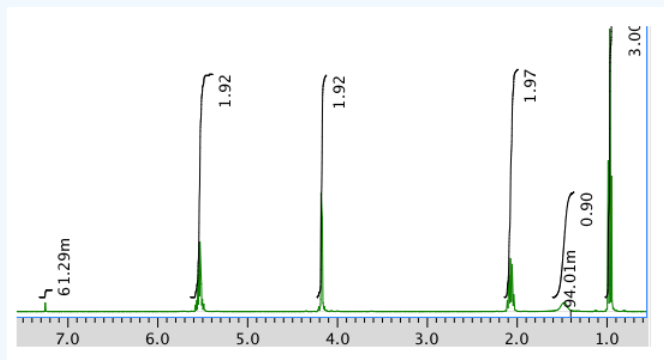


## 5.6: More Practice with 2D

The following problems involve real samples. Note that you may need to check for peaks due to solvent. Helpful tables may be found [here](#).

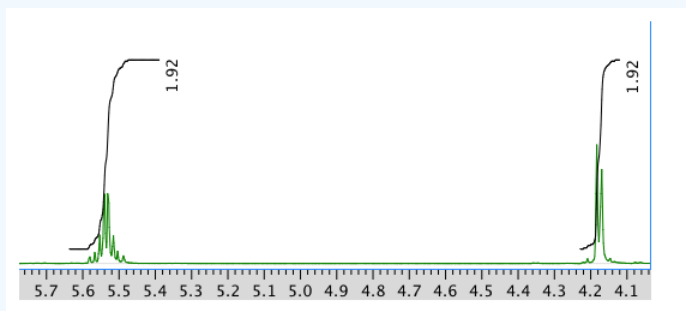
### Exercise 5.6.1

The following problems involve real samples. Note that you may need to check for peaks due to solvent. Helpful tables may be found [here](#).

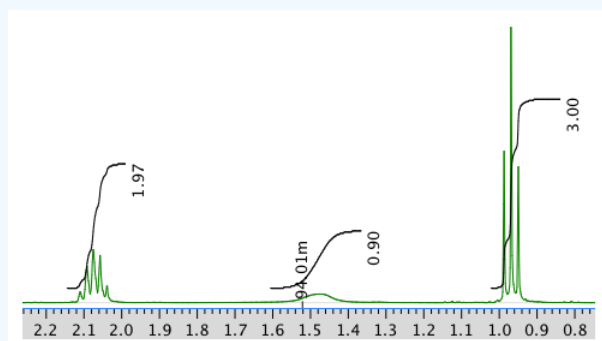


Note that the m beside one of the integral values stands for milli (one thousandth).  $61.29\text{m} = 0.06129$  units.

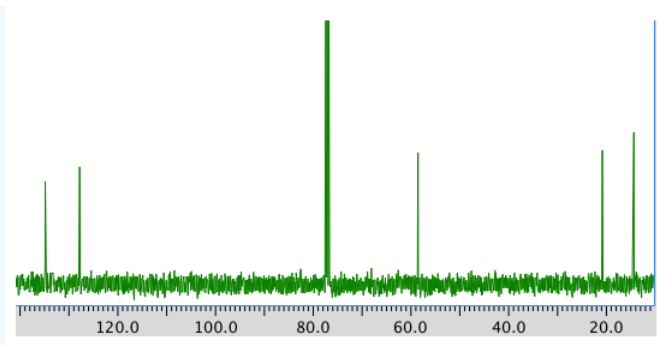
An expansion of the  $^1\text{H}$  NMR spectrum:



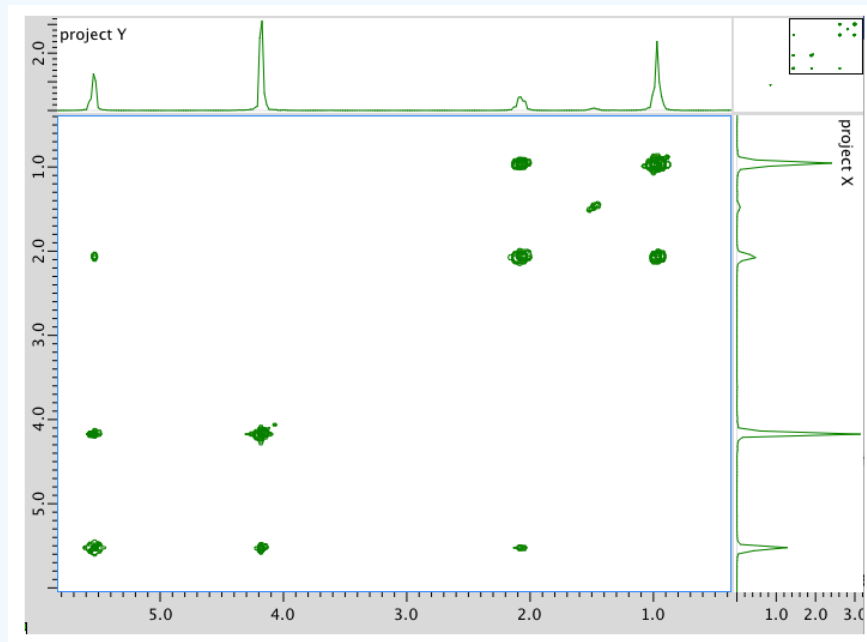
Another expansion:



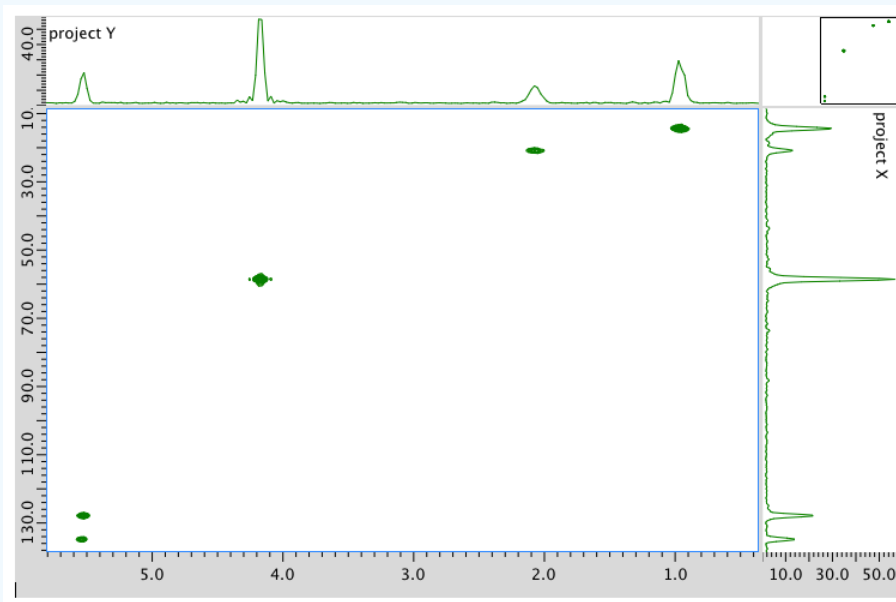
A  $^{13}\text{C}$  NMR spectrum:



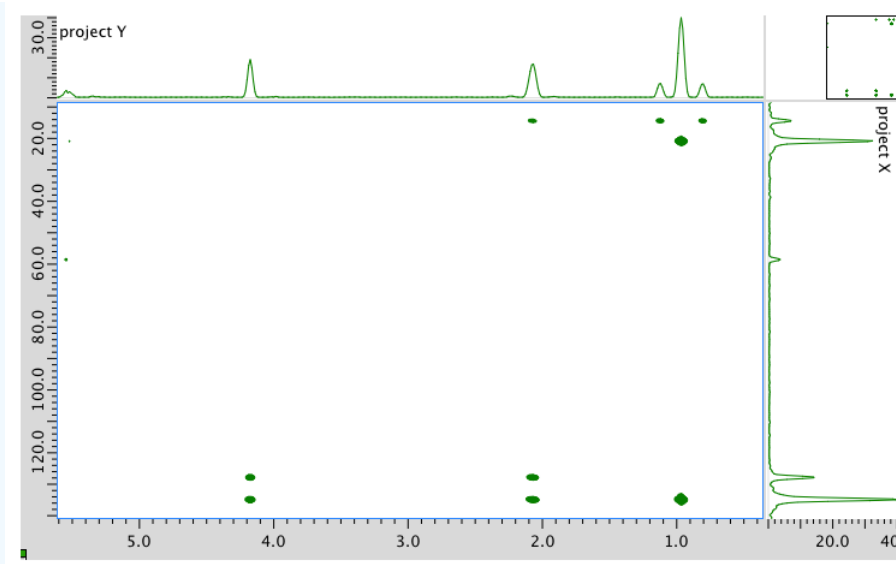
COSY spectrum:



HMQC spectrum:



HMBC spectrum:



### Answer

$^1\text{H}$  NMR

Chemical shift (ppm)	Integration	Multiplicity	Partial Structure
5.54	2H	multiplet	$\text{CH}=\text{C}$ (x 2)
4.17	2H	doublet	$\text{O}-\text{CH}_2-\text{CH}$
2.08	2H	quintet	$\text{CH}-\text{CH}_2-\text{CH}_3$
1.47	1H	broad singlet	OH
0.95	3H	triplet	$\text{CH}_2-\text{CH}_3$

\* total # H: 10

$^{13}\text{C}$  NMR

Chemical shift (ppm)	Type of carbon
134	$\text{sp}^2$
128	$\text{sp}^2$
58	$\text{sp}^3-\text{O}$
21	$\text{sp}^3$
14	$\text{sp}^3$

\*total # C: 5

COSY

Assignment	$^1\text{H}$	COSY
A	0.95	2.08
B	2.08	0.95, 5.54
C	4.17	5.54

D	5.54	2.08
E	5.54	4.17

\*HMQC indicates two hydrogens at 5.54 are in two different environments

HMQC

Assignment	$^{13}\text{C}$	$^1\text{H}$
A	14	0.95
B	21	2.08
C	58	4.17
D	128	5.54
E	134	5.54

Formula:

$\text{C}_5\text{H}_{10}\text{O}$  (1 O indicated from shift in  $^{13}\text{C}$ ,  $^1\text{H}$  NMR)

$\text{FW} = 5 \times 12 + (10 \times 1) + (1 \times 16) = 86$

Compare  $\text{C}_5\text{H}_{10}$  ratio to  $\text{C}_5\text{H}_{12}$  in hydrocarbon

Degrees of unsaturation =  $\frac{(2 \times 5) + 2 - 10}{2} = 1$  unit (1 double bond)

The data tables should be consistent with this structure:

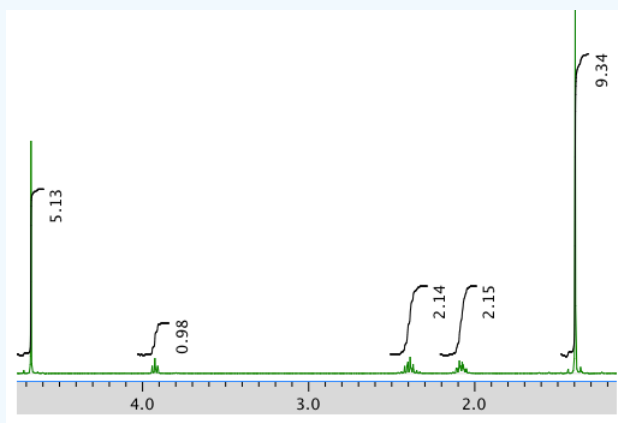
pent-2-en-1-ol (could be *cis* or *trans* based on this analysis)

### Exercise 5.6.2

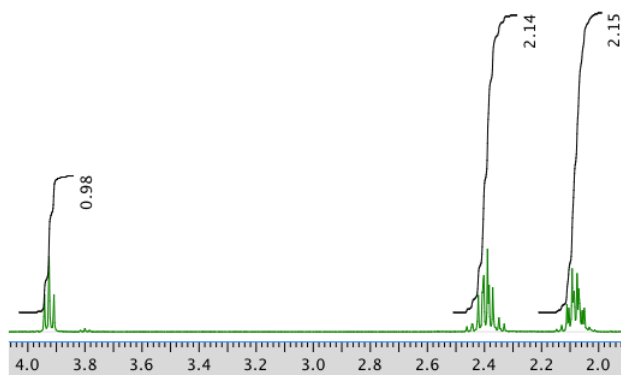
Present an analysis of the following data and propose a structure.

MW: 202 amu

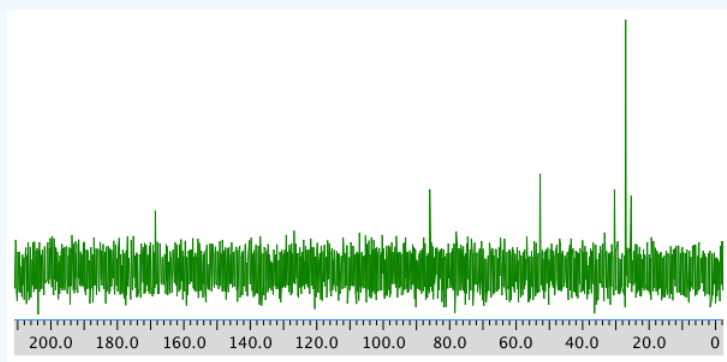
The full  $^1\text{H}$  NMR spectrum in  $\text{D}_2\text{O}$ :



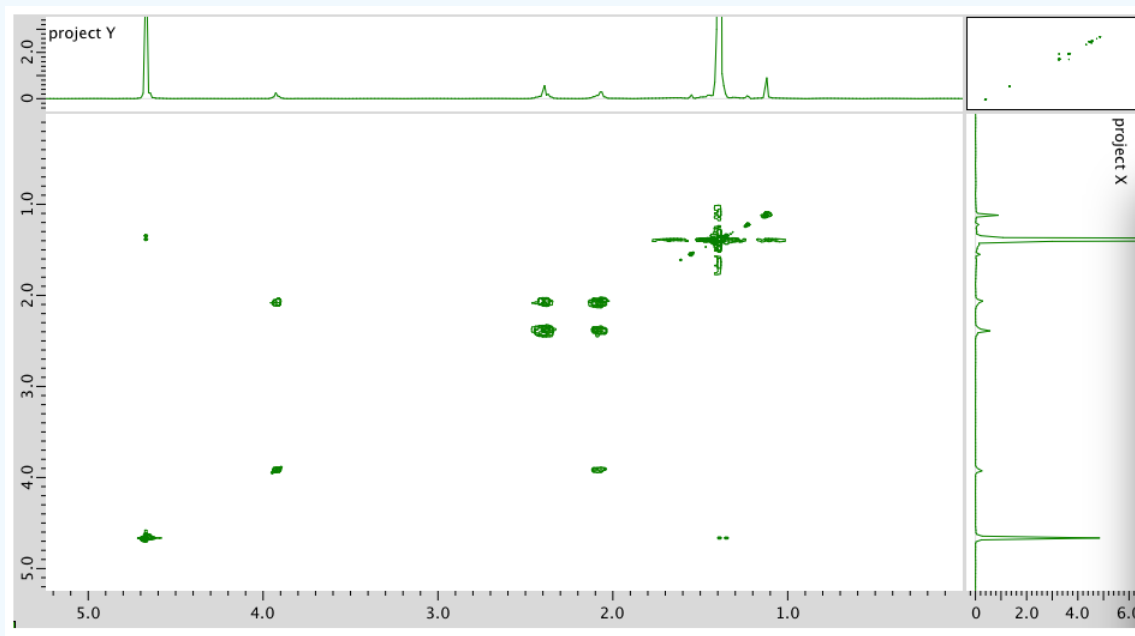
An expansion:



$^{13}\text{C}$  NMR:



COSY:



Answer

$^1\text{H}$  NMR:

Chemical shift (ppm)	Integration	Multiplicity	Partial structure

4.7	5H	singlet	solvent
3.93	1H	triplet	CH <sub>2</sub> -CH-N
2.40	2H	multiplet	CH <sub>2</sub> -CH <sub>2</sub> ?
2.09	2H	multiplet	CH <sub>2</sub> -CH <sub>2</sub> ?
1.4	9H	singlet	C(CH <sub>3</sub> ) <sub>3</sub>

\*Total number of H: 19 H

<sup>13</sup>C NMR:

Chemical shift (ppm)	Type of carbon
170	sp <sup>2</sup> (C=O)
80	sp <sup>3</sup> (C-O)
52	sp <sup>3</sup> (C-N)
32	sp <sup>3</sup>
28	sp <sup>3</sup>
26	sp <sup>3</sup>

\*Total number of C: 6 apparent, but two more suggested by symmetry (3 methyl groups in <sup>1</sup>H NMR) for 8 C; a third extra suggested by MW fit for 9 C

COSY:

Assignment	<sup>1</sup> H	COSY
Solvent	4.7	--
B	3.93	2.40
D	2.40	2.09
C	2.09	2.40, 2.09
A	1.4	--

Formula:

C<sub>9</sub>H<sub>18</sub>O<sub>3</sub>N<sub>2</sub> (extra O indicated from shift in <sup>13</sup>C, <sup>1</sup>H NMR; second O suggested by C=O in <sup>13</sup>C NMR; additional CO needed to fit MW)

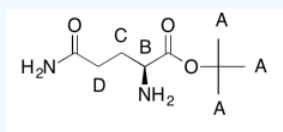
$$FW = (9 \times 12) + (18 \times 1) + (3 \times 16) + (2 \times 14) = 202$$

$$FW = ((9 \times 12) + (18 \times 1) + (3 \times 16) + (2 \times 14) = 202)$$

Compare C<sub>9</sub>H<sub>18</sub> to C<sub>9</sub>H<sub>22</sub> for the corresponding hydrocarbon corrected for two nitrogens (therefore two extra hydrogens)

$$\text{Degrees of unsaturation} = \frac{(2 \times 9) + 2 + 2 - 10}{2} = 2 \text{ units (2 double bonds)}$$

The data tables should be consistent with this structure:

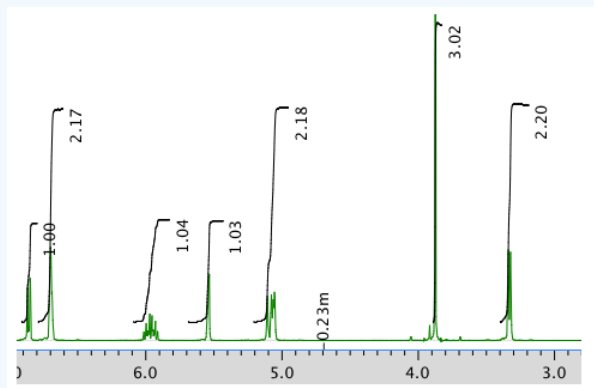


### Exercise 5.6.3

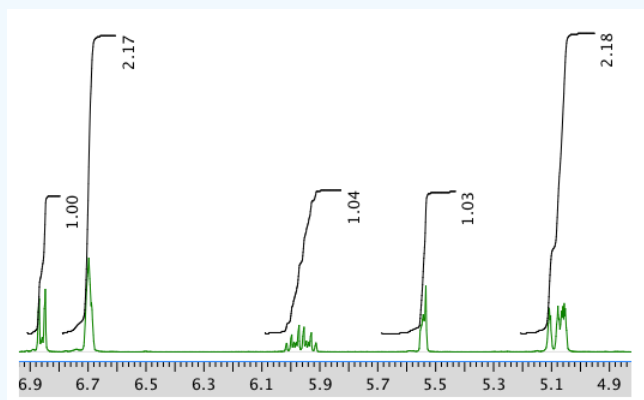
Present an analysis of the following data and propose a structure.

MW: 164 amu

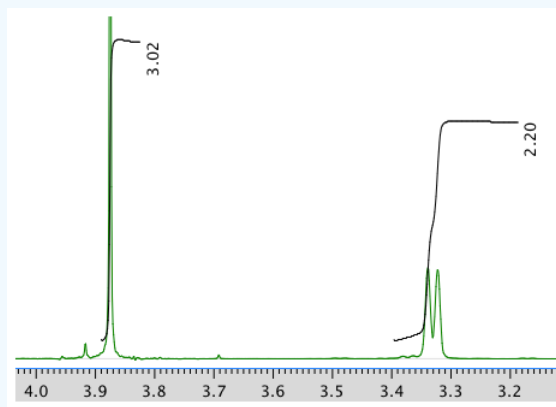
The full  $^1\text{H}$  NMR spectrum in  $\text{CDCl}_3$ :



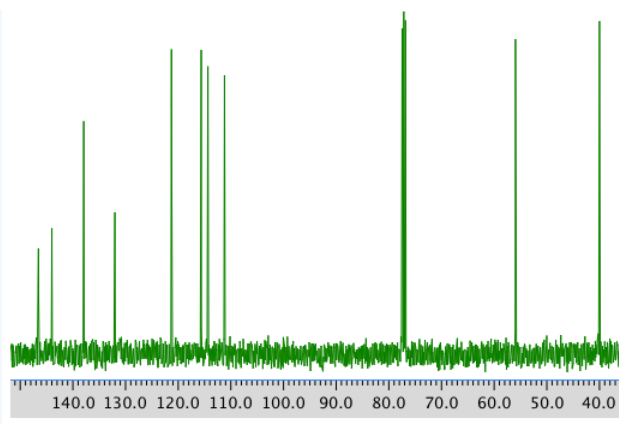
An expansion of the  $^1\text{H}$  NMR spectrum:



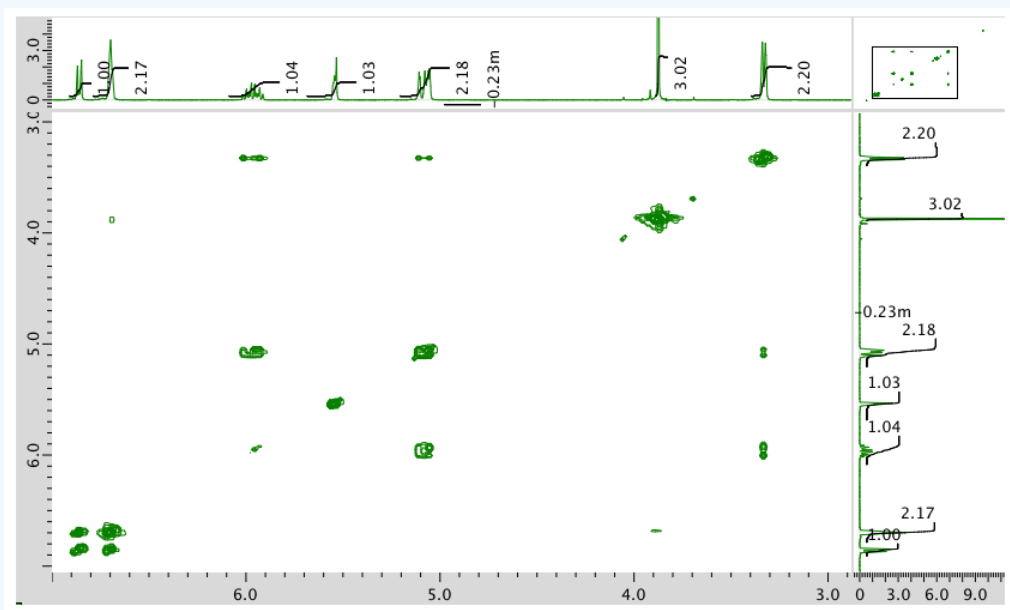
Another expansion:



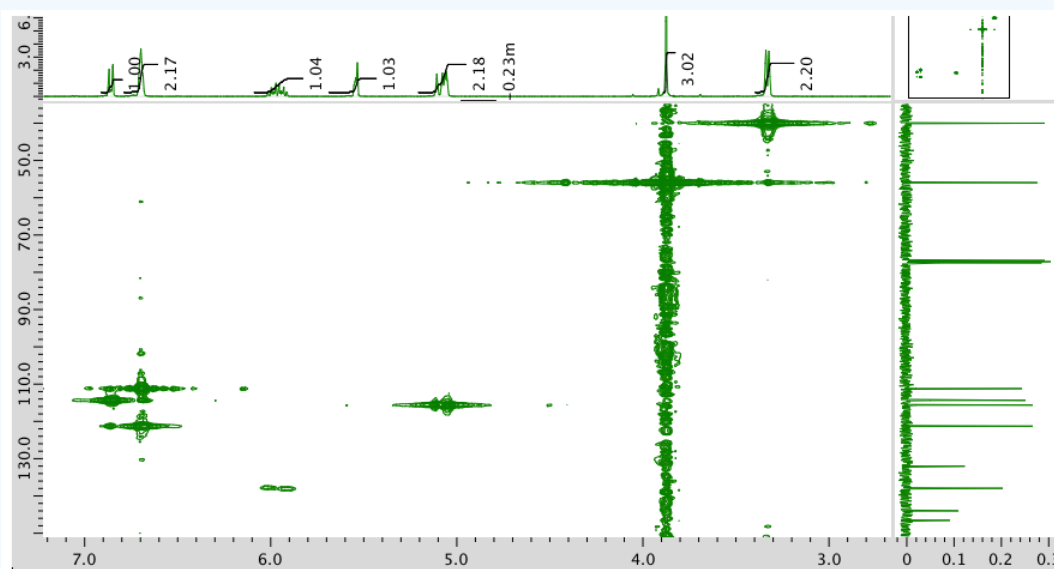
$^{13}\text{C}$  NMR spectrum:



COSY spectrum:

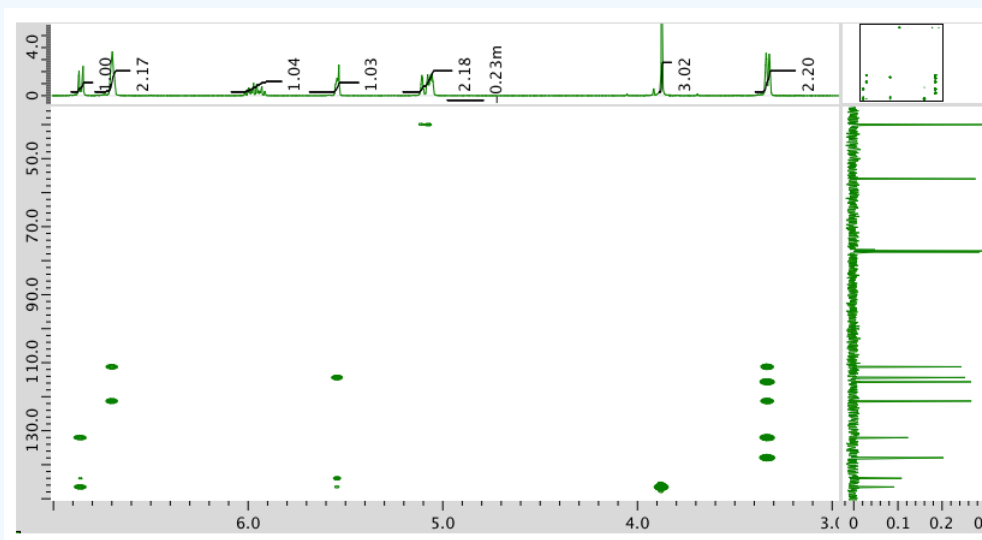


HMQC:



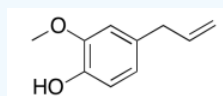


HMBC:



**Answer**

The data should be consistent with this structure:

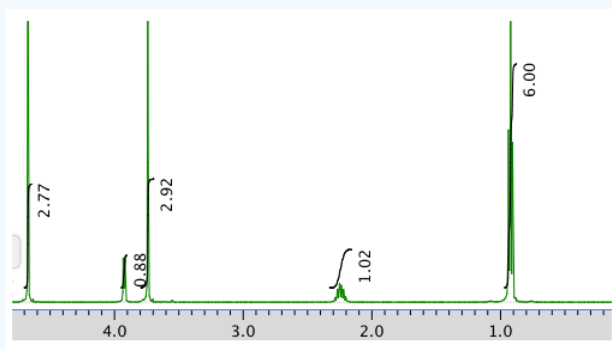


#### Exercise 5.6.4

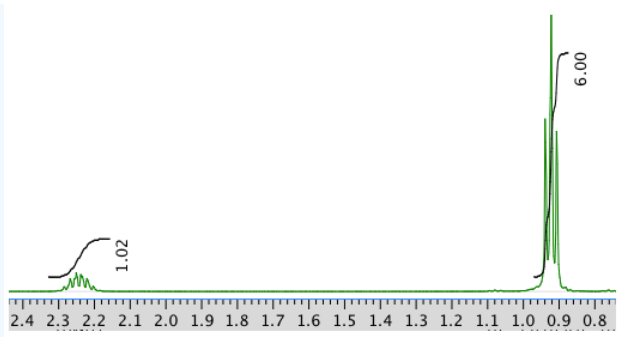
Present an analysis of the following data and propose a structure.

MW: 131 amu

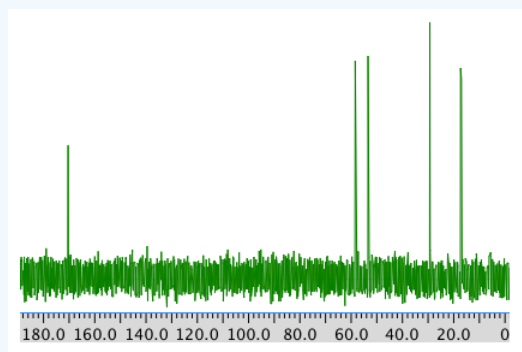
The full  $^1\text{H}$  NMR spectrum in  $\text{D}_2\text{O}$ :



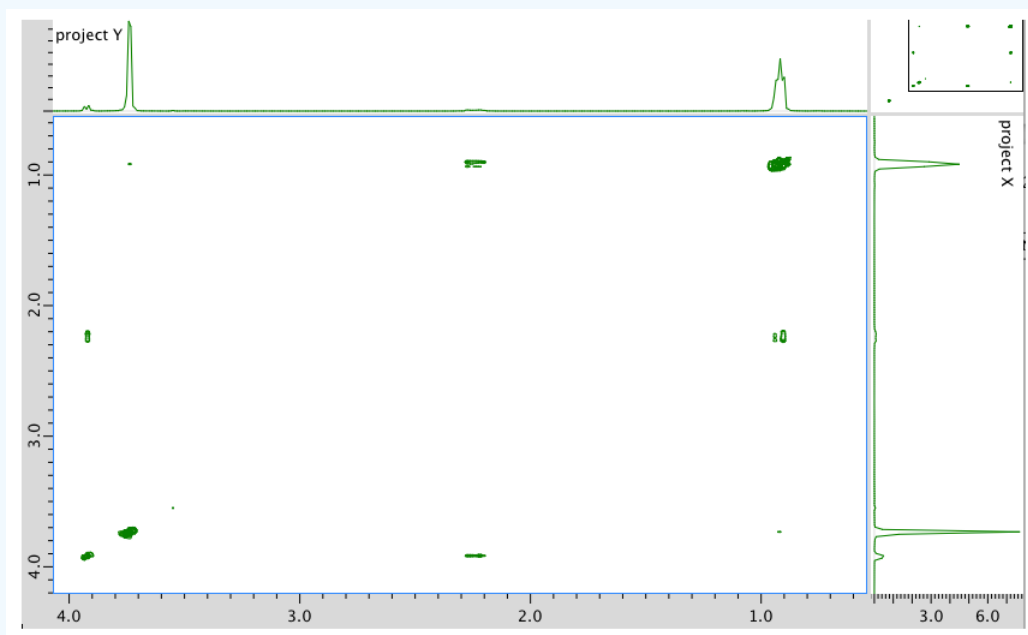
An expansion:



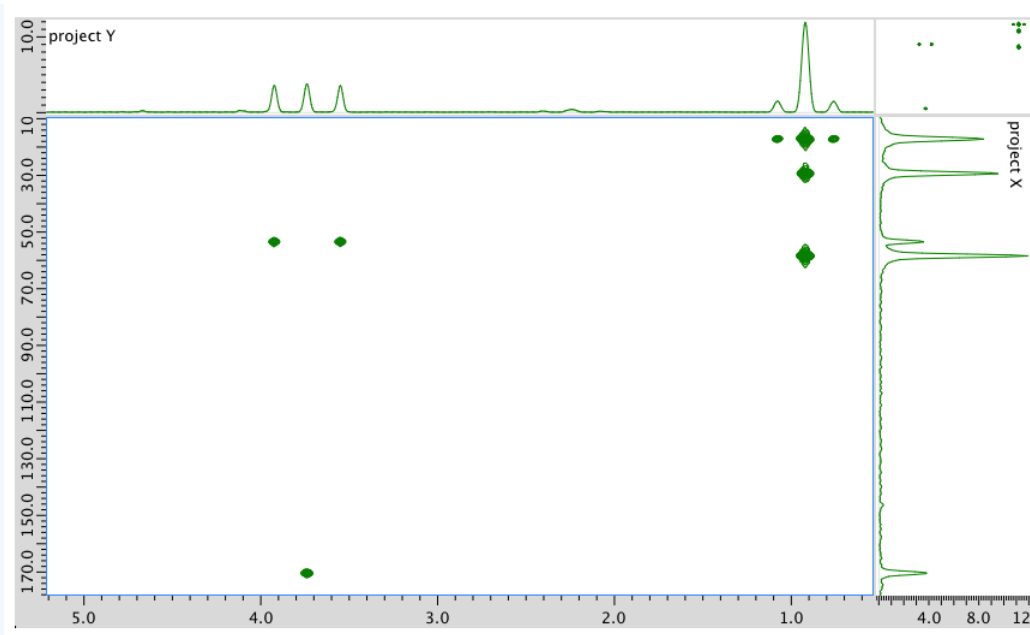
<sup>13</sup>C NMR:



COSY:



HMBC:



### Answer

$^1\text{H}$  NMR:

Chemical shift (ppm)	Integration	Multiplicity	Partial Structure
4.71	--	singlet	solvent
4.17	1H	doublet?	CO-CH-N
3.75	3H	singlet	O-CH <sub>3</sub>
2.25	1H	multiplet	CH-CH-(CH <sub>3</sub> ) <sub>2</sub>
0.92	6H	triplet?	2 x CH <sub>3</sub>

$^{13}\text{C}$  NMR:

Chemical shift (ppm)	Type of carbon
170	sp <sup>2</sup> C=O
60	sp <sup>3</sup> C-N
52	sp <sup>3</sup> C-O
30	sp <sup>3</sup> C
19	sp <sup>3</sup> C

COSY:

Assignment	$^1\text{H}$	COSY
A	4.17	2.25
B	3.75	--
C	2.25	4.17
D	0.92	2.25

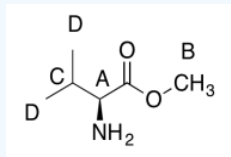
Formula:

$C_6H_{13}O_2N$  (1 O indicated from shift in  $^{13}C$ ,  $^1H$  NMR)

$FW = (6 \times 120) + (13 \times 1) + (2 \times 16) + (1 \times 14) = 131$

Degrees of unsaturation =  $\frac{(2 \times 6) + 2 + 1 - 13}{2} = 1$  unit (1 double bond)

The data should be consistent with this structure:

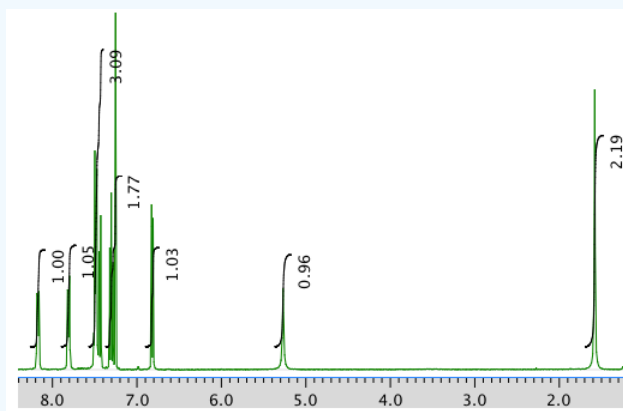


### Exercise 5.6.5

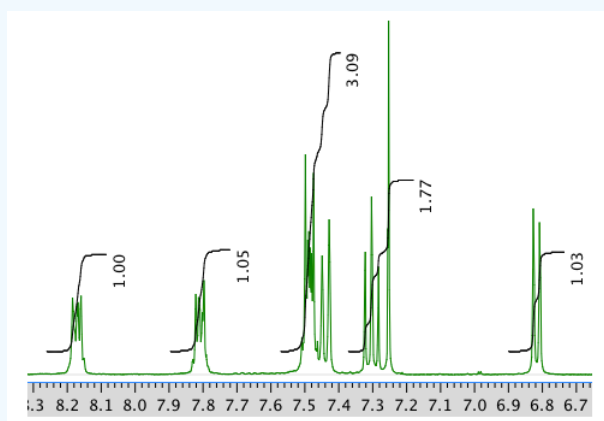
Present an analysis of the following data and propose a structure.

MW: 144 amu

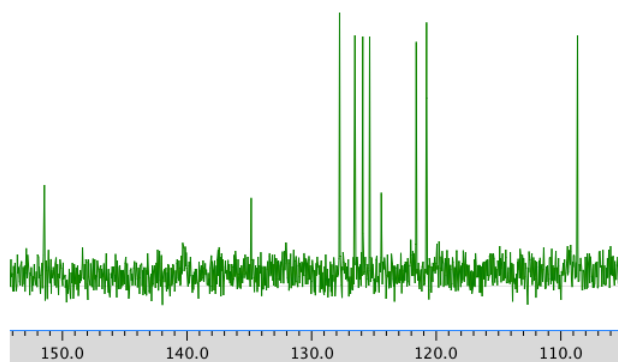
The  $^1H$  NMR spectrum in  $CDCl_3$ :



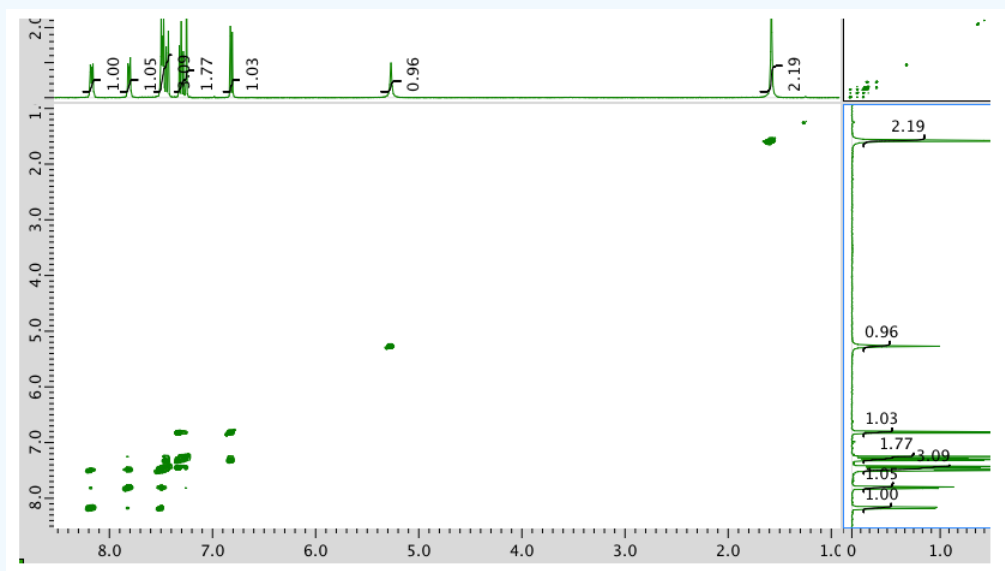
An expansion of the  $^1H$  NMR spectrum:



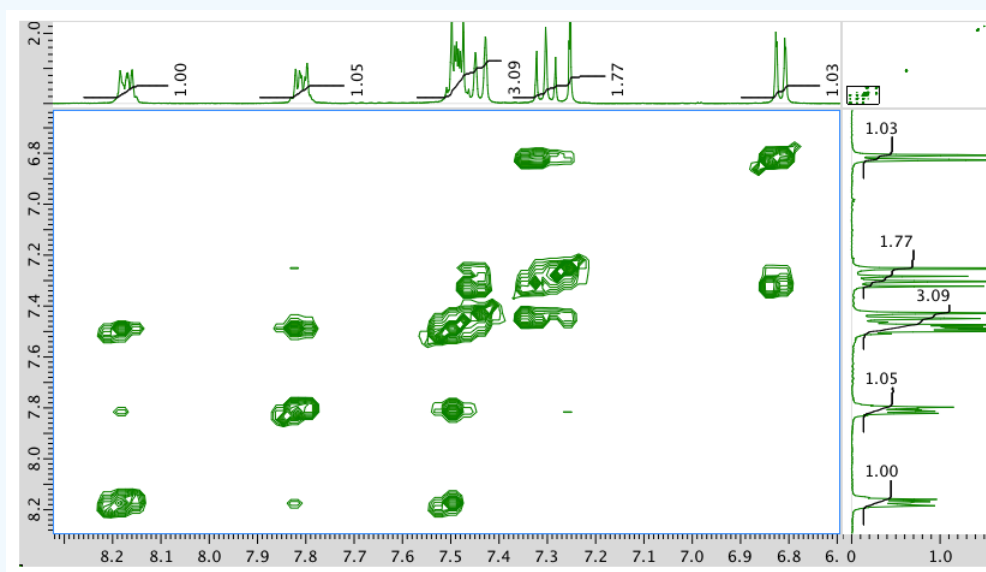
$^{13}C$  NMR spectrum:



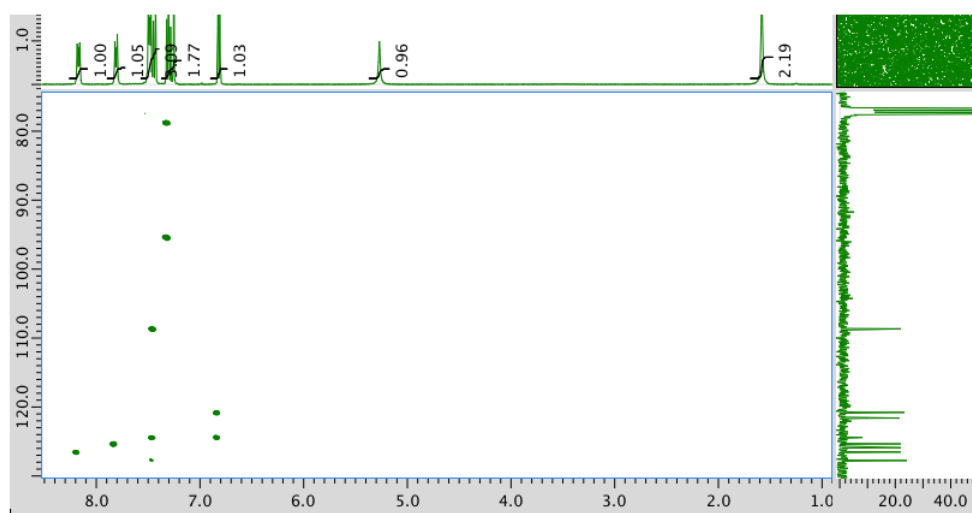
COSY:



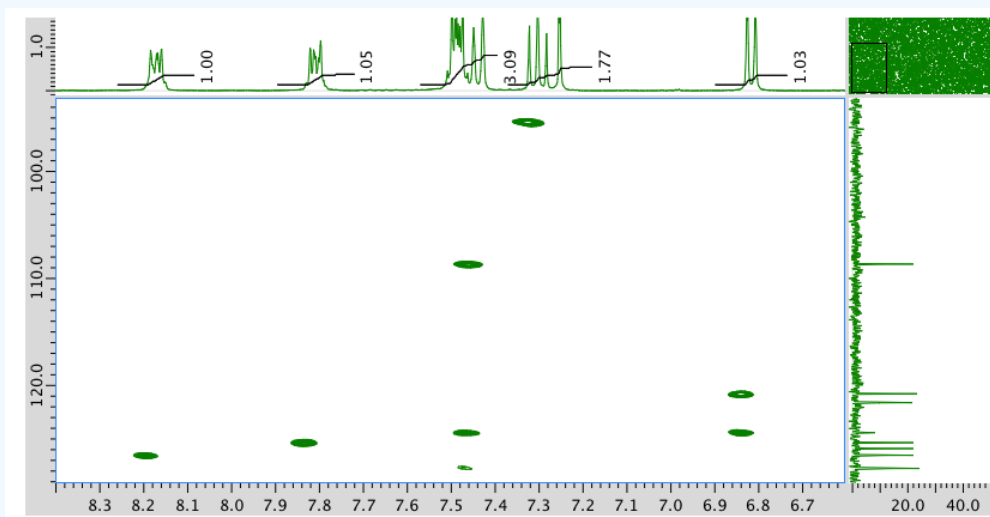
An expansion of the COSY spectrum:



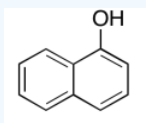
HMBC:



An expansion of the HMBC spectrum:



**Answer**

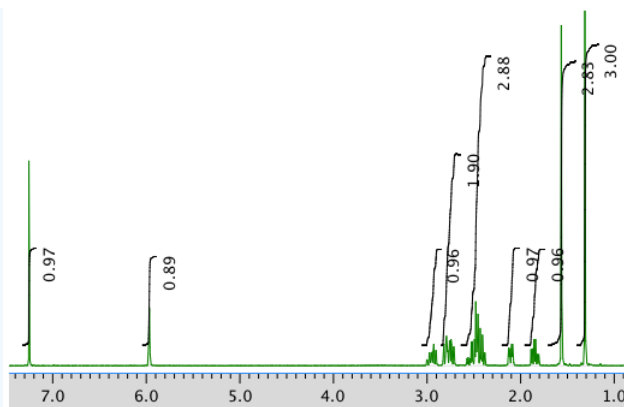


#### Exercise 5.6.6

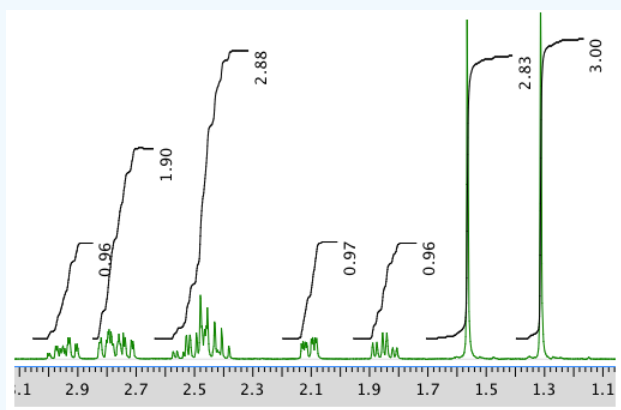
Present an analysis of the following data and propose a structure.

MW: 164 amu

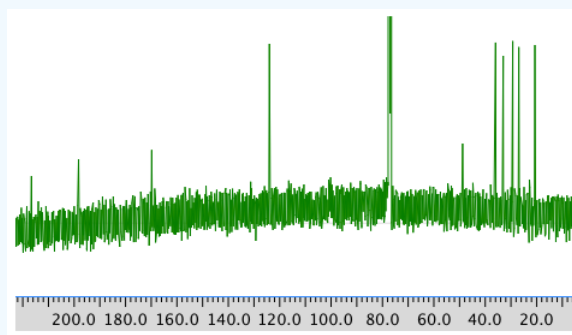
The full  $^1\text{H}$  NMR spectrum in  $\text{CDCl}_3$ :



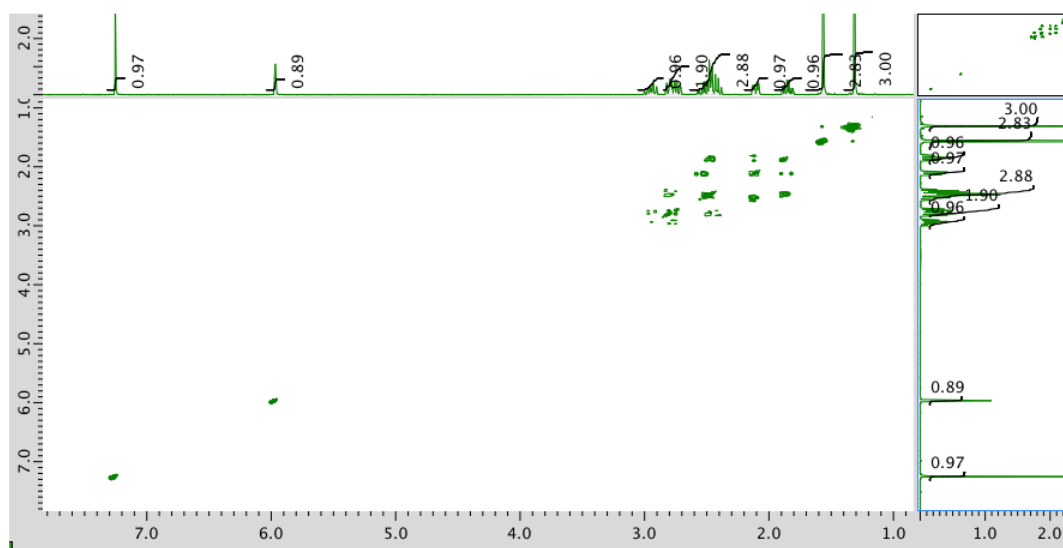
AN expansion:



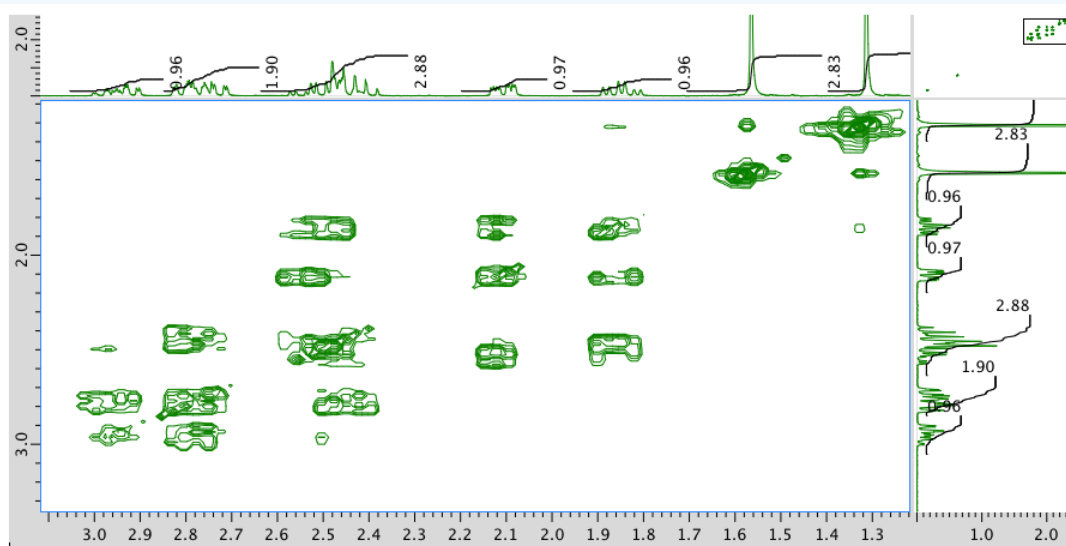
<sup>13</sup>C NMR:



COSY:

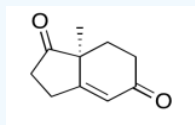


Expansion of COSY spectrum:



### Answer

The data should be consistent with this structure:



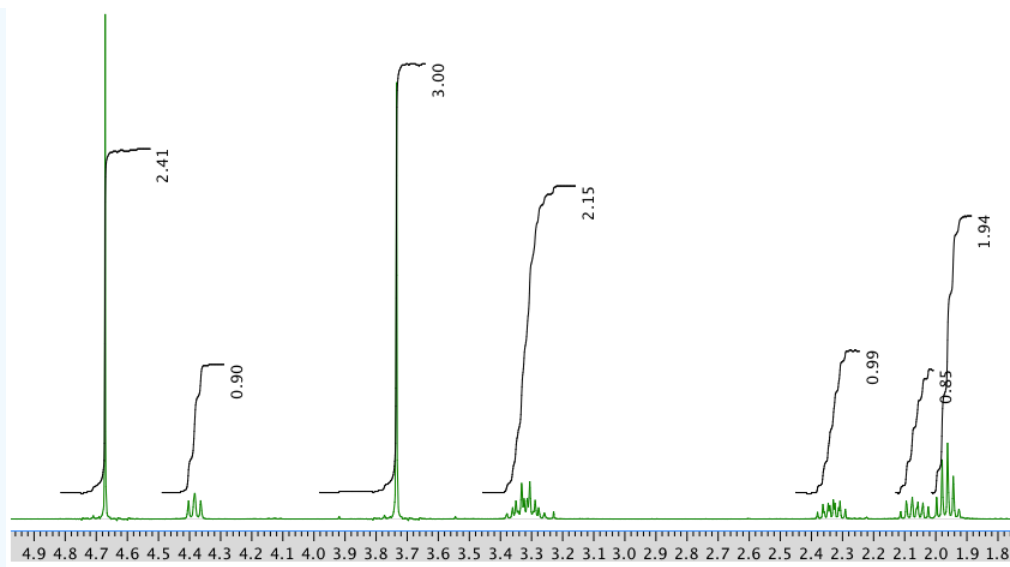
### Exercise 5.6.7

Present an analysis of the following data and propose a structure.

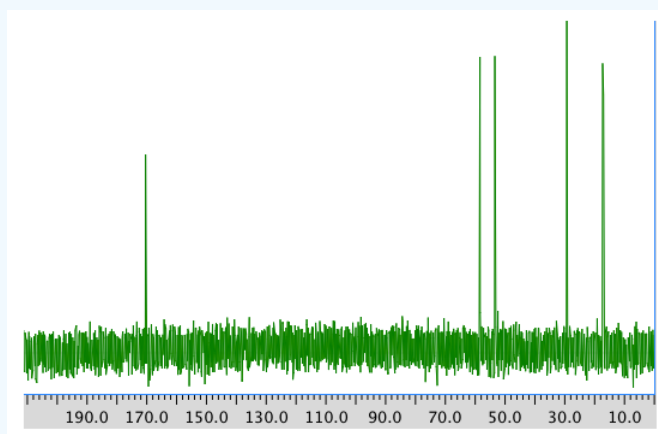
MW: 129 amu

The full  $^1\text{H}$  NMR spectrum in  $\text{D}_2\text{O}$ :

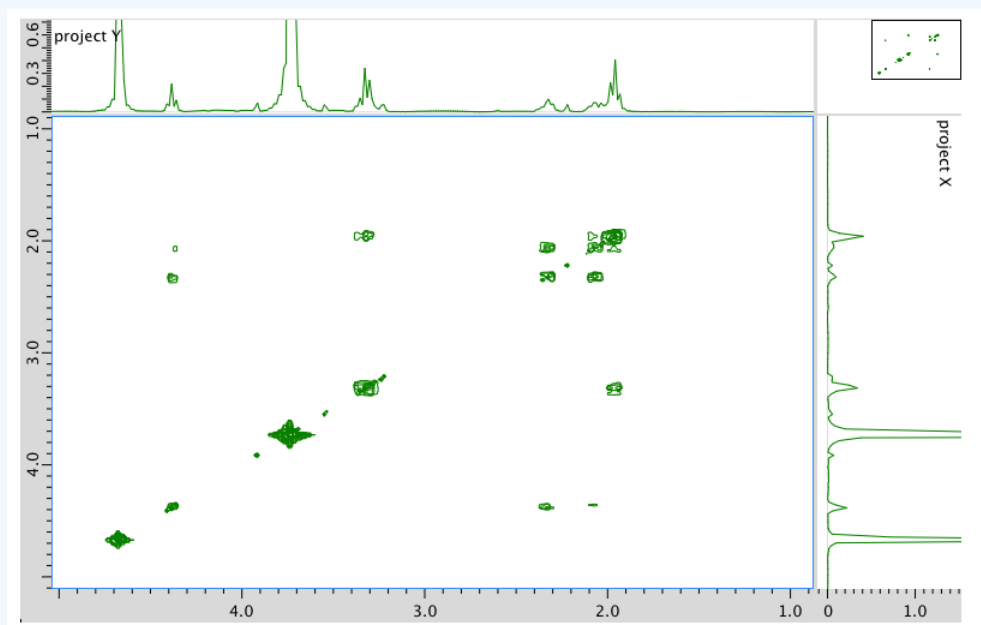




<sup>13</sup>C NMR spectrum:

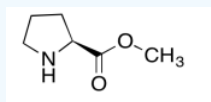


COSY spectrum:



## Answer

The data should be consistent with this structure:

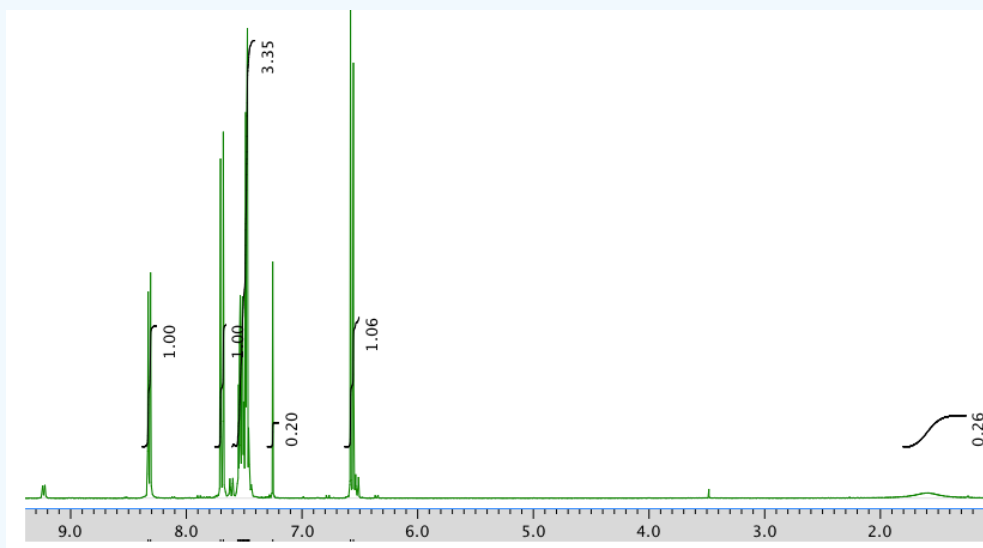


## Exercise 5.6.8

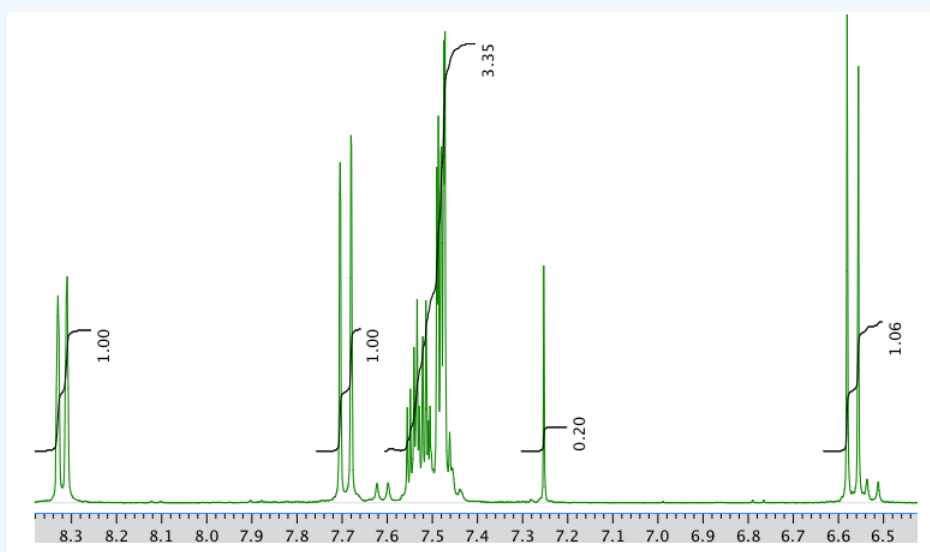
Present an analysis of the following data and propose a structure.

MW: 173 amu

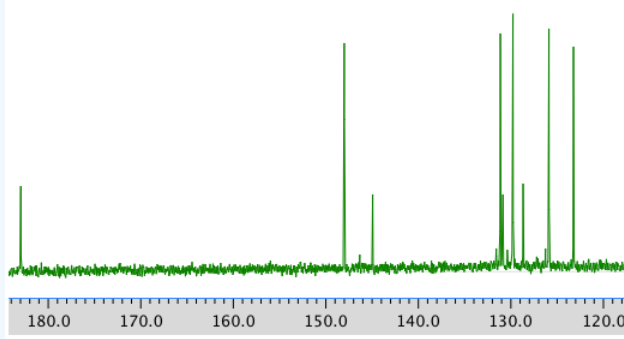
The full  $^1\text{H}$  NMR spectrum in  $\text{CDCl}_3$ :



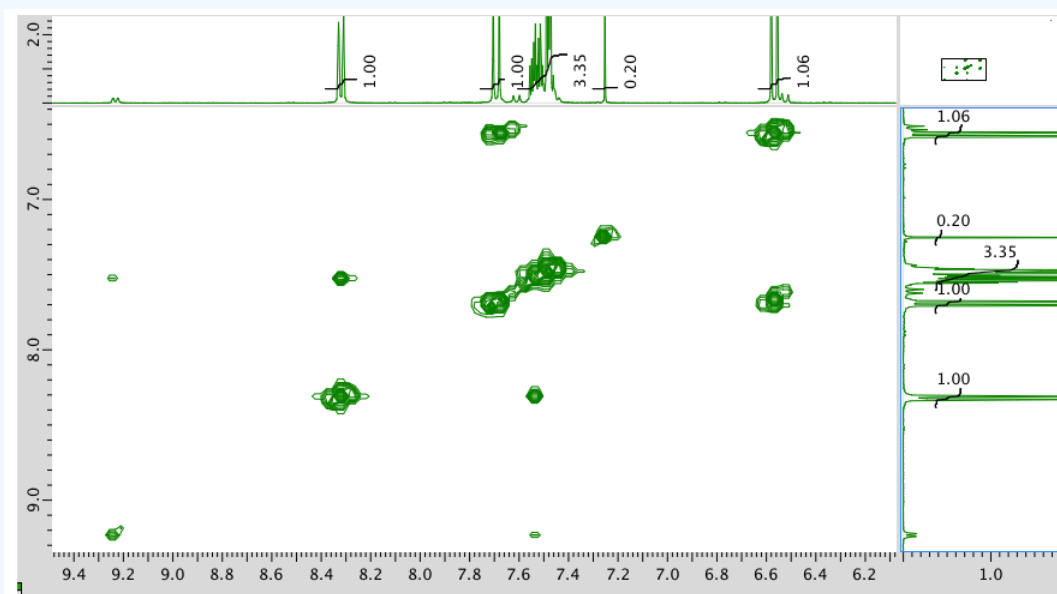
An expansion of the  $^1\text{H}$  NMR spectrum:



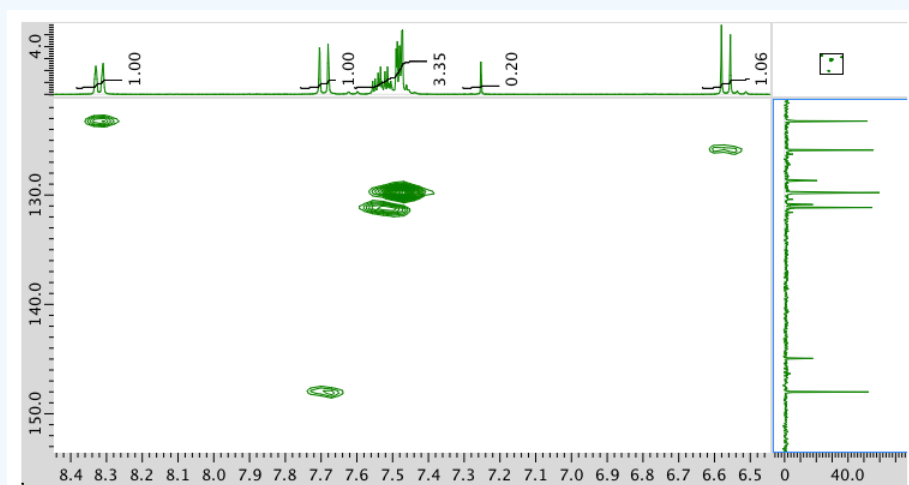
$^{13}\text{C}$  NMR spectrum:



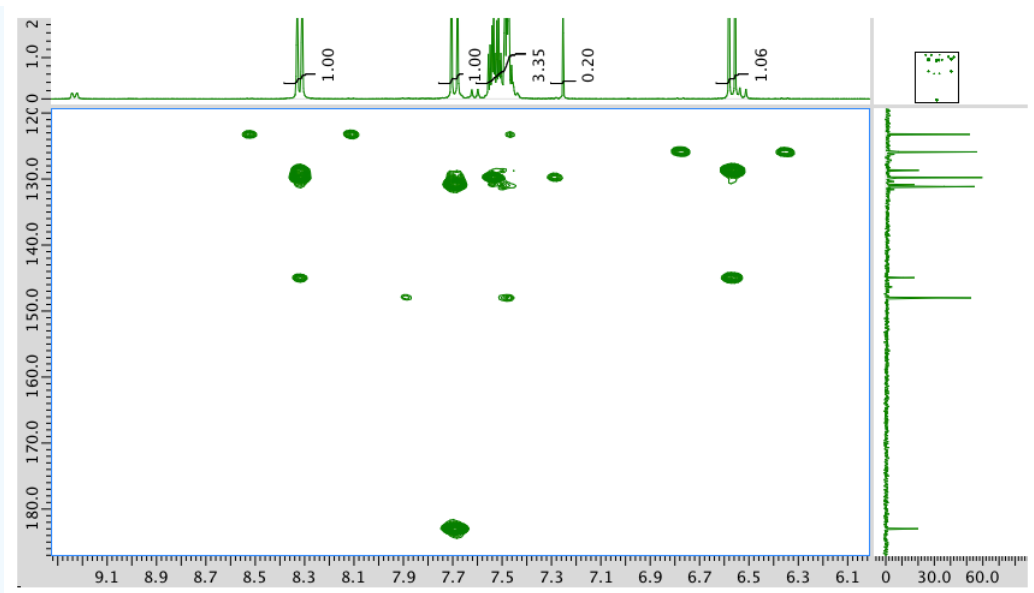
COSY:



HMQC:

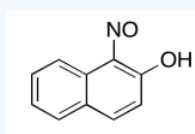


HMBC:



### Answer

The data should be consistent with this structure:

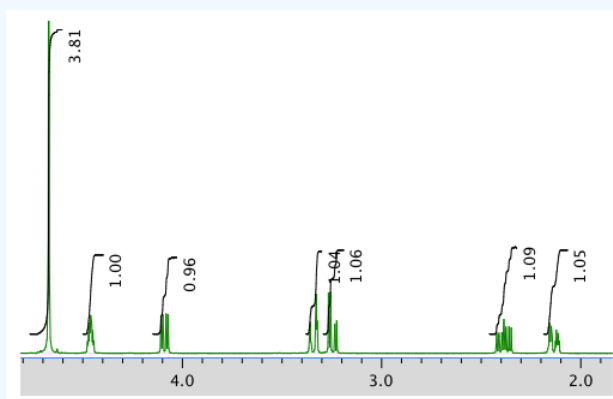


### Exercise 5.6.9

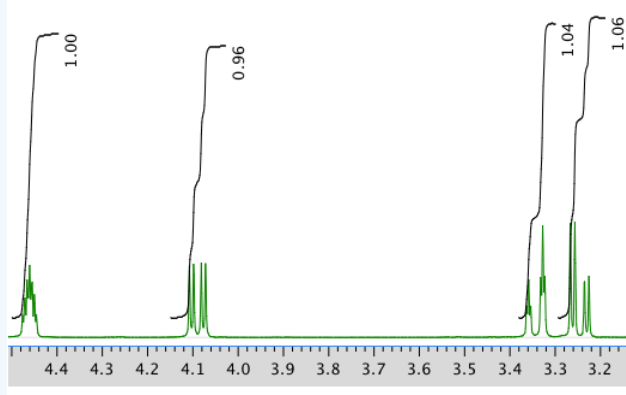
Present an analysis of the following data and propose a structure.

MW: 145 amu

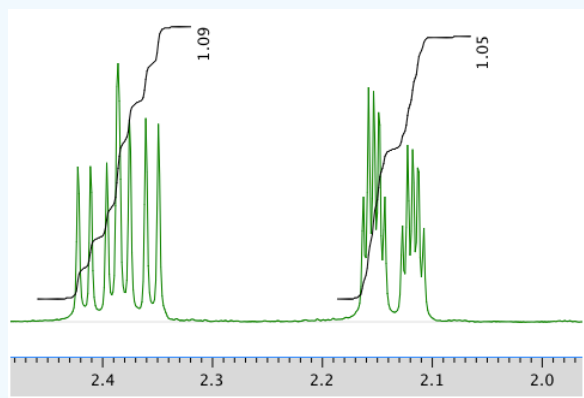
The full  $^1\text{H}$  NMR spectrum in  $\text{D}_2\text{O}$ :



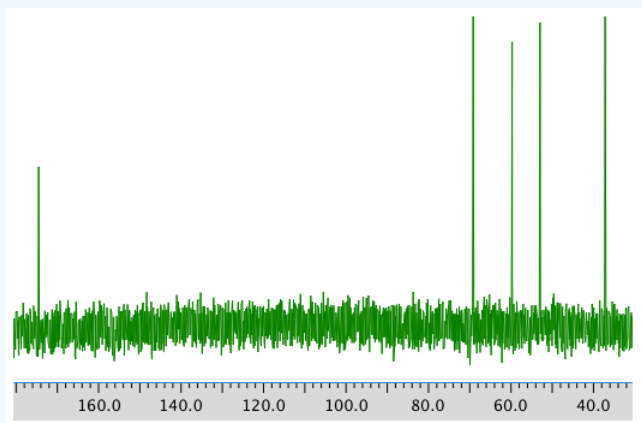
An expanded view of  $^1\text{H}$  NMR spectrum:



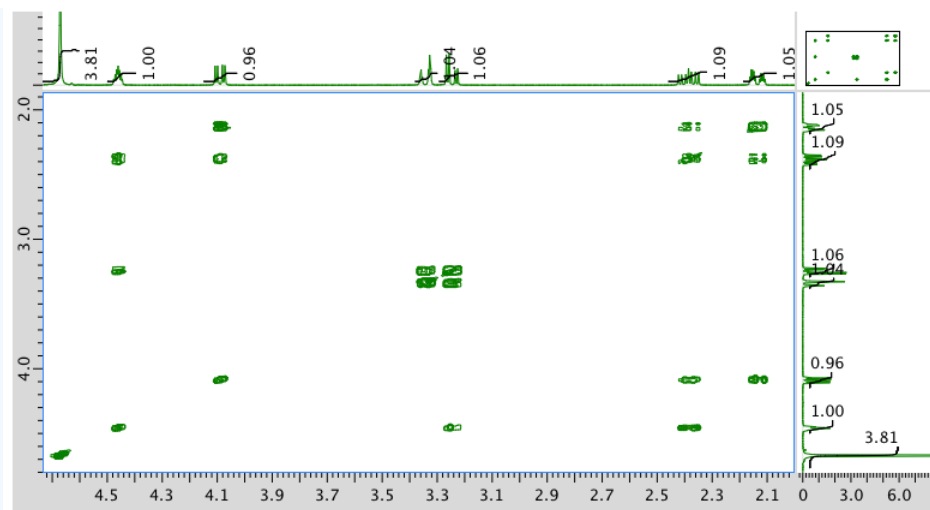
Another expanded view:



$^{13}\text{C}$  NMR spectrum:

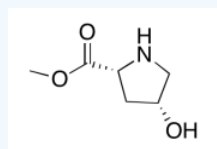


COSY spectrum:



### Answer

The data should be consistent with this structure:

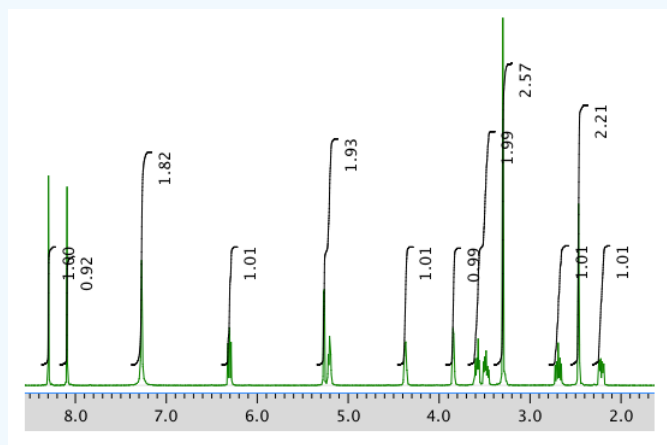


### Exercise 5.6.10

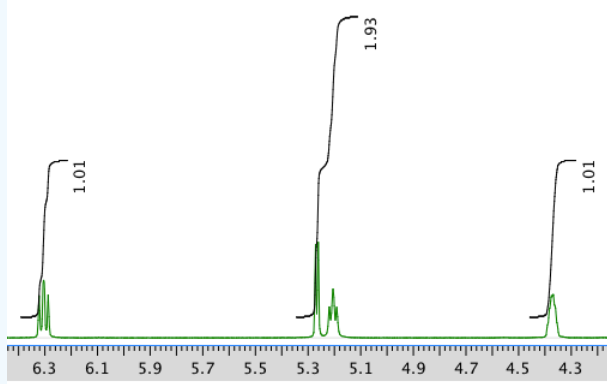
Present an analysis of the following data and propose a structure.

MW: 251 amu

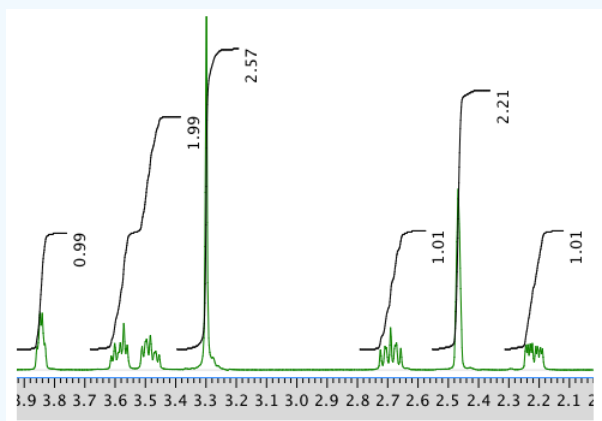
The full  $^1\text{H}$  NMR spectrum in  $\text{DMSO}-d_6$ :



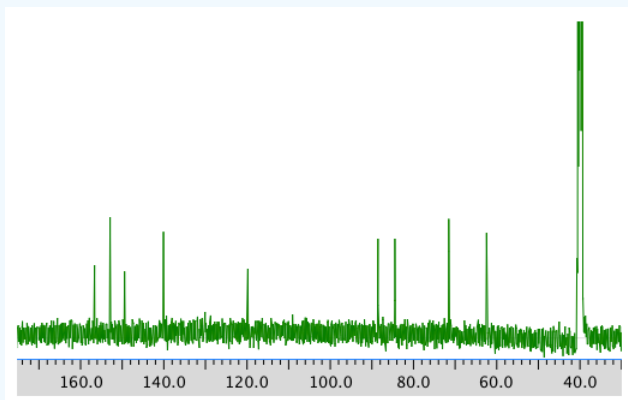
An expanded view of the  $^1\text{H}$  NMR spectrum:



Another expansion:



$^{13}\text{C}$  NMR spectrum:

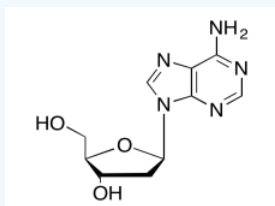


COSY spectrum:





The data should be consistent with this structure:

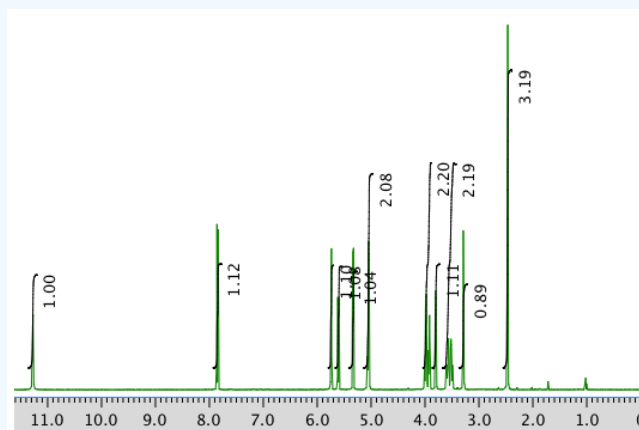


### Exercise 5.6.11

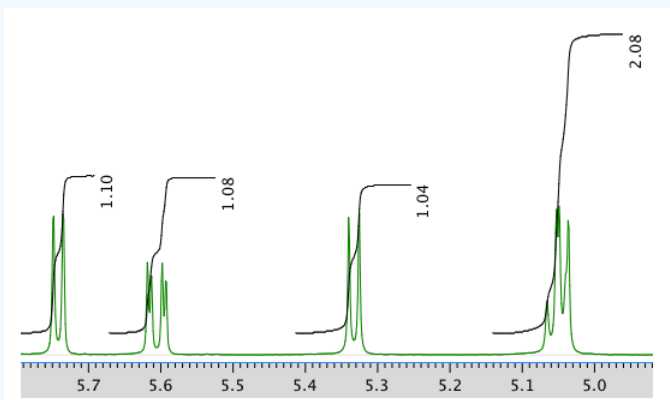
Present an analysis of the following data and propose a structure.

MW: 244 amu

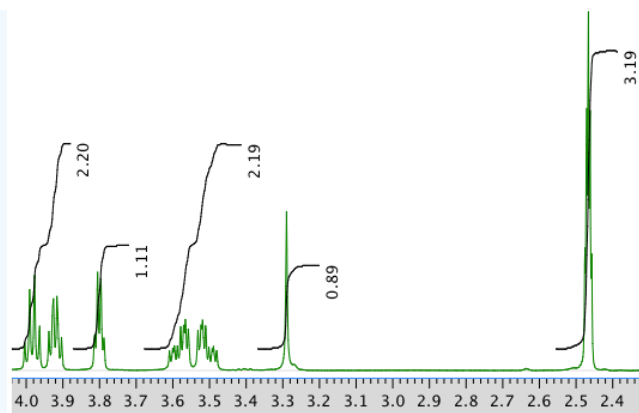
The full  $^1\text{H}$  NMR spectrum in  $\text{DMSO}-d_6$ :



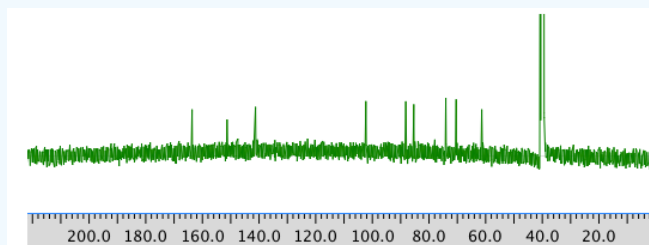
An expanded view of the  $^1\text{H}$  NMR spectrum:



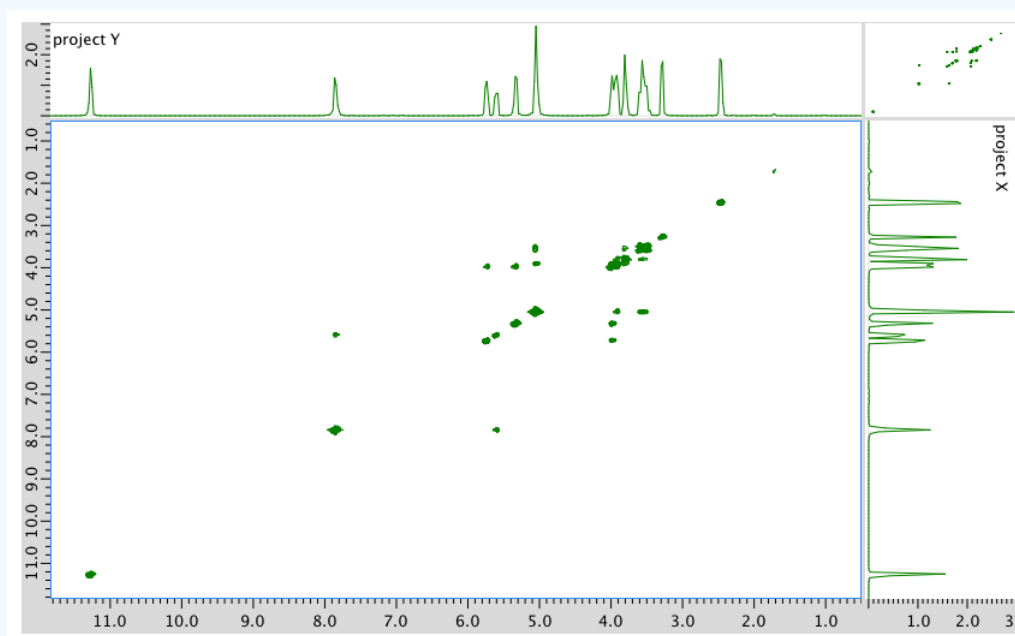
Another expansion of the  $^1\text{H}$  NMR spectrum:



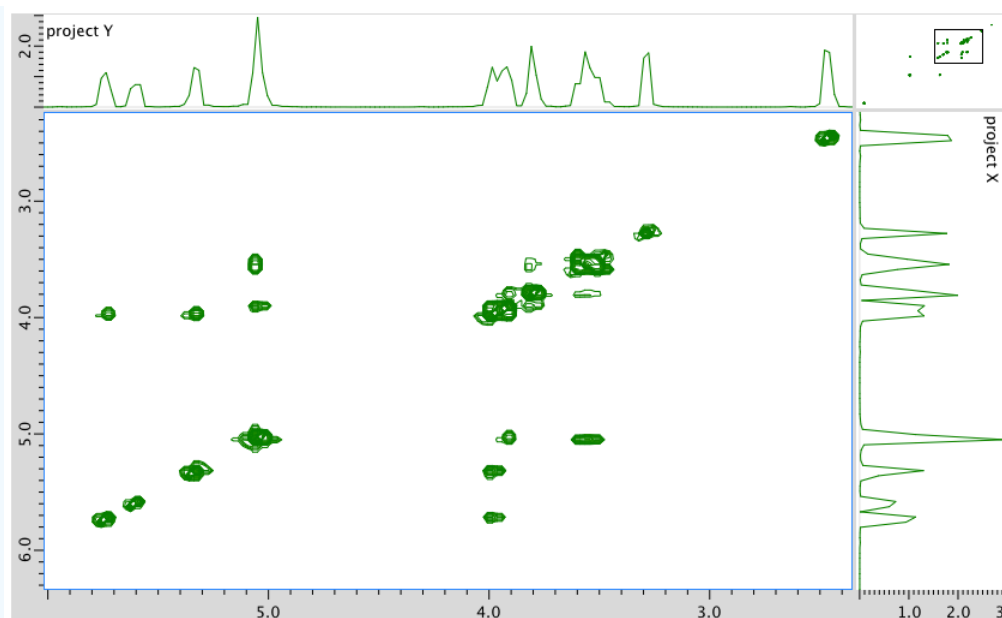
$^{13}\text{C}$  NMR spectrum:



COSY:

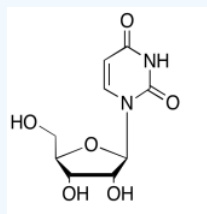


An expanded view of the COSY spectrum:



### Answer

The data should be consistent with this structure:

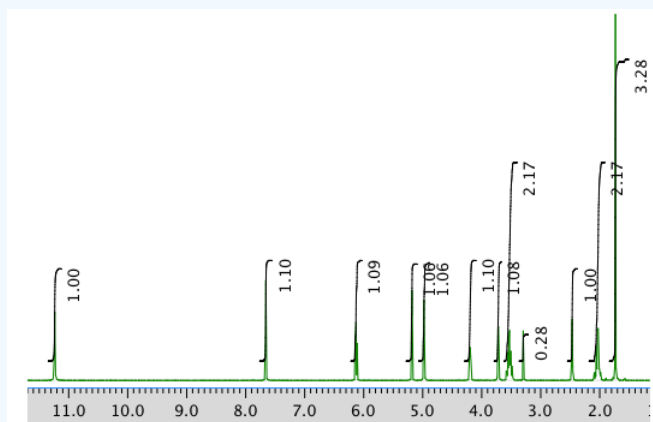


### Exercise 5.6.12

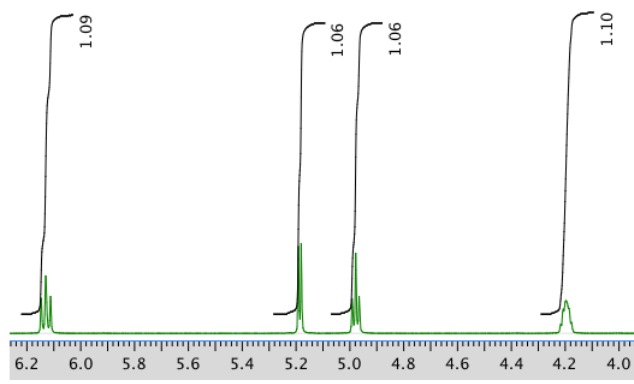
Present an analysis of the following data and propose a structure.

MW: 242 amu

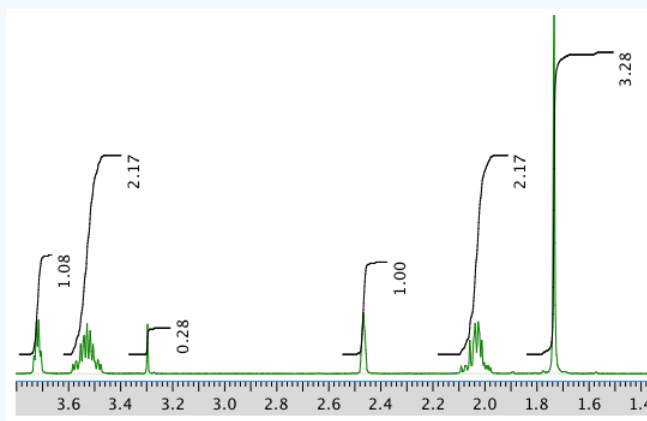
The full  $^1\text{H}$  NMR spectrum in  $\text{DMSO}-d_6$ :



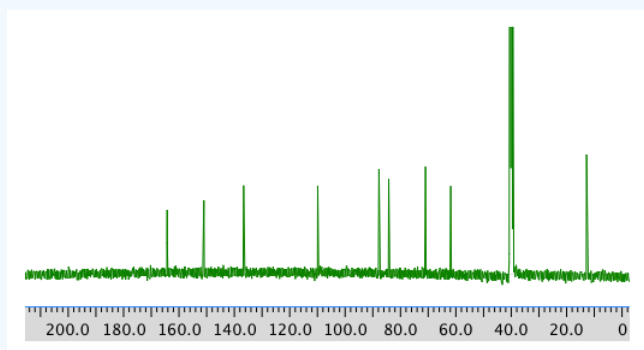
Expansion of the  $^1\text{H}$  NMR spectrum:



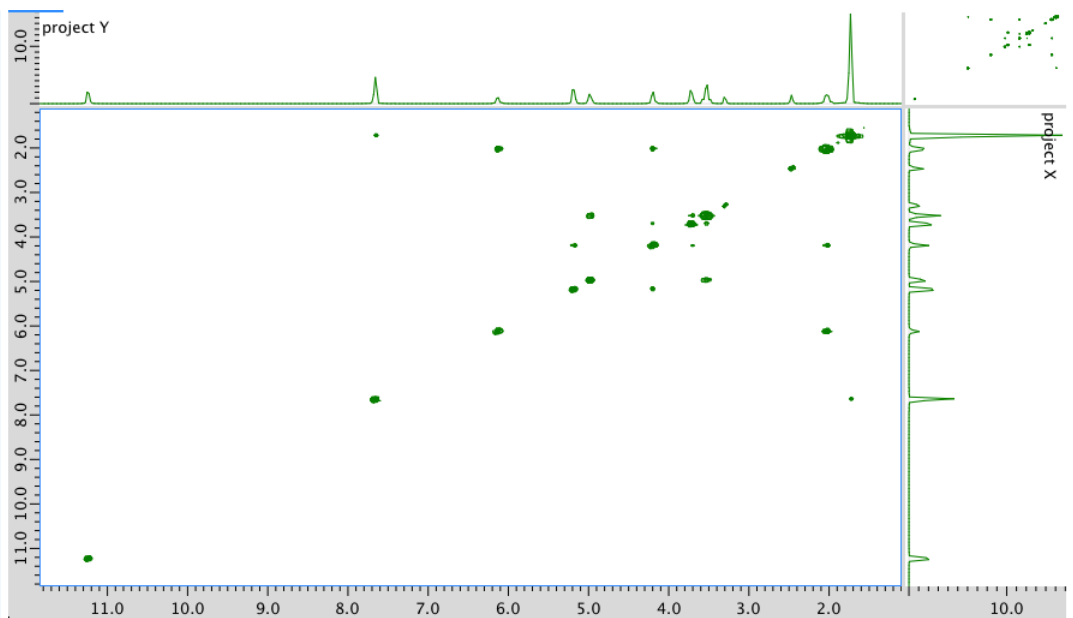
Another expansion:



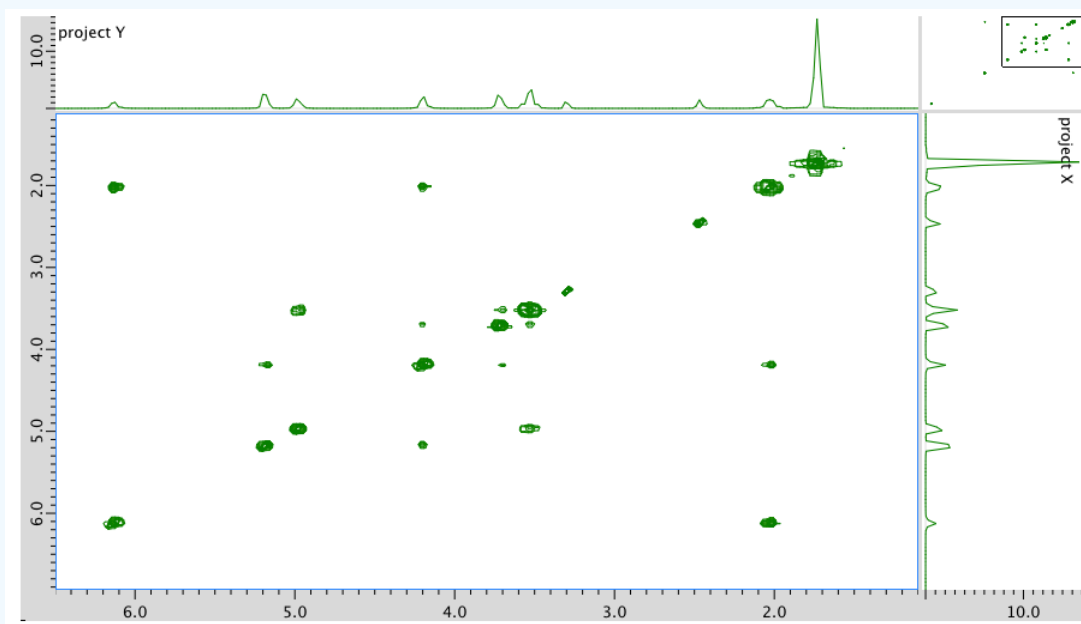
$^{13}\text{C}$  NMR spectrum:



COSY:

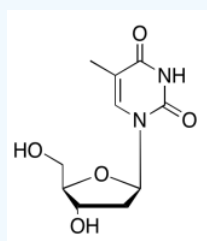


An expansion of the COSY spectrum:



**Answer**

The data should be consistent with this structure:

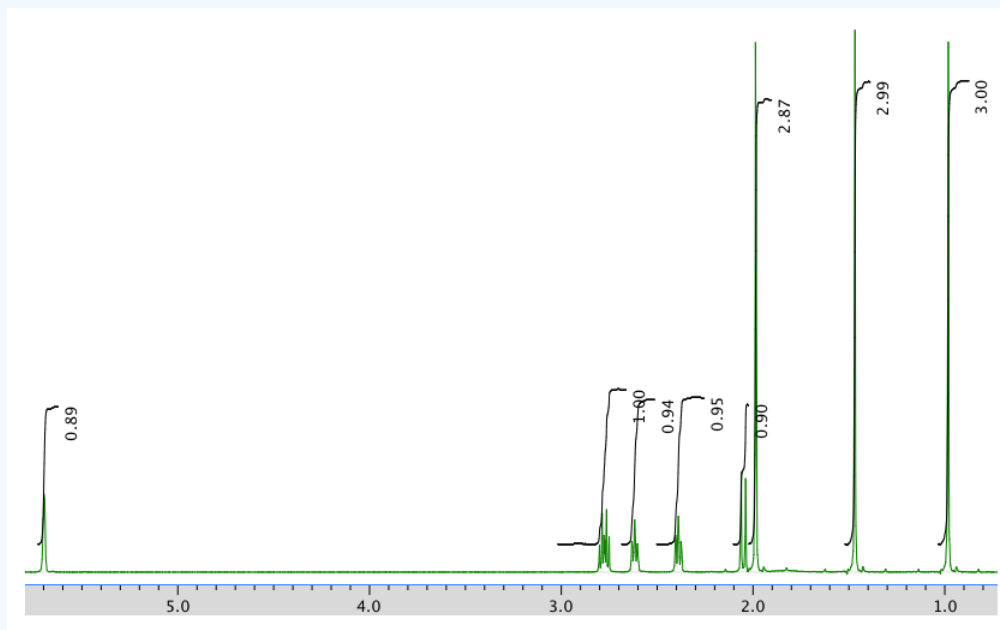


### Exercise 5.6.13

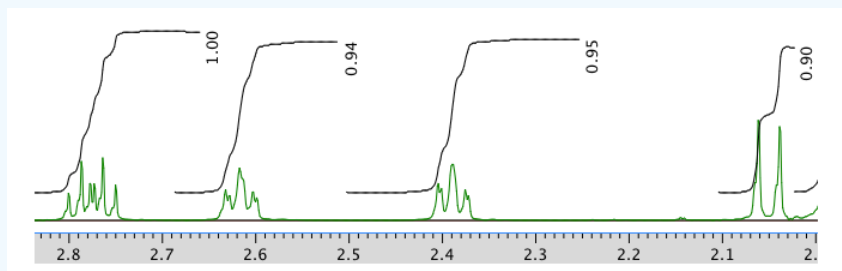
Present an analysis of the following data and propose a structure.

MW: 152 amu

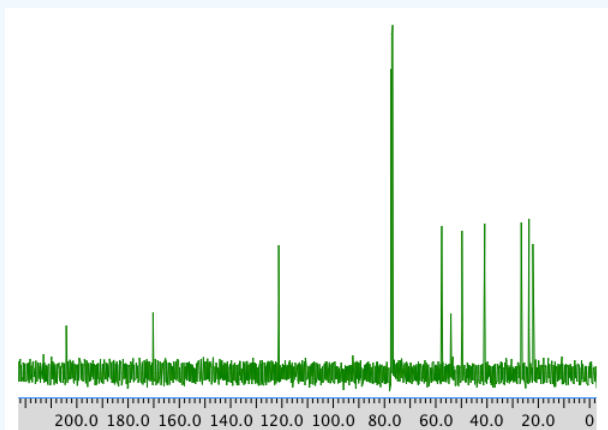
The full  $^1\text{H}$  NMR spectrum in  $\text{CDCl}_3$ :



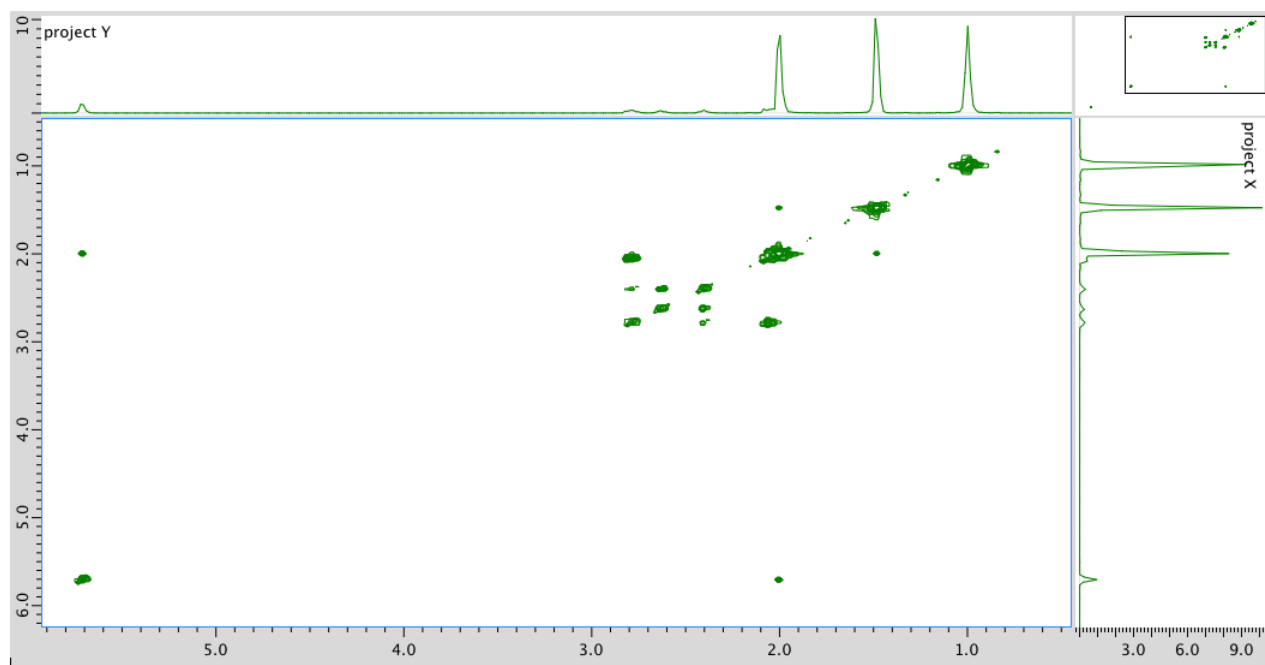
An expansion of the  $^1\text{H}$  NMR spectrum:



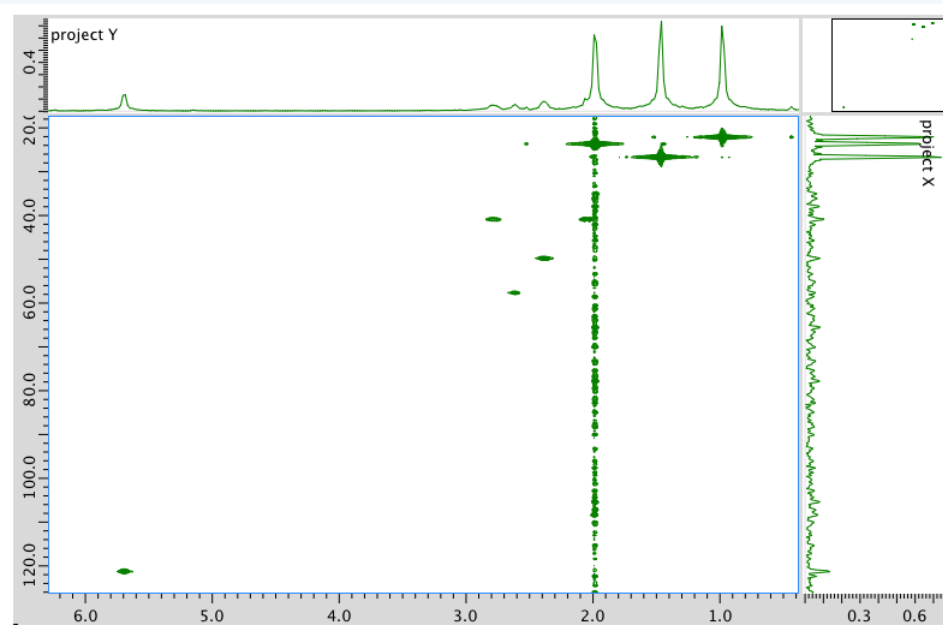
$^{13}\text{C}$  NMR spectrum in  $\text{CDCl}_3$ :



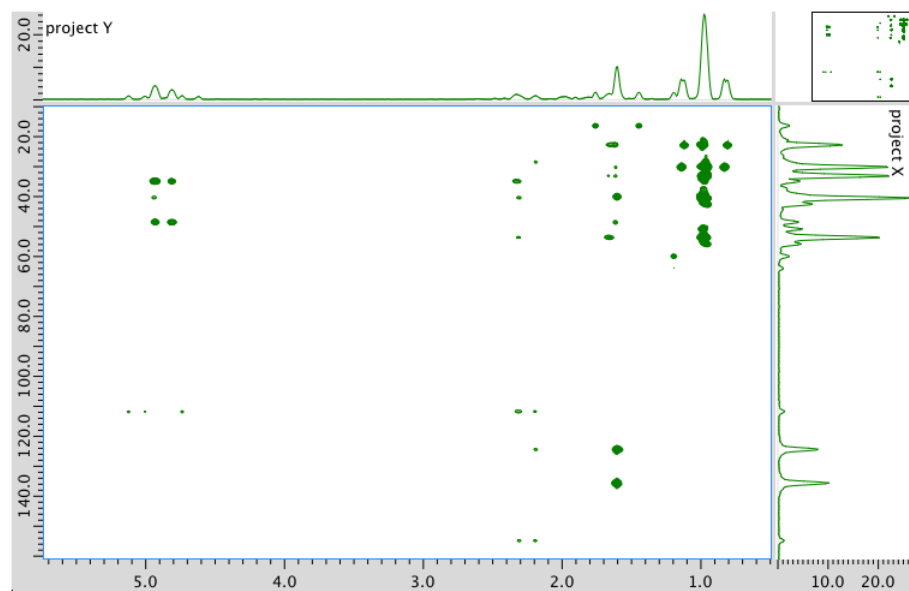
COSY spectrum:



HMOC spectrum:



HMBC spectrum:



### Answer

$^1\text{H}$  NMR:

Chemical shift (ppm)	Integration	Multiplicity	Partial structure
5.7	1H	singlet	$\text{C}=\text{CH}-\text{CO}$
2.77	1H	multiplet	$\text{CH}_2-\text{CH}-\text{CO}$
2.62	1H	multiplet	$\text{C}-\text{CH}-\text{C}$
2.39	1H	multiplet	$\text{C}-\text{CH}-\text{C}$
2.05	1H	doublet?	$\text{C}-\text{CH}-\text{CH}?$
1.96	3H	singlet	$\text{C}-\text{CH}_3$
1.48	3H	singlet	$\text{C}-\text{CH}_3$
0.98	3H	singlet	$\text{C}-\text{CH}_3$

$^{13}\text{C}$  NMR:

Chemical shift (ppm)	Type of carbon
204	$\text{sp}^2 \text{ C}=\text{O}$
170	$\text{sp}^2$
121	$\text{sp}^2$
59	$\text{sp}^3$
55	$\text{sp}^3$
50	$\text{sp}^3$
41	$\text{sp}^3$
28	$\text{sp}^3$
24	$\text{sp}^3$



22

$sp^3$

COSY:

Assignment	$^1\text{H}$	COSY
1	2.39	2.77, 2.62?
3	5.7	2.05?
5	2.62	2.62?
7a	2.77	2.05
7b	2.05	2.77
8	1.48	--
9	0.98	--
10	1.96	--

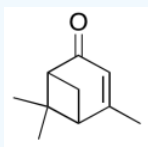
Formula:

$\text{C}_{10}\text{H}_{14}\text{O}$  (1 O indicated from shift in  $^{13}\text{C}$ ,  $^1\text{H}$  NMR)

$\text{FW} = (10 \times 12) + (14 \times 1) + (1 \times 16) = 150$

Degrees of unsaturation =  $\frac{(2 \times 10) + 2 - 14}{2} = 4$  units (e.g. 2 rings, 2 double bonds)

The data should be consistent with this structure:

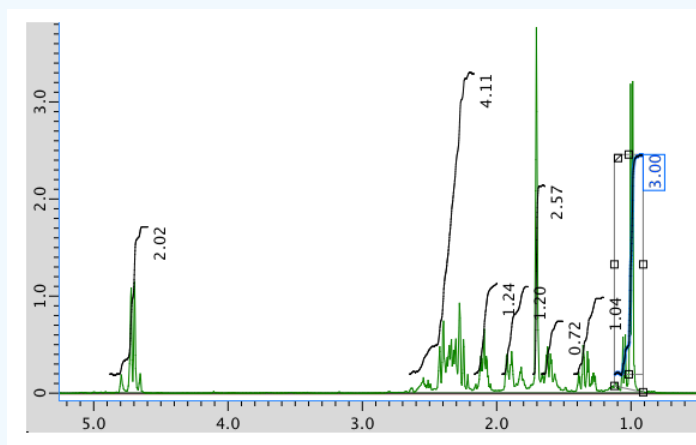


### Exercise 5.6.14

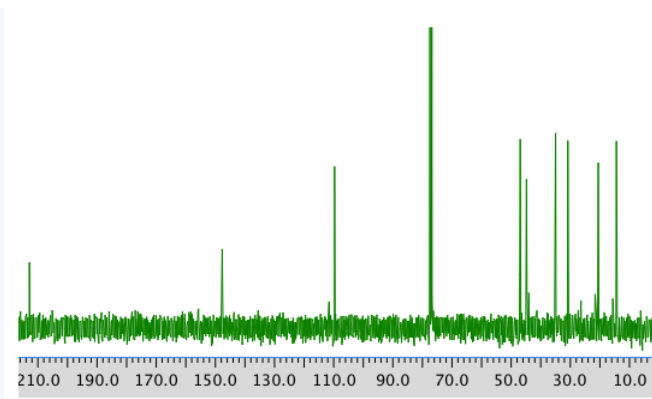
Present an analysis of the following data and propose a structure.

MW: 152 amu

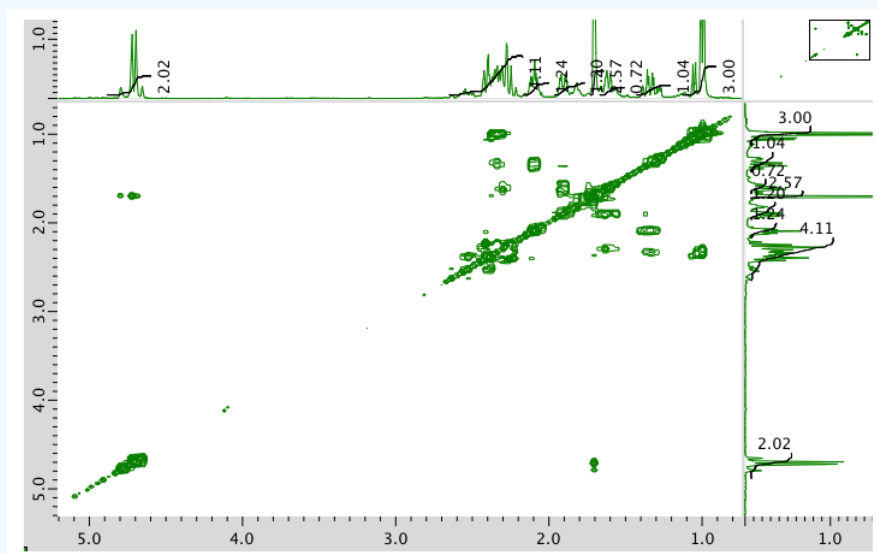
The full  $^1\text{H}$  NMR spectrum in  $\text{CDCl}_3$ :



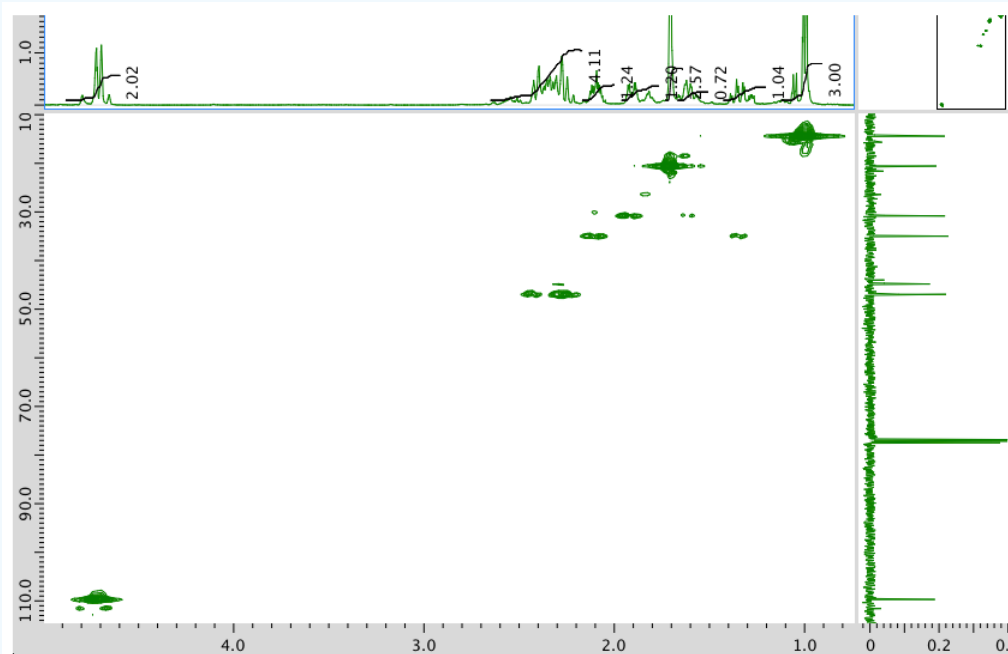
$^{13}\text{C}$  NMR spectrum:



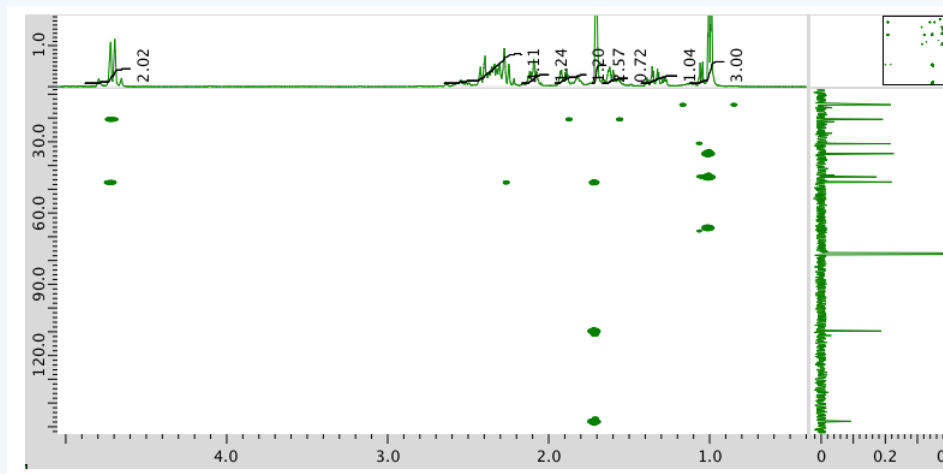
COSY spectrum:



HMQC spectrum:

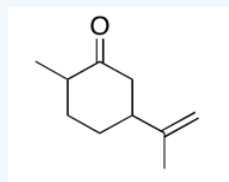


HMBC spectrum:



**Answer**

The data should be consistent with this structure:

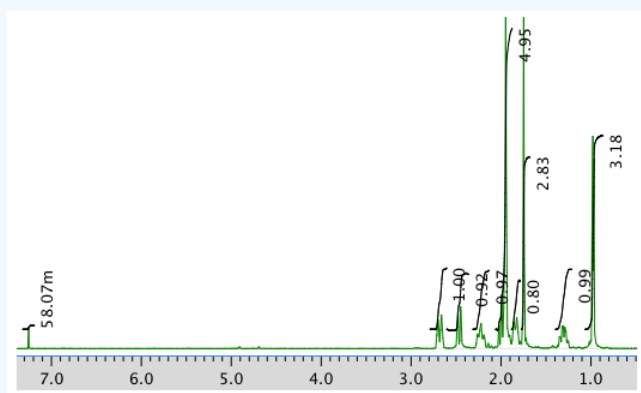


### Exercise 5.6.15

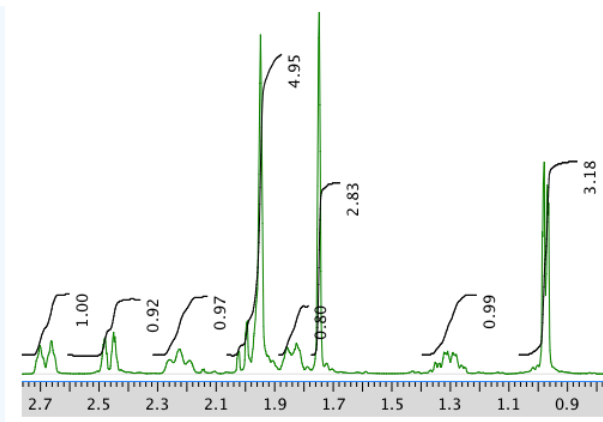
Present an analysis of the following data and propose a structure.

MW: 150 amu

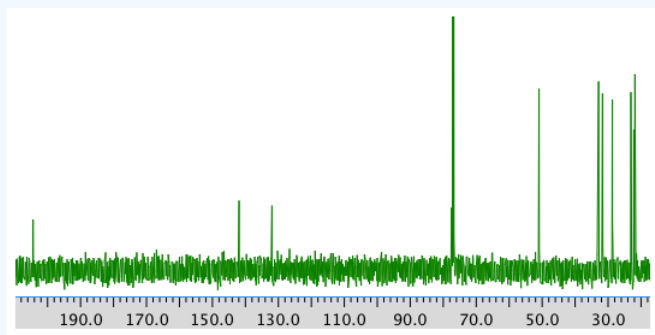
The full  $^1\text{H}$  NMR spectrum in  $\text{CDCl}_3$ :



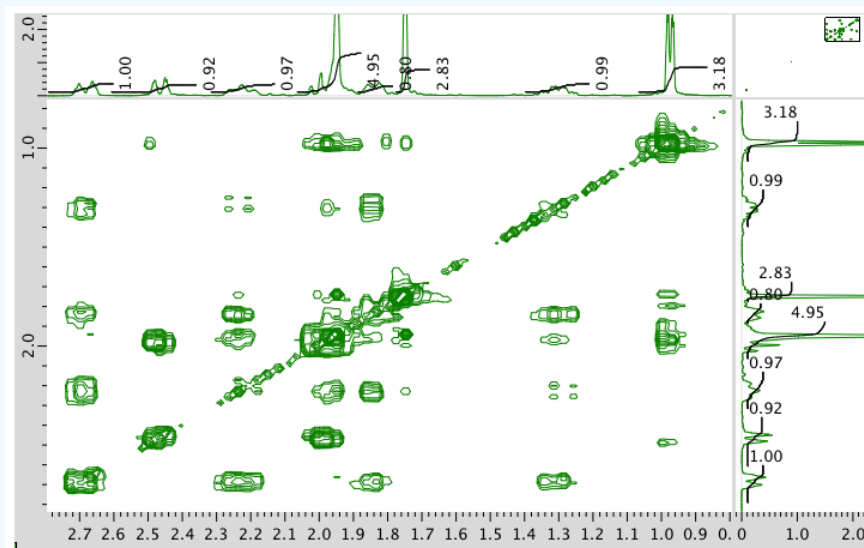
An expansion of the  $^1\text{H}$  NMR spectrum:



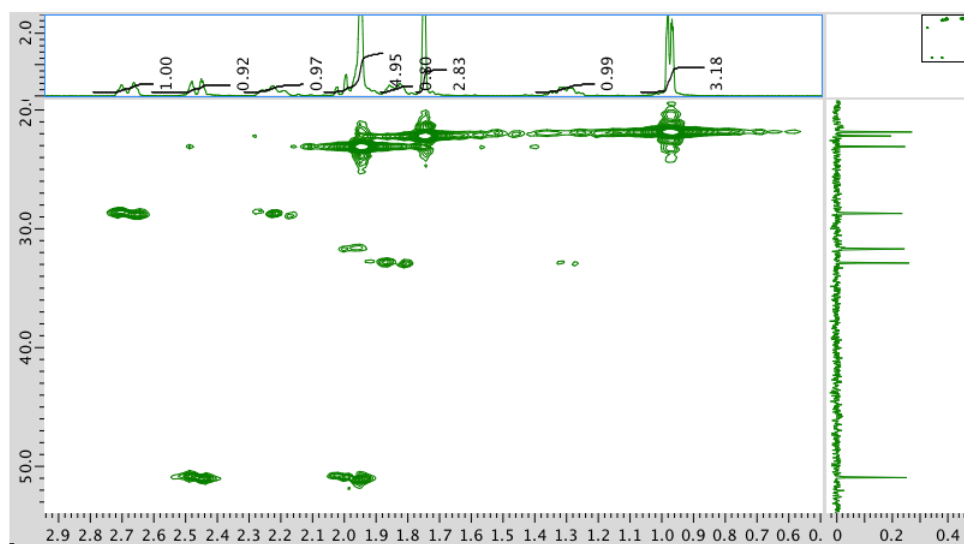
<sup>13</sup>C NMR spectrum:



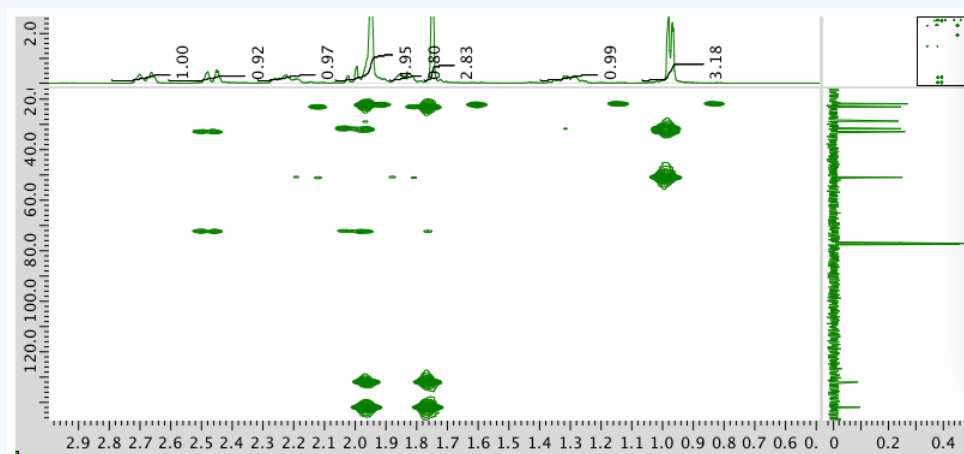
COSY spectrum:



HMQC spectrum:

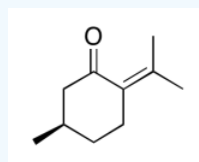


HMBC spectrum:



### Answer

The data should be consistent with this structure:



NMR spectra obtained on a JEOL 400 MHz NMR spectrometer.

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