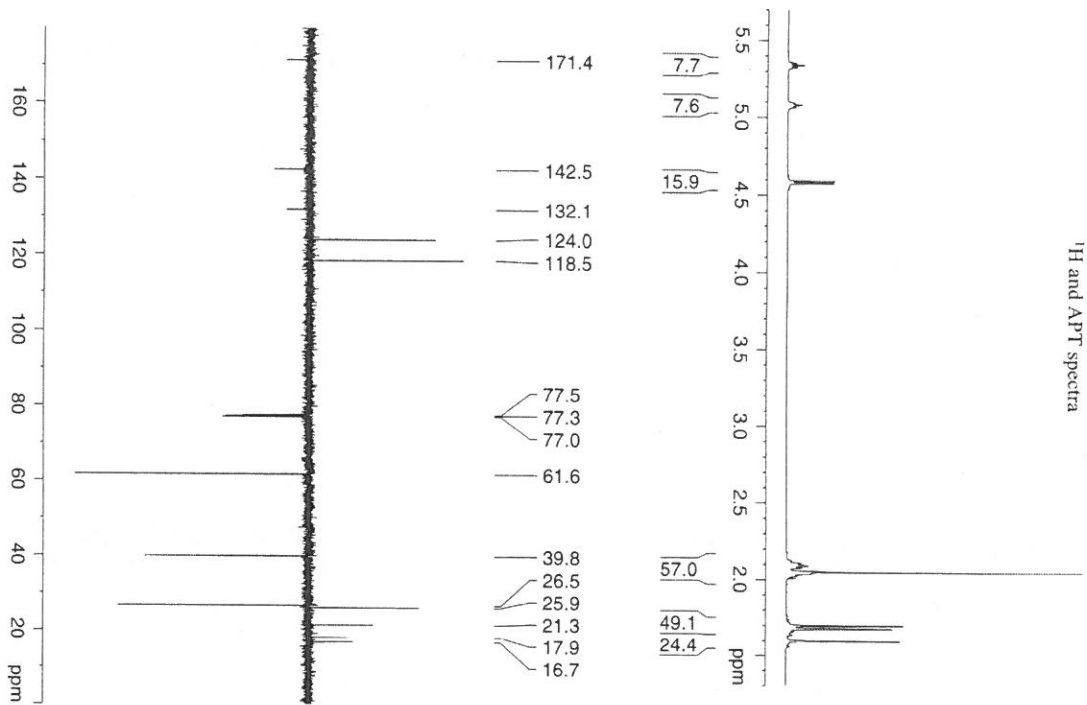
**Problem 27:**  $C_{12}H_{11}NO_3$   
 IR: 1631, 1714, 2216, 3230  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra



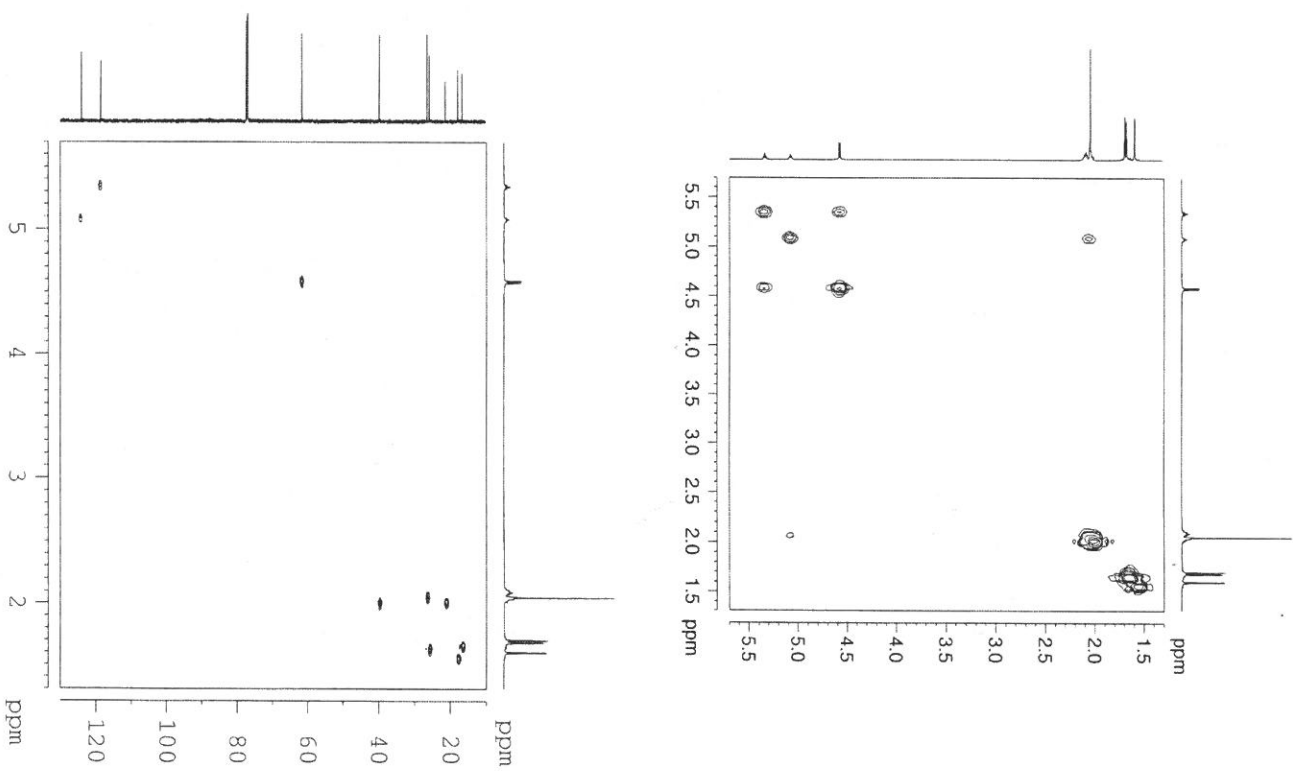
**Problem 27:**  $C_{12}H_{11}NO_3$   
 500 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



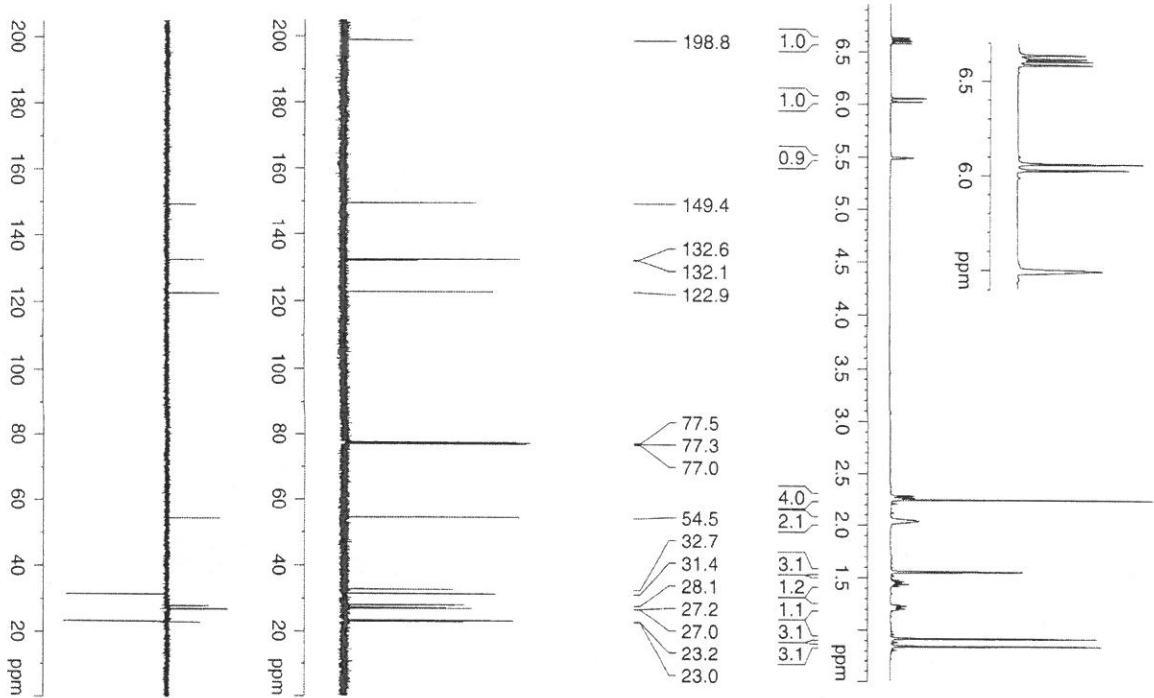
**Problem 28:**  $C_{12}H_{20}O_2$   
 IR: 1680, 1740  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$  and APT spectra



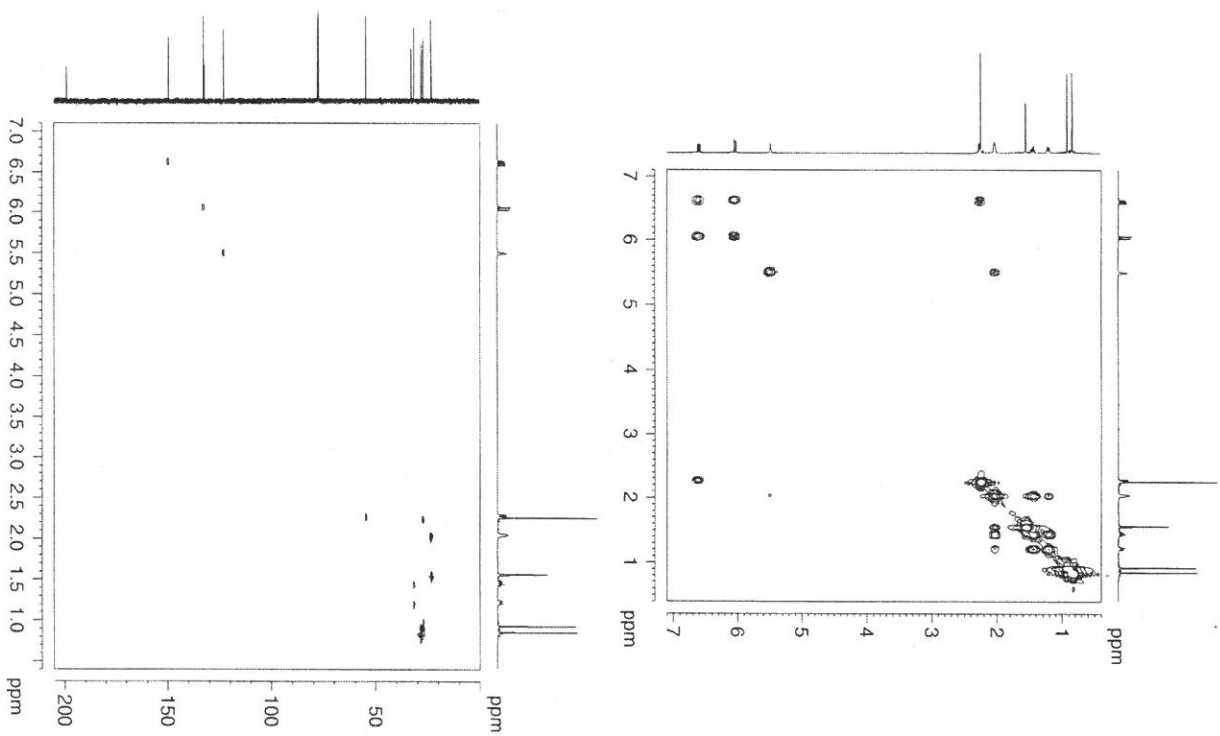
**Problem 28:**  $C_{12}H_{20}O_2$   
 500 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



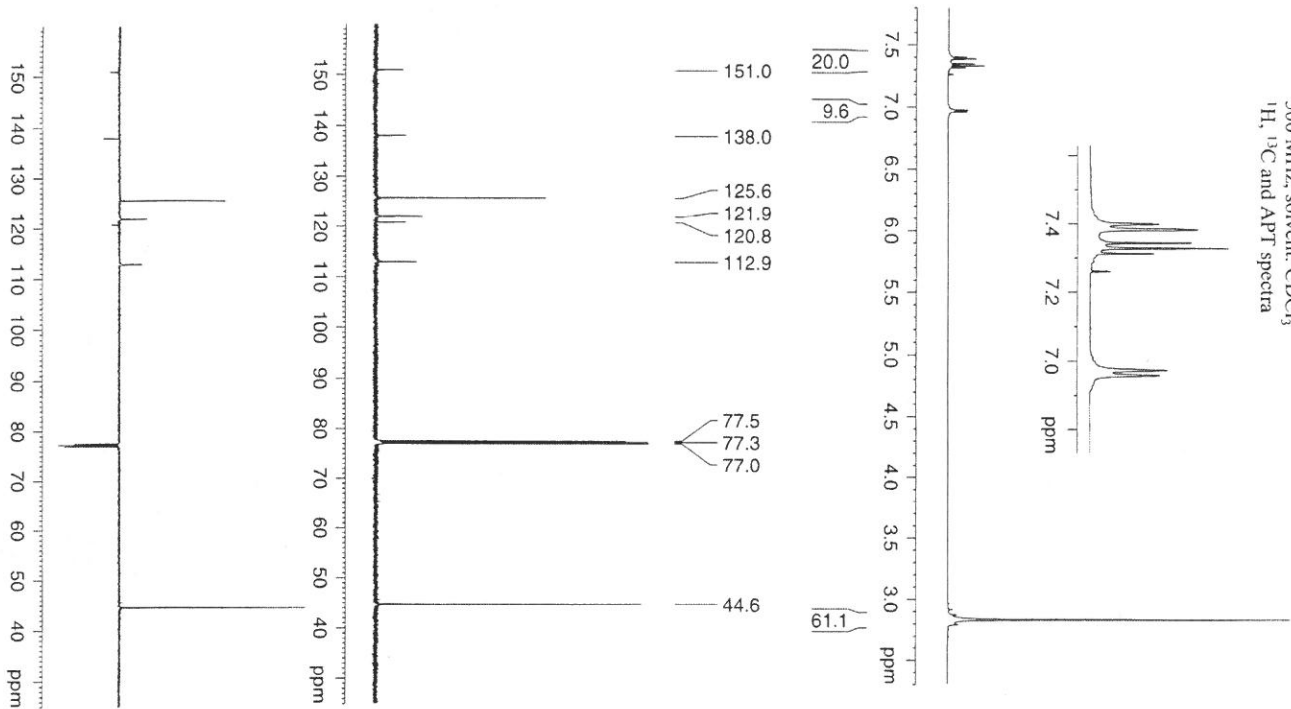
**Problem 29:**  $C_{13}H_{20}O$   
 IR: 1620, 1674  $cm^{-1}$   
 400 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra



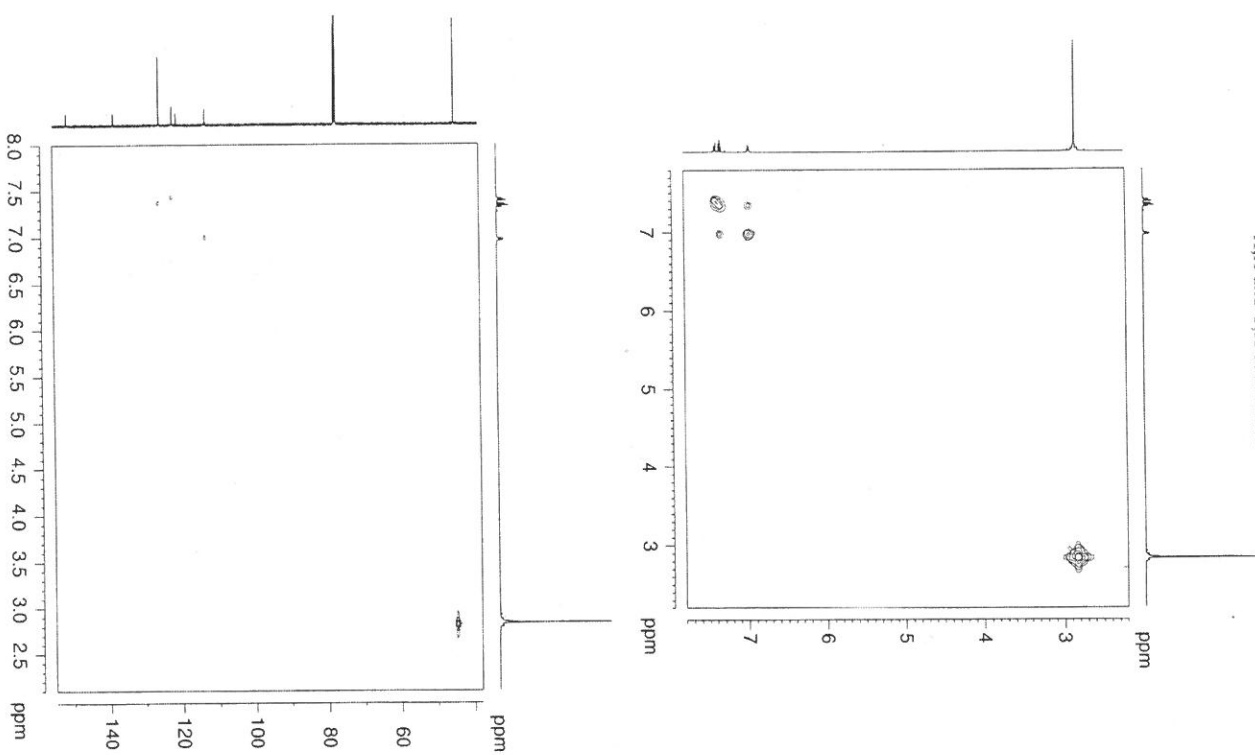
**Problem 29:**  $C_{13}H_{20}O$   
 400 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  correlation



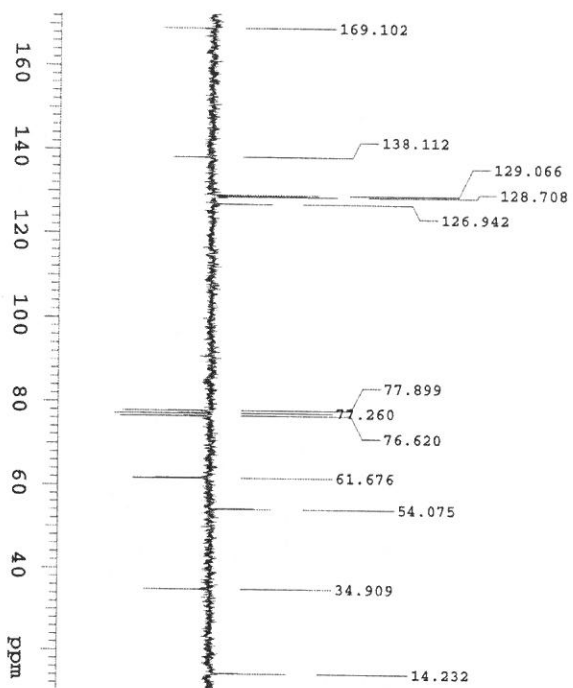
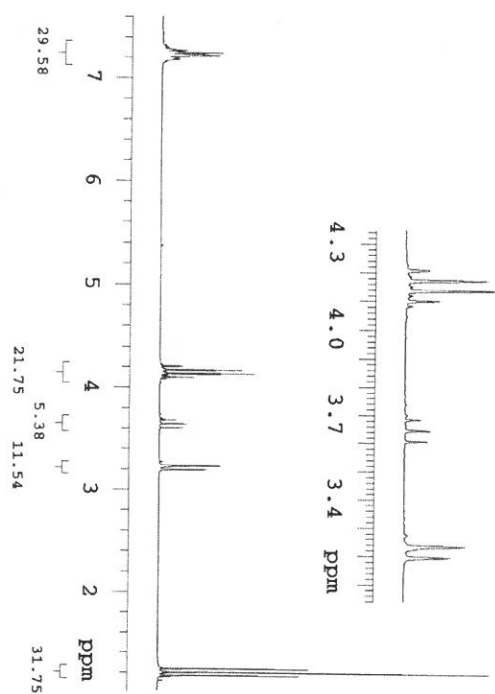
**Problem 30:**  $C_{14}H_{18}N_2$   
 IR: no bands characteristic of functional groups  
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and APT spectra



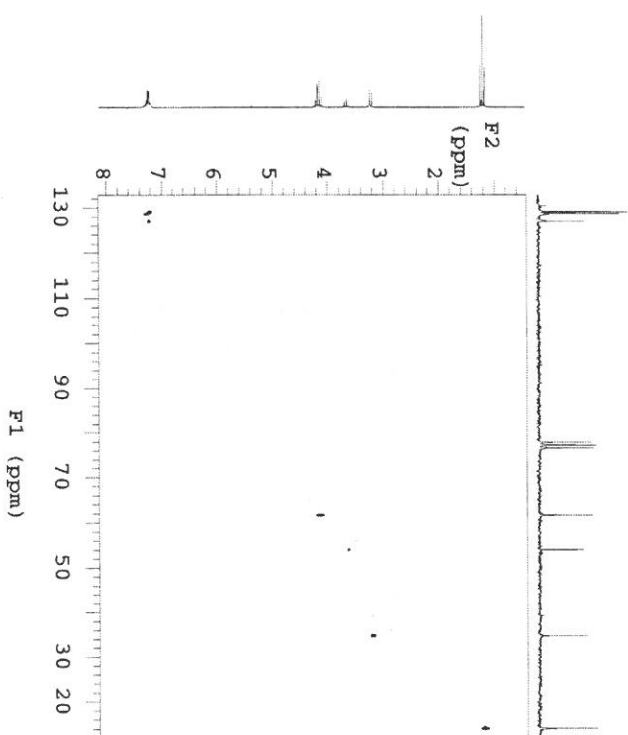
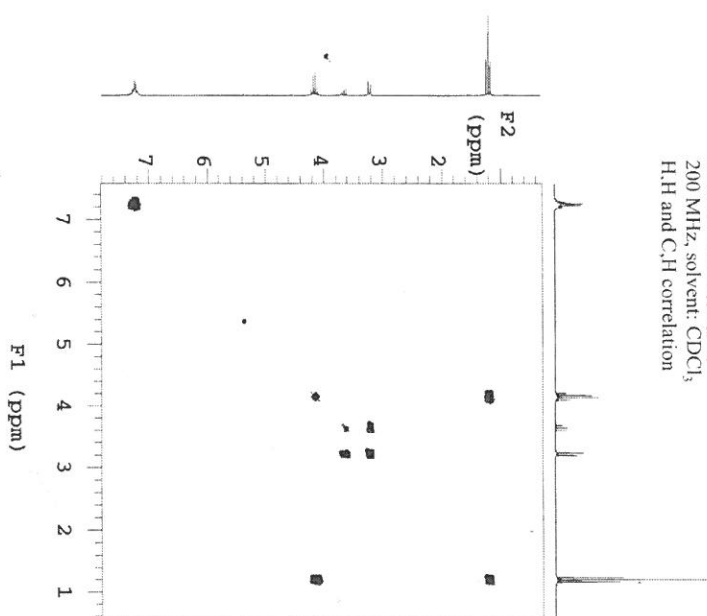
**Problem 30:**  $C_{14}H_{18}N_2$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  correlation



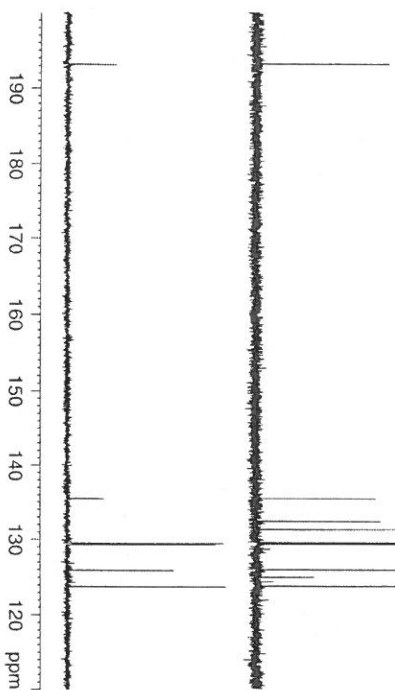
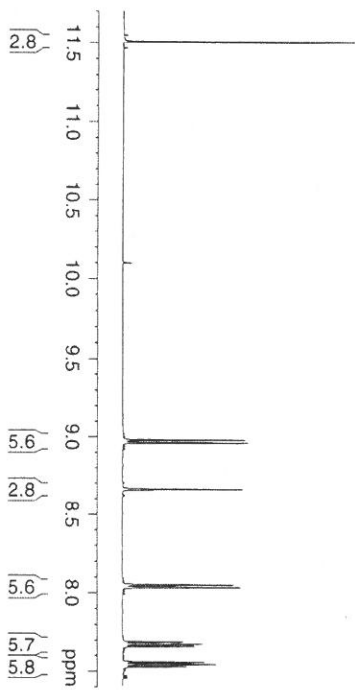
**Problem 31:**  $C_{14}H_{18}O_4$   
 IR:  $1735\text{ cm}^{-1}$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  with expansion and APT spectra



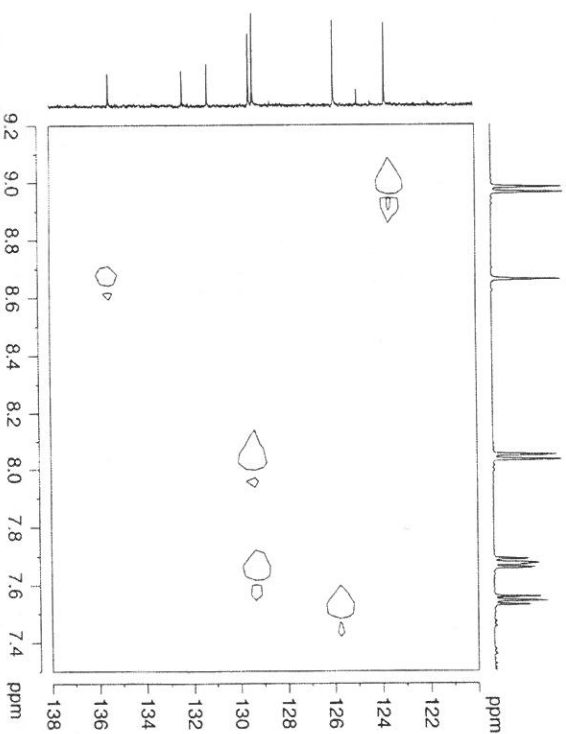
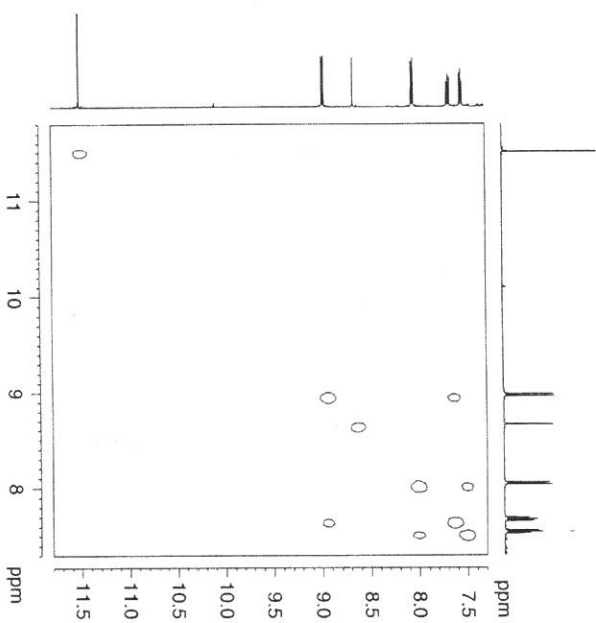
**Problem 31:**  $C_{14}H_{18}O_4$   
 200 MHz, solvent:  $CDCl_3$   
 $^1H$  and  $^{13}C$  correlation



**Problem 32:**  $C_{15}H_{10}O$   
 IR: 1668 (strong)  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra

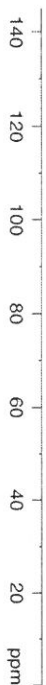
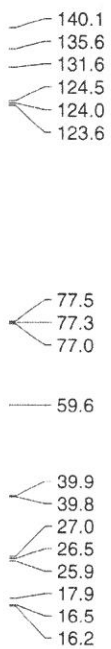
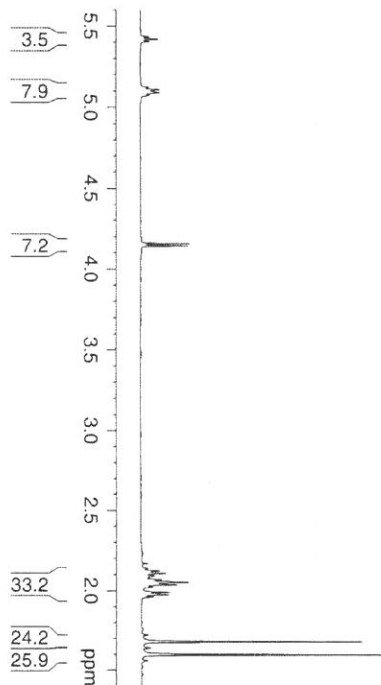


**Problem 32:**  $C_{15}H_{10}O$   
 500 MHz, solvent:  $CDCl_3$   
 $H,H$  correlation  
 aromatic part of the  $C,H$  correlation

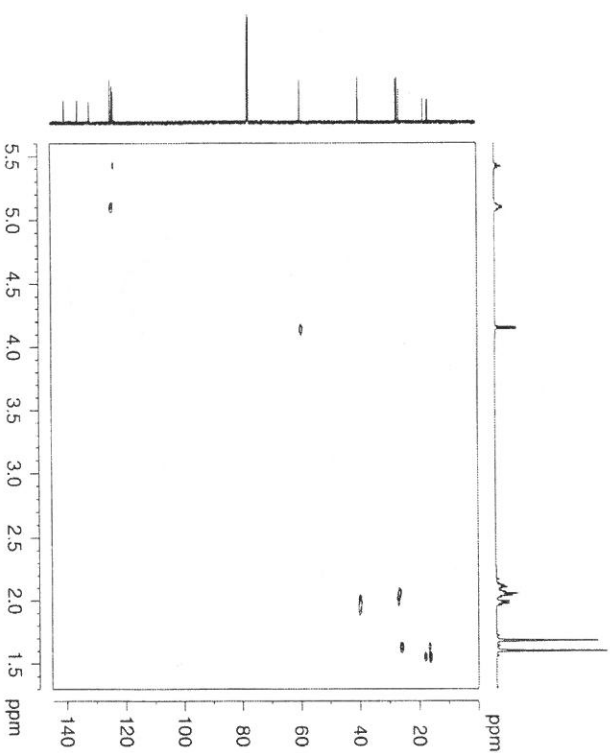
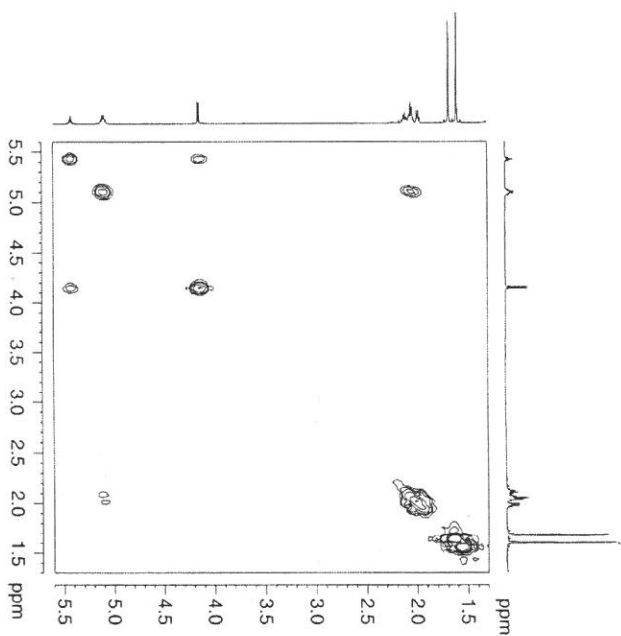




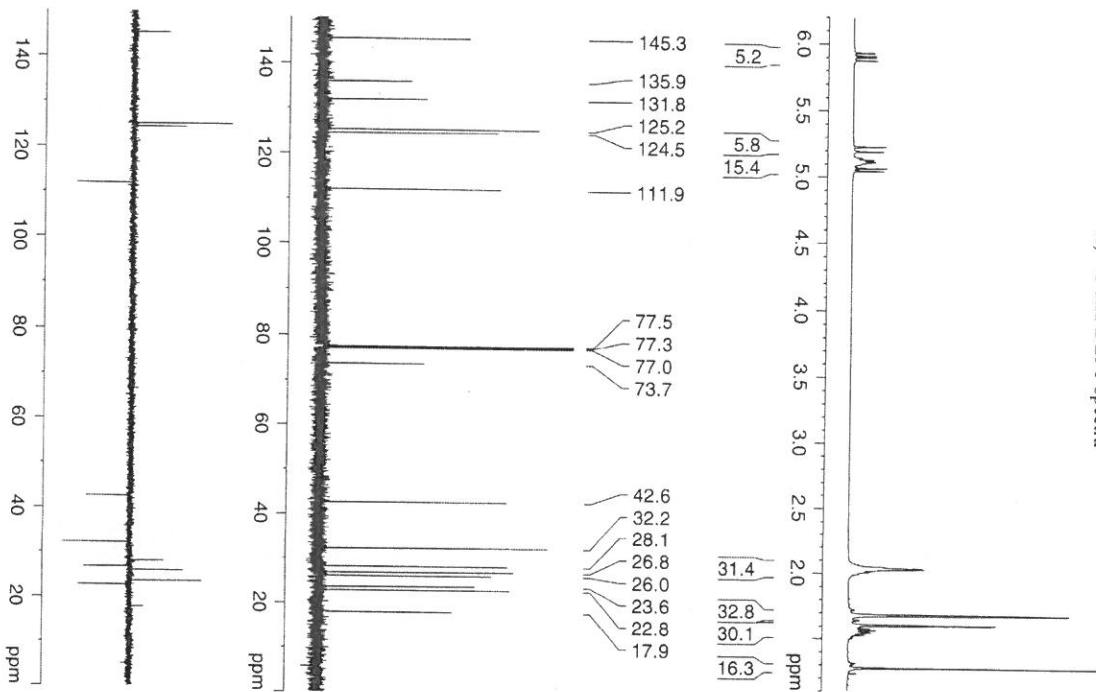
**Problem 33: C<sub>15</sub>H<sub>26</sub>O**  
 IR: 1668, 3366 (broad) cm<sup>-1</sup>  
 500 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H and APT spectra



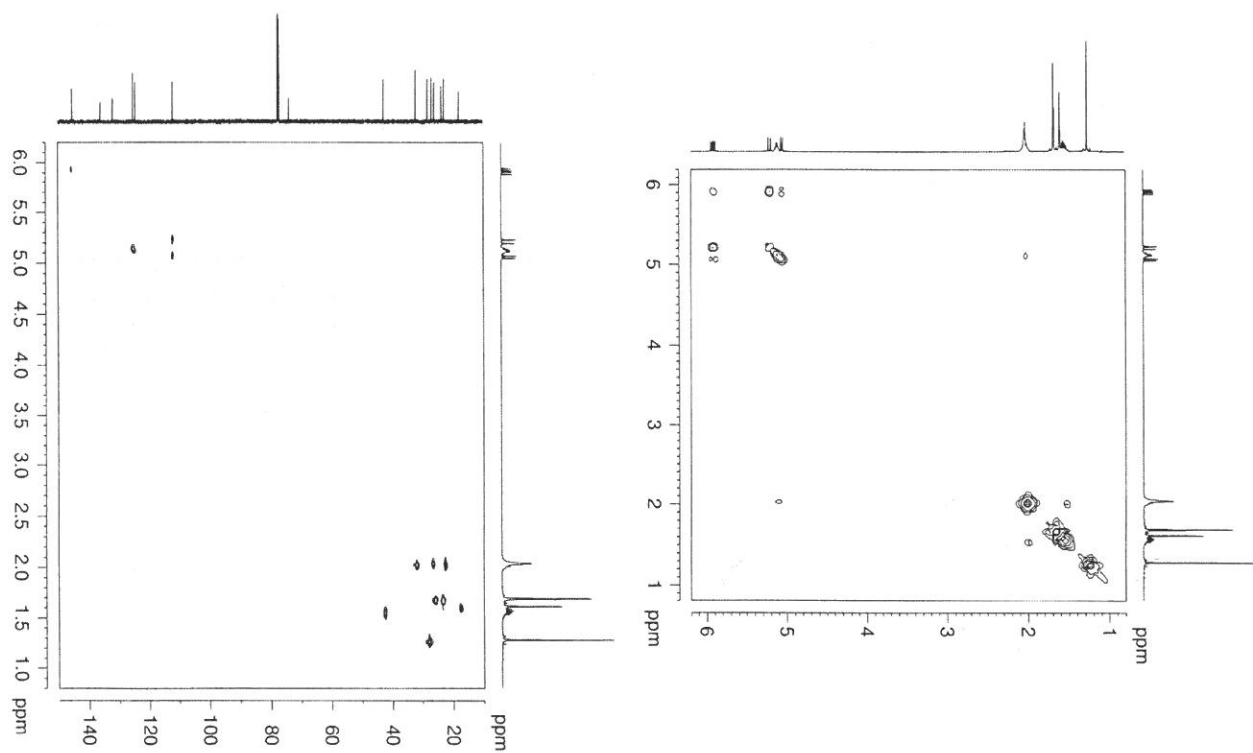
**Problem 33: C<sub>15</sub>H<sub>26</sub>O**  
 500 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H and <sup>13</sup>C correlation



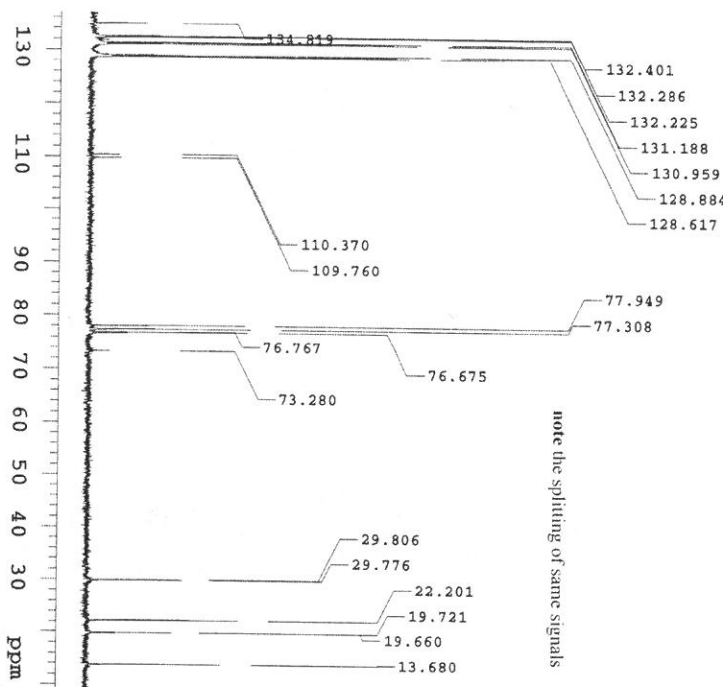
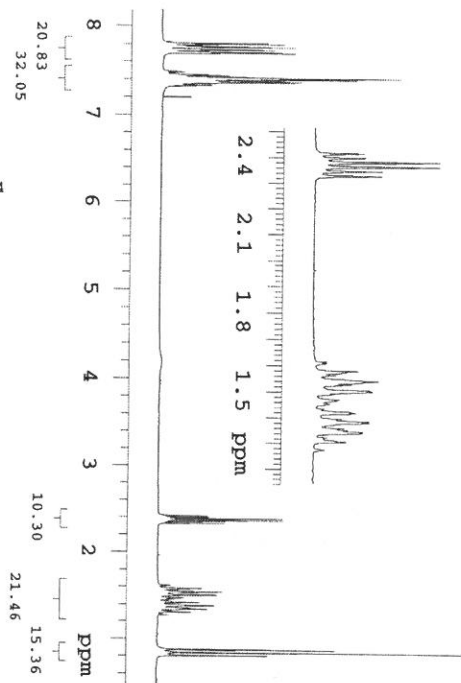
**Problem 34:  $C_{15}H_{26}O$**   
 IR: 1675, 3406 (broad)  $cm^{-1}$   
 500 MHz, solvent:  $CDCl_3$   
 $^1H$ ,  $^{13}C$  and DEPT spectra



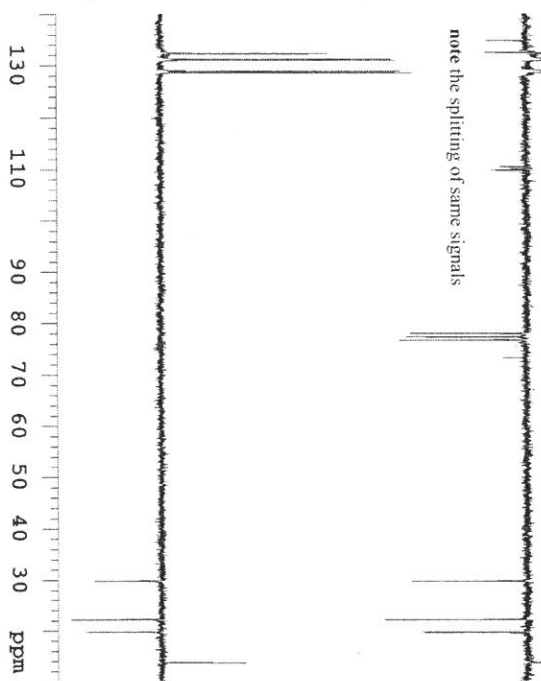
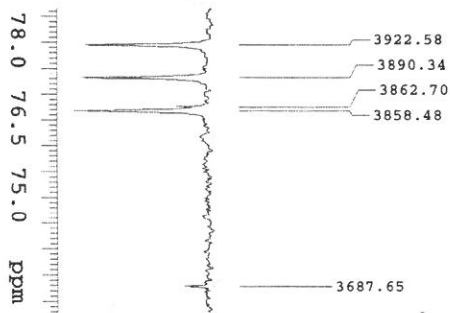
**Problem 34:  $C_{15}H_{26}O$**   
 500 MHz, solvent:  $CDCl_3$   
 $H,H$  and  $C,H$  correlation



**Problem 35: C<sub>18</sub>H<sub>16</sub>OP**  
<sup>31</sup>P NMR: δ = 8.6 ppm  
 IR: 1195, 2194 cm<sup>-1</sup>  
 200 MHz, solvent: CDCl<sub>3</sub>  
<sup>1</sup>H and <sup>13</sup>C spectra



**Problem 35: C<sub>18</sub>H<sub>16</sub>OP**  
 200 MHz, solvent: CDCl<sub>3</sub>  
 APT and DEPT spectra  
 expansion: peak frequency in Hz

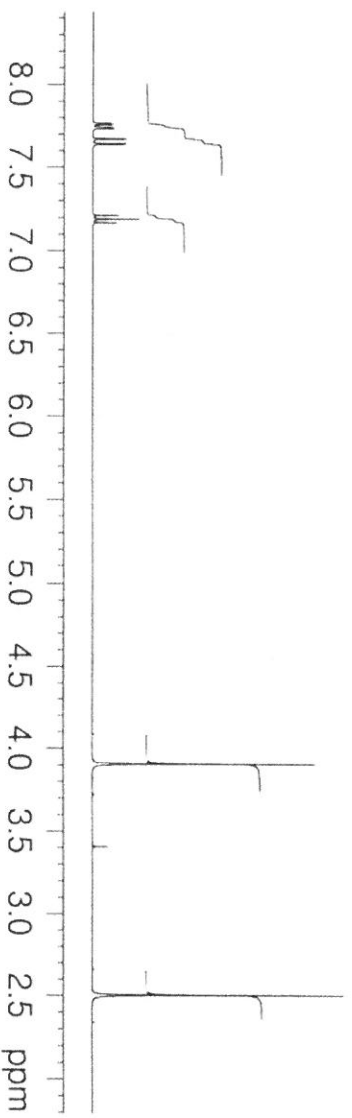


# Problem 42

Identify the following compound.

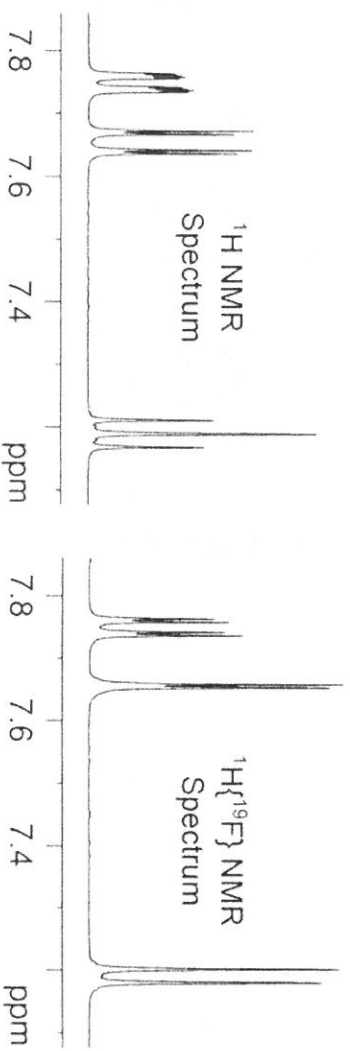
Molecular Formula:  $C_9H_9FO_2$

IR:  $1681\text{ cm}^{-1}$



$^1\text{H}$  NMR Spectrum  
(DMSO- $d_6$ , 400 MHz)

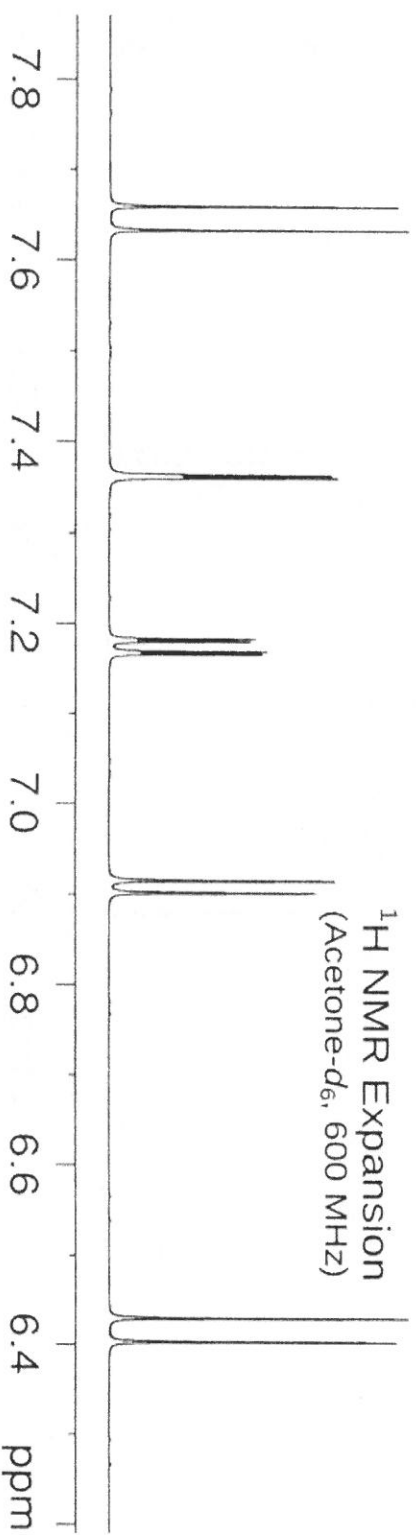
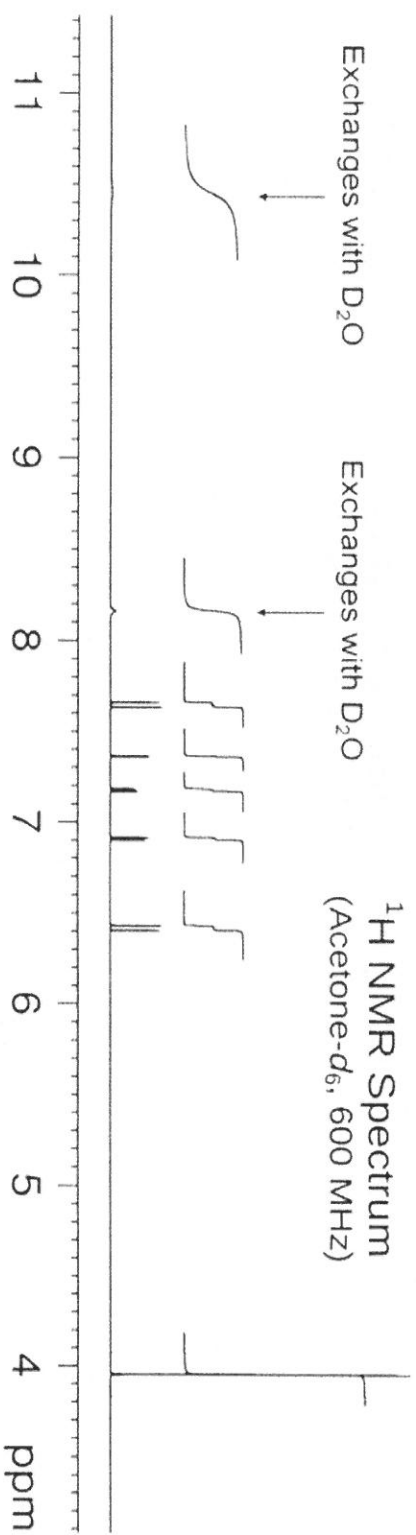
$^1\text{H}$  NMR Expansions  
(DMSO- $d_6$ , 400 MHz)



# Problem 43

Identify the following compound.

Molecular Formula:  $C_{10}H_{10}O_4$



# Problem 55

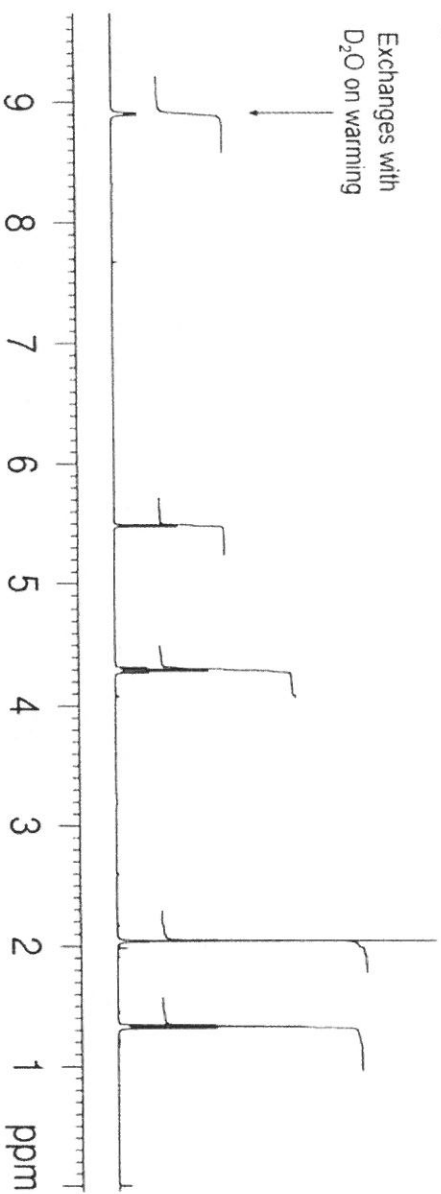
Identify the following compound.

Molecular Formula:  $C_7H_{10}N_2O_3$

IR: 2930, 2258, 1740, 1698, 1643  $cm^{-1}$

$^1H$  NMR Spectrum  
( $CDCl_3/DMSO-d_6$ , 500 MHz)

Exchanges with  
 $D_2O$  on warming



### Exercise 4

The COSY spectrum and the one-dimensional  $^1\text{H}$  NMR spectrum at the top of it have been recorded from glutathione ( $\gamma$ -L-glutamyl-L-cysteinylglycine, 17) in  $\text{D}_2\text{O}$ . Assign the signals as far as possible. Why do the signals at  $\delta = 2.3 - 2.4$  and  $\delta = 2.6 - 2.7$  appear as strongly split multiplets?

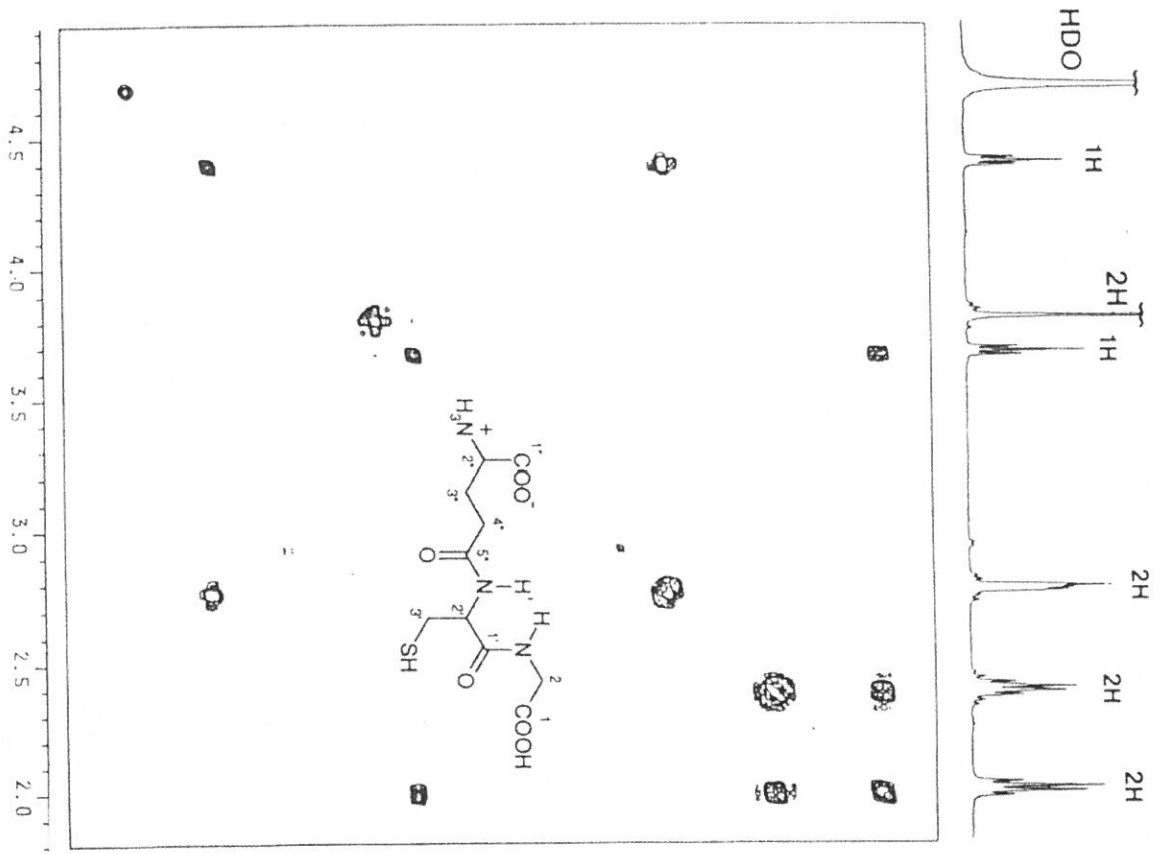


Fig. 4.4.  $^1\text{H}$  NMR and COSY spectrum of glutathione (17) in  $\text{D}_2\text{O}$ . Acetone- $d_6$  in  $\text{D}_2\text{O}$  ( $\delta = 2.04$ ) has been used as external standard, i.e., measured in a separate sample tube.

### Exercise 5

In the previous exercise 4 the COSY spectrum of glutathione (17) recorded in  $\text{D}_2\text{O}$  allowed the assignment of the  $^1\text{H}$  resonances belonging to each individual amino acid. If 17 is dissolved in  $\text{DMSO}-d_6$  the two NH protons of the amide bond appear as separate signals due to their slower exchange rate in that strongly complexing solvent.

Assign the signals of the NH protons and use them to prove the amino acid sequence in glutathione (17). Note: All spectra were recorded with suppression of the water peak resulting in a reduction of ca. 90% of the very broad water peak at  $\delta = 3 - 4.5$ ; there is a residual spike artifact at  $\delta = 3.78$ .

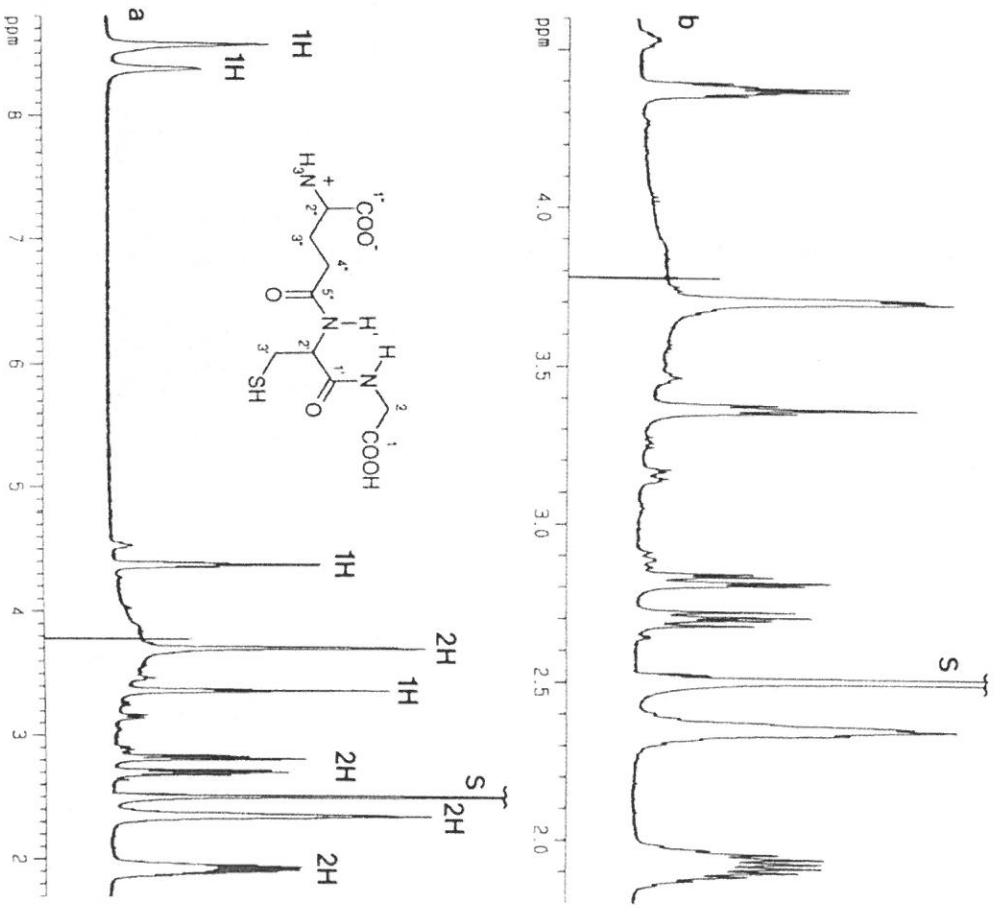


Fig. 4.5.1a. 500 MHz  $^1\text{H}$  NMR spectrum of 17, in  $\text{DMSO}-d_6$ ; b section of the same spectrum; S: solvent

### Exercise 13

The NMR spectra depicted in Figs. 4.13.1 - 4.13.3 belong to a pleasant smelling liquid. What is the structure of this compound **22** which is a natural product with the molecular formula  $C_{15}H_{20}O$ ?

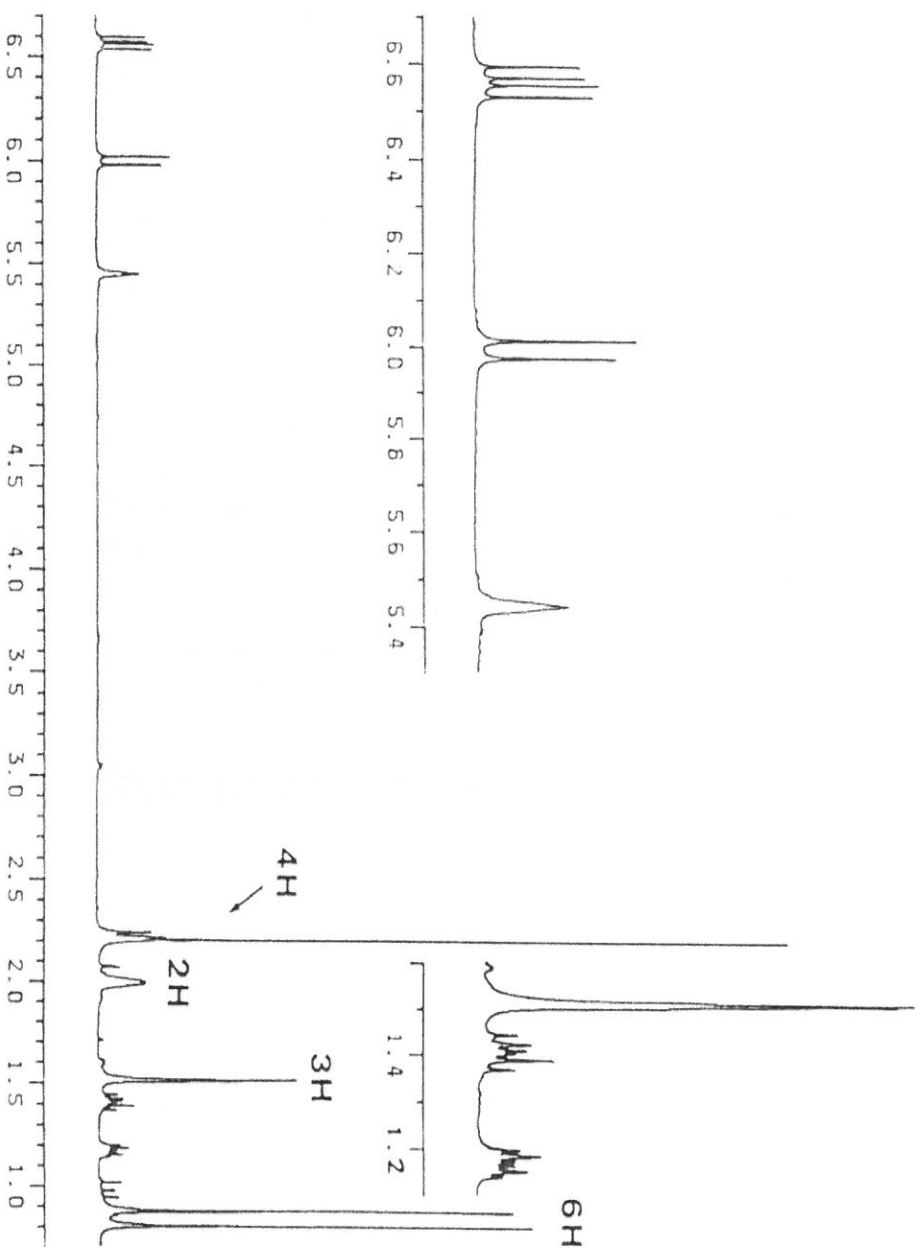


Fig. 4.13.1. 400 MHz  $^1H$  NMR spectrum of **22**, in  $CDCl_3$ .



### Exercise 15

Compound 24 has been isolated from the plant *Aristolochia argentina*, and its molecular formula is  $C_{12}H_{18}O_2$ . What is the structure of 24?

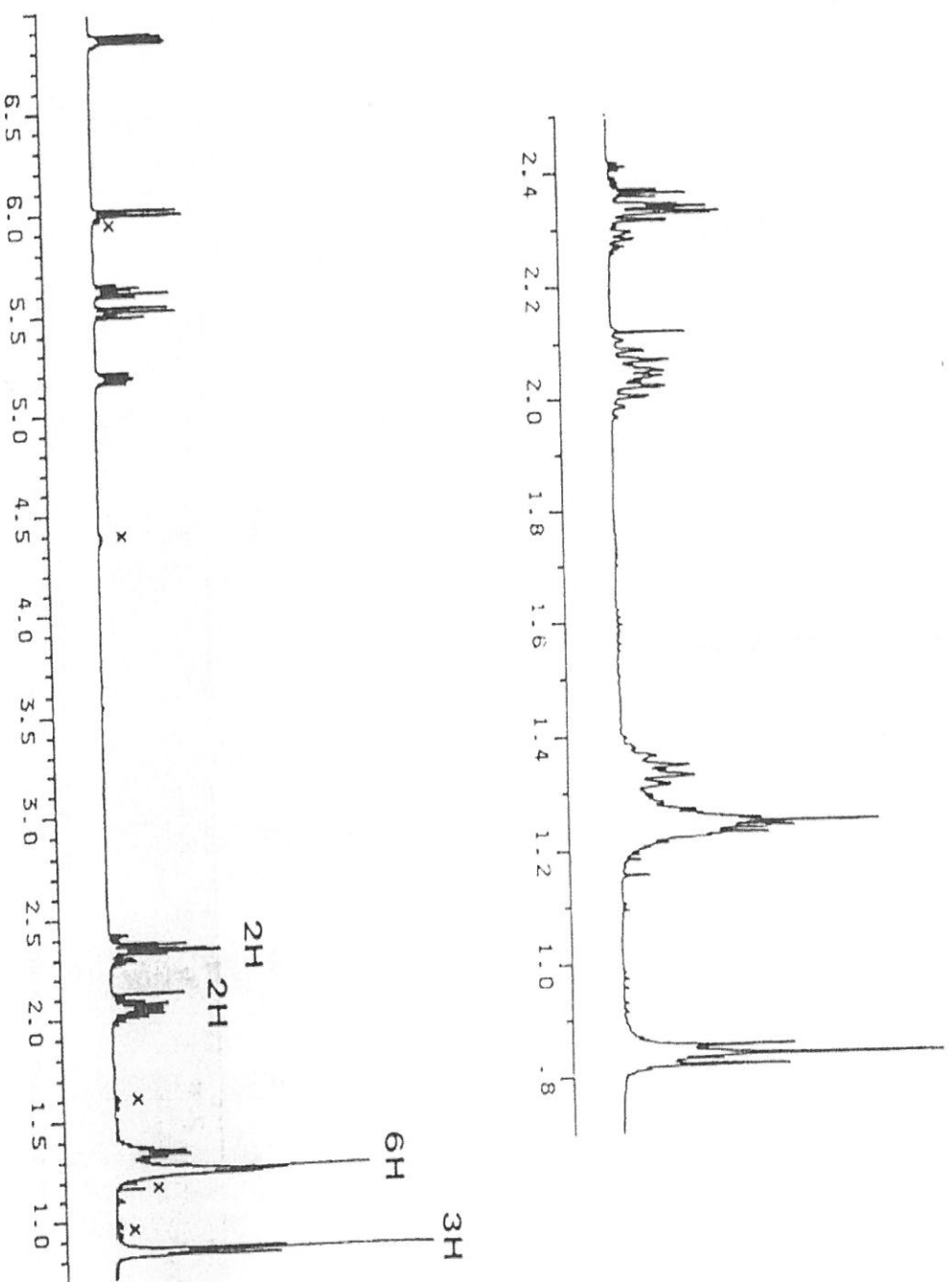


Fig. 4.15.1. 400 MHz  $^1\text{H}$  NMR spectrum of 24, in  $\text{CDCl}_3$ ; the little peaks marked by "x" belong to an impurity.