Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Selective Palladium-Catalysed Arylation of Dihalopyridines Using N-Heterocyclic Carbene Ligands

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Felix-Hausdorff-Straße 4, D-17487 Greifswald, Germany.

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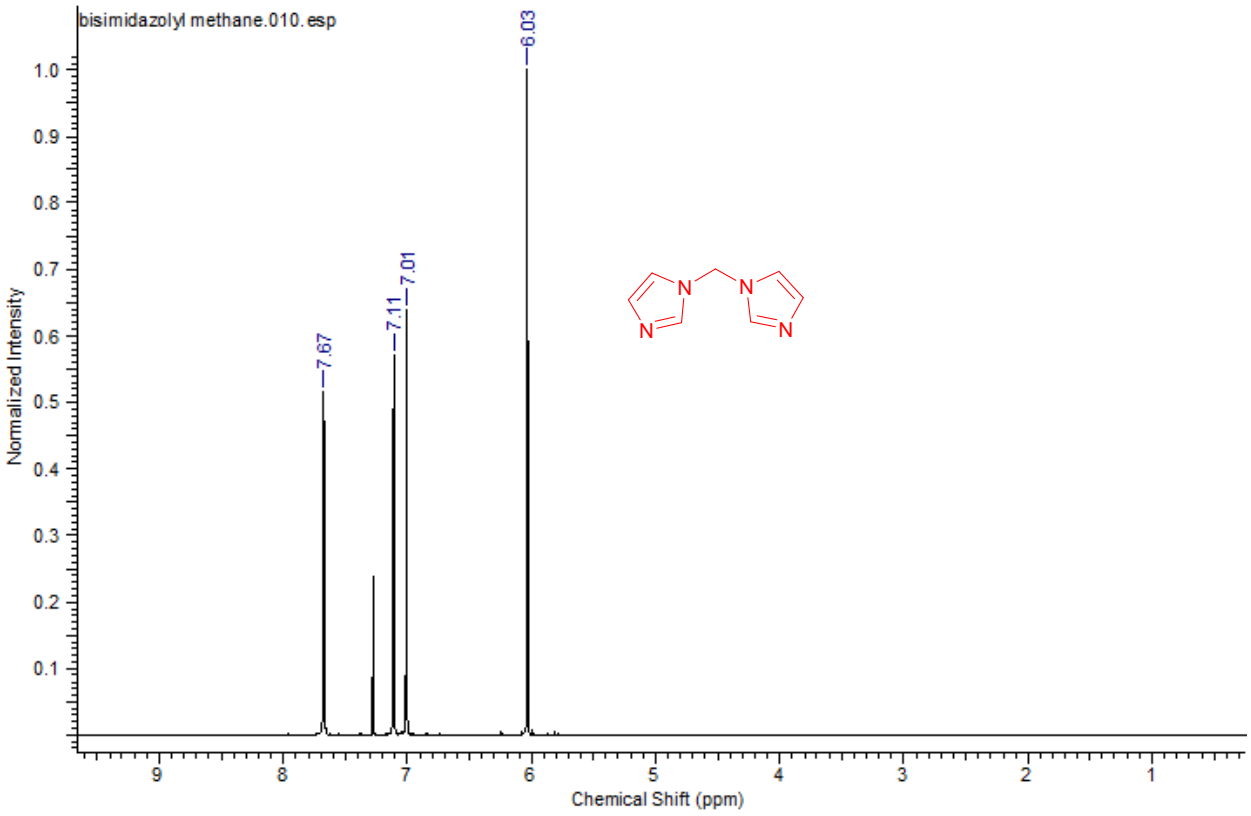
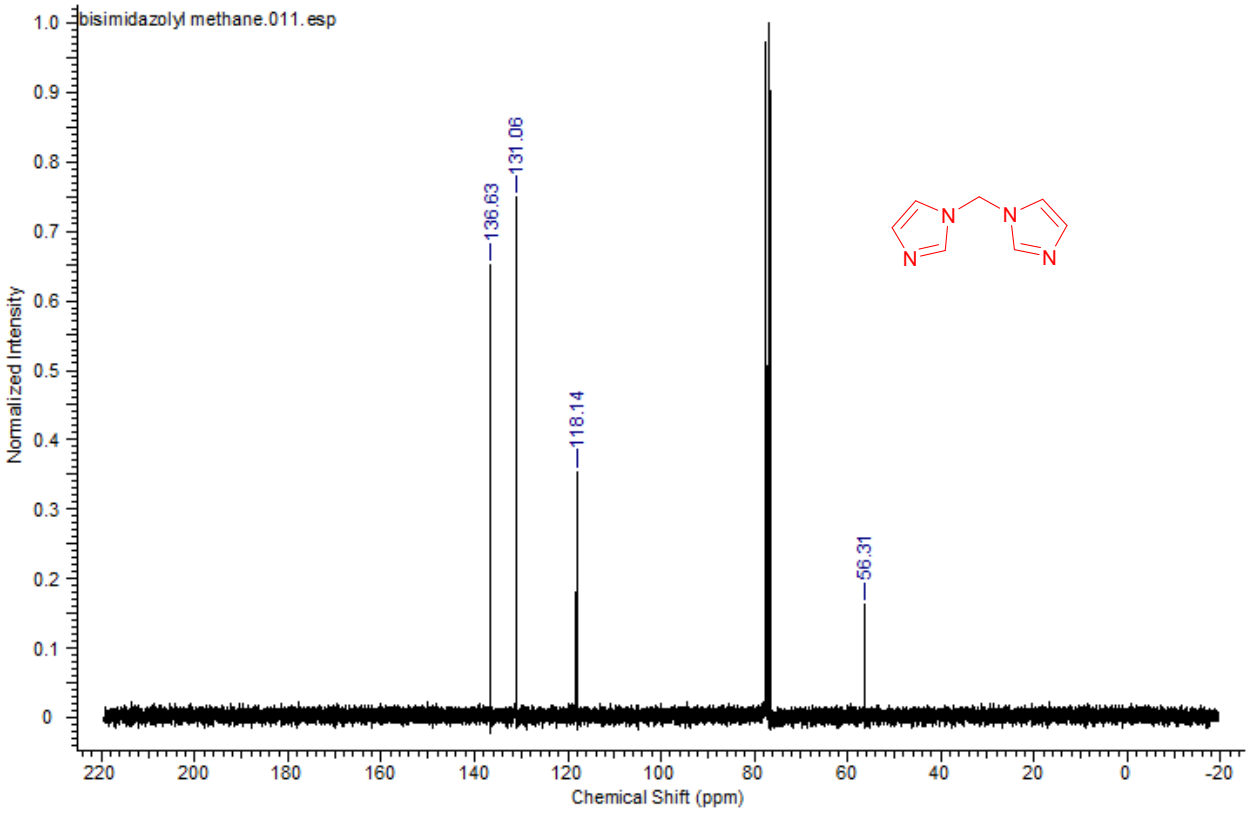
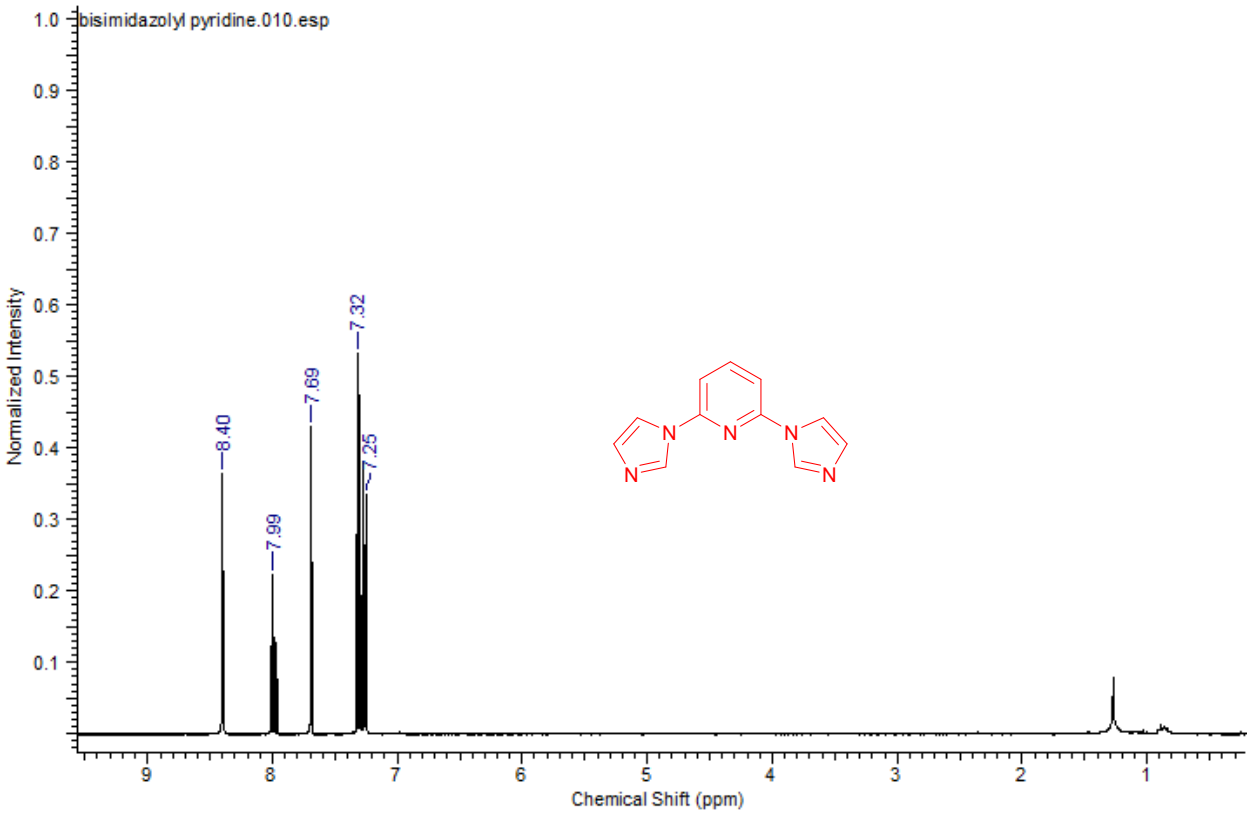
1H, 13C NMRs for all the compounds



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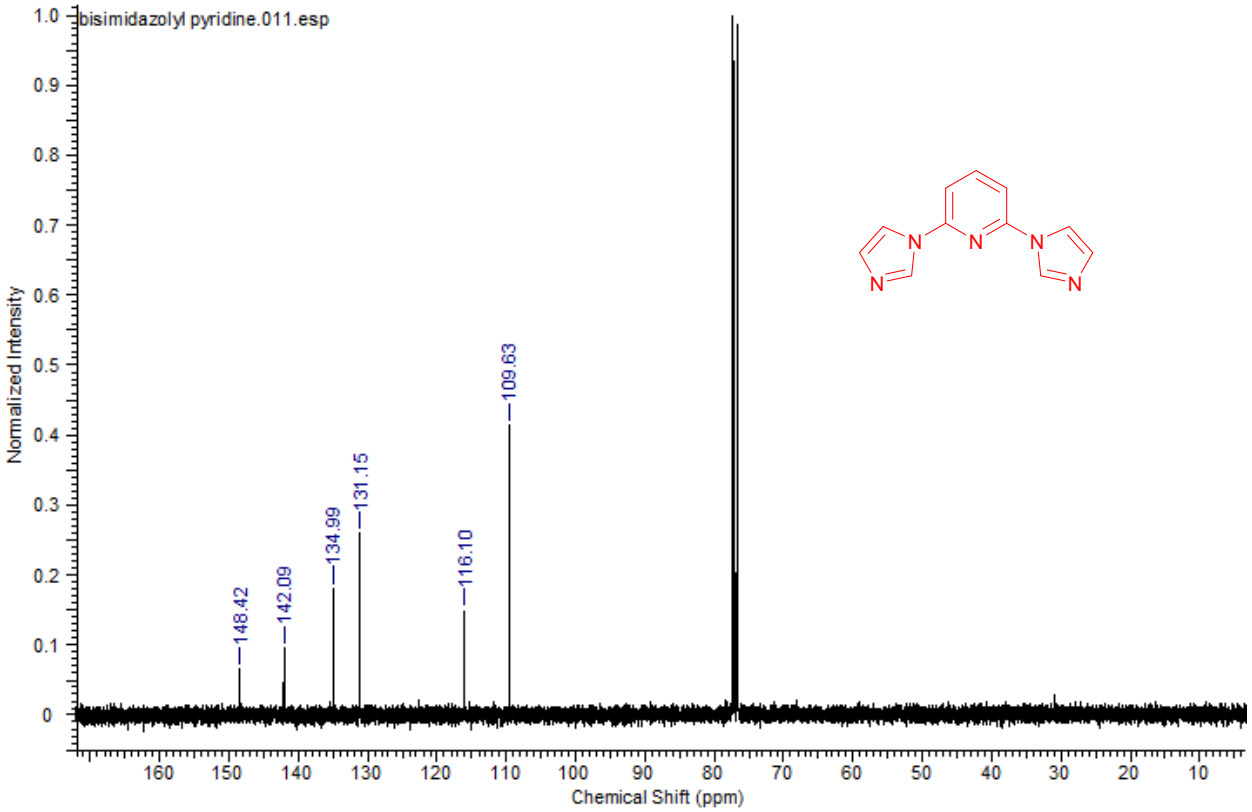
N
N
N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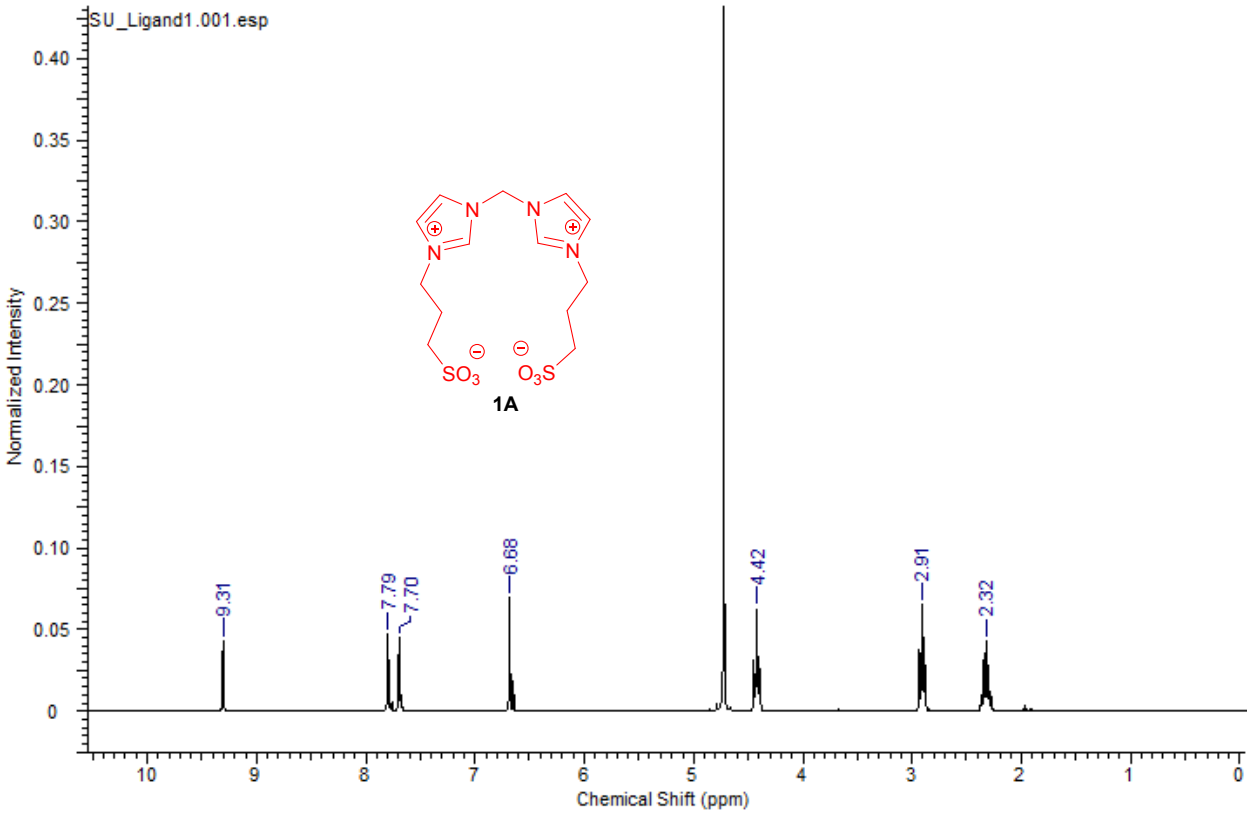
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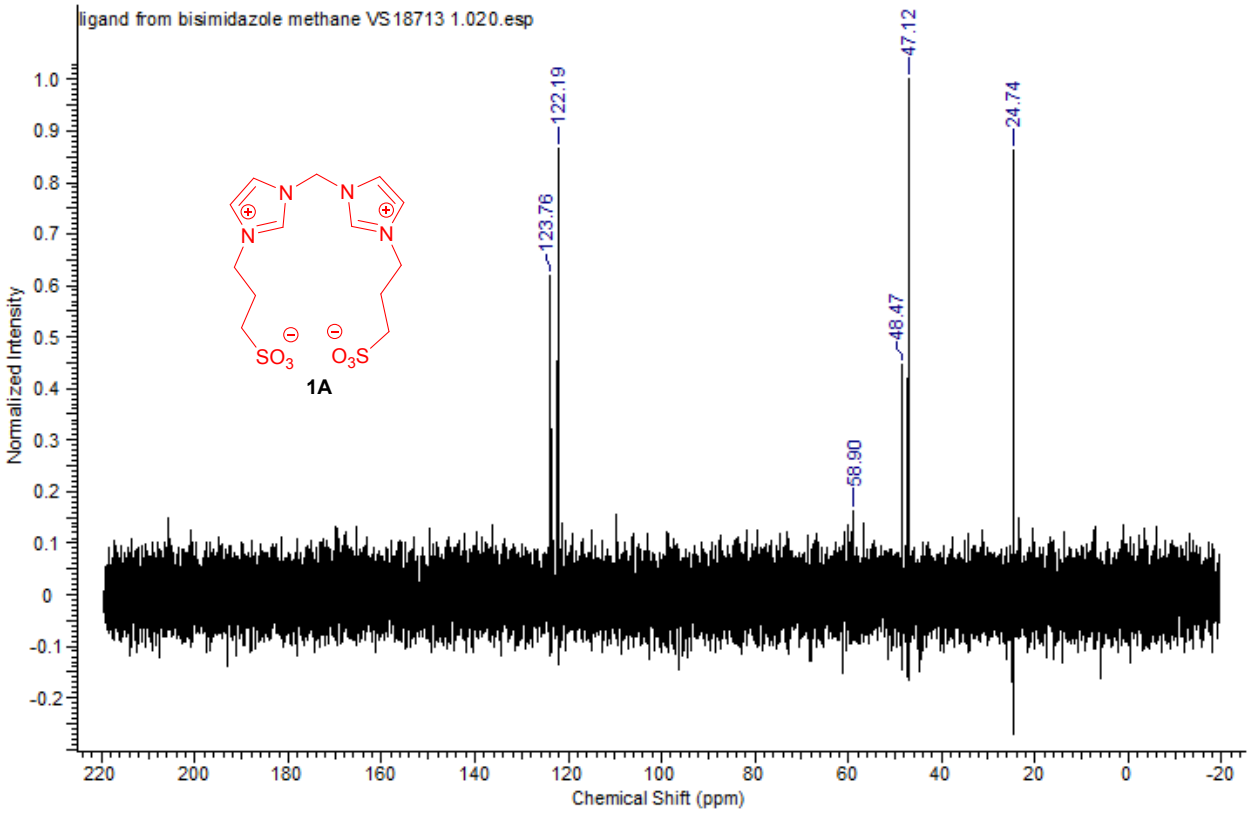
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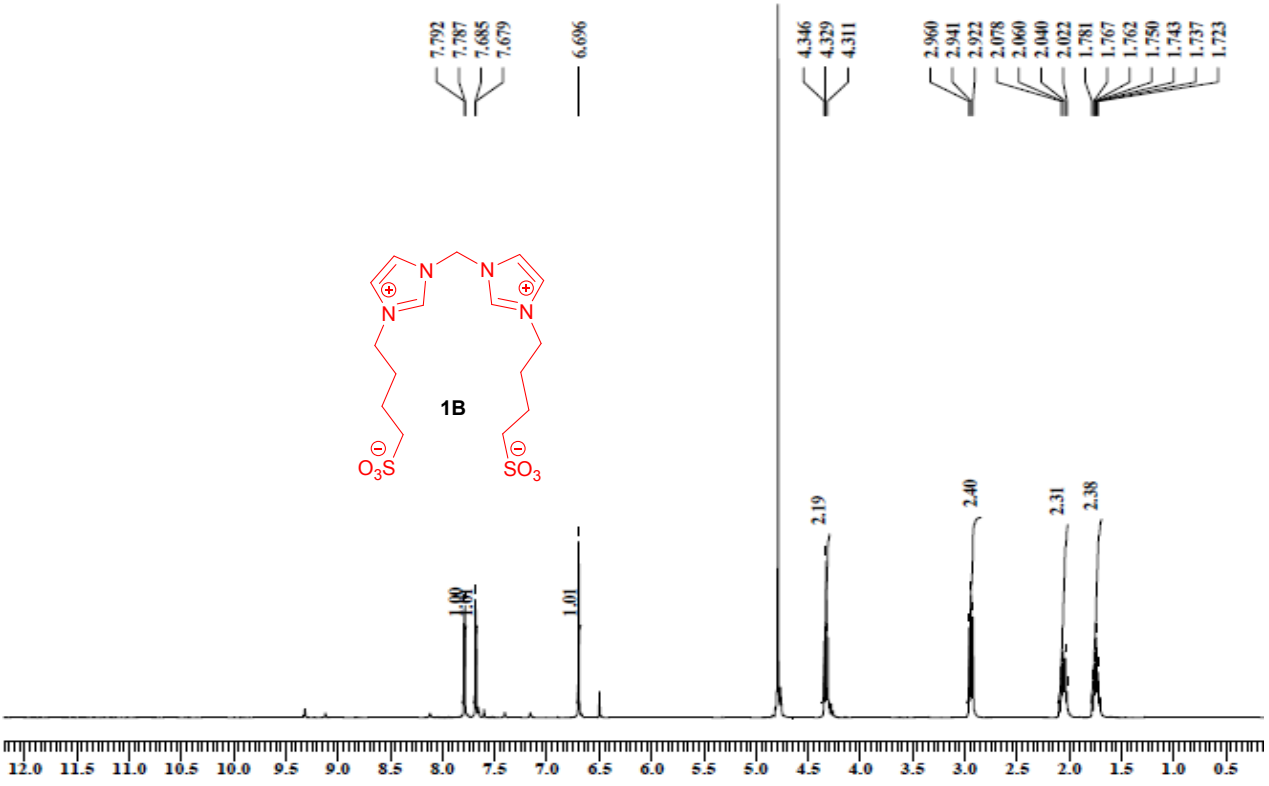
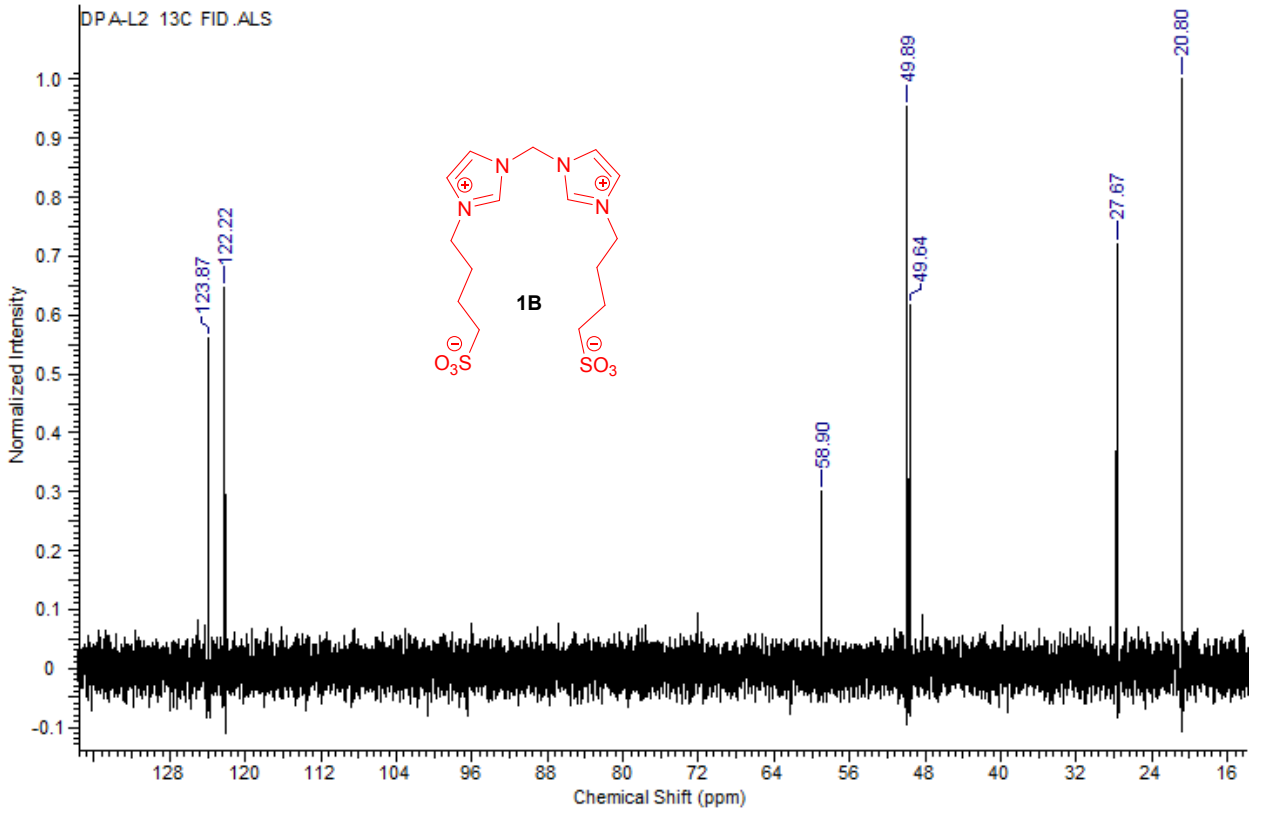
1A

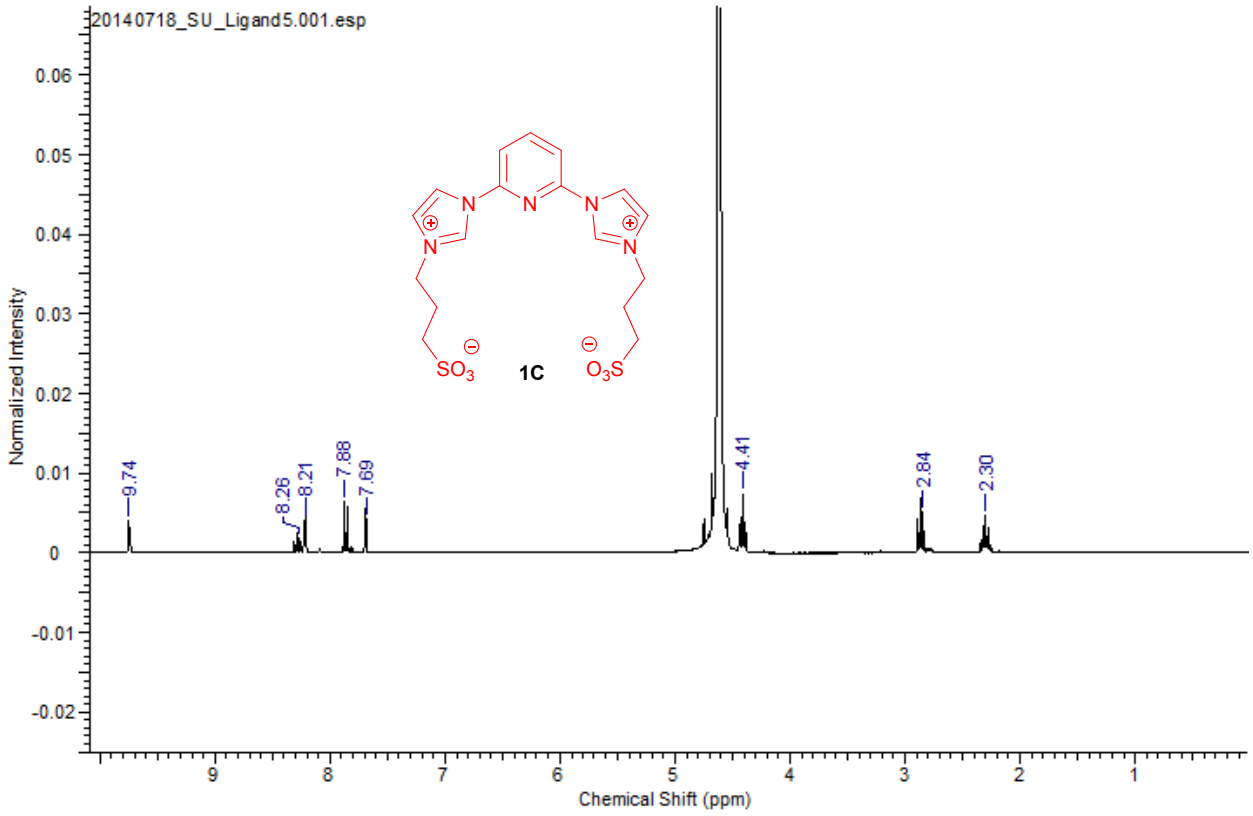
SAPALA ORGANICS PVT LTD

DPA-L2 1H NMR D2O

Analysed By : MSR



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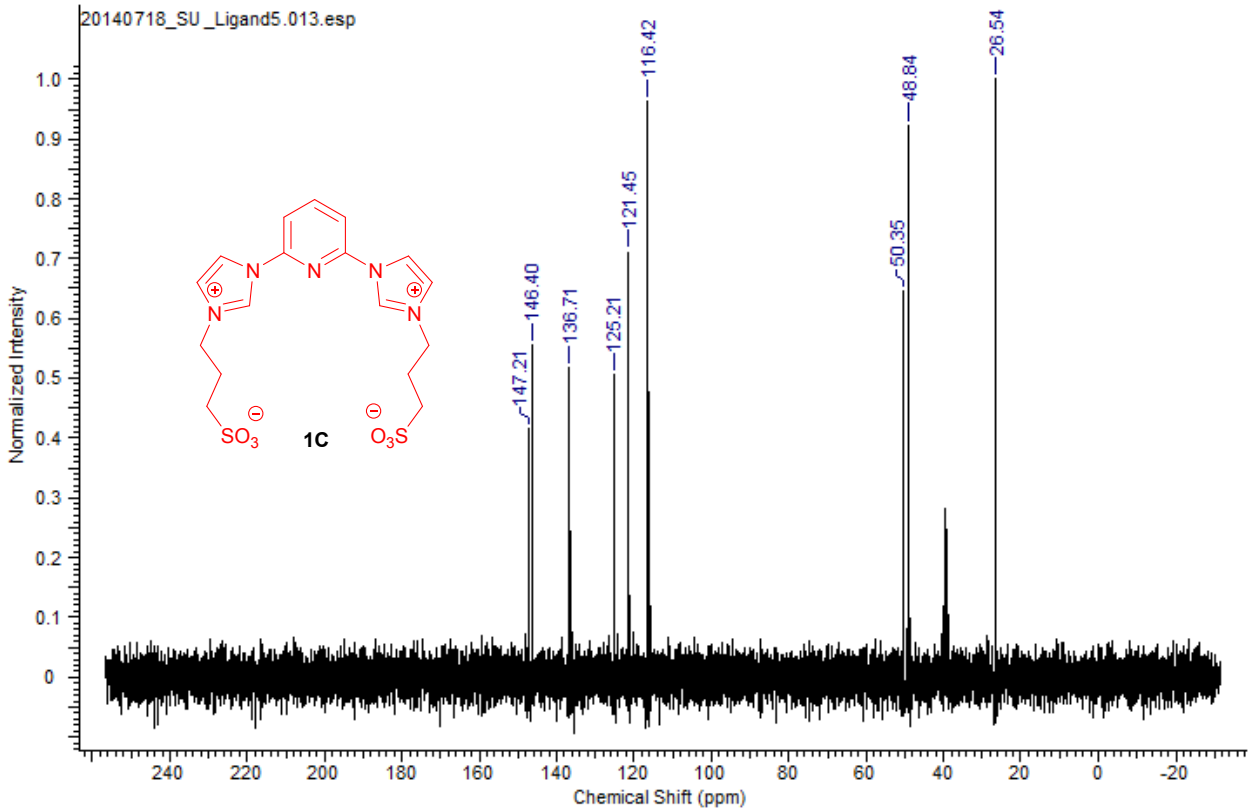
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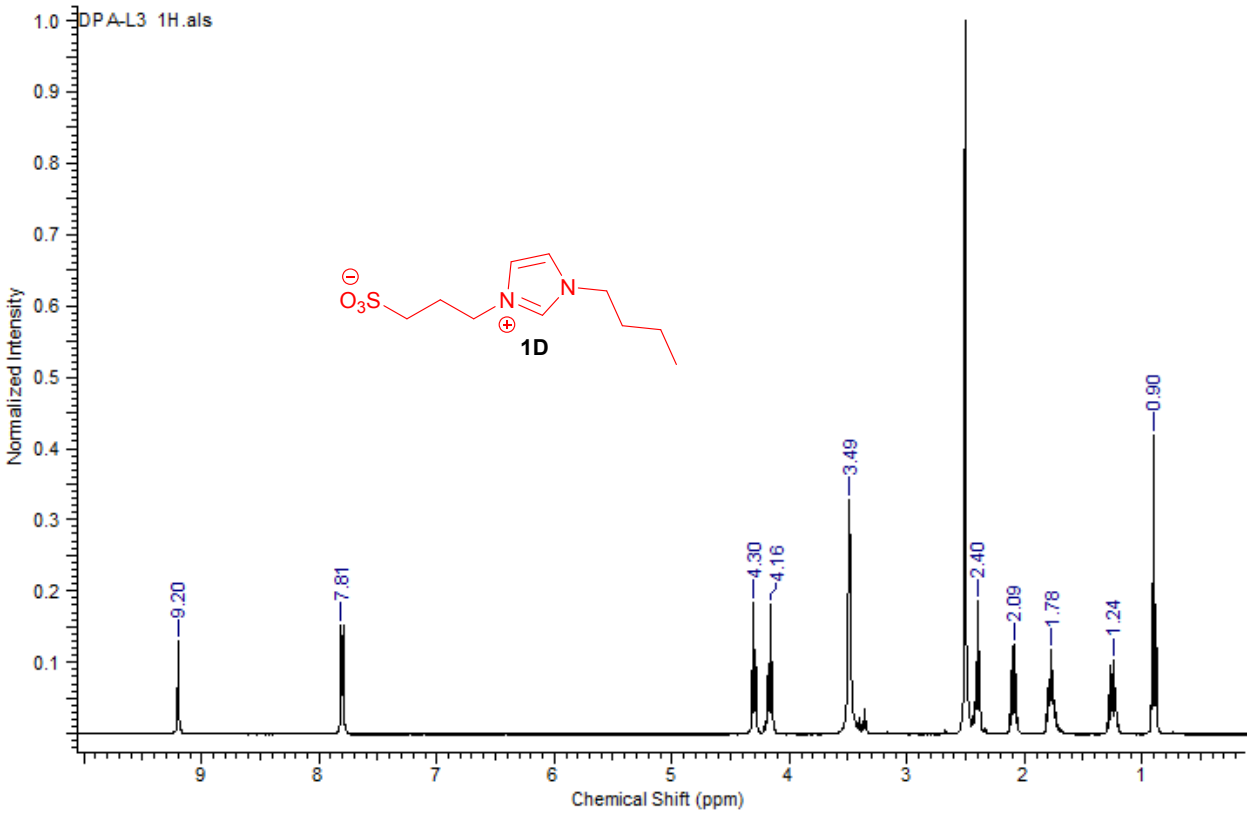
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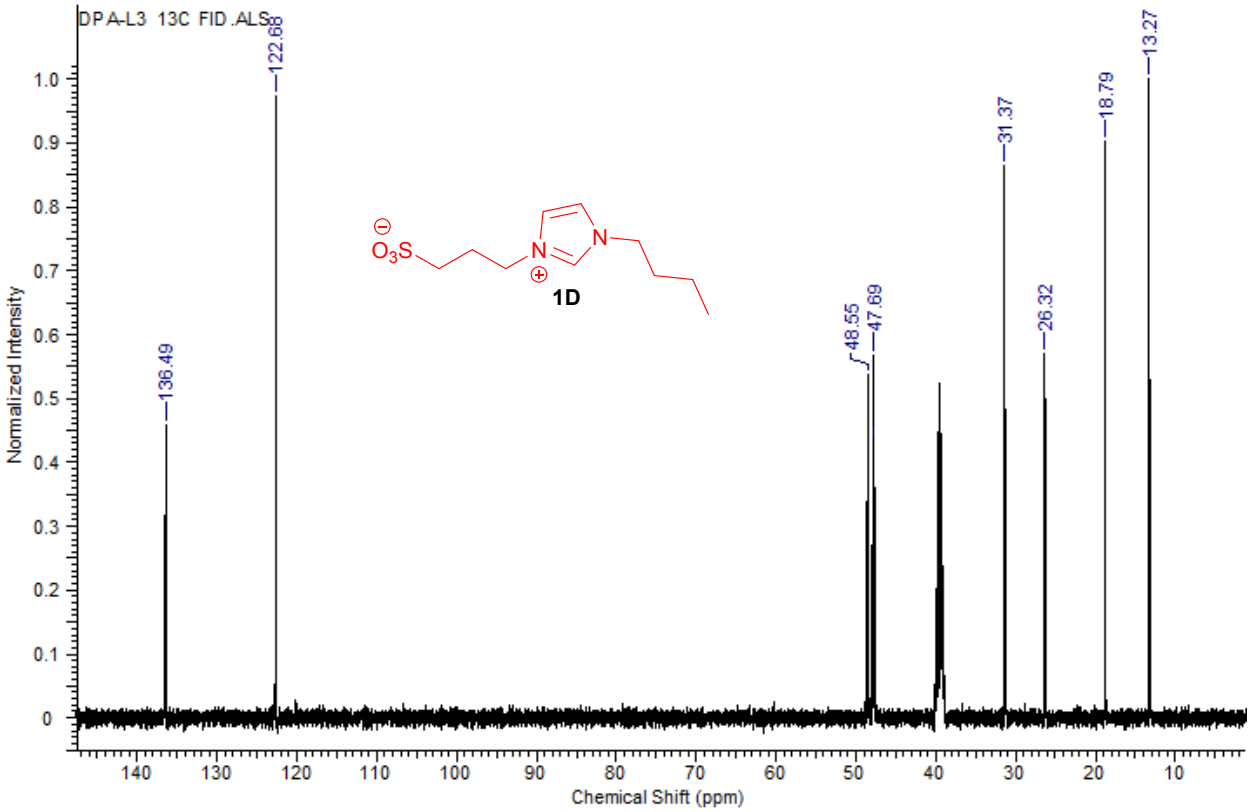
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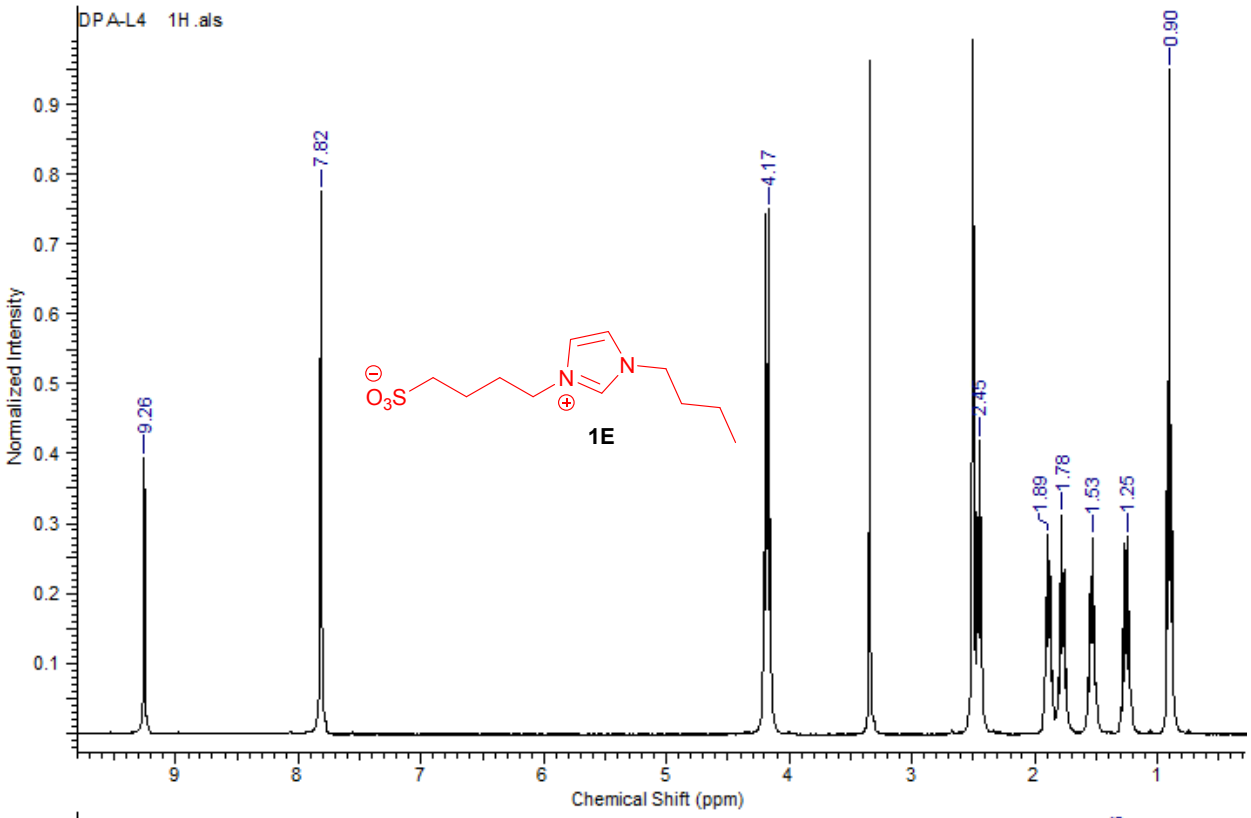
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0.90
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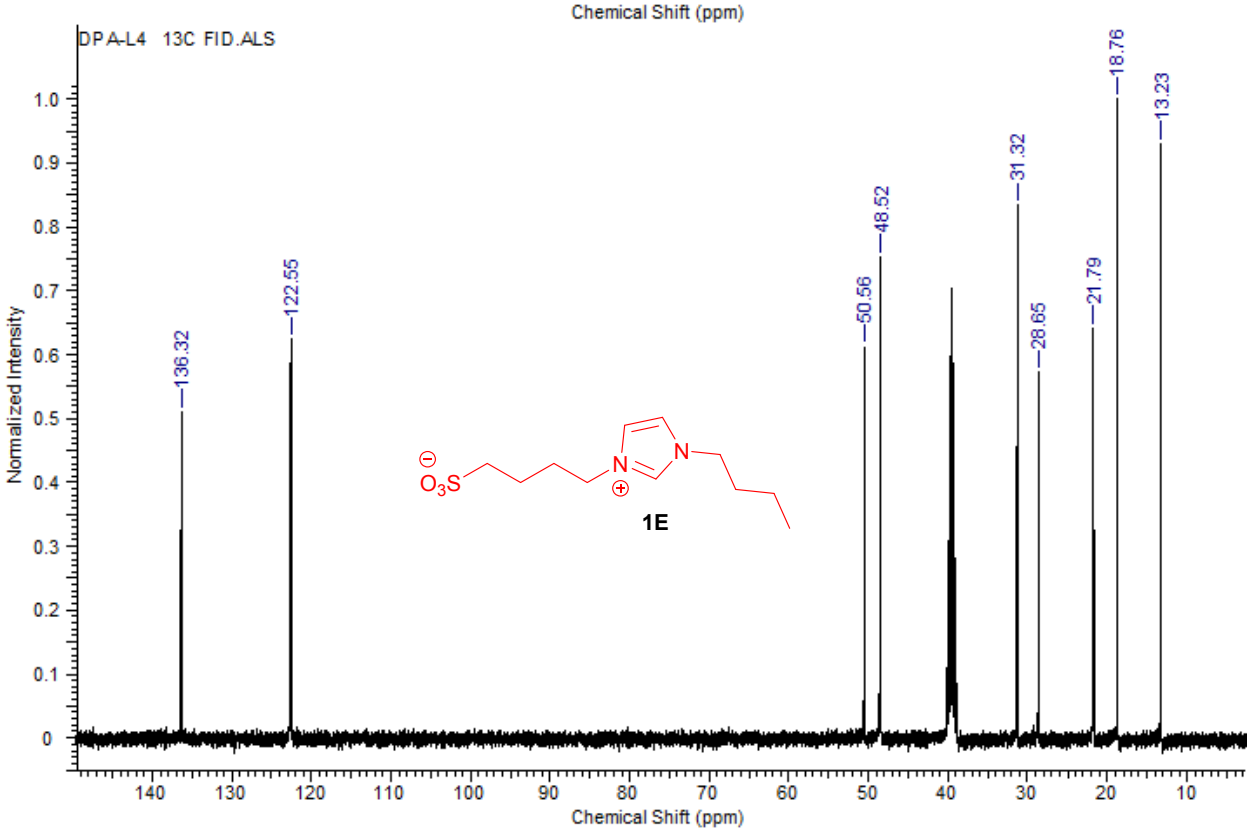
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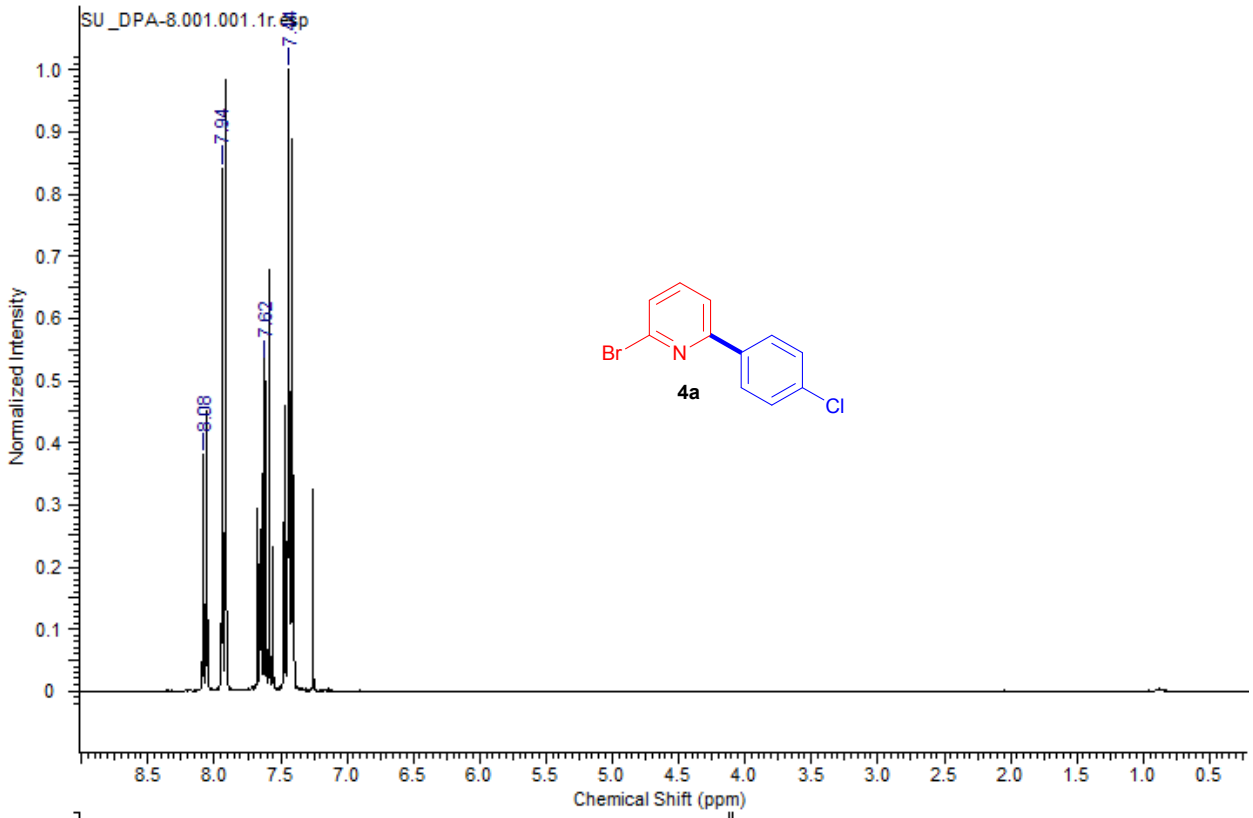
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Cl
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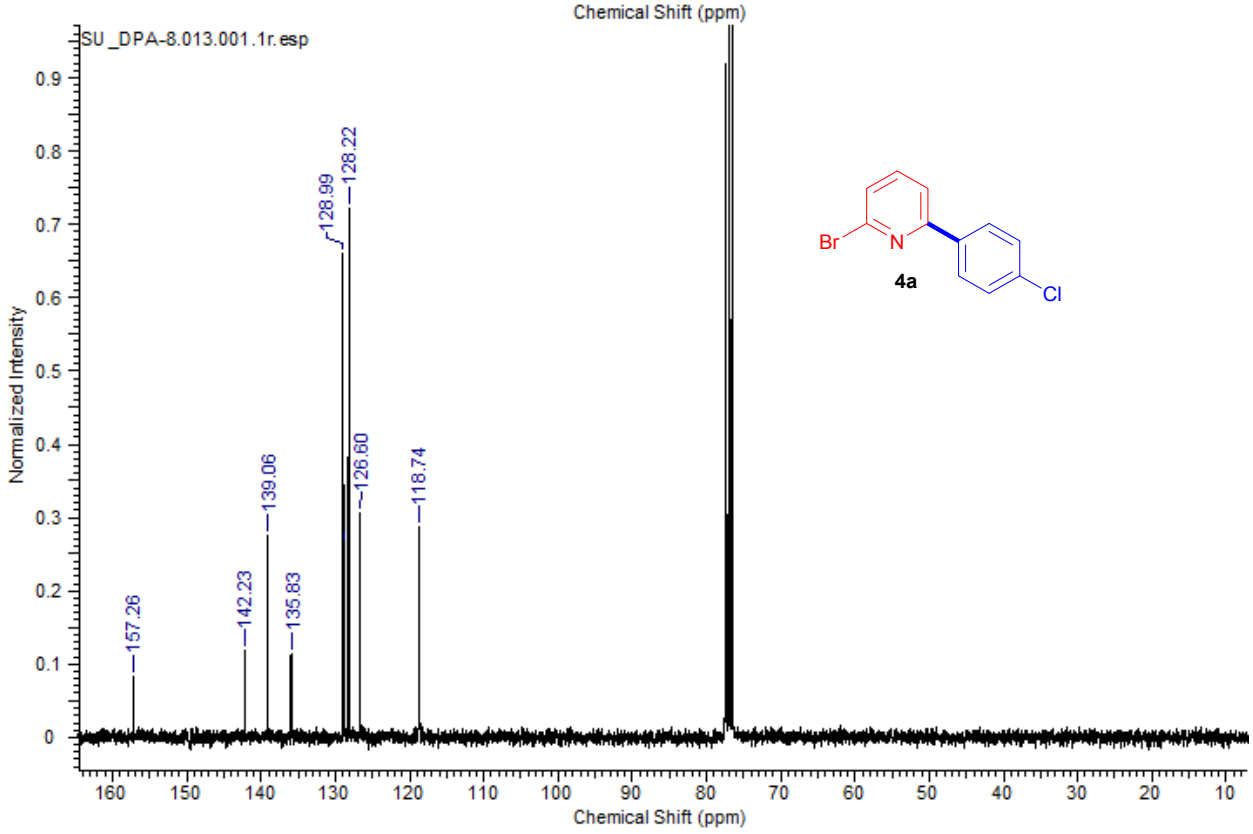
Chemical Shift (ppm)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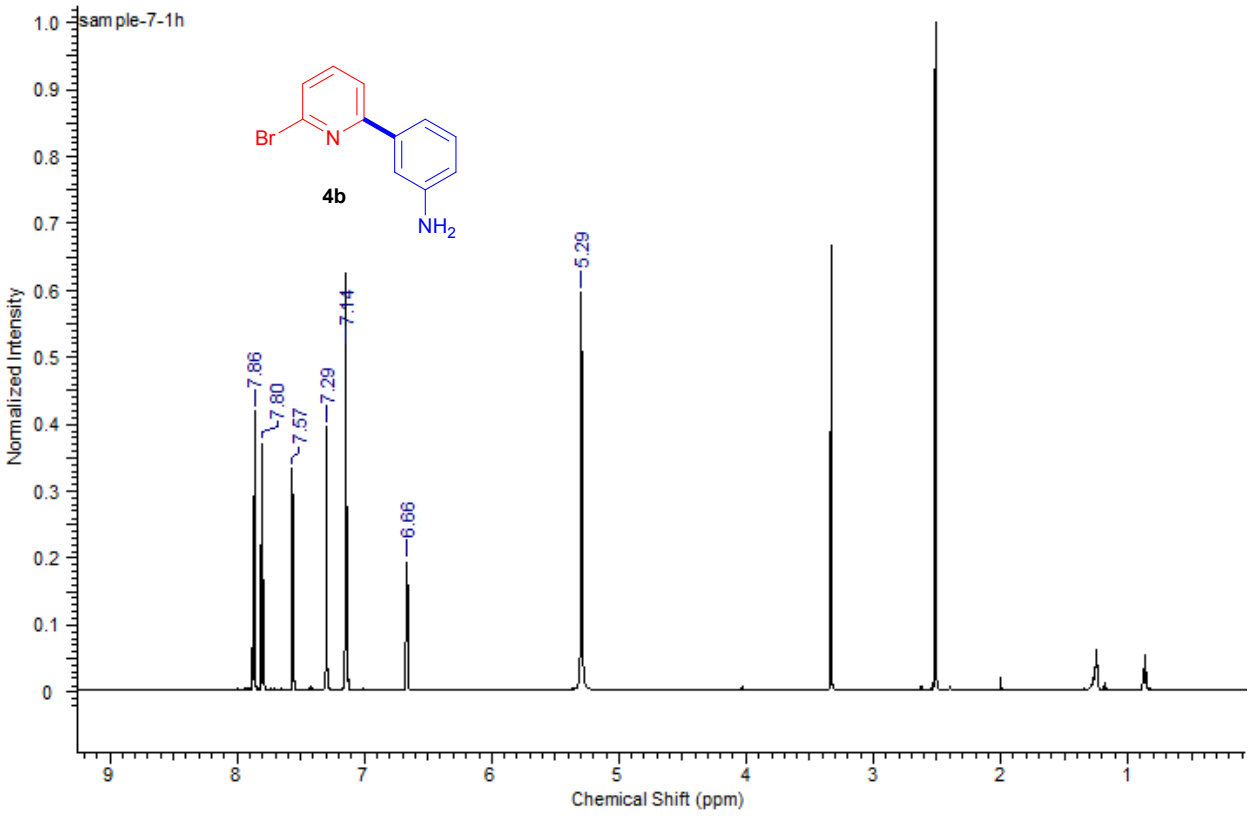
Cl
4a
Chemical Shift (ppm)

.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NH2
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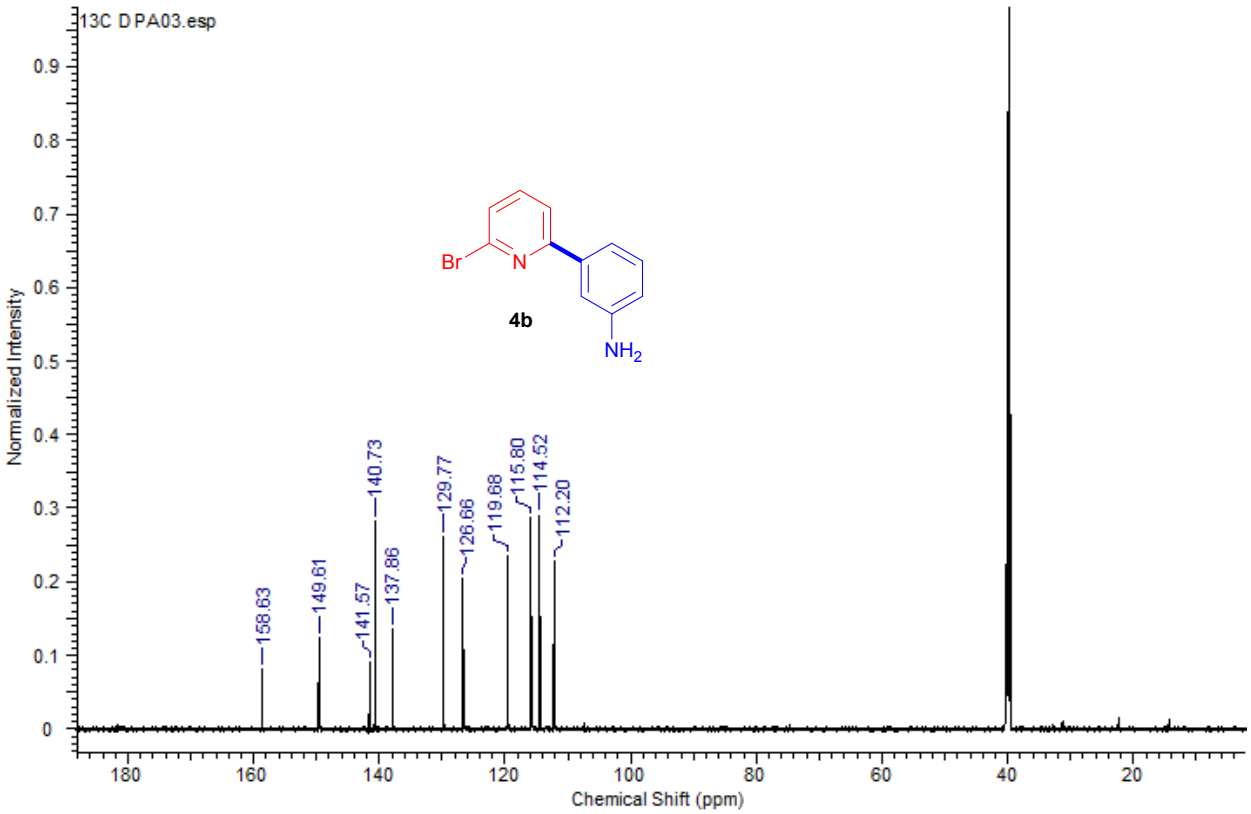
7.14
6.66
5.29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NH2
4b
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114.52
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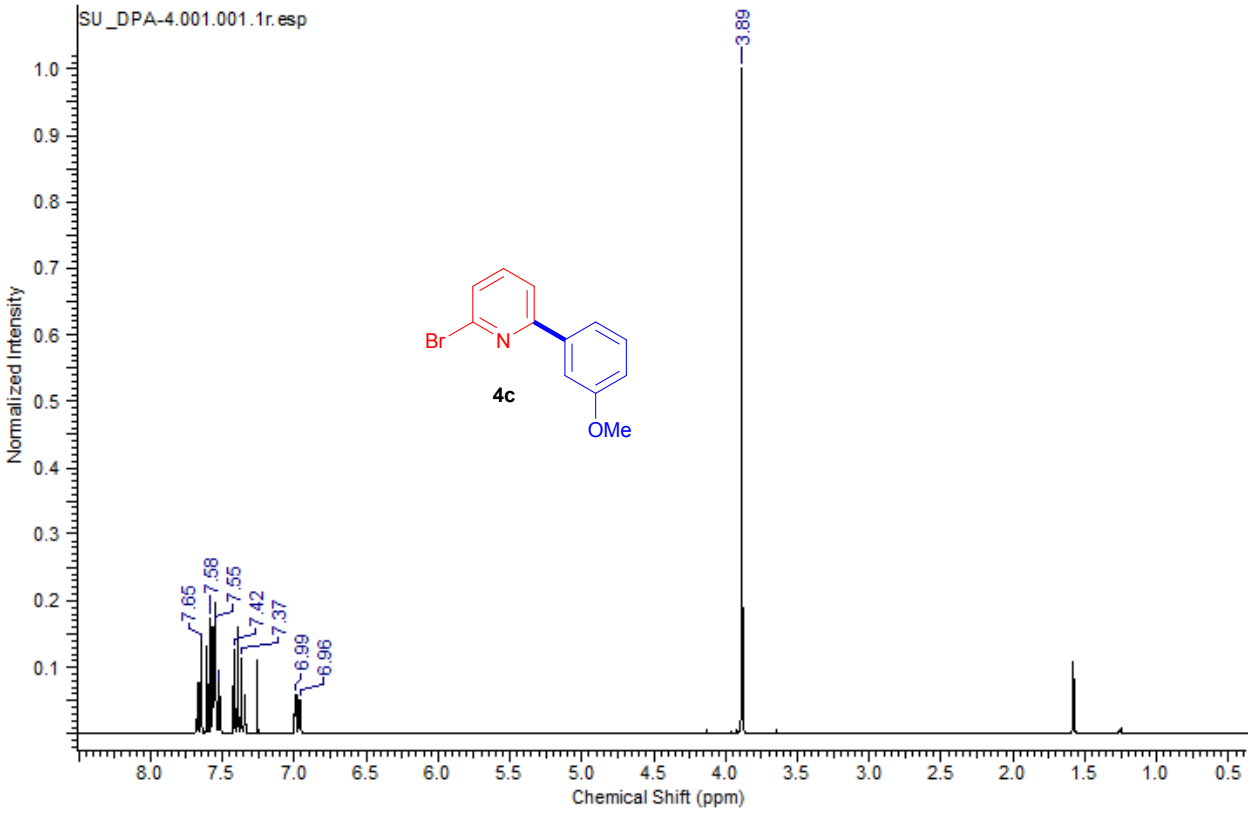
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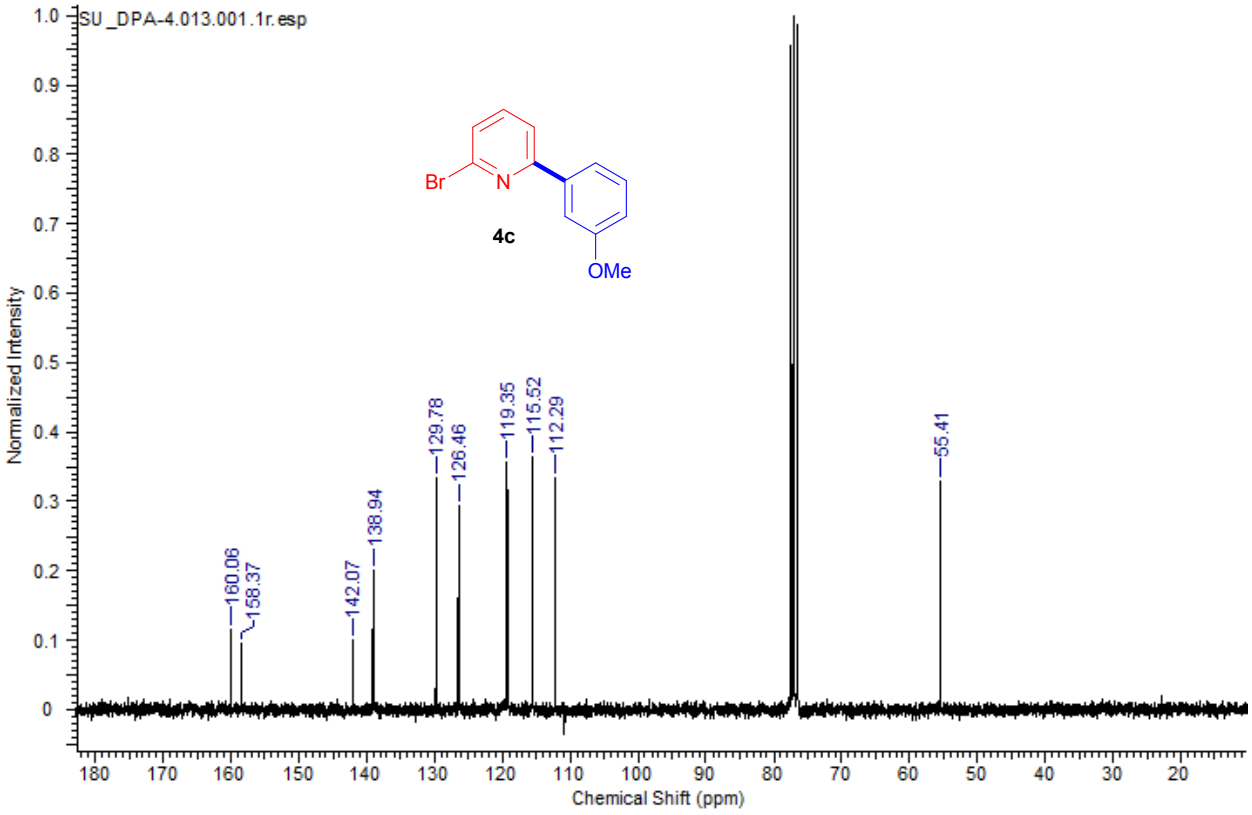
OMe
4c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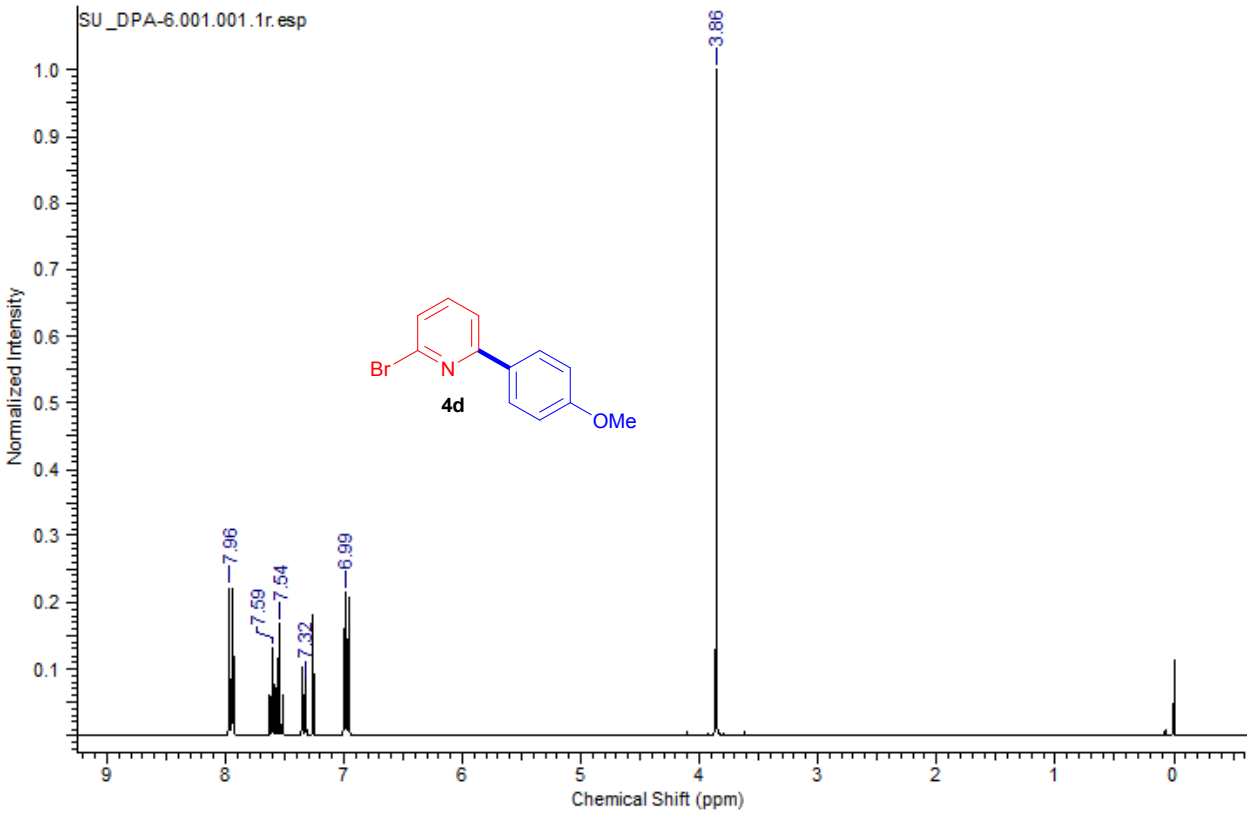
N
OMe
4c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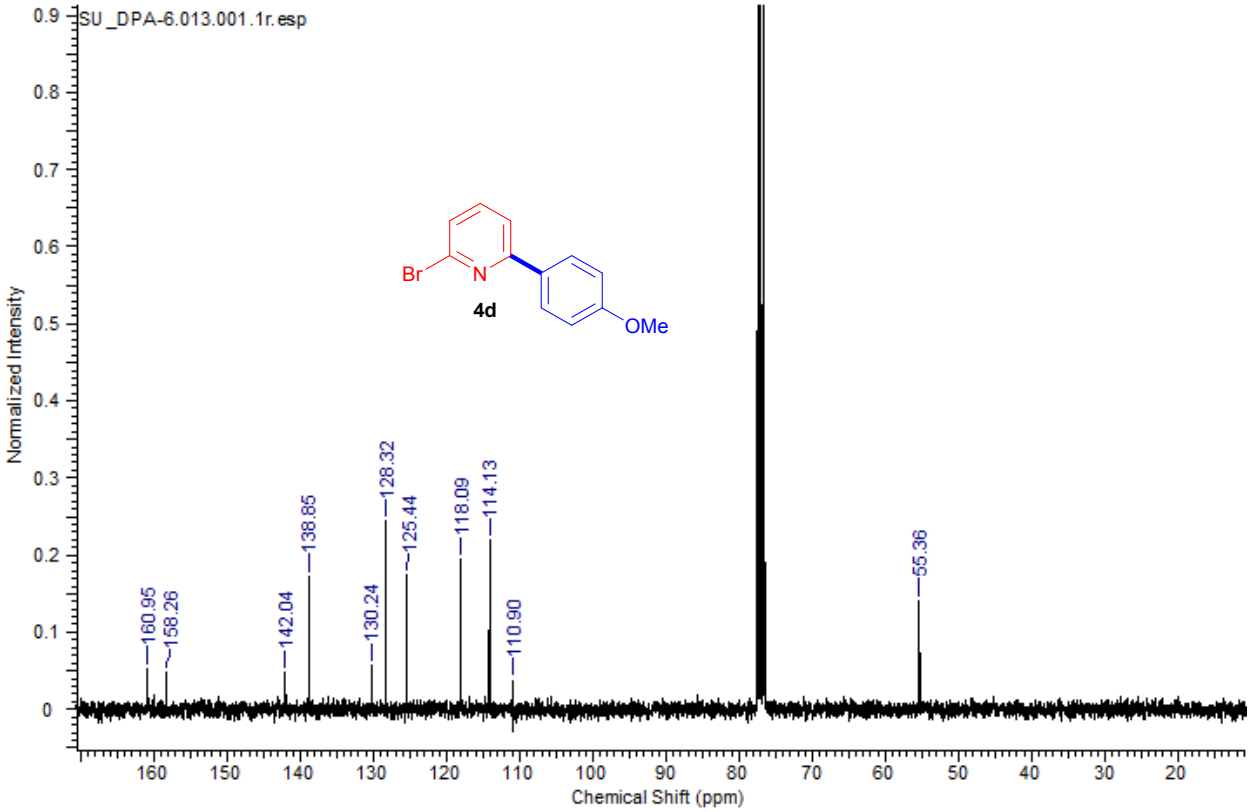
OMe
4d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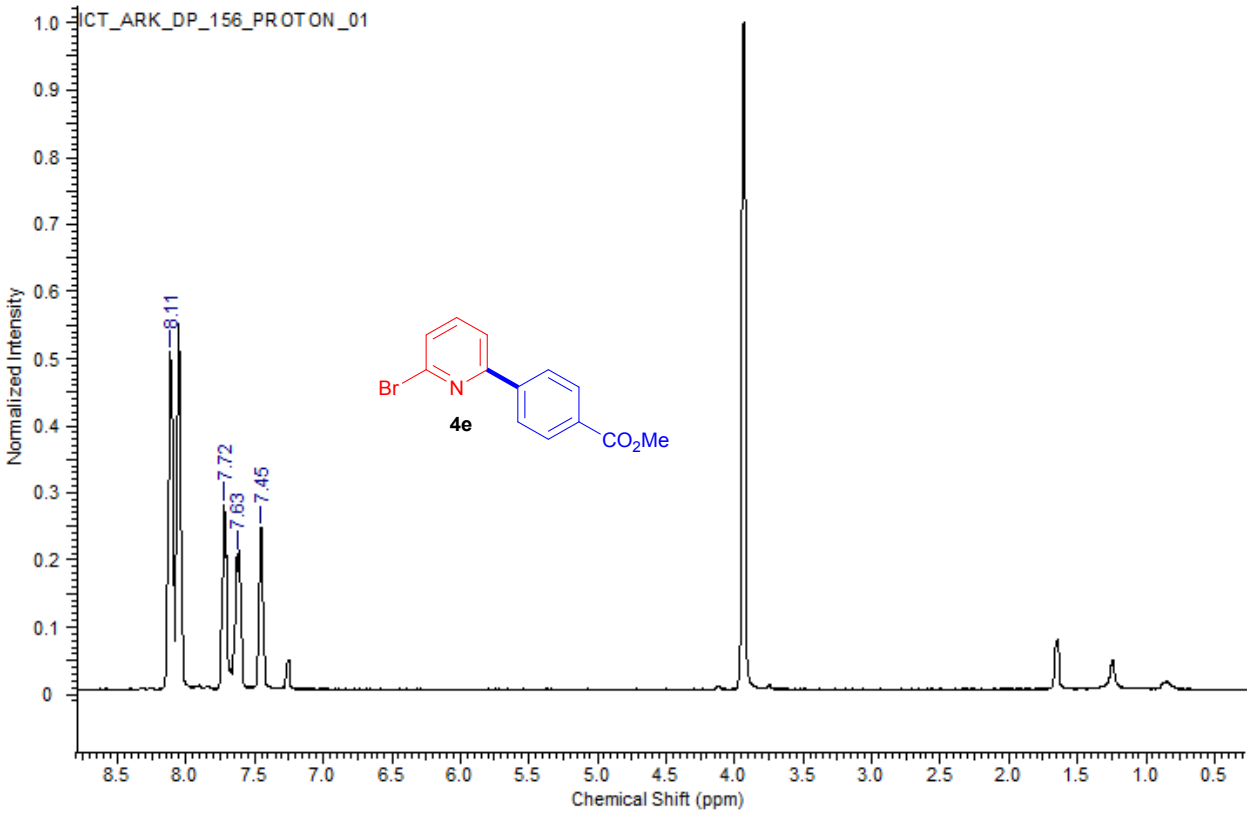
110.90
55.36
Br
N
OMe
4d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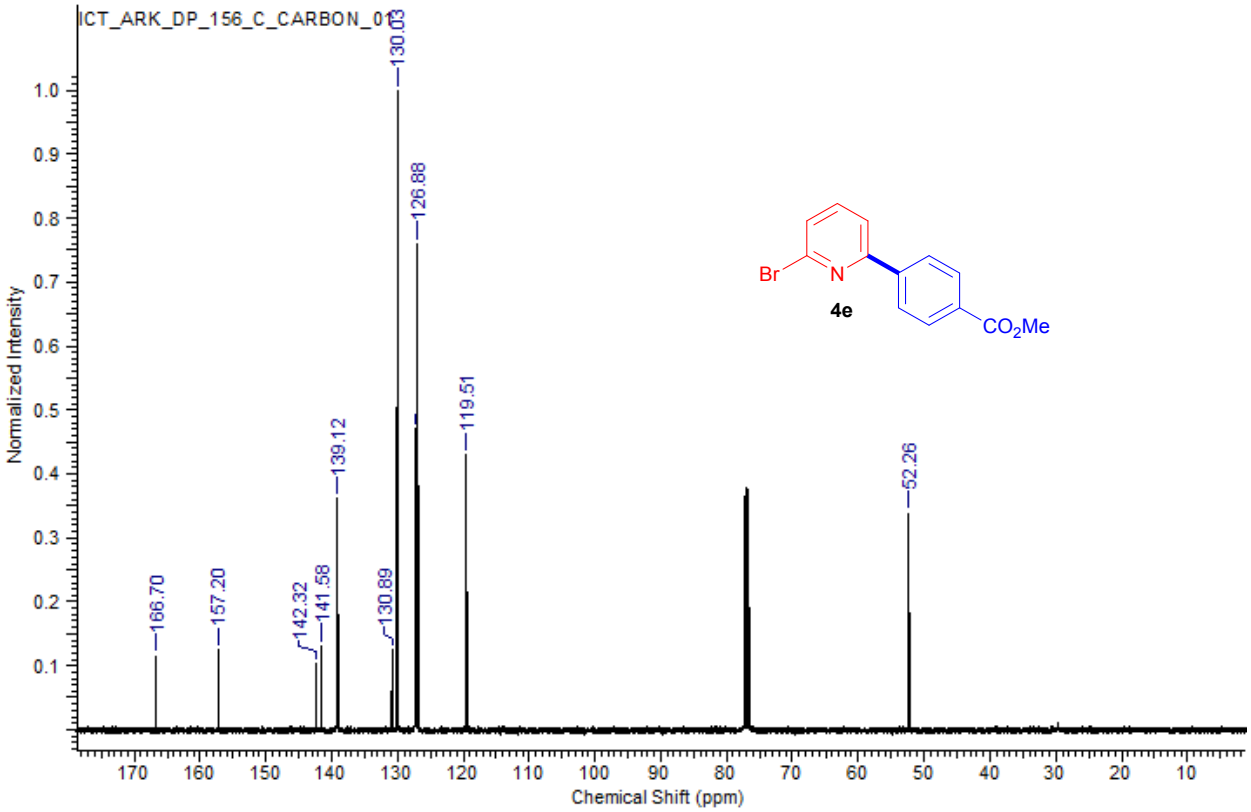
Br
N
4e
CO2Me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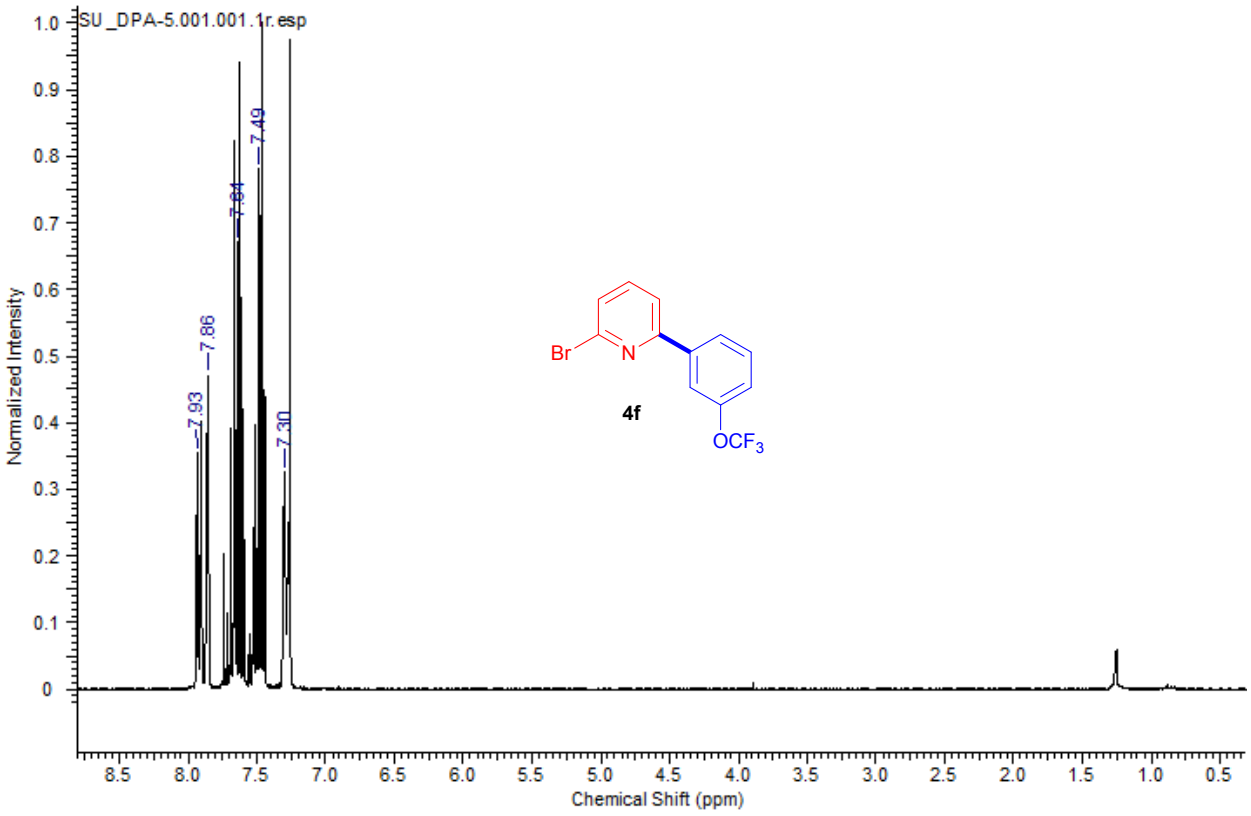
52.26
Br
N
4e
CO2Me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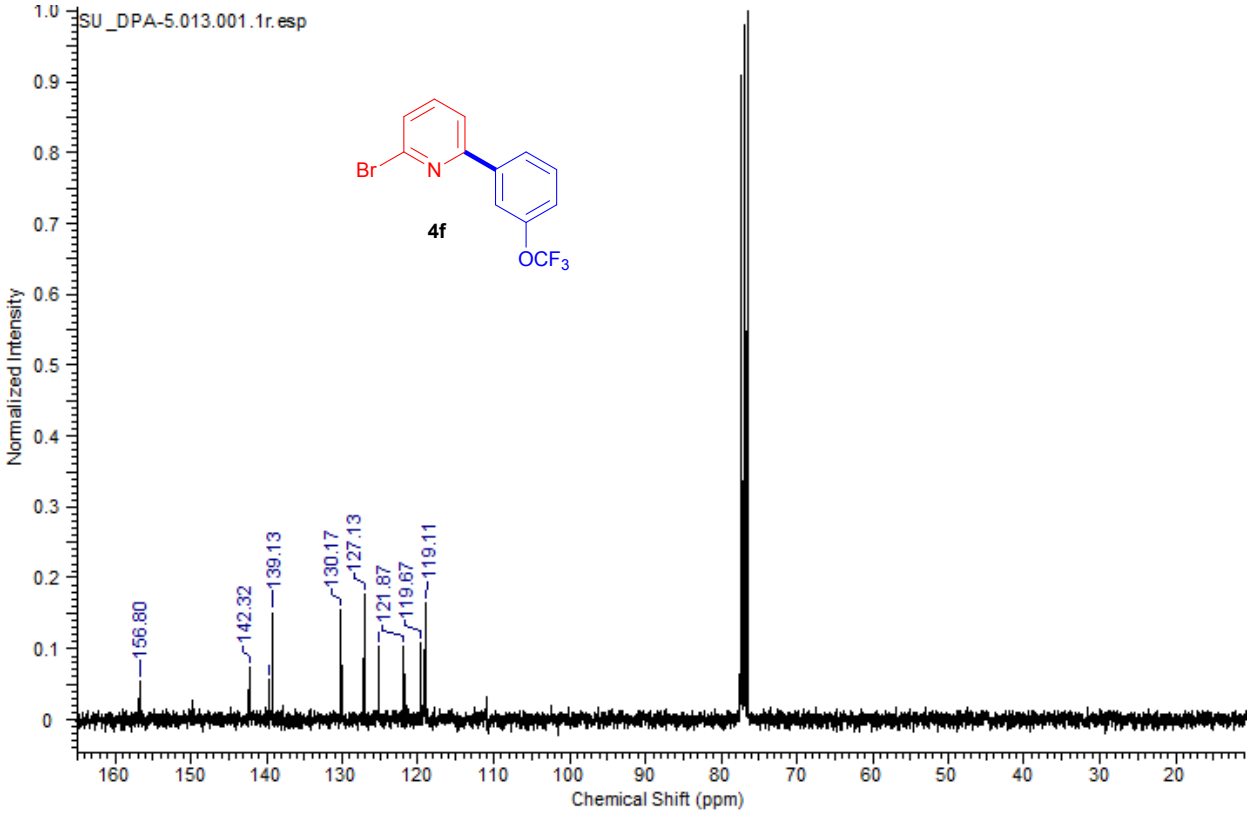
N
OCF3
4f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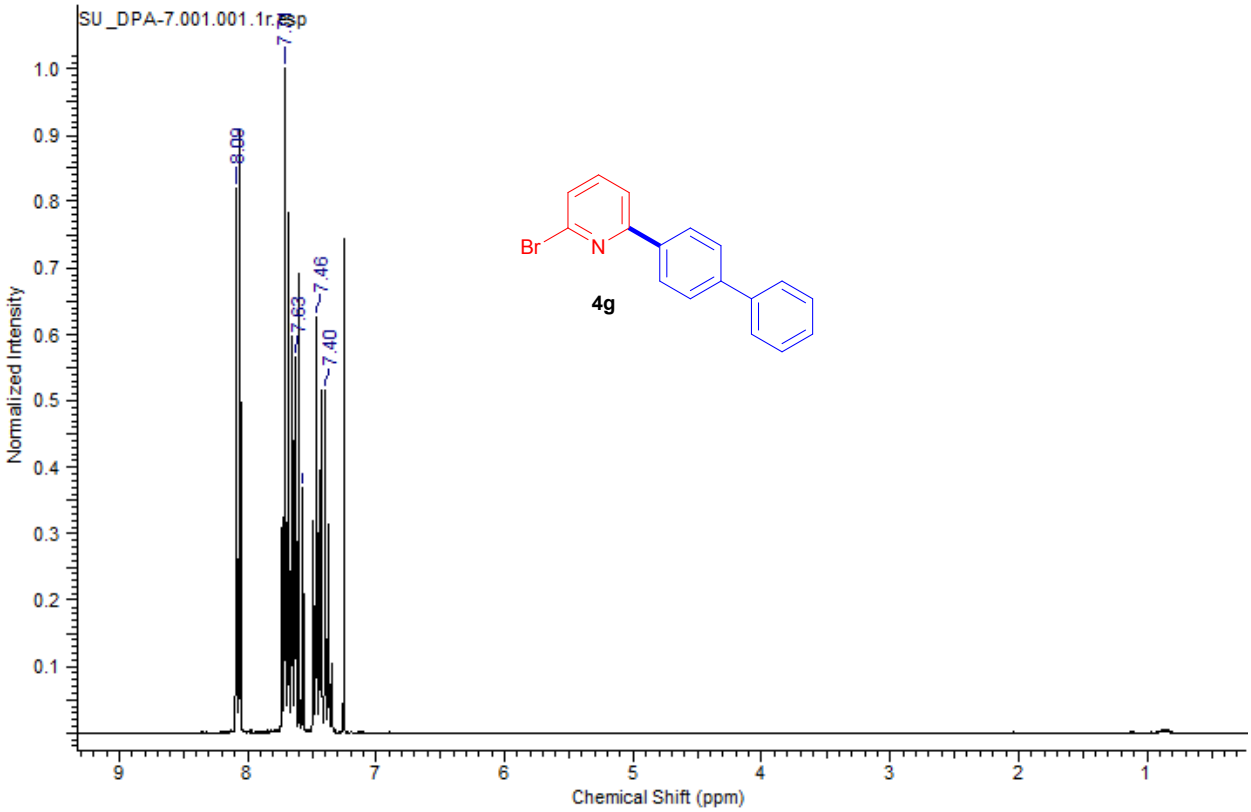
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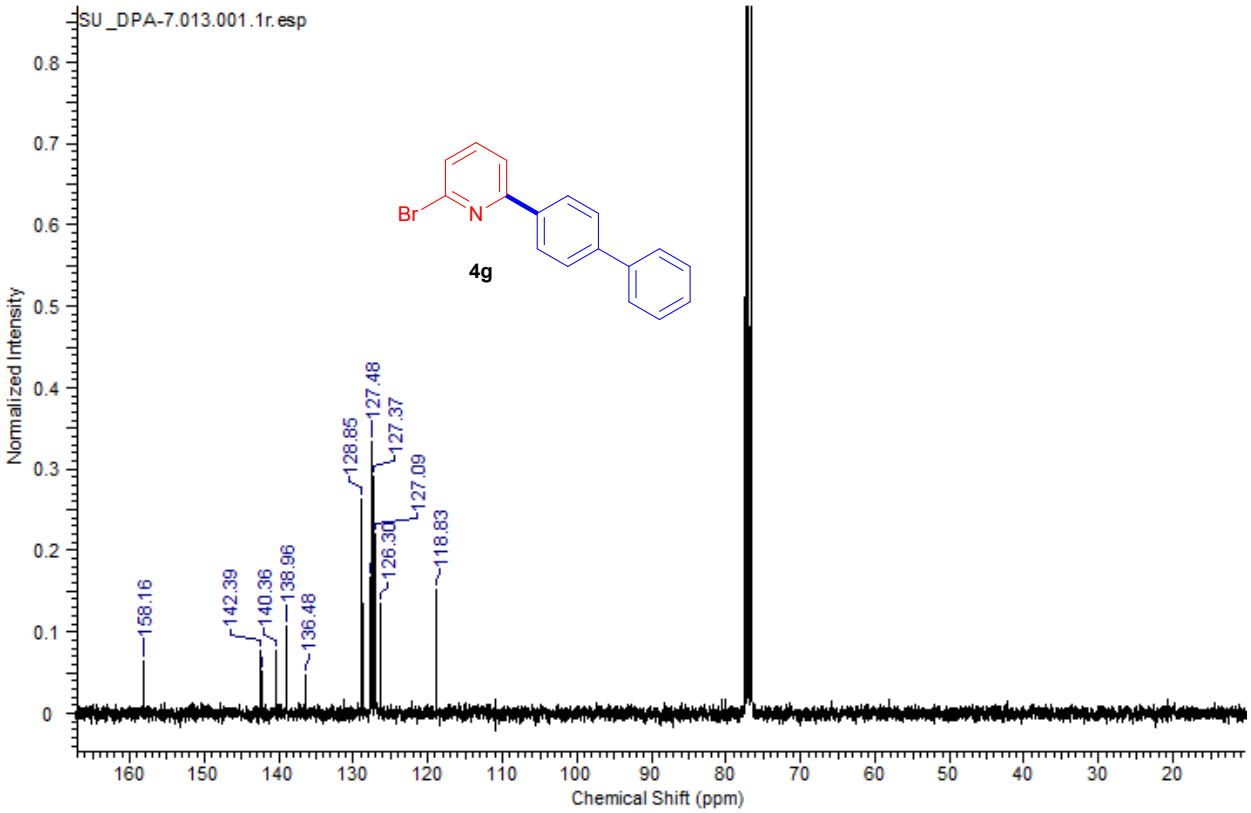
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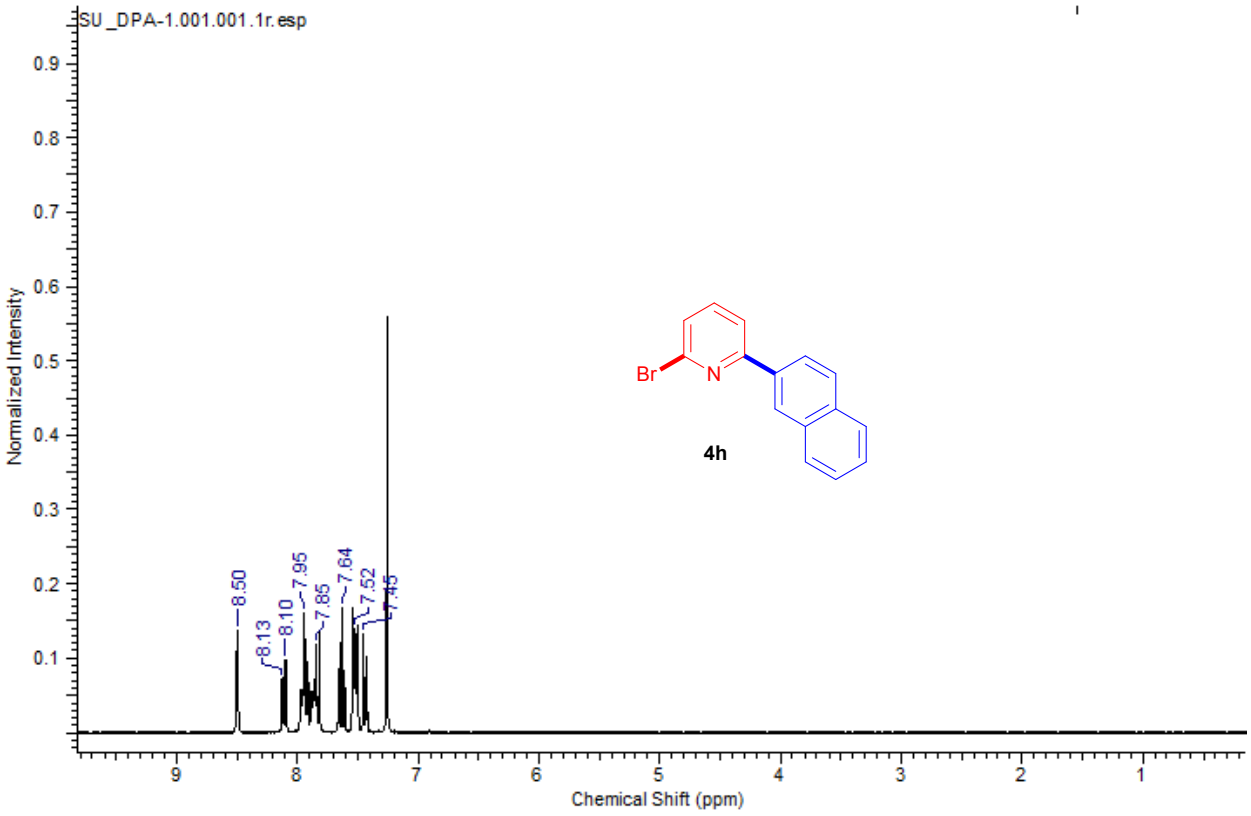
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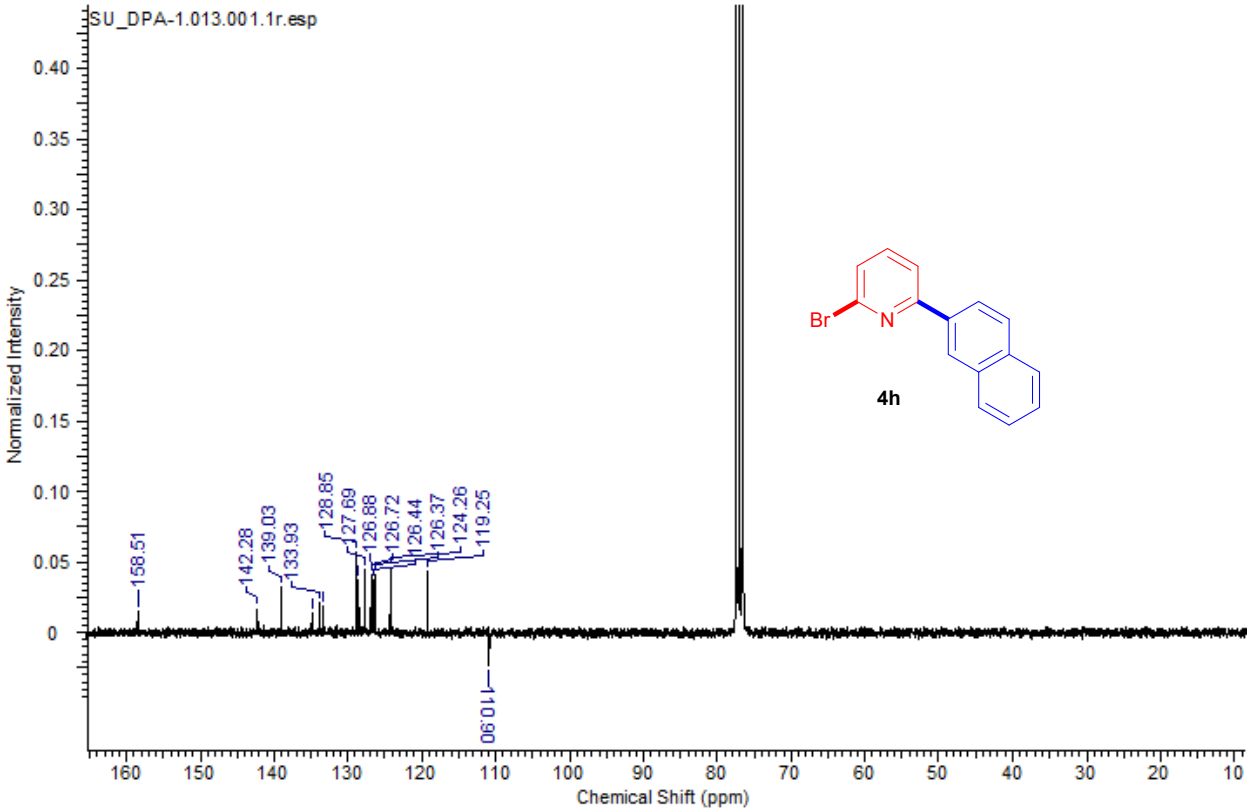
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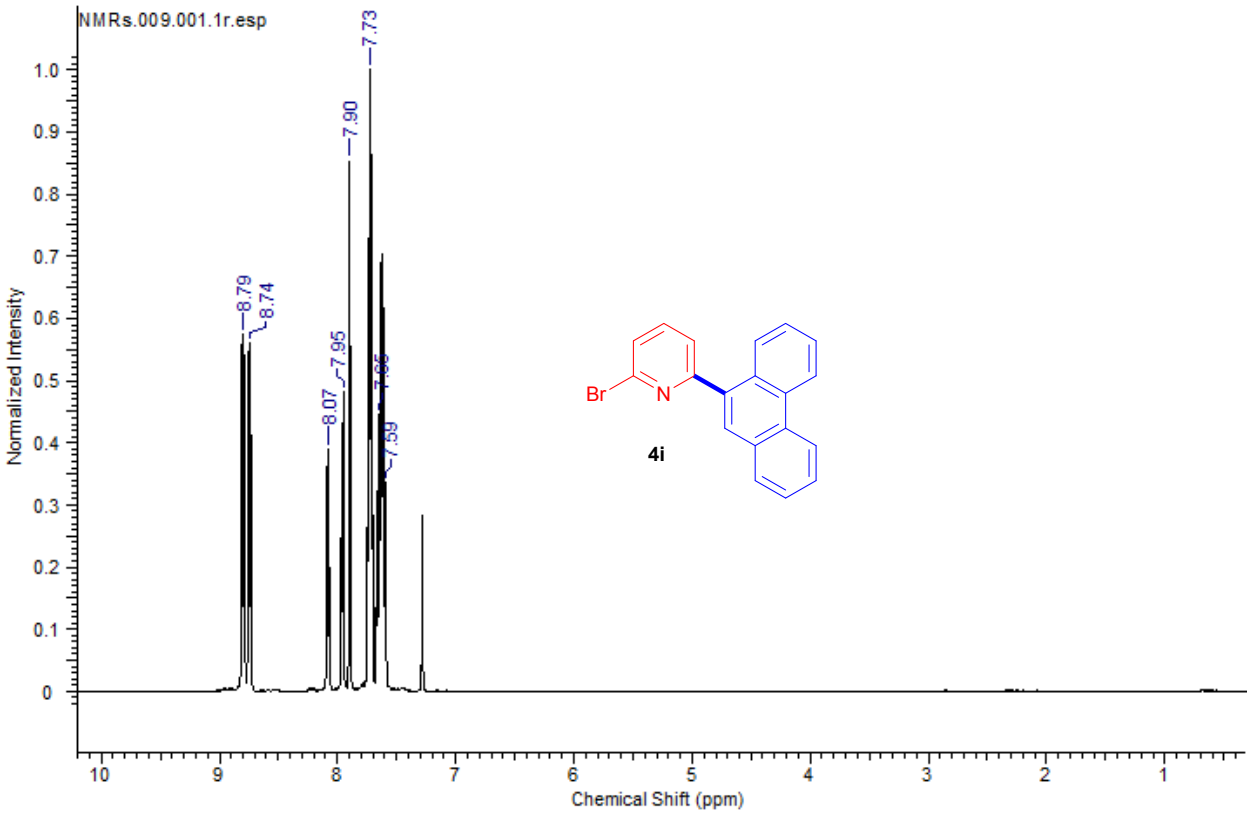
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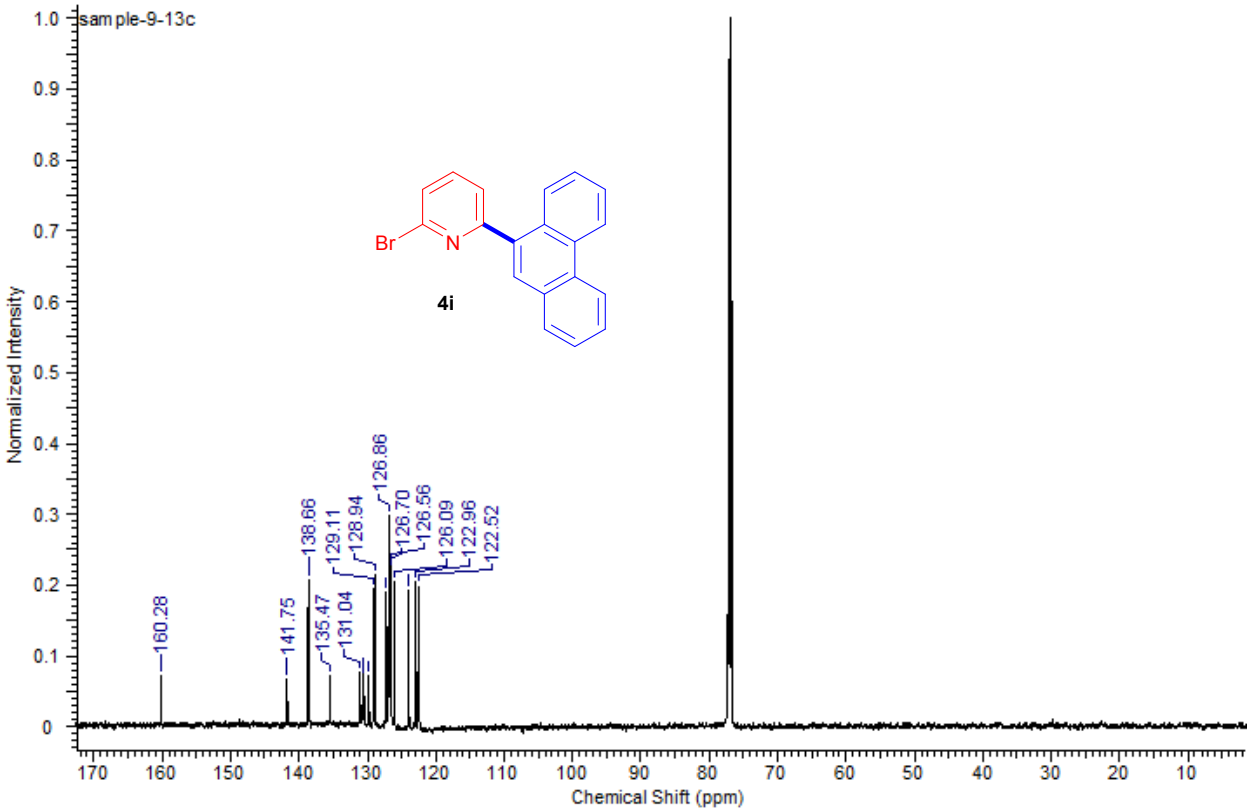
4i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Chemical Shift (ppm)
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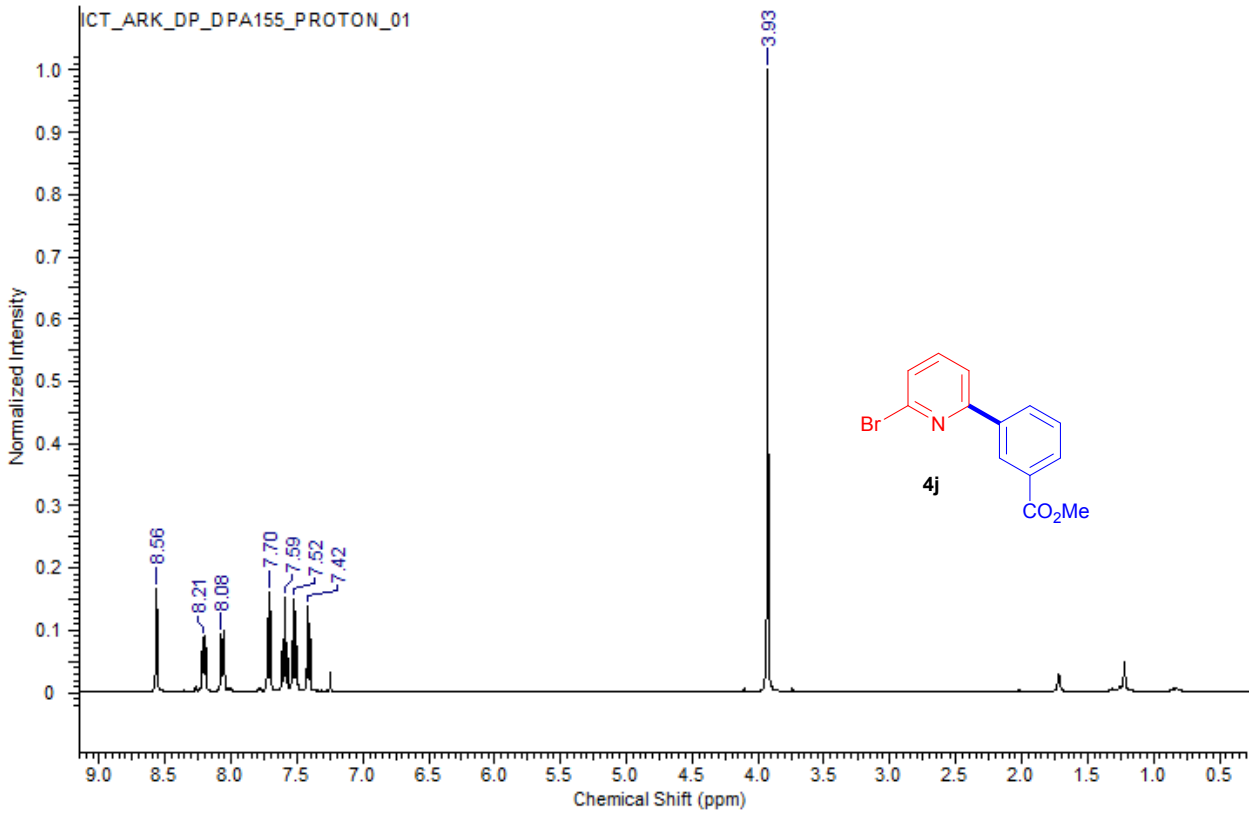
122.96
122.52
Br
N
4i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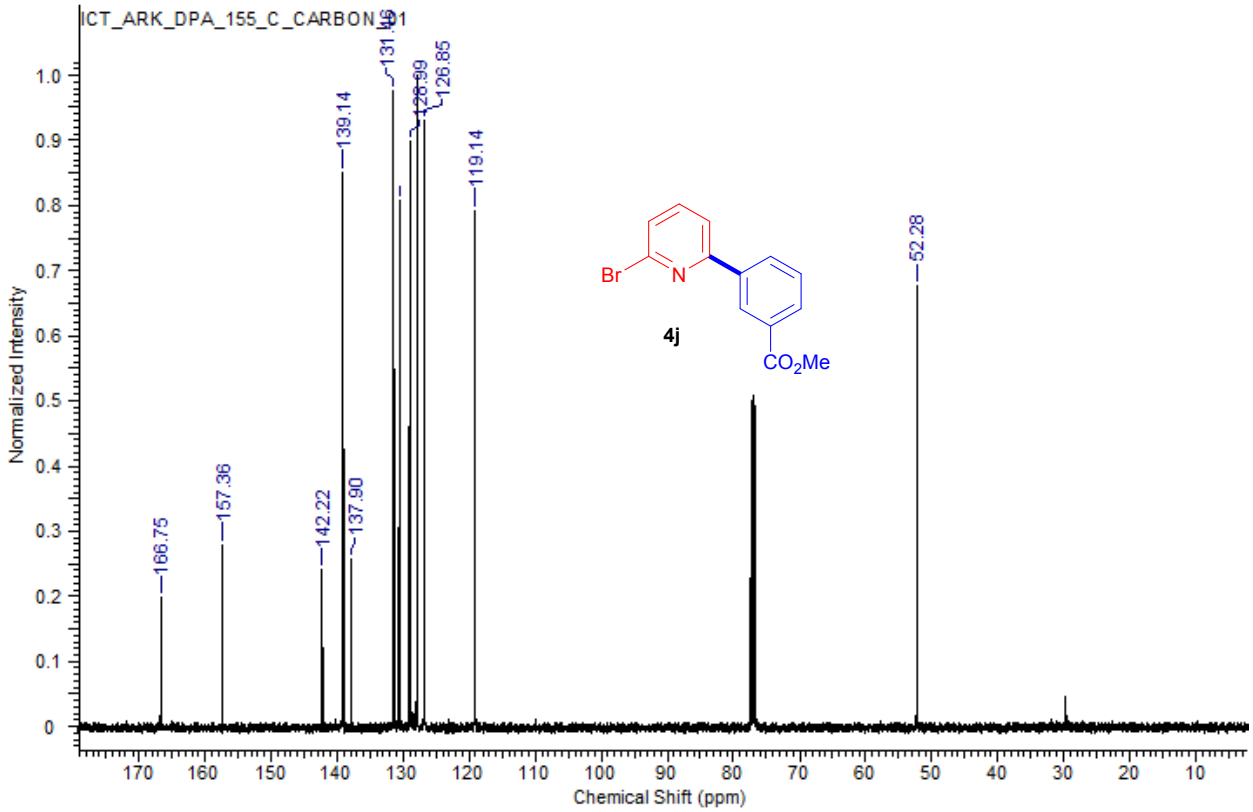
CO2Me
4j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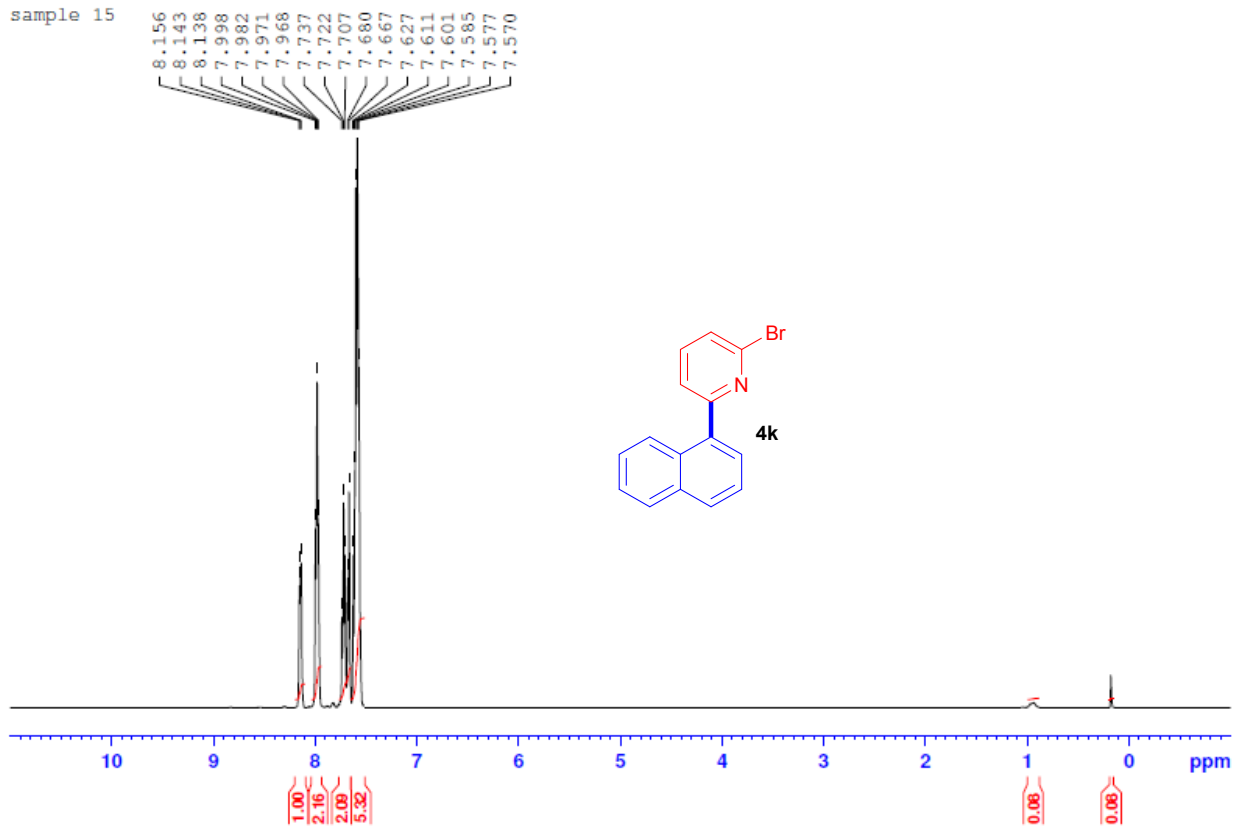
52.28
Br
N
CO2Me
4j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8.143
8.138
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7.722
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7.601
7.585
7.577
7.570
Br
N
4k
10
9
8
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3
2
1
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ppm
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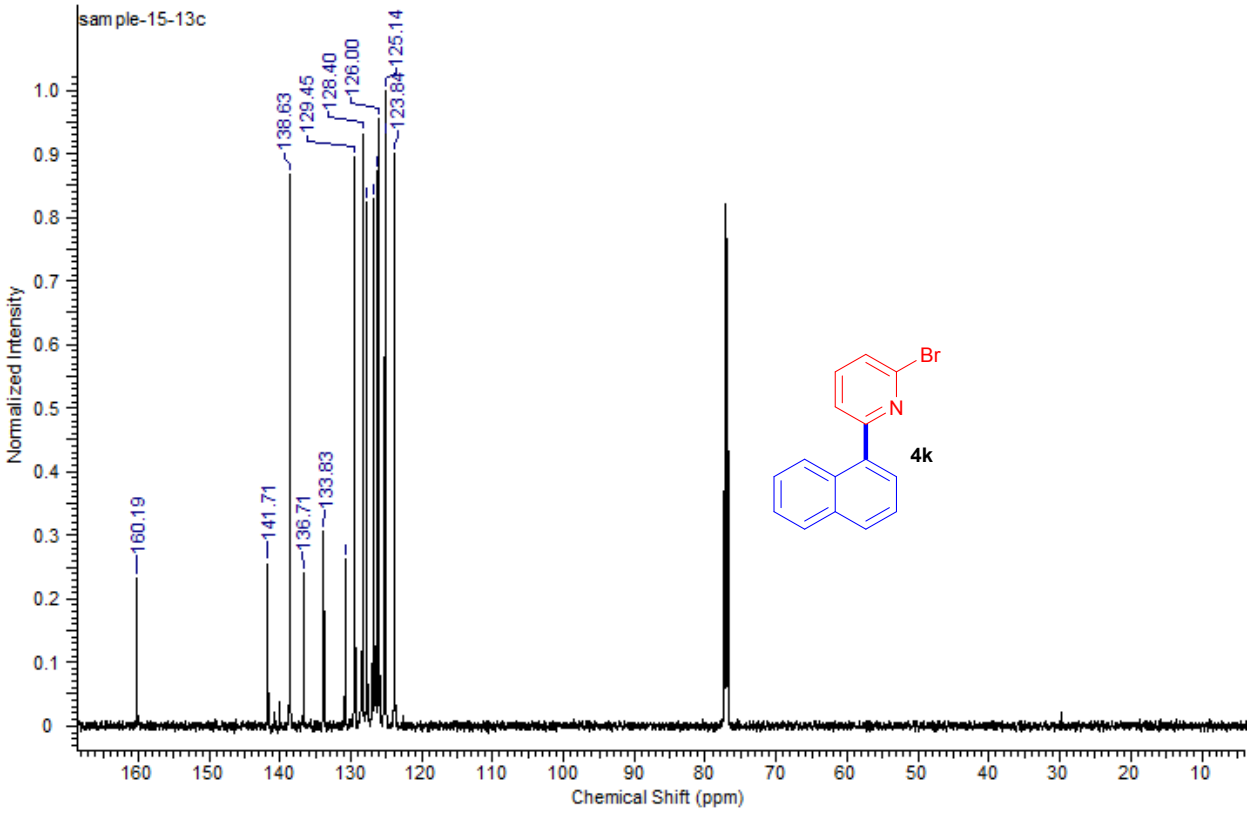
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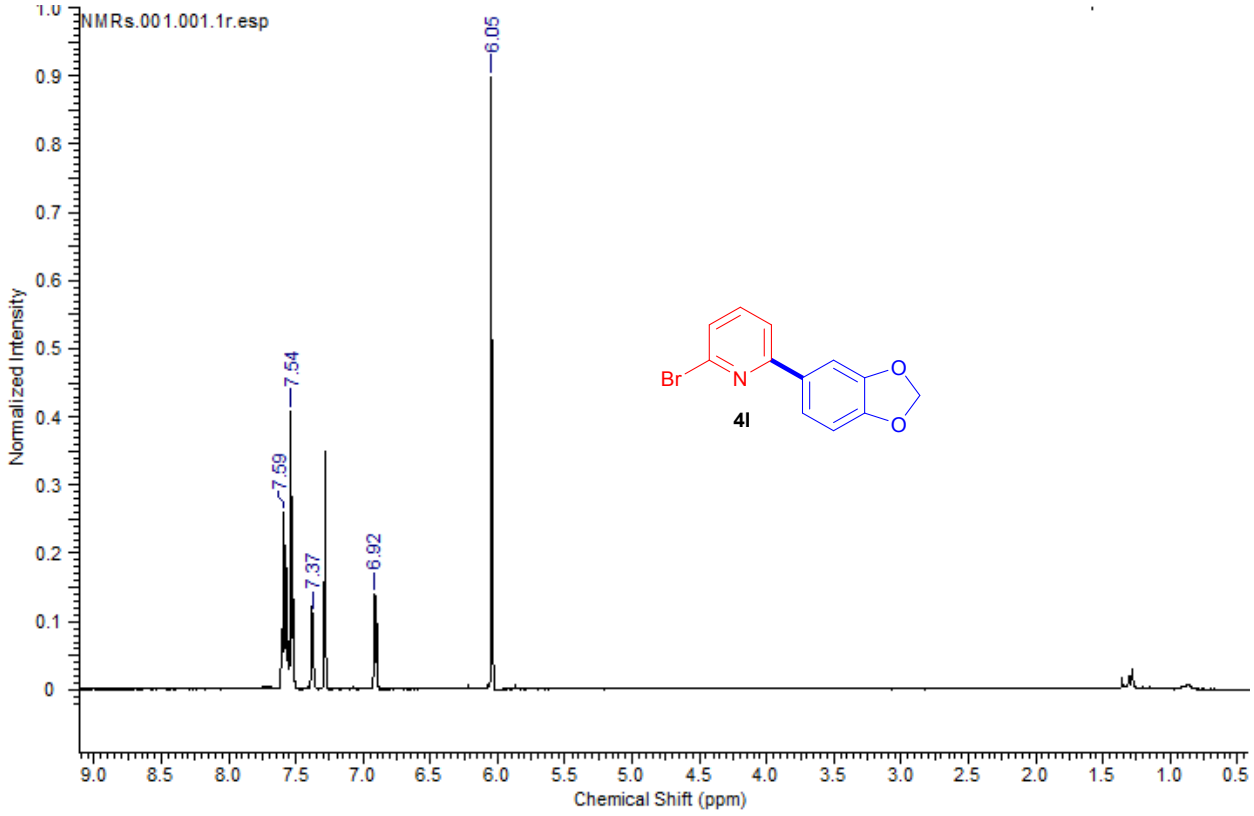
125.14
Br
N
4k
Chemical Shift (ppm)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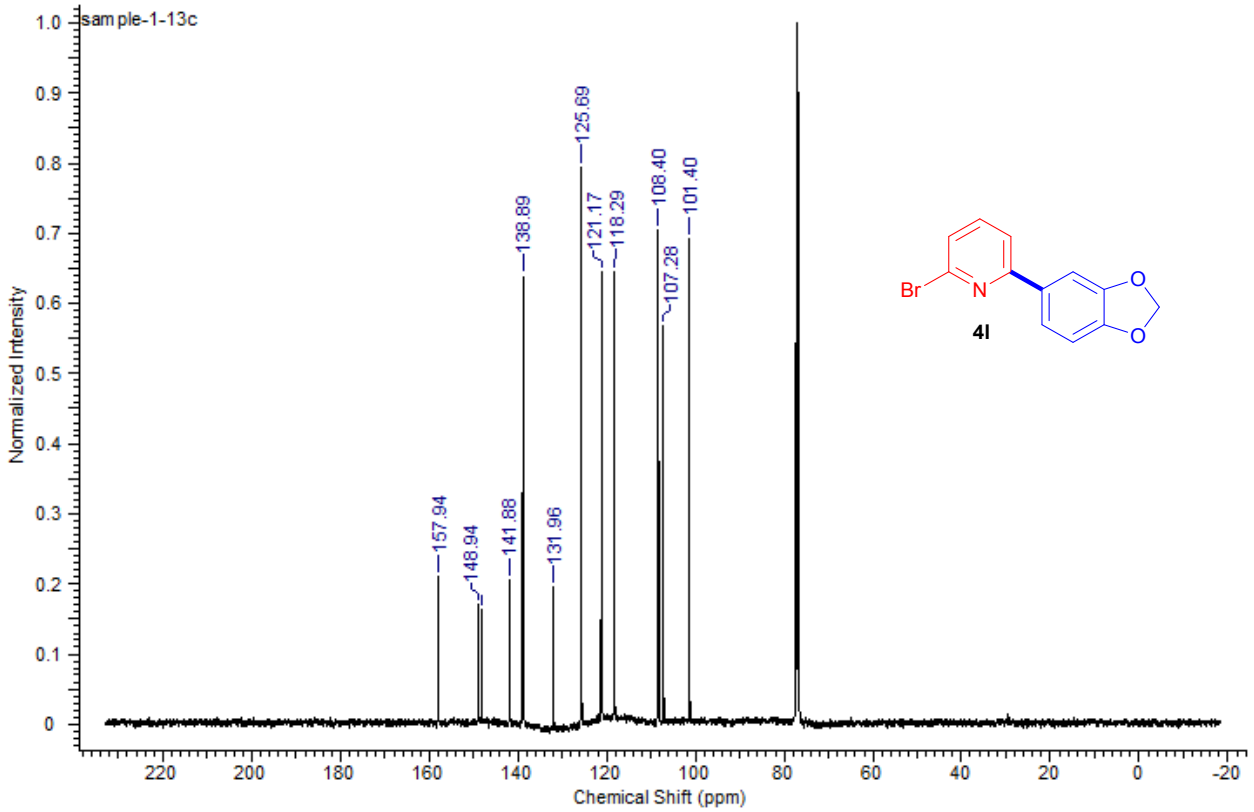
O
O
4l


sample-1-13c
Normalized Intensity
Chemical Shift (ppm)
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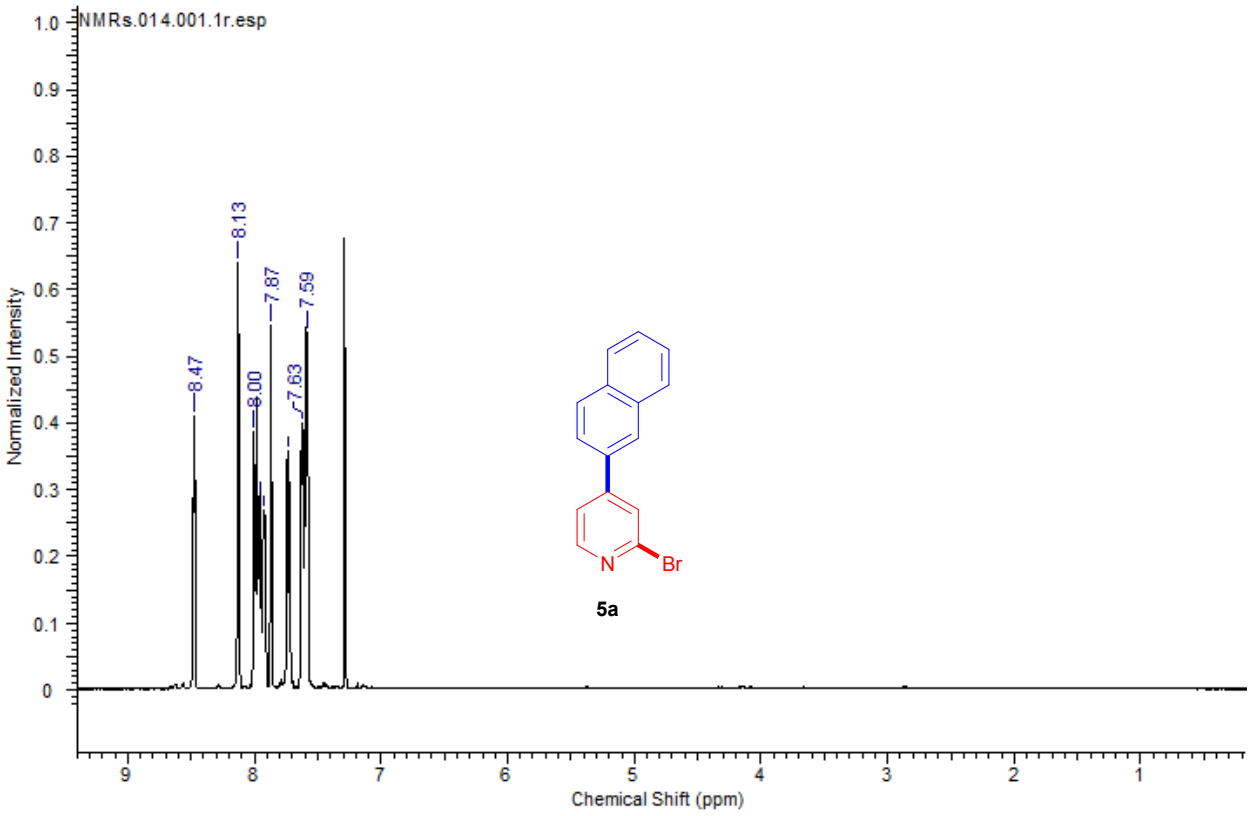
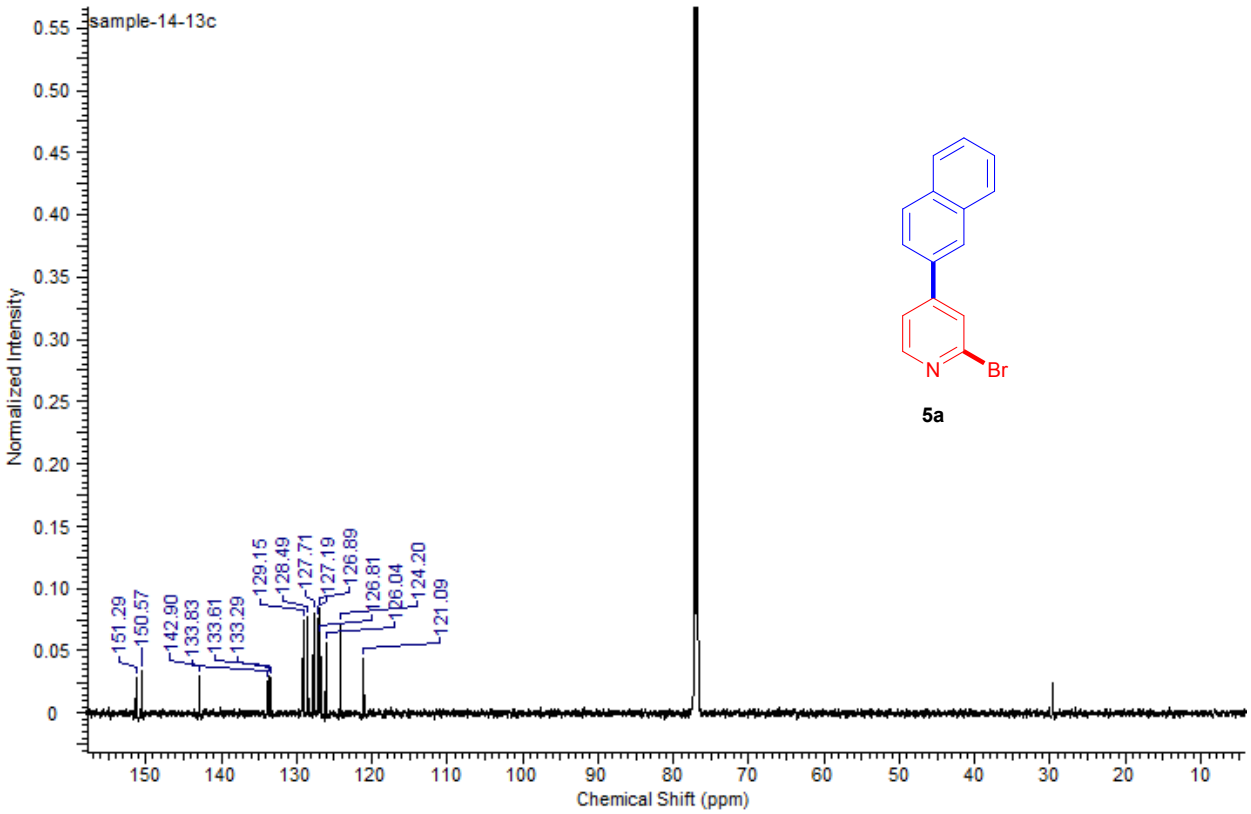
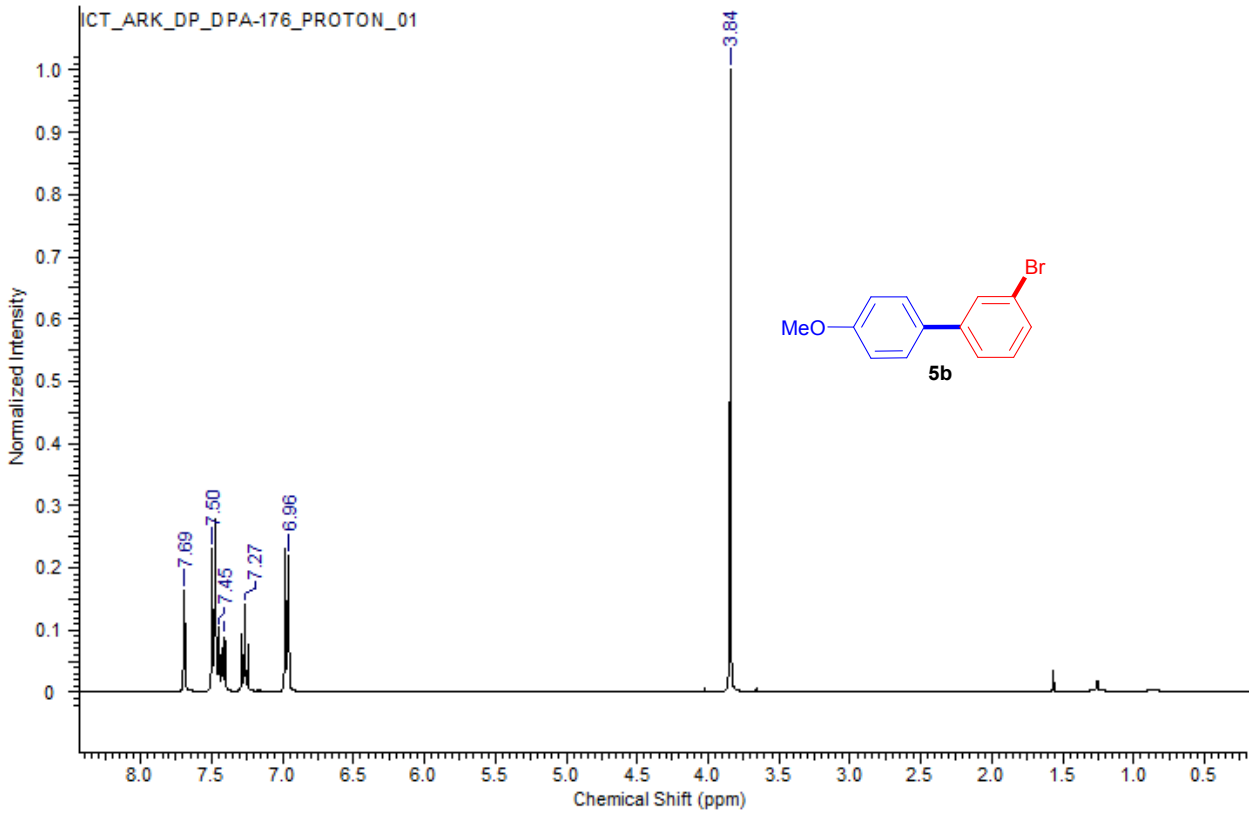
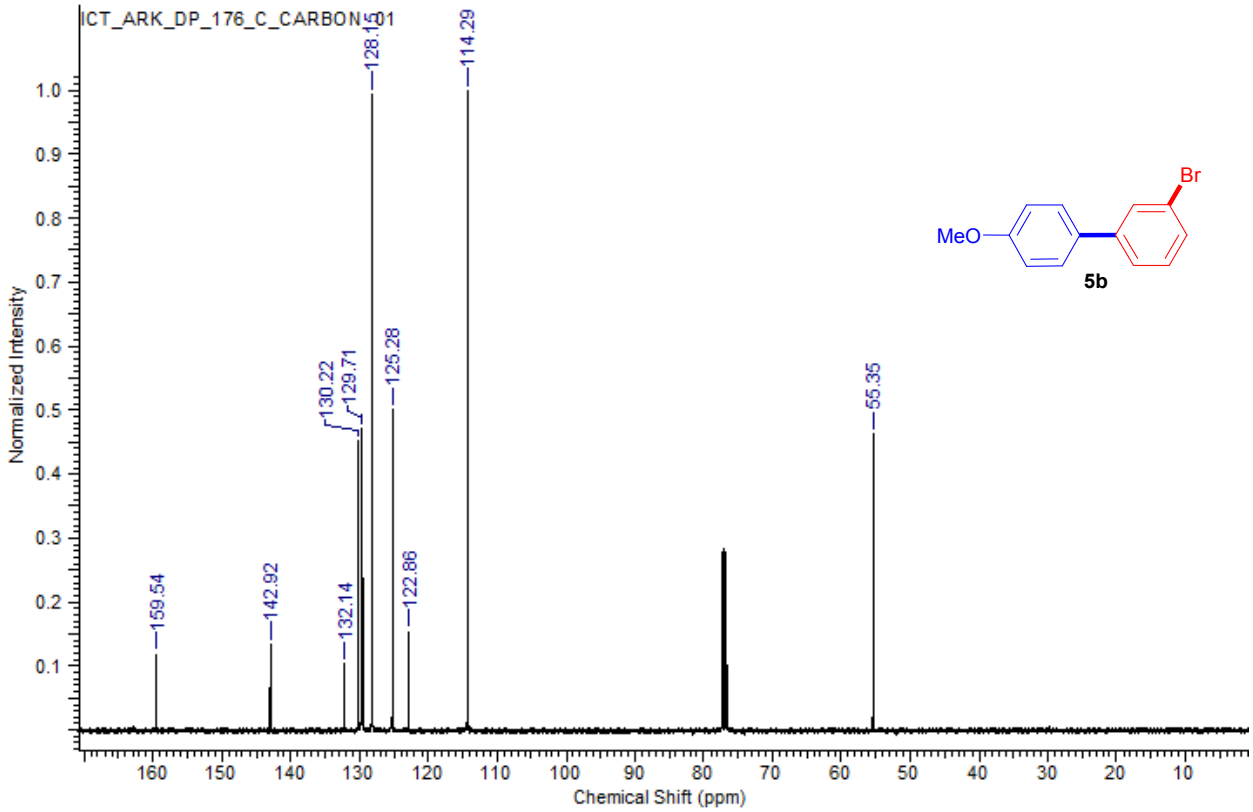
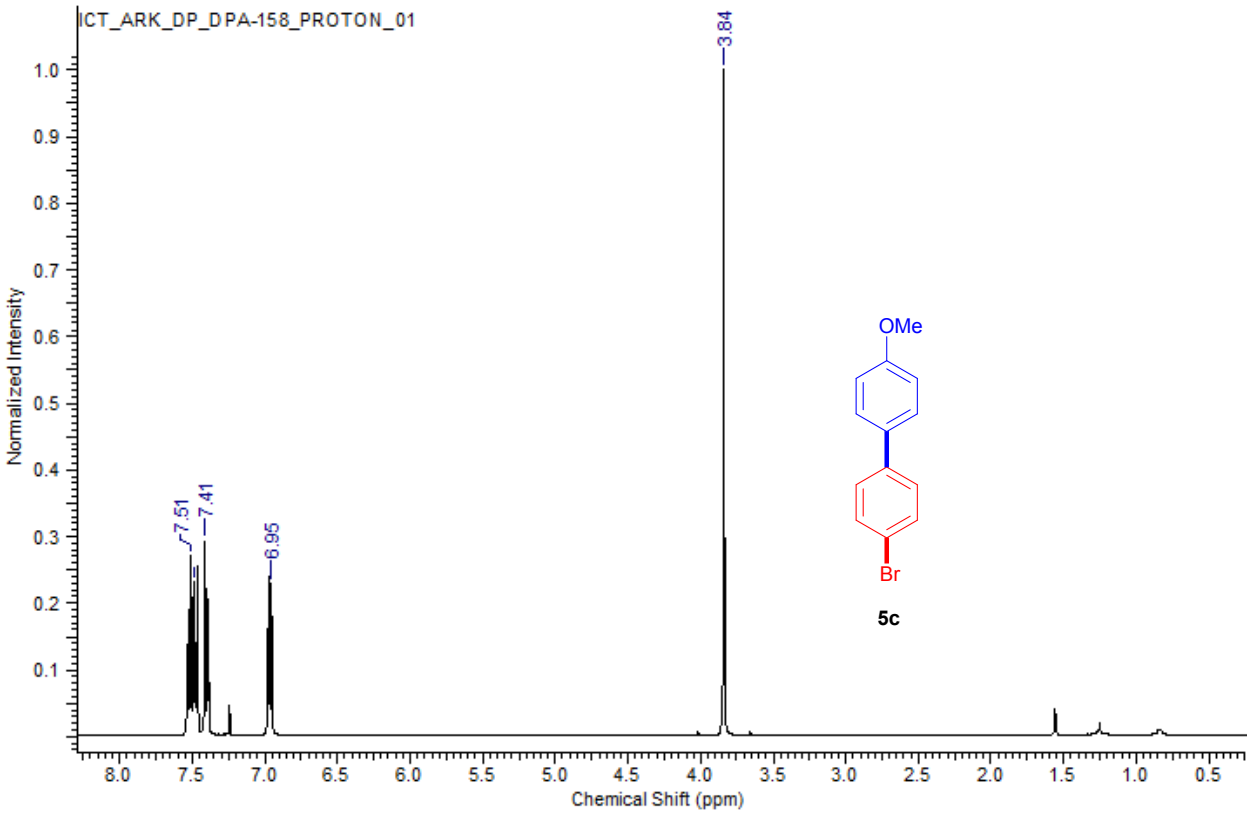
148.94
141.88
138.89
131.96
125.69
121.17
118.29
108.40
107.28
101.40
Br
N
O
O
4l

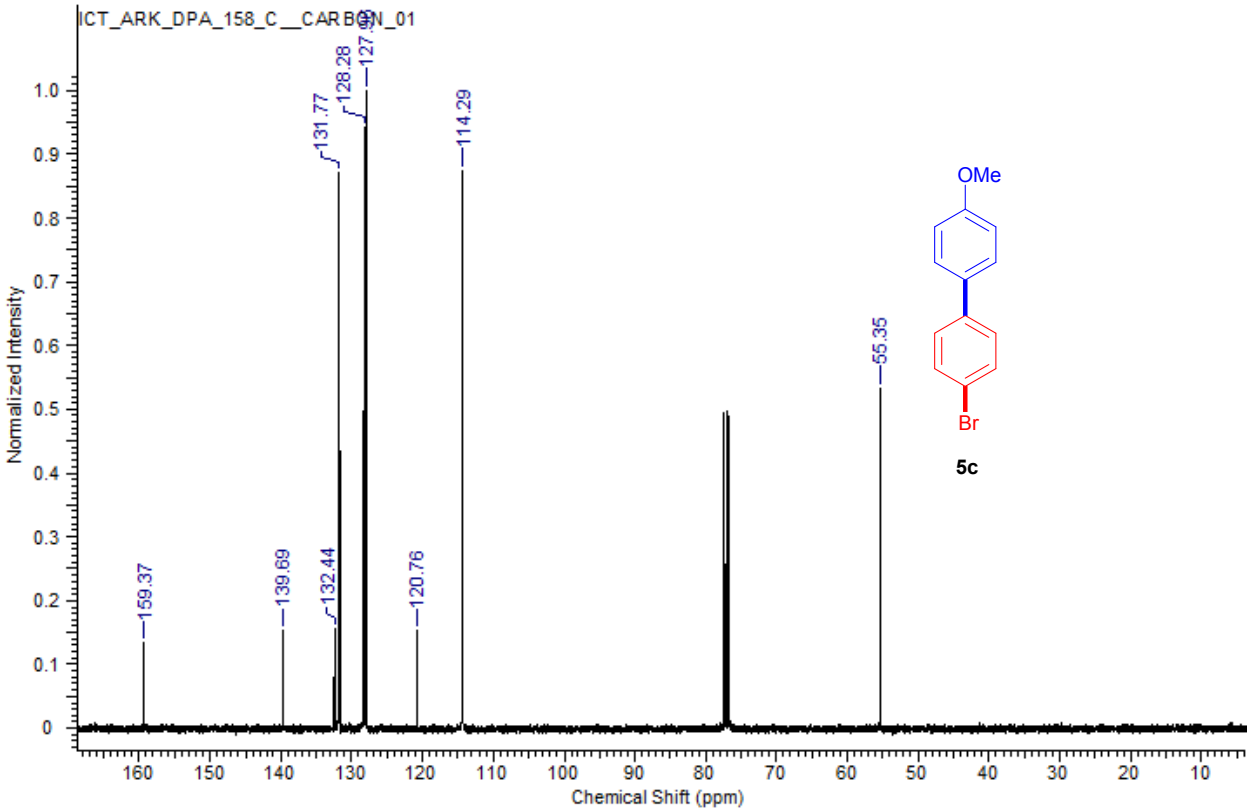

NMRs.014.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.47
8.13
8.00
7.87
7.63
7.59
N
Br
5a

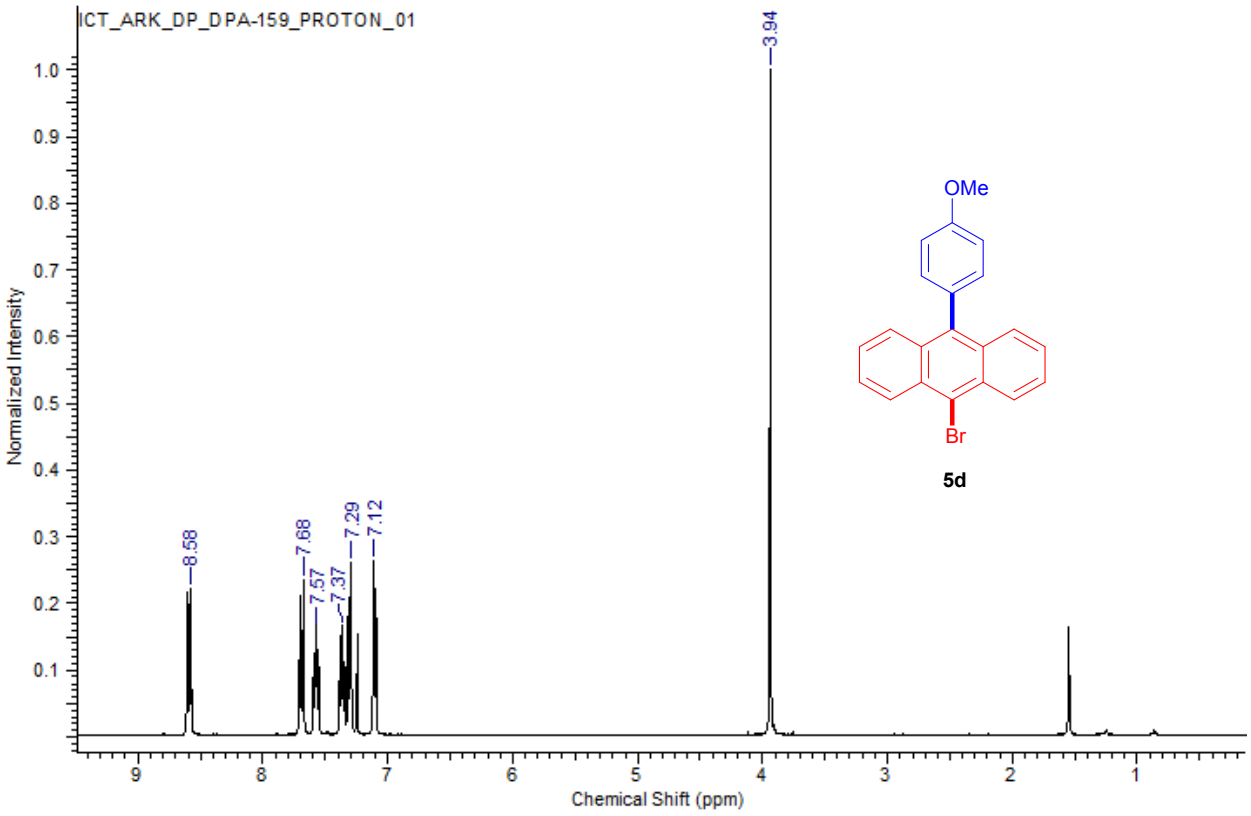

sample-14-13c
Normalized Intensity
Chemical Shift (ppm)
151.29
150.57
142.90
133.83
133.61
133.29
129.15
128.49
127.71
127.19
126.89
126.81
126.04
124.20
121.09
N
Br
5a

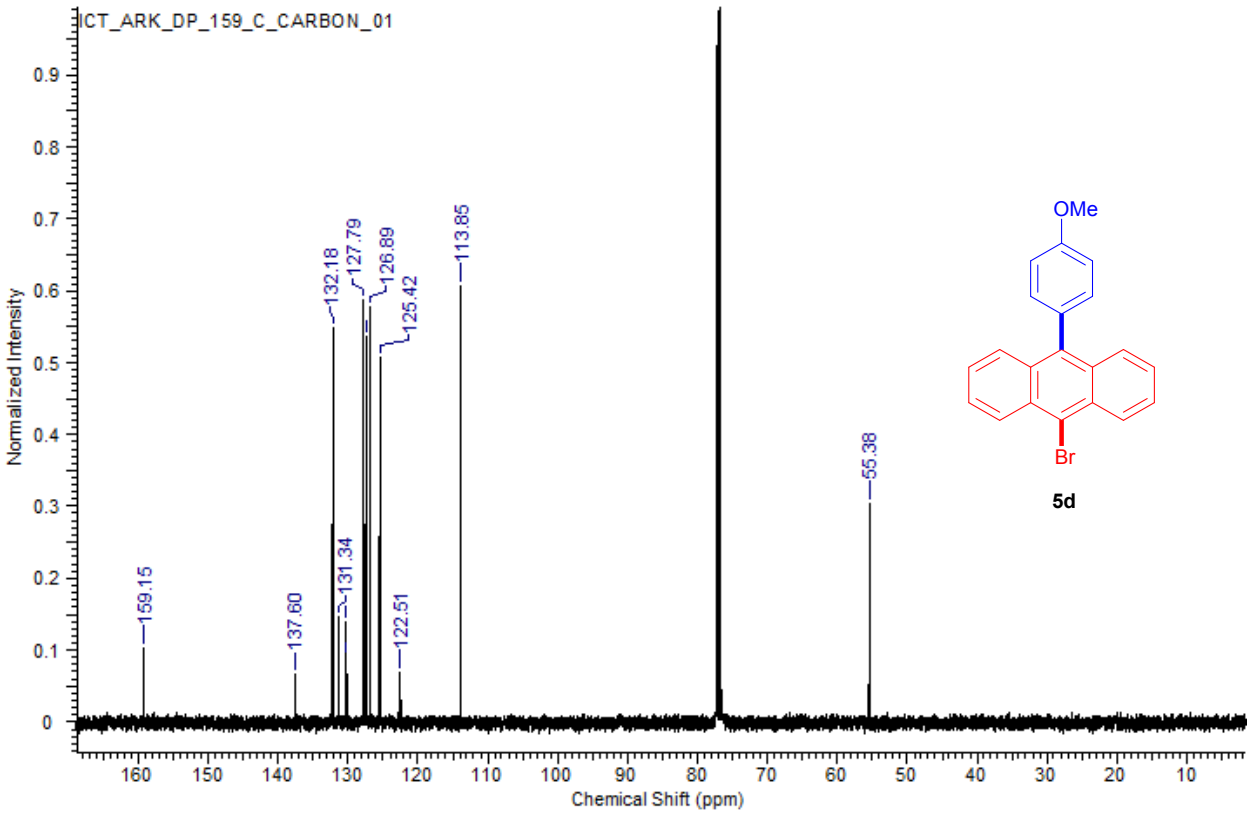

ICT_ARK_DP_DPA-176_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
3.84
7.69
7.50
7.45
7.27
6.96
MeO
Br
5b

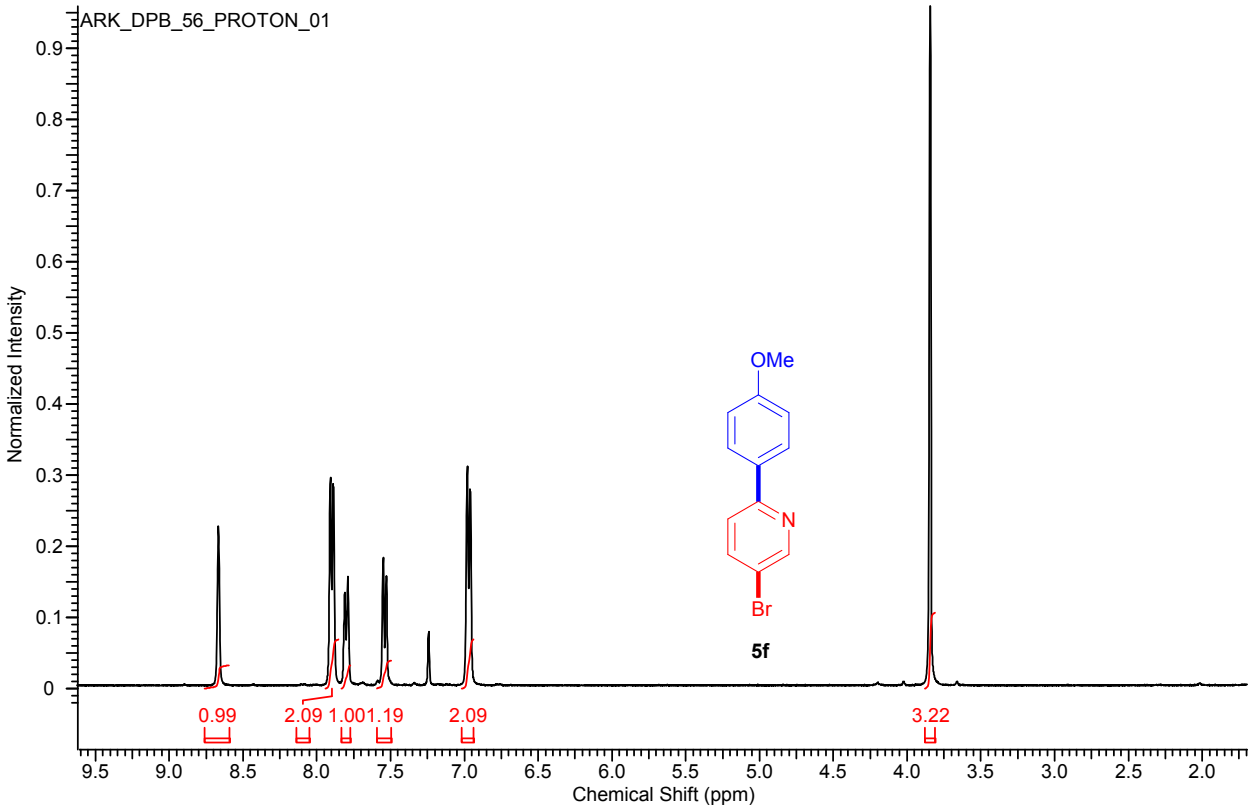

ICT_ARK_DP_176_C_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
159.54
142.92
132.14
130.22
129.71
128.15
125.28
122.86
114.29
55.35
MeO
Br
5b

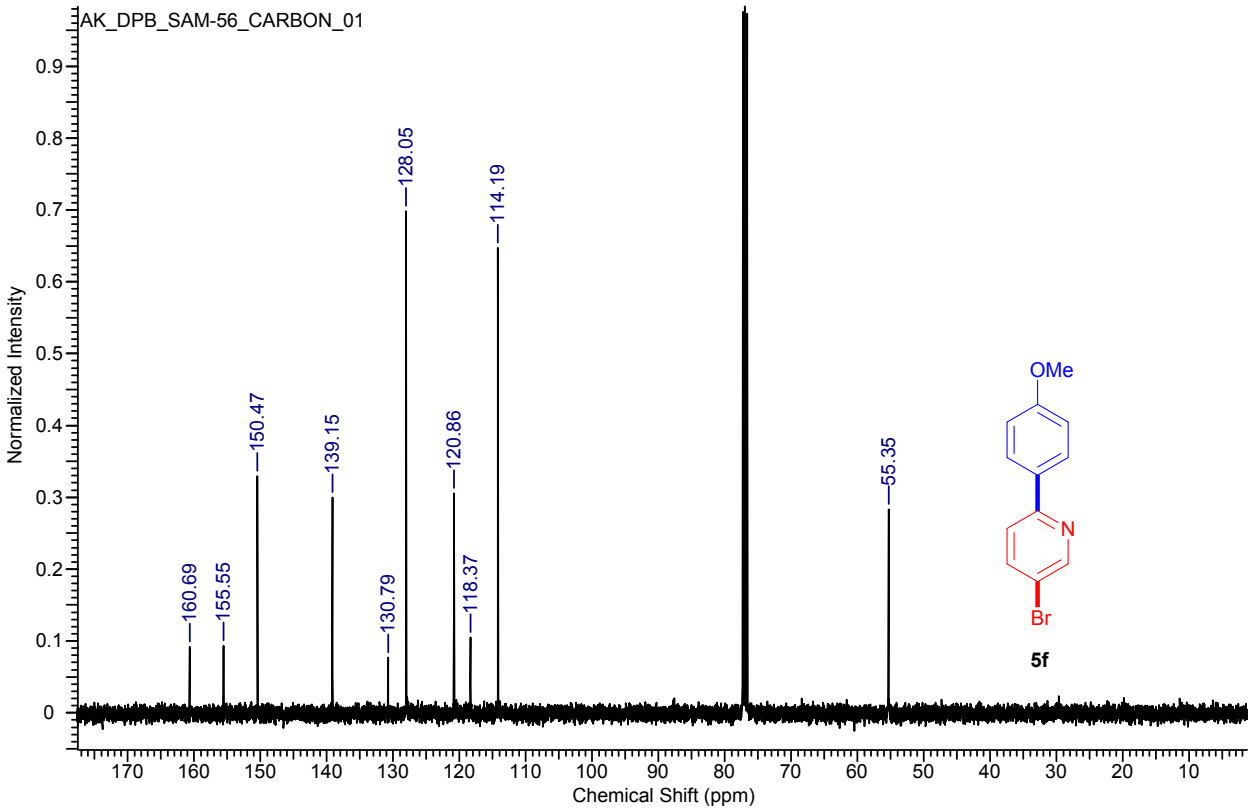

ICT_ARK_DP_DPA-158_PROTON_01
3.84
7.51
7.41
6.95
OMe
Br
5c
Normalized Intensity
Chemical Shift (ppm)

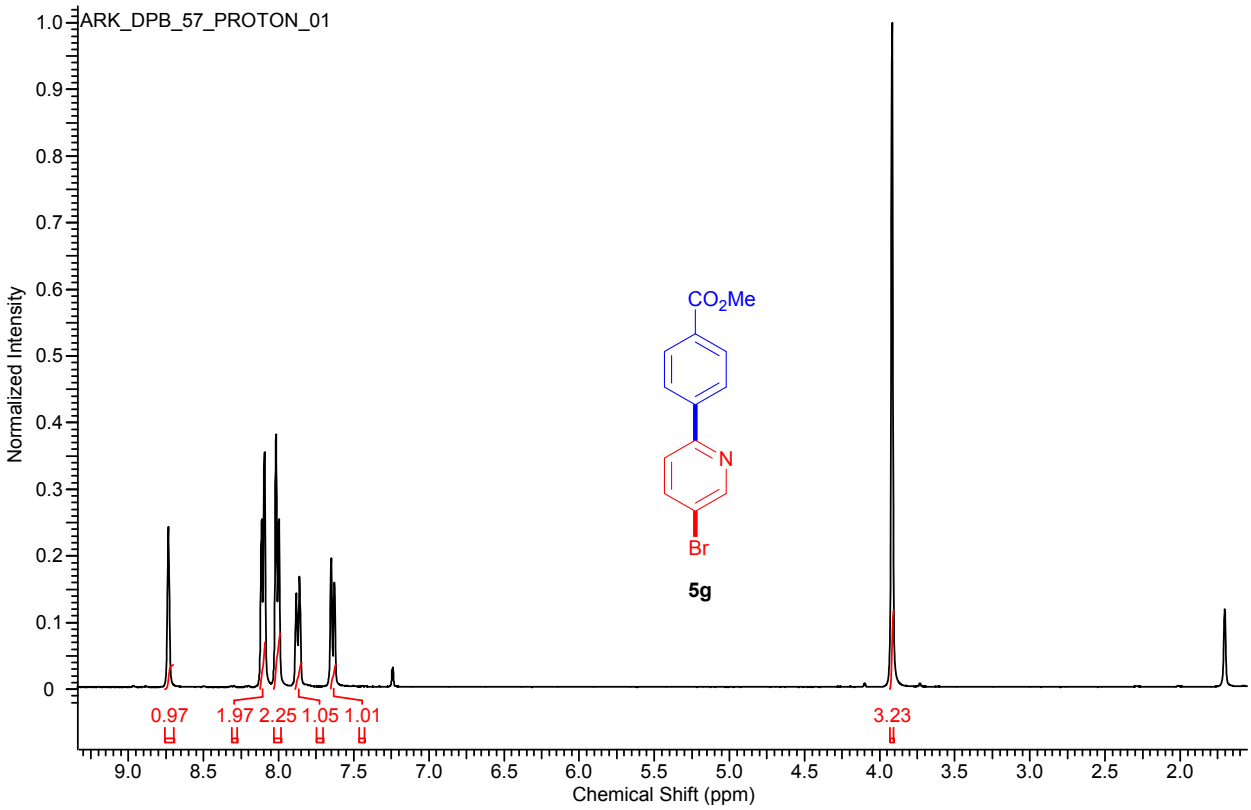

ICT_ARK_DPA_158_C__CARBON_01
159.37
139.69
132.44
131.77
128.28
127.49
120.76
114.29
55.35
OMe
Br
5c
Normalized Intensity
Chemical Shift (ppm)

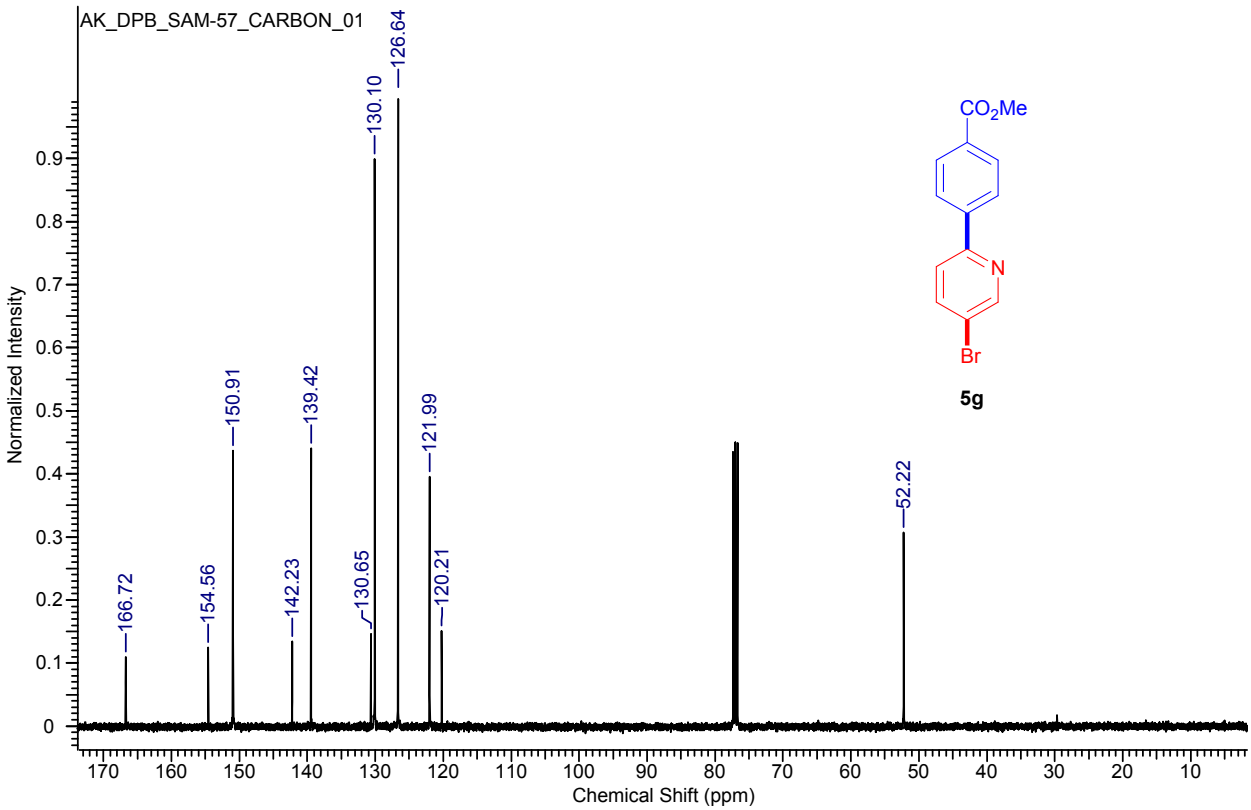

ICT_ARK_DP_DPA-159_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
3.94
8.58
7.68
7.57
7.37
7.29
7.12
OMe
Br
5d

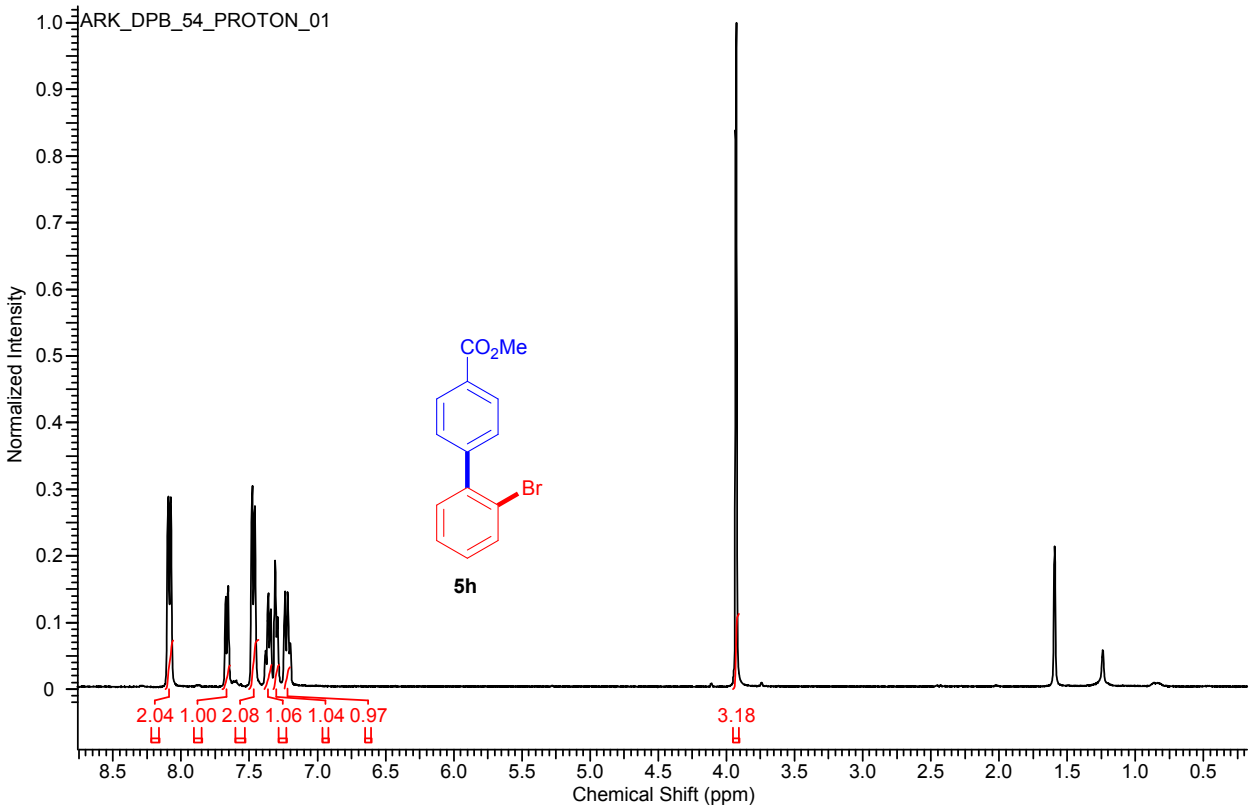

ICT_ARK_DP_159_C_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
159.15
137.60
132.18
131.34
127.79
126.89
125.42
122.51
113.85
55.38
OMe
Br
5d

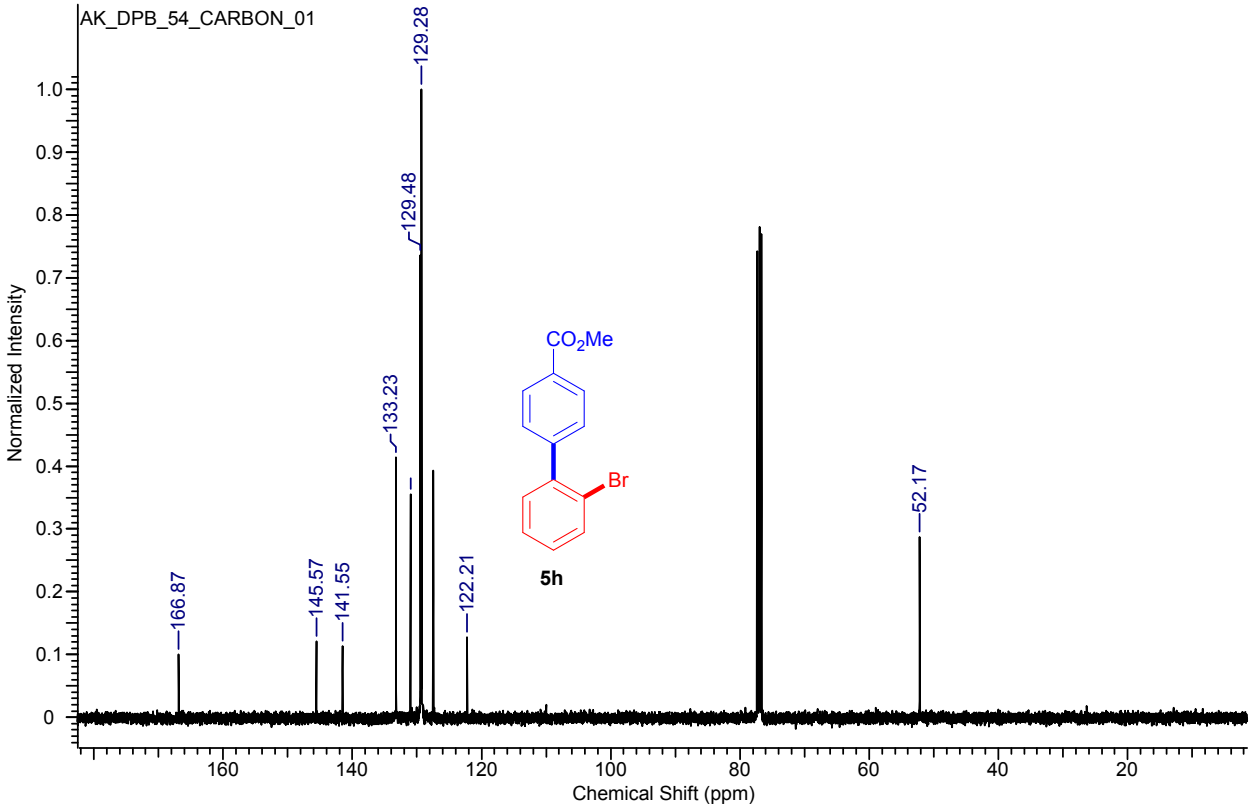

ARK_DPB_56_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
OMe
N
Br
5f
0.99
2.09
1.00
1.19
2.09
3.22

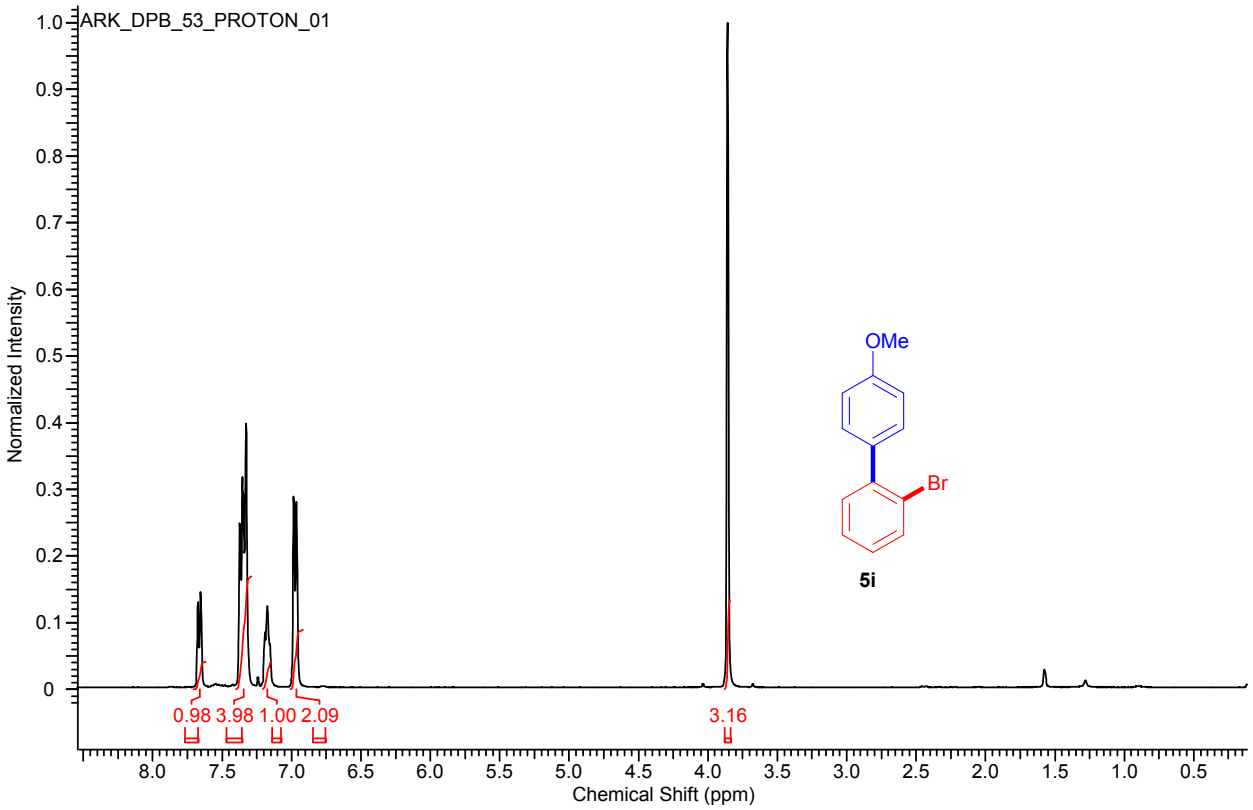

AK_DPB_SAM-56_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
160.69
155.55
150.47
139.15
130.79
128.05
120.86
118.37
114.19
55.35
OMe
N
Br
5f

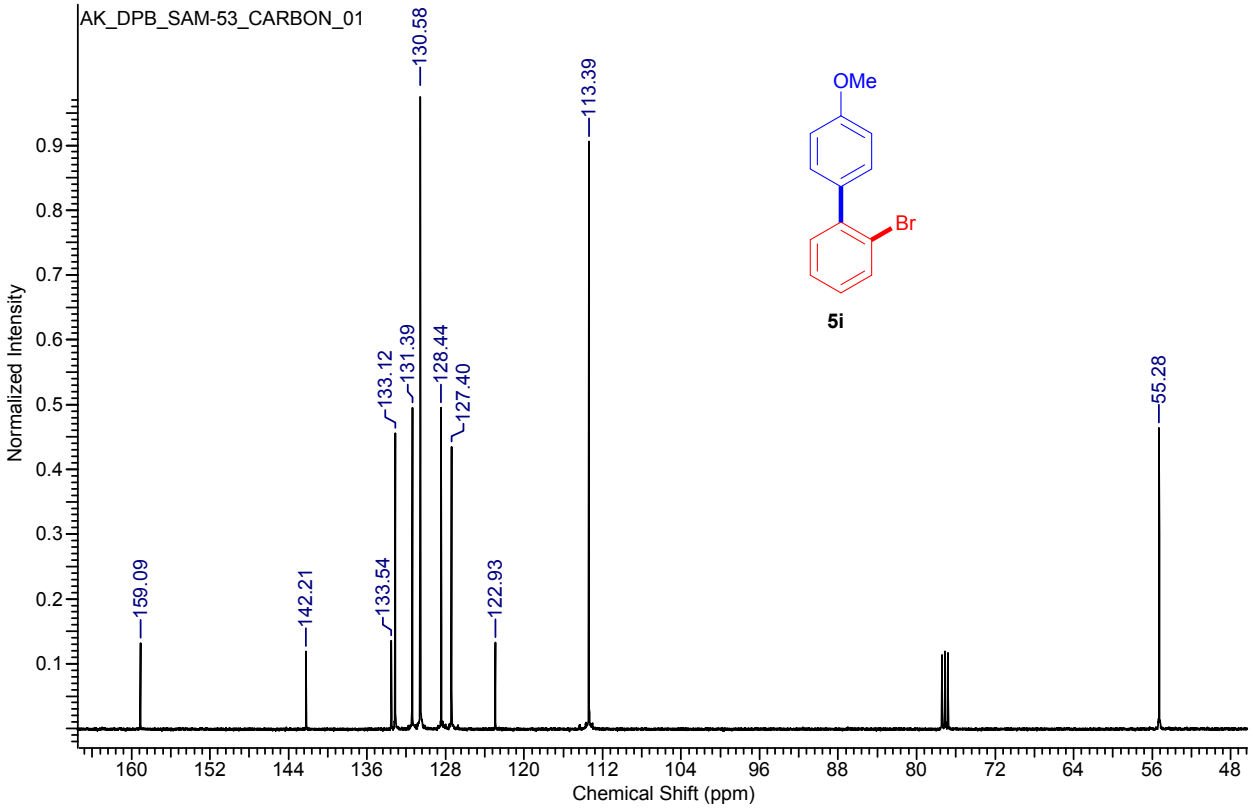

ARK_DPB_57_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
0.97
1.97
2.25
1.05
1.01
3.23
CO2Me
N
Br
5g

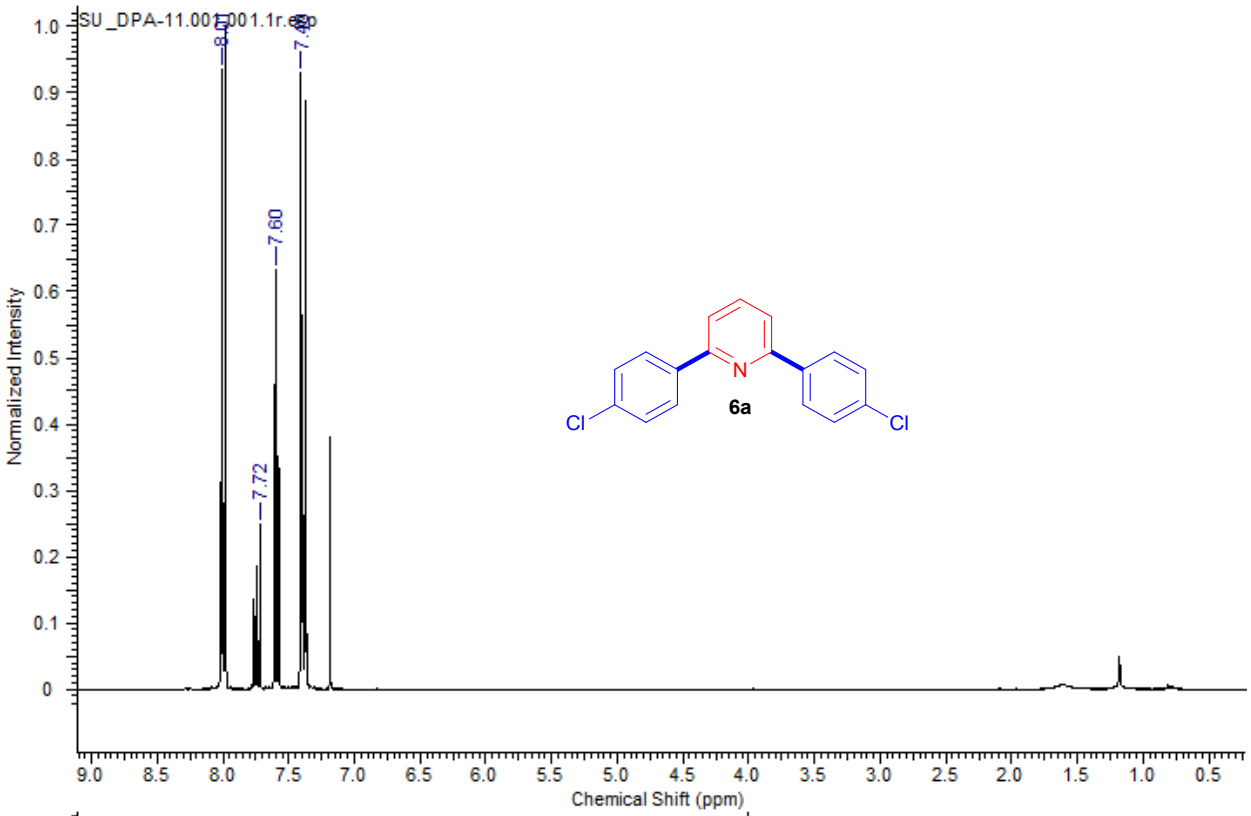

AK_DPB_SAM-57_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
166.72
154.56
150.91
142.23
139.42
130.65
130.10
126.64
121.99
120.21
52.22
CO2Me
N
Br
5g

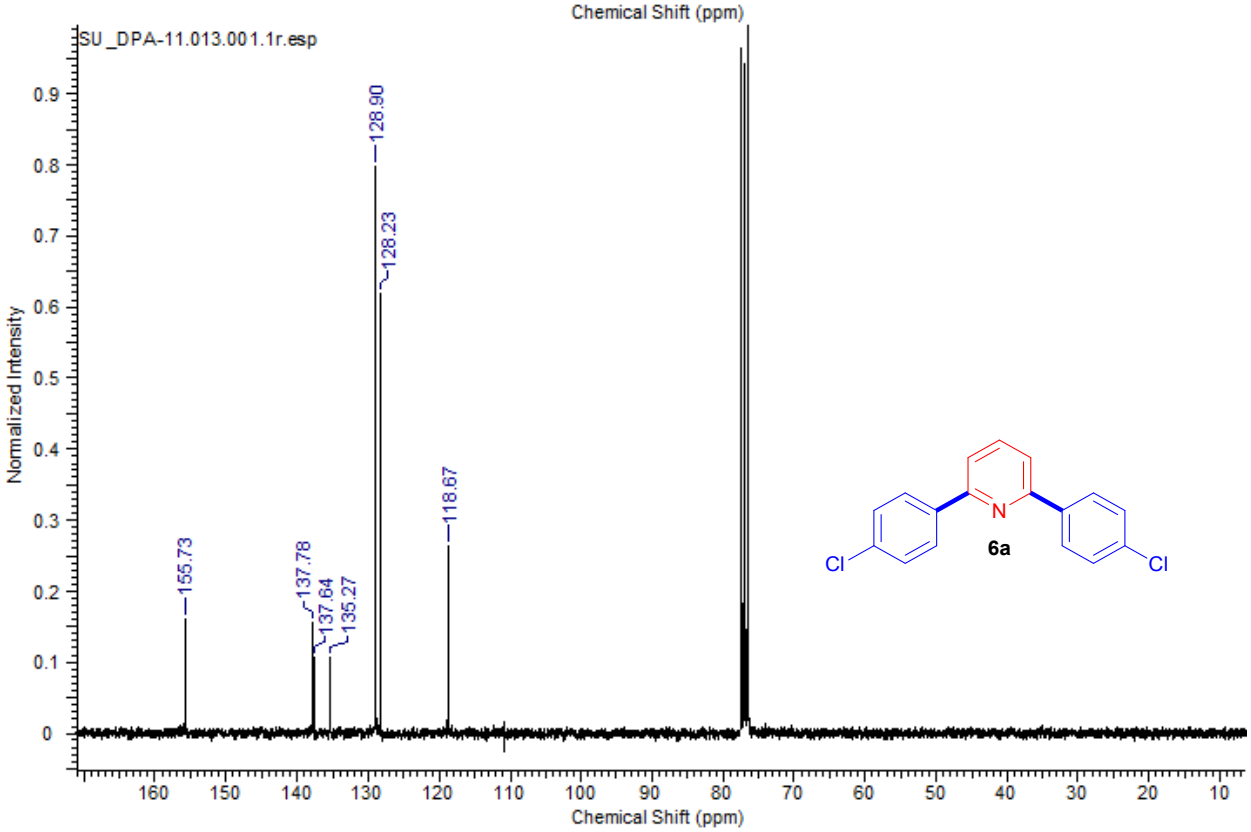

ARK_DPB_54_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
CO2Me
Br
5h
2.04 1.00 2.08 1.06 1.04 0.97
3.18

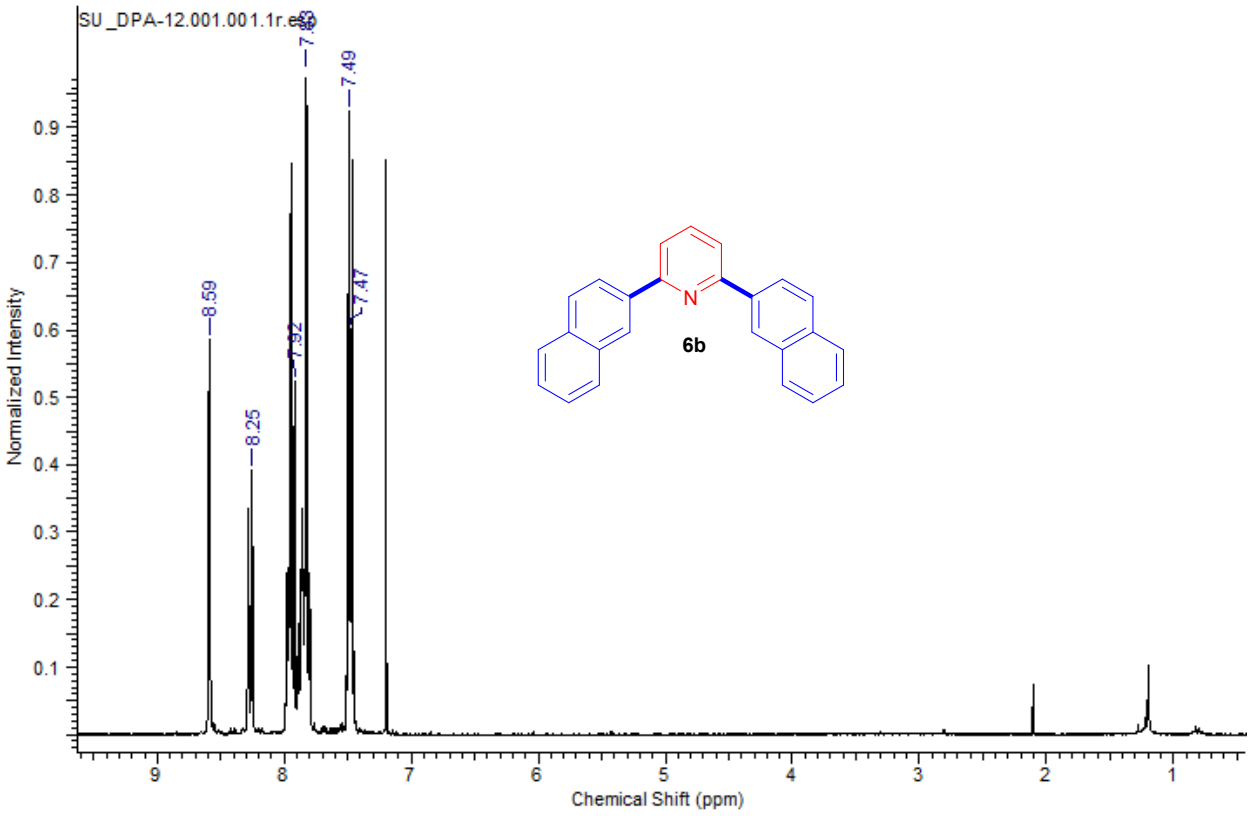

AK_DPB_54_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
CO2Me
Br
5h
166.87
145.57
141.55
133.23
129.48
129.28
122.21
52.17

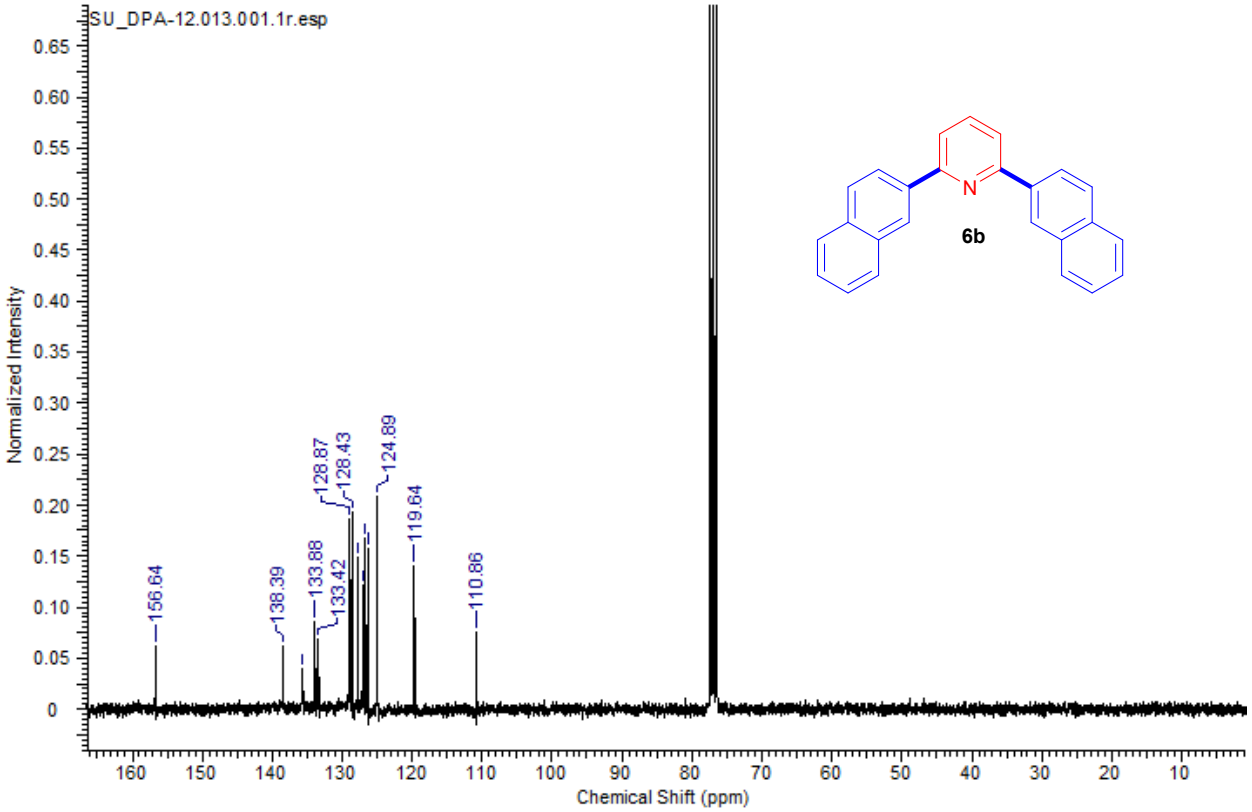

ARK_DPB_53_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
0.98 3.98 1.00 2.09
3.16
OMe
Br
5i

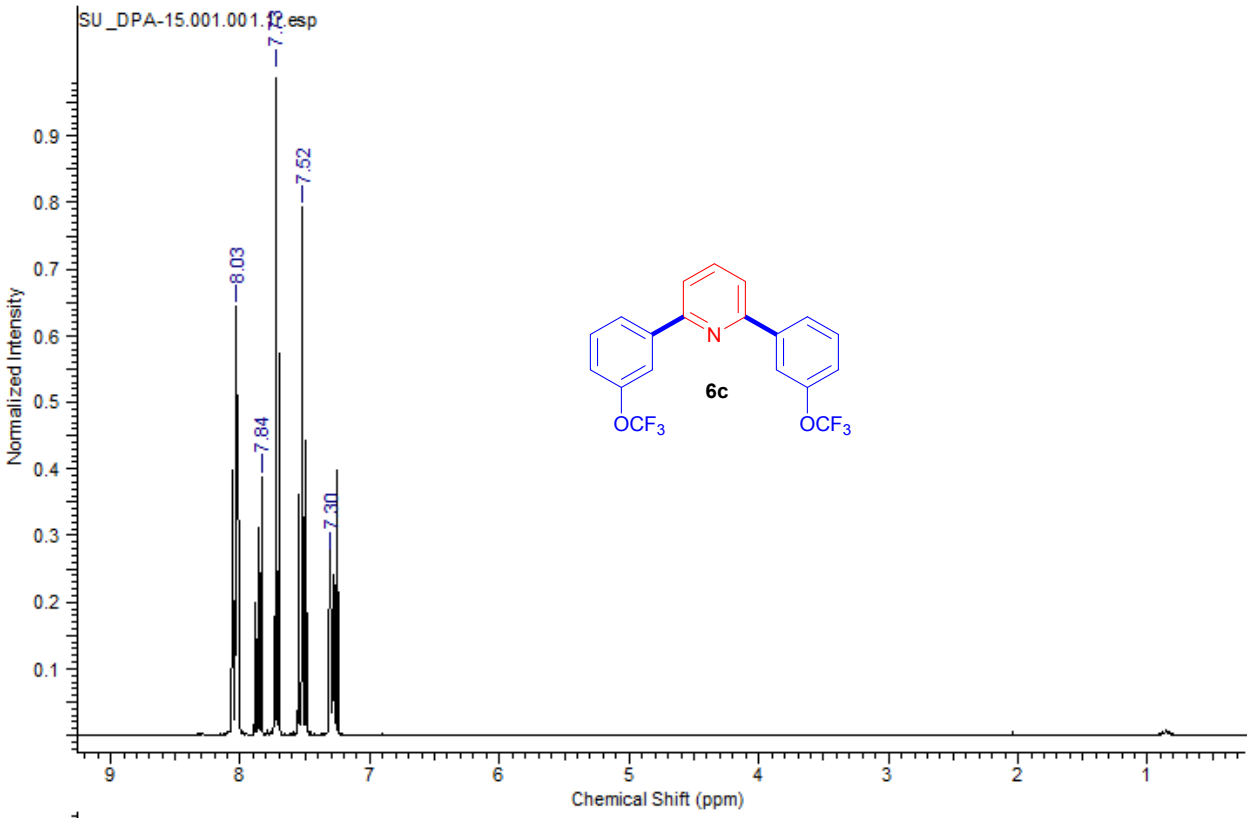

AK_DPB_SAM-53_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
159.09
142.21
133.54
133.12
131.39
130.58
128.44
127.40
122.93
113.39
55.28
OMe
Br
5i

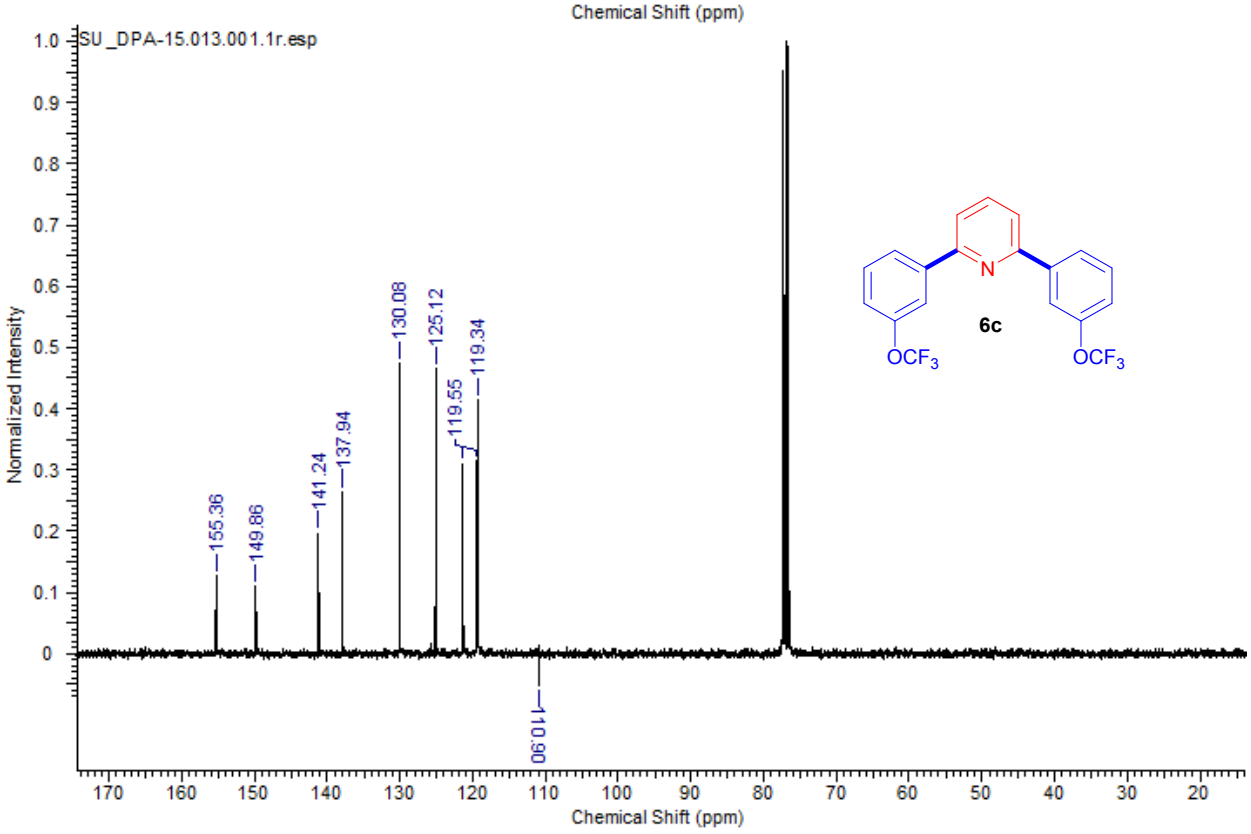

SU _DPA-11.001.001.1r.esp
Normalized Intensity
7.72
7.60
Chemical Shift (ppm)
N
Cl
Cl
6a

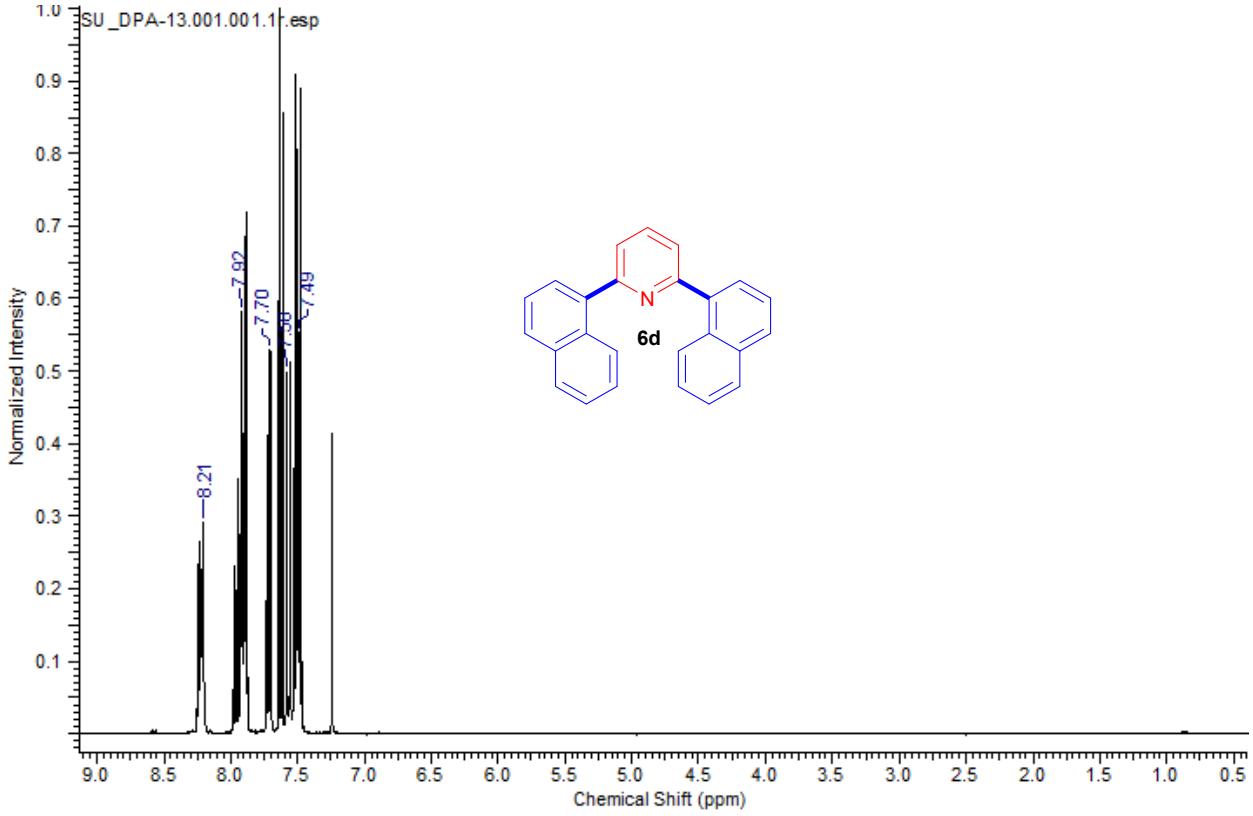

SU _DPA-11.013.001.1r.esp
Normalized Intensity
155.73
137.78
137.64
135.27
128.90
128.23
118.67
Chemical Shift (ppm)
N
Cl
Cl
6a

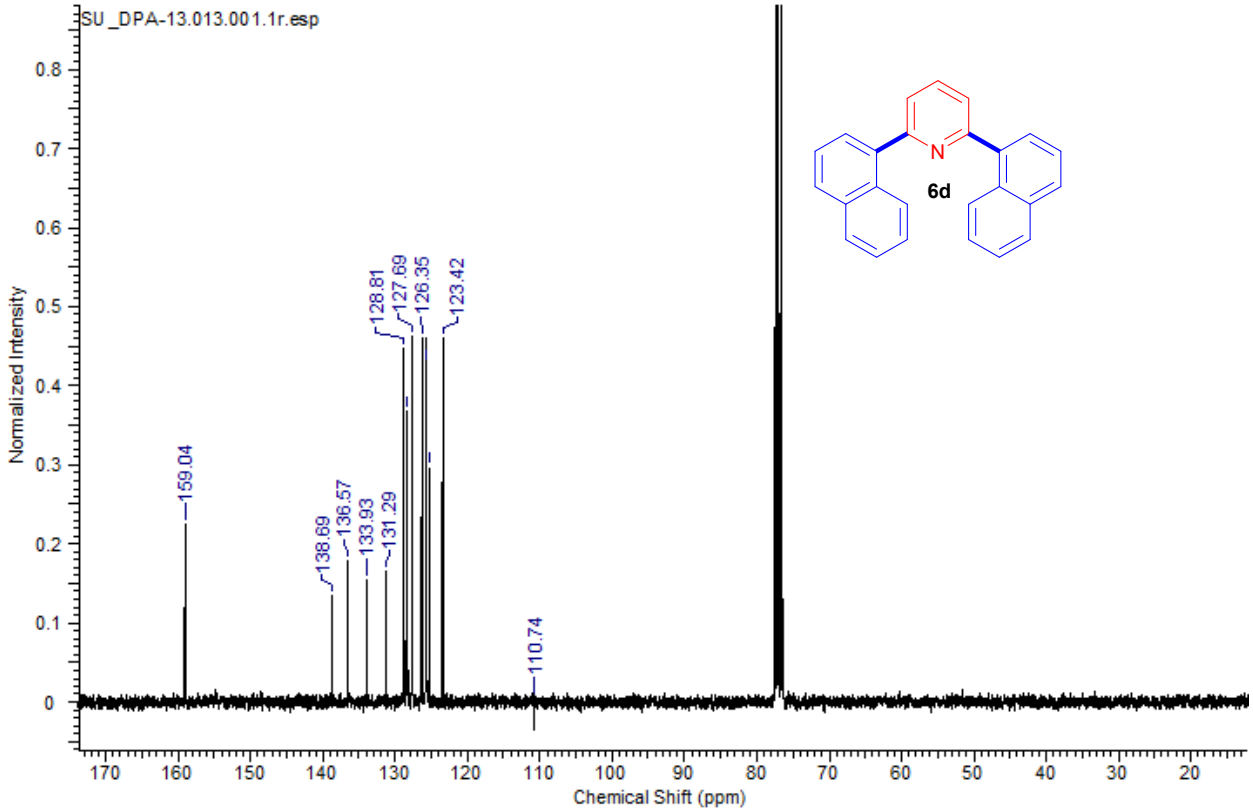

SU_DPA-12.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.59
8.25
7.92
7.49
7.47
6b

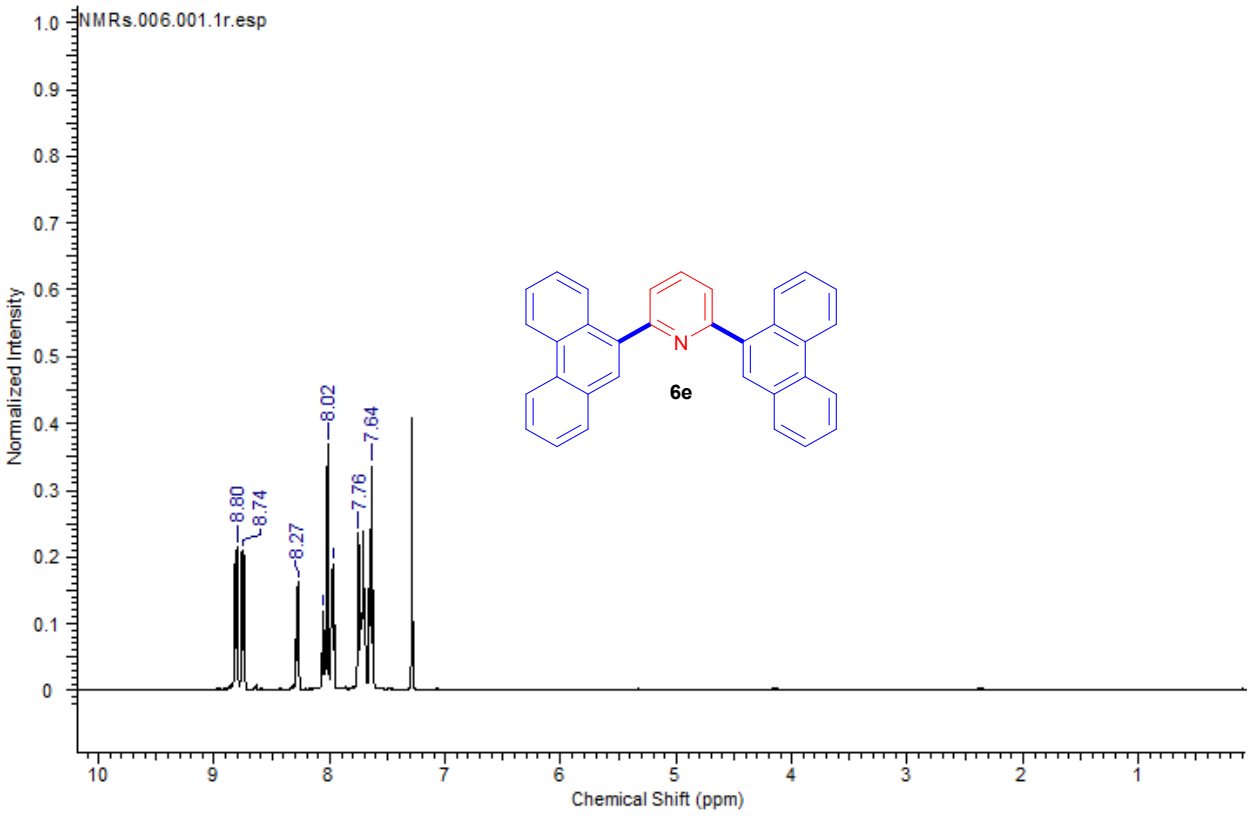

SU_DPA-12.013.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
156.64
138.39
133.88
133.42
128.87
128.43
124.89
119.64
110.86
6b

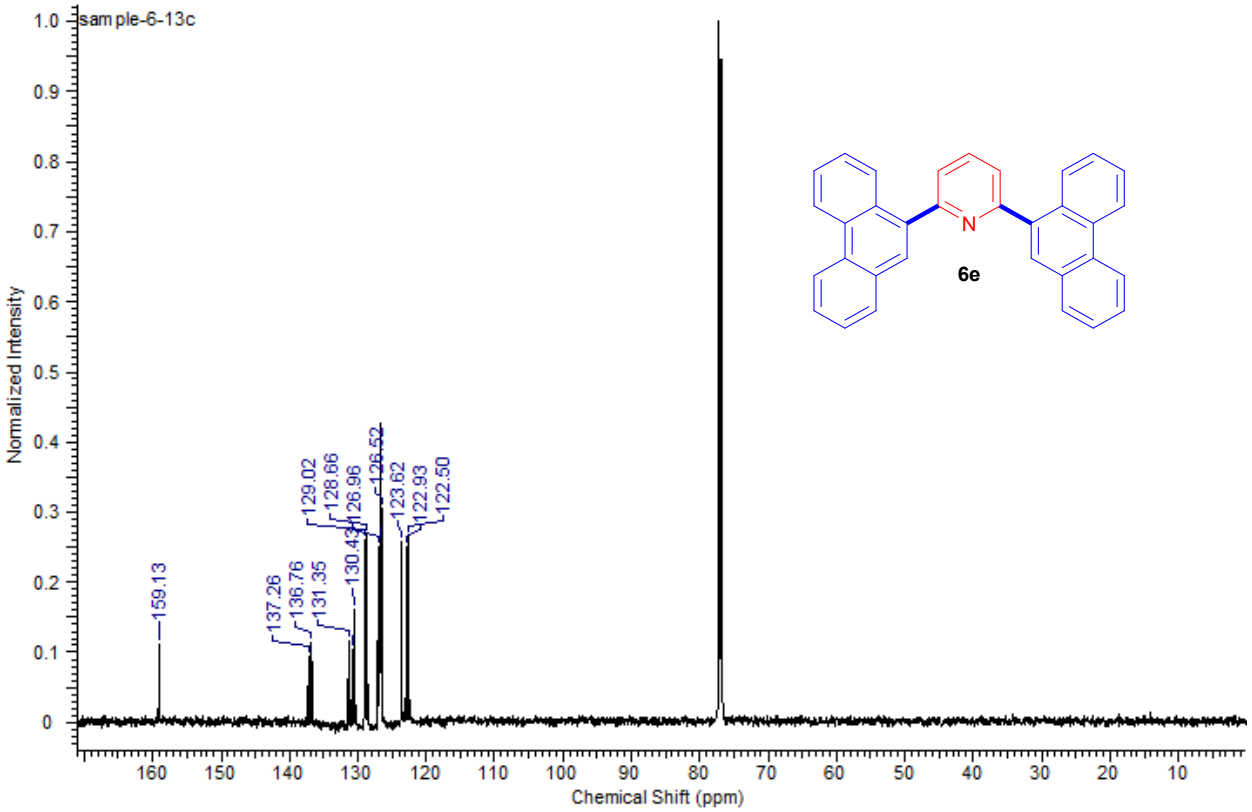

SU _DPA-15.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.03
7.84
7.52
7.30
6c
OCF3
OCF3

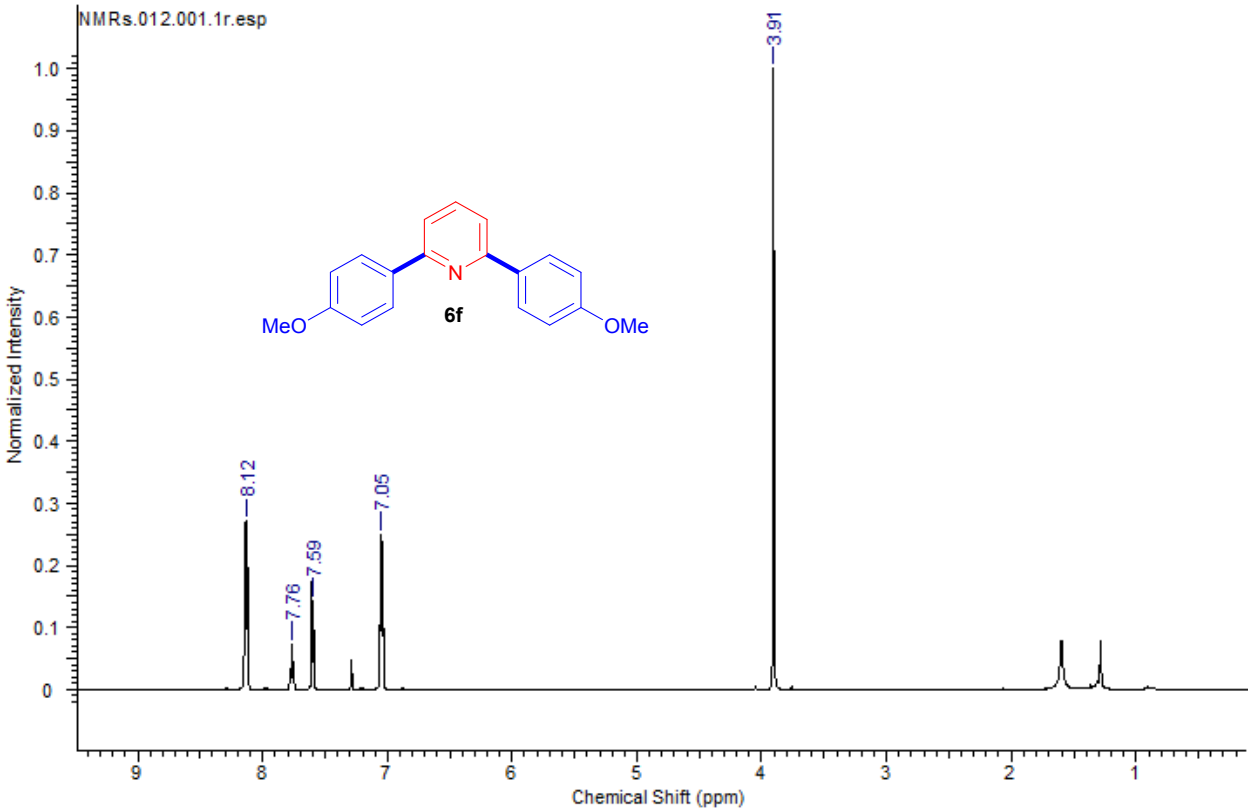

SU _DPA-15.013.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
155.36
149.86
141.24
137.94
130.08
125.12
119.55
119.34
110.90
6c
OCF3
OCF3

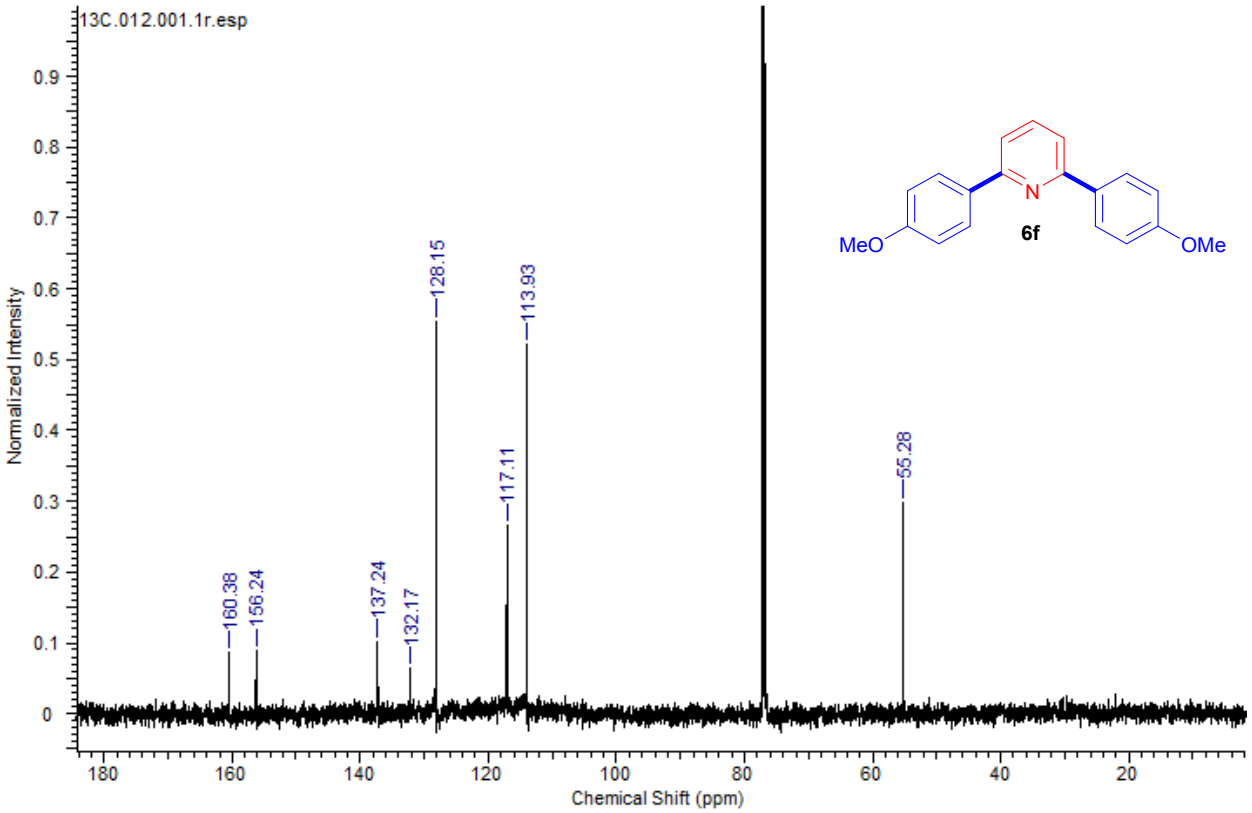

SU _DPA-13.001.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.21
7.92
7.70
7.49
N
6d

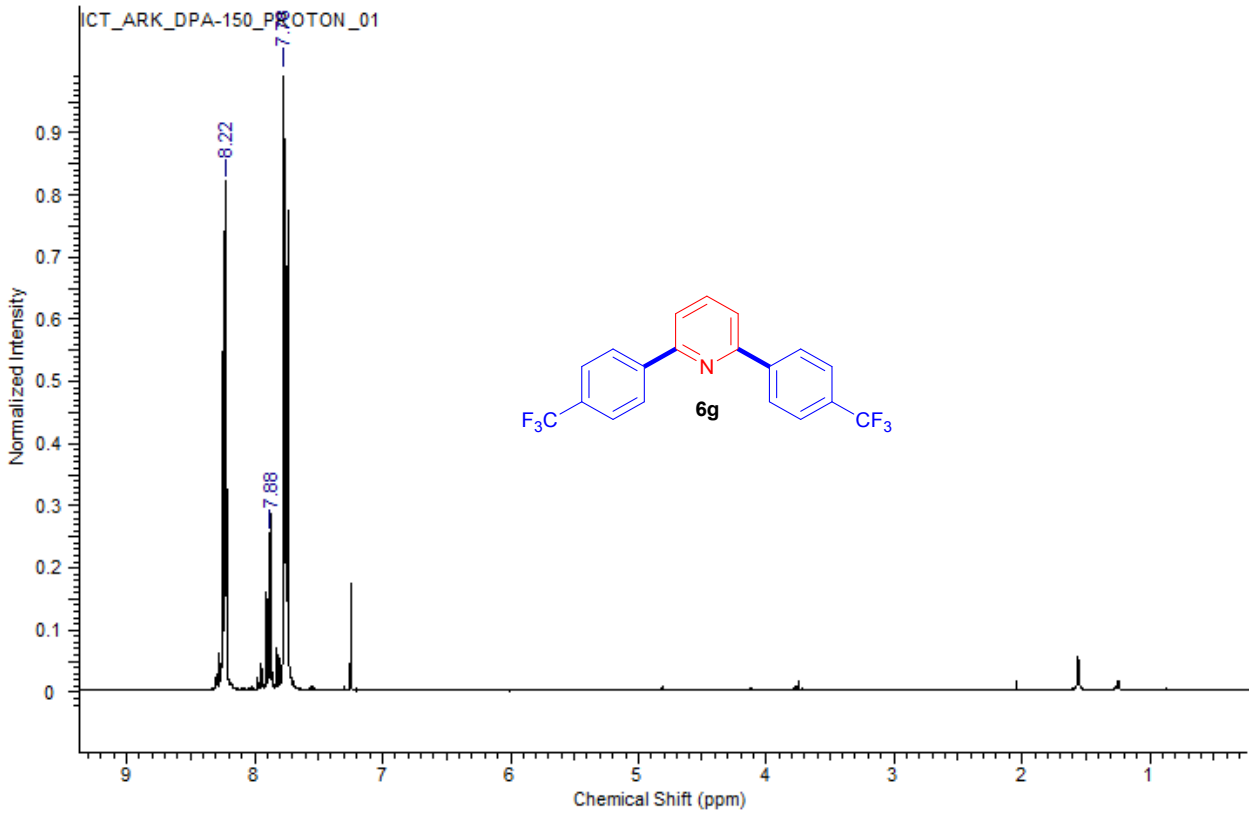

SU _DPA-13.013.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
159.04
138.69
136.57
133.93
131.29
128.81
127.69
126.35
123.42
110.74
N
6d

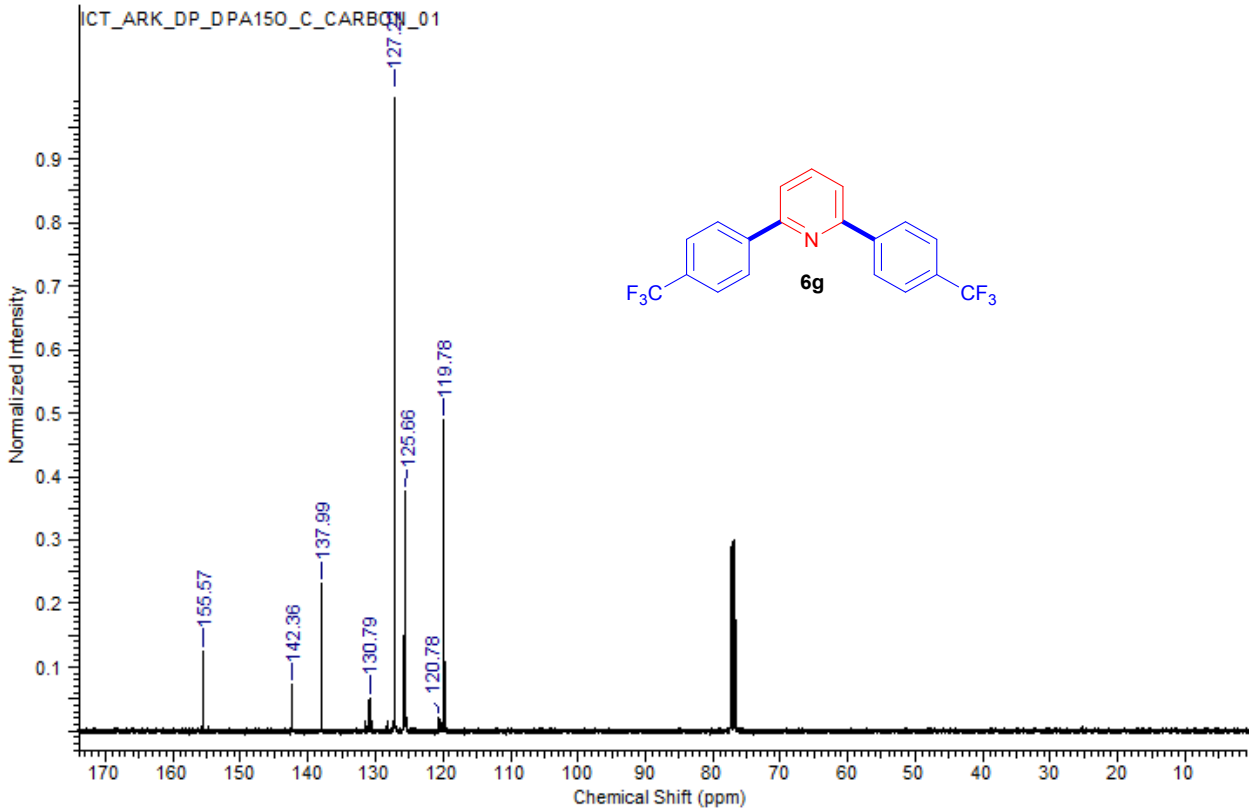

NMRs.006.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.80
8.74
8.27
8.02
7.76
7.64
N
6e

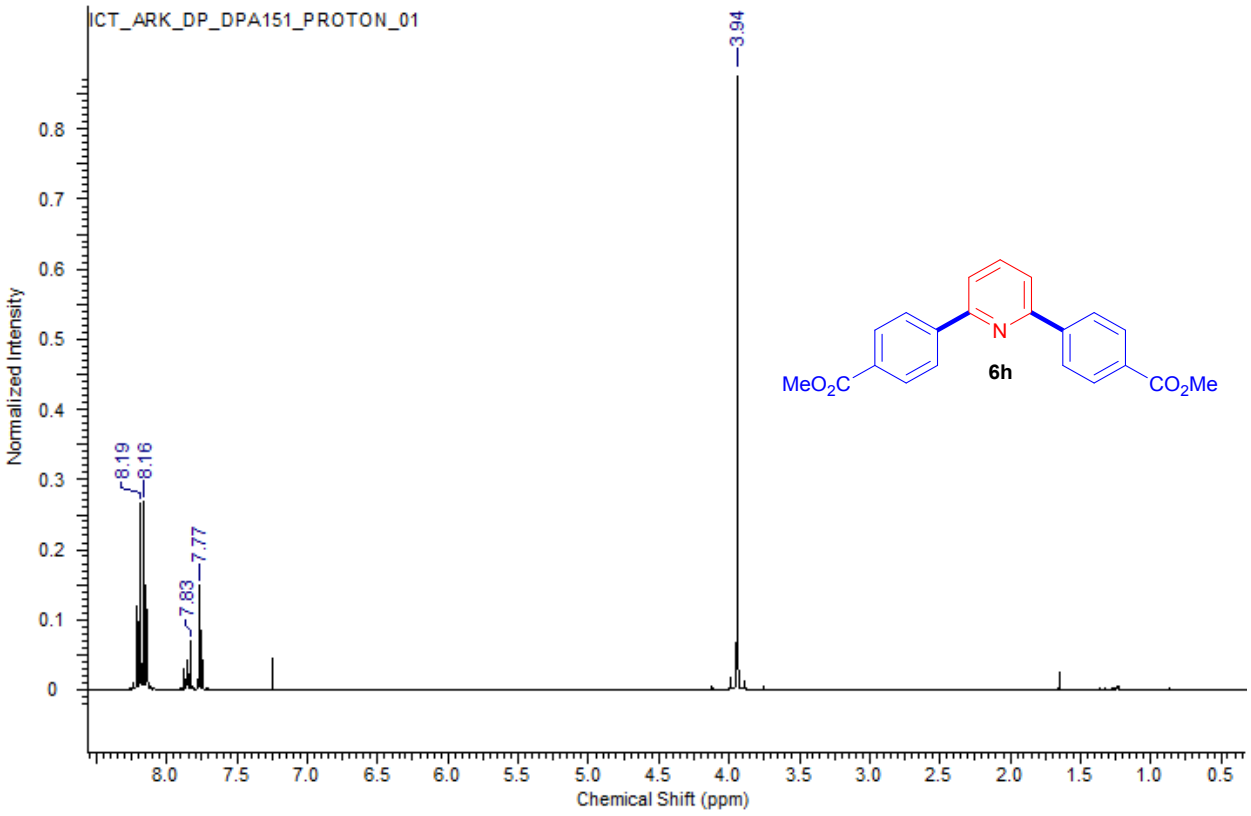

sample-6-13c
Normalized Intensity
Chemical Shift (ppm)
159.13
137.26
136.76
131.35
130.43
129.02
128.66
126.96
126.52
123.62
122.93
122.50
N
6e

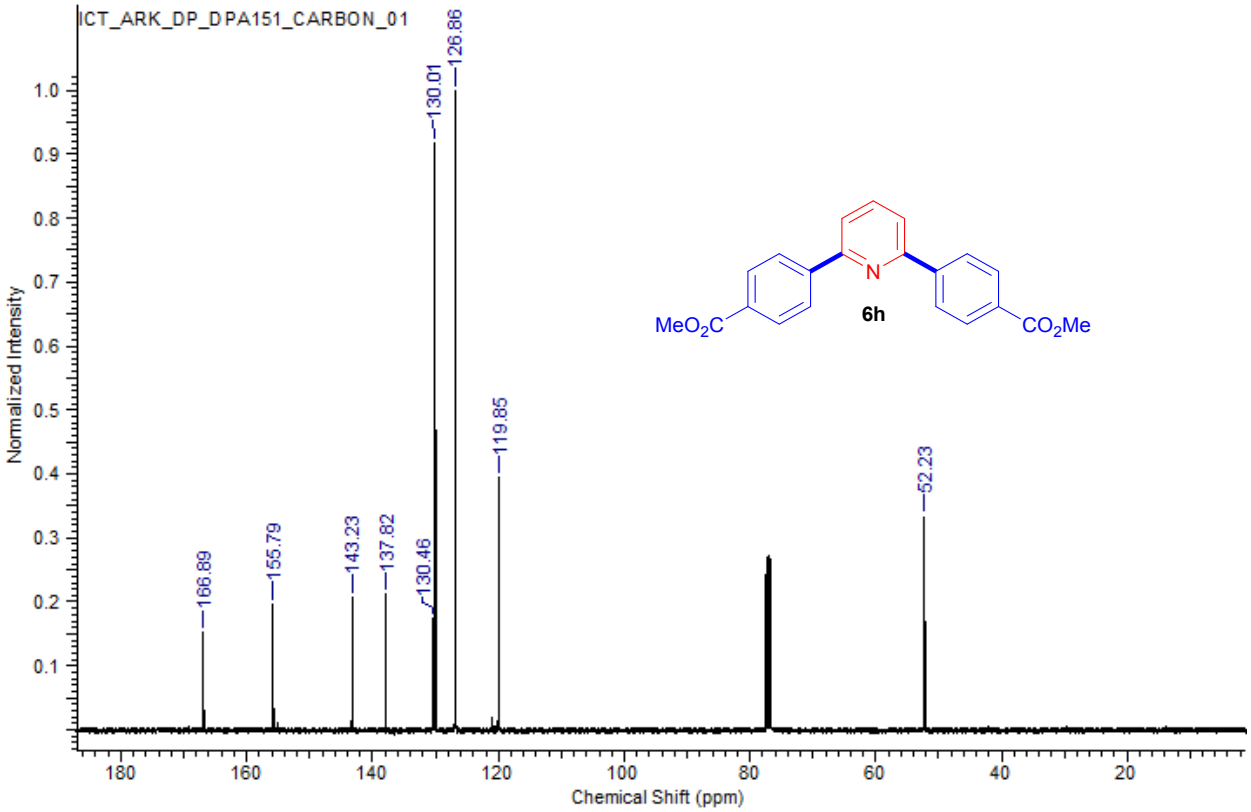

NMRs.012.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
3.91
8.12
7.76
7.59
7.05
MeO
OMe
6f

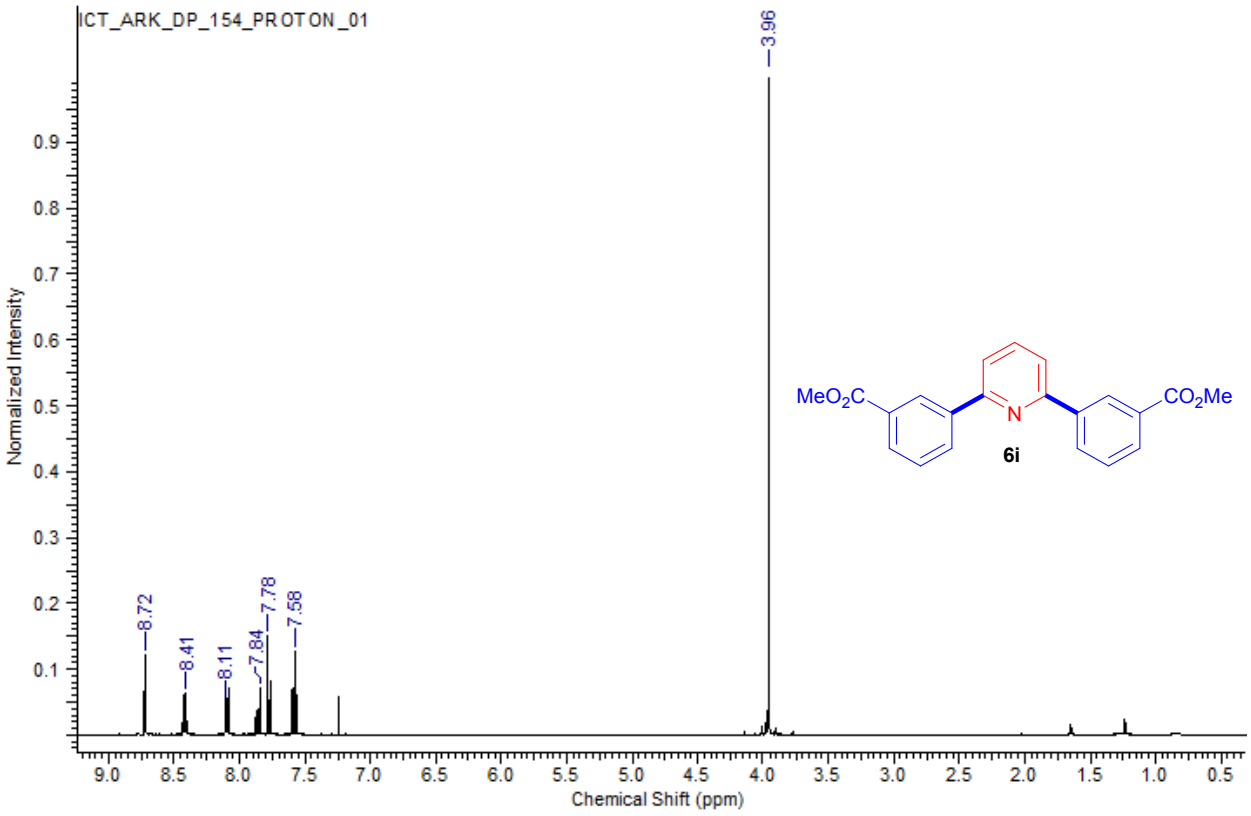

13C.012.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.38
156.24
137.24
132.17
128.15
117.11
113.93
55.28
MeO
OMe
6f

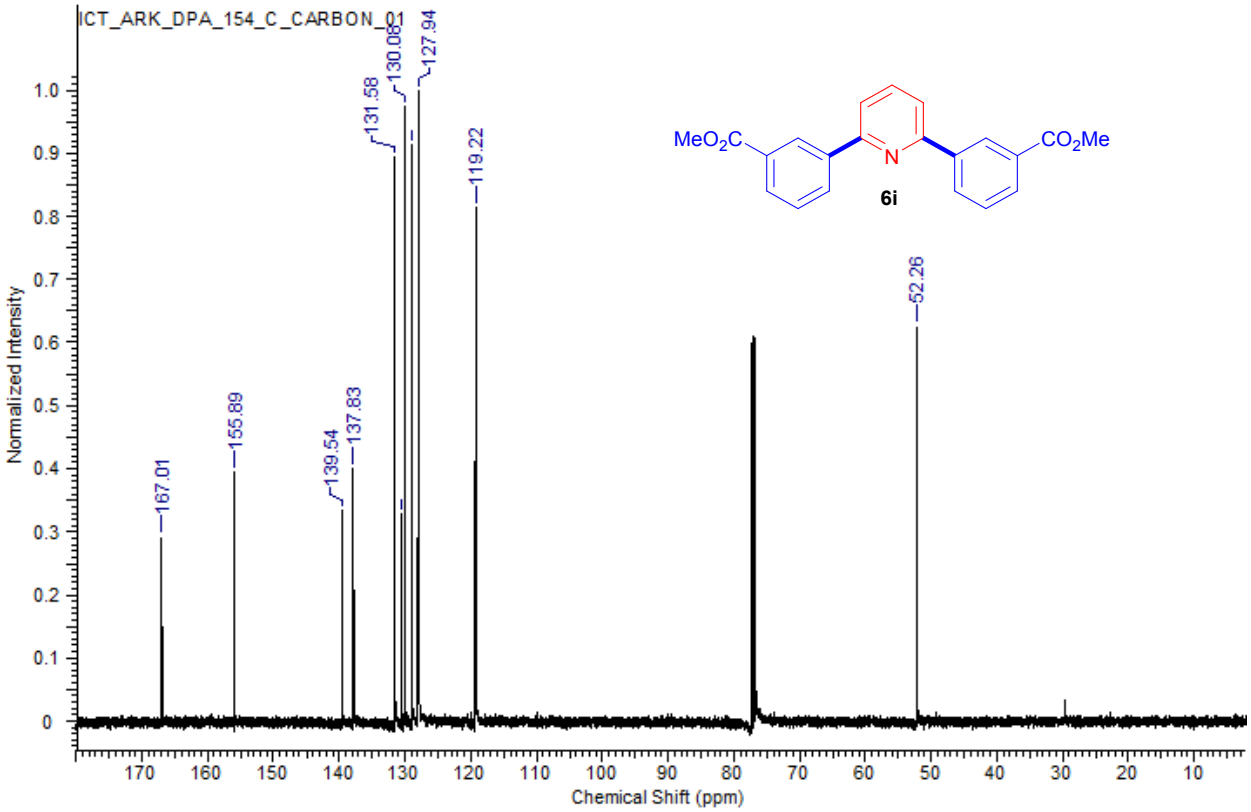

ICT_ARK_DPA-150_PROTON_01
Normalized Intensity
8.22
7.88
Chemical Shift (ppm)
6g

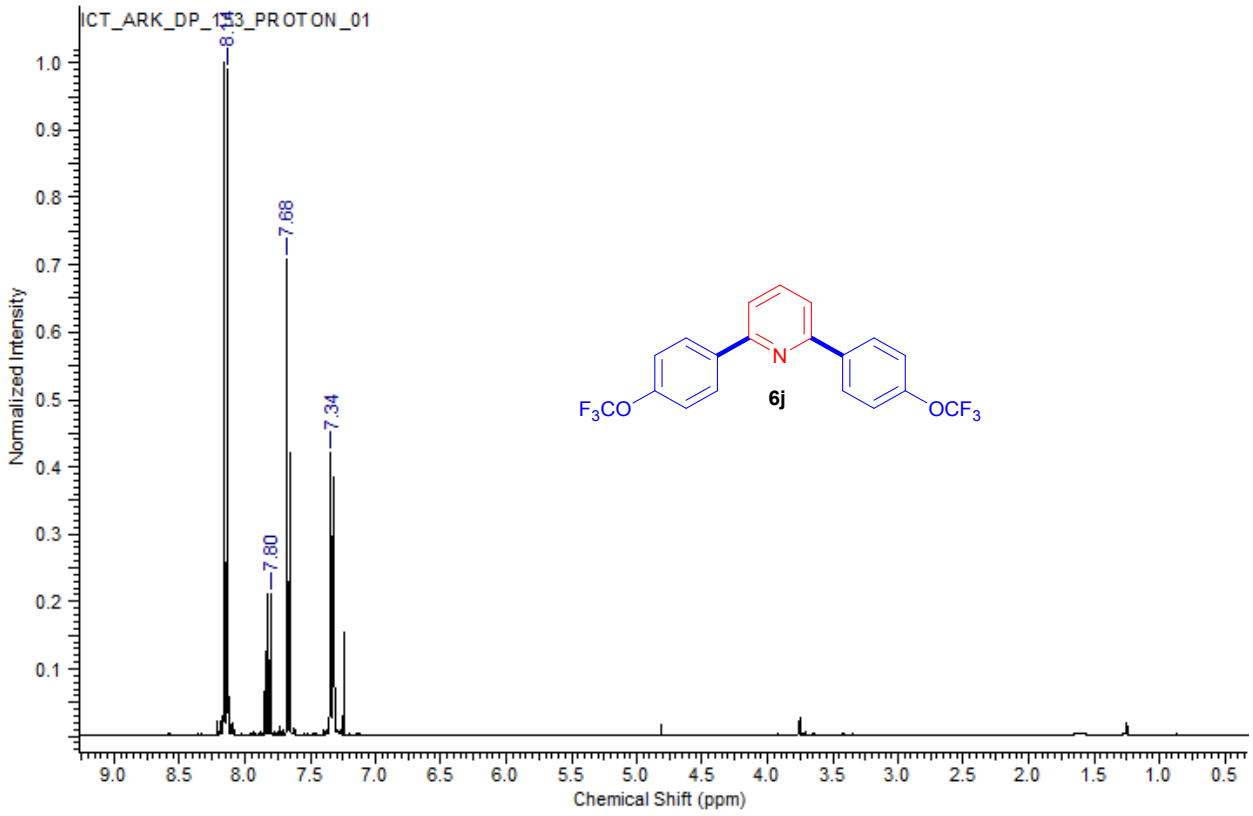

ICT_ARK_DP_DPA15O_C_CARBON_01
Normalized Intensity
127.22
125.66
119.78
155.57
142.36
137.99
130.79
120.78
Chemical Shift (ppm)
6g

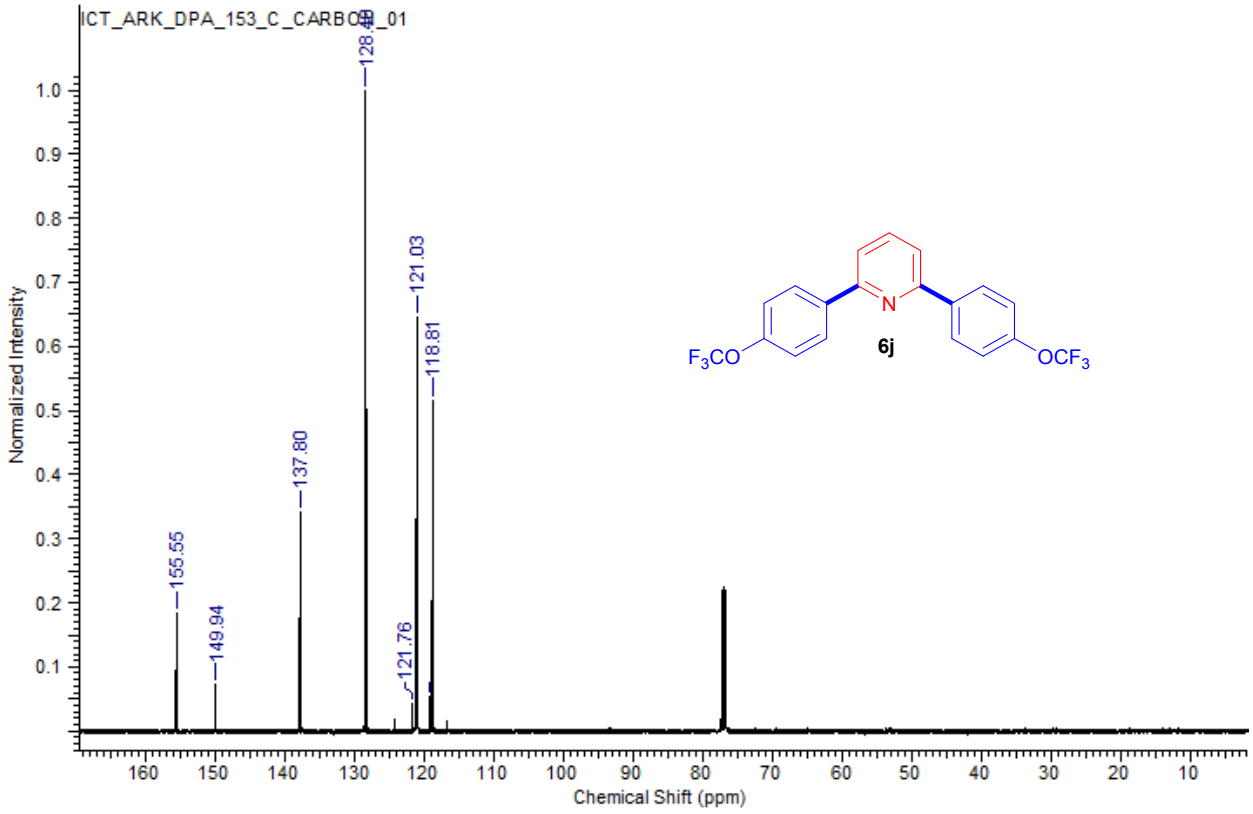

ICT_ARK_DP_DPA151_PROTON_01
3.94
8.19
8.16
7.83
7.77
Normalized Intensity
Chemical Shift (ppm)
N
MeO2C
CO2Me
6h

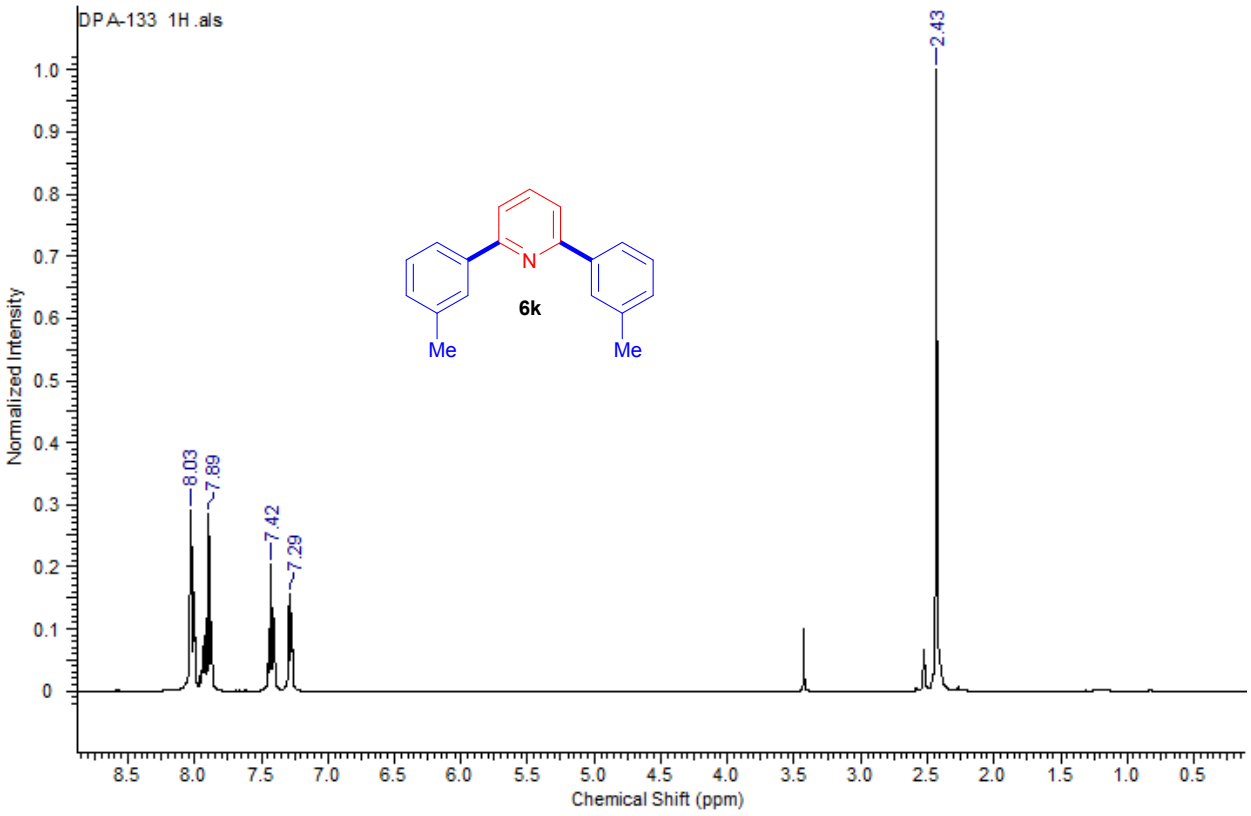

ICT_ARK_DP_DPA151_CARBON_01
126.86
130.01
119.85
52.23
166.89
155.79
143.23
137.82
130.46
Normalized Intensity
Chemical Shift (ppm)
N
MeO2C
CO2Me
6h

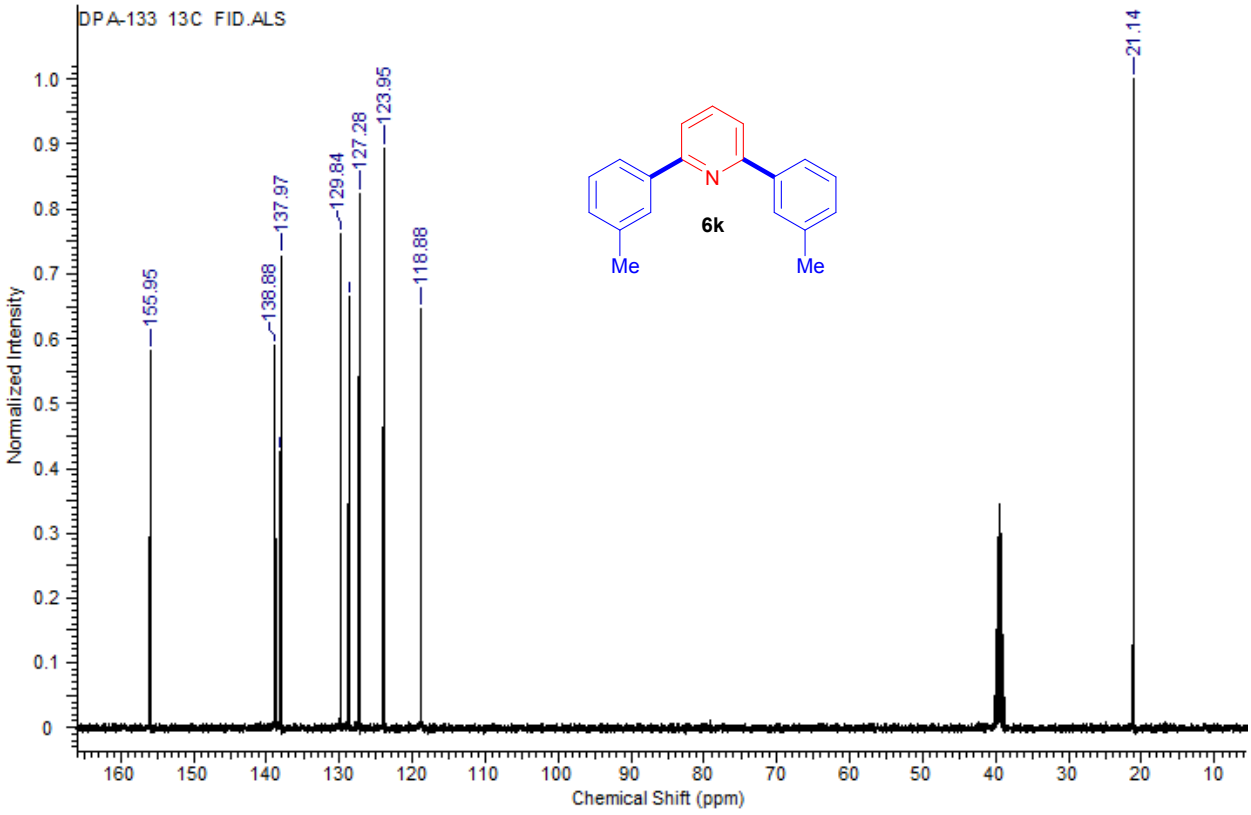

ICT_ARK_DP_154_PROTON_01
3.96
8.72
8.41
8.11
7.84
7.78
7.58
Normalized Intensity
Chemical Shift (ppm)
MeO2C
CO2Me
N
6i

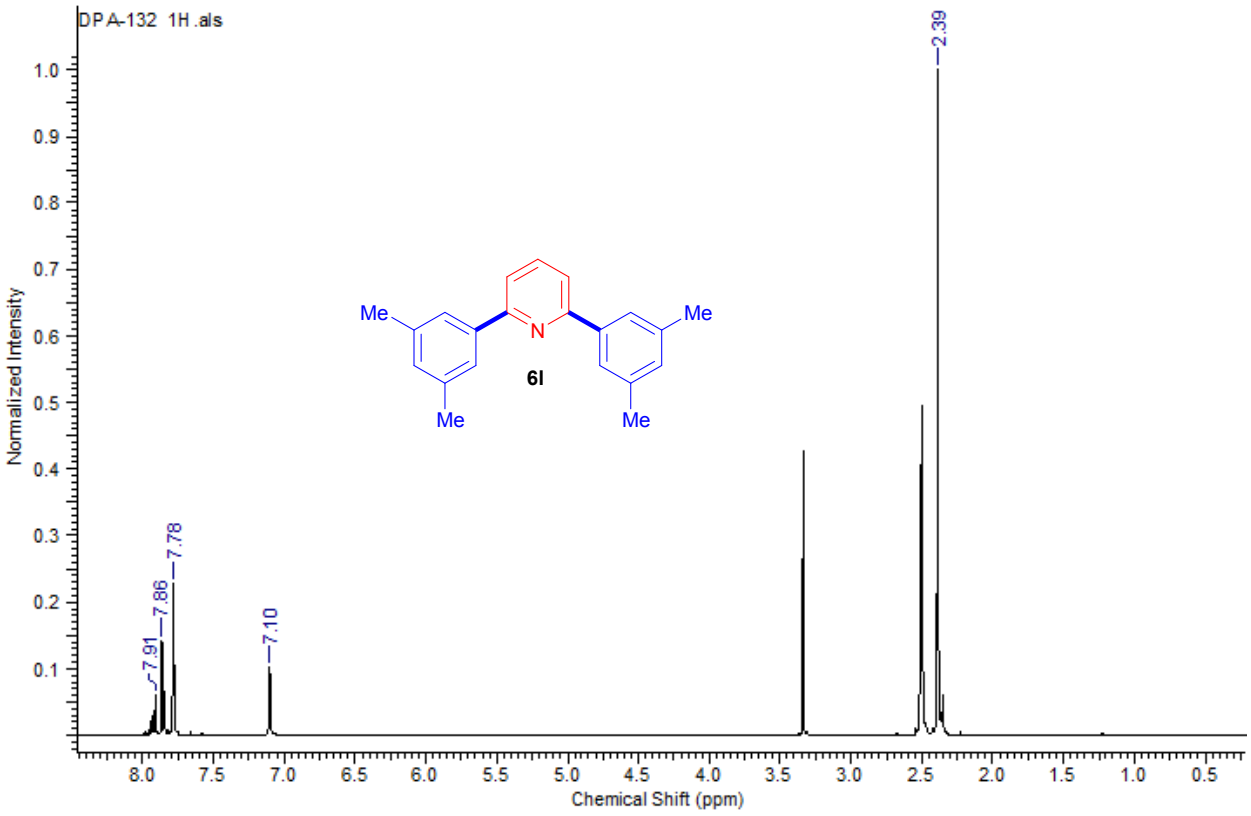

ICT_ARK_DPA_154_C_CARBON_01
167.01
155.89
139.54
137.83
131.58
130.08
127.94
119.22
52.26
Normalized Intensity
Chemical Shift (ppm)
MeO2C
CO2Me
N
6i

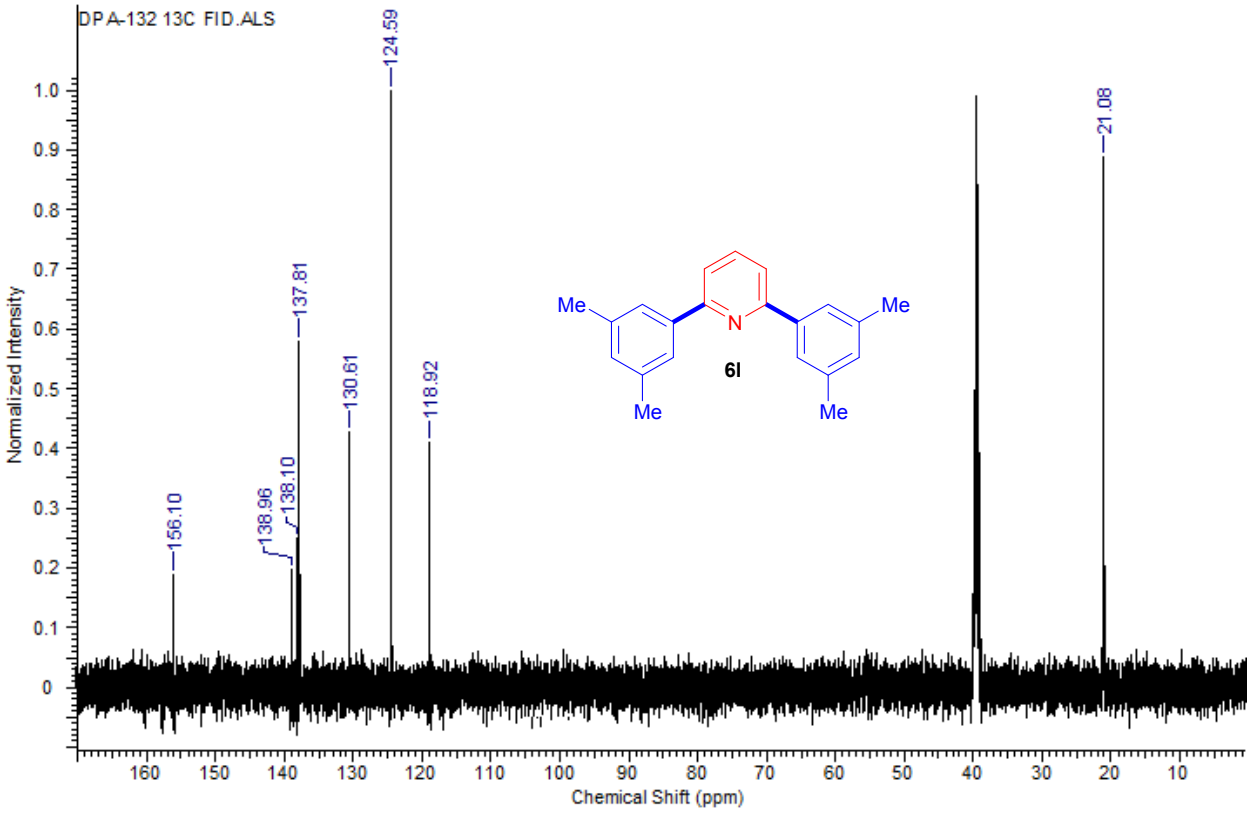

ICT_ARK_DP_153_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
8.11
7.68
7.80
7.34
6j
F3CO
OCF3

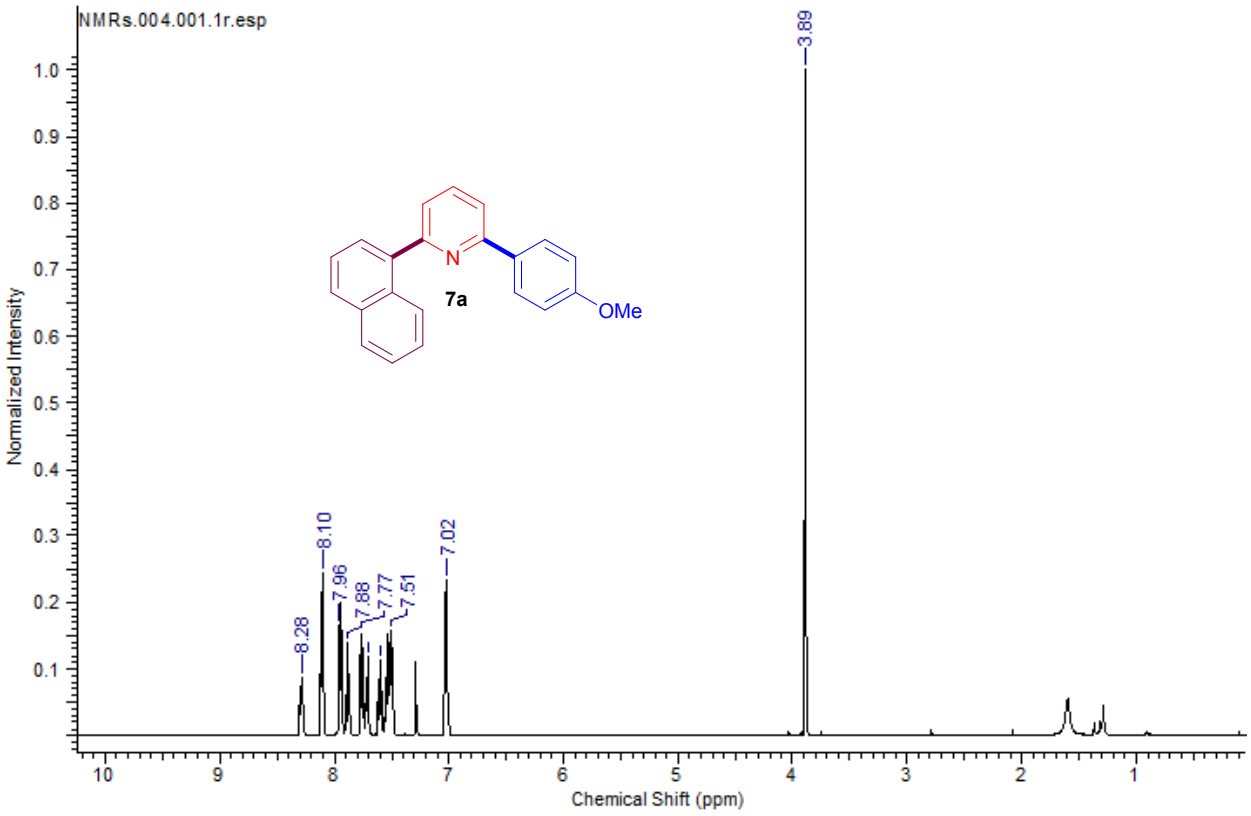

ICT_ARK_DPA_153_C_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
128.49
121.03
118.81
137.80
155.55
149.94
121.76
6j
F3CO
OCF3

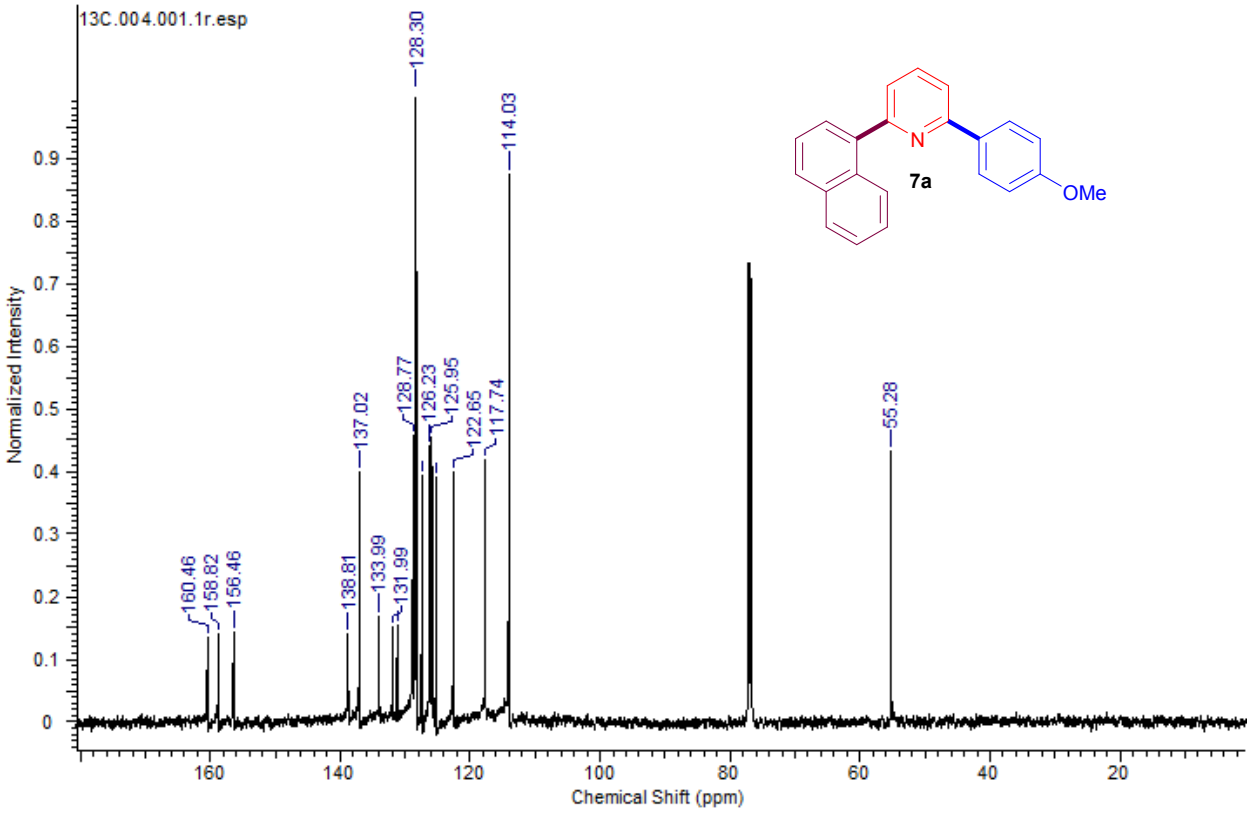

DPA-133 1H.als
Normalized Intensity
Chemical Shift (ppm)
8.03
7.89
7.42
7.29
2.43
N
Me
Me
6k

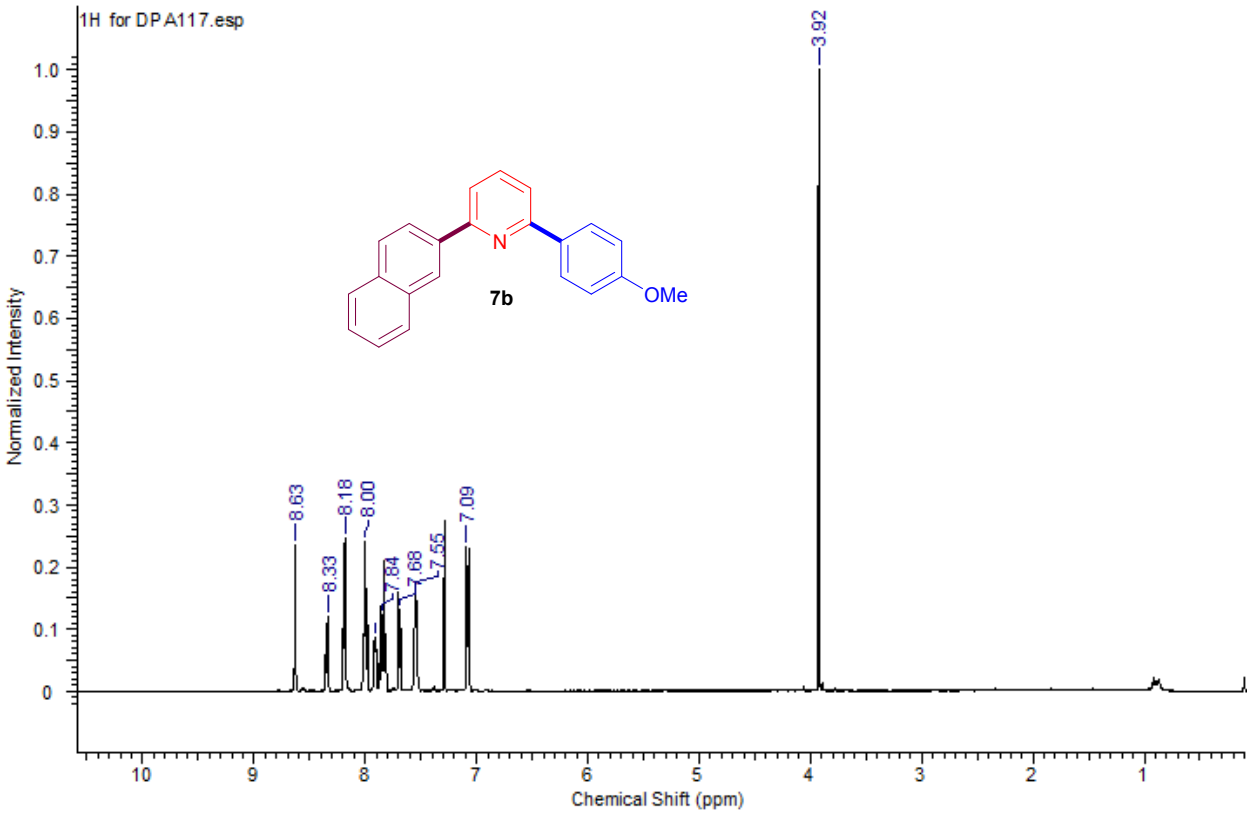

DPA-133 13C FID.ALS
Normalized Intensity
Chemical Shift (ppm)
155.95
138.88
137.97
129.84
127.28
123.95
118.88
21.14
N
Me
Me
6k

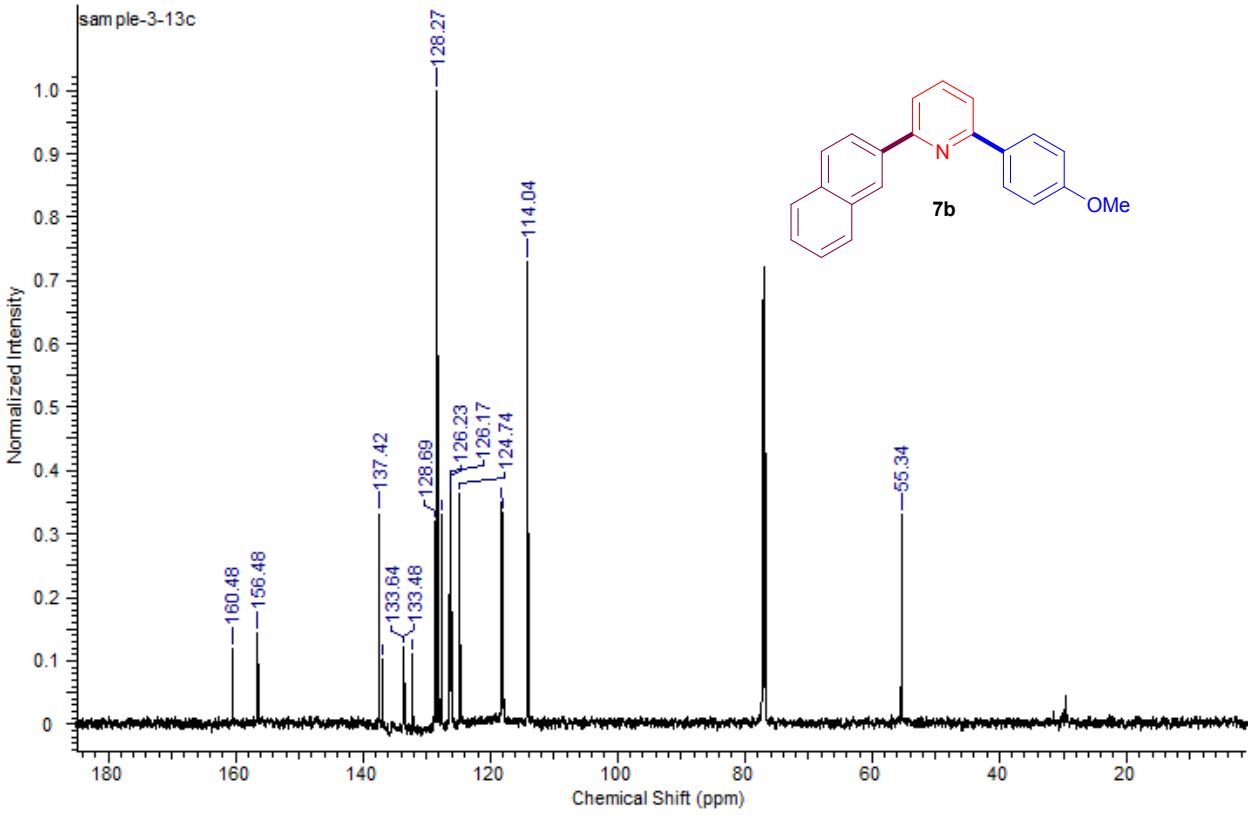

DPA-132 1H.als
Normalized Intensity
Chemical Shift (ppm)
2.39
7.10
7.78
7.86
7.91
Me
Me
Me
Me
N
6l

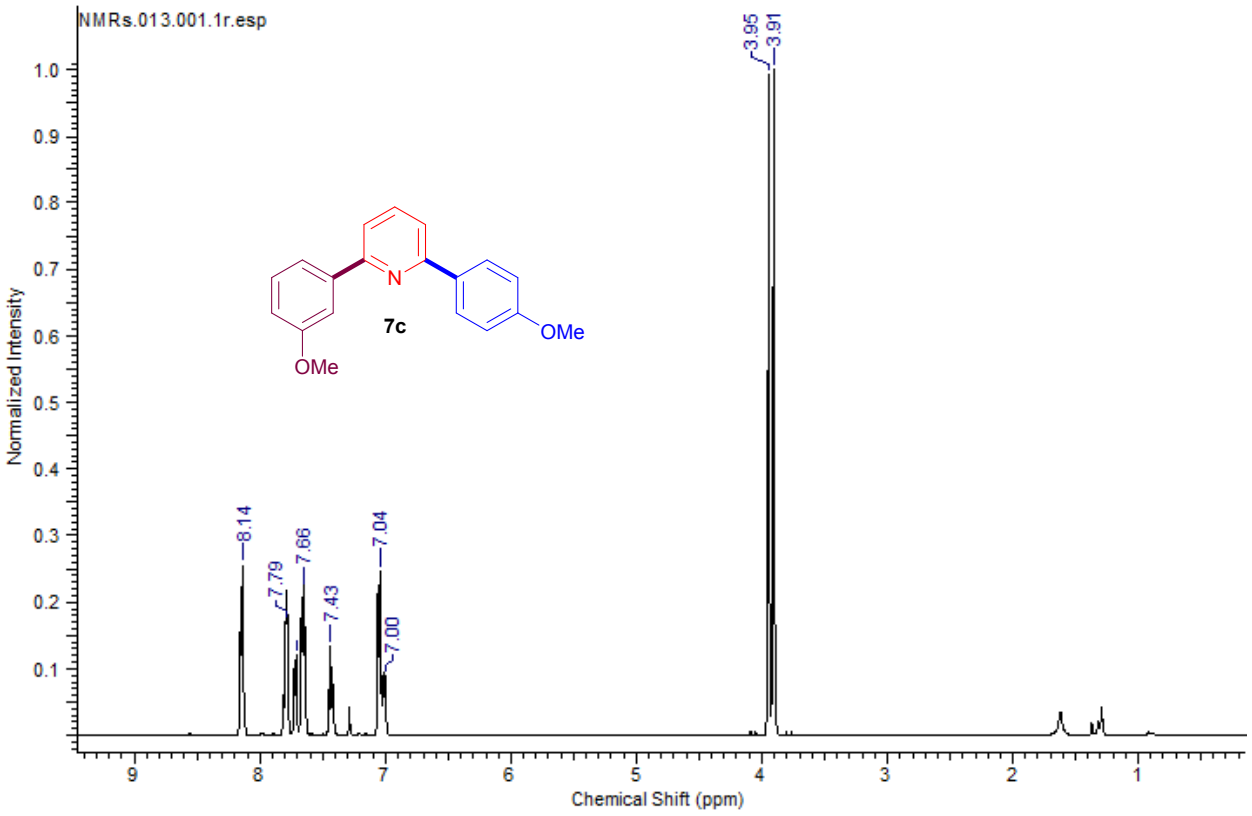

DPA-132 13C FID.ALS
Normalized Intensity
Chemical Shift (ppm)
124.59
137.81
130.61
118.92
156.10
138.96
138.10
21.08
Me
Me
Me
Me
N
6l

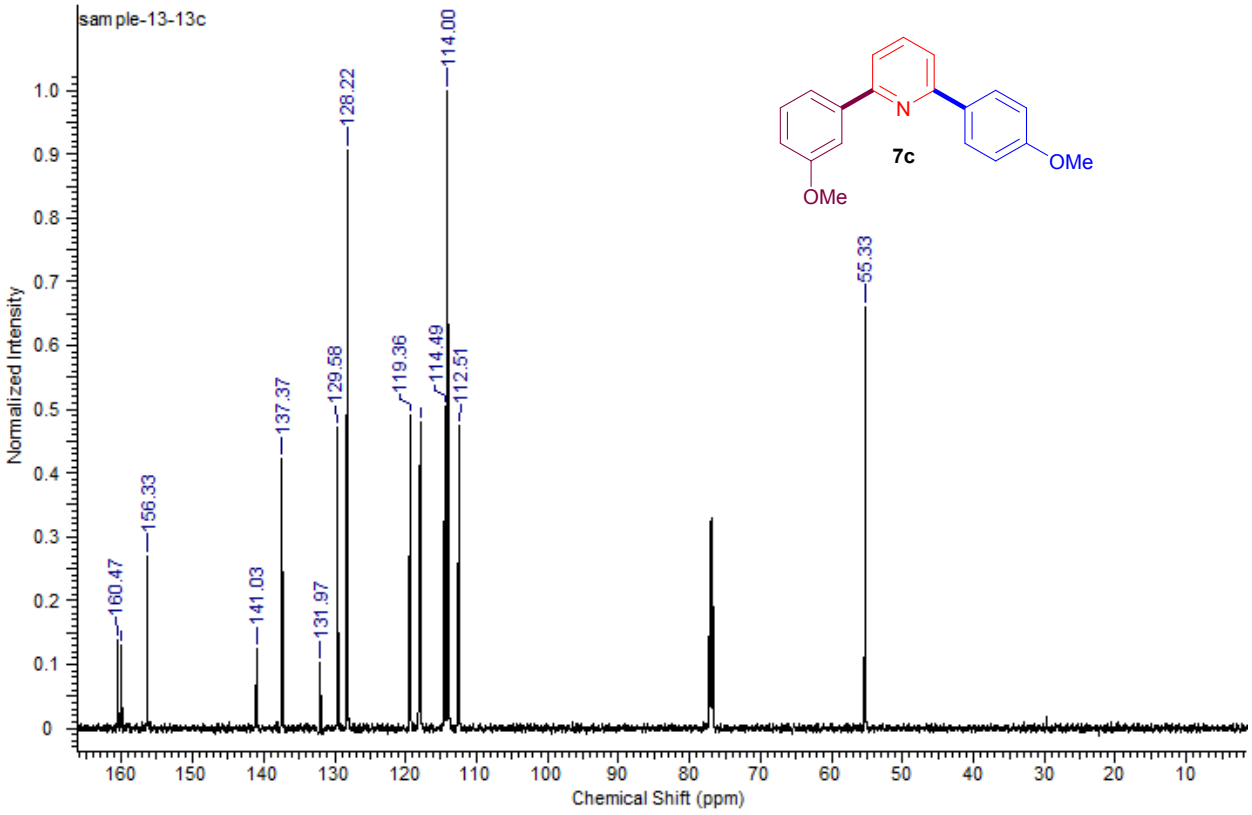

NMRs.004.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
3.89
8.28
8.10
7.96
7.88
7.77
7.51
7.02
N
OMe
7a

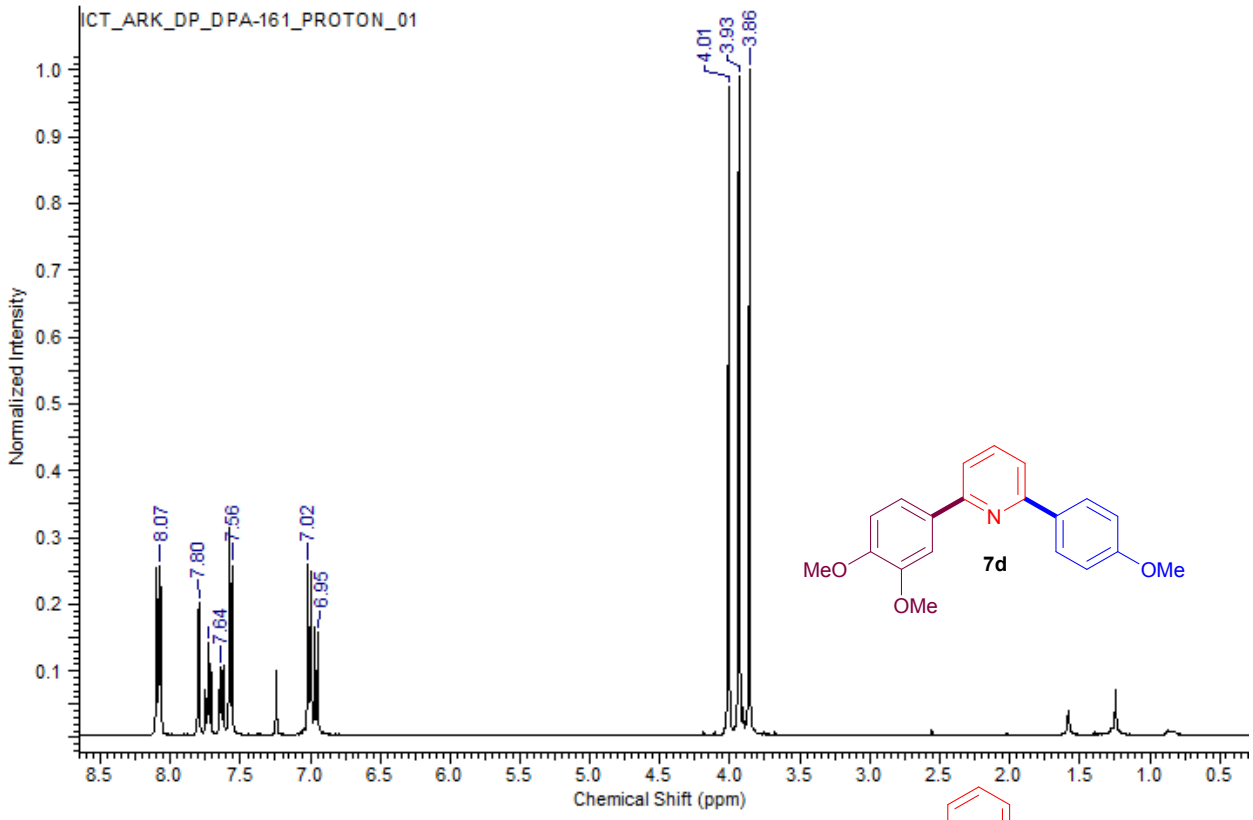

13C.004.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
160.46
158.82
156.46
138.81
137.02
133.99
131.99
128.30
128.77
126.23
125.95
122.65
117.74
114.03
55.28
N
OMe
7a

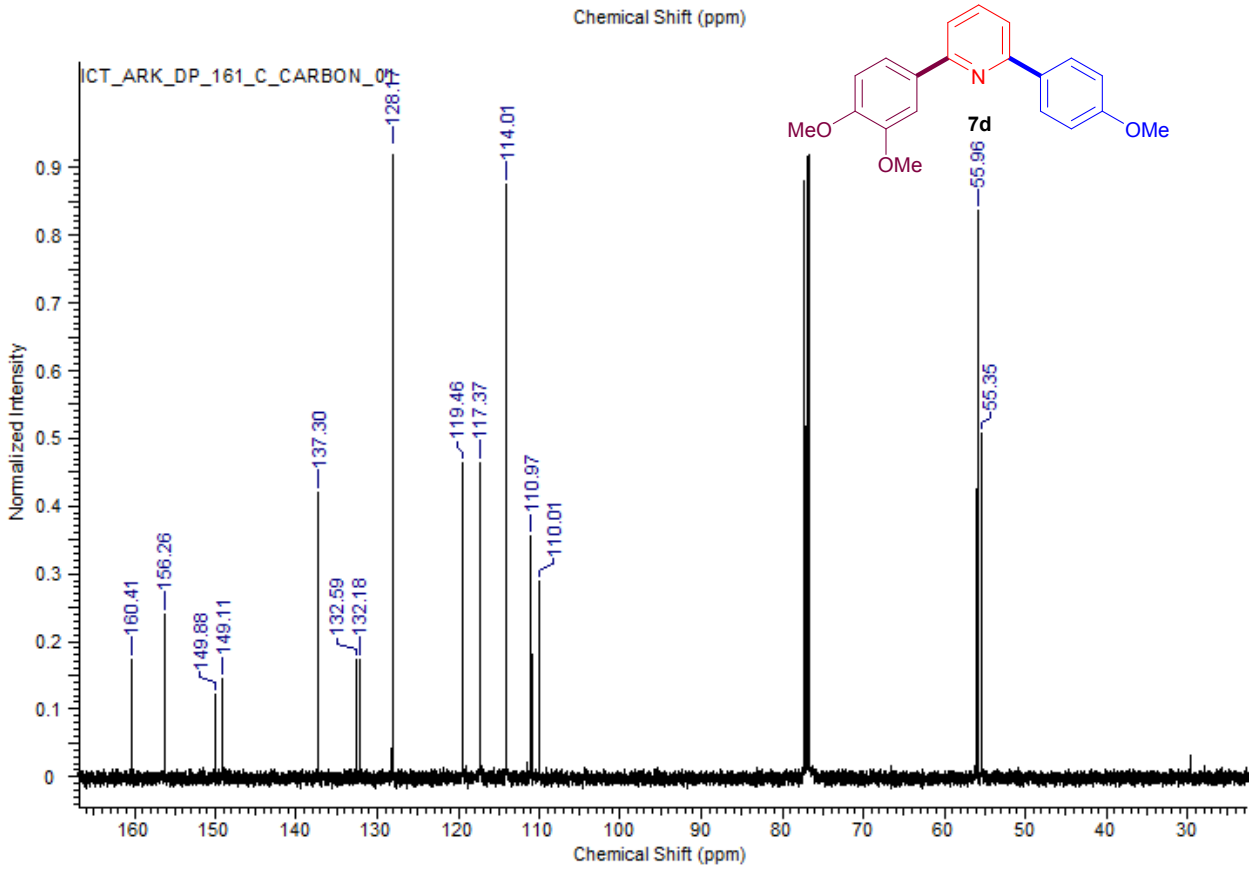

1H for DPA117.esp
Normalized Intensity
Chemical Shift (ppm)
3.92
8.63
8.33
8.18
8.00
7.84
7.68
7.55
7.09
N
OMe
7b

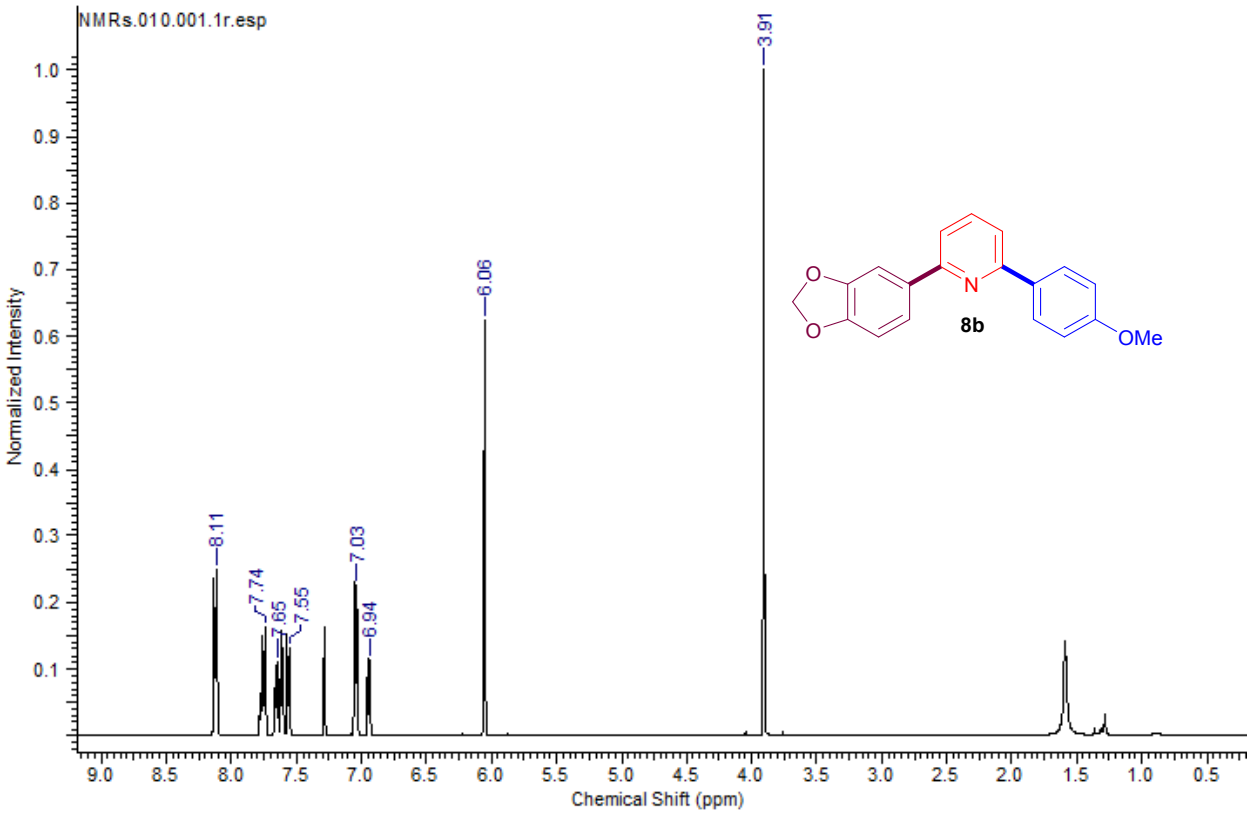

sample-3-13c
Normalized Intensity
Chemical Shift (ppm)
160.48
156.48
137.42
133.64
133.48
128.69
128.27
126.23
126.17
124.74
114.04
55.34
N
OMe
7b

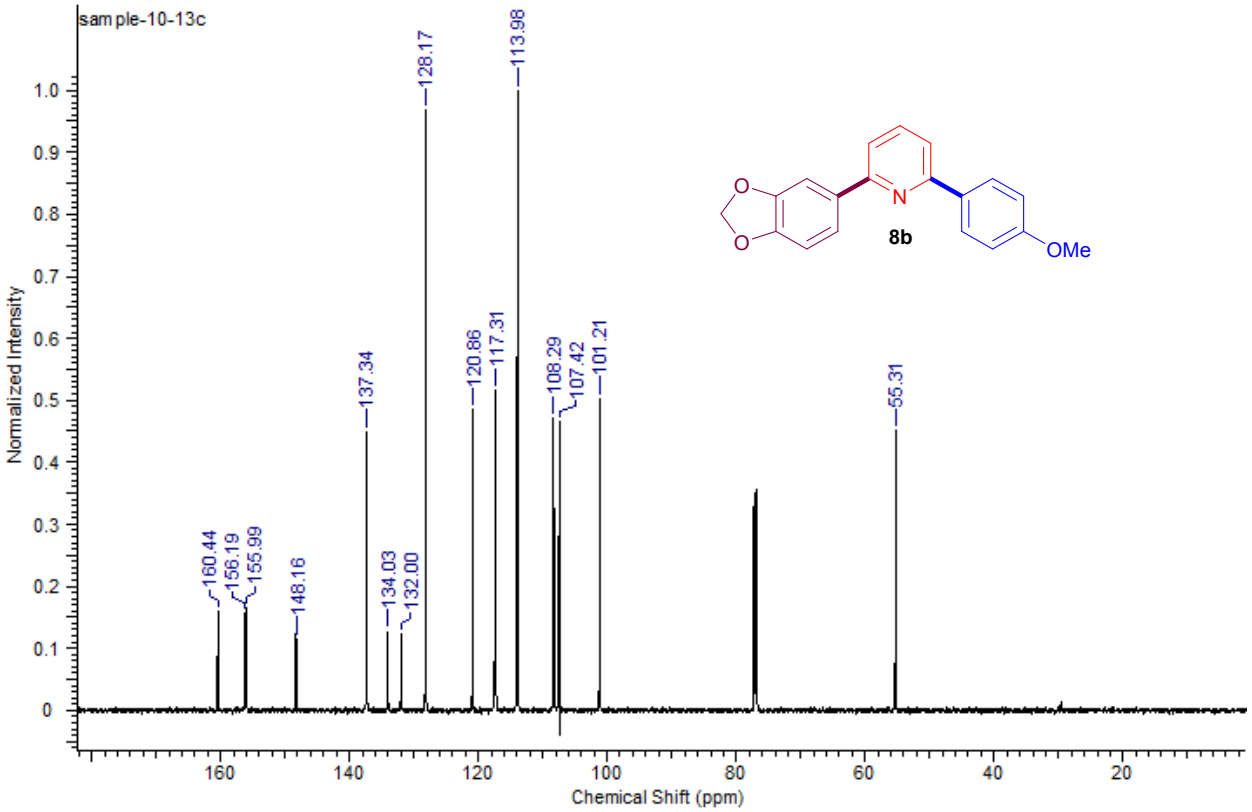

NMRs.013.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
3.95
3.91
8.14
7.79
7.66
7.43
7.04
7.00
7c
OMe
OMe

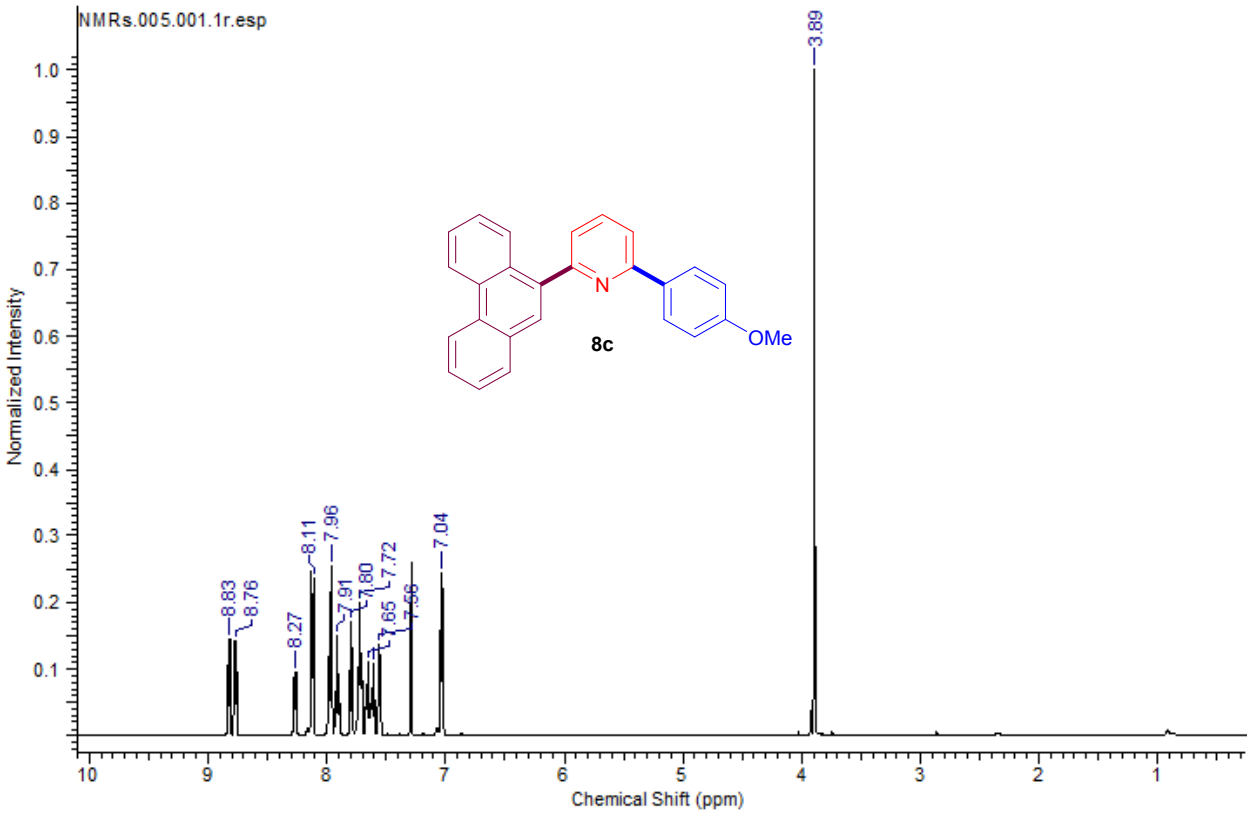

sam ple-13-13c
Normalized Intensity
Chemical Shift (ppm)
160.47
156.33
141.03
137.37
131.97
129.58
128.22
119.36
114.49
114.00
112.51
55.33
7c
OMe
OMe

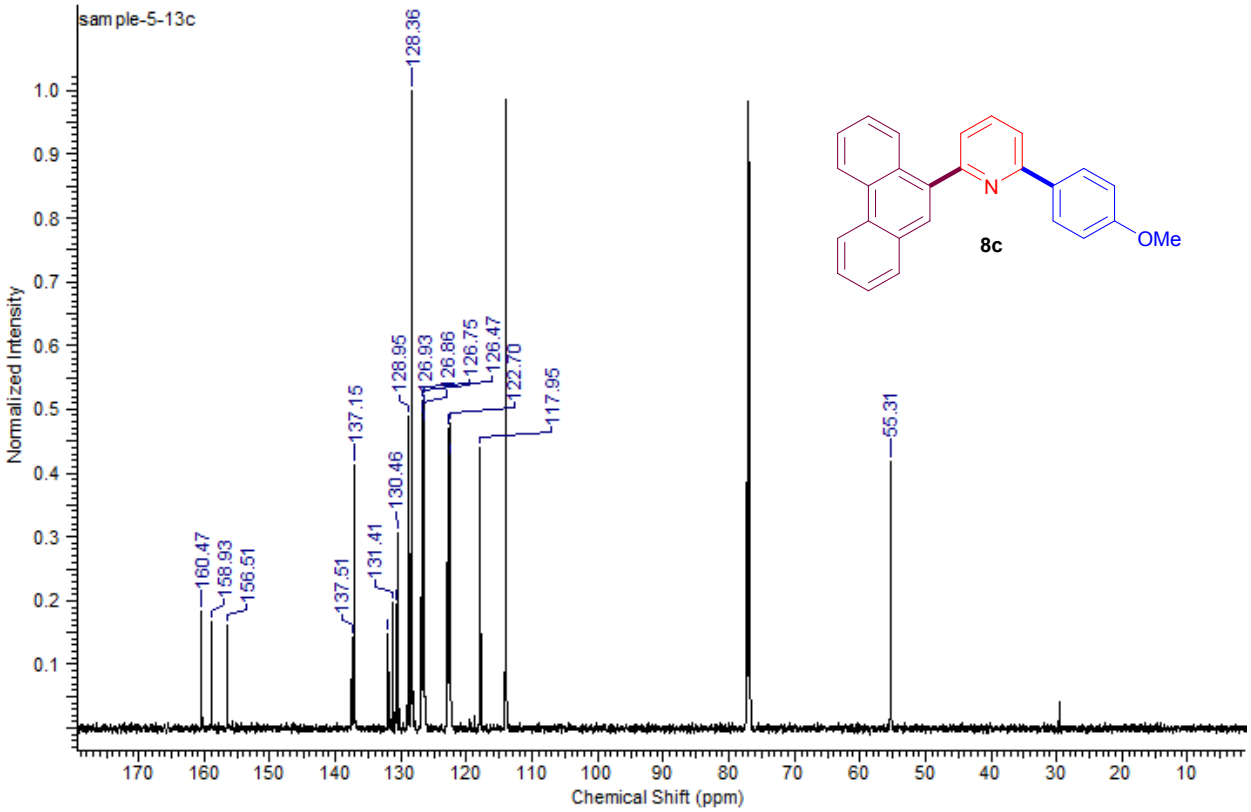

ICT_ARK_DP_DPA-161_PROTON_01
Normalized Intensity
Chemical Shift (ppm)
4.01
3.93
3.86
8.07
7.80
7.64
7.56
7.02
6.95
MeO
OMe
OMe
N
7d


ICT_ARK_DP_161_C_CARBON_01
Normalized Intensity
Chemical Shift (ppm)
160.41
156.26
149.88
149.11
137.30
132.59
132.18
128.17
119.46
117.37
114.01
110.97
110.01
55.96
55.35
MeO
OMe
OMe
N
7d


NMRs.010.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
8.11
7.74
7.65
7.55
7.03
6.94
6.06
3.91
O
O
N
8b
OMe


sample-10-13c
Normalized Intensity
Chemical Shift (ppm)
160.44
156.19
155.99
148.16
137.34
134.03
132.00
128.17
120.86
117.31
113.98
108.29
107.42
101.21
55.31
O
O
N
8b
OMe


NMRs.005.001.1r.esp
Normalized Intensity
Chemical Shift (ppm)
3.89
8.83
8.76
8.27
8.11
7.96
7.91
7.80
7.72
7.65
7.56
7.04
N
OMe
8c


sam ple-5-13c
Normalized Intensity
Chemical Shift (ppm)
128.36
128.95
126.93
126.86
126.75
126.47
122.70
117.95
137.15
130.46
131.41
137.51
160.47
158.93
156.51
55.31
N
OMe
8c

X-ray data for 4g

Table 1. Crystal data and structure refinement for **4g**.

Identification code	shelx
Empirical formula	C17 H12 Br N
Formula weight	310.19
Temperature	170(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 21/c
Unit cell dimensions	a = 28.494(6) A alpha = 90 deg. b = 7.2296(14) A beta = 92.33(3) deg. c = 6.2594(13) A gamma = 90 deg.
Volume	1288.4(5) A^3
Z, Calculated density	4, 1.599 Mg/m^3
Absorption coefficient	3.174 mm^-1
F(000)	624
Crystal size	0.482 x 0.465 x 0.241 mm
Theta range for data collection	3.543 to 29.362 deg.
Limiting indices	-32<=h<=38, -9<=k<=9, -7<=l<=8
Reflections collected / unique	12856 / 3336 [R(int) = 0.0980]
Completeness to theta = 25.000	94.7 %
Absorption correction	Numerical
Max. and min. transmission	0.7225 and 0.0907
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3336 / 0 / 172

Goodness-of-fit on F^2 1.054

Final R indices [I>2sigma(I)] R1 = 0.0378, wR2 = 0.0913

R indices (all data) R1 = 0.0527, wR2 = 0.0995

Extinction coefficient n/a

Largest diff. peak and hole 0.424 and -1.112 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **4g** U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	967(1)	4881(3)	6489(4)	26(1)
C(2)	869(1)	5238(4)	4352(4)	33(1)
C(3)	1252(1)	5552(4)	3113(4)	32(1)
C(4)	1699(1)	5509(3)	4059(4)	28(1)
C(5)	1755(1)	5137(3)	6231(3)	21(1)
C(6)	2226(1)	5083(3)	7322(3)	19(1)
C(7)	2616(1)	5875(3)	6407(3)	24(1)
C(8)	3055(1)	5827(3)	7435(4)	24(1)
C(9)	3129(1)	4998(3)	9445(3)	19(1)
C(10)	2736(1)	4192(3)	10341(3)	22(1)
C(11)	2295(1)	4231(3)	9306(3)	22(1)
C(12)	3600(1)	4994(3)	10545(3)	19(1)
C(13)	3985(1)	5831(3)	9624(4)	26(1)
C(14)	4428(1)	5797(3)	10627(4)	29(1)
C(15)	4500(1)	4922(3)	12582(4)	29(1)
C(16)	4123(1)	4123(3)	13539(4)	29(1)
C(17)	3678(1)	4165(3)	12544(4)	26(1)
Br(1)	451(1)	4436(1)	8264(1)	38(1)
N(1)	1386(1)	4815(3)	7446(3)	24(1)

Table 3. Bond lengths [A] and angles [deg] for **4g**

C(1)-N(1)	1.314(3)
C(1)-C(2)	1.380(4)
C(1)-Br(1)	1.906(2)
C(2)-C(3)	1.385(4)
C(3)-C(4)	1.382(3)
C(4)-C(5)	1.389(3)
C(5)-N(1)	1.344(3)
C(5)-C(6)	1.482(3)
C(6)-C(11)	1.393(3)
C(6)-C(7)	1.393(3)
C(7)-C(8)	1.384(3)
C(8)-C(9)	1.402(3)
C(9)-C(10)	1.398(3)
C(9)-C(12)	1.483(3)
C(10)-C(11)	1.391(3)
C(12)-C(17)	1.397(3)
C(12)-C(13)	1.399(3)
C(13)-C(14)	1.387(3)
C(14)-C(15)	1.385(4)
C(15)-C(16)	1.377(4)
C(16)-C(17)	1.392(3)
N(1)-C(1)-C(2)	126.4(2)
N(1)-C(1)-Br(1)	115.82(17)
C(2)-C(1)-Br(1)	117.74(19)

C(1)-C(2)-C(3)	116.1(2)
C(4)-C(3)-C(2)	119.4(2)
C(3)-C(4)-C(5)	119.4(2)
N(1)-C(5)-C(4)	121.7(2)
N(1)-C(5)-C(6)	116.79(19)
C(4)-C(5)-C(6)	121.5(2)
C(11)-C(6)-C(7)	117.6(2)
C(11)-C(6)-C(5)	120.94(19)
C(7)-C(6)-C(5)	121.49(19)
C(8)-C(7)-C(6)	121.28(19)
C(7)-C(8)-C(9)	121.9(2)
C(10)-C(9)-C(8)	116.4(2)
C(10)-C(9)-C(12)	122.44(19)
C(8)-C(9)-C(12)	121.16(19)
C(11)-C(10)-C(9)	121.84(19)
C(10)-C(11)-C(6)	121.0(2)
C(17)-C(12)-C(13)	117.1(2)
C(17)-C(12)-C(9)	121.6(2)
C(13)-C(12)-C(9)	121.23(19)
C(14)-C(13)-C(12)	121.4(2)
C(15)-C(14)-C(13)	120.5(2)
C(16)-C(15)-C(14)	119.1(2)
C(15)-C(16)-C(17)	120.6(2)
C(16)-C(17)-C(12)	121.3(2)
C(1)-N(1)-C(5)	116.96(19)

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **4g**

The anisotropic displacement factor exponent takes the form -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	17(1)	32(1)	29(1)	-1(1)	3(1)	-1(1)
C(2)	22(1)	44(1)	32(1)	2(1)	-6(1)	-2(1)
C(3)	28(1)	44(1)	25(1)	4(1)	-4(1)	-5(1)
C(4)	26(1)	34(1)	22(1)	0(1)	0(1)	-5(1)
C(5)	20(1)	21(1)	22(1)	-1(1)	1(1)	0(1)
C(6)	17(1)	20(1)	21(1)	-1(1)	2(1)	1(1)
C(7)	21(1)	29(1)	22(1)	5(1)	1(1)	-3(1)
C(8)	19(1)	28(1)	24(1)	5(1)	3(1)	-4(1)
C(9)	19(1)	18(1)	20(1)	0(1)	3(1)	2(1)
C(10)	18(1)	27(1)	21(1)	4(1)	3(1)	-1(1)
C(11)	18(1)	26(1)	21(1)	2(1)	4(1)	-2(1)
C(12)	17(1)	19(1)	22(1)	-1(1)	2(1)	2(1)
C(13)	18(1)	33(1)	27(1)	5(1)	3(1)	1(1)
C(14)	19(1)	36(1)	32(1)	4(1)	2(1)	-1(1)
C(15)	20(1)	34(1)	31(1)	-2(1)	-5(1)	3(1)
C(16)	28(1)	33(1)	26(1)	5(1)	-7(1)	0(1)
C(17)	25(1)	29(1)	24(1)	4(1)	1(1)	-4(1)
Br(1)	16(1)	64(1)	35(1)	1(1)	3(1)	-2(1)
N(1)	19(1)	30(1)	23(1)	-1(1)	1(1)	1(1)

X-ray data for 6d

Table 1. Crystal data and structure refinement for **6d**.

Identification code	shelx
Empirical formula	C25 H17 N
Formula weight	331.39
Temperature	170(2) K
Wavelength	0.71073 A
Crystal system, space group	Orthorhombic, P 21 21 21
Unit cell dimensions	a = 6.4697(13) A alpha = 90 deg. b = 11.008(2) A beta = 90 deg. c = 25.007(5) A gamma = 90 deg.
Volume	1781.0(6) A^3
Z, Calculated density	4, 1.236 Mg/m^3
Absorption coefficient	0.071 mm^-1
F(000)	696
Crystal size	0.447 x 0.419 x 0.389 mm
Theta range for data collection	3.252 to 26.756 deg.
Limiting indices	-8<=h<=7, -13<=k<=13, -30<=l<=31
Reflections collected / unique	14188 / 3767 [R(int) = 0.0711]
Completeness to theta = 25.242	99.7 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3767 / 0 / 235
Goodness-of-fit on F^2	0.924
Final R indices [I>2sigma(I)]	R1 = 0.0387, wR2 = 0.0800

R indices (all data)	R1 = 0.0602, wR2 = 0.0861
Extinction coefficient	n/a
Largest diff. peak and hole	0.155 and -0.158 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for dpa13.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2985(3)	8243(2)	2897(1)	32(1)
C(2)	2359(4)	8783(2)	3386(1)	38(1)
C(3)	817(4)	9637(2)	3393(1)	49(1)
C(4)	-193(4)	9978(2)	2922(2)	58(1)
C(5)	394(4)	9504(3)	2448(1)	52(1)
C(6)	2029(4)	8642(2)	2414(1)	40(1)
C(7)	2715(4)	8187(3)	1922(1)	46(1)
C(8)	4266(4)	7341(3)	1899(1)	46(1)
C(9)	5175(4)	6919(2)	2372(1)	38(1)
C(10)	4580(3)	7344(2)	2863(1)	30(1)
C(11)	5608(3)	6857(2)	3350(1)	30(1)
C(12)	7751(4)	6827(2)	3391(1)	33(1)
C(13)	8648(4)	6360(2)	3848(1)	37(1)
C(14)	7385(4)	5949(2)	4256(1)	37(1)
C(15)	5264(4)	6005(2)	4191(1)	30(1)

C(16)	3809(4)	5582(2)	4616(1)	32(1)
C(17)	3671(4)	6182(2)	5092(1)	37(1)
C(18)	2248(4)	5832(2)	5488(1)	41(1)
C(19)	972(4)	4855(2)	5407(1)	37(1)
C(20)	1097(4)	4184(2)	4926(1)	30(1)
C(21)	-156(4)	3154(2)	4832(1)	38(1)
C(22)	-18(4)	2510(2)	4370(1)	41(1)
C(23)	1383(4)	2863(2)	3974(1)	38(1)
C(24)	2618(4)	3852(2)	4044(1)	33(1)
C(25)	2521(4)	4547(2)	4523(1)	30(1)
N(1)	4370(3)	6447(2)	3744(1)	31(1)

Table 3. Bond lengths [A] and angles [deg] for dpa13.

C(1)-C(2)	1.418(3)
C(1)-C(6)	1.425(3)
C(1)-C(10)	1.433(3)
C(2)-C(3)	1.371(4)
C(3)-C(4)	1.398(4)
C(4)-C(5)	1.350(4)
C(5)-C(6)	1.424(4)
C(6)-C(7)	1.400(4)
C(7)-C(8)	1.370(4)
C(8)-C(9)	1.400(4)
C(9)-C(10)	1.368(3)
C(10)-C(11)	1.488(3)
C(11)-N(1)	1.348(3)
C(11)-C(12)	1.390(3)
C(12)-C(13)	1.382(3)
C(13)-C(14)	1.383(4)
C(14)-C(15)	1.383(3)
C(15)-N(1)	1.348(3)
C(15)-C(16)	1.494(3)
C(16)-C(17)	1.364(3)
C(16)-C(25)	1.430(3)
C(17)-C(18)	1.407(4)

C(18)-C(19)	1.371(4)
C(19)-C(20)	1.415(3)
C(20)-C(21)	1.413(3)
C(20)-C(25)	1.421(3)
C(21)-C(22)	1.358(3)
C(22)-C(23)	1.397(4)
C(23)-C(24)	1.361(3)
C(24)-C(25)	1.423(3)

C(2)-C(1)-C(6)	118.5(2)
C(2)-C(1)-C(10)	123.1(2)
C(6)-C(1)-C(10)	118.4(2)
C(3)-C(2)-C(1)	120.5(2)
C(2)-C(3)-C(4)	120.8(3)
C(5)-C(4)-C(3)	120.3(3)
C(4)-C(5)-C(6)	121.2(3)
C(7)-C(6)-C(5)	121.7(2)
C(7)-C(6)-C(1)	119.7(2)
C(5)-C(6)-C(1)	118.6(3)
C(8)-C(7)-C(6)	120.9(2)
C(7)-C(8)-C(9)	119.8(3)
C(10)-C(9)-C(8)	121.7(2)
C(9)-C(10)-C(1)	119.5(2)
C(9)-C(10)-C(11)	119.0(2)
C(1)-C(10)-C(11)	121.5(2)

N(1)-C(11)-C(12)	122.1(2)
N(1)-C(11)-C(10)	116.97(19)
C(12)-C(11)-C(10)	121.0(2)
C(13)-C(12)-C(11)	119.2(2)
C(12)-C(13)-C(14)	118.9(2)
C(13)-C(14)-C(15)	119.0(2)
N(1)-C(15)-C(14)	122.6(2)
N(1)-C(15)-C(16)	115.6(2)
C(14)-C(15)-C(16)	121.8(2)
C(17)-C(16)-C(25)	119.3(2)
C(17)-C(16)-C(15)	120.7(2)
C(25)-C(16)-C(15)	120.1(2)
C(16)-C(17)-C(18)	121.7(2)
C(19)-C(18)-C(17)	120.3(2)
C(18)-C(19)-C(20)	120.1(2)
C(21)-C(20)-C(19)	121.8(2)
C(21)-C(20)-C(25)	118.7(2)
C(19)-C(20)-C(25)	119.5(2)
C(22)-C(21)-C(20)	121.4(2)
C(21)-C(22)-C(23)	120.0(2)
C(24)-C(23)-C(22)	120.8(2)
C(23)-C(24)-C(25)	120.7(2)
C(20)-C(25)-C(24)	118.3(2)
C(20)-C(25)-C(16)	119.2(2)
C(24)-C(25)-C(16)	122.5(2)

C(15)-N(1)-C(11)	118.1(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for dpa13.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	26(1)	29(1)	41(1)	8(1)	0(1)	-5(1)
C(2)	35(1)	30(1)	48(2)	6(1)	6(1)	0(1)
C(3)	46(2)	32(1)	70(2)	1(1)	16(2)	2(1)
C(4)	39(2)	36(1)	97(3)	17(2)	7(2)	8(1)
C(5)	36(2)	44(2)	75(2)	24(2)	-8(2)	-1(1)
C(6)	31(1)	38(1)	52(2)	15(1)	-7(1)	-9(1)
C(7)	44(2)	54(2)	40(2)	13(1)	-8(1)	-16(1)
C(8)	45(2)	55(2)	38(2)	2(1)	3(1)	-15(1)
C(9)	33(1)	39(1)	42(2)	-1(1)	4(1)	-6(1)
C(10)	26(1)	30(1)	35(1)	3(1)	2(1)	-4(1)
C(11)	26(1)	26(1)	37(1)	0(1)	2(1)	0(1)
C(12)	27(1)	28(1)	43(1)	-1(1)	3(1)	0(1)
C(13)	25(1)	34(1)	52(2)	-1(1)	-2(1)	2(1)
C(14)	35(1)	30(1)	45(1)	3(1)	-7(1)	3(1)
C(15)	32(1)	24(1)	35(1)	0(1)	-3(1)	0(1)
C(16)	32(1)	28(1)	35(1)	2(1)	-4(1)	3(1)

C(17)	42(1)	32(1)	39(2)	-2(1)	-5(1)	-1(1)
C(18)	52(2)	38(1)	34(1)	-4(1)	0(1)	8(1)
C(19)	41(2)	37(1)	34(1)	2(1)	2(1)	7(1)
C(20)	29(1)	29(1)	33(1)	6(1)	-1(1)	5(1)
C(21)	32(1)	37(1)	43(2)	4(1)	2(1)	0(1)
C(22)	34(1)	39(1)	50(2)	-2(1)	-1(1)	-6(1)
C(23)	38(1)	37(1)	38(1)	-3(1)	-4(1)	-2(1)
C(24)	32(1)	33(1)	33(1)	4(1)	2(1)	1(1)
C(25)	26(1)	28(1)	35(1)	3(1)	-5(1)	4(1)
N(1)	27(1)	31(1)	36(1)	2(1)	1(1)	1(1)

Table 5. Hydrogen bonds for dpa13 [A and deg.].

Donor --- H....Acceptor	D - H	H...A	D...A	D - H...A
C(2) --H(2) ..N(1)	0.93	2.49	3.0189	116