Problem 47

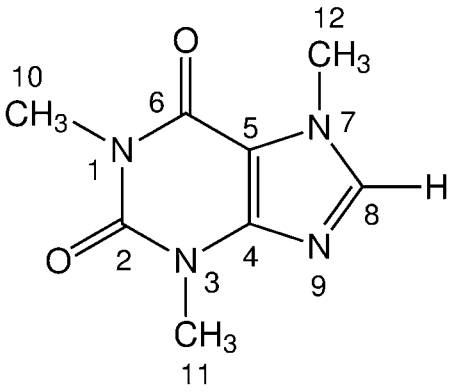
The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra of caffeine ($C_8H_{10}N_4O_2$) recorded in DMSO-d_6 solution at 298 K and 500 MHz are given below.

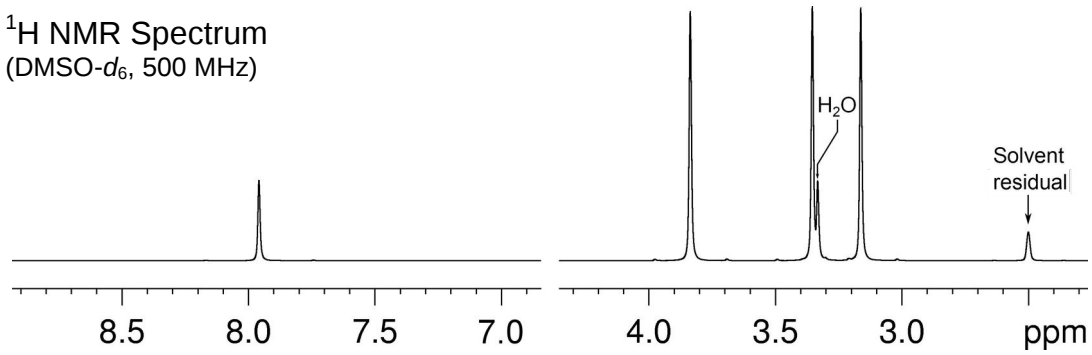
The ^{1}H NMR spectrum has signals at δ 3.16, 3.35, 3.84 and 7.96 ppm.

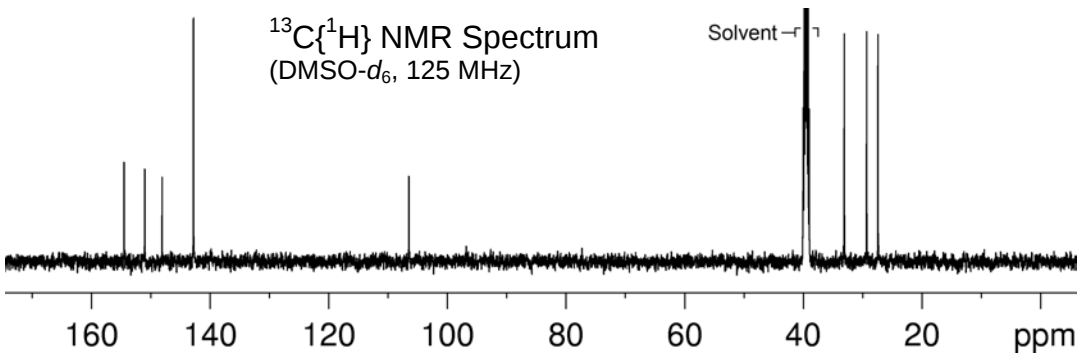
The $^{13}C\{^{1}H\}$ NMR spectrum has signals at δ 27.4, 29.3, 33.1, 106.5, 142.7, 148.0, 150.9 and 154.4 ppm.

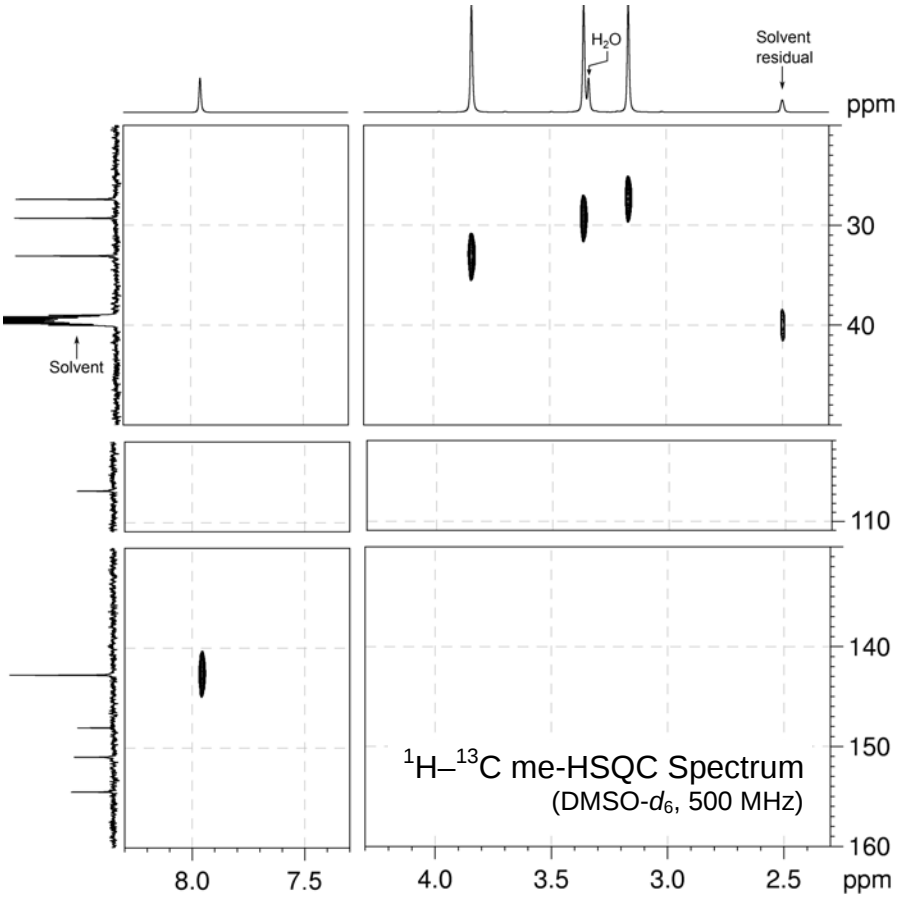
The ^{15}N NMR spectrum has signals at δ 112.9, 149.6, 156.1 and 231.2 ppm.

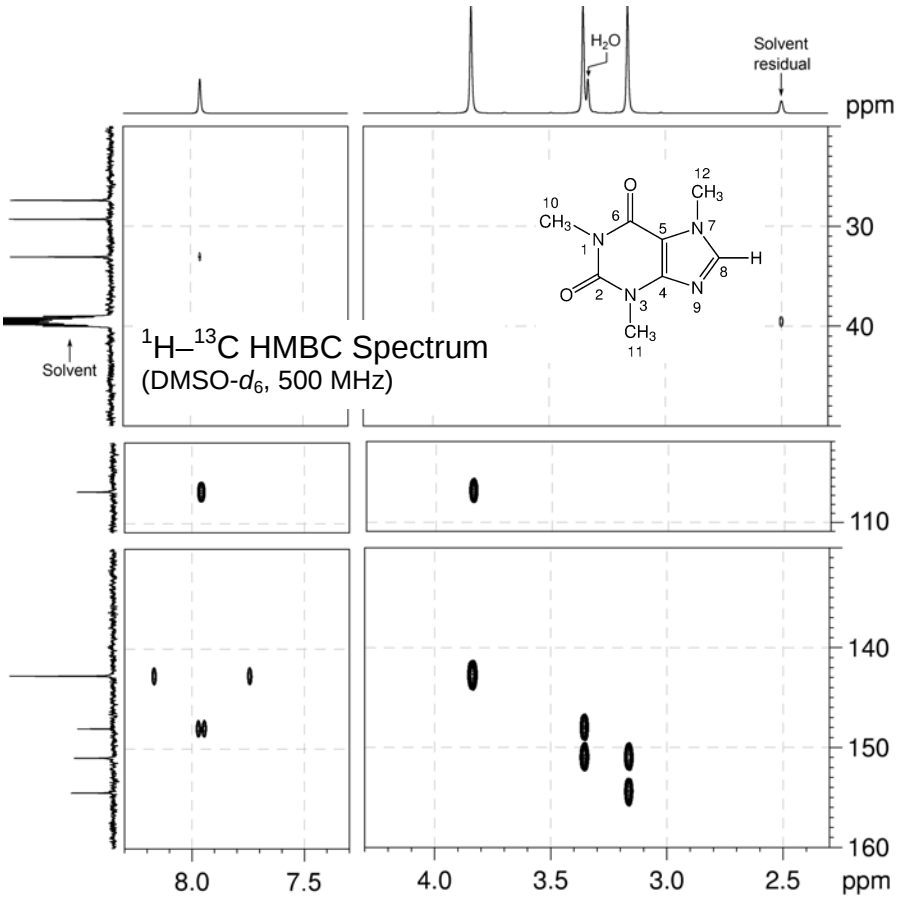
The multiplicity-edited ^{1}H–^{13}C HSQC, ^{1}H–^{13}C HMBC and ^{1}H–^{15}N HMBC spectra are given on the following pages. Use these spectra to assign each ^{1}H, ^{13}C and ^{15}N resonance to its corresponding nucleus.

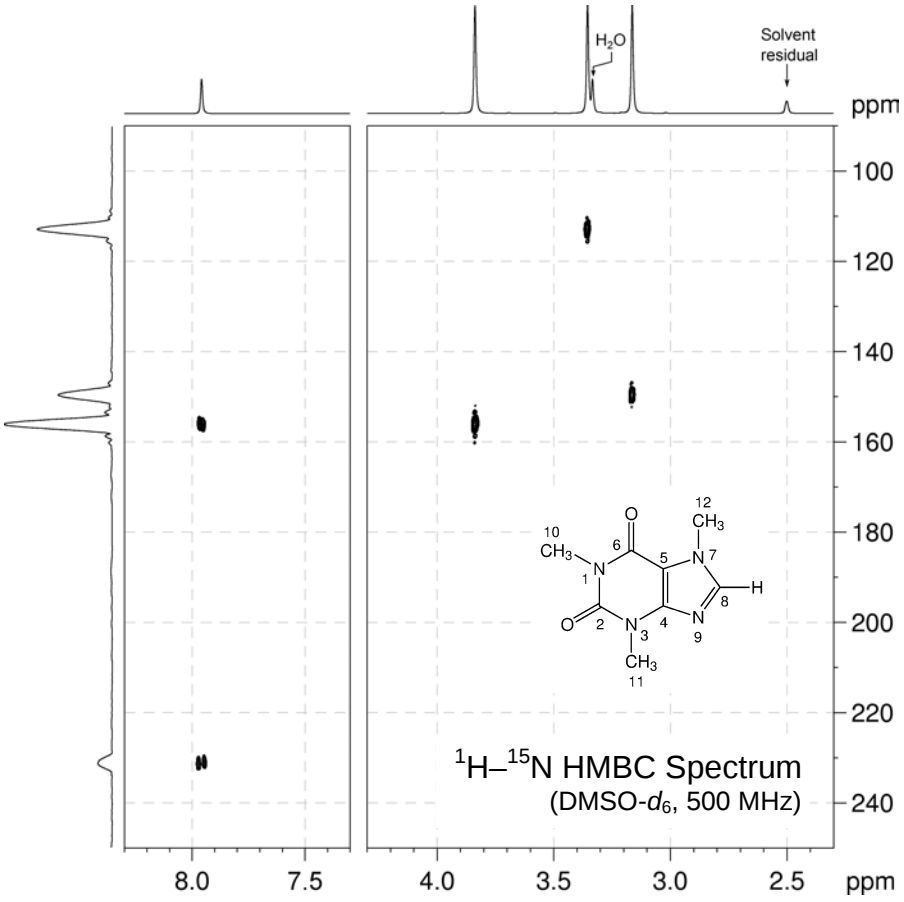







H2O
Solvent residual
ppm
30
40
Solvent
110
140
150
160
8.0
7.5
4.0
3.5
3.0
2.5
ppm
1H–13C me-HSQC Spectrum
(DMSO-d6, 500 MHz)


H2O
Solvent residual
ppm
30
40
Solvent
110
140
150
160
8.0
7.5
4.0
3.5
3.0
2.5
ppm
1H–13C HMBC Spectrum
(DMSO-d6, 500 MHz)
O
12
CH3
10
CH3
6
5
N
7
N
1
H
8
O
2
N
4
N
3
9
CH3
11



Proton	Chemical Shift (ppm)	Nucleus	Chemical Shift (ppm)
		N_1	
		C_2	
		N_3	
		C_4	
		C_5	
		C_6	
		N_7	
H_8		C_8	
		N_9	
H_{10}		C_{10}	
H_{11}		C_{11}	
H_{12}		C_{12}	