SUPPORTING INFORMATION

The unexpected reactivity of 9-iodo-*nido*-carborane: from nucleophilic substitution reactions to the synthesis of tricobalt tris(dicarbollide) Na[4,4′,4″-$(MeOCH_2CH_2O)_3$-3,3′,3″-$Co_3(\mu^3\text{-}O)(\mu^3\text{-}S)(1,2\text{-}C_2B_9H_{10})$]

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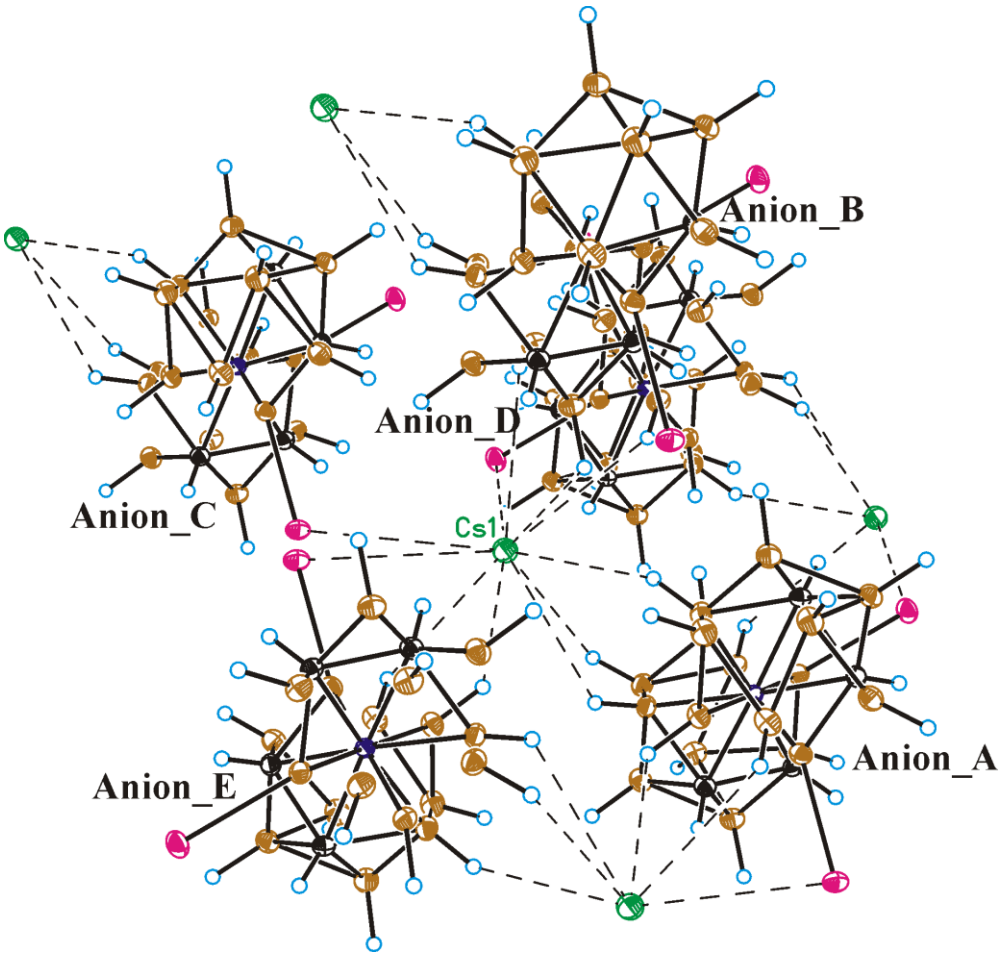


Figure 1S. Crystal structure fragment of Si18F showing close contacts formed by Cs atom which links 5 dicarbollide anions

Cs…Anion_A : Cs1…H7, 3.03(2)Å; Cs1…H7', 3.08(2)Å; Cs1…H12', 3.42(2)Å
Cs…Anion_B : Cs1…H6', 3.25(2)Å; Cs1…H10', 3.21(2)Å
Cs…Anion_C : Cs1…I1, 4.0869(11)Å
Cs…Anion_D : Cs1…I2, 3.7708(12)Å; Cs1…H9', 3.39(2)Å
Cs…Anion_E : Cs1…I2, 4.1904(12)Å; Cs1…H8, 3.24(2)Å; Cs1…H9, 3.04(2)Å

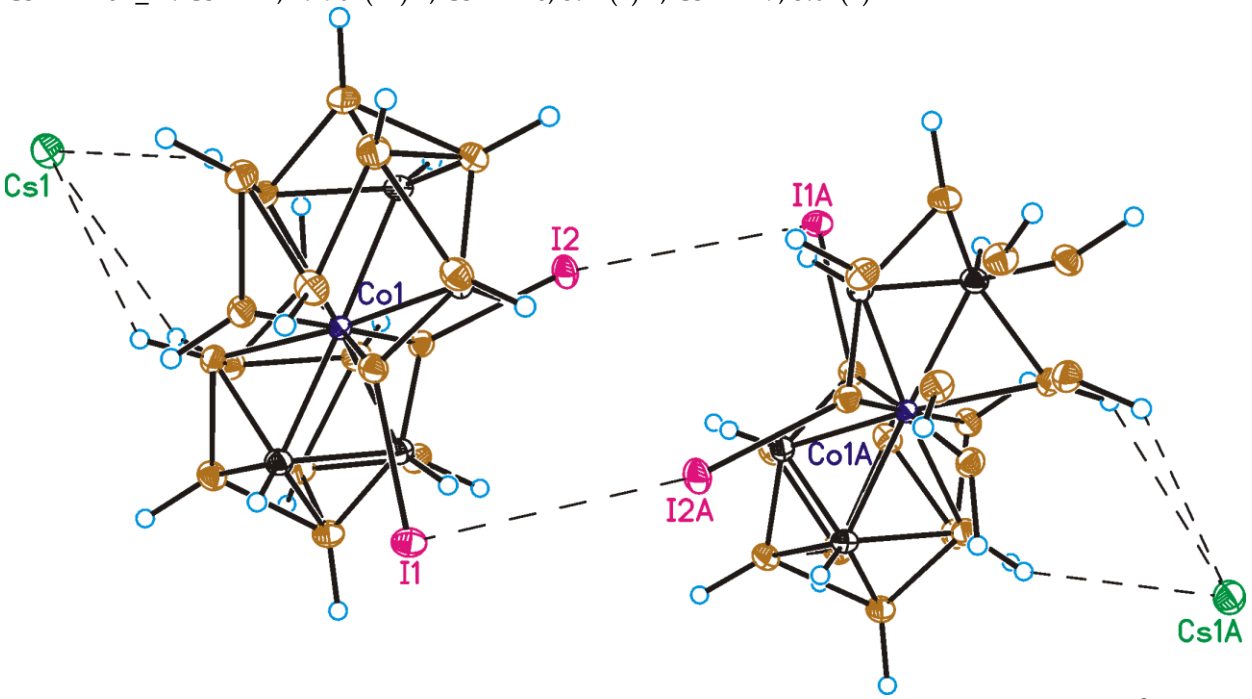


Figure 2S. Centrosymmetric dimer formed by I…I shortened contacts (I1…I2A, 4.0803(7)Å)

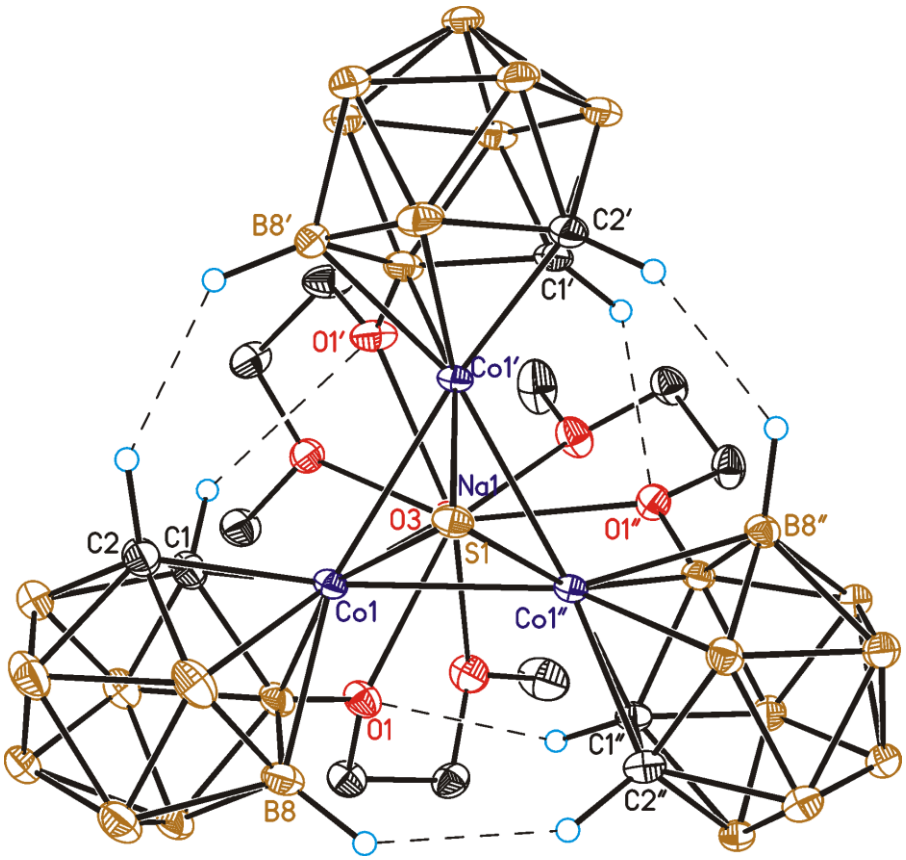


Figure 3S. Intramolecular noncovalent bonding in Na[12].

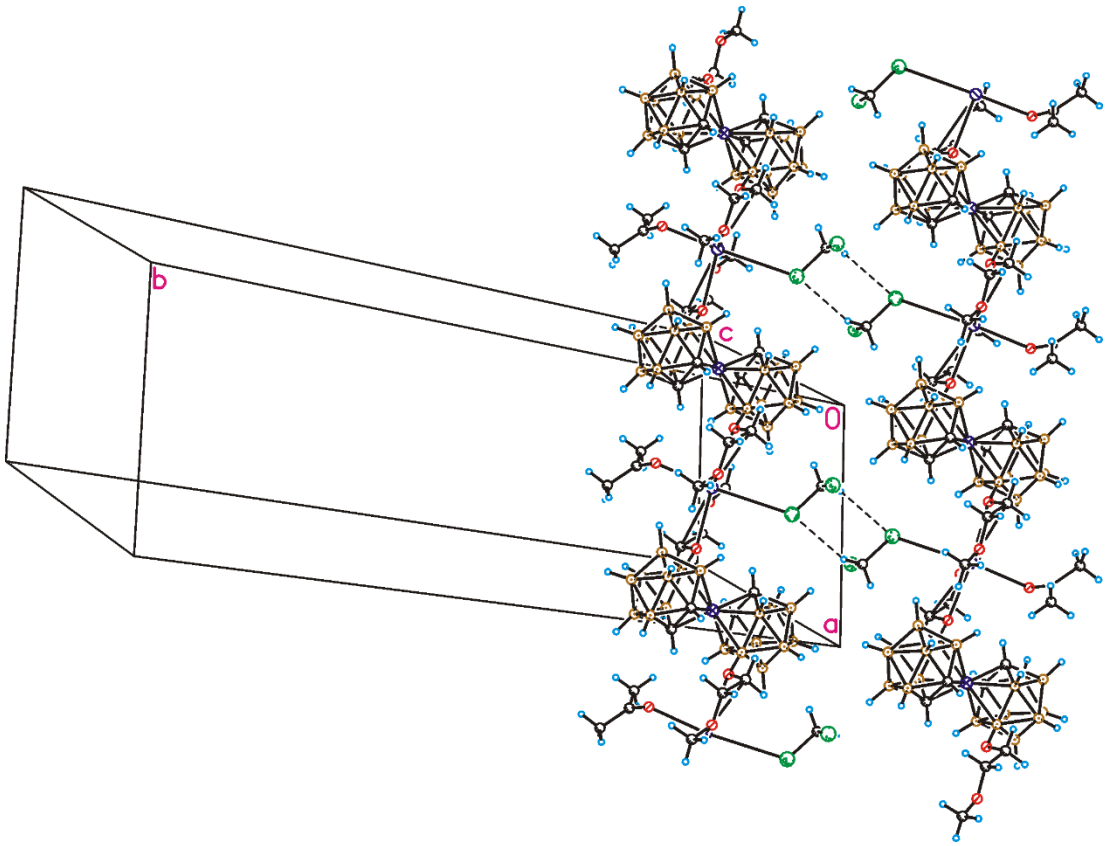


Figure 4S. Interchain bonding in crystal structure of $\{K(Me_2CO)(CH_2Cl_2)[\mathbf{14}]\}_n$.

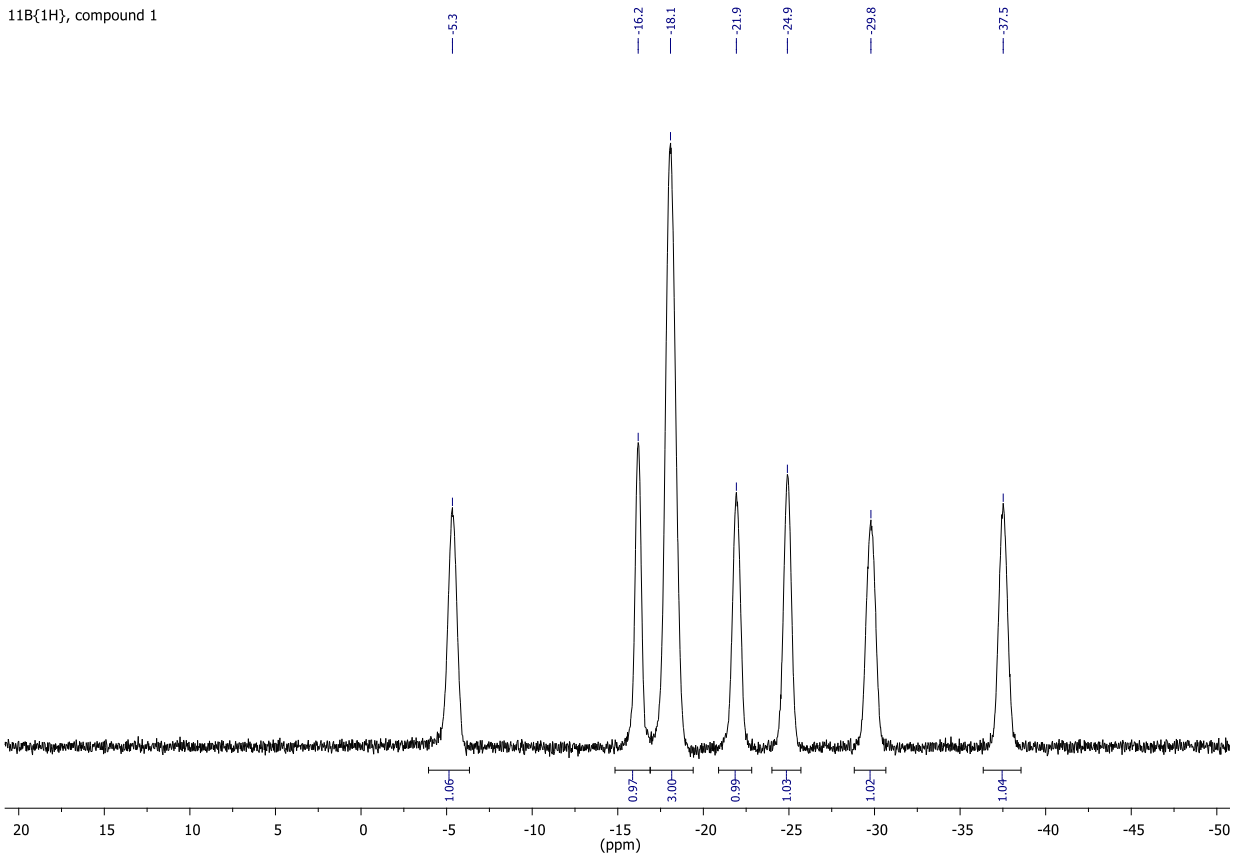
Table 1S. Selected characteristics of all optimized (DFT/BP86/cc-pvDZ) isomers of diiodo cobalt bis(dicarbollide), and key properties for all found intercage bond critical points (BCP').

	4-4'- isomer					4-7'- isomer			8-8' isomer		
rotamer	*cisoid*	*gauche*	*transoid*	*gauche*-2	*cisoid*-2	*cisoid*	*gauche*	*transoid*	*cisoid*	*gauche*	*transoid*
B(8')-B(10')-B(10)-B(8) dihedral [a]	36	93	161	116	45	46	107	180	42	113	180
Deviation from ideal [b]	0	15	19	8	9	10	1	0	6	5	0
B(I')-B(10')-B(10)-B(I) dihedral	96	34	63	104	173	45	103	180	42	113	180
Deviation from ideal	12	2	27	4	7	9	5	0	6	5	0
ΔE, kcal·mol^{-1} [c]	17.3	17.4	12.8	5.4	11.8	16.5	10.4	12.3	13.8	4.2	0.0
ΔG, kcal·mol^{-1} [c]	16.7	17.1	11.8	5.2	11.5	16.1	10.2	12.0	13.5	4.1	0.0
CH···I BCP											
r, Å [d]			2.646 2.646	2.882 3.001 2.882 3.001	2.775 2.774	2.785	2.816 3.059			2.849 2.850	2.935 2.935 2.936 2.934
ρ_{BCP}, a.u.			0.017 0.017	0.010 0.008 0.010 0.008	0.013 0.013	0.012	0.012 0.007			0.011 0.011	0.009 0.009 0.009 0.009
E_{BCP}, kcal·mol^{-1} [e]			-3.3 -3.3	-1.8 -1.4 -1.8 -1.4	-2.4 -2.4	-2.3	-2.1 -1.3			-2.0 -2.0	-1.6 -1.6 -1.6 -1.6
DI [f]			0.075 0.075	0.037 0.025 0.037 0.025	0.049 0.049	0.052	0.044 0.021			0.040 0.040	0.031 0.031 0.031 0.031
I···I BCP											
r, Å [d]		4.225				4.013			4.002		
ρ_{BCP}, a.u.		0.003				0.005			0.006		
E_{BCP}, kcal·mol^{-1} [e]		-0.6				-1.0			-1.0		
DI [f]		0.017				0.030			0.036		
BH···I BCP											
r, Å [d]		3.069 3.069				2.970	3.122	2.941 2.940	3.061 3.061		
ρ_{BCP}, a.u.		0.008 0.008				0.009	0.007	0.010 0.010	0.008 0.008		
E_{BCP}, kcal·mol^{-1} [e]		-1.3 -1.3				-1.7	-1.3	-1.9 -1.9	-1.4 -1.4		
DI [f]		0.029 0.029				0.036	0.025	0.038 0.038	0.029 0.029		

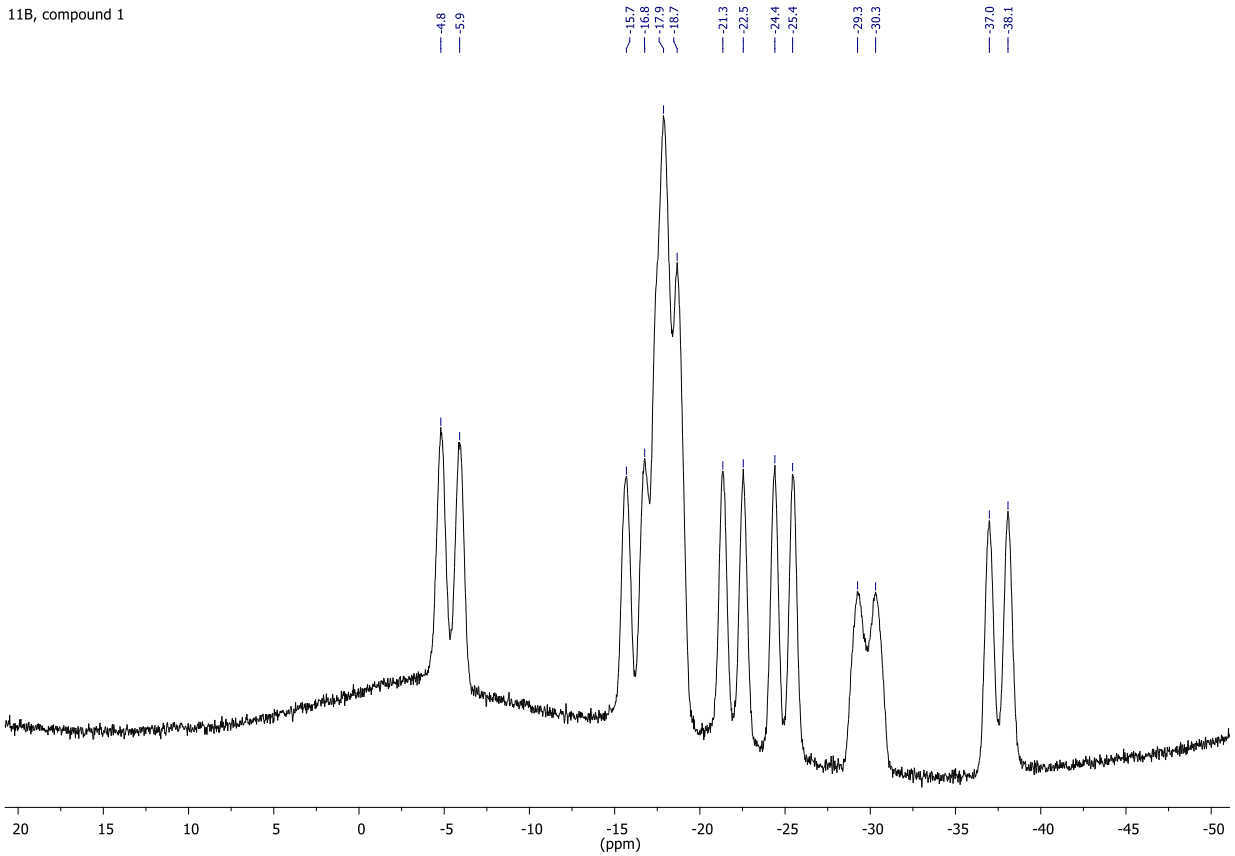
[a,b] ideal angles are 36° for *cisoid*, 108° for *gauche* and 180° for *transoid* rotamers [c] all energies are respective to most favored 8,8'-*transoid* isomer.
[d] H···I and I···I distances respectively [e] energy of interaction calculated as $E_{BCP}=0.5\cdot V_{BCP}$ [f] QTAIM electron delocalization index (formal bond order)

Table 2. Selected geometry parameters (distances in Å, angles in deg.) for Na[**12**]. Comparison of experiment and theory.

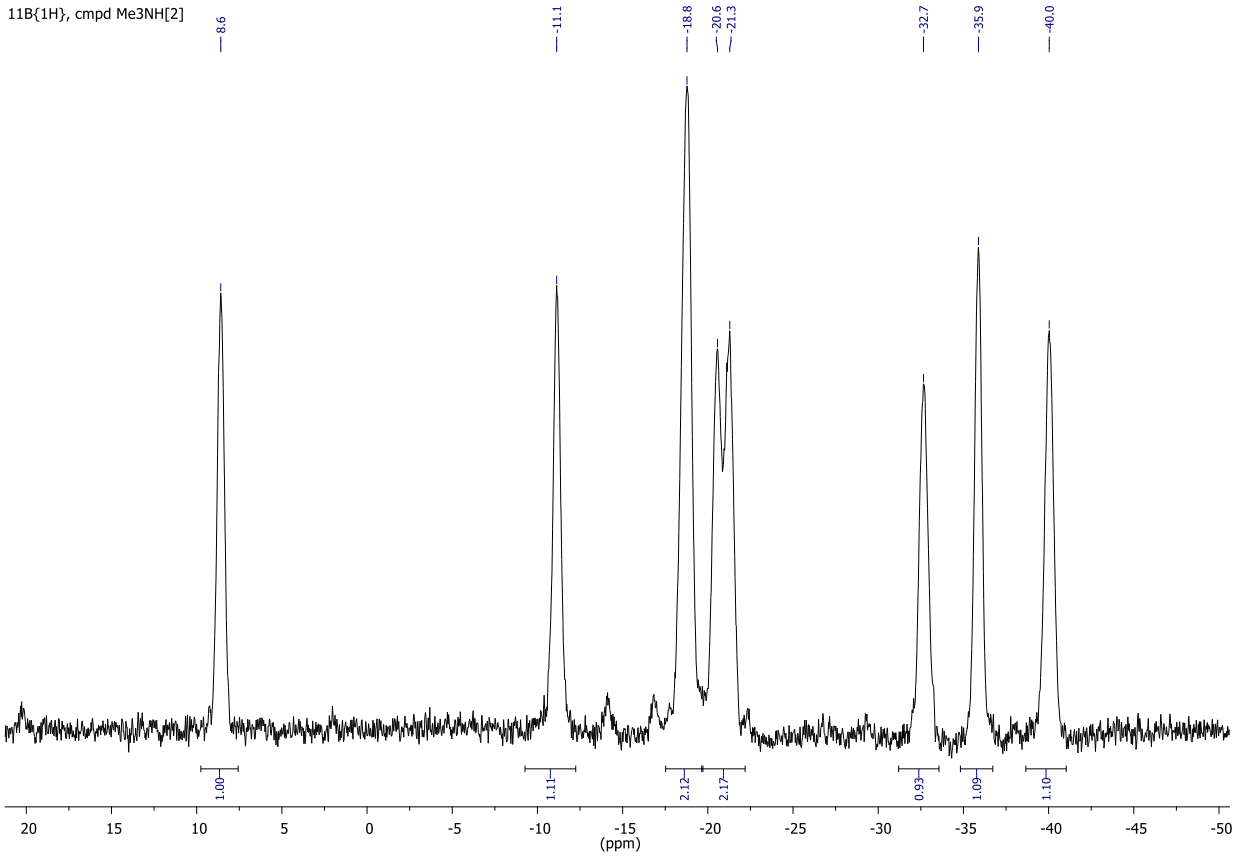
Bond	X-ray	Calc	Torsion angle or noncovalent bond	X-ray	Calc
Co1-Co1'	2.5401(6)	2.557	B8-Co1-Co1"-C2	-6.6(3)	-10.6
Co1-Co1"	2.5436(6)	2.557	C2-Co1-Co1'-B8'	-12.1(3)	-10.6
Co1'-Co1"	2.5339(6)	2.557	C2'-Co1'-Co1"-B8"	-14.7(3)	-10.5
Co1-S1	2.1637(8)	2.161	B8-B4-O1-C3	97.4(4)	92.5
Co1'-S1	2.1614(9)	2.161	B8'-B4'-O1'-C3'	99.3(4)	92.5
Co1"-S1	2.1636(8)	2.162	B8"-B4"-O1"-C3"	93.4(4)	92.4
Co1-O3	1.835(2)	1.823	O1…H1"	2.33	2.45
Co1'-O3	1.8408(19)	1.823	O1'…H1	2.68	2.46
Co1"-O3	1.844(2)	1.822	O1"…H1'	2.40	2.46
Co1-C1	2.015(3)	1.984	H8….H2"	2.13	2.07
Co1-C2	2.021(3)	1.982	H8"…H2'	2.22	2.06
Co1-B4	2.096(3)	2.083	H8'…H2	2.19	2.06
Co1-B8	2.104(4)	2.075			
Co1-B7	2.112(4)	2.097			
Co1'-C1'	2.014(3)	1.984			
Co1'-C2'	2.012(3)	1.982			
Co1'-B4'	2.109(4)	2.083			
Co1'-B8'	2.107(4)	2.076			
Co1'-B7'	2.112(3)	2.097			
Co1"-C1"	2.029(3)	1.983			
Co1"-C2"	2.029(3)	1.982			
Co1"-B4"	2.101(3)	2.084			
Co1"-B8"	2.110(3)	2.076			
Co1"-B7"	2.111(4)	2.098			

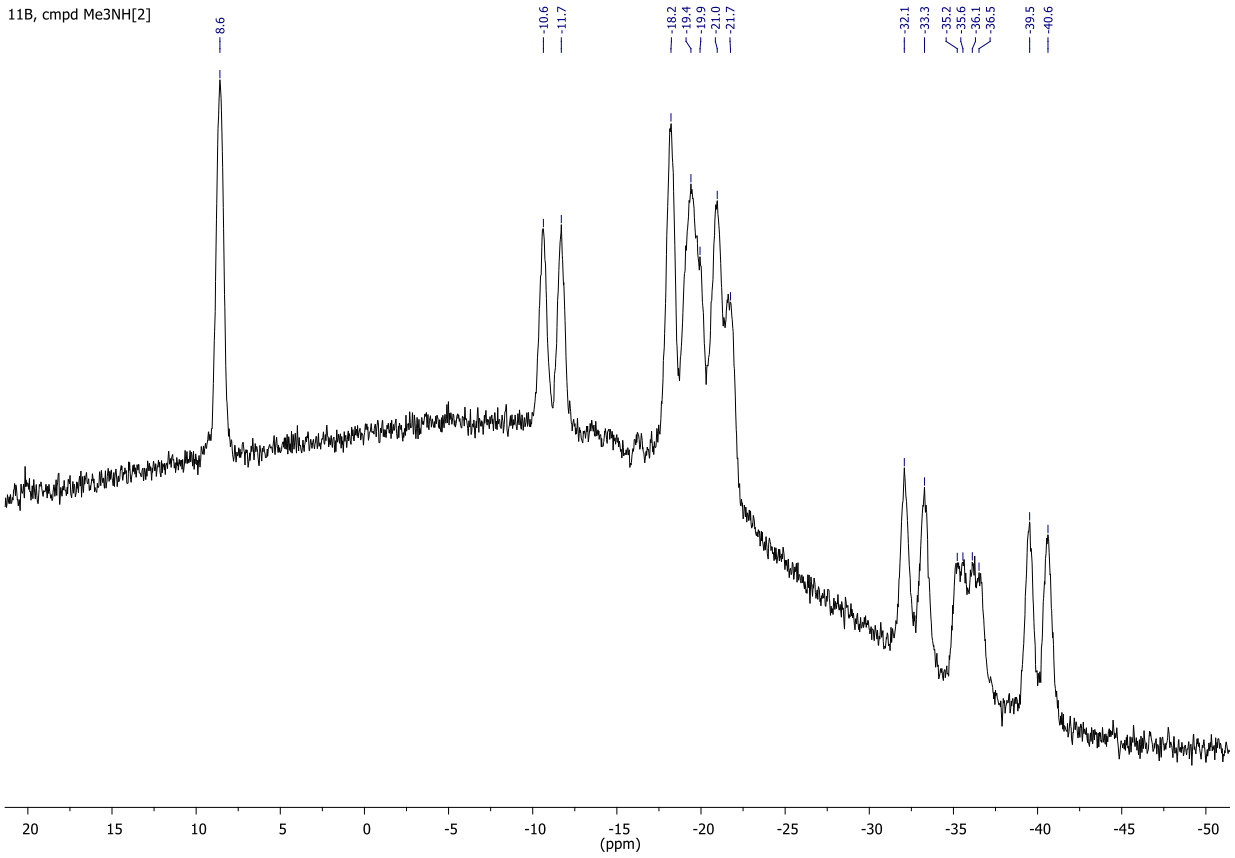

11B{1H}, compound 1
-5.3
-16.2
-18.1
-21.9
-24.9
-29.8
-37.5
1.06
0.97
3.00
0.99
1.03
1.02
1.04
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
(ppm)

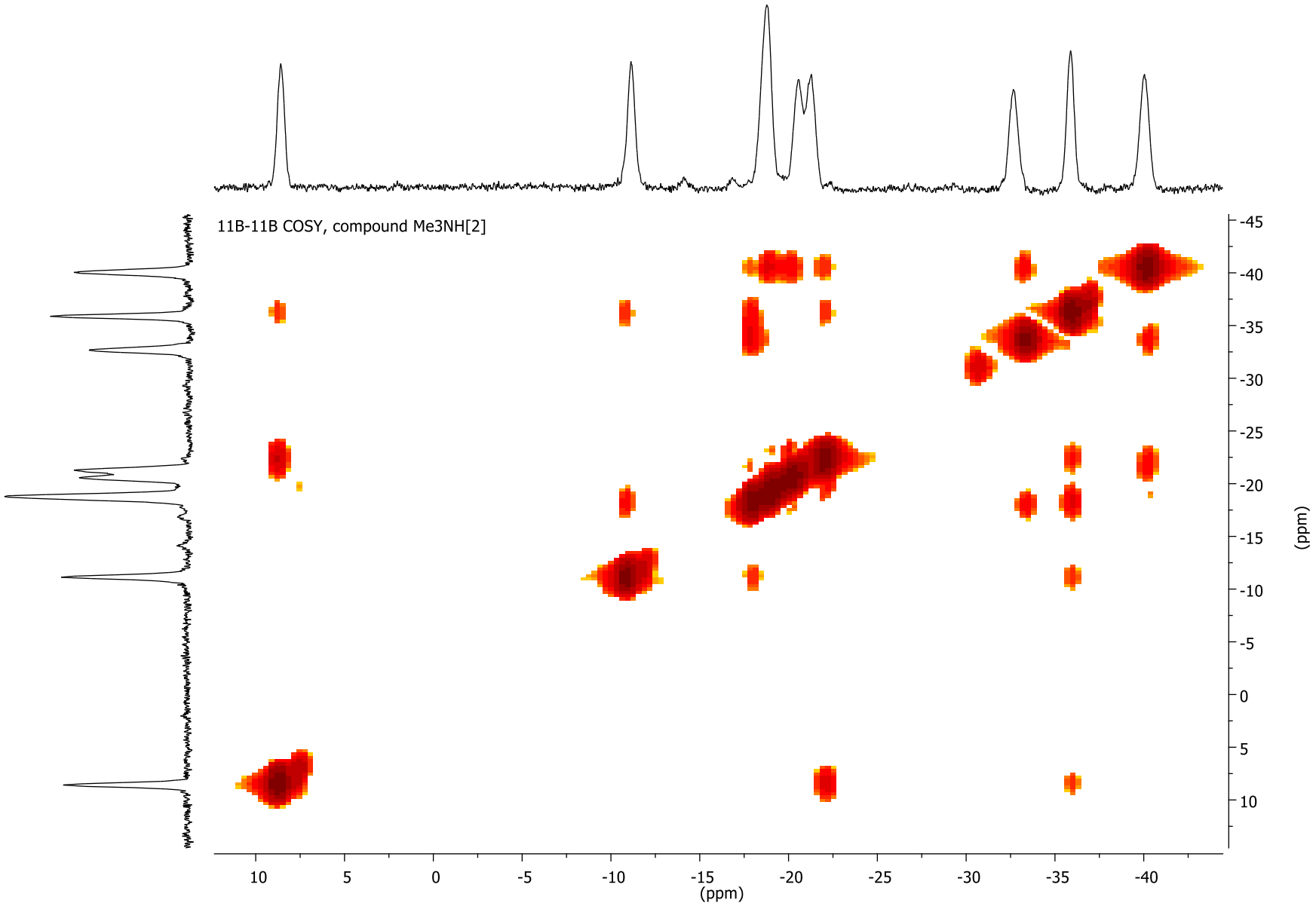
11B, compound 1



11B{1H}, cmpd Me3NH[2]




11B, cmpd Me3NH[2]
8.6
-10.6
-11.7
-18.2
-19.4
-19.9
-21.0
-21.7
-32.1
-33.3
-35.2
-35.6
-36.1
-36.5
-39.5
-40.6
20
15
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
-45
-50
(ppm)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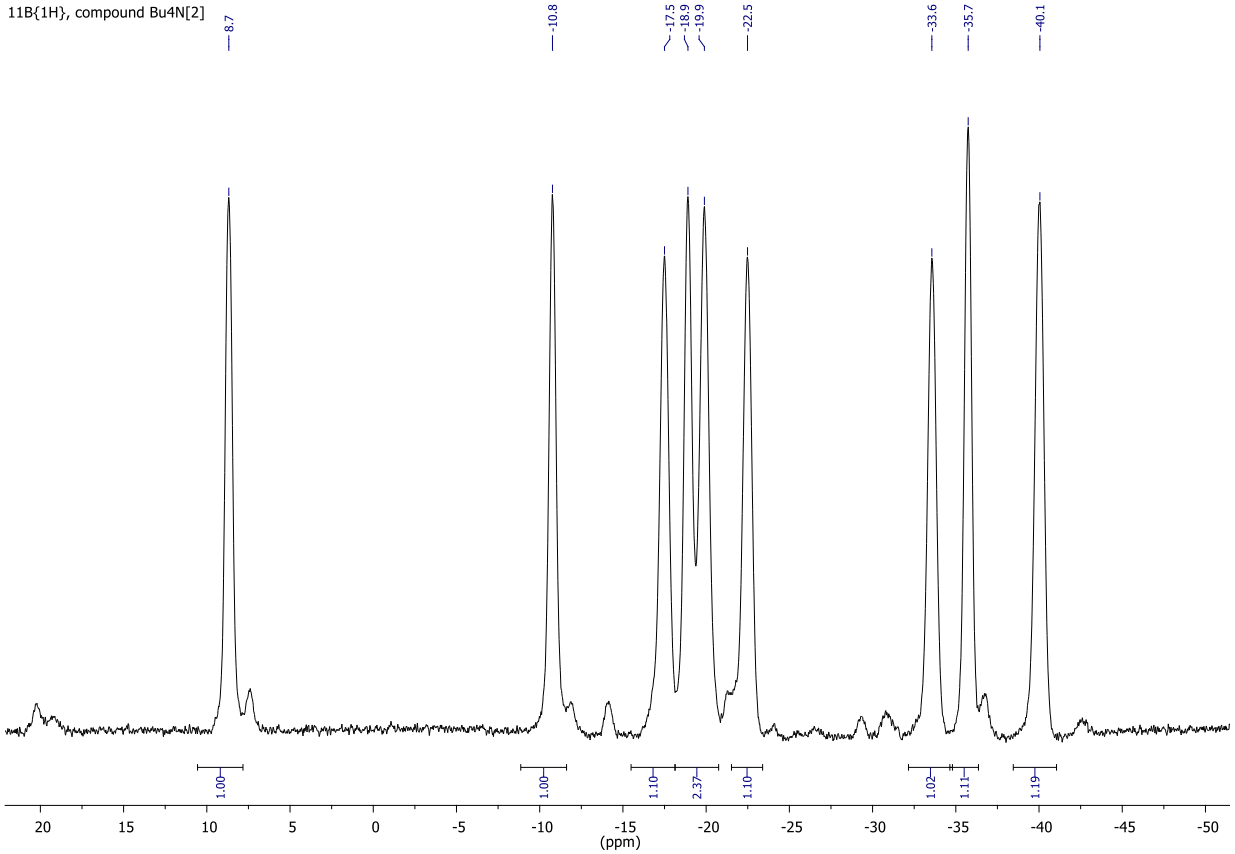
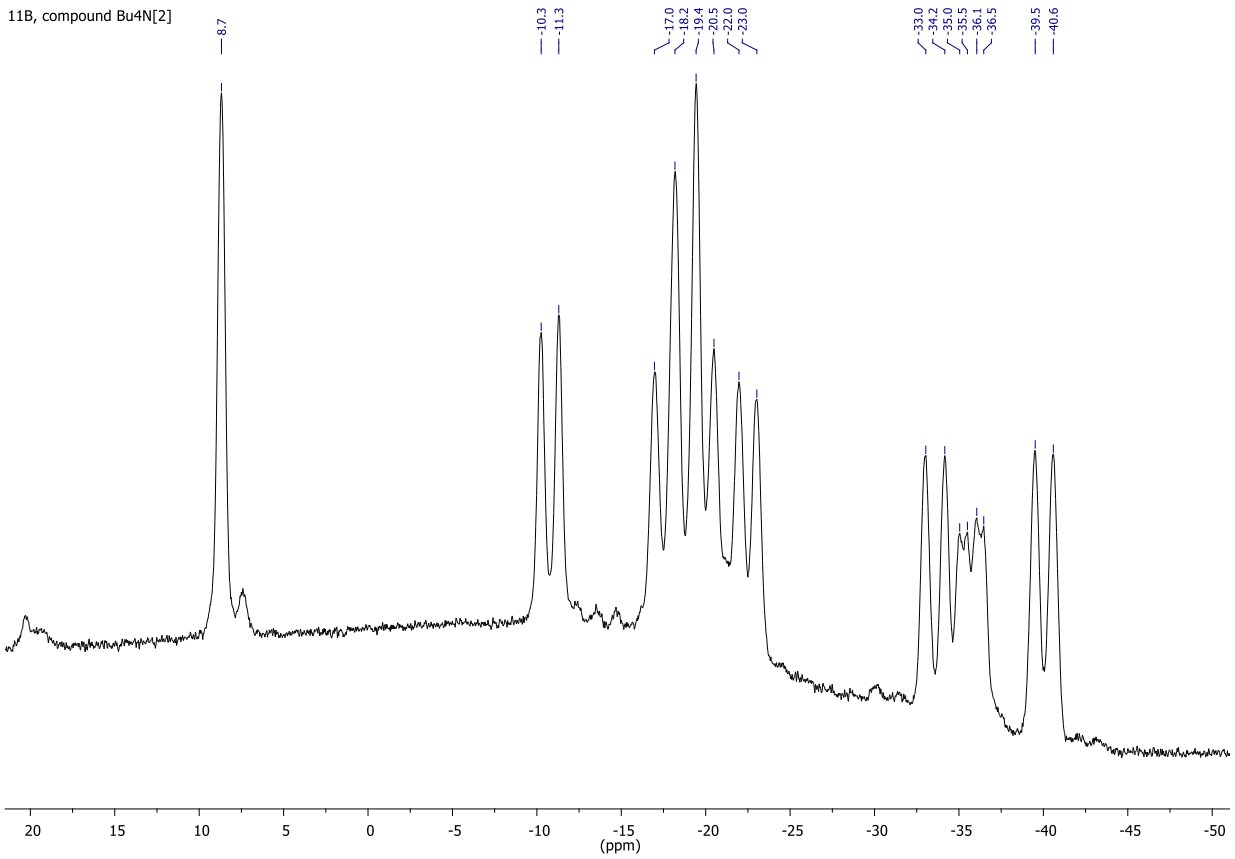
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
(ppm)
-45
-40
-35
-30
-25
-20
-15
-10
-5
0
5
10
(ppm)

11B{1H}, compound Bu4N[2]



11B, compound Bu4N[2]



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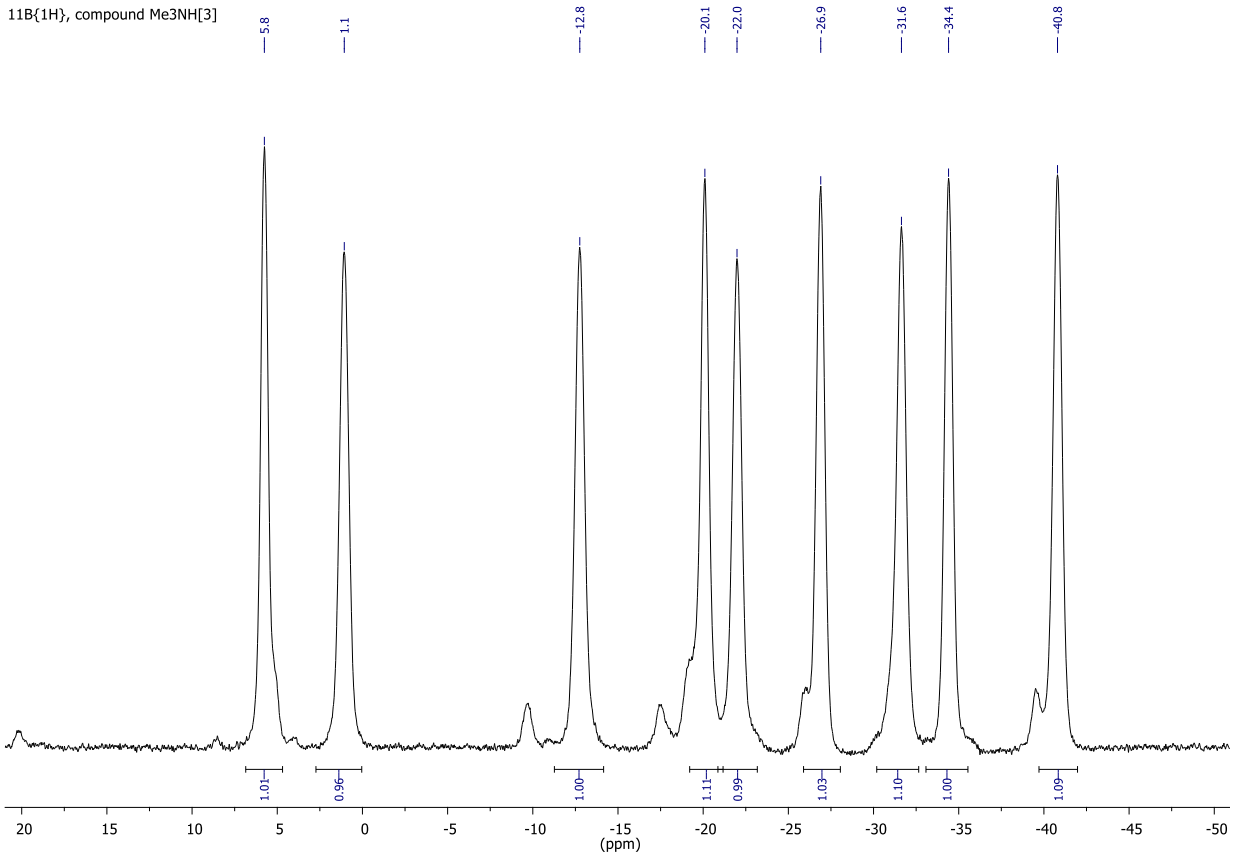
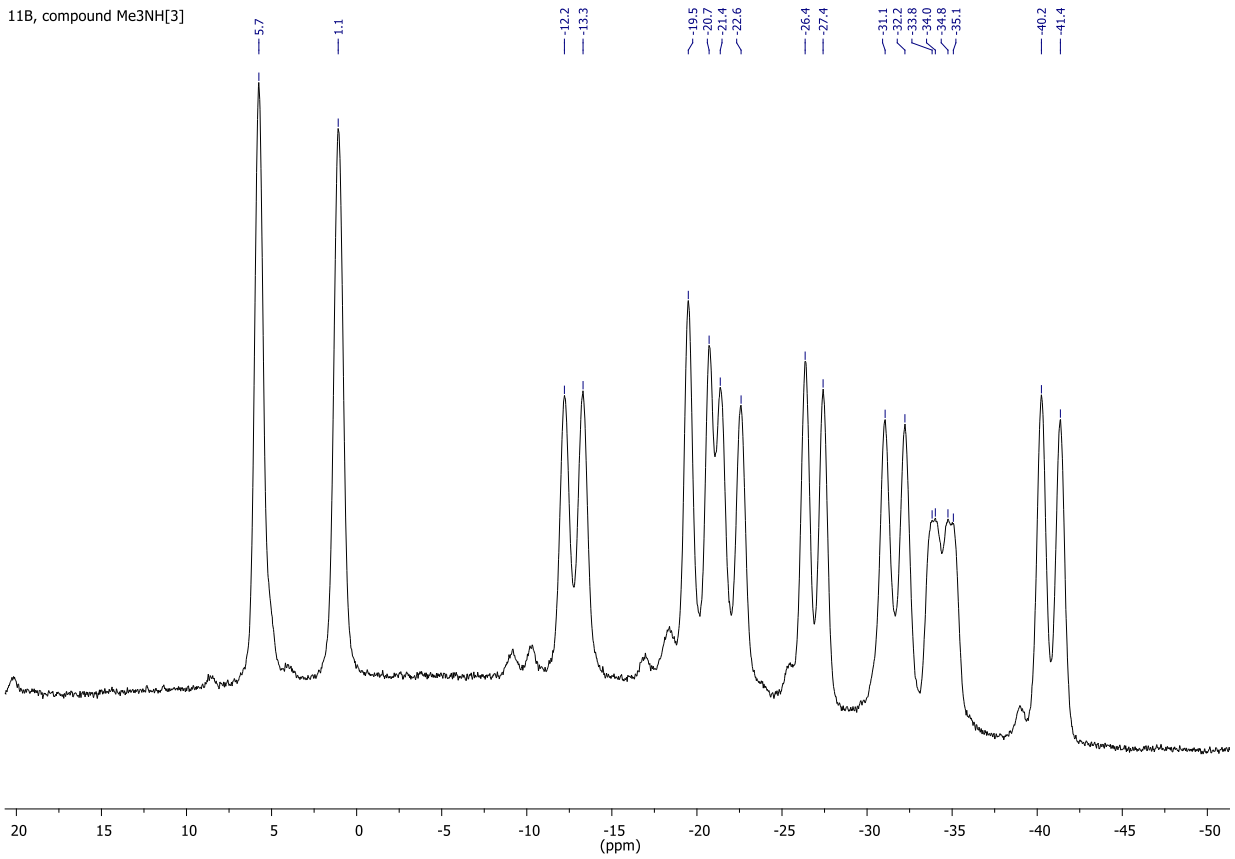
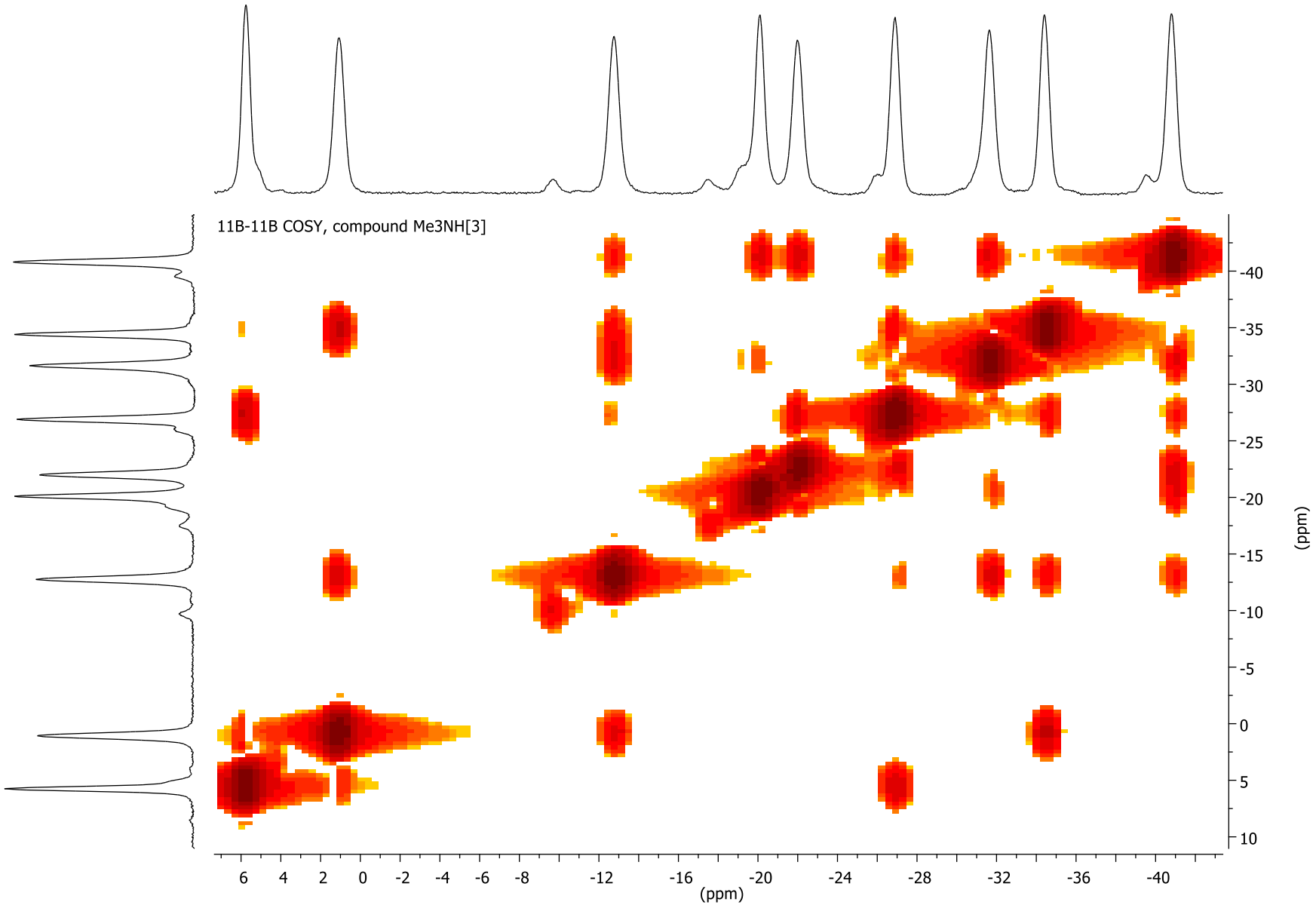
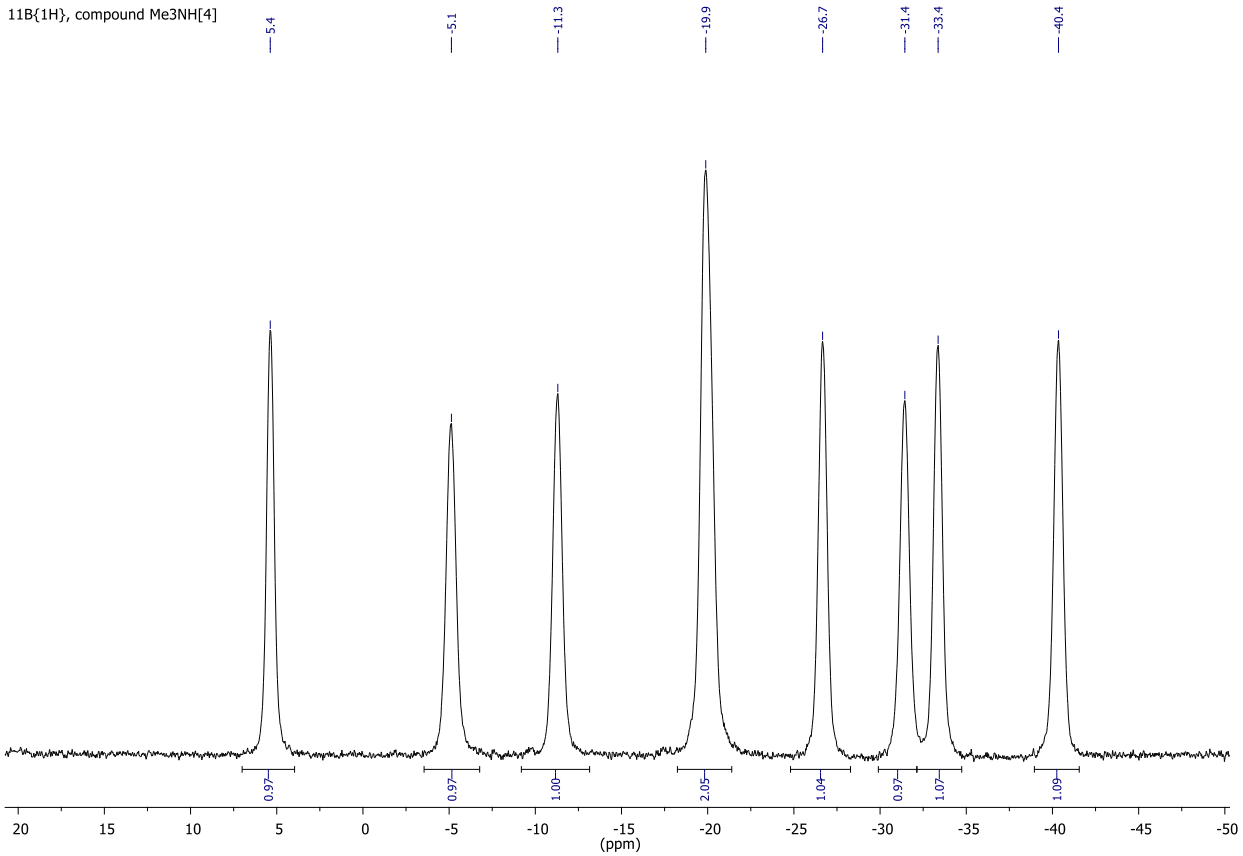


11B, compound Me3NH[3]

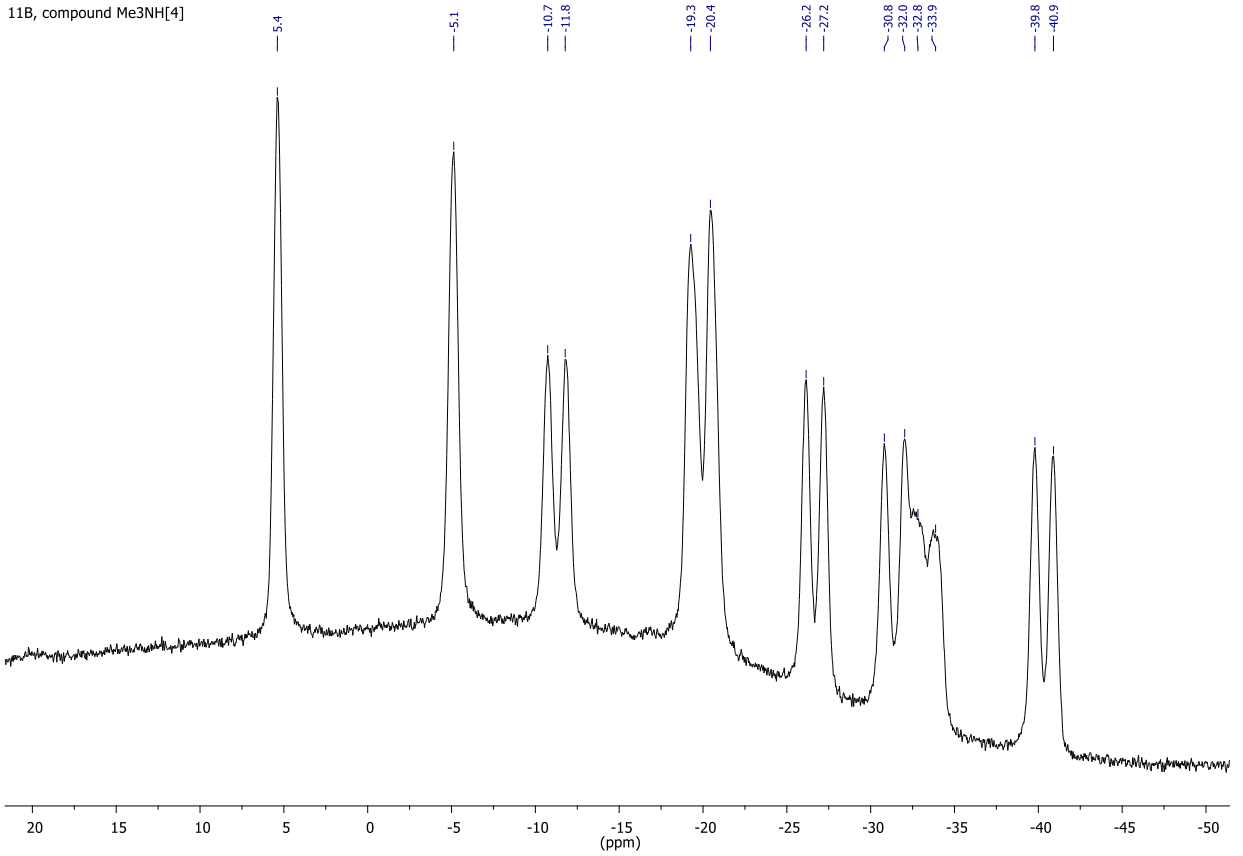


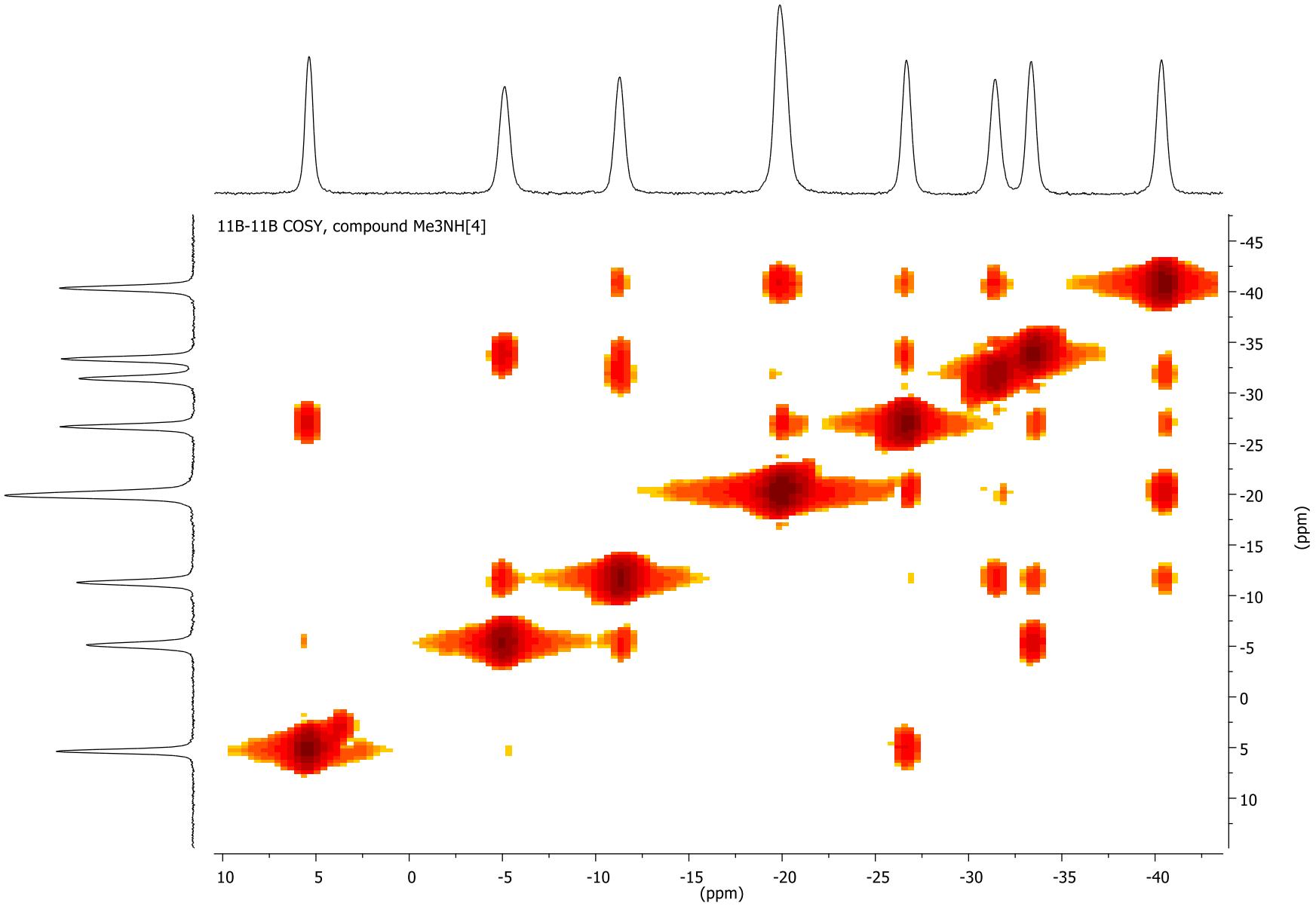

11B-11B COSY, compound Me3NH[3]
6
4
2
0
-2
-4
-6
-8
-12
-16
-20
-24
-28
-32
-36
-40
(ppm)
-40
-35
-30
-25
-20
-15
-10
-5
0
5
10
(ppm)

11B{1H}, compound Me3NH[4]

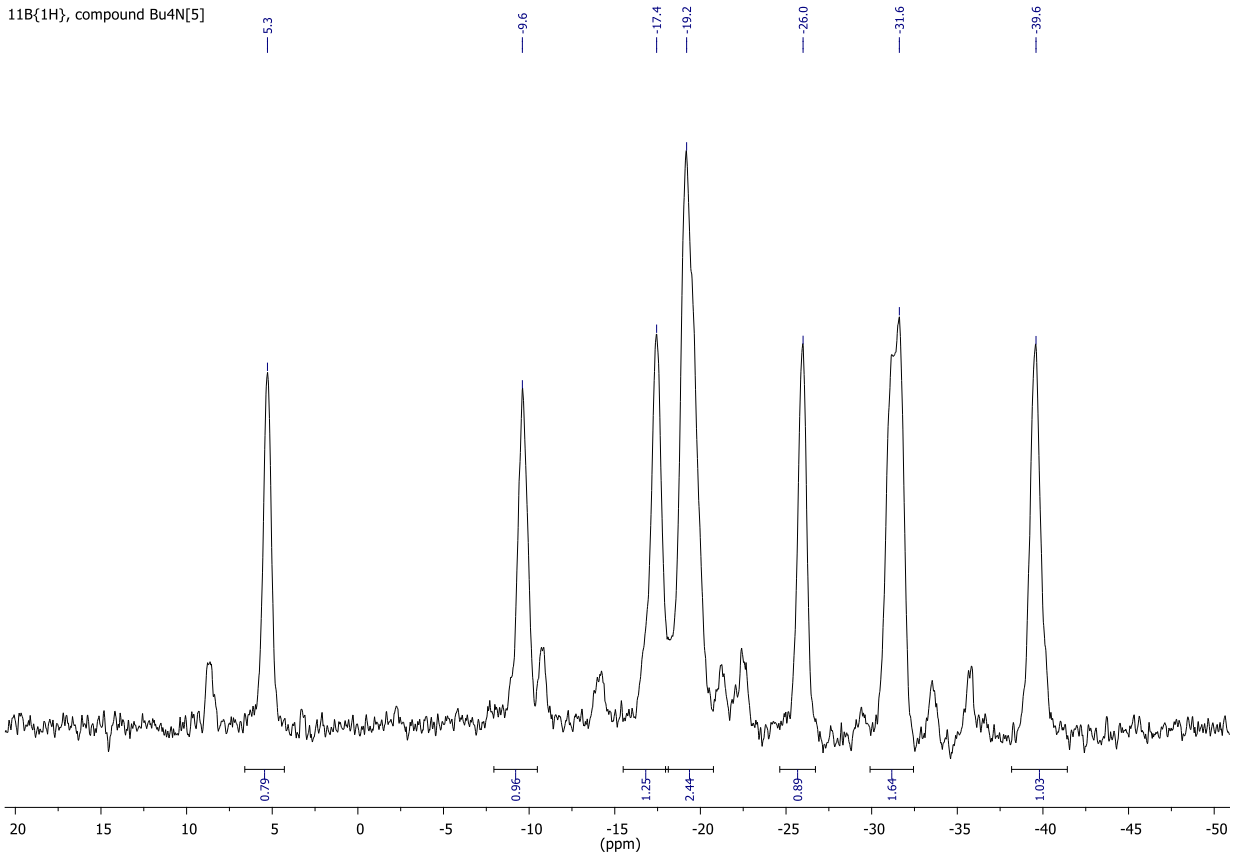


11B, compound Me3NH[4]

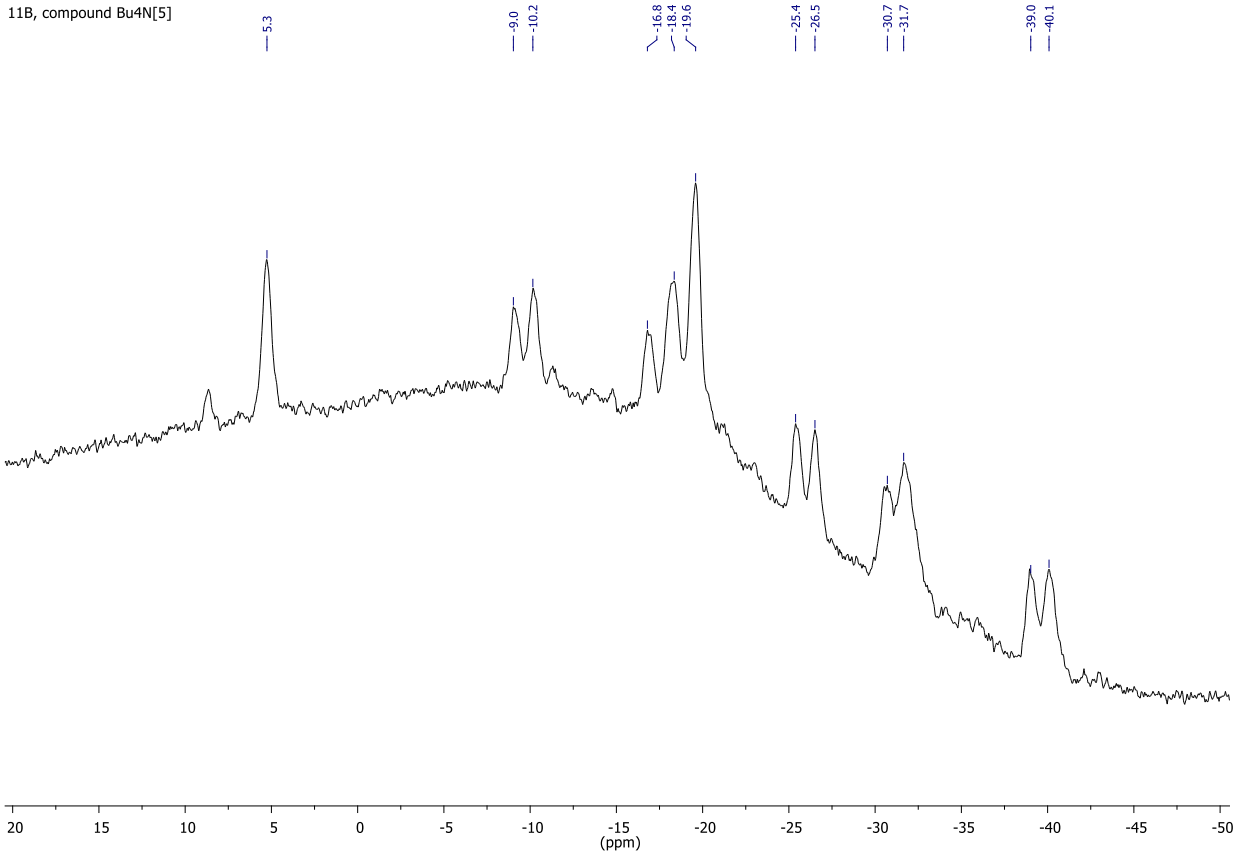


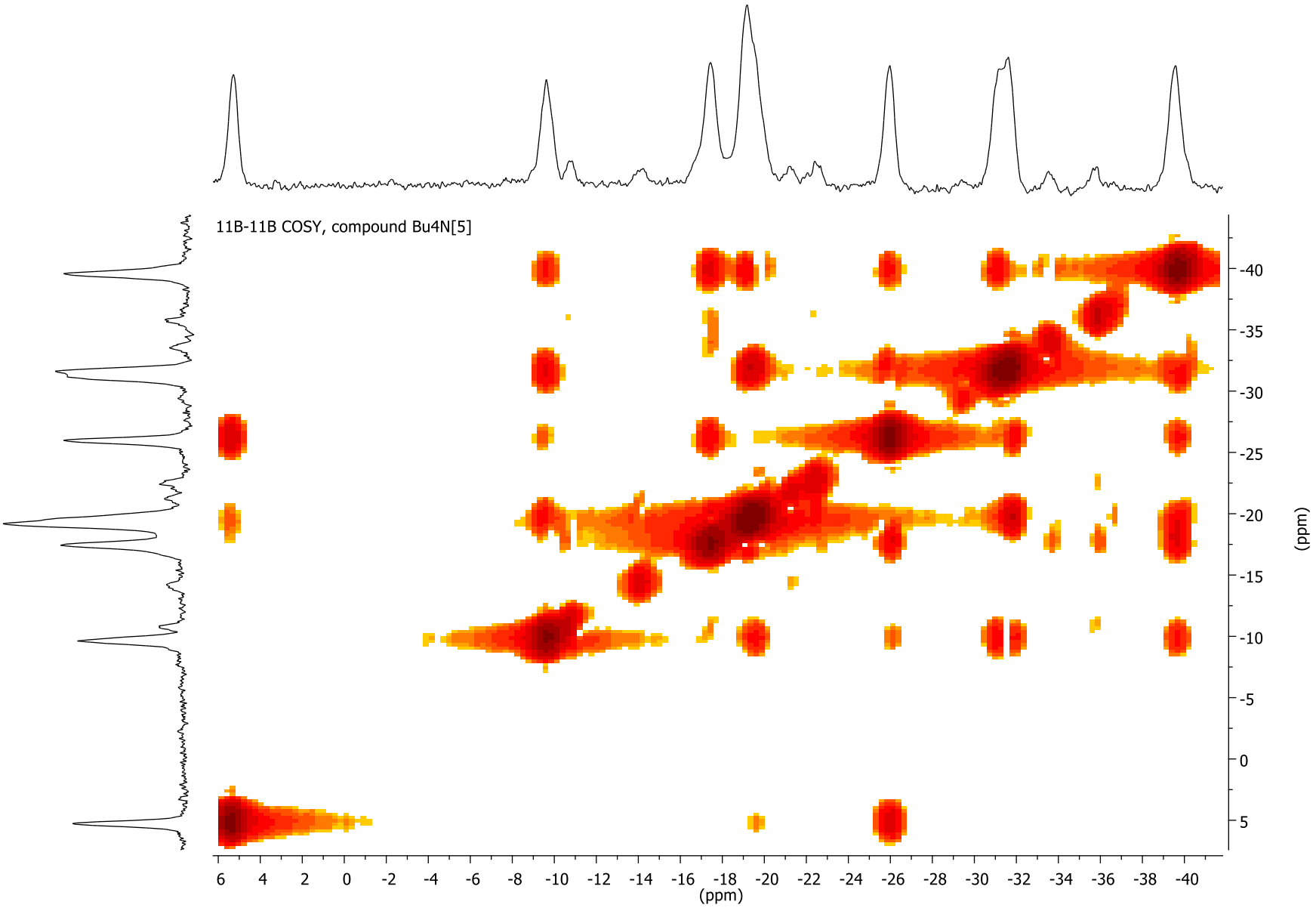

11B-11B COSY, compound Me3NH[4]
10
5
0
-5
-10
-15
-20
-25
-30
-35
-40
(ppm)
-45
-40
-35
-30
-25
-20
-15
-10
-5
0
5
10
(ppm)

11B{1H}, compound Bu4N[5]

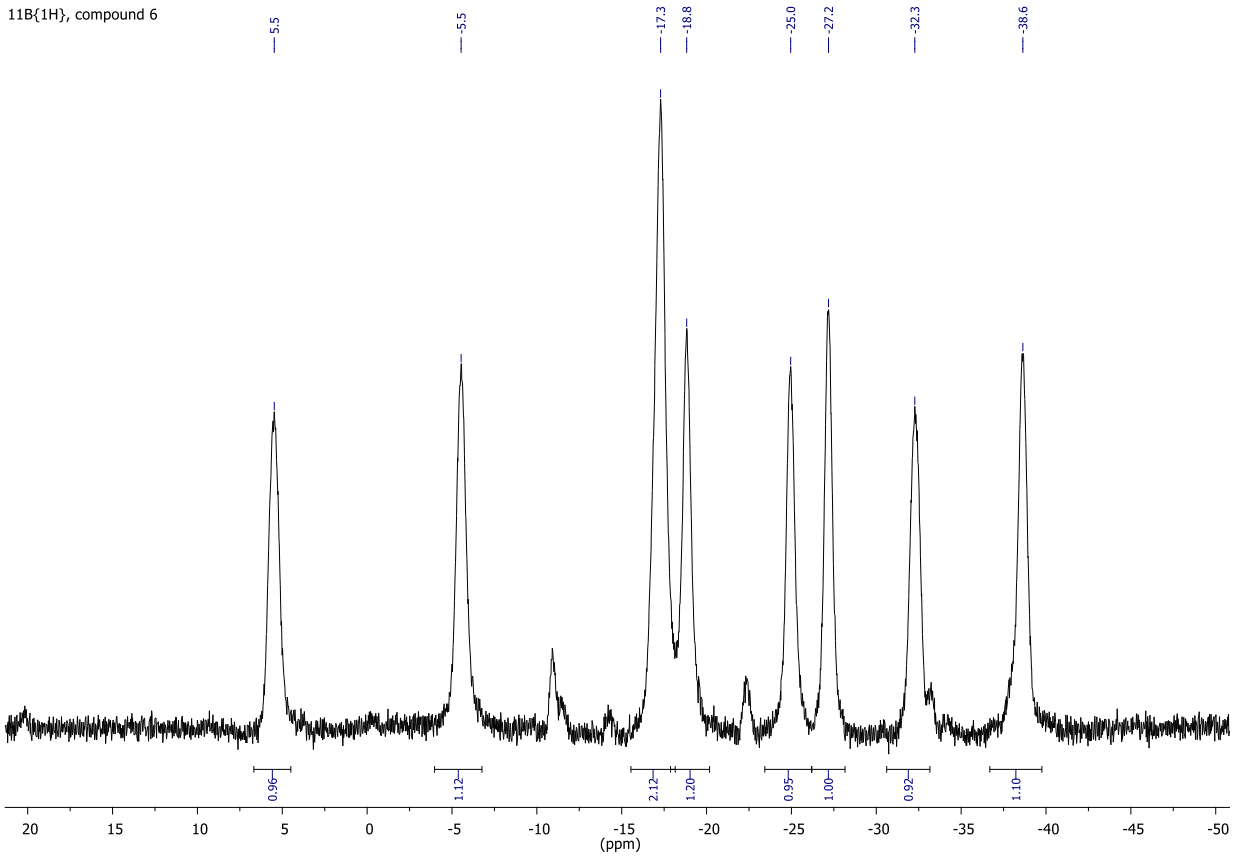


11B, compound Bu4N[5]

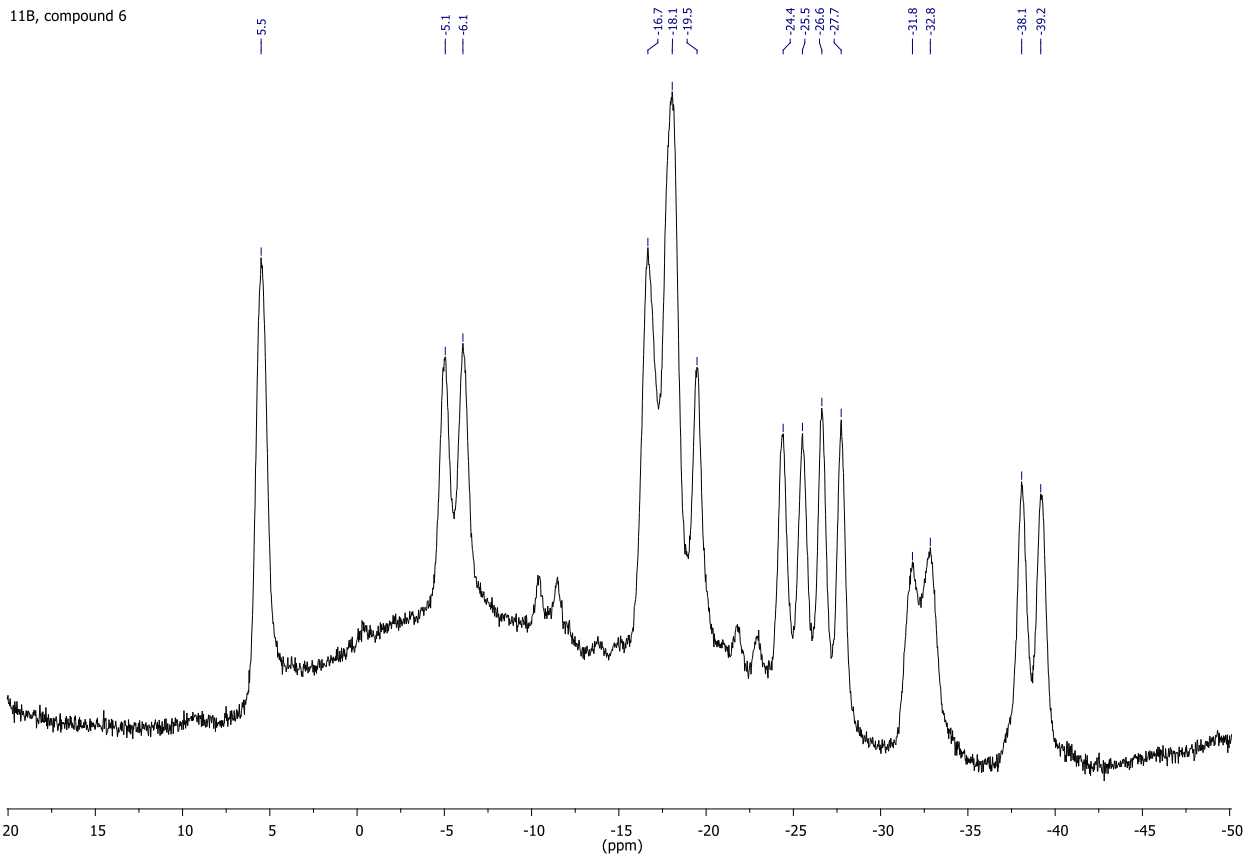



11B-11B COSY, compound Bu4N[5]
6 4 2 0 -2 -4 -6 -8 -10 -12 -14 -16 -18 -20 -22 -24 -26 -28 -30 -32 -34 -36 -38 -40
(ppm)
-40 -35 -30 -25 -20 -15 -10 -5 0 5
(ppm)

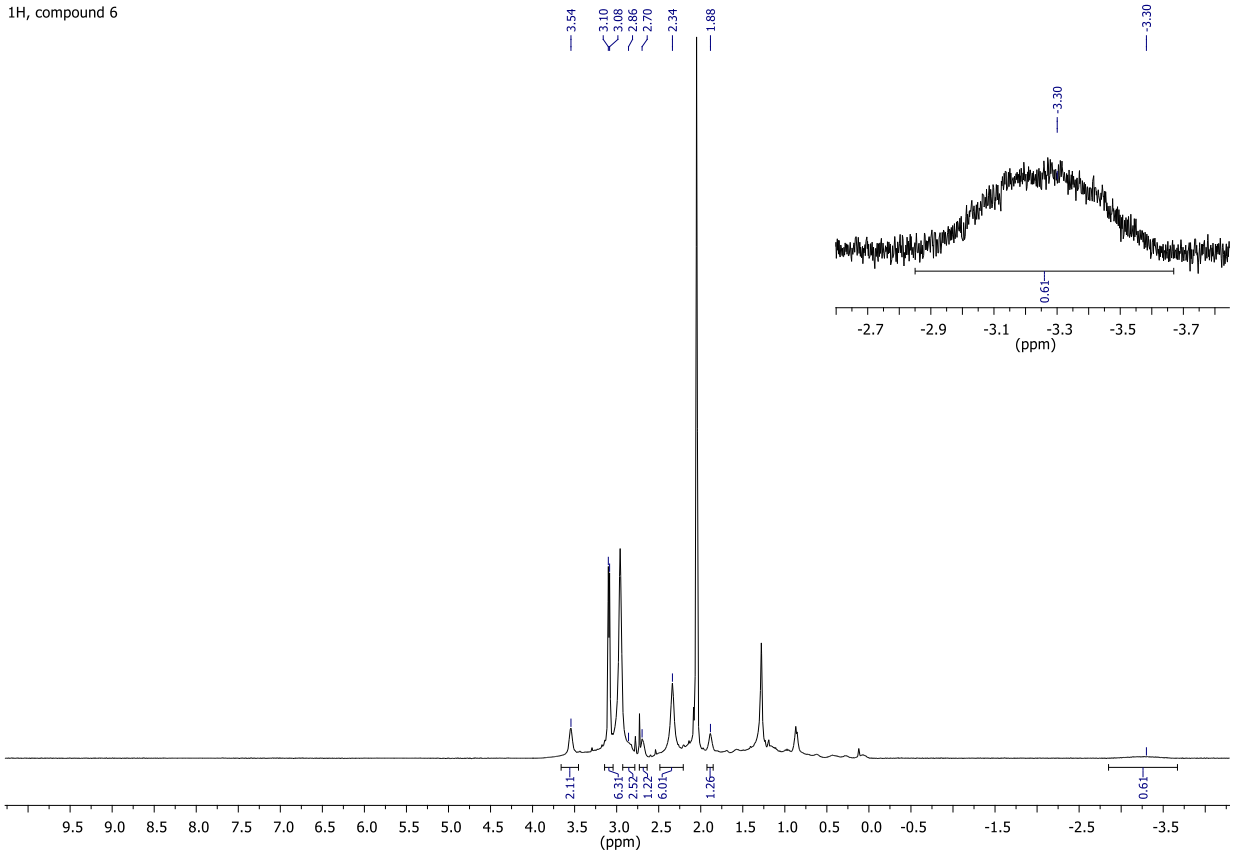
11B{1H}, compound 6



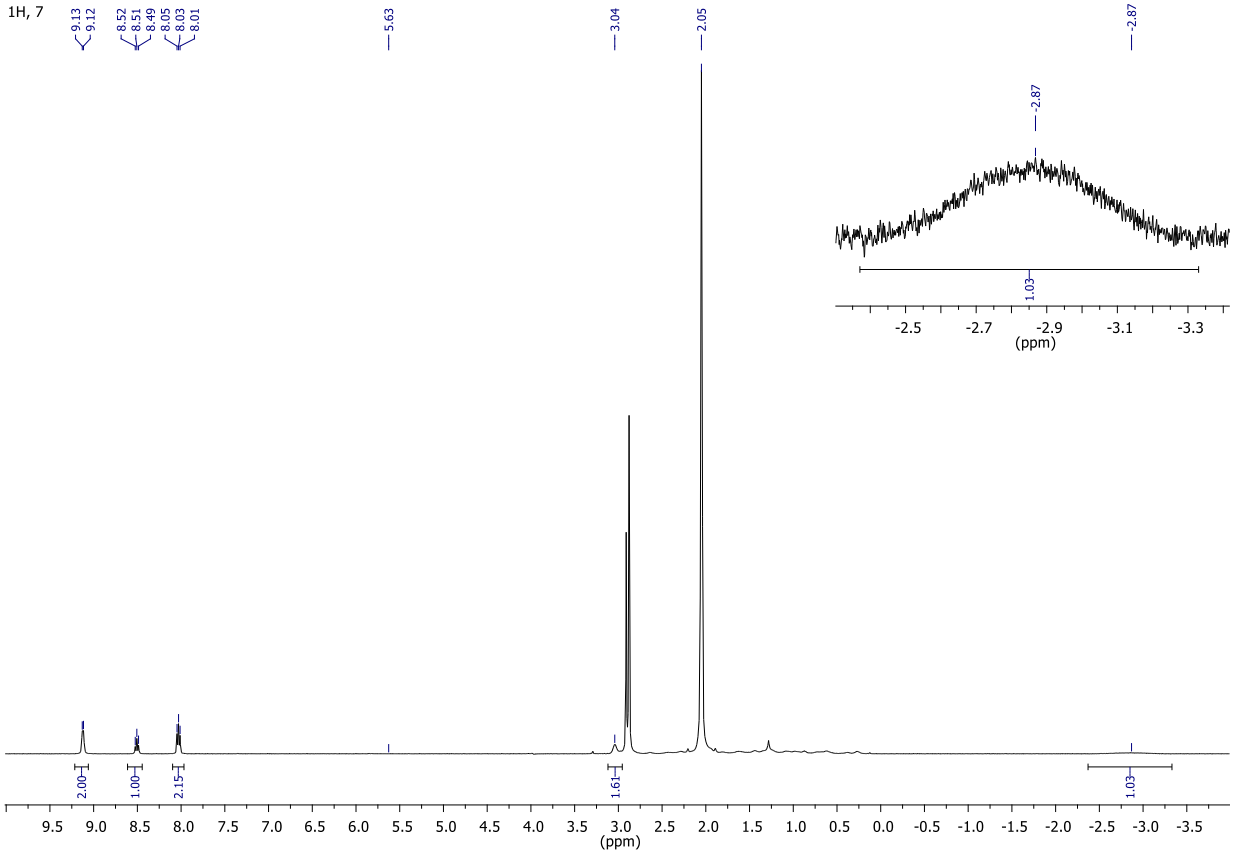
11B, compound 6



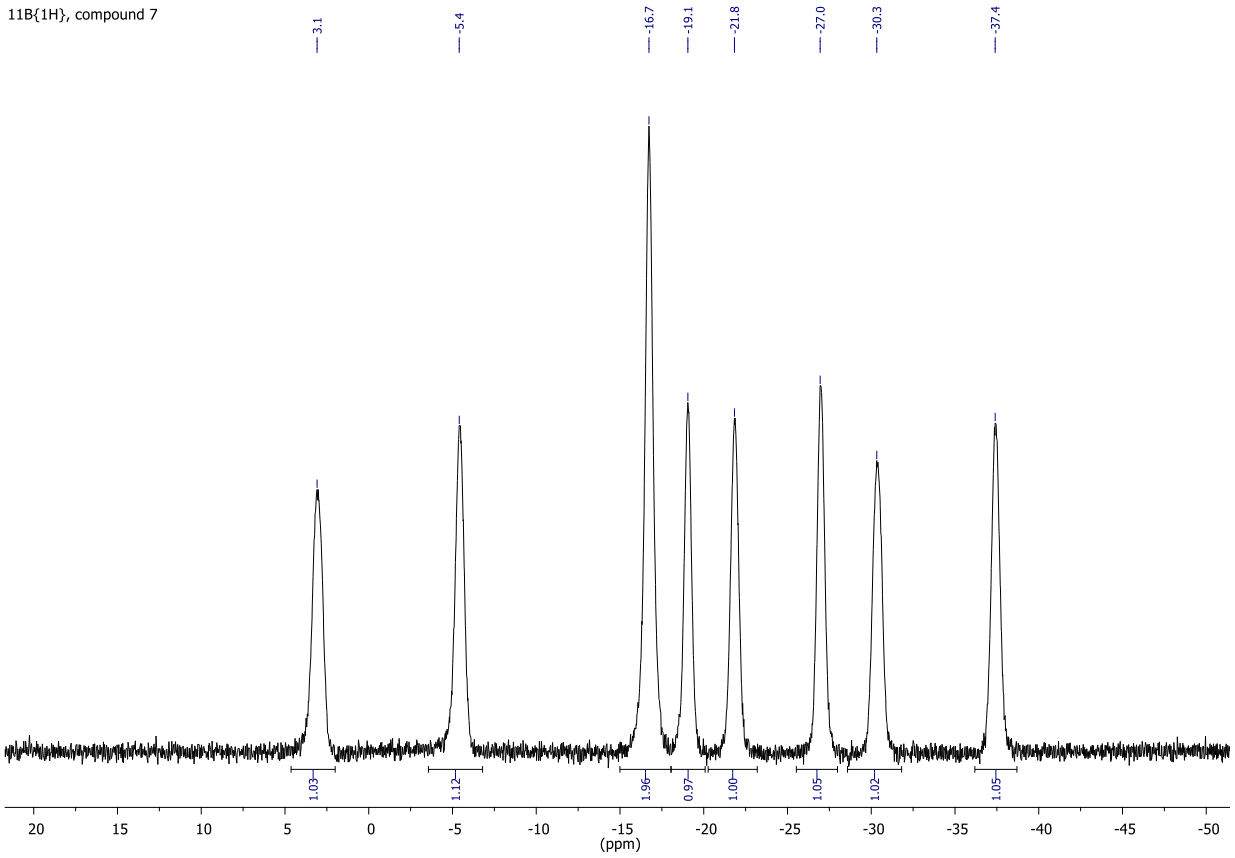
1H, compound 6



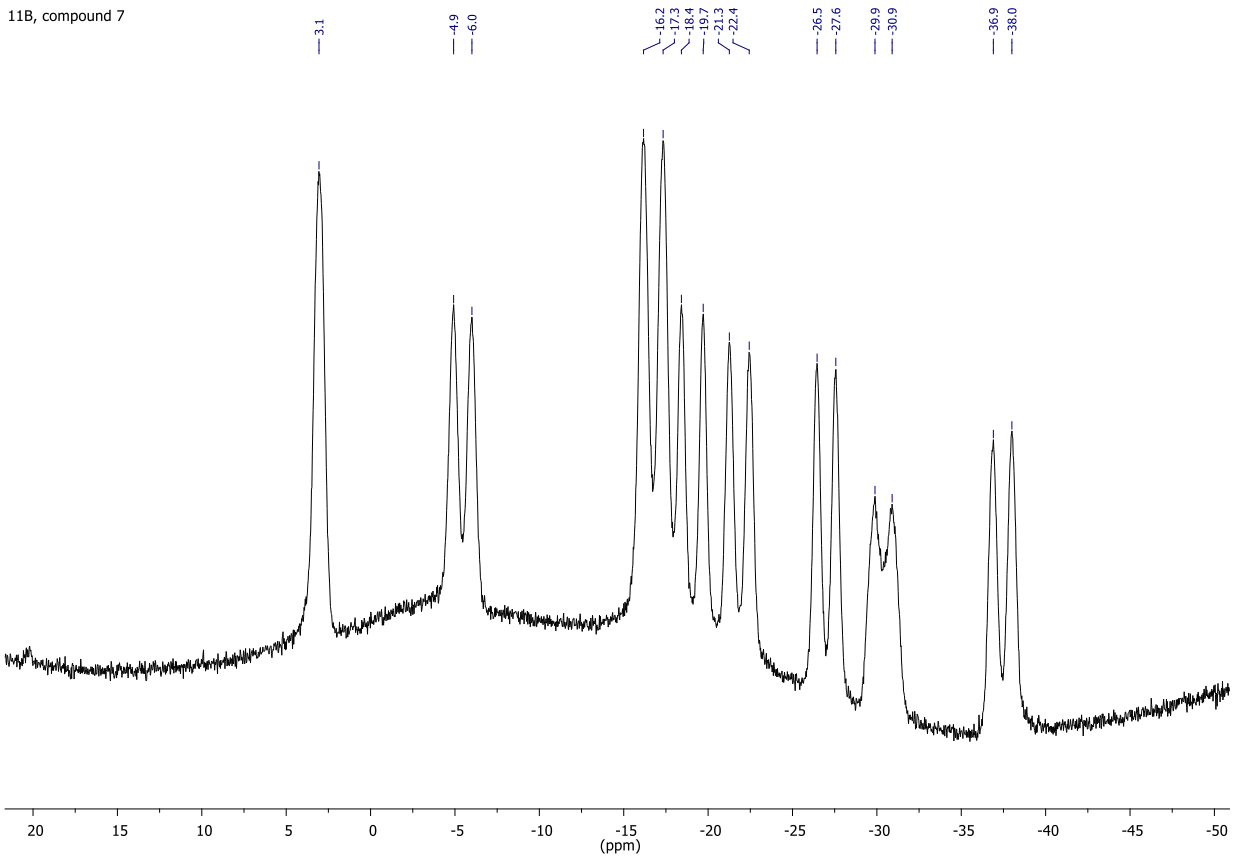
1H, 7



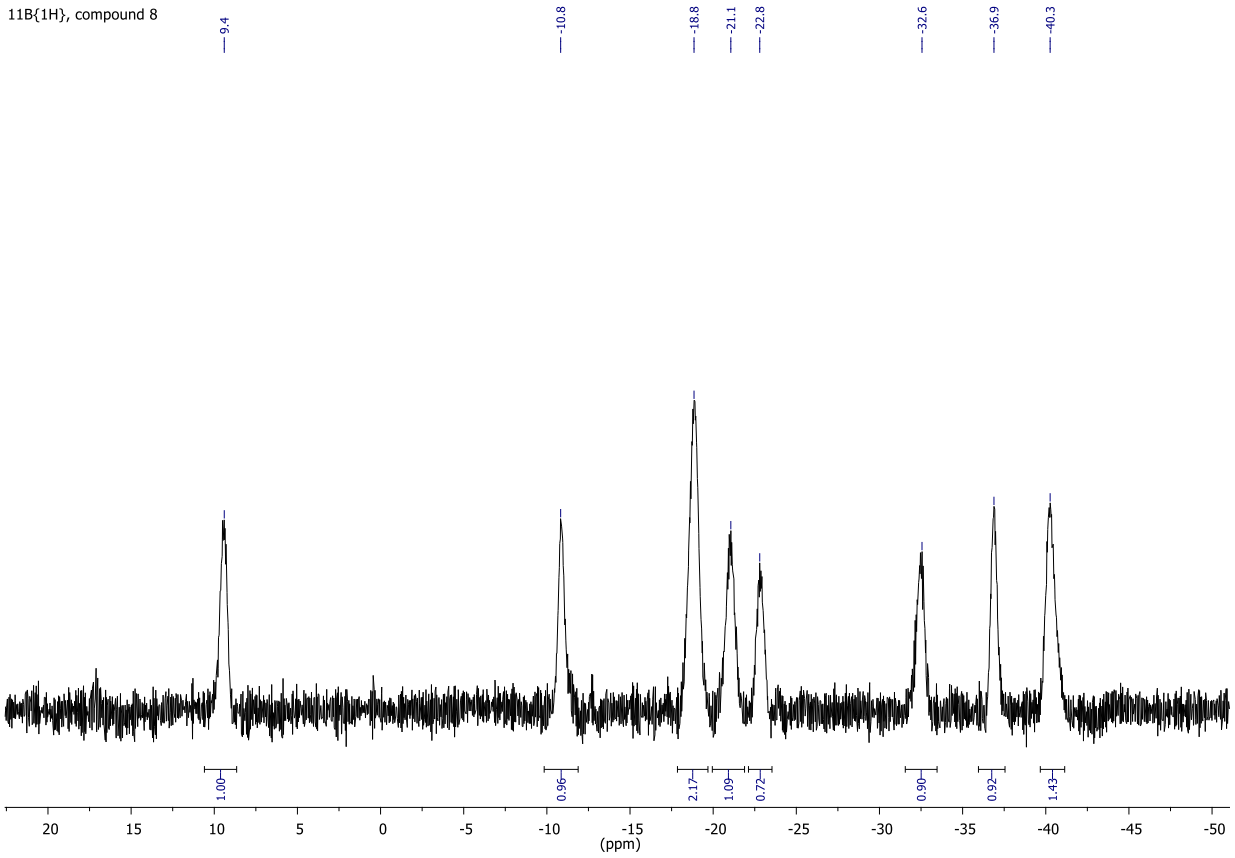
11B{1H}, compound 7



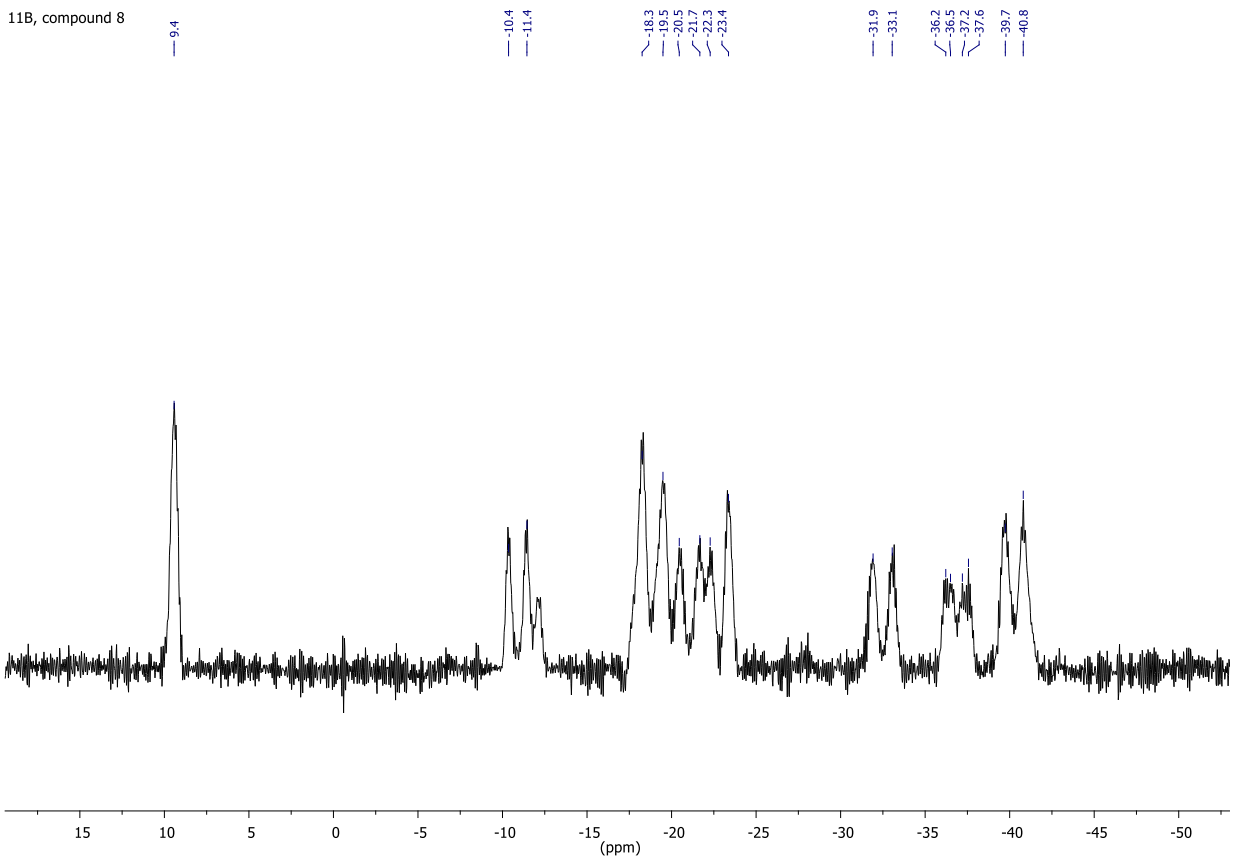
11B, compound 7



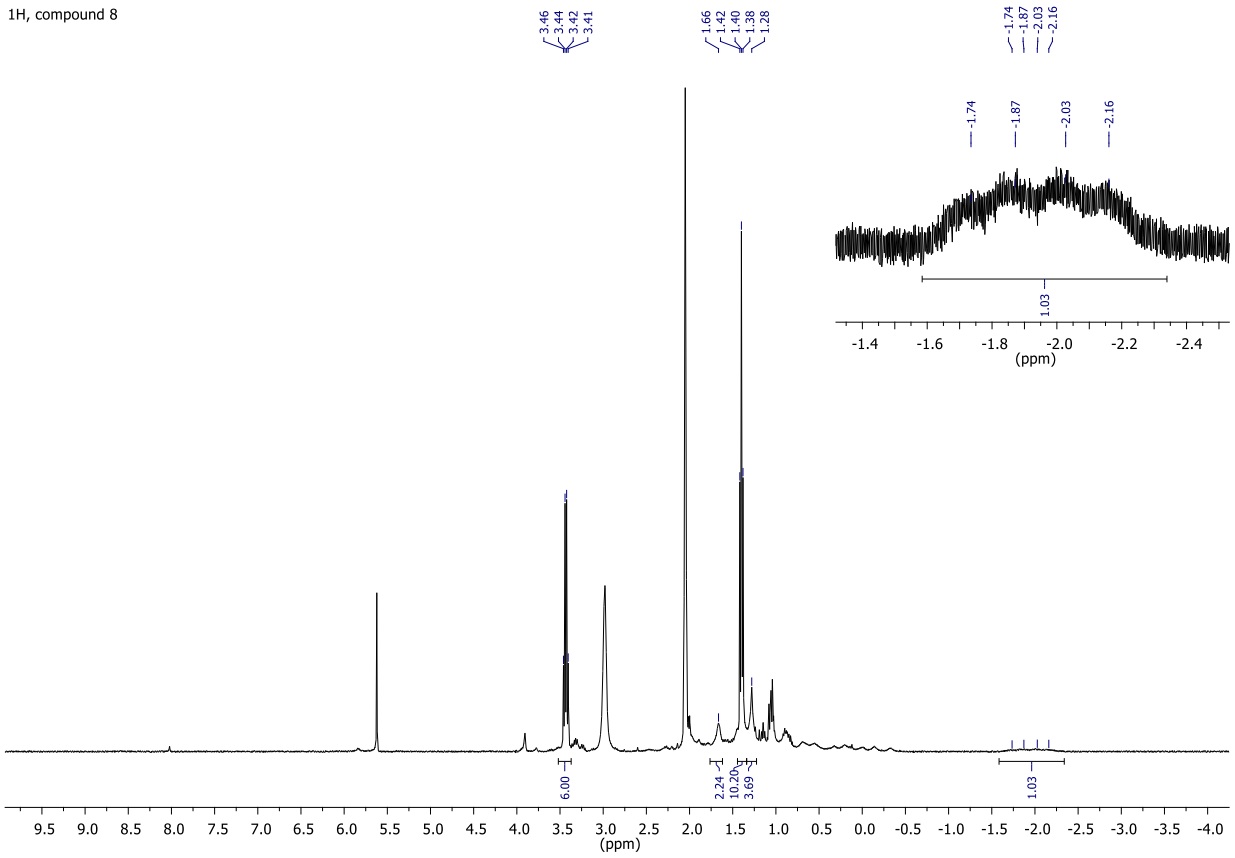
11B{1H}, compound 8



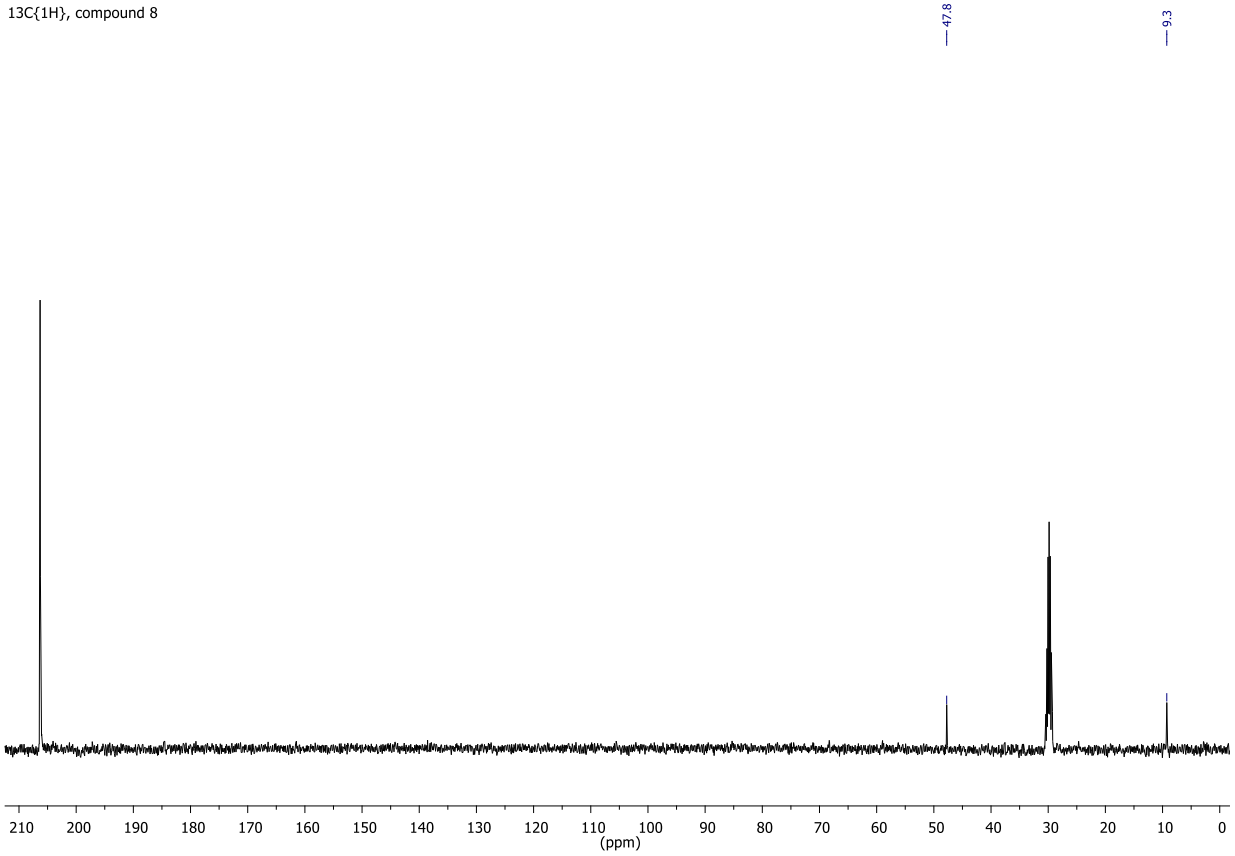
11B, compound 8

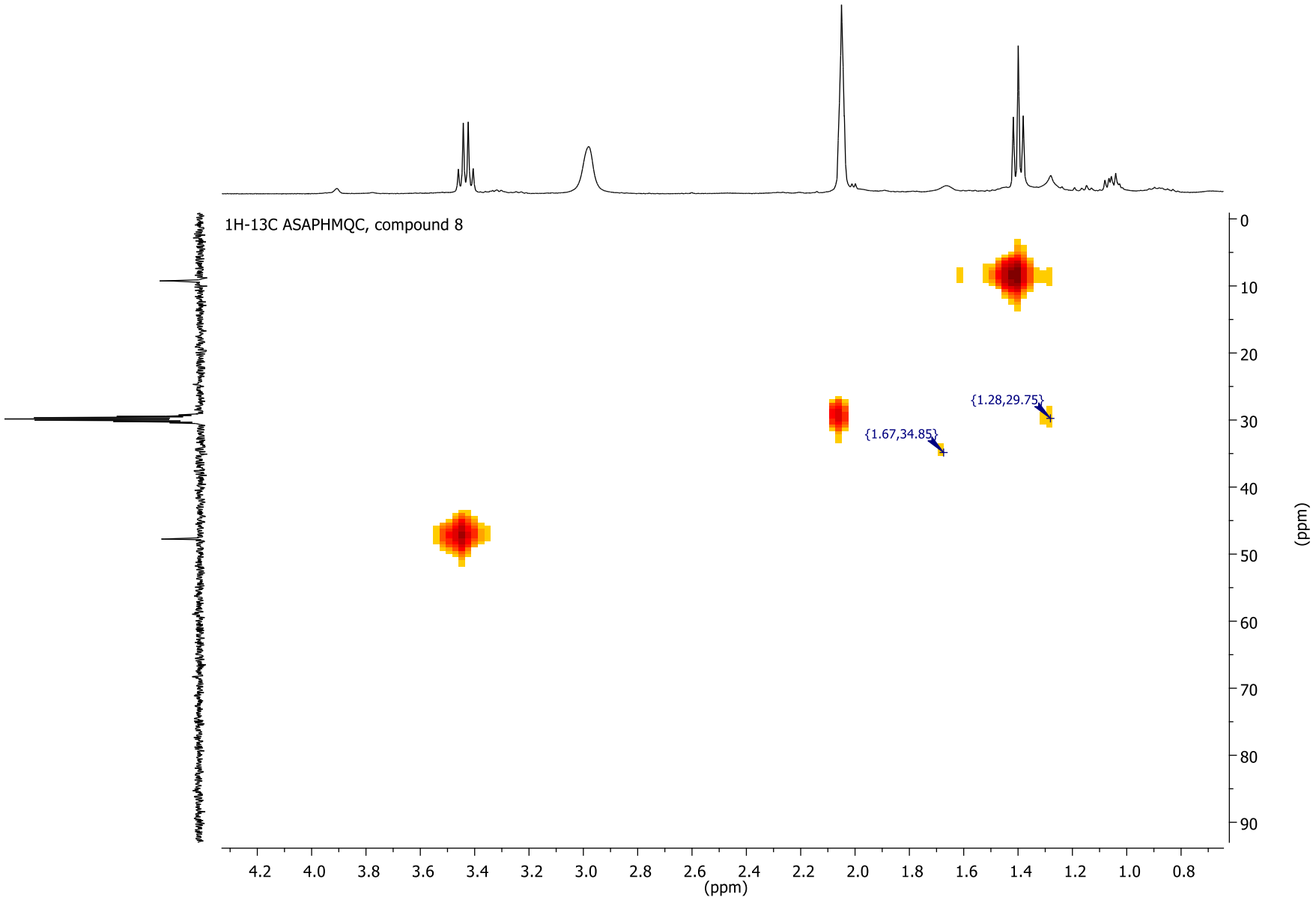


1H, compound 8

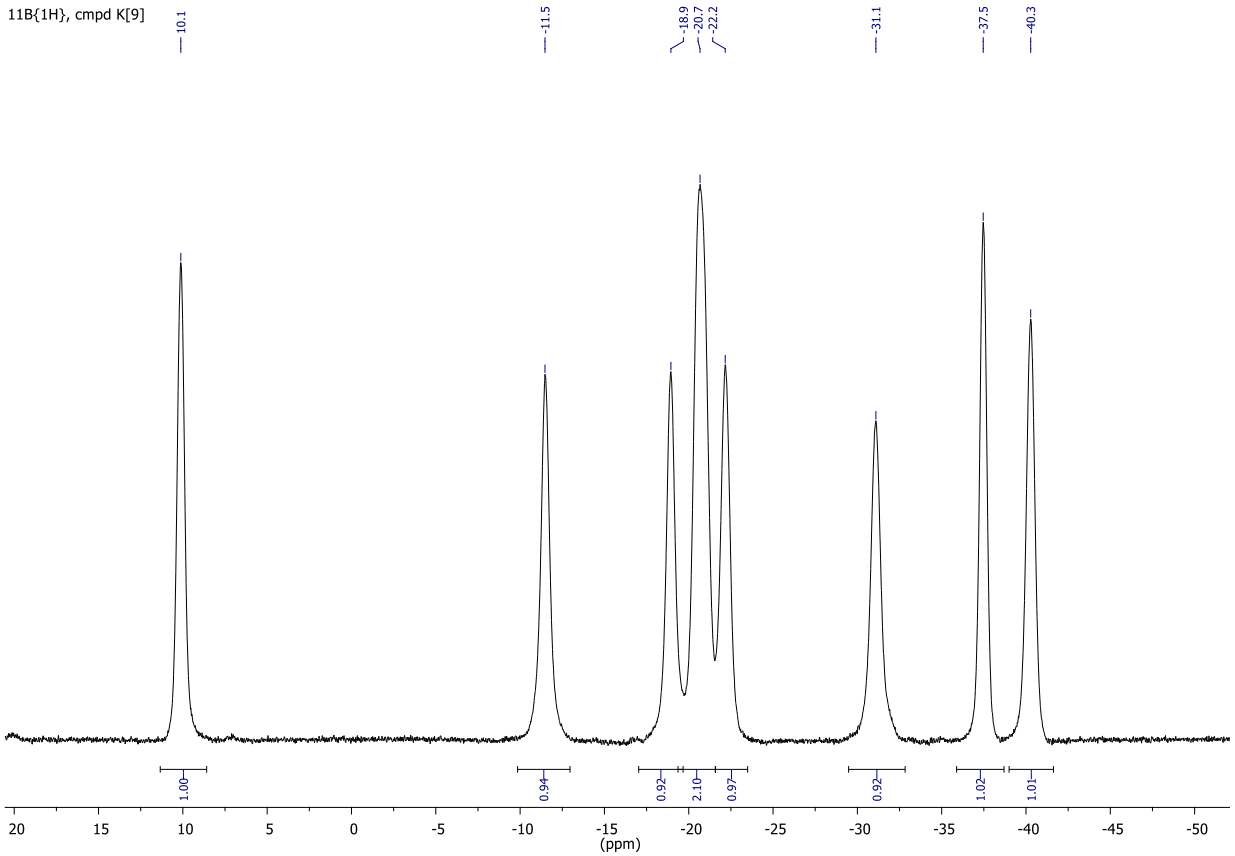


13C{1H}, compound 8

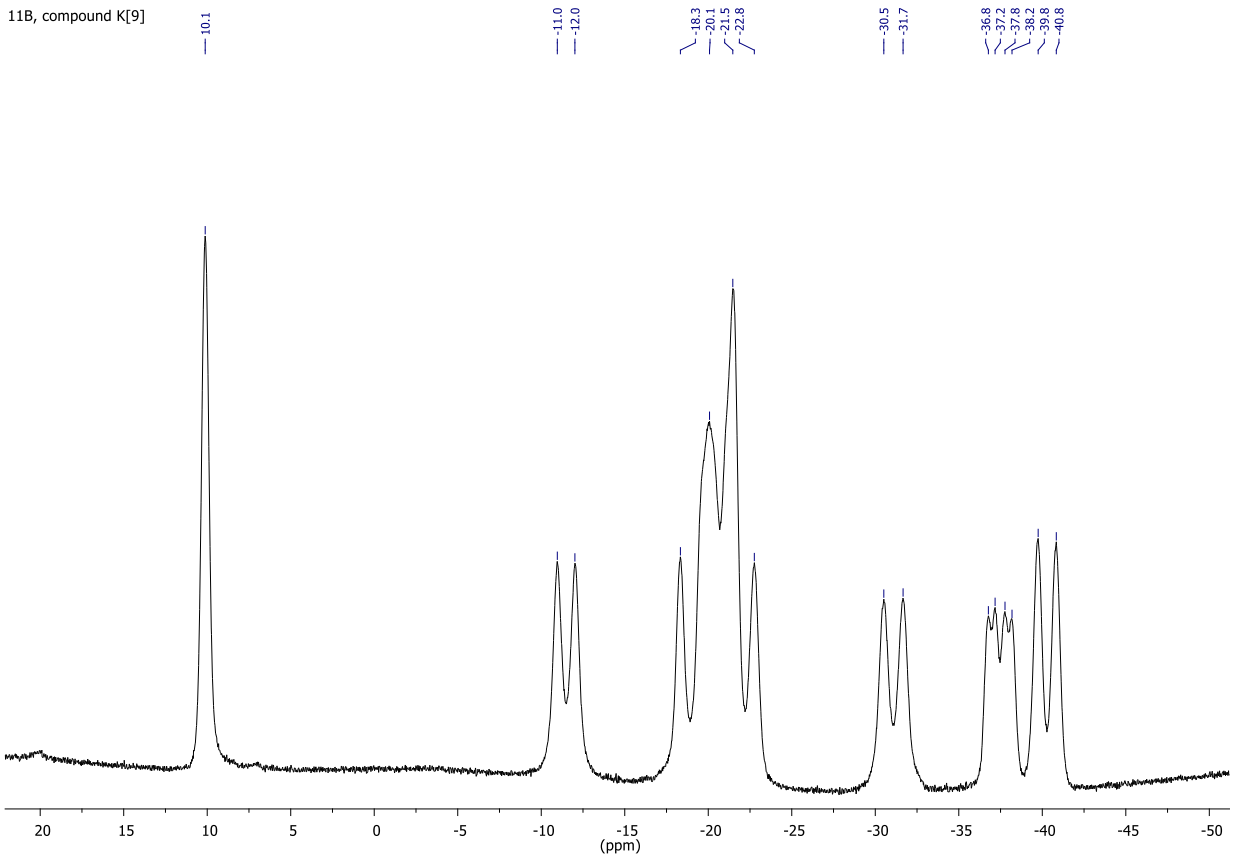


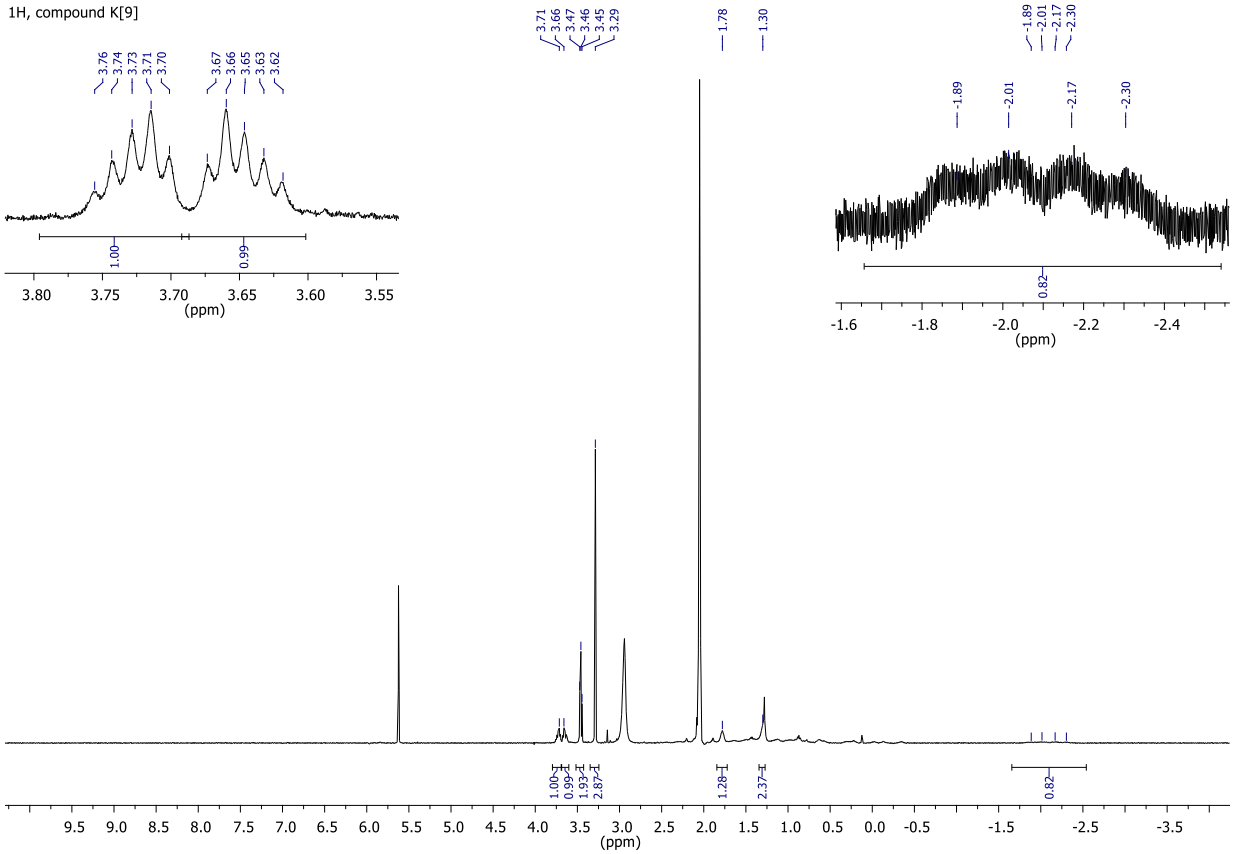

1H-13C ASAPHMQC, compound 8
{1.28,29.75}
{1.67,34.85}
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
(ppm)
0
10
20
30
40
50
60
70
80
90
(ppm)

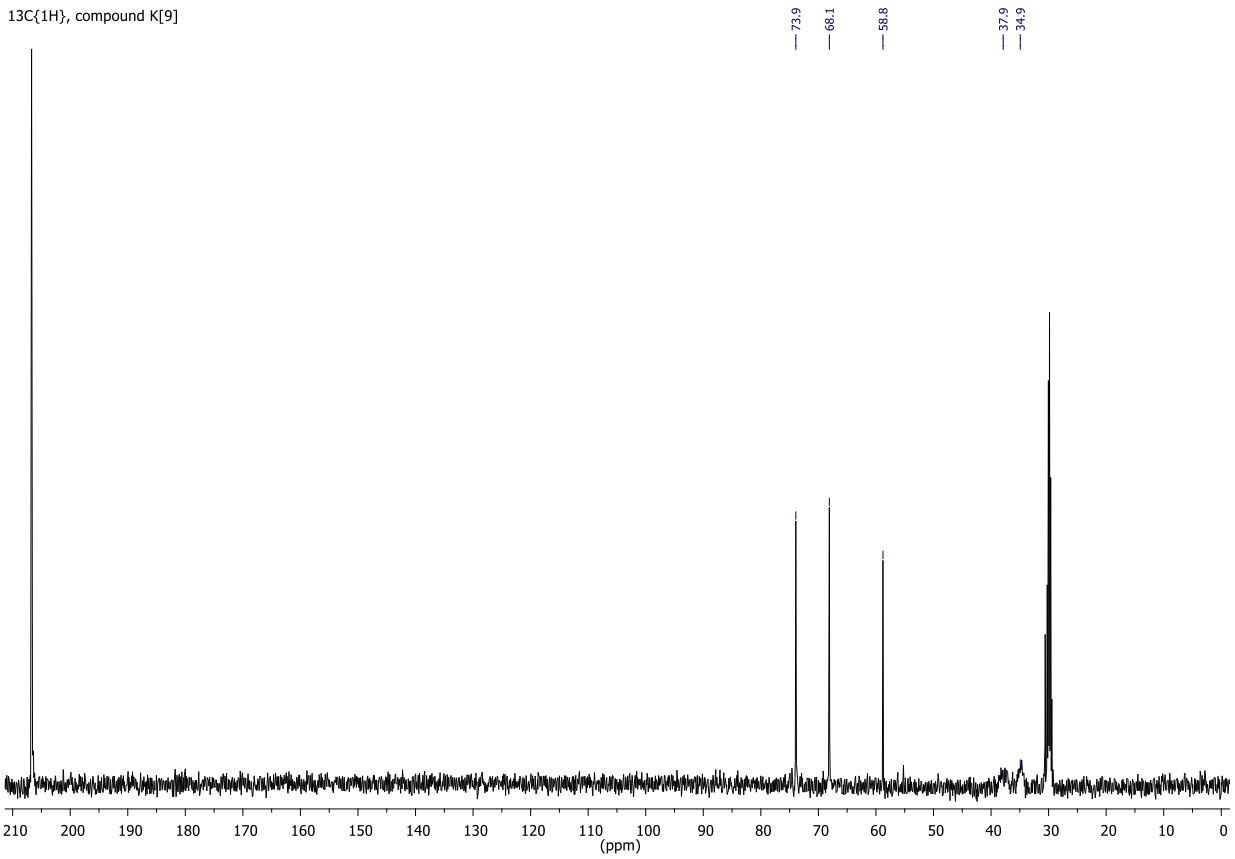
11B{1H}, cmpd K[9]

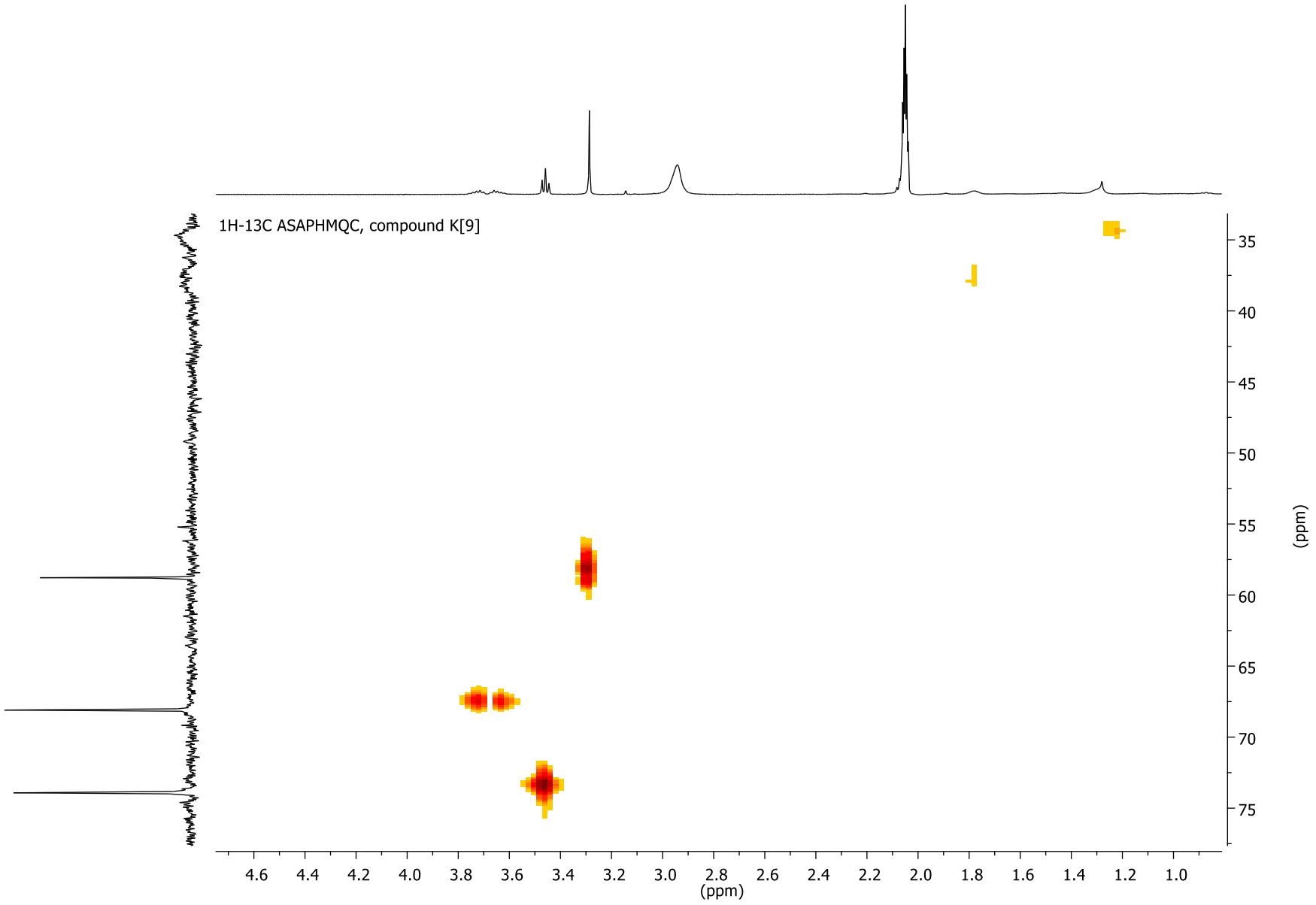


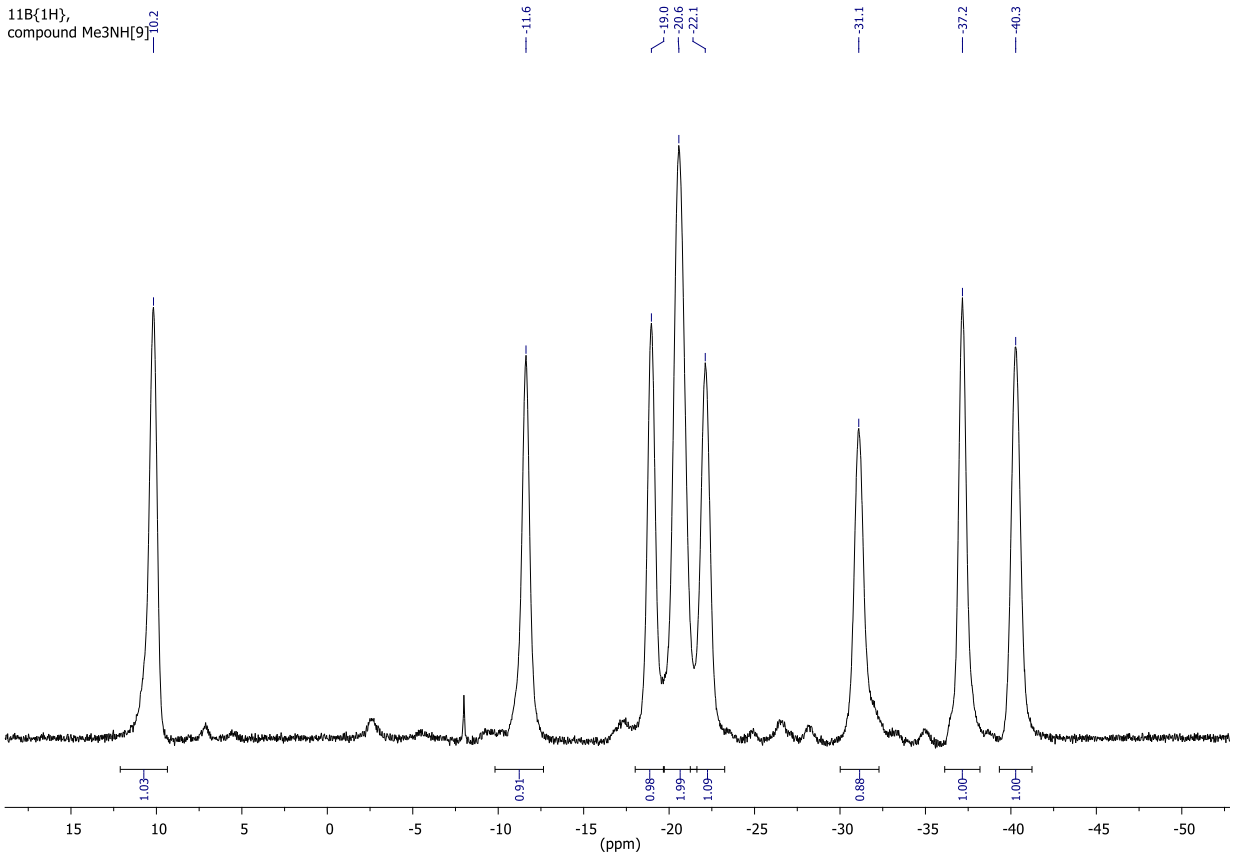
11B, compound K[9]

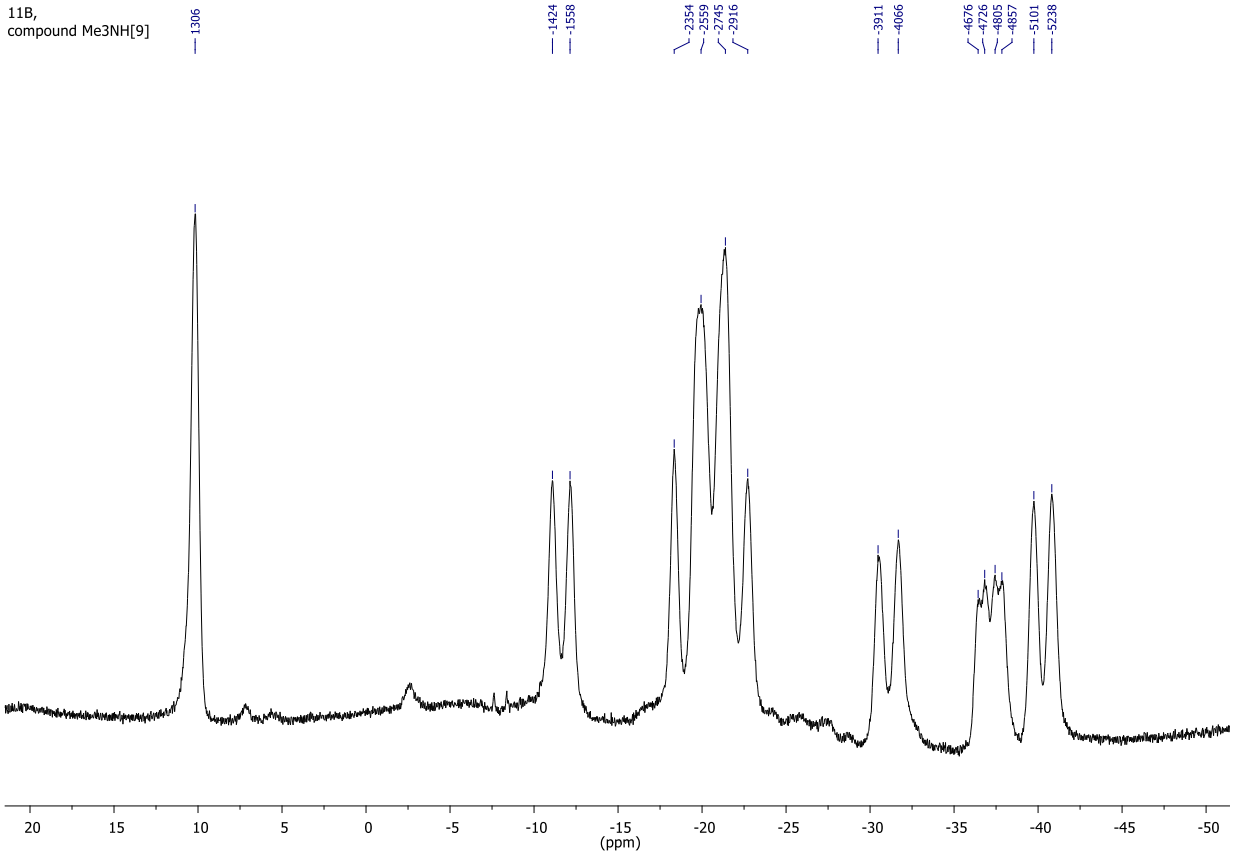



1H, compound K[9]
3.76
3.74
3.73
3.71
3.70
3.67
3.66
3.65
3.63
3.62
1.00
0.99
3.80
3.75
3.70
3.65
3.60
3.55
(ppm)
3.71
3.66
3.47
3.46
3.45
3.29
1.78
1.30
1.89
2.01
2.17
2.30
0.82
-1.6
-1.8
-2.0
-2.2
-2.4
(ppm)
1.00
0.99
1.93
2.87
1.28
2.37
0.82
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.5
-2.5
-3.5
(ppm)

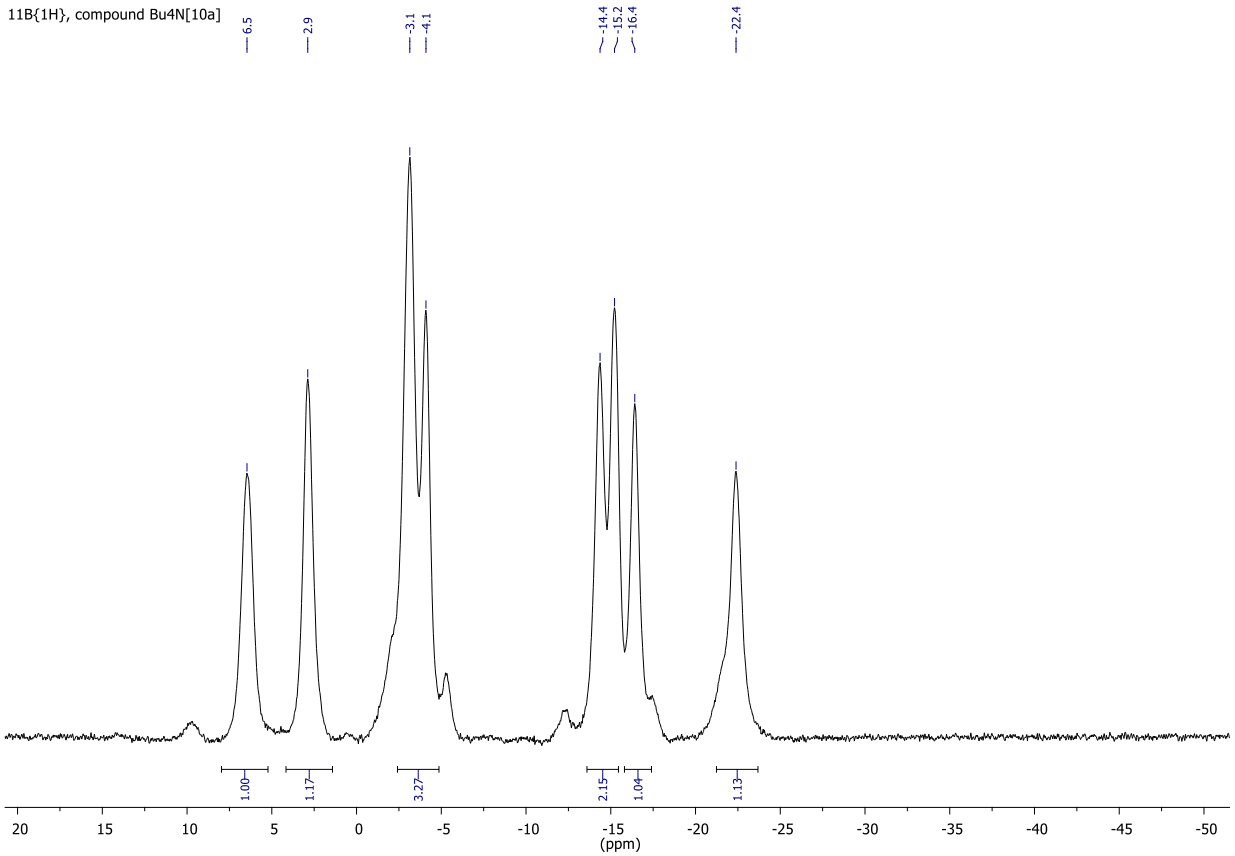

13C{1H}, compound K[9]
73.9
68.1
58.8
37.9
34.9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
(ppm)


1H-13C ASAPHMQC, compound K[9]
4.6
4.4
4.2
4.0
3.8
3.6
3.4
3.2
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
(ppm)
35
40
45
50
55
60
65
70
75
(ppm)

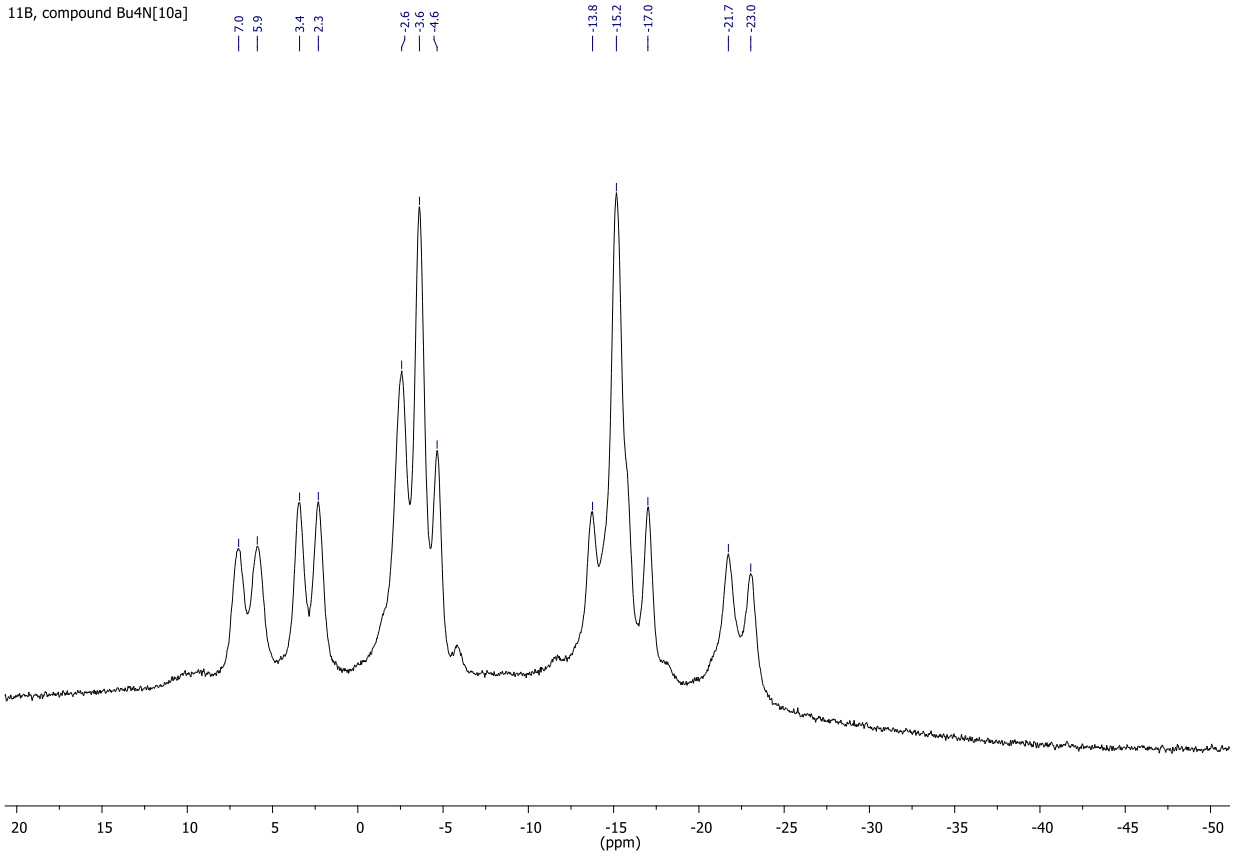

11B{1H},
compound Me3NH[9]
10.2
-11.6
-19.0
-20.6
-22.1
-31.1
-37.2
-40.3
1.03
0.91
0.98
1.99
1.09
0.88
1.00
1.00
15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)


11B,
compound Me3NH[9]
1306
-1424
-1558
-2354
-2559
-2745
-2916
-3911
-4066
-4676
-4726
-4805
-4857
-5101
-5238
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

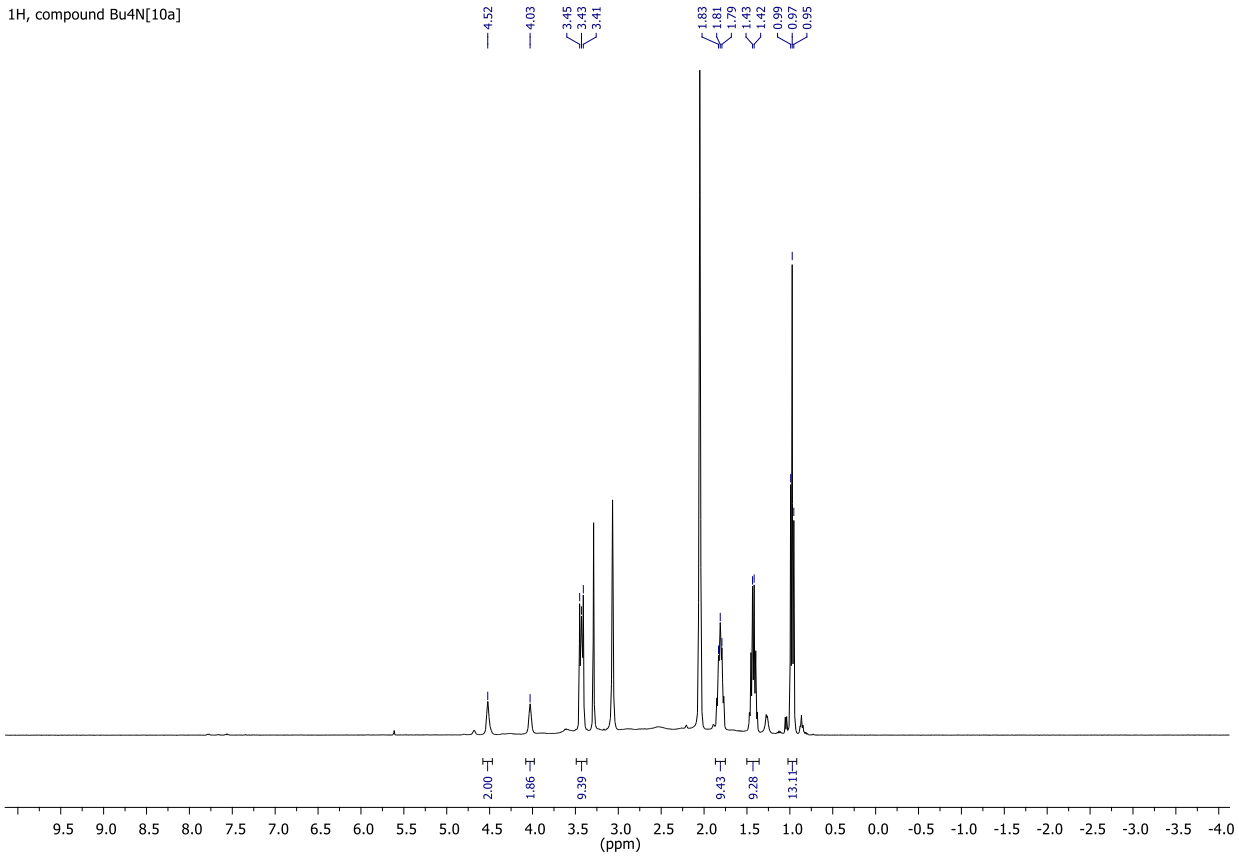
11B{1H}, compound Bu4N[10a]



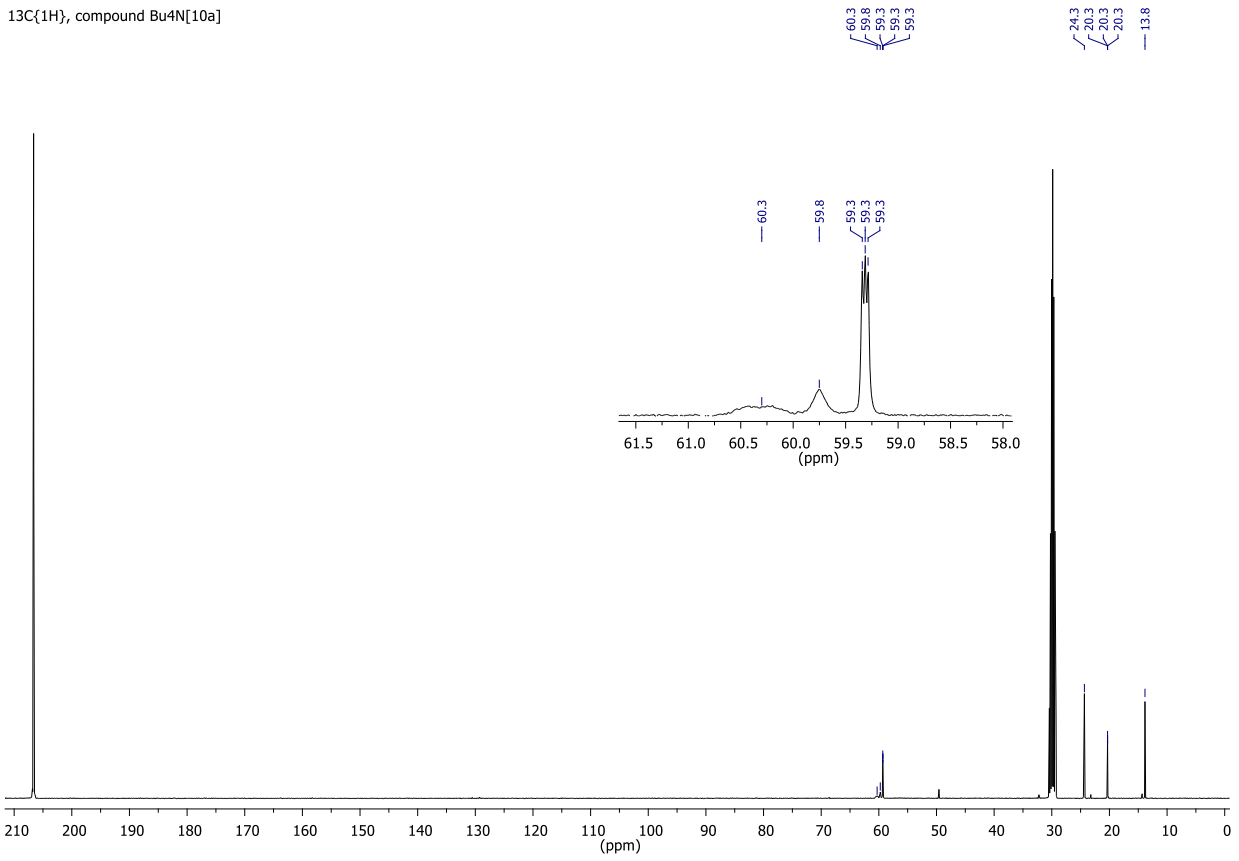
11B, compound Bu4N[10a]

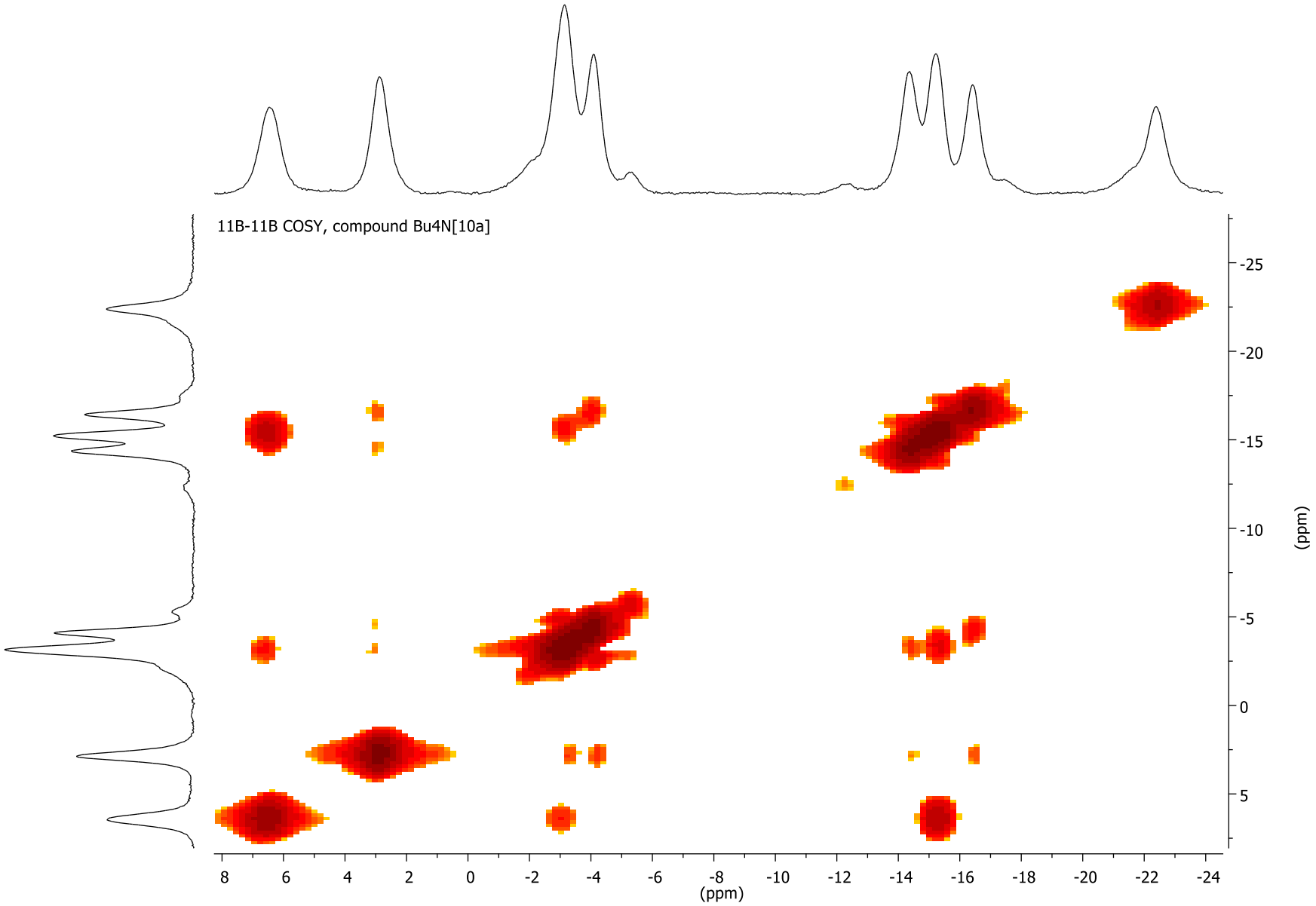


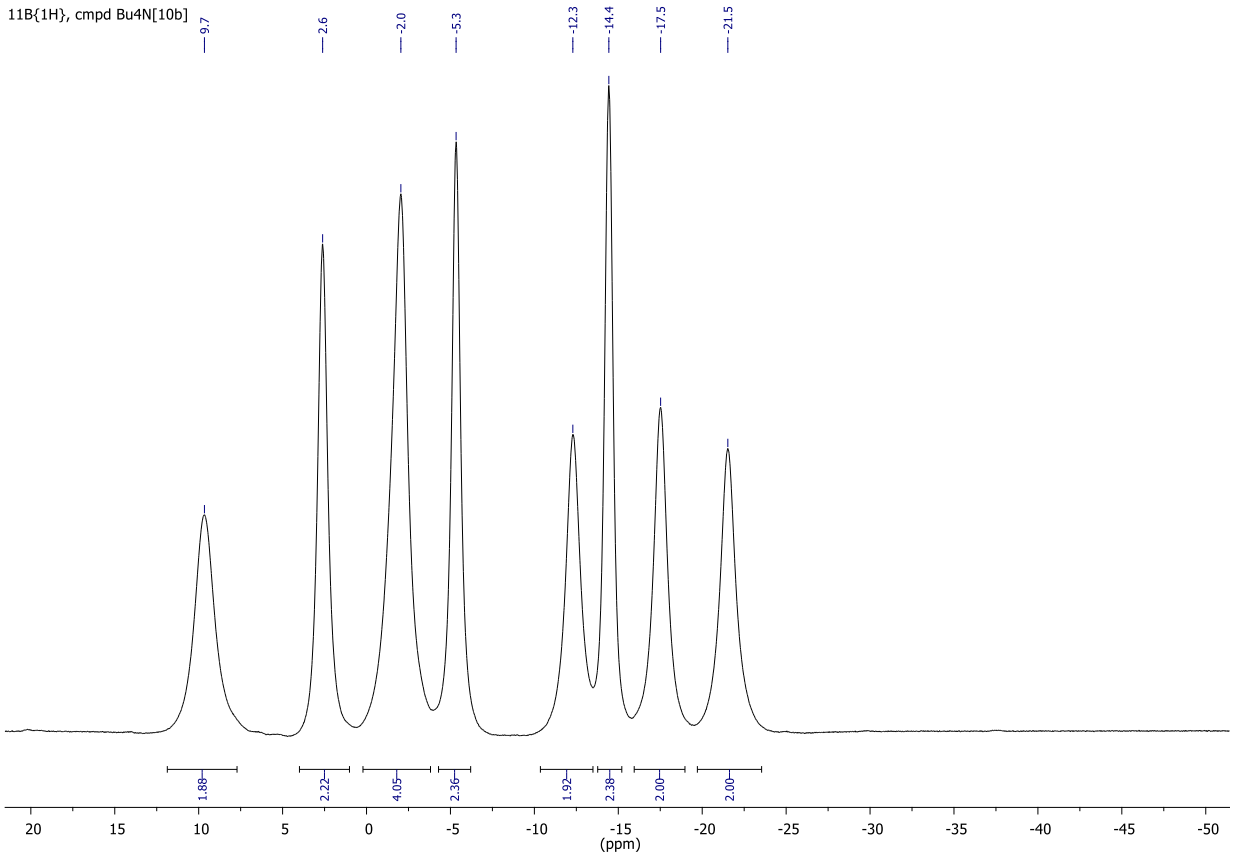
1H, compound Bu4N[10a]

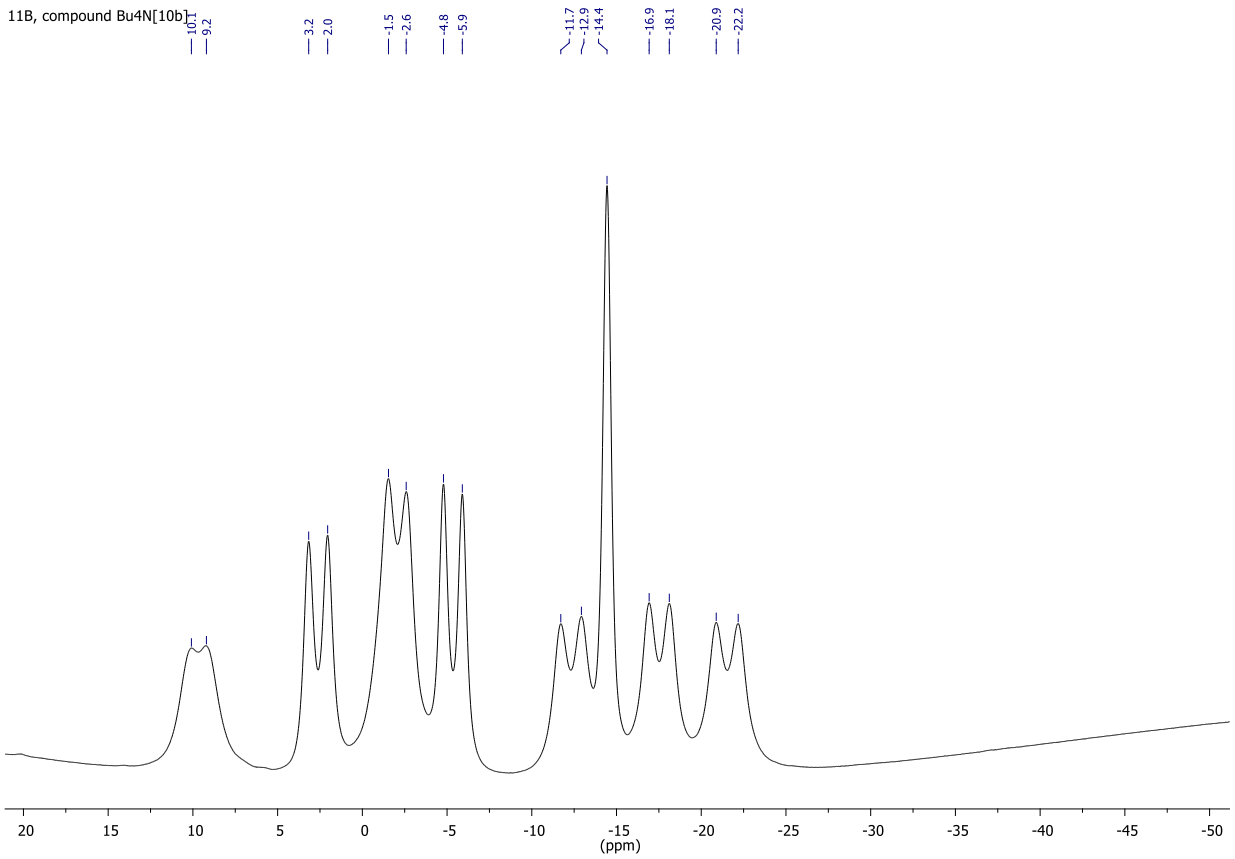


13C{1H}, compound Bu4N[10a]

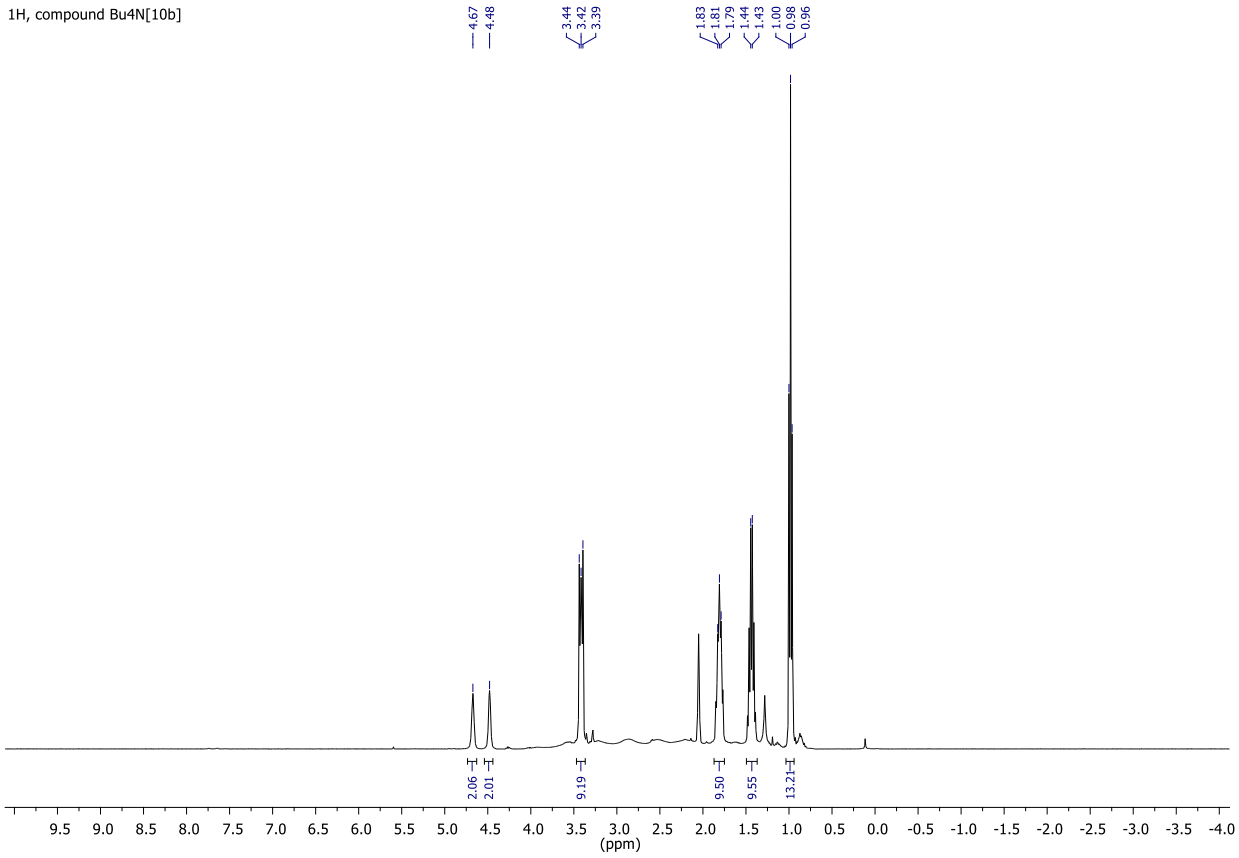



11B-11B COSY, compound Bu4N[10a]
8
6
4
2
0
-2
-4
-6
-8
-10
-12
-14
-16
-18
-20
-22
-24
(ppm)
-25
-20
-15
-10
-5
0
5
(ppm)

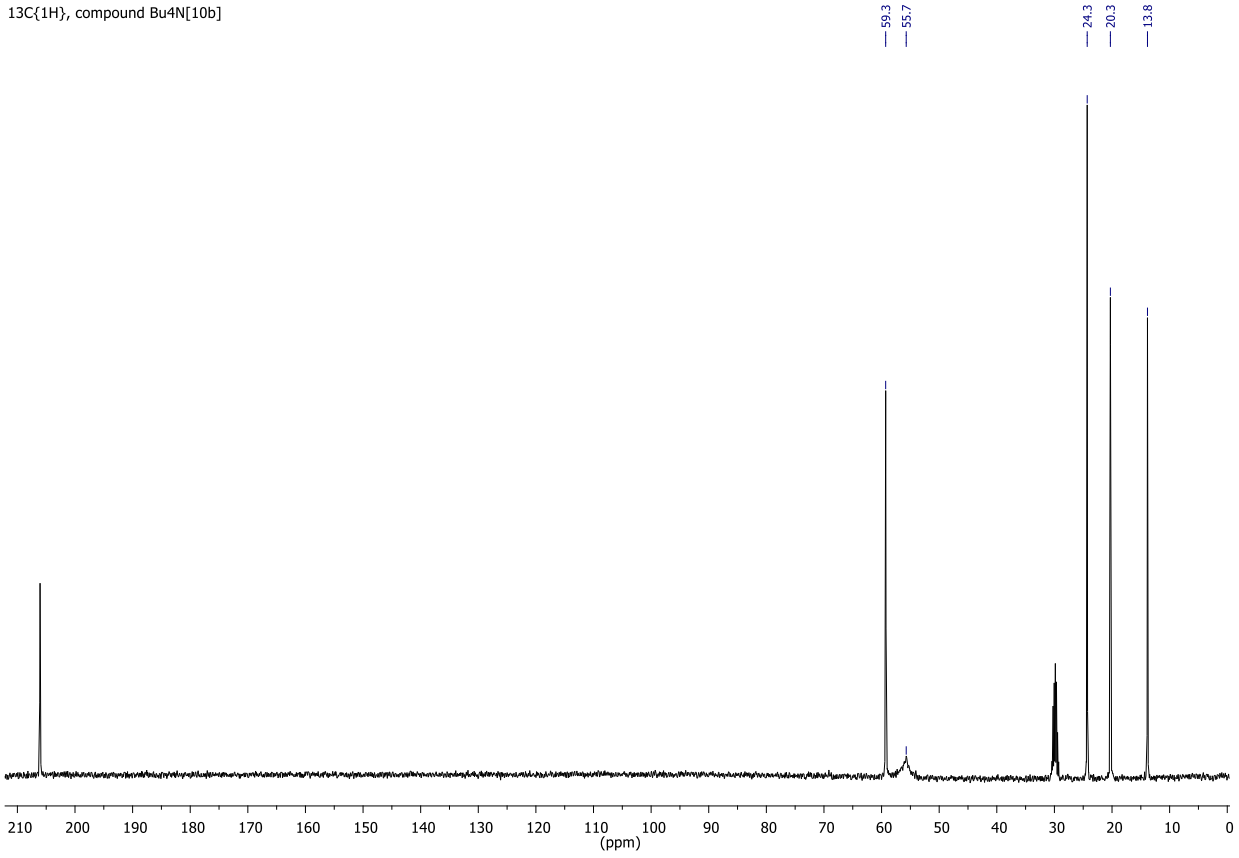

11B{1H}, cmpd Bu4N[10b]
9.7
2.6
-2.0
-5.3
-12.3
-14.4
-17.5
-21.5
1.88
2.22
4.05
2.36
1.92
2.38
2.00
2.00
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

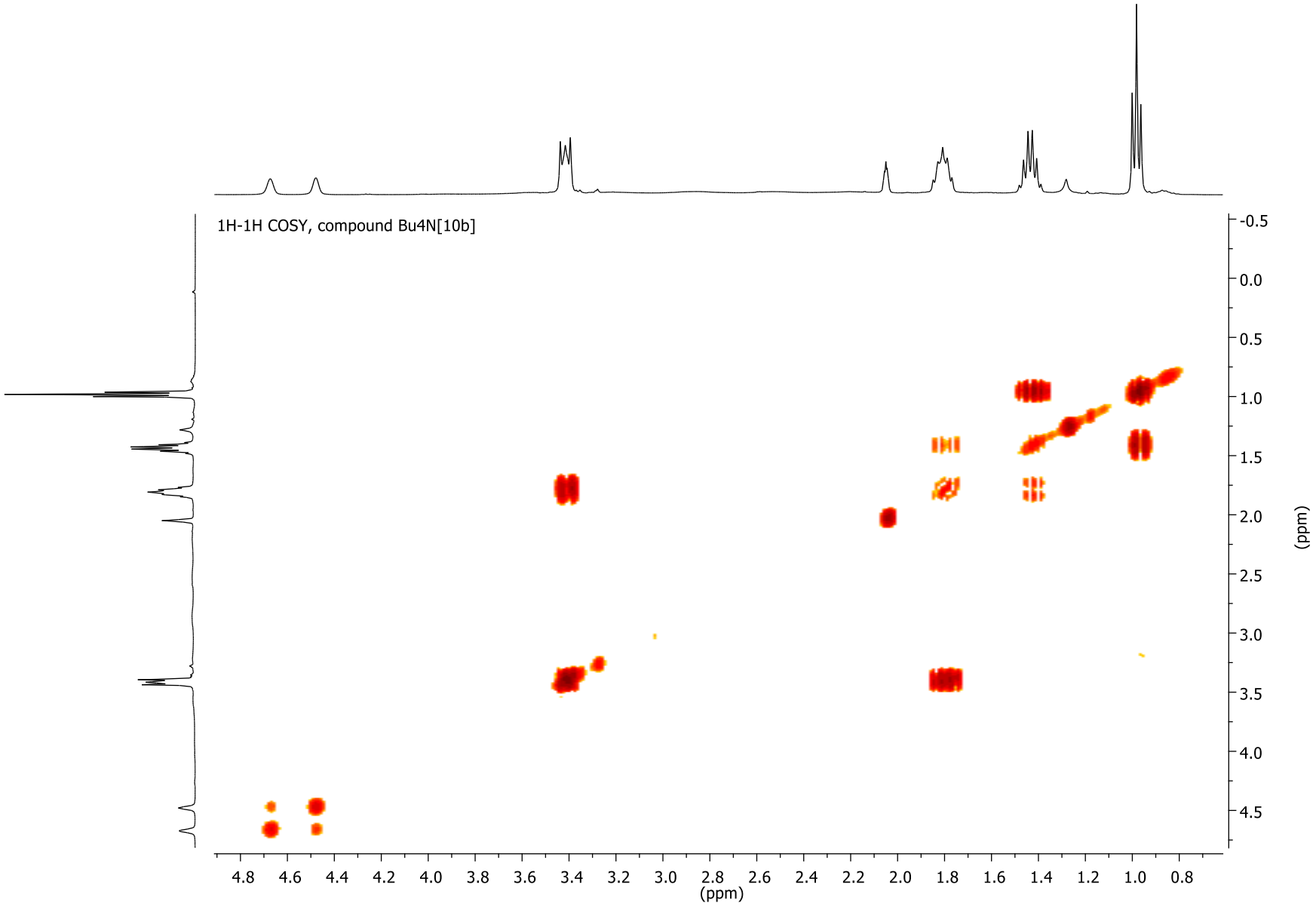

11B, compound Bu4N[10b]
10.1
9.2
3.2
2.0
-1.5
-2.6
-4.8
-5.9
-11.7
-12.9
-14.4
-16.9
-18.1
-20.9
-22.2
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

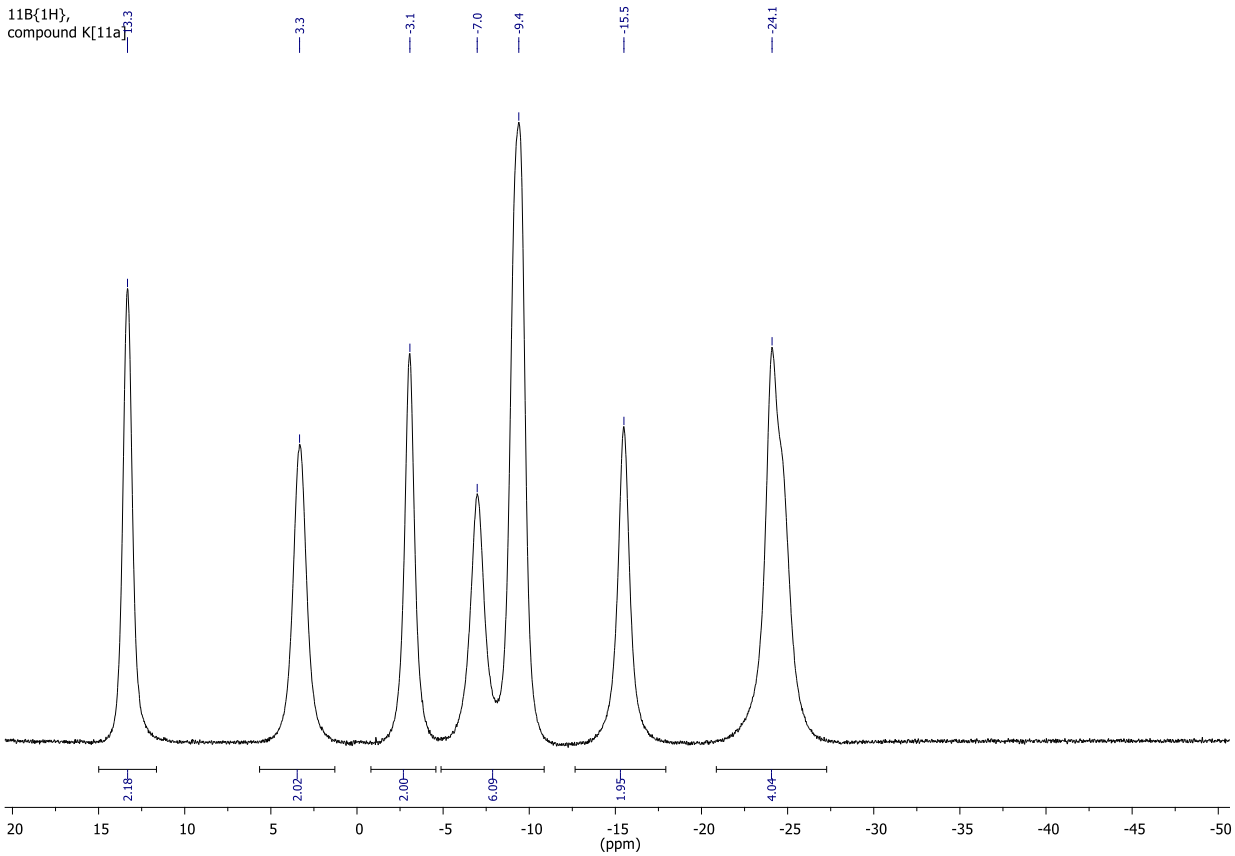
1H, compound Bu4N[10b]

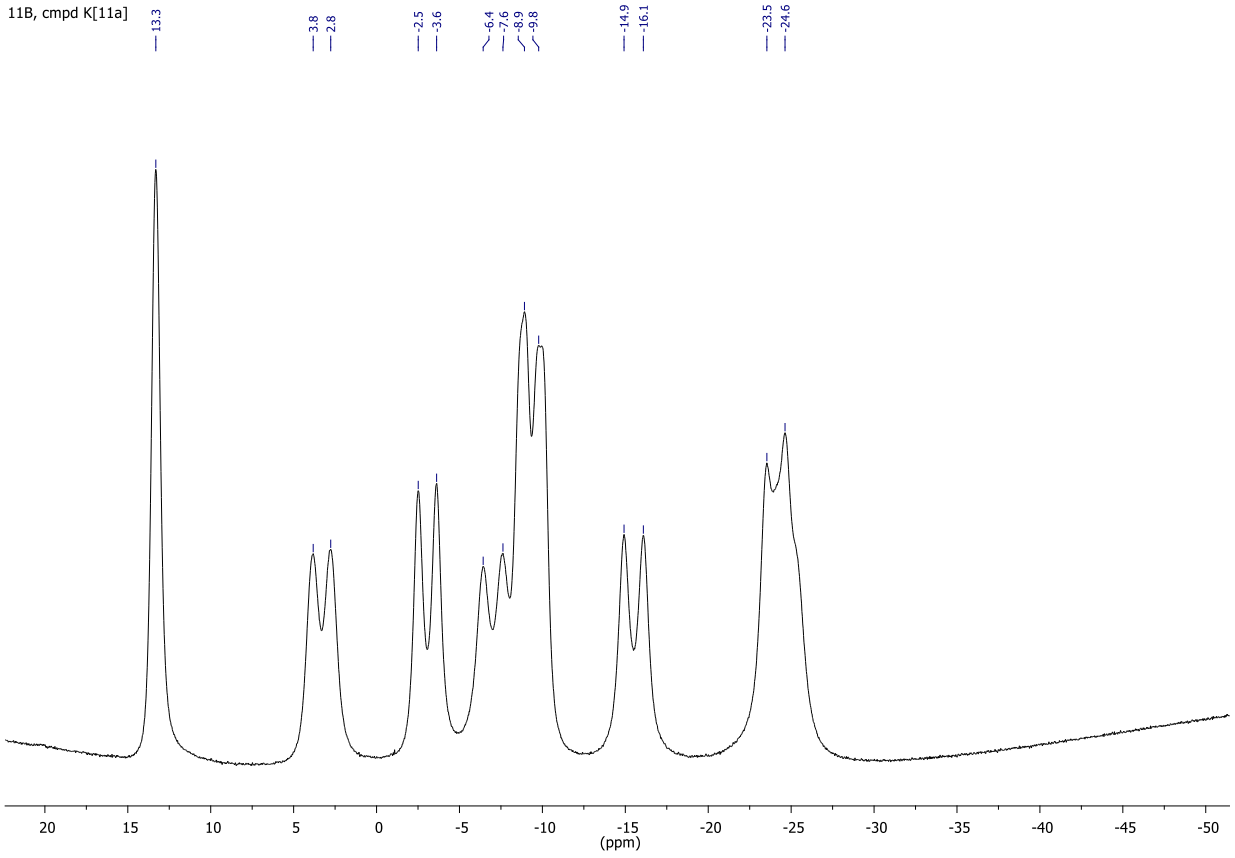


13C{1H}, compound Bu4N[10b]

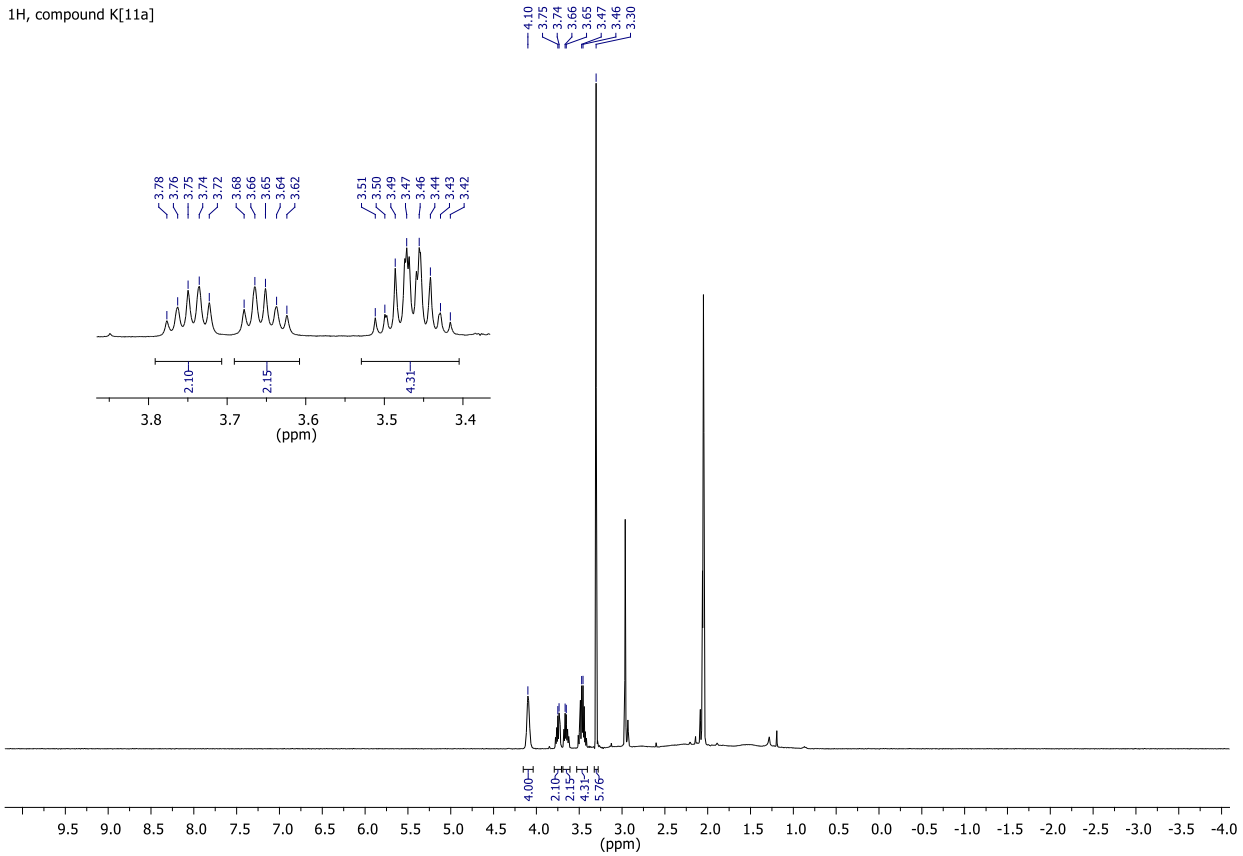



1H-1H COSY, compound Bu4N[10b]
4.8 4.6 4.4 4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0 1.8 1.6 1.4 1.2 1.0 0.8
(ppm)
-0.5 0.0 0.5 1.0 1.5 2.0 2.5 3.0 3.5 4.0 4.5
(ppm)

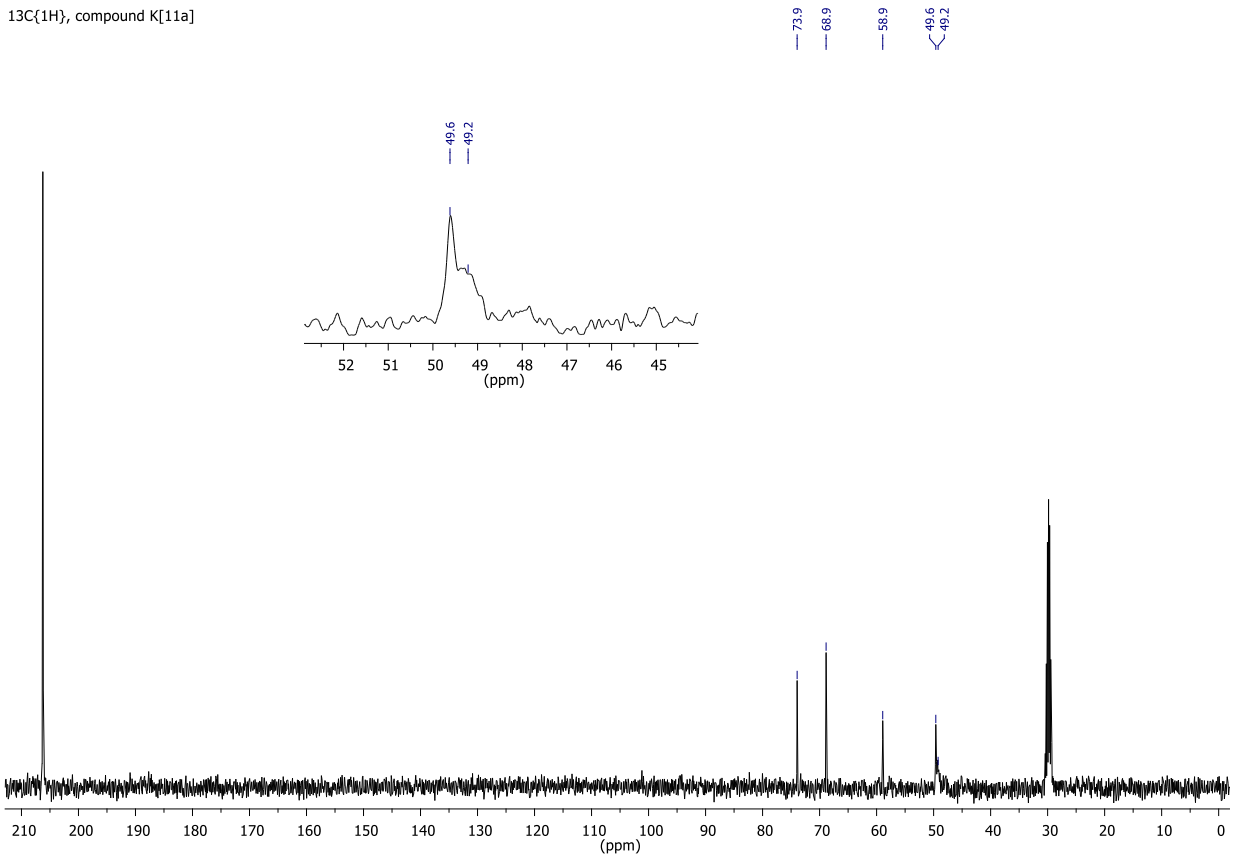

11B{1H},
compound K[11a]
13.3
3.3
-3.1
-7.0
-9.4
-15.5
-24.1
2.18
2.02
2.00
6.09
1.95
4.04
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

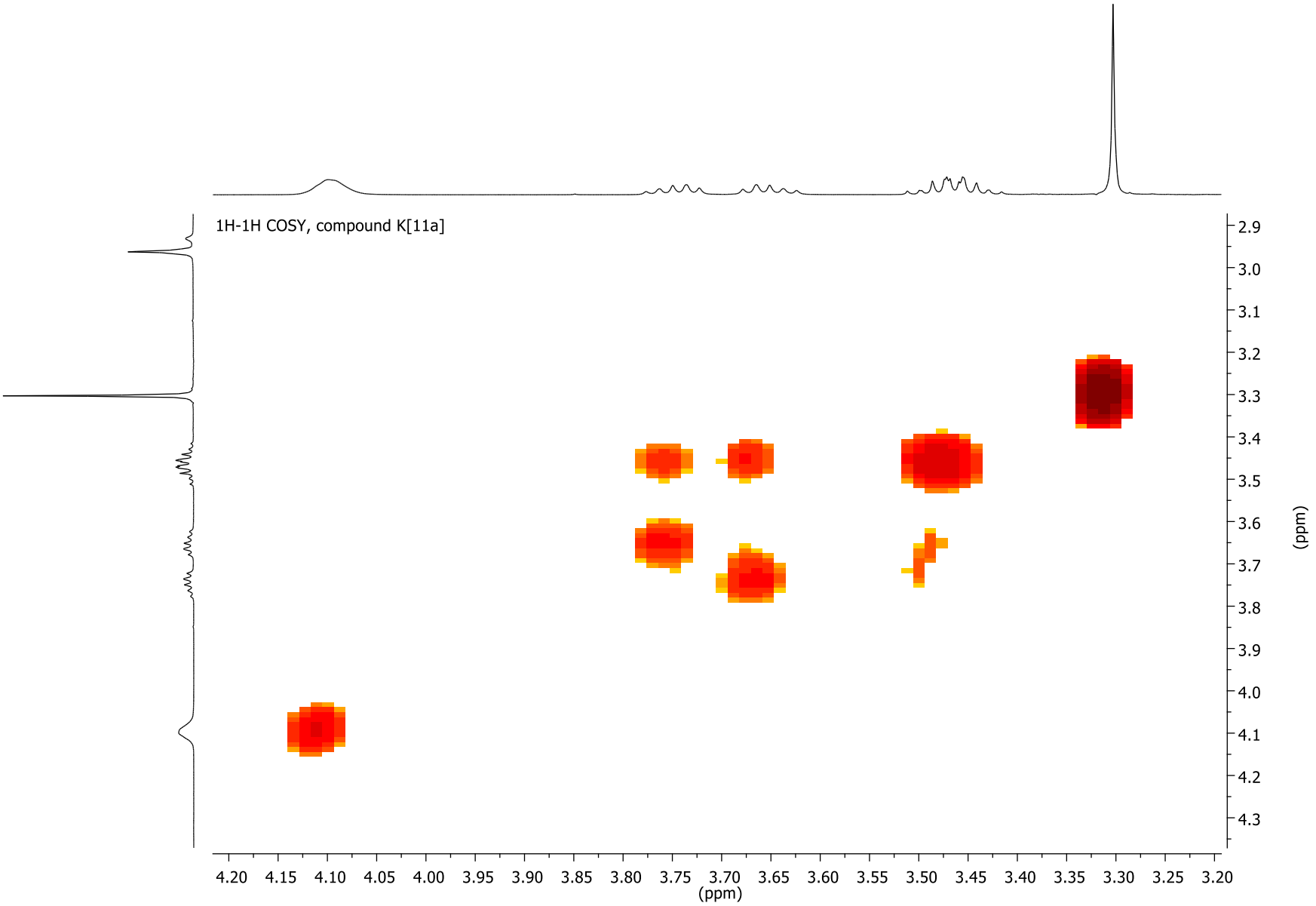

11B, cmpd K[11a]
13.3
3.8
2.8
-2.5
-3.6
-6.4
-7.6
-8.9
-9.8
-14.9
-16.1
-23.5
-24.6
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

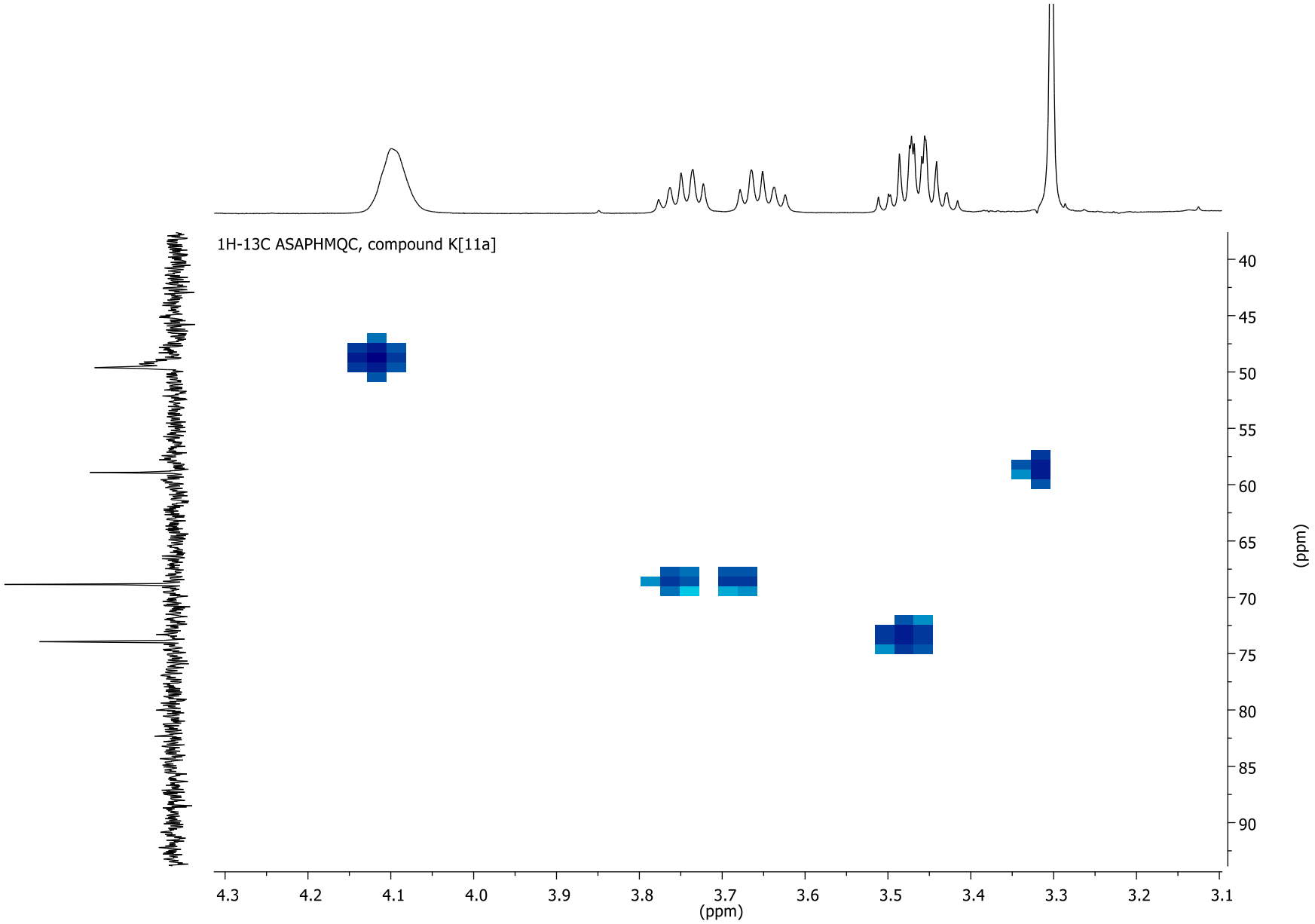
1H, compound K[11a]

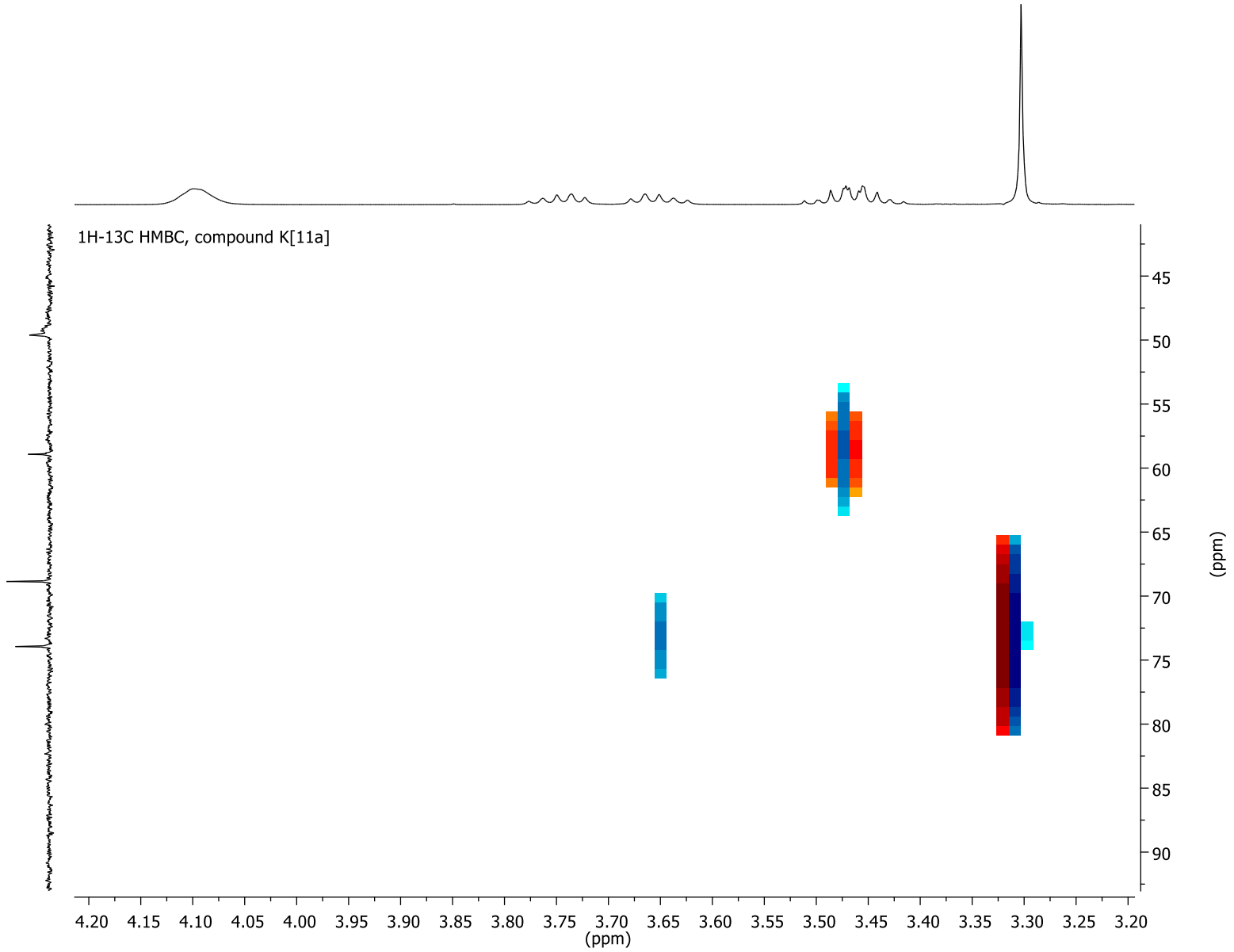


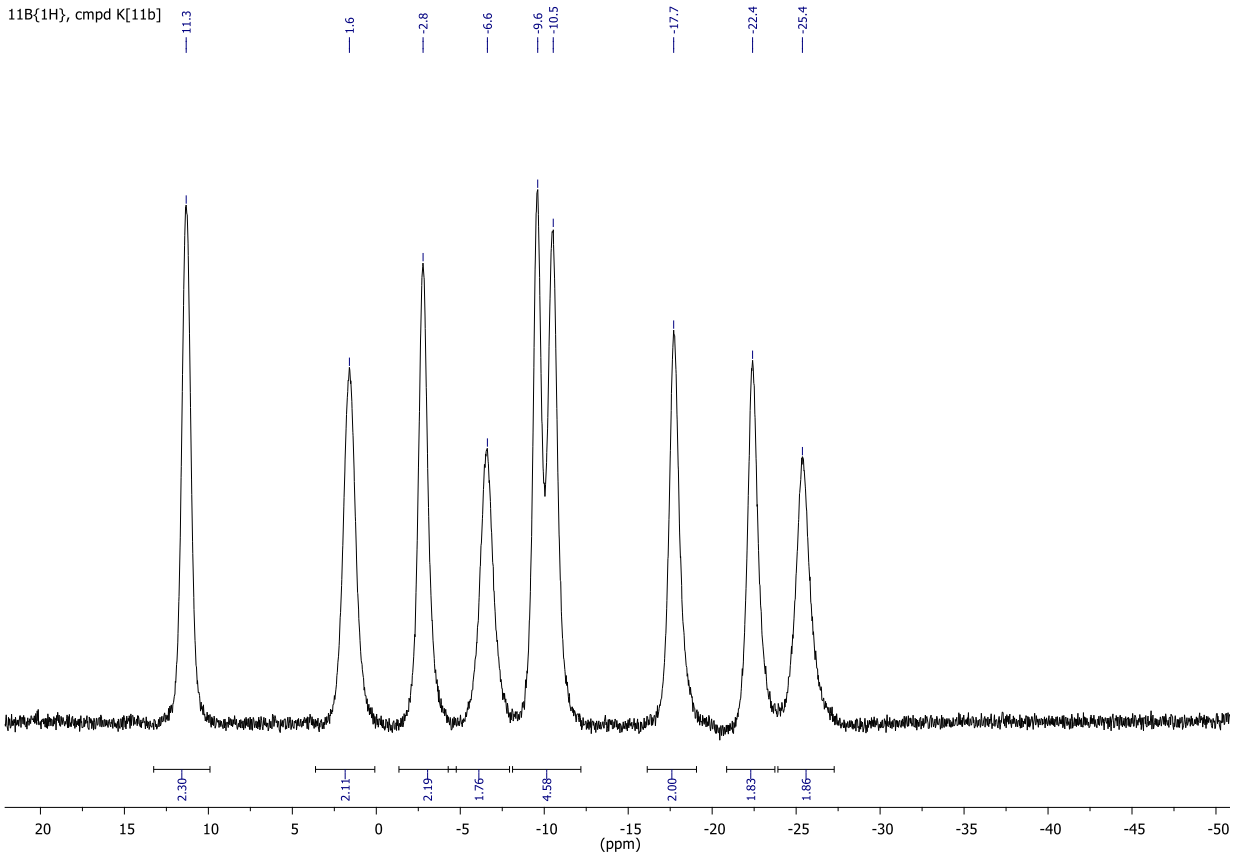
13C{1H}, compound K[11a]

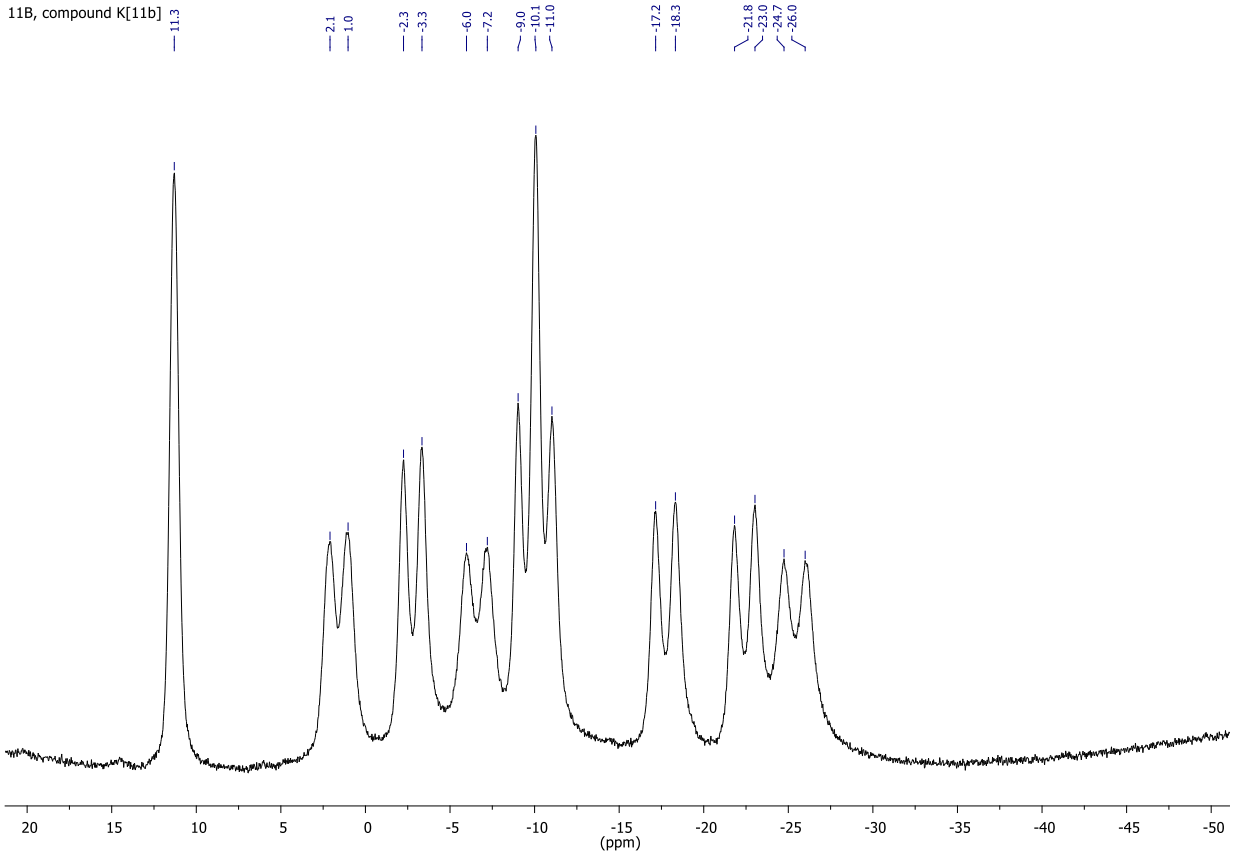



1H-1H COSY, compound K[11a]
4.20 4.15 4.10 4.05 4.00 3.95 3.90 3.85 3.80 3.75 3.70 3.65 3.60 3.55 3.50 3.45 3.40 3.35 3.30 3.25 3.20
(ppm)
2.9 3.0 3.1 3.2 3.3 3.4 3.5 3.6 3.7 3.8 3.9 4.0 4.1 4.2 4.3
(ppm)

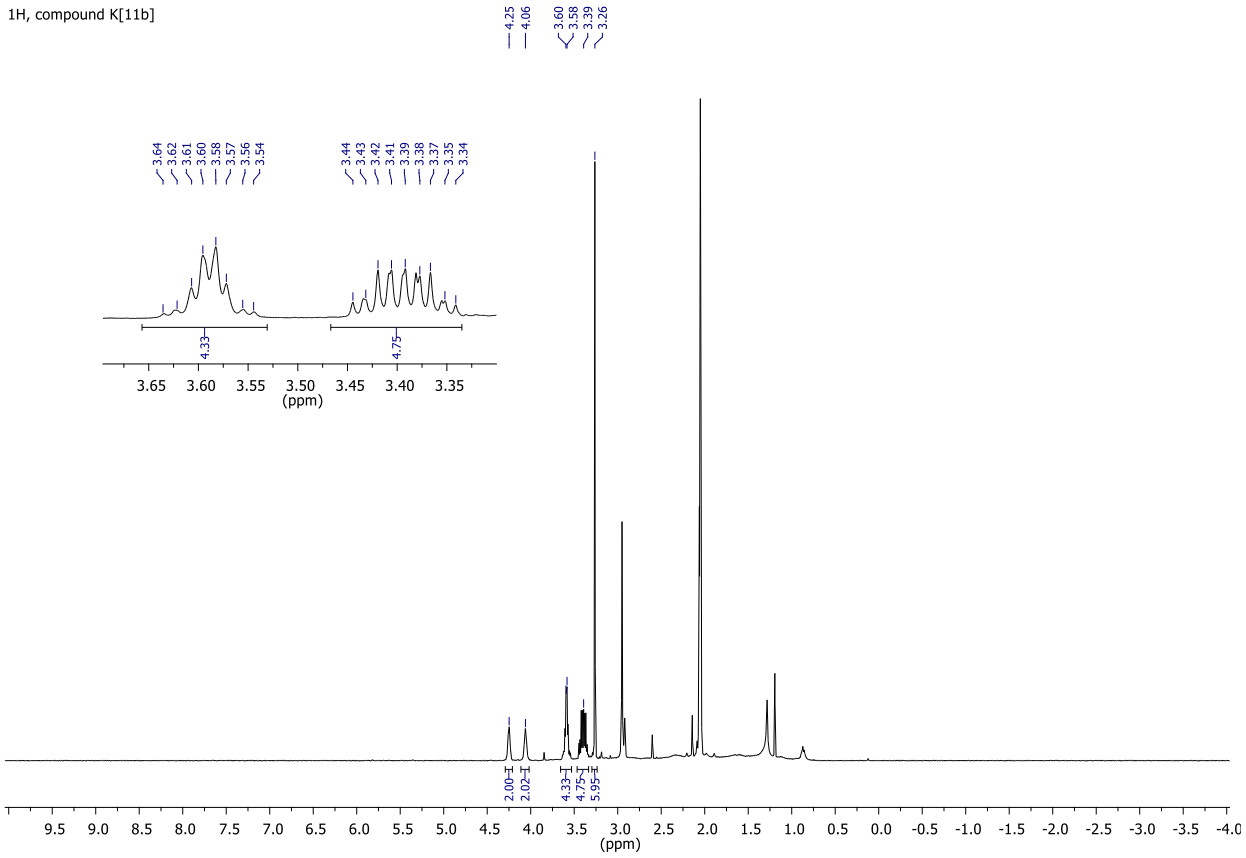

1H-13C ASAPHMQC, compound K[11a]
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
(ppm)
40
45
50
55
60
65
70
75
80
85
90
(ppm)


1H-13C HMBC, compound K[11a]
4.20 4.15 4.10 4.05 4.00 3.95 3.90 3.85 3.80 3.75 3.70 3.65 3.60 3.55 3.50 3.45 3.40 3.35 3.30 3.25 3.20
(ppm)
45 50 55 60 65 70 75 80 85 90
(ppm)

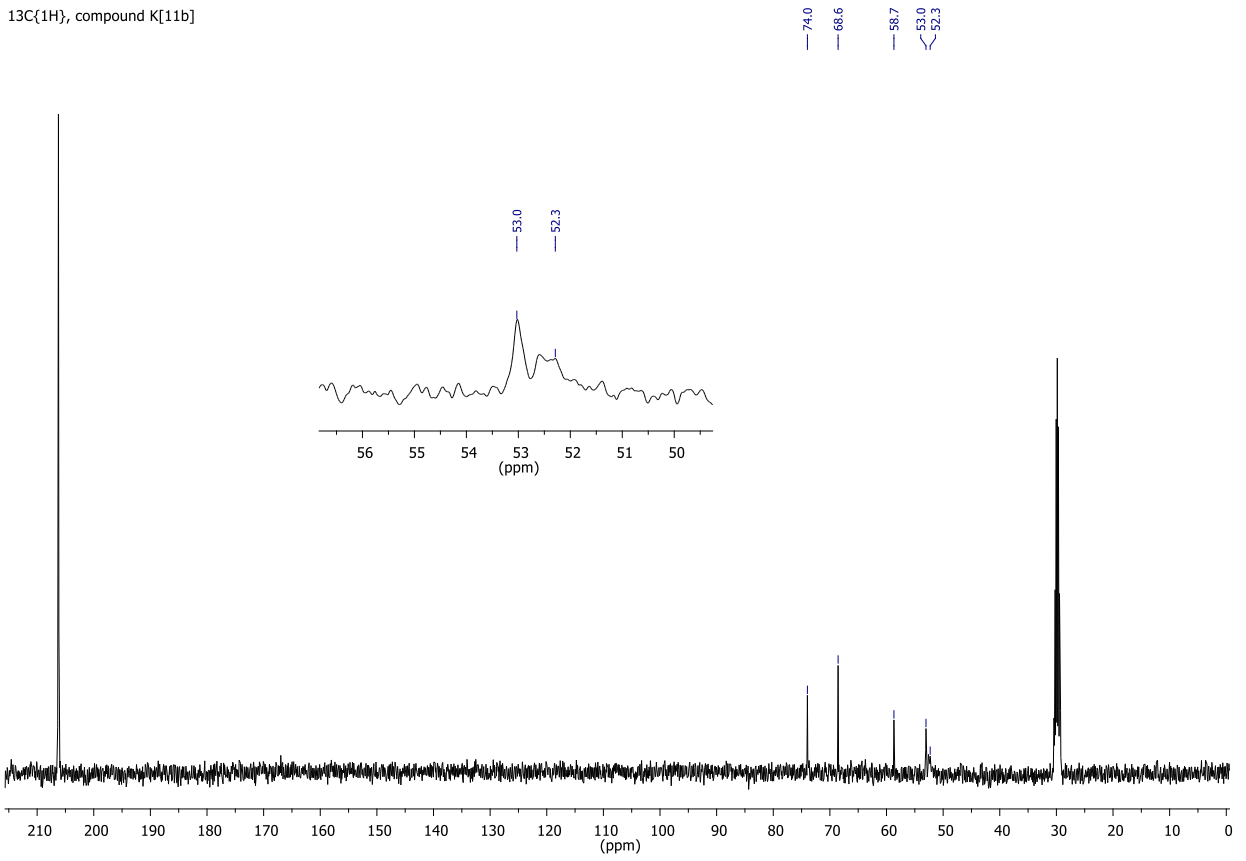

11B{1H}, cmpd K[11b]
11.3
1.6
-2.8
-6.6
-9.6
-10.5
-17.7
-22.4
-25.4
2.30
2.11
2.19
1.76
4.58
2.00
1.83
1.86
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

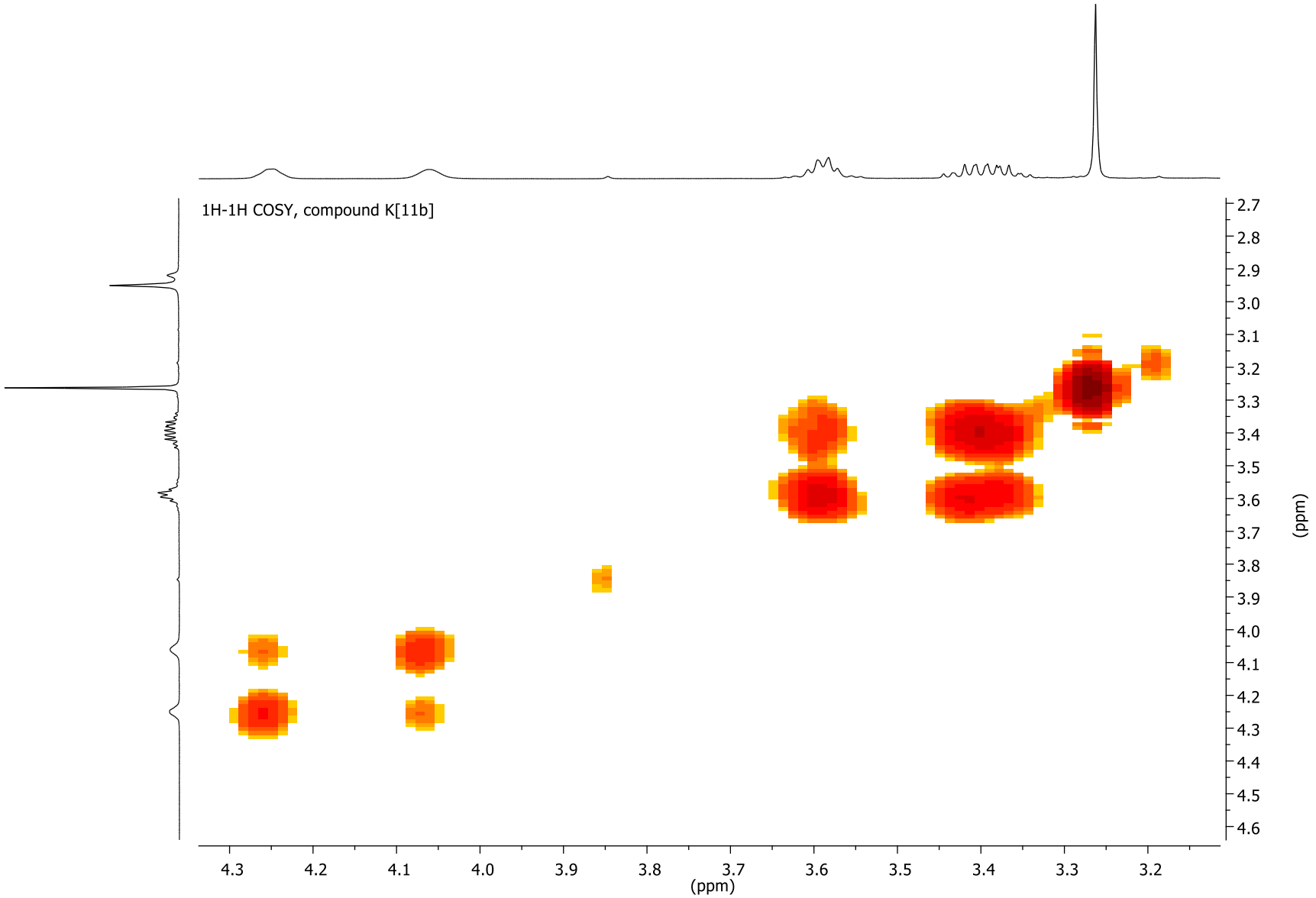

11B, compound K[11b]
11.3
2.1
1.0
-2.3
-3.3
-6.0
-7.2
-9.0
-10.1
-11.0
-17.2
-18.3
-21.8
-23.0
-24.7
-26.0
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

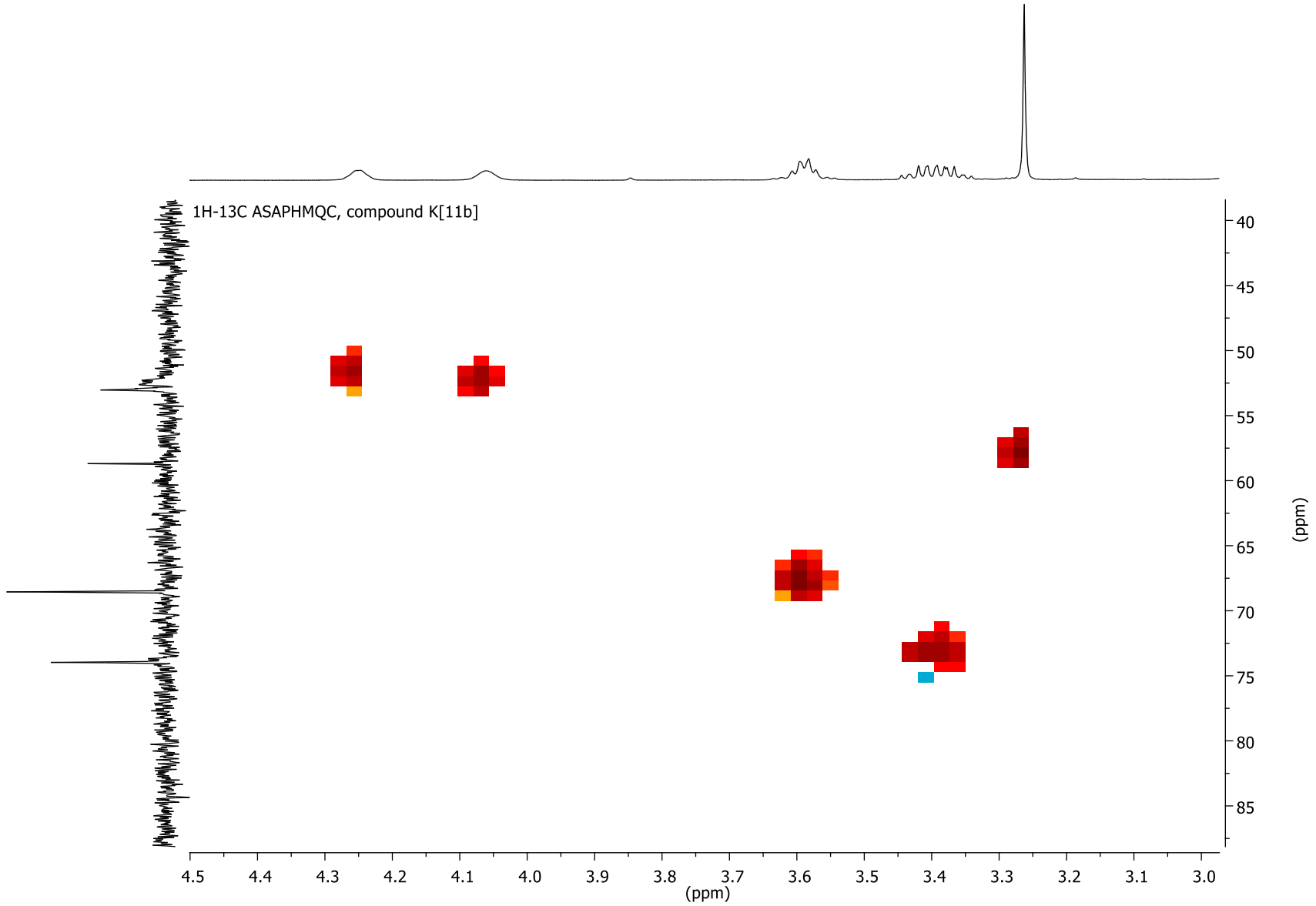
1H, compound K[11b]

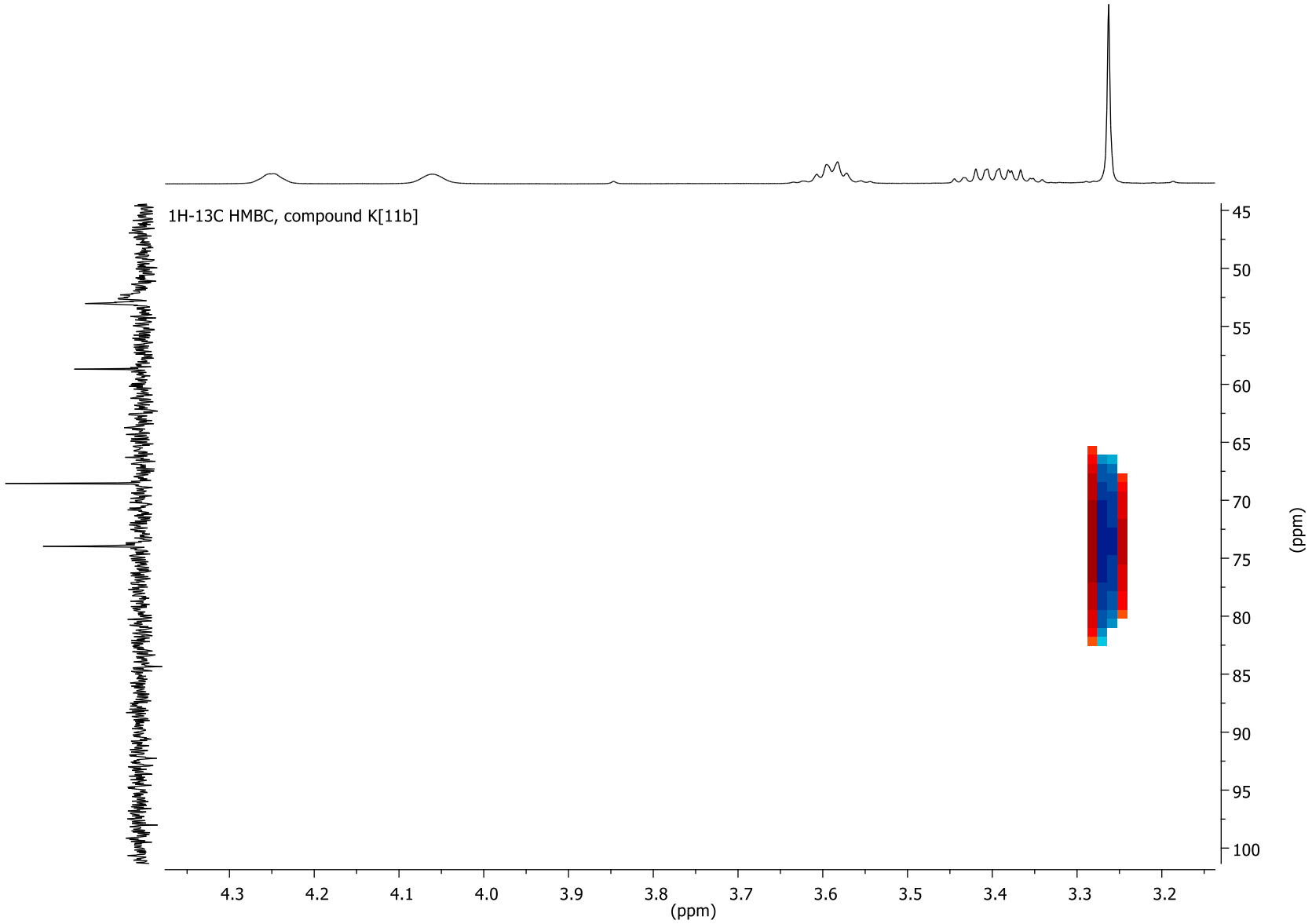


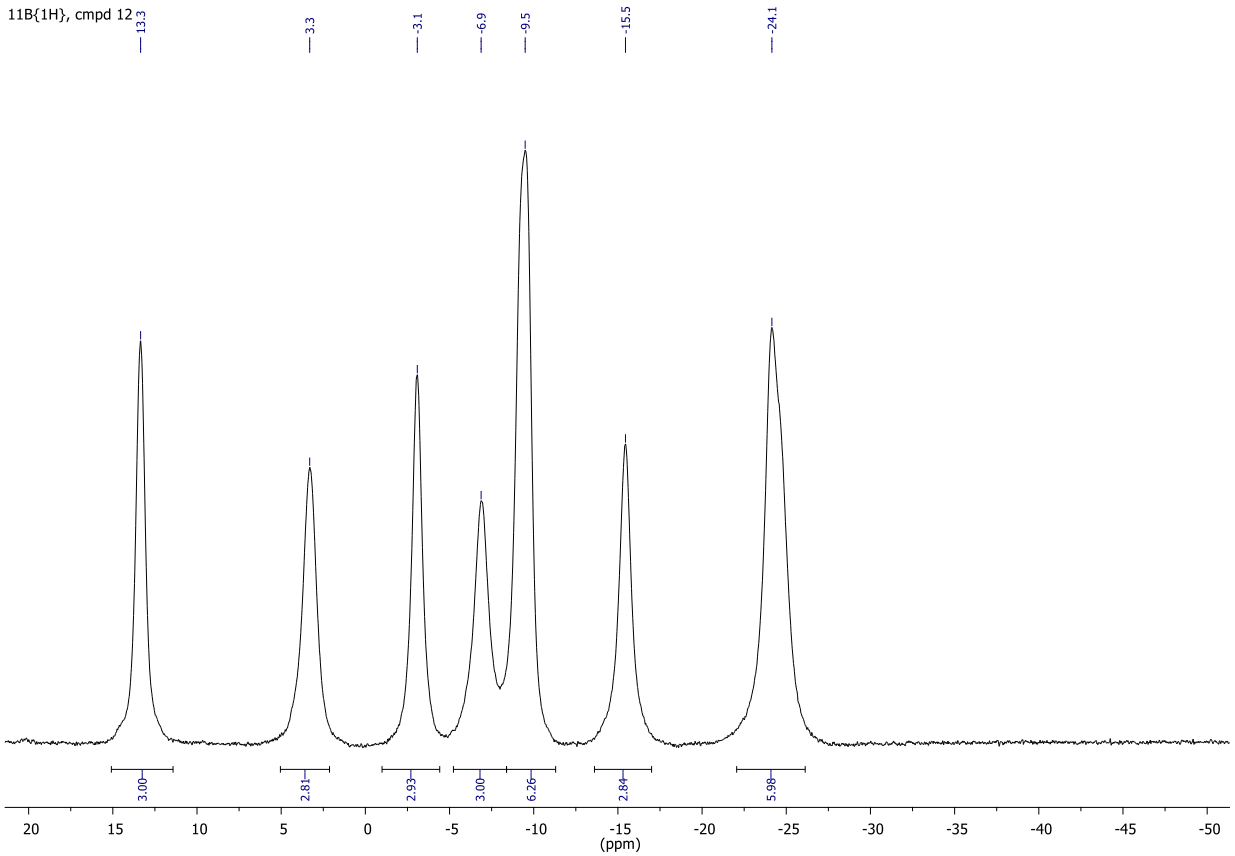
13C{1H}, compound K[11b]

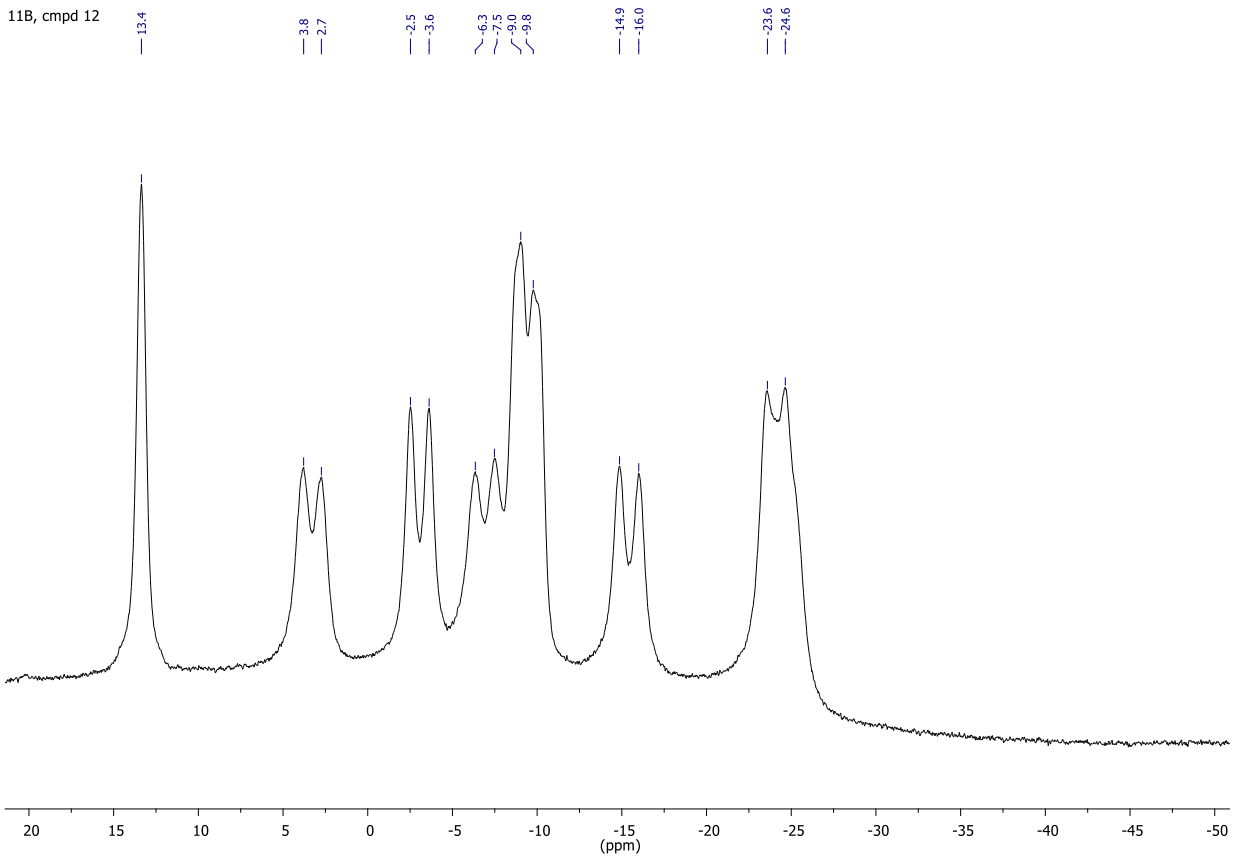



1H-1H COSY, compound K[11b]
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
(ppm)
2.7
2.8
2.9
3.0
3.1
3.2
3.3
3.4
3.5
3.6
3.7
3.8
3.9
4.0
4.1
4.2
4.3
4.4
4.5
4.6
(ppm)

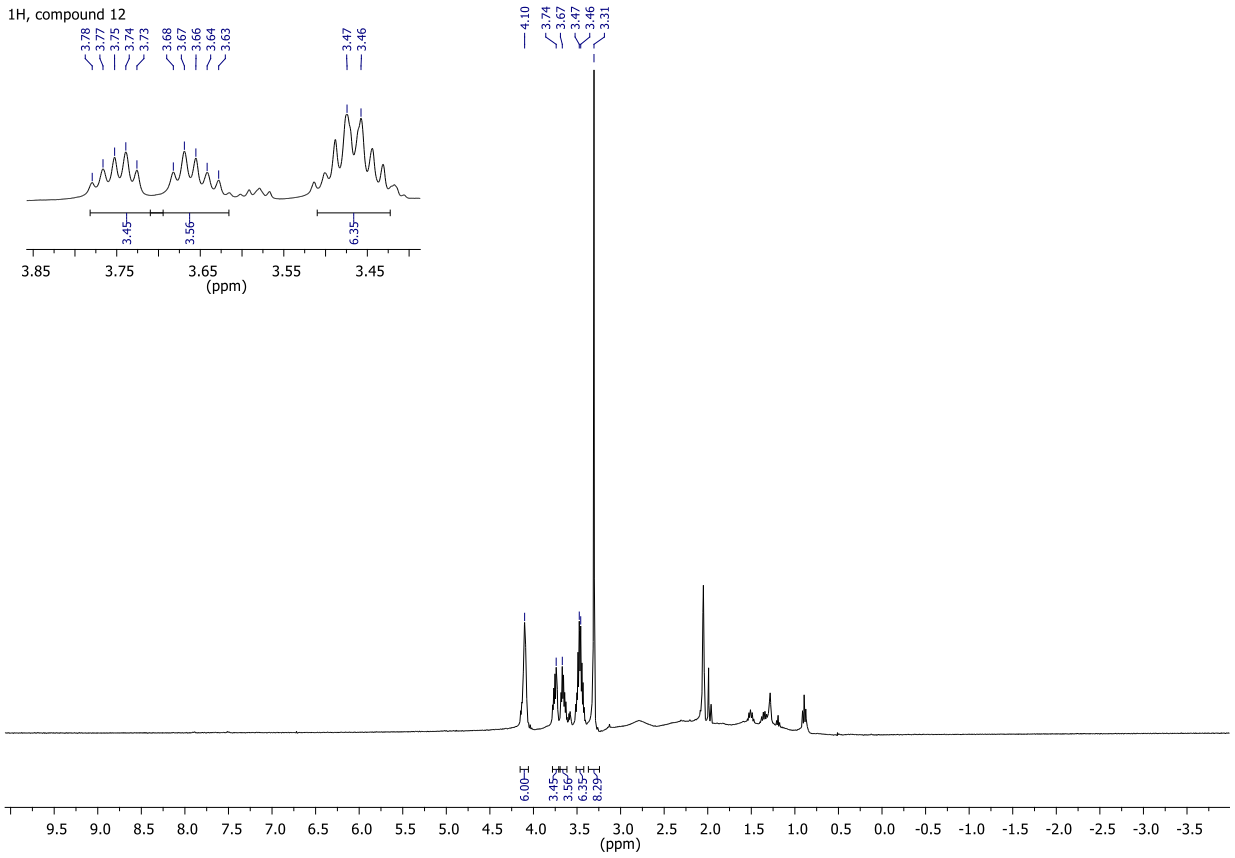

1H-13C ASAPHMQC, compound K[11b]
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
(ppm)
40
45
50
55
60
65
70
75
80
85
(ppm)


1H-13C HMBC, compound K[11b]
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
(ppm)
45
50
55
60
65
70
75
80
85
90
95
100
(ppm)

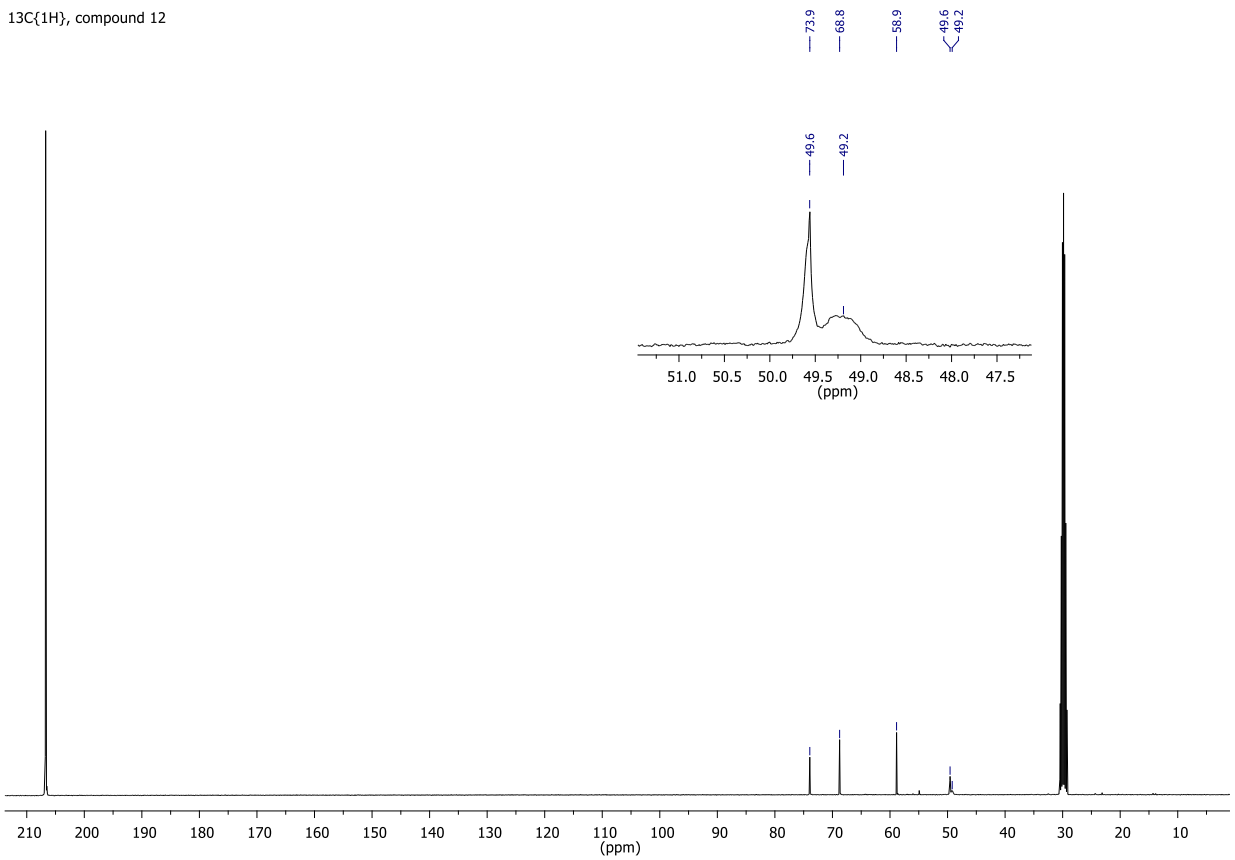

11B{1H}, cmpd 12
13.3
3.3
-3.1
-6.9
-9.5
-15.5
-24.1
3.00
2.81
2.93
3.00
6.26
2.84
5.98
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

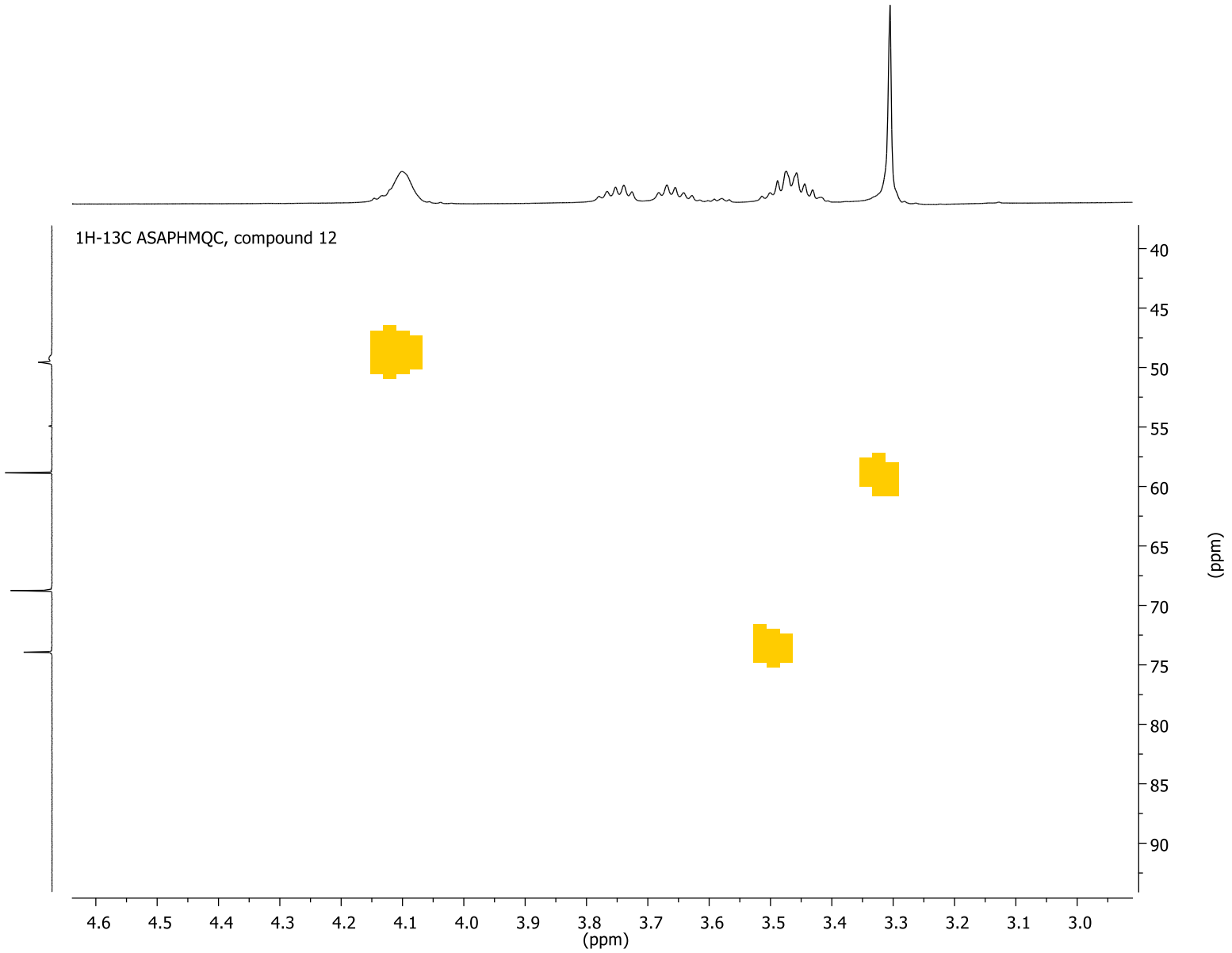

11B, cmpd 12
13.4
3.8
2.7
-2.5
-3.6
-6.3
-7.5
-9.0
-9.8
-14.9
-16.0
-23.6
-24.6
20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

1H, compound 12

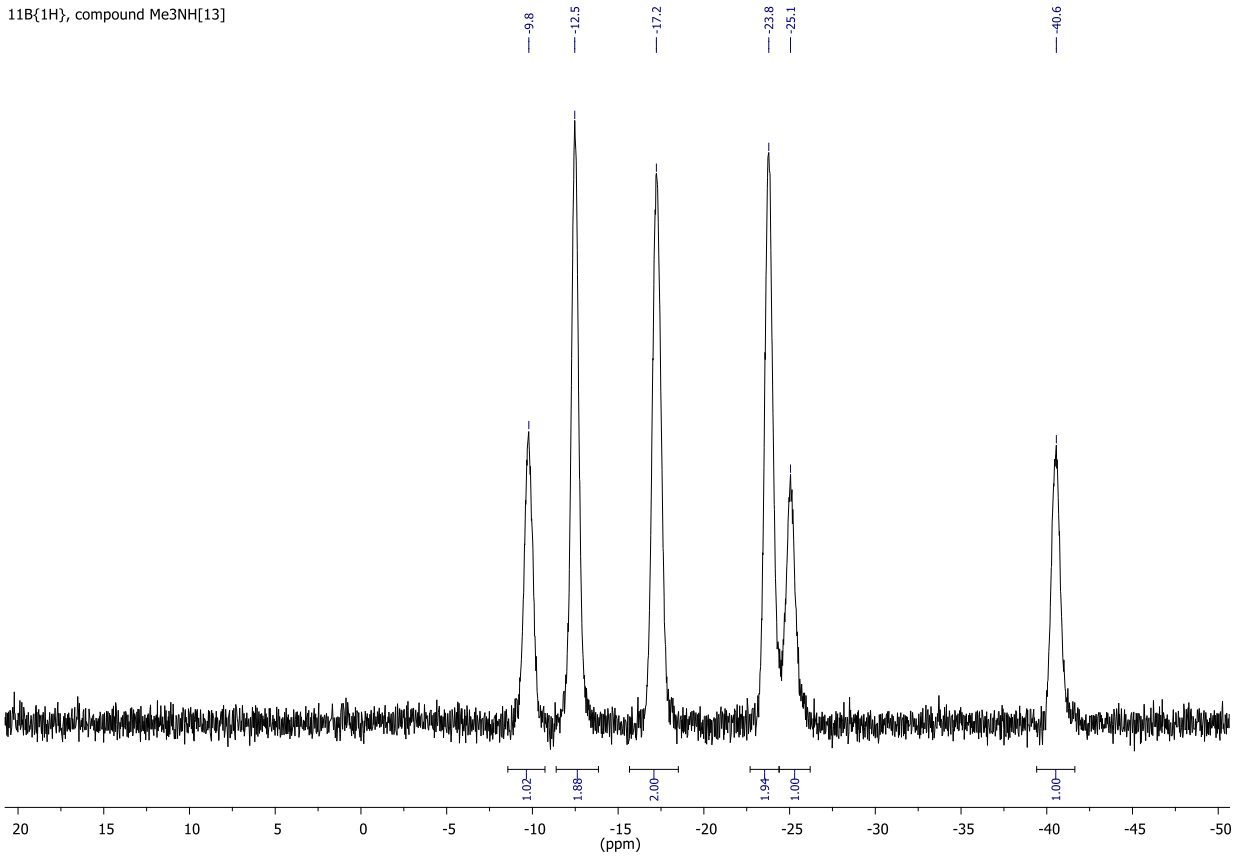


13C{1H}, compound 12

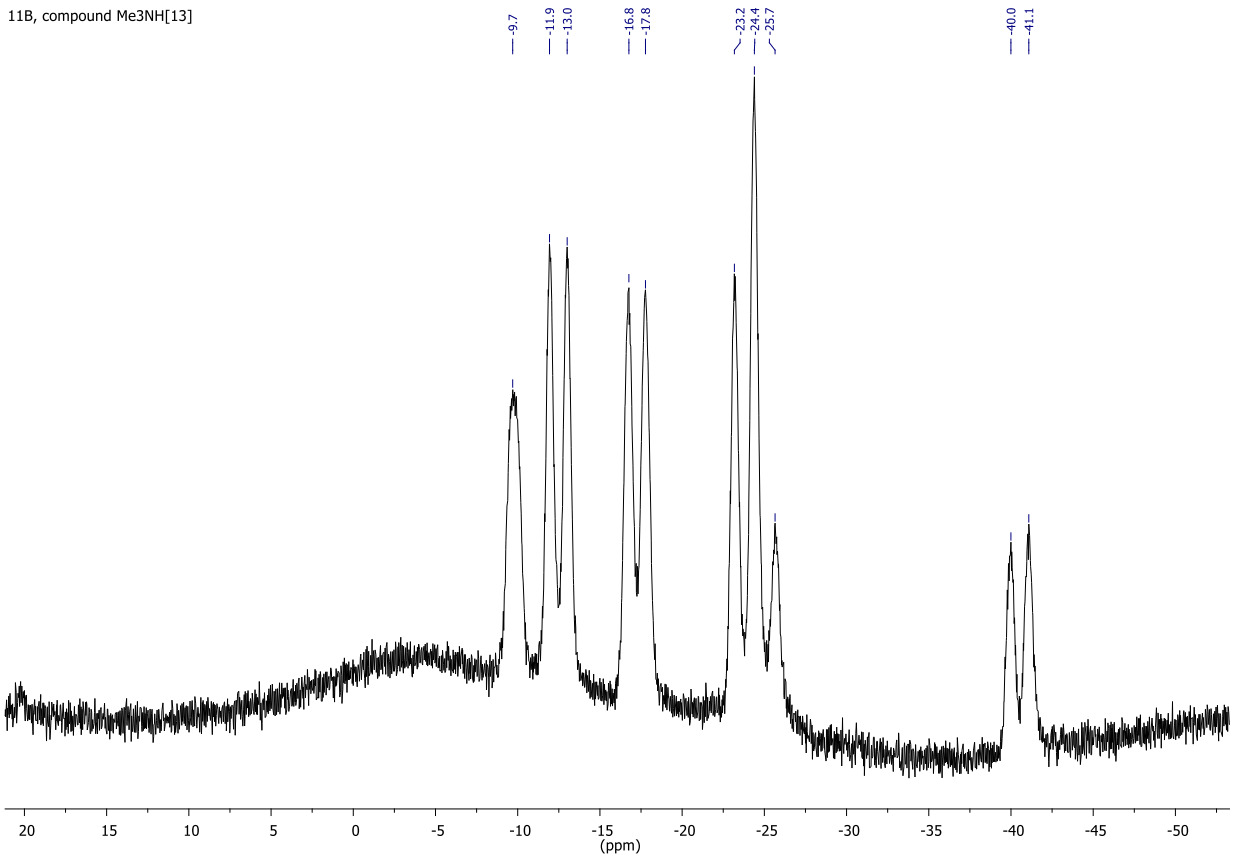



1H-13C ASAPHMQC, compound 12
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
(ppm)
40
45
50
55
60
65
70
75
80
85
90
(ppm)

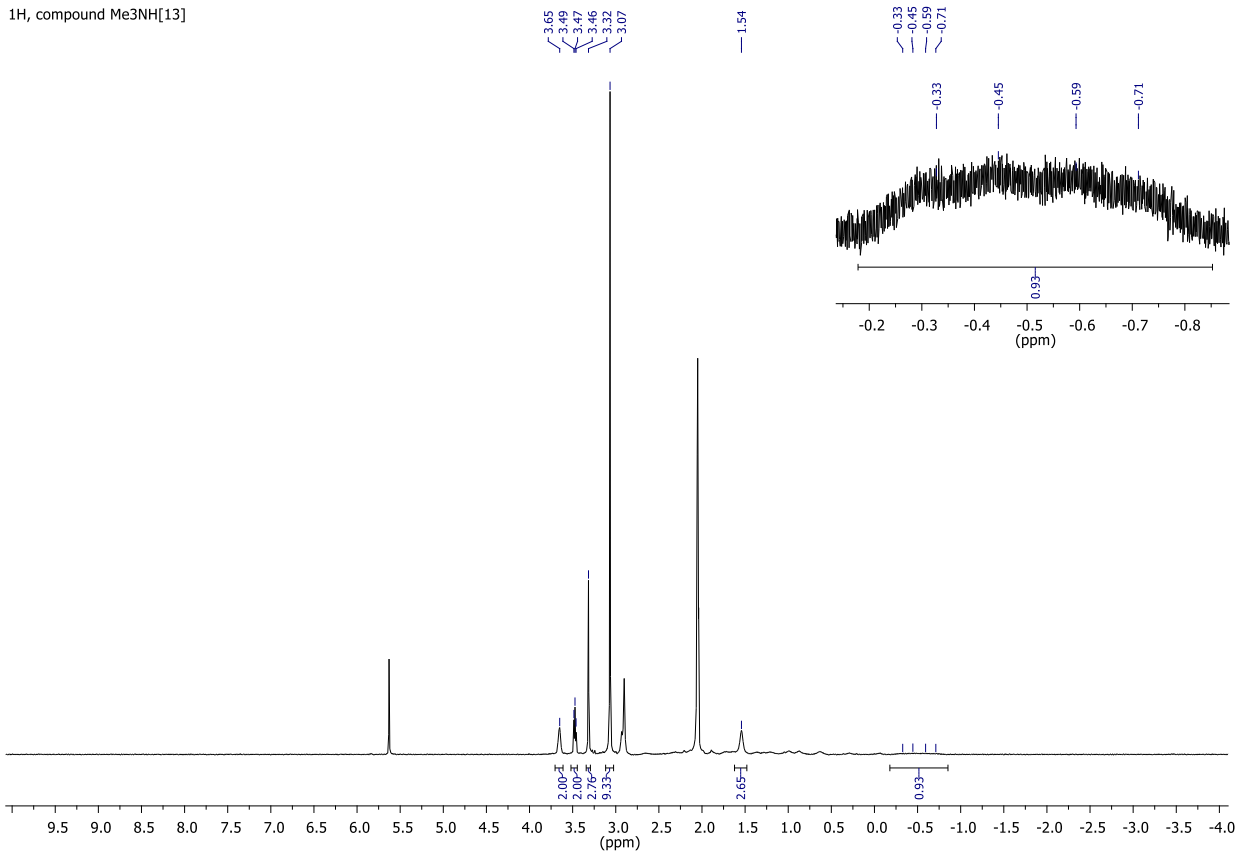
11B{1H}, compound Me3NH[13]



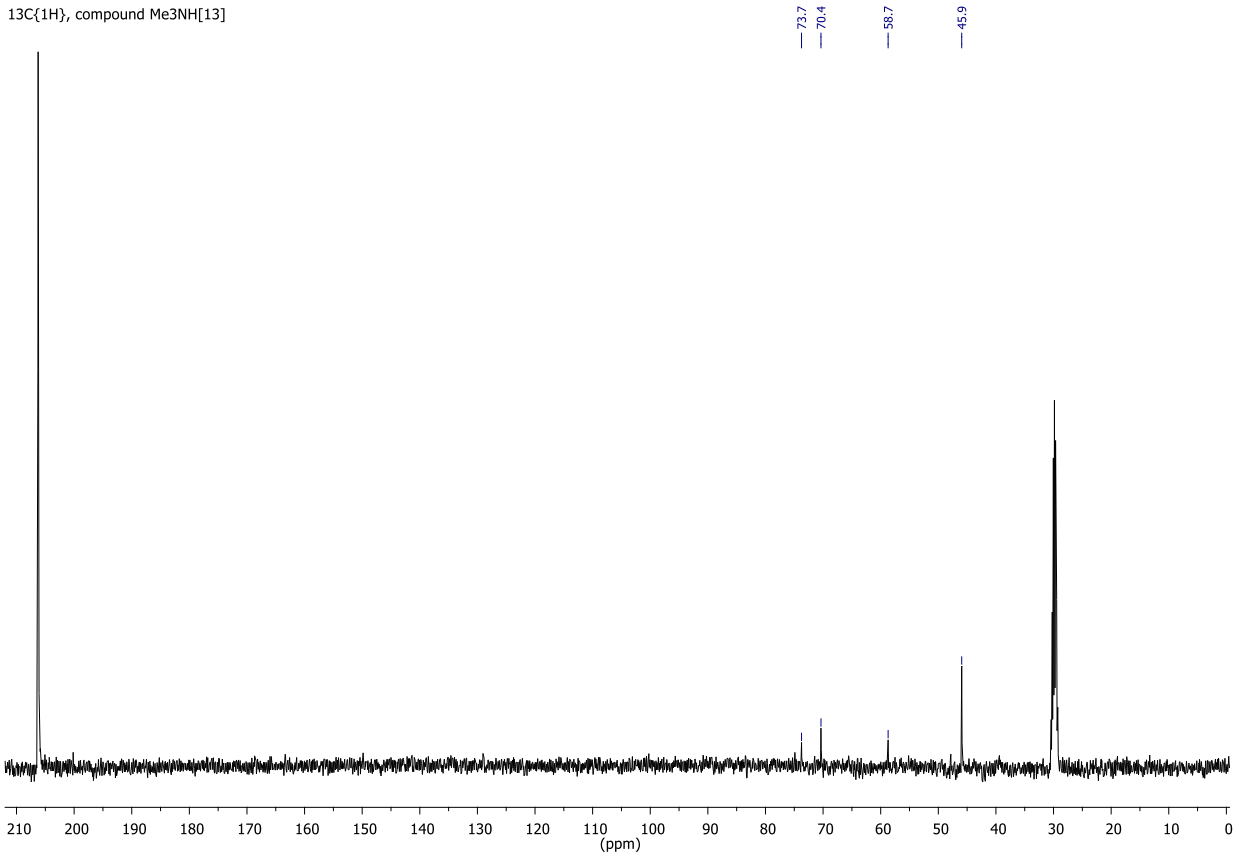
11B, compound Me3NH[13]

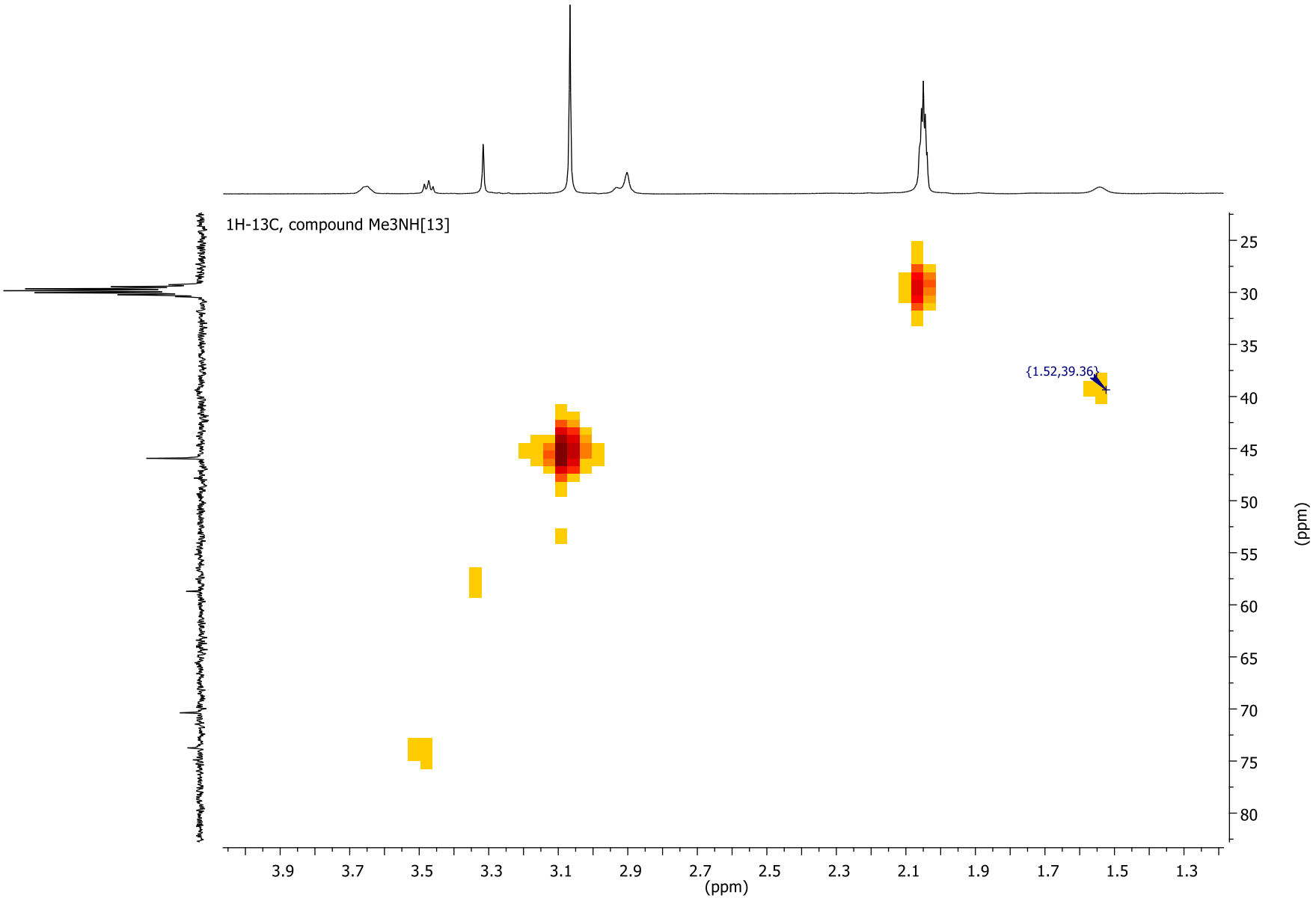


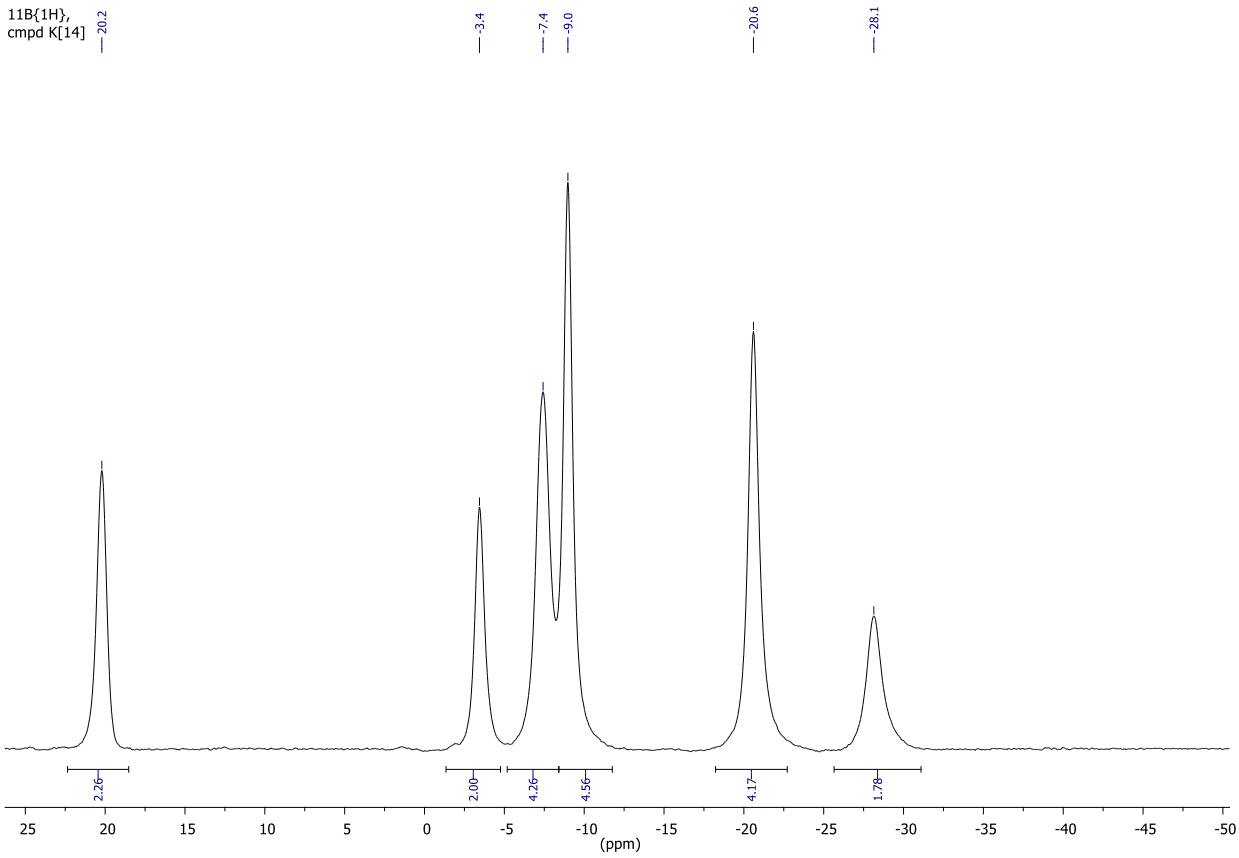
1H, compound Me3NH[13]

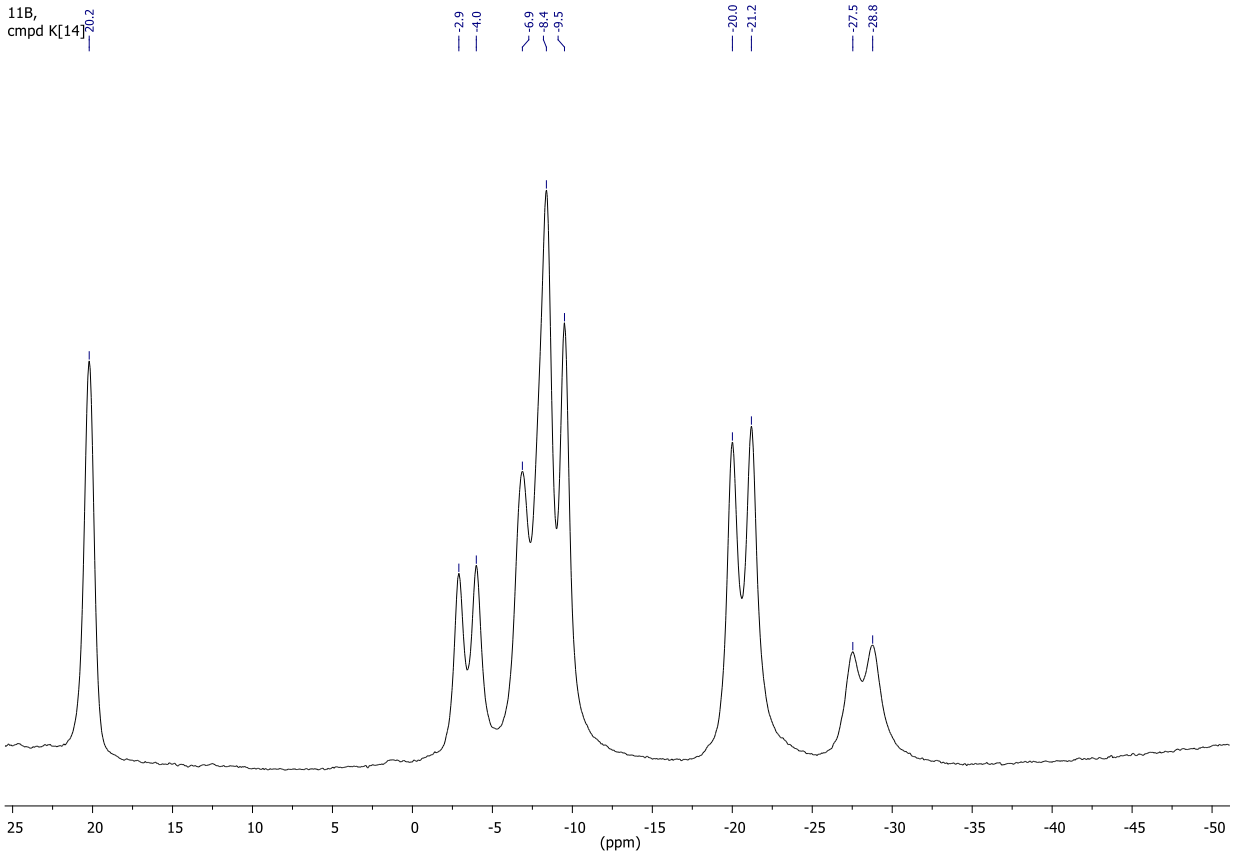


13C{1H}, compound Me3NH[13]

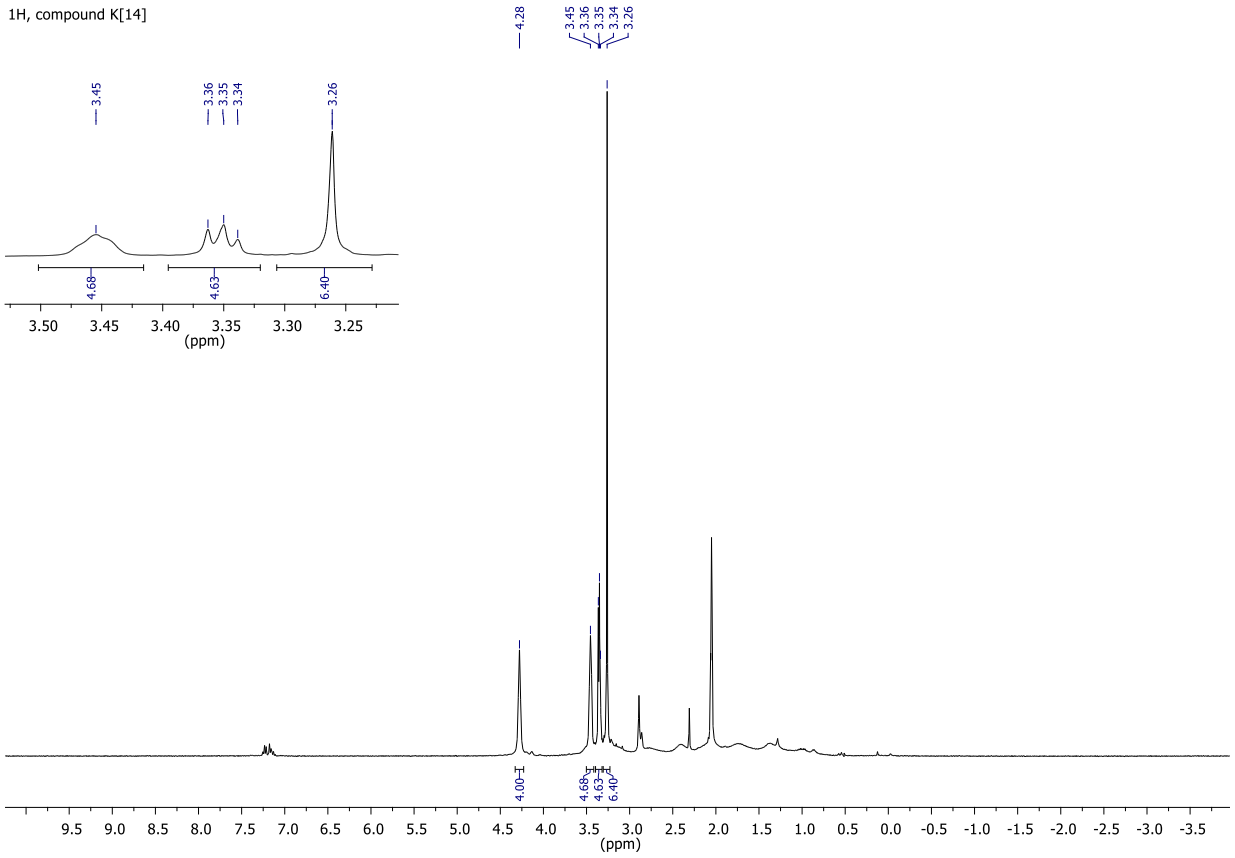



1H-13C, compound Me3NH[13]
{1.52,39.36}
3.9
3.7
3.5
3.3
3.1
2.9
2.7
2.5
2.3
2.1
1.9
1.7
1.5
1.3
(ppm)
25
30
35
40
45
50
55
60
65
70
75
80
(ppm)

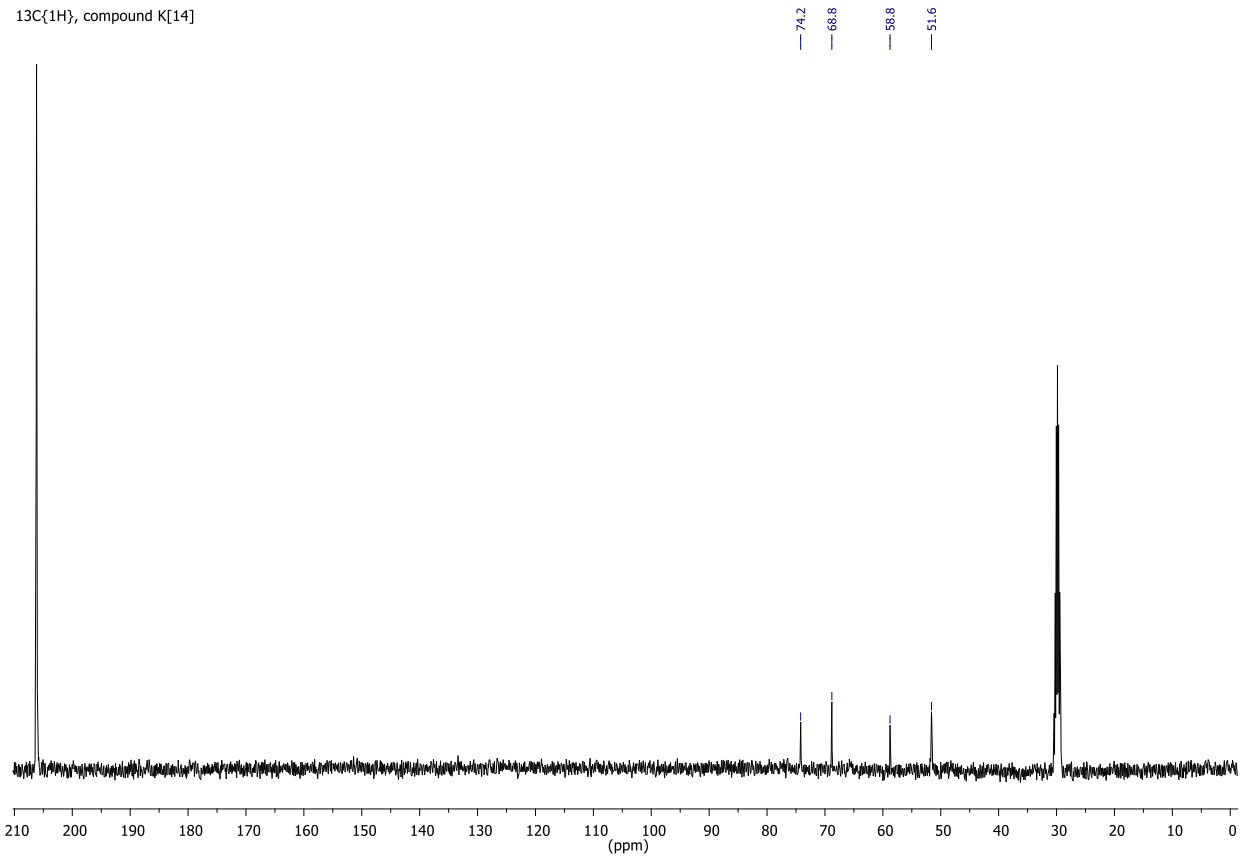

11B{1H},
cmpd K[14]
20.2
-3.4
-7.4
-9.0
-20.6
-28.1
2.26
2.00
4.26
4.56
4.17
1.78
25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

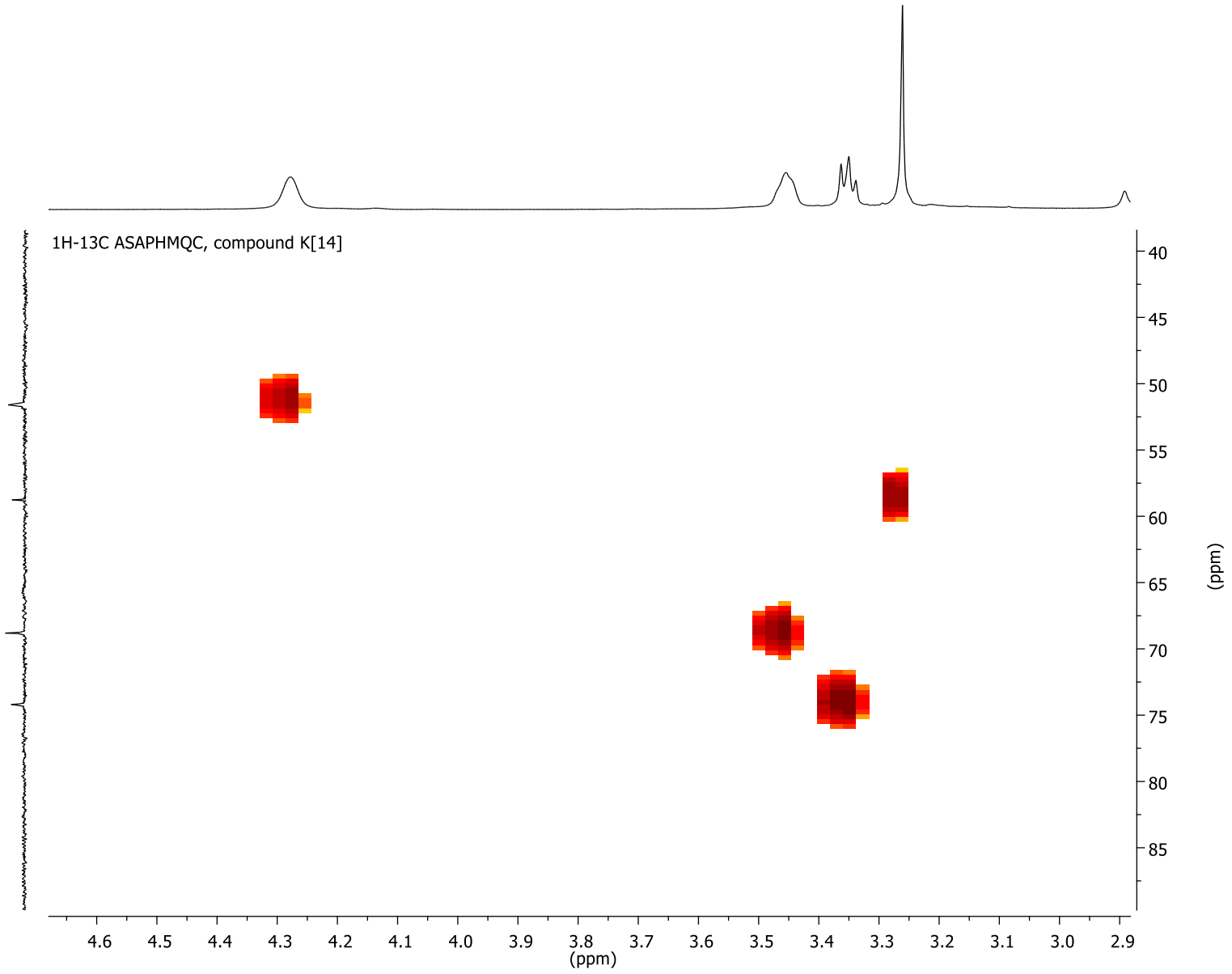

11B,
cmpd K[14]
20.2
-2.9
-4.0
-6.9
-8.4
-9.5
-20.0
-21.2
-27.5
-28.8
25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 -50
(ppm)

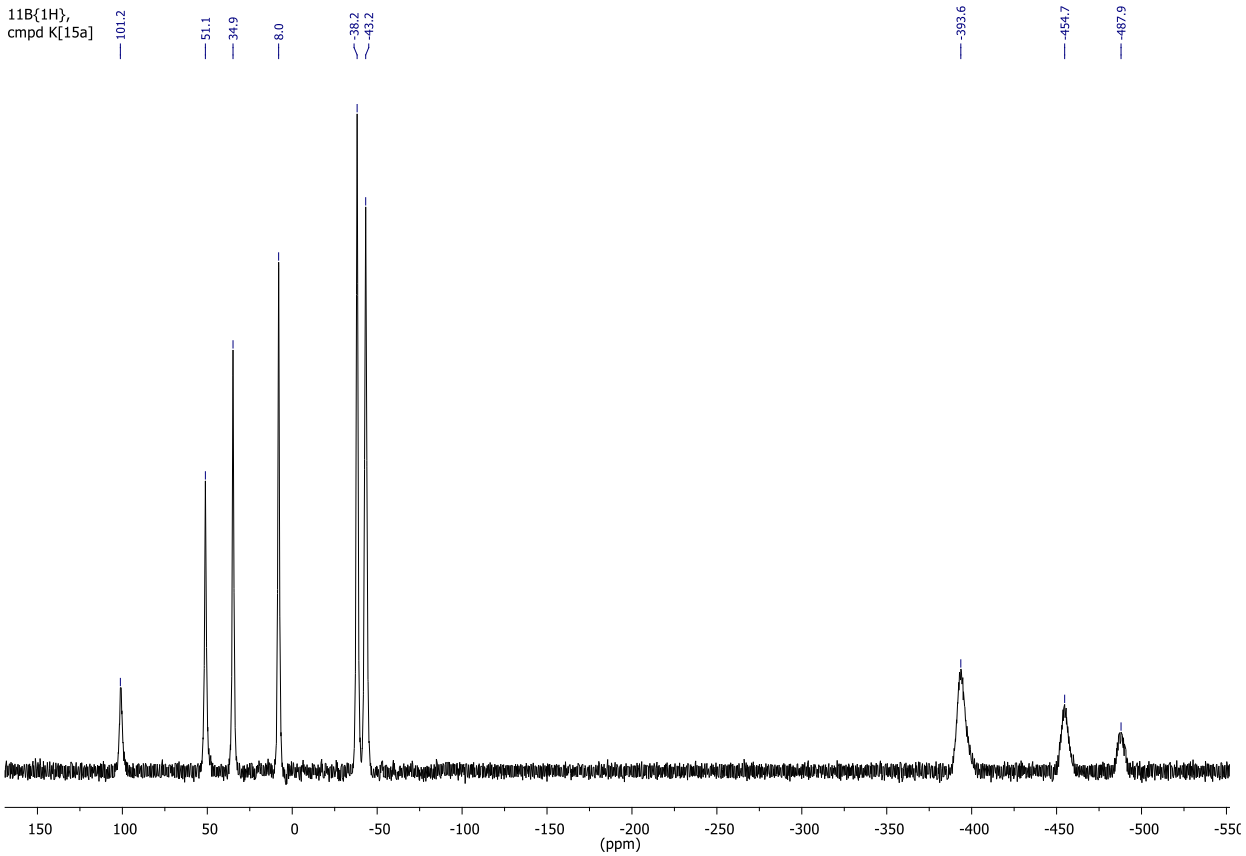
1H, compound K[14]

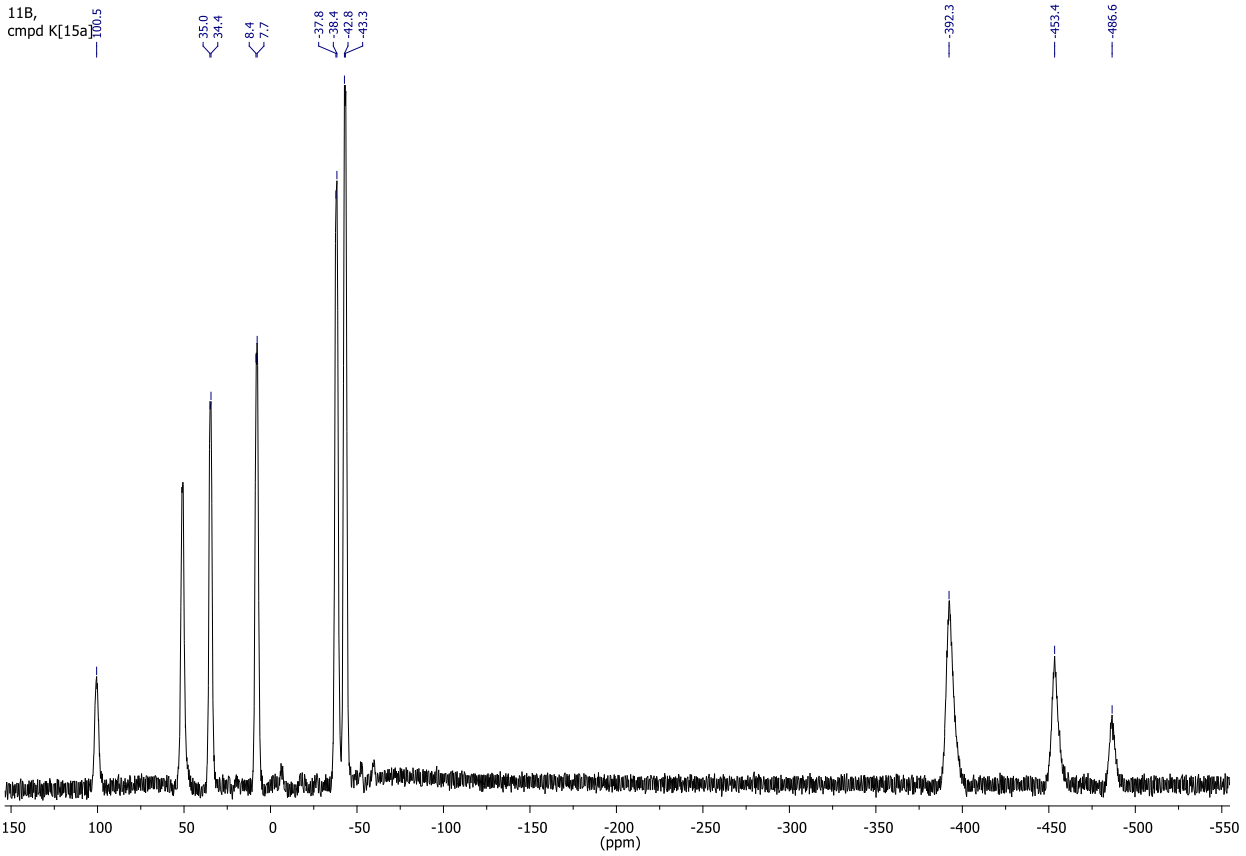


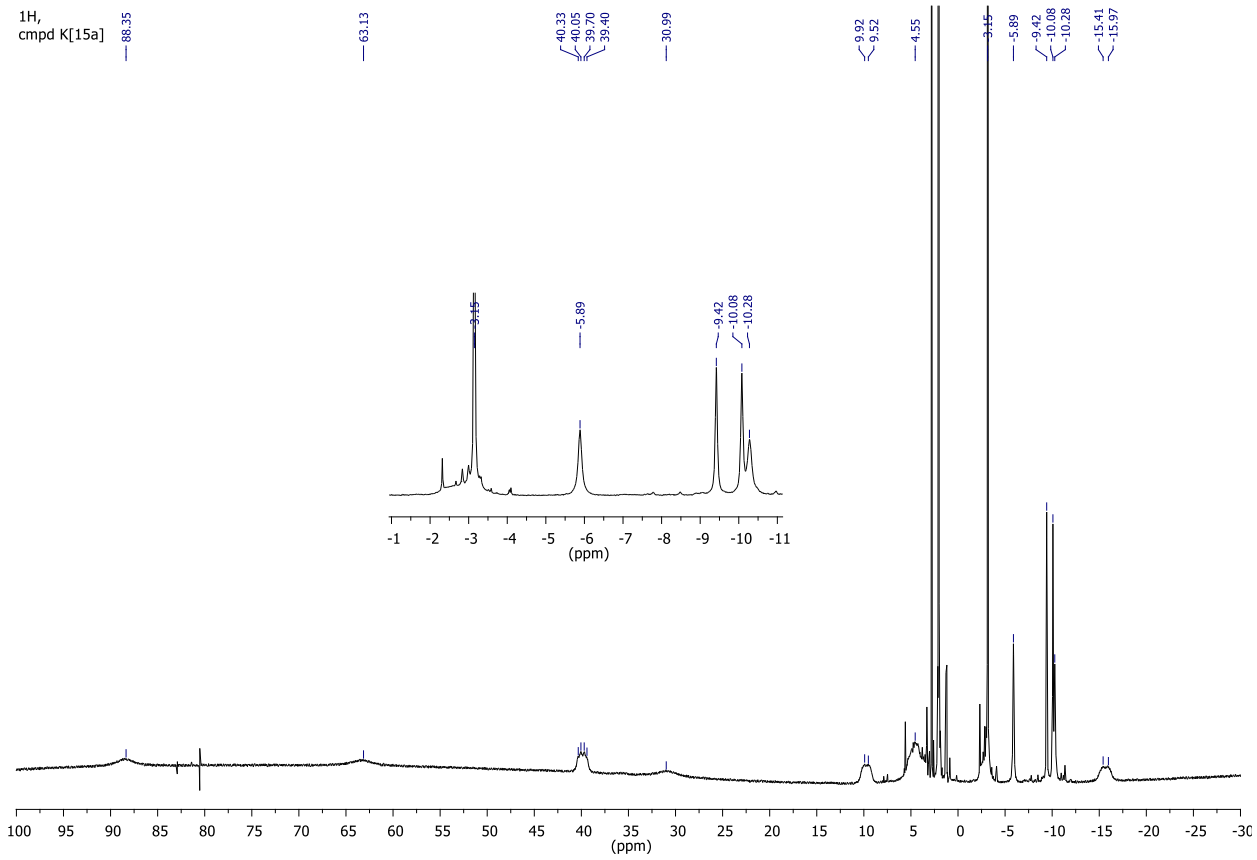
13C{1H}, compound K[14]

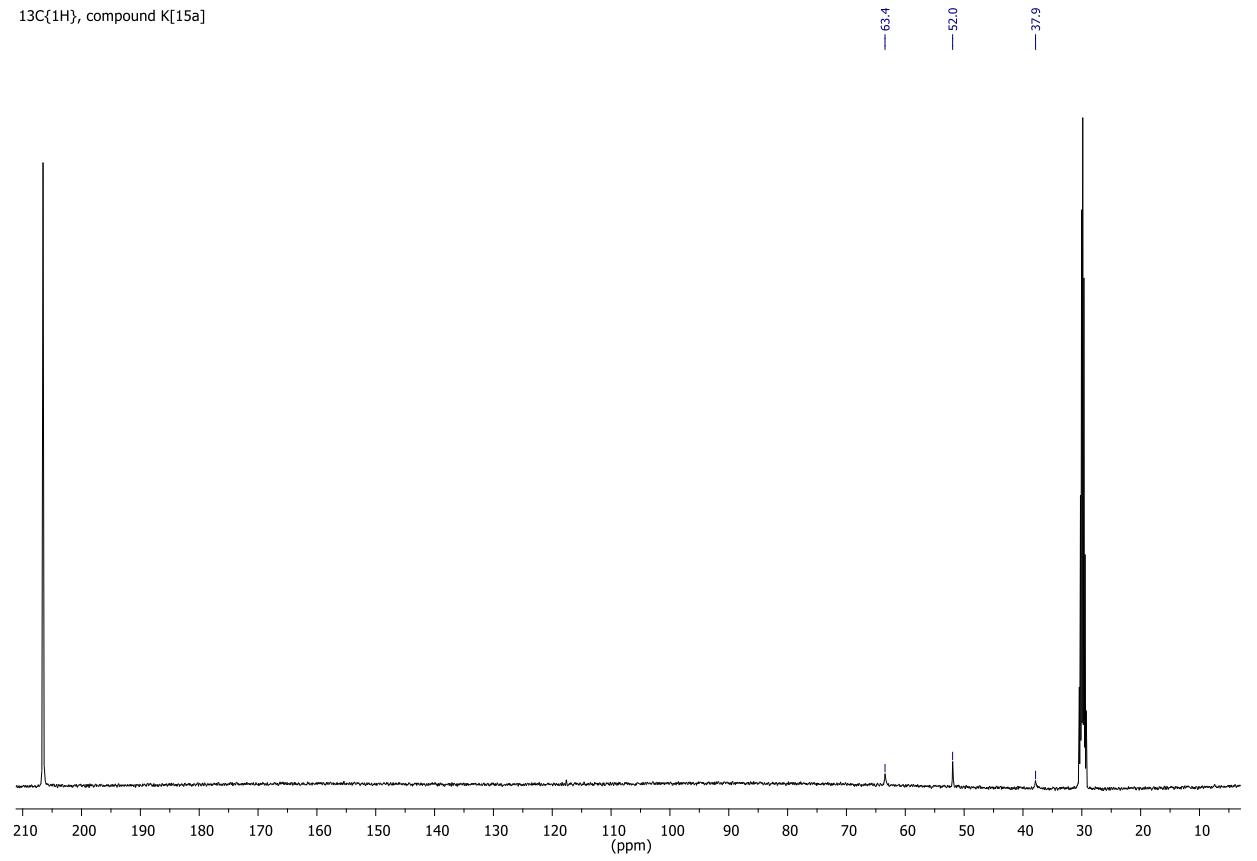


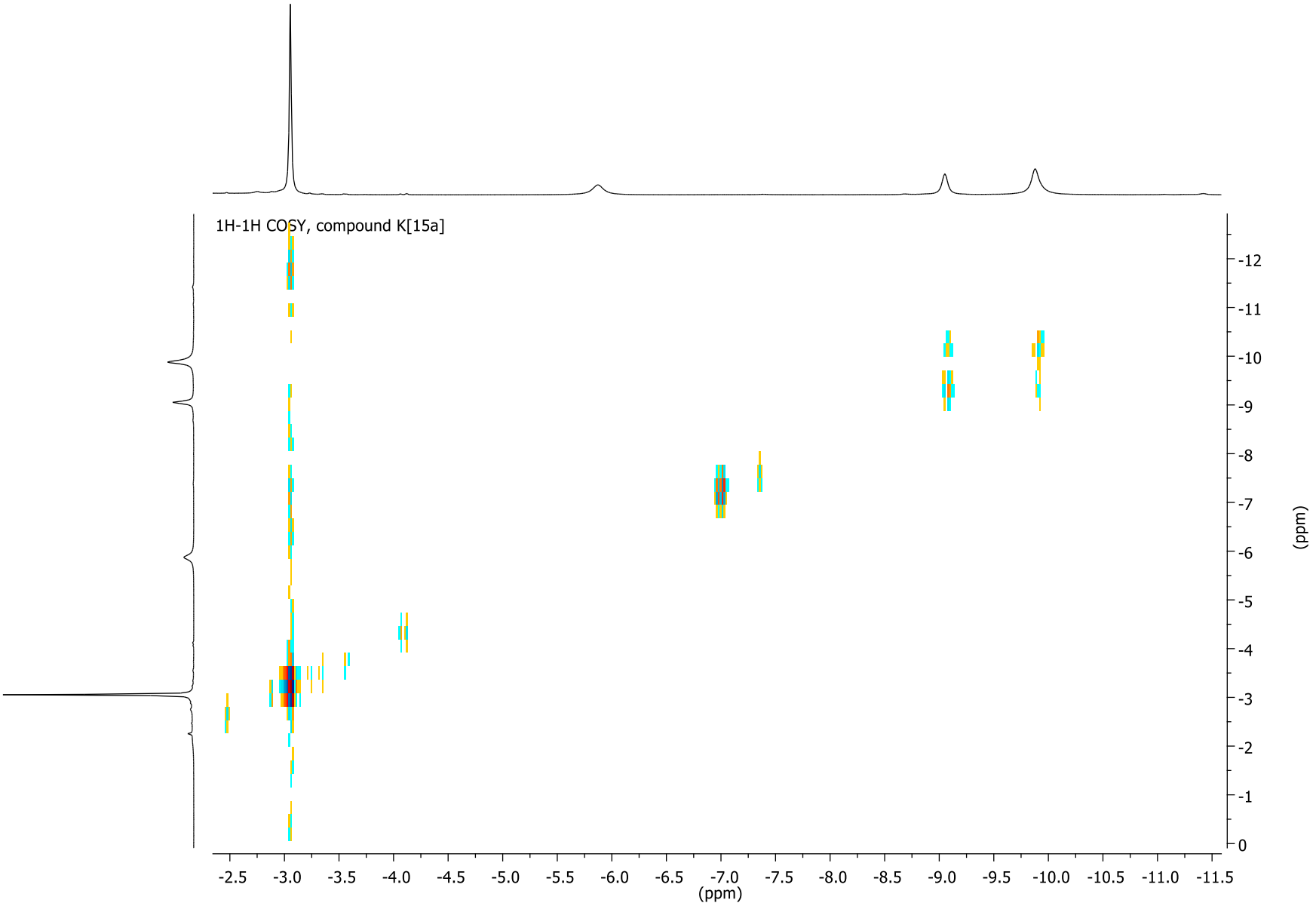

1H-13C ASAPHMQC, compound K[14]
4.6
4.5
4.4
4.3
4.2
4.1
4.0
3.9
3.8
3.7
3.6
3.5
3.4
3.3
3.2
3.1
3.0
2.9
(ppm)
40
45
50
55
60
65
70
75
80
85
(ppm)

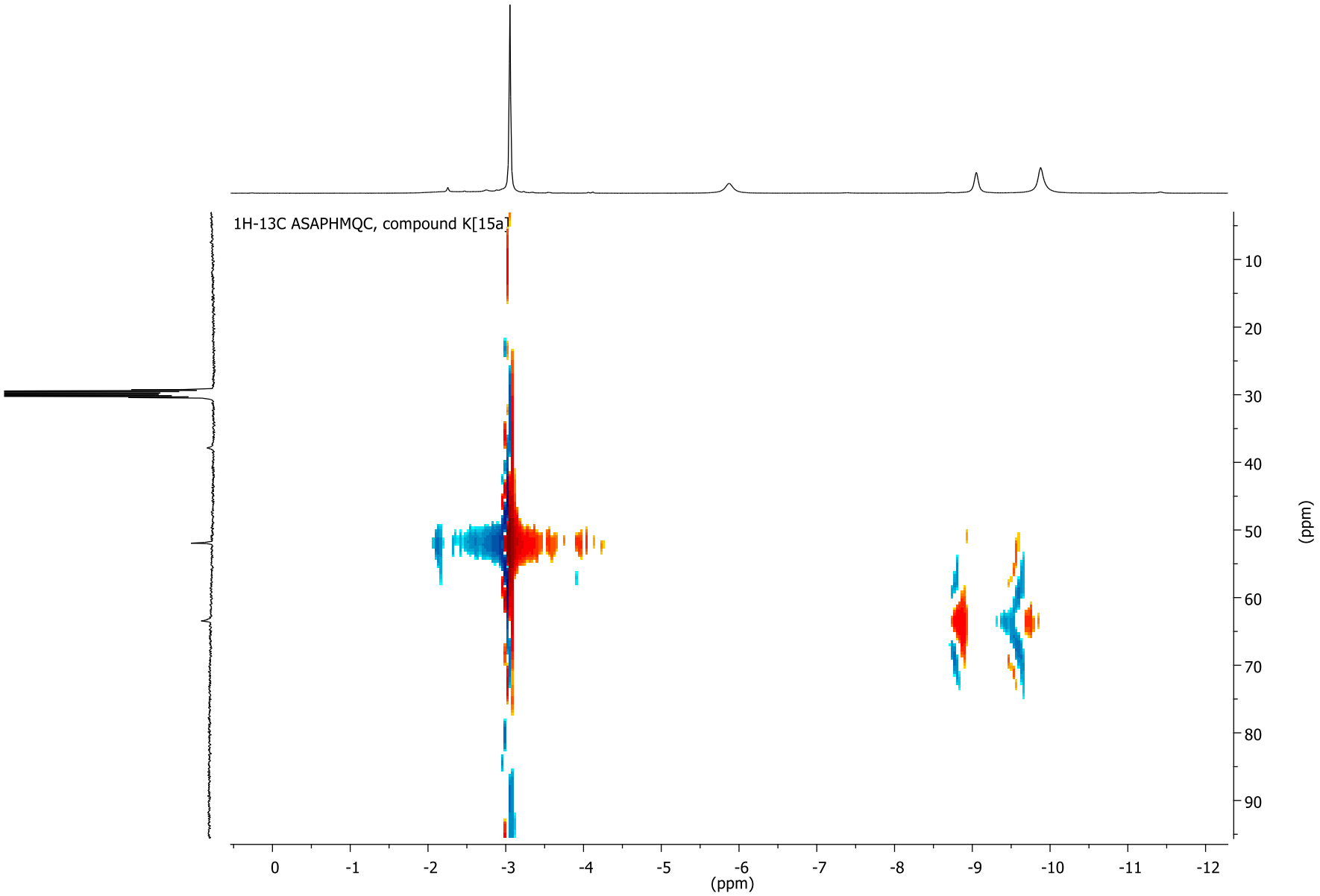

11B{1H},
cmpd K[15a]
101.2
51.1
34.9
8.0
-38.2
-43.2
-393.6
-454.7
-487.9
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
-450
-500
(ppm)

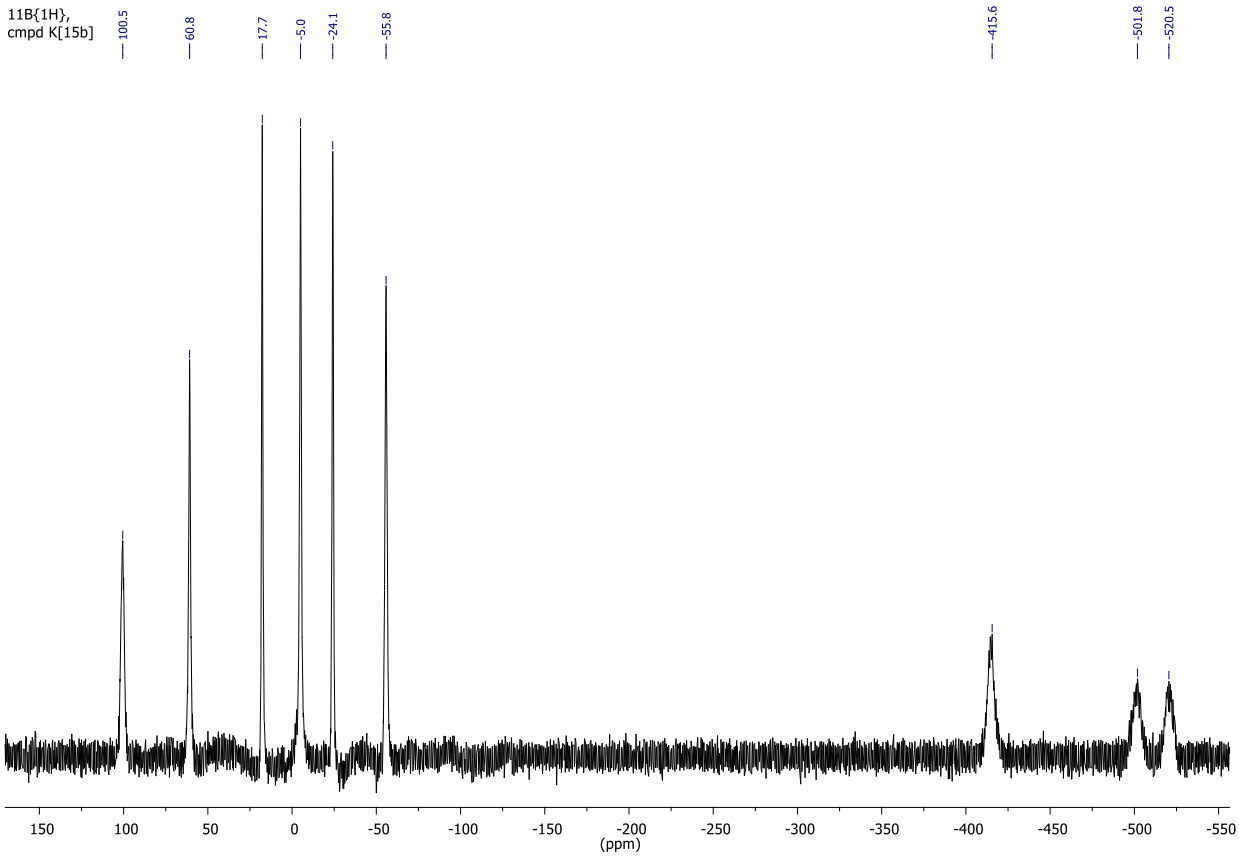

11B,
cmpd K[15a]
100.5
35.0
34.4
8.4
7.7
-37.8
-38.4
-42.8
-43.3
-392.3
-453.4
-486.6
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
-450
-500
-550
(ppm)

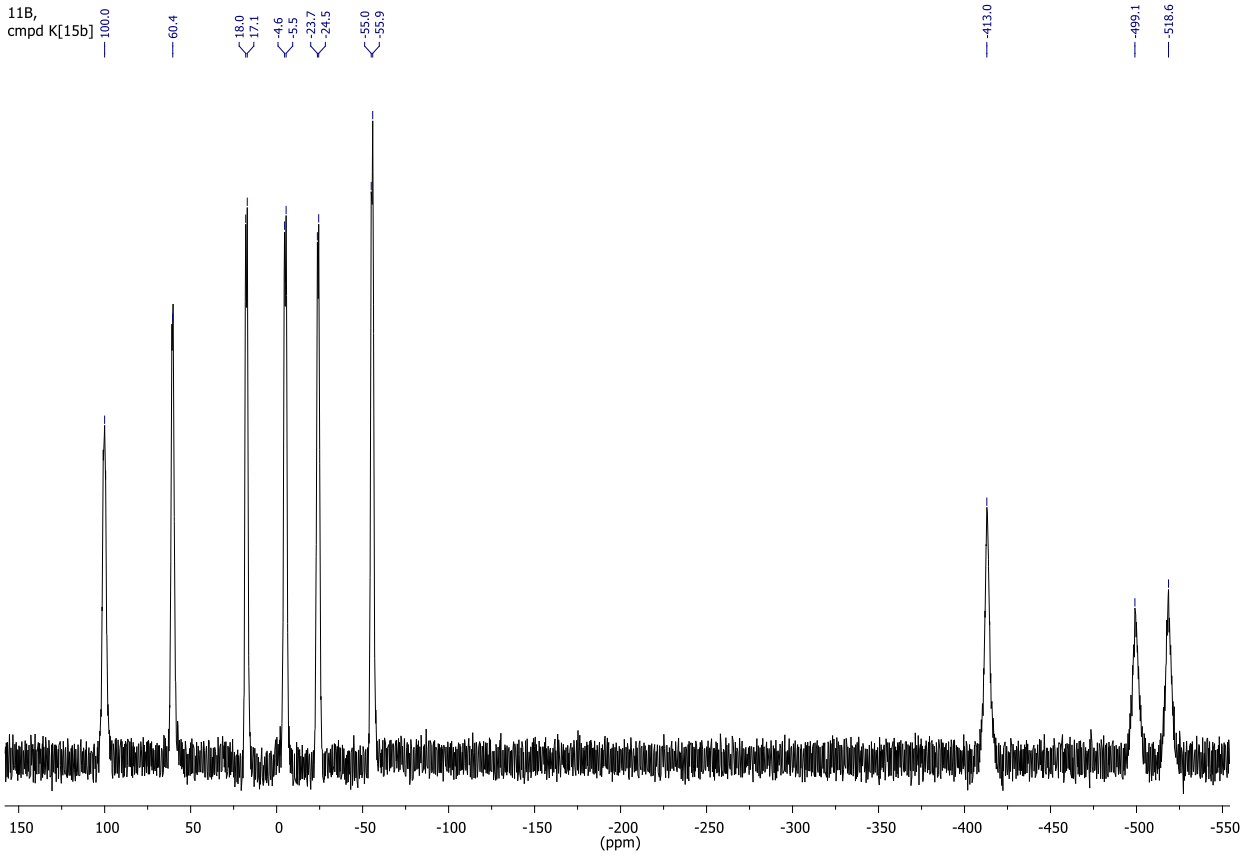

1H,
cmpd K[15a]
88.35
63.13
40.33
40.05
39.70
39.40
30.99
9.92
9.52
4.55
3.15
-5.89
-9.42
-10.08
-10.28
-15.41
-15.97
(ppm)

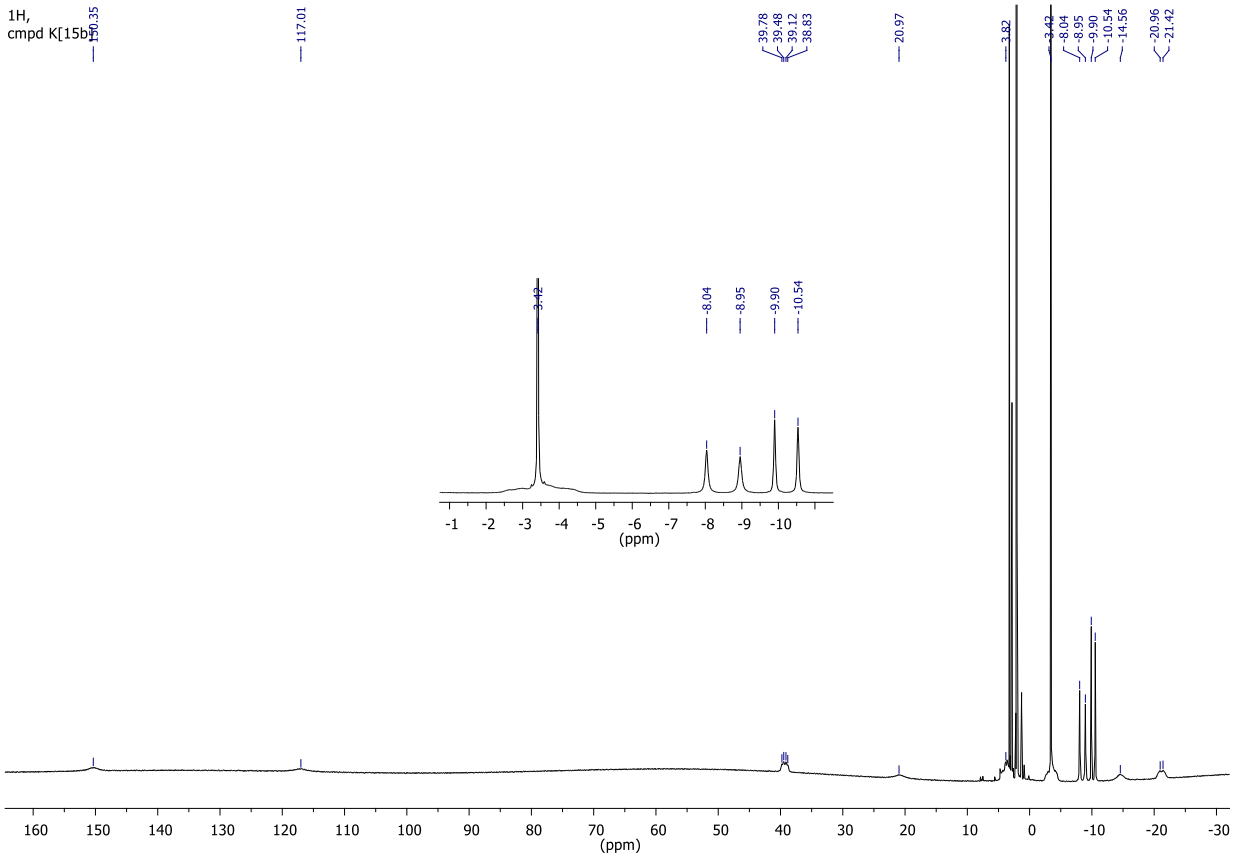

13C{1H}, compound K[15a]
63.4
52.0
37.9
(ppm)

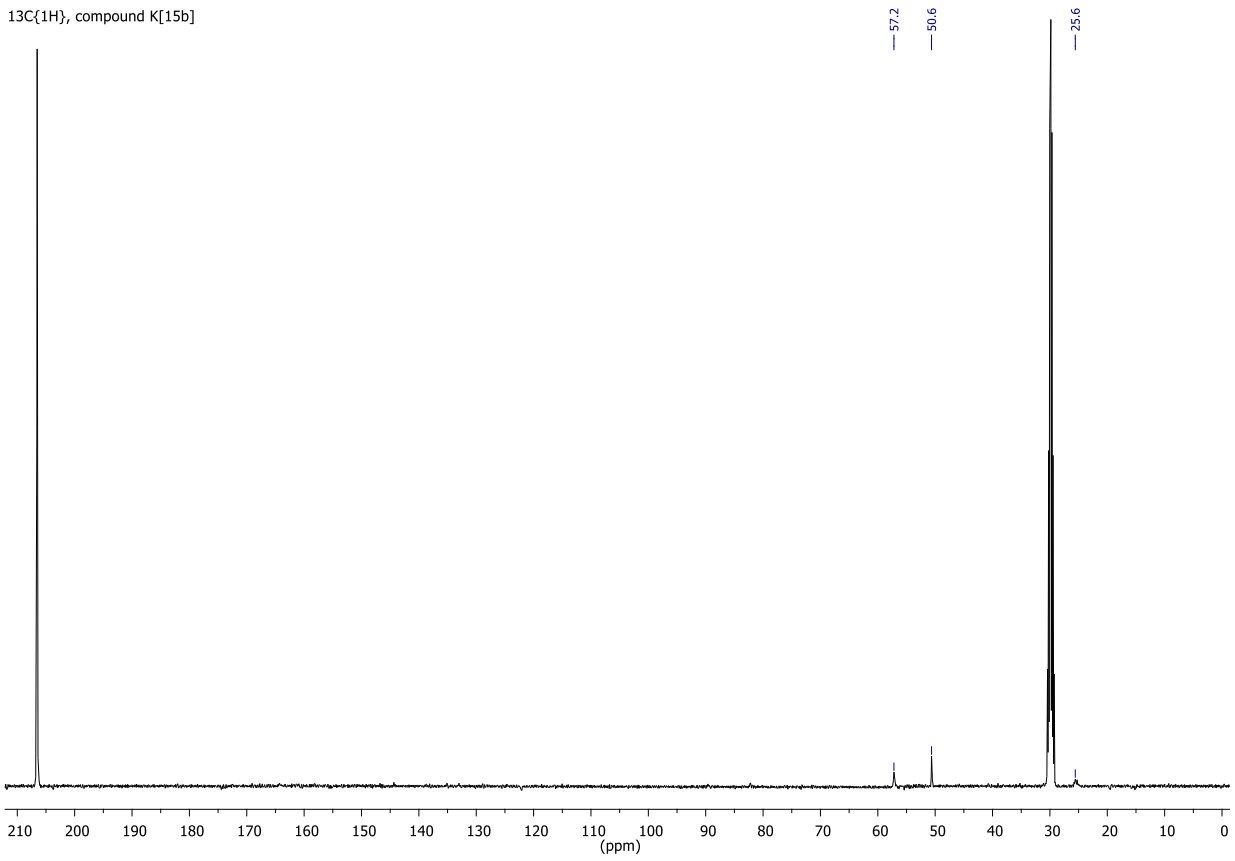

1H-1H COSY, compound K[15a]
-12
-11
-10
-9
-8
-7
-6
-5
-4
-3
-2
-1
0
(ppm)
-2.5
-3.0
-3.5
-4.0
-4.5
-5.0
-5.5
-6.0
-6.5
-7.0
-7.5
-8.0
-8.5
-9.0
-9.5
-10.0
-10.5
-11.0
-11.5
(ppm)

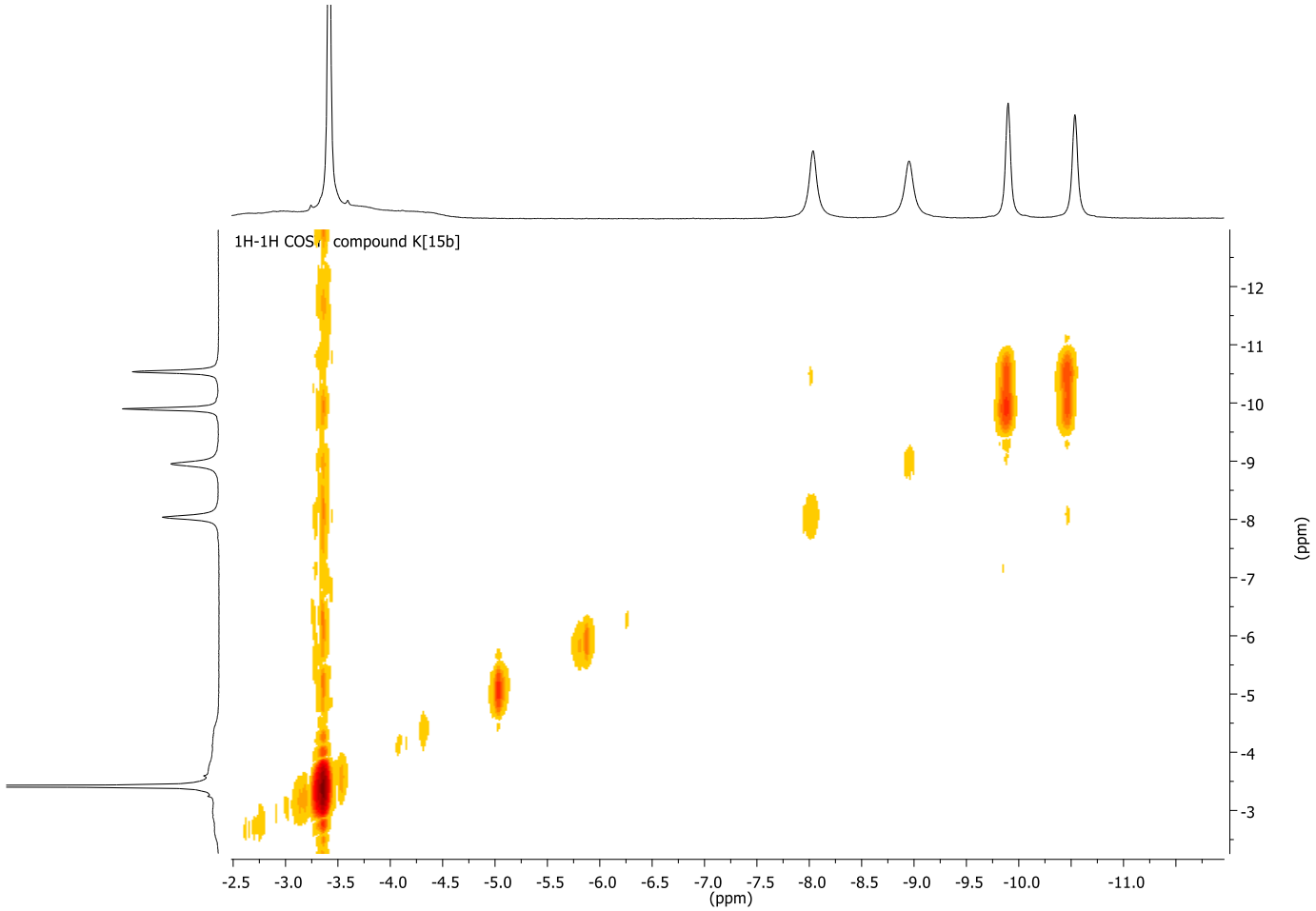

1H-13C ASAPHMQC, compound K[15a]
0
-1
-2
-3
-4
-5
-6
-7
-8
-9
-10
-11
-12
(ppm)
10
20
30
40
50
60
70
80
90
(ppm)

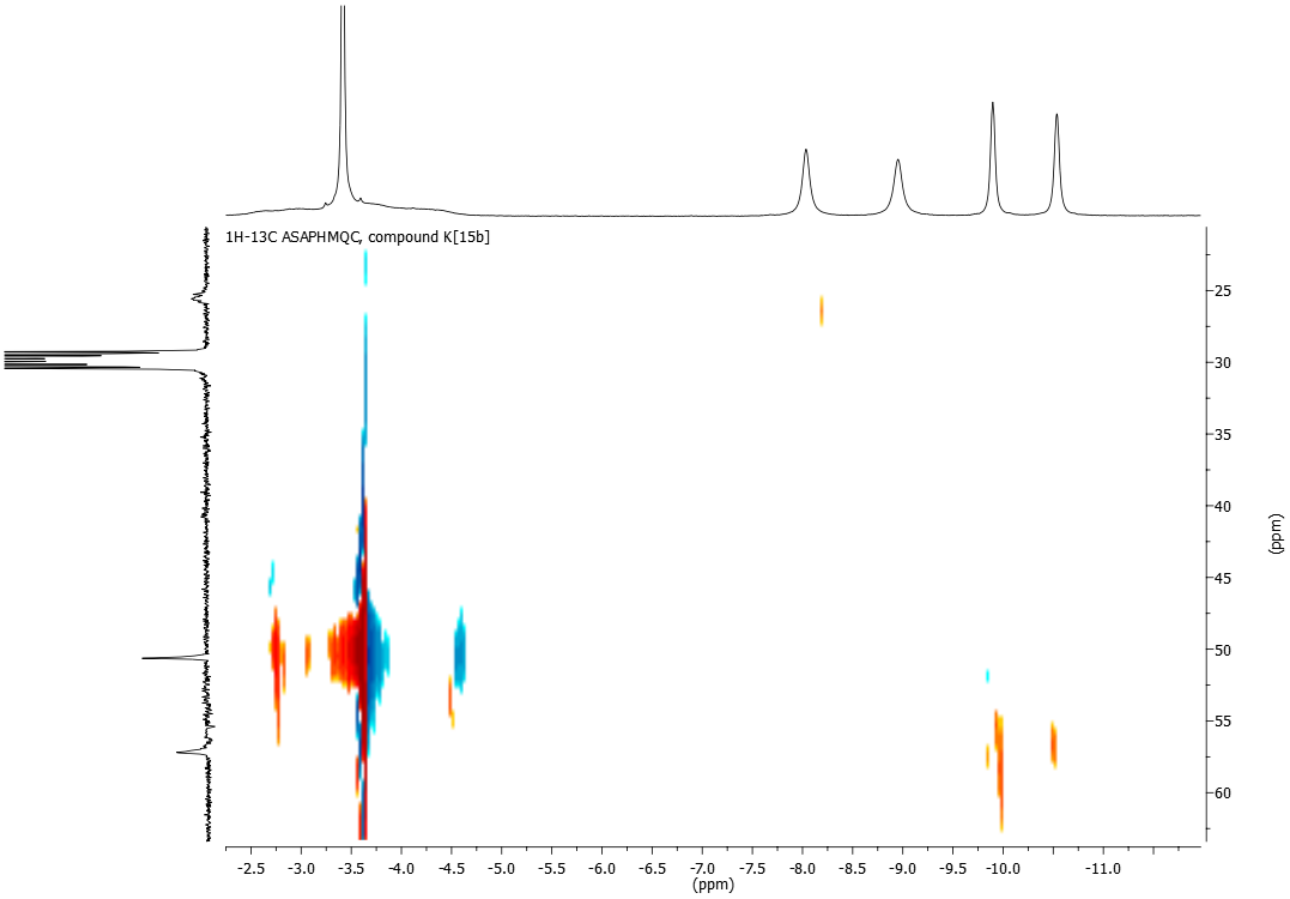

11B{1H},
cmpd K[15b]
100.5
60.8
17.7
-5.0
-24.1
-55.8
-415.6
-501.8
-520.5
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
-450
-500
-550
(ppm)


11B,
cmpd K[15b]
100.0
60.4
18.0
17.1
-4.6
-5.5
-23.7
-24.5
-55.0
-55.9
-413.0
-499.1
-518.6
150
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
-450
-500
-550
(ppm)


1H,
cmpd K[15b]
150.35
117.01
39.78
39.48
39.12
38.83
20.97
3.82
3.42
-8.04
-8.95
-9.90
-10.54
-14.56
-20.96
-21.42
(ppm)


13C{1H}, compound K[15b]
57.2
50.6
25.6
(ppm)


1H-1H COSY, compound K[15b]
-12
-11
-10
-9
-8
-7
-6
-5
-4
-3
(ppm)
-2.5 -3.0 -3.5 -4.0 -4.5 -5.0 -5.5 -6.0 -6.5 -7.0 -7.5 -8.0 -8.5 -9.0 -9.5 -10.0 -11.0
(ppm)


1H-13C ASAPHMQC, compound K[15b]
25
30
35
40
45
50
55
60
(ppm)
-2.5 -3.0 -3.5 -4.0 -4.5 -5.0 -5.5 -6.0 -6.5 -7.0 -7.5 -8.0 -8.5 -9.0 -9.5 -10.0 -11.0
(ppm)

^{11}B NMR study of reaction of [9-I-7,8-$C_2B_9H_{11}$]$^-$ with 1,2-dimethoxyethane.