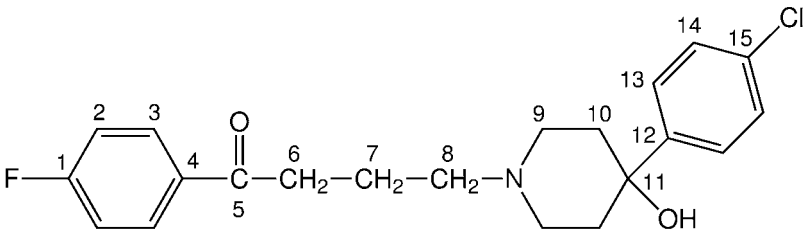
Problem 66

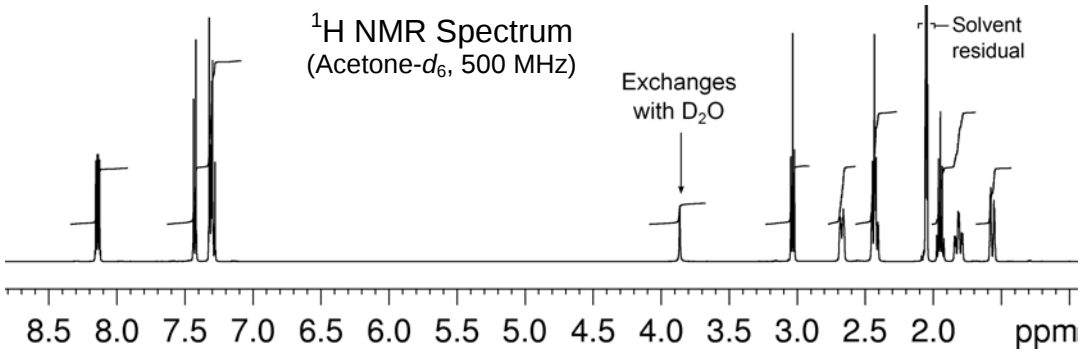
The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of haloperidol ($C_{21}H_{23}ClFNO_2$) recorded in acetone-d_6 solution at 298 K and 500 MHz are given below.

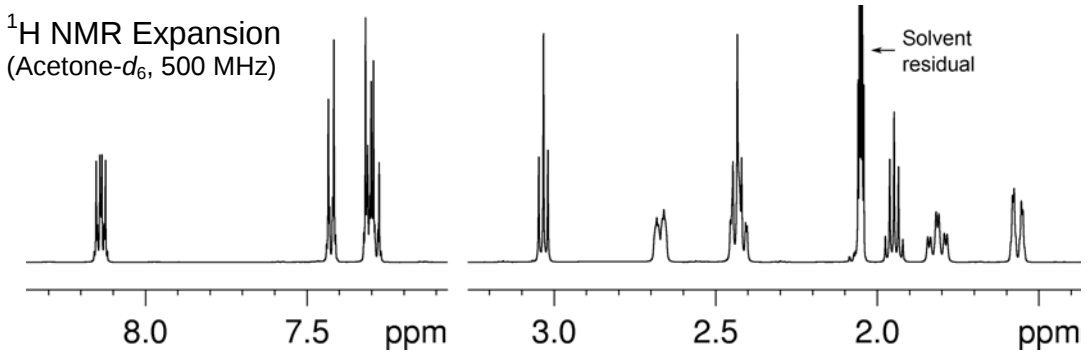
The ^{1}H NMR spectrum has signals at δ 1.57, 1.81, 1.95, 2.43, 2.44, 2.67, 3.03, 3.86, 7.29, 7.31, 7.43 and 8.14 ppm.

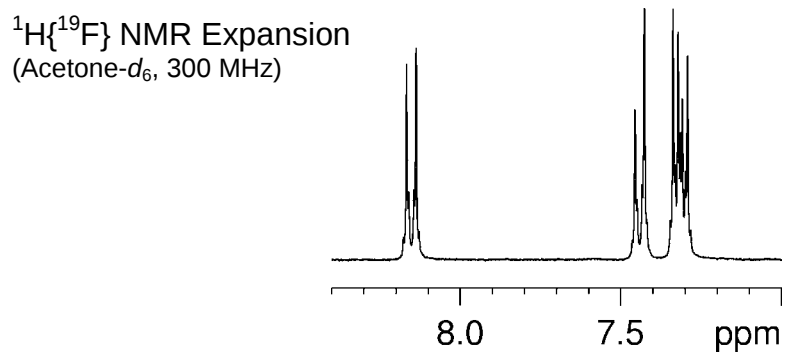
The $^{13}C\{^{1}H\}$ NMR spectrum has signals at δ 22.3, 35.6, 38.4, 49.2, 57.6, 70.2, 115.3, 126.6, 127.8, 130.8, 131.4, 134.5, 149.1, 165.4 and 197.8 ppm.

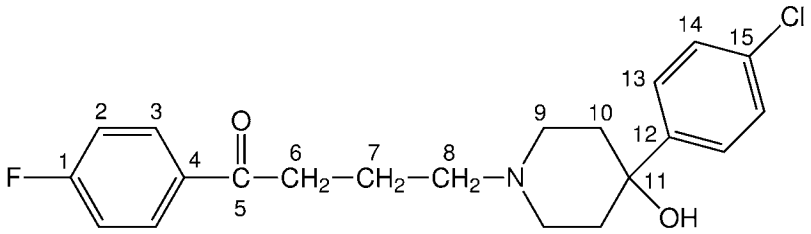
Use the spectra below to assign each proton and carbon resonance.

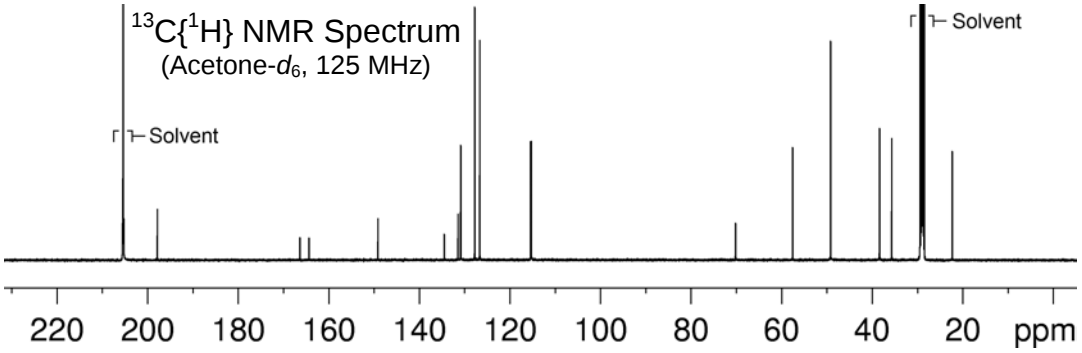


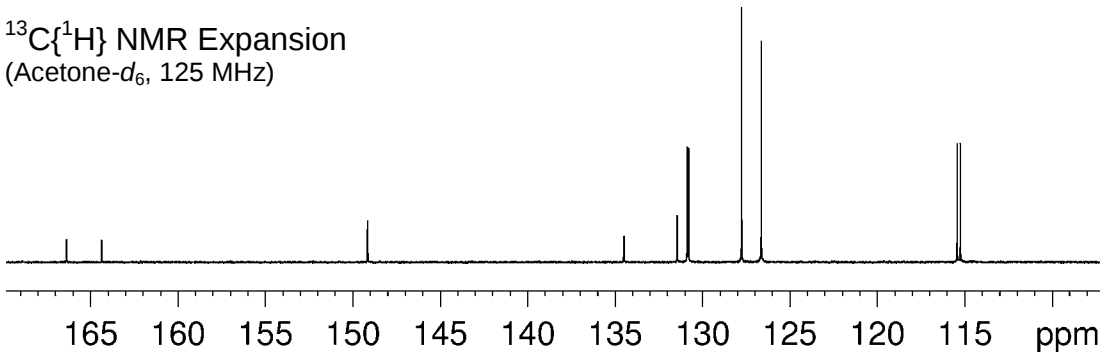


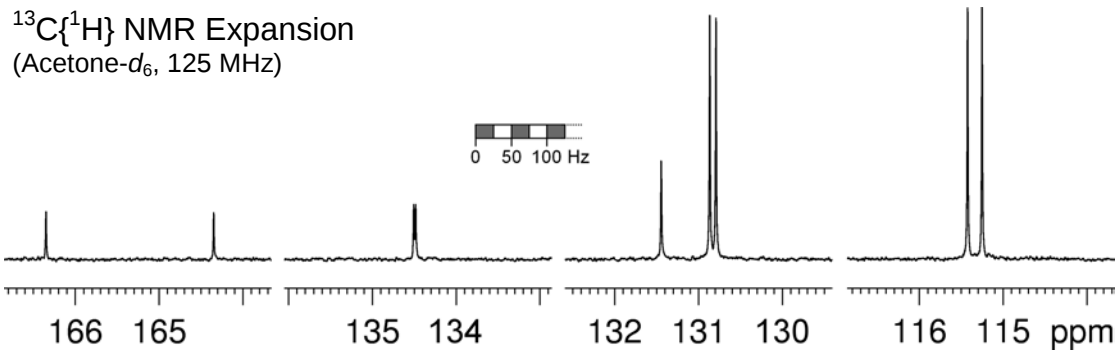


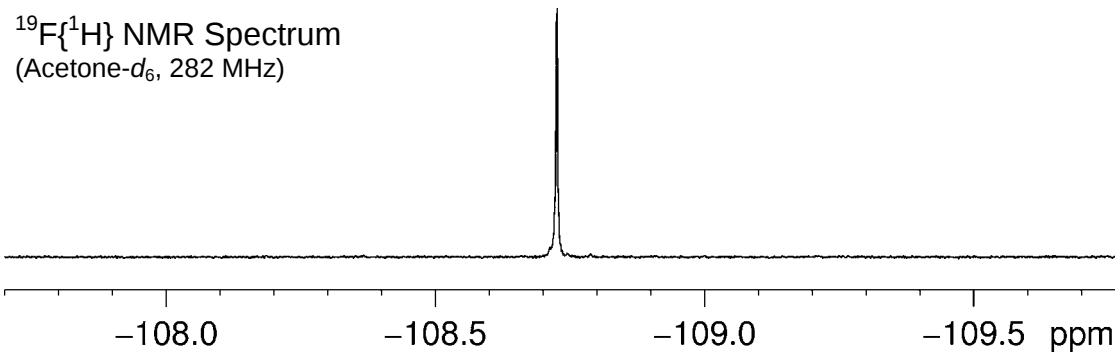


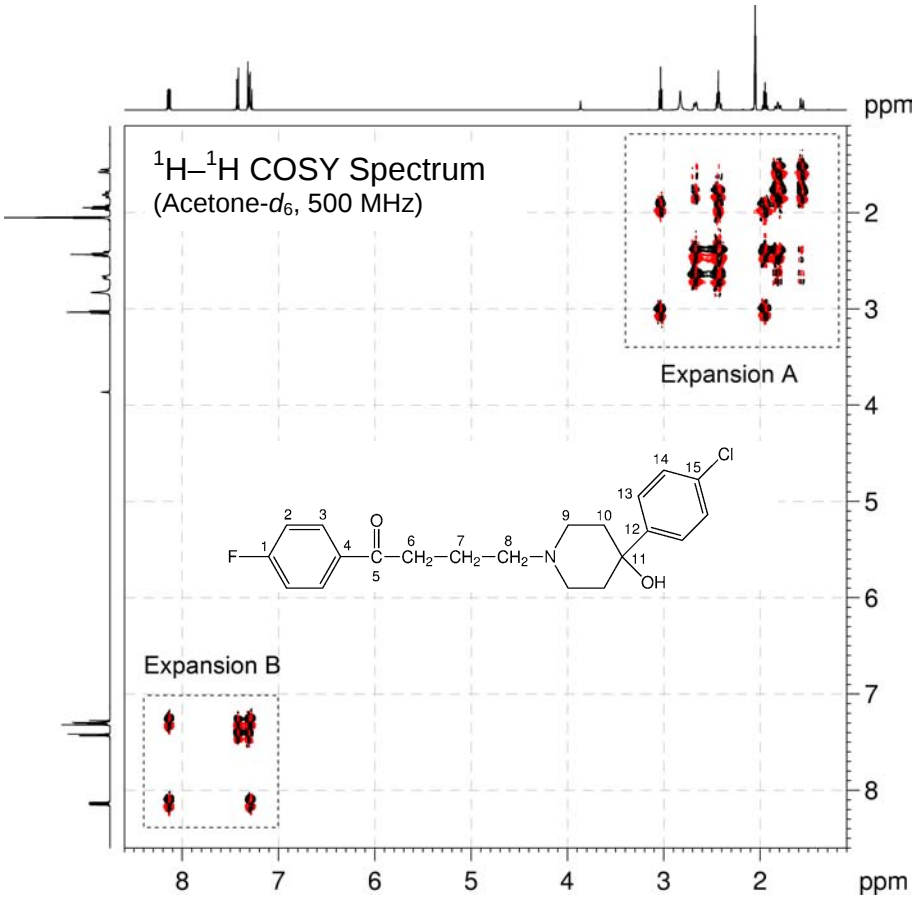

F
O
OH
Cl
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15

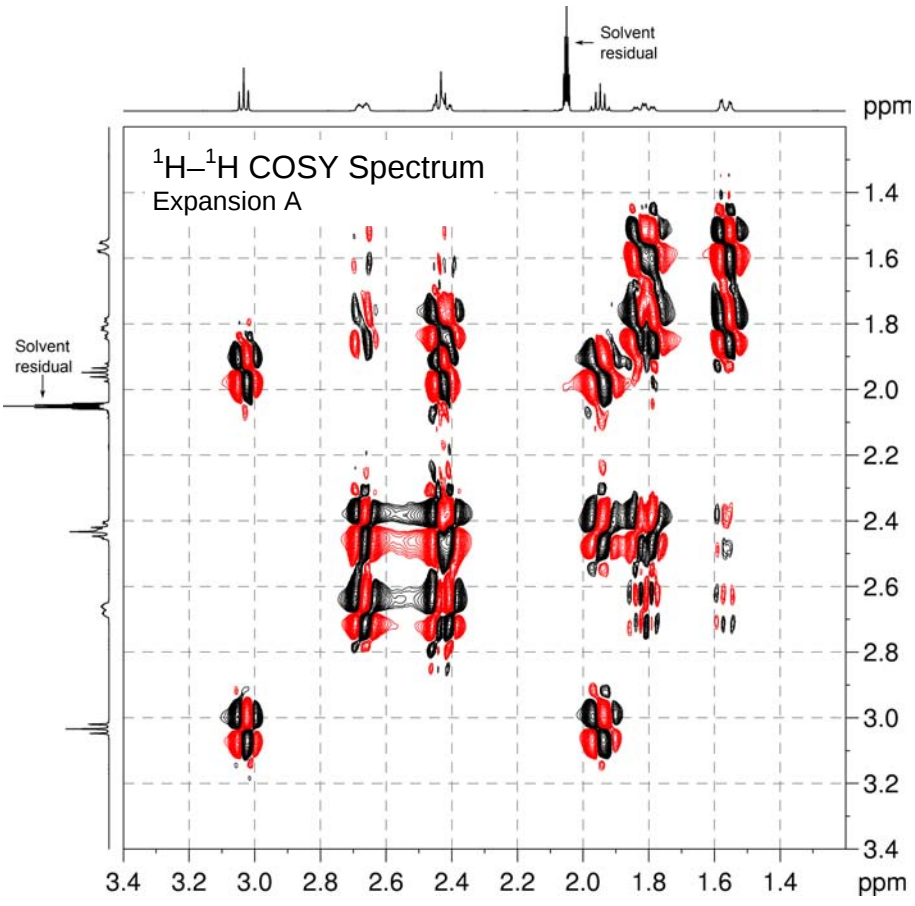

13C{1H} NMR Spectrum
(Acetone-d6, 125 MHz)
Solvent
Solvent
220 200 180 160 140 120 100 80 60 40 20 ppm

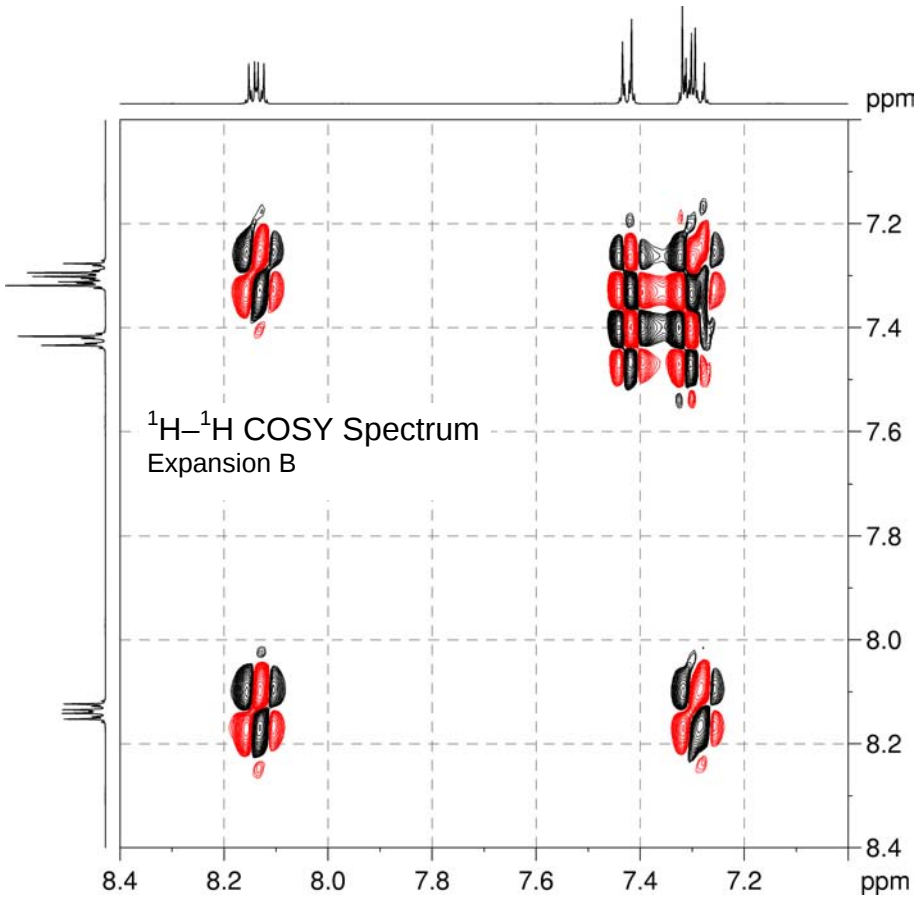

13C{1H} NMR Expansion
(Acetone-d6, 125 MHz)
165 160 155 150 145 140 135 130 125 120 115 ppm

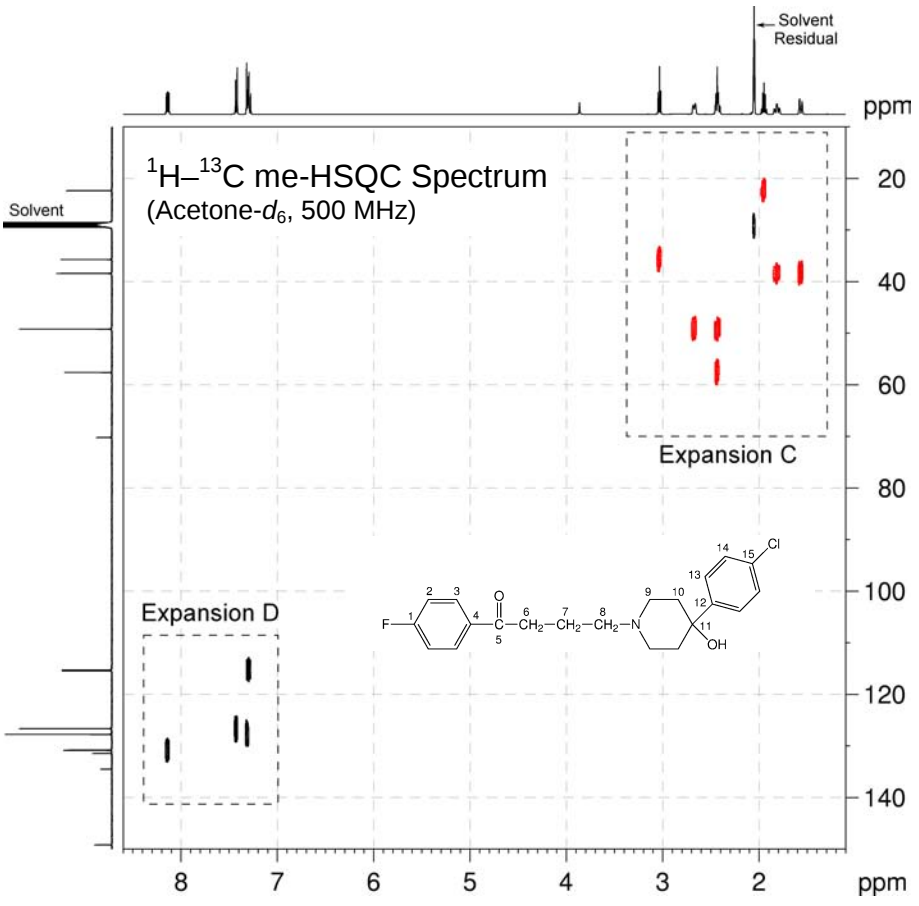

13C{1H} NMR Expansion
(Acetone-d6, 125 MHz)
0 50 100 Hz
166 165 135 134 132 131 130 116 115 ppm

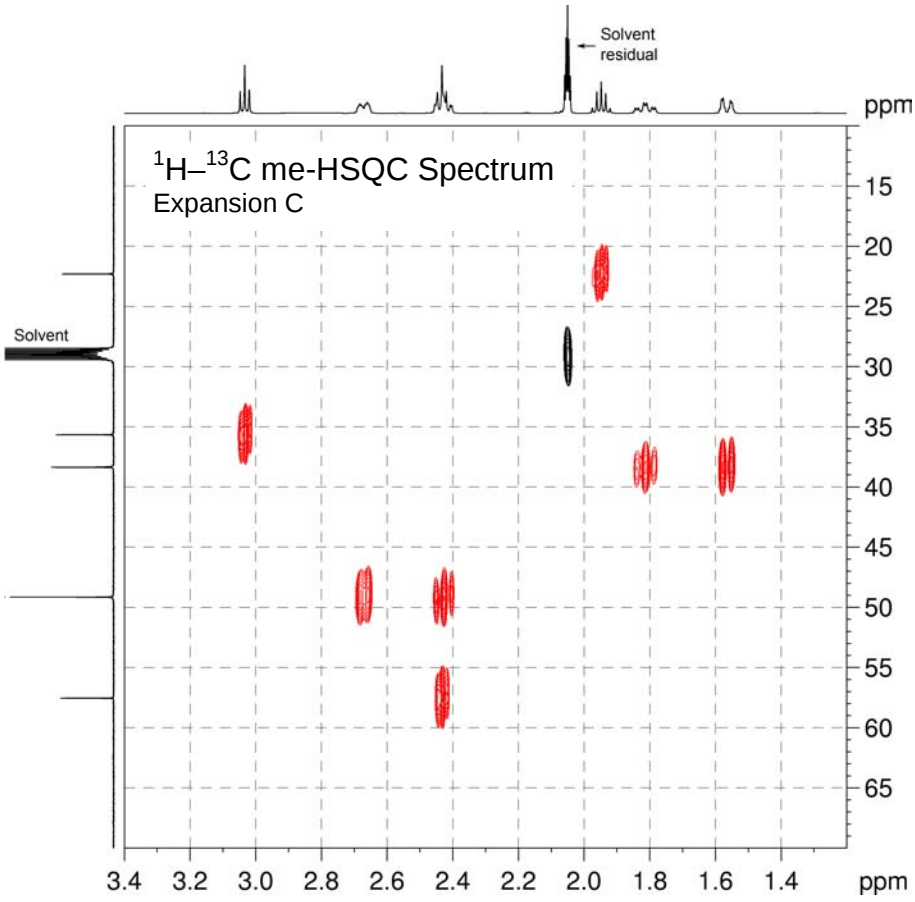

19F{1H} NMR Spectrum
(Acetone-d6, 282 MHz)
−108.0 −108.5 −109.0 −109.5 ppm

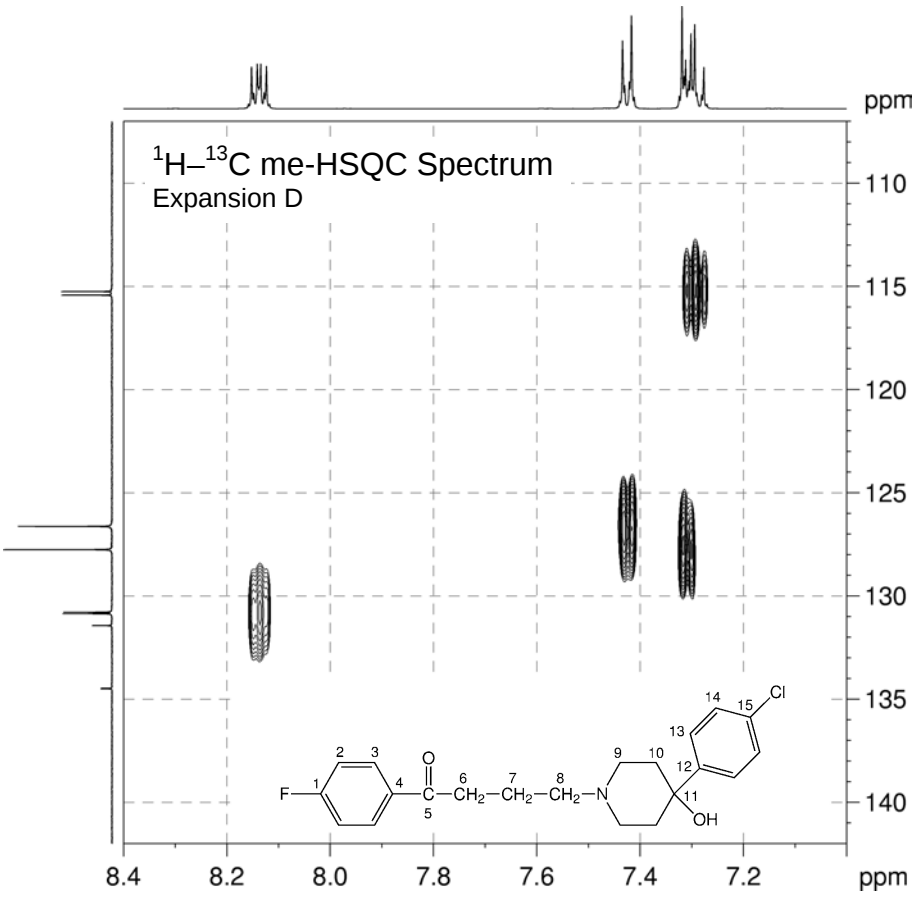

^{1}H–^{1}H COSY Spectrum
(Acetone-d_6, 500 MHz)
Expansion A
Expansion B
ppm

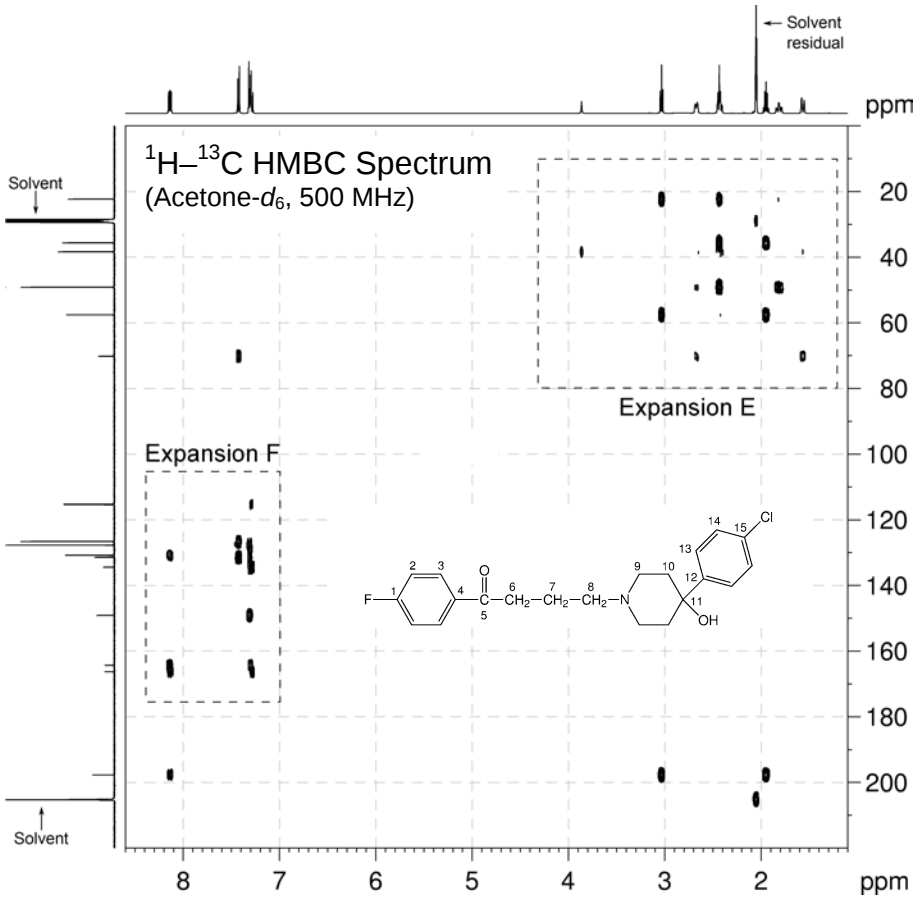

Solvent residual
^{1}H–^{1}H COSY Spectrum
Expansion A
Solvent residual
ppm

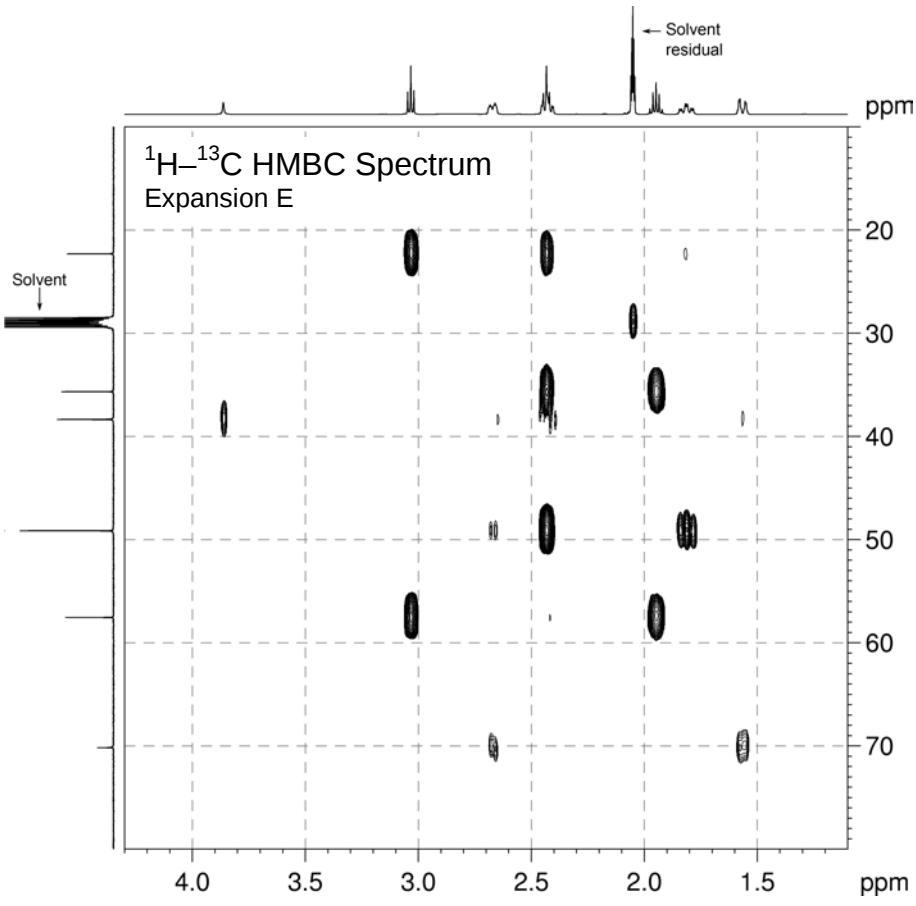

ppm
7.2
7.4
7.6
7.8
8.0
8.2
8.4
^{1}H–^{1}H COSY Spectrum
Expansion B
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm

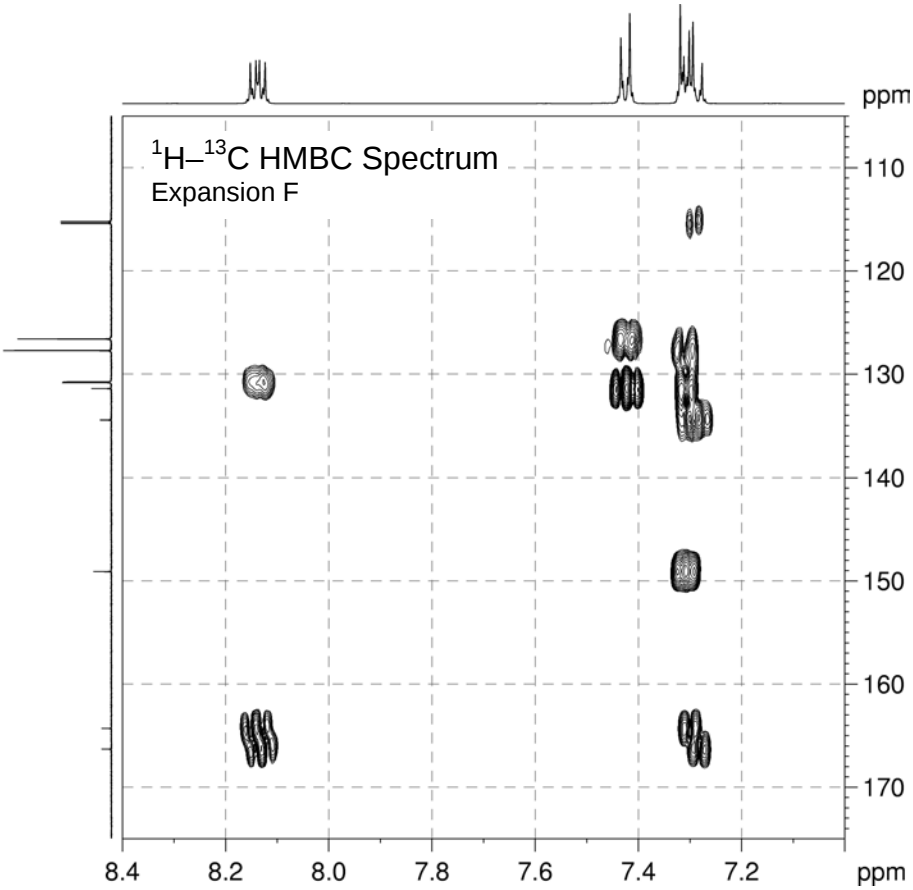

Solvent
Residual
ppm
^{1}H–^{13}C me-HSQC Spectrum
(Acetone-d_6, 500 MHz)
Solvent
20
40
60
80
100
120
140
Expansion C
Expansion D
F
Cl
OH
N
8
7
6
5
4
3
2
ppm

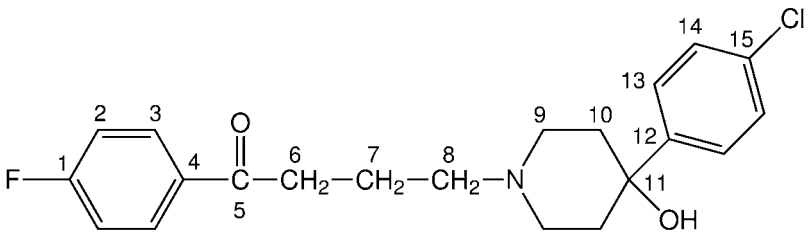

Solvent residual
ppm
^{1}H–^{13}C me-HSQC Spectrum
Expansion C
Solvent
15
20
25
30
35
40
45
50
55
60
65
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
ppm

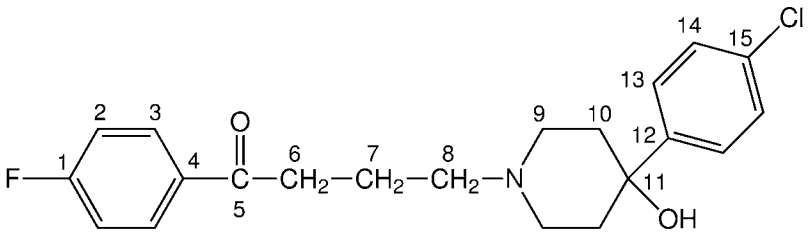

ppm
^{1}H–^{13}C me-HSQC Spectrum
Expansion D
110
115
120
125
130
135
140
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm
F
Cl
OH
N


Solvent residual
ppm
^{1}H–^{13}C HMBC Spectrum
(Acetone-d_6, 500 MHz)
Solvent
Expansion E
Expansion F
Solvent
20
40
60
80
100
120
140
160
180
200
8
7
6
5
4
3
2
ppm


Solvent residual
ppm
^{1}H–^{13}C HMBC Spectrum
Expansion E
Solvent
20
30
40
50
60
70
4.0
3.5
3.0
2.5
2.0
1.5
ppm


^{1}H–^{13}C HMBC Spectrum
Expansion F
ppm
110
120
130
140
150
160
170
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm


F
O
C
OH
Cl
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
N



Proton	Chemical Shift (ppm)	Carbon	Chemical Shift (ppm)
		C_1	
H_2		C_2	
H_3		C_3	
		C_4	
		C_5	
H_6		C_6	
H_7		C_7	
H_8		C_8	
H_9		C_9	
H_{10}		C_{10}	
		C_{11}	
		C_{12}	
H_{13}		C_{13}	
H_{14}		C_{14}	
		C_{15}	
OH			