

Supporting Information for:

Highly efficient heterogeneous copper-catalyzed decarboxylative cross-coupling of potassium polyfluorobenzoates with aryl halides leading to polyfluorobiaryls

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The Spectral Data of Polyfluorobiaryls 3a-3u:

2,3,4,5,6-Pentafluorobiphenyl (3a).¹ White solid, Mp: 110-111 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.52-7.39 (m, 5H). ¹³C NMR (100 MHz, CDCl₃): δ 144.3 (dm, *J*_F = 246.1 Hz), 140.6 (dm, *J*_F = 240.9 Hz), 137.9 (dm, *J*_F = 250.0 Hz), 130.2, 129.4, 128.8, 126.5, 116.1 (td, *J*_F = 17.3, 4.0 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -143.3 (dd, *J*_F = 22.8, 8.2 Hz, 2F), -155.8 (t, *J*_F = 20.9 Hz, 1F), -162.4 (td, *J*_F = 22.8, 8.3 Hz, 2F).

2,3,4,5,6-Pentafluoro-4'-nitrobiphenyl (3b).¹ Yellow solid, Mp: 87-88 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.26 (d, *J* = 8.8 Hz, 2H), 7.56 (d, *J* = 8.4 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 148.4, 144.3 (dm, *J*_F = 248.4 Hz), 141.5 (dm, *J*_F = 254.8 Hz), 138.2 (dm, *J*_F = 253.0 Hz), 133.1, 131.5, 124.0, 114.0 (td, *J*_F = 16.7, 4.2 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -142.7 (dd, *J*_F = 22.3, 8.0 Hz, 2F), -152.9 (t, *J*_F = 20.8 Hz, 1F), -161.2 (td, *J*_F = 21.2, 7.1 Hz, 2F).

2',3',4',5',6'-Pentafluorobiphenyl-4-carbonitrile (3c).² Yellow solid, Mp: 124-125 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.81 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 2H).

^{13}C NMR (100 MHz, CDCl_3): δ 144.2 (dm, $J_{\text{F}} = 249.9$ Hz), 141.3 (dm, $J_{\text{F}} = 254.6$ Hz), 138.1 (dm, $J_{\text{F}} = 250.7$ Hz), 132.6, 131.3, 131.1, 118.2, 114.3 (td, $J_{\text{F}} = 16.6, 4.1$ Hz), 113.5. ^{19}F NMR (376 MHz, CDCl_3): δ -142.8 (dd, $J = 22.2, 7.9$ Hz, 2F), -153.1 (t, $J = 20.9$ Hz, 1F), -161.1 (td, $J = 21.5, 7.2$ Hz, 2F).

4'-Chloro-2,3,4,5,6-pentafluorobiphenyl (3d).³ White solid, Mp: 84-85 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.46 (d, $J = 8.4$ Hz, 2H), 7.36 (d, $J = 8.4$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 144.2 (dm, $J_{\text{F}} = 246.3$ Hz), 140.8 (dm, $J_{\text{F}} = 255.0$ Hz), 138.0 (dm, $J_{\text{F}} = 249.6$ Hz), 135.8, 131.6, 129.2, 124.9, 115.0 (td, $J_{\text{F}} = 17.0, 4.1$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -143.2 (dd, $J_{\text{F}} = 22.5, 7.9$ Hz, 2F), -155.0 (t, $J_{\text{F}} = 20.9$ Hz, 1F), -162.0 (td, $J_{\text{F}} = 22.6, 8.3$ Hz, 2F).

2,3,4,4',5,6-Hexafluorobiphenyl (3e).³ White solid, Mp: 114-115 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.45-7.37 (m, 2H), 7.22-7.14 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 163.3 (d, $J_{\text{F}} = 249.9$ Hz), 144.3 (dm, $J_{\text{F}} = 246.1$ Hz), 140.6 (dm, $J_{\text{F}} = 252.3$ Hz), 137.9 (dm, $J_{\text{F}} = 249.7$ Hz), 132.2 (d, $J_{\text{F}} = 8.4$ Hz), 122.4, 116.1 (d, $J_{\text{F}} = 22.0$ Hz), 115.1 (td, $J_{\text{F}} = 17.2, 4.0$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -111.4 (s, 1F), -143.4 (dd, $J_{\text{F}} = 22.7, 8.2$ Hz, 2F), -155.4 (t, $J_{\text{F}} = 20.9$ Hz, 1F), -162.2 (td, $J_{\text{F}} = 22.7, 8.2$ Hz, 2F).

2,3,4,5,6-Pentafluoro-4'-(trifluoromethyl)biphenyl (3f).⁴ White solid, Mp: 57-58 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.76 (d, $J = 8.4$ Hz, 2H), 7.57 (d, $J = 8.0$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 144.3 (dm, $J_{\text{F}} = 247.5$ Hz), 141.1 (dm, $J_{\text{F}} = 253.8$ Hz), 138.1 (dm, $J_{\text{F}} = 250.0$ Hz), 131.6 (q, $J_{\text{F}} = 32.9$ Hz), 130.7, 130.2, 125.8 (q, $J_{\text{F}} = 3.8$ Hz), 123.9 (q, $J_{\text{F}} = 272.4$ Hz), 114.7 (td, $J_{\text{F}} = 17.1, 4.0$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -63.1 (s, 3F), -143.0 (dd, $J_{\text{F}} = 22.6, 8.3$ Hz, 2F), -153.9 (t, $J_{\text{F}} = 20.8$ Hz, 1F), -161.6 (td, $J_{\text{F}} = 22.9, 7.9$ Hz, 2F).

2,3,4,5,6-Pentafluoro-3'-nitrobiphenyl (3g).³ Yellow solid, Mp: 107-108 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.37-8.33 (m, 2H), 7.81 (d, $J = 8.0$ Hz, 1H), 7.74 (t, $J = 8.4$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 148.5, 144.3 (dm, $J_{\text{F}} = 248.0$ Hz), 141.4

(dm, $J_F = 254.1$ Hz), 138.1 (dm, $J_F = 254.1$ Hz), 136.2, 130.0, 128.2, 125.3, 124.3, 113.8 (td, $J_F = 16.8, 3.8$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -143.1 (dd, $J_F = 22.5, 8.3$ Hz, 2F), -153.2 (t, $J_F = 20.8$ Hz, 1F), -161.2 (td, $J_F = 21.4, 7.2$ Hz, 2F).

2,3,4,5,6-Pentafluoro-4'-methylbiphenyl (3h).⁴ White solid, Mp: 117-118 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.29 (s, 4H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 144.3 (dm, $J_F = 245.7$ Hz), 140.2 (dm, $J_F = 250.5$ Hz), 139.5, 138.0 (dm, $J_F = 249.8$ Hz), 130.0, 129.5, 123.5, 116.1 (td, $J_F = 17.3, 4.1$ Hz), 21.4. ^{19}F NMR (376 MHz, CDCl_3): δ -143.4 (dd, $J_F = 22.9, 8.2$ Hz, 2F), -156.3 (t, $J_F = 20.8$ Hz, 1F), -162.6 (td, $J_F = 21.5, 7.8$ Hz, 2F).

2,3,4,5,6-Pentafluoro-4'-methoxybiphenyl (3i).⁴ White solid, Mp: 122-123 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.25 (d, $J = 8.4$ Hz, 2H), 6.90 (d, $J = 8.8$ Hz, 2H), 3.74 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 160.5, 144.3 (dm, $J_F = 245.1$ Hz), 140.1 (dm, $J_F = 251.5$ Hz), 138.0 (dm, $J_F = 248.7$ Hz), 131.5, 118.5, 115.9 (td, $J_F = 17.1, 3.9$ Hz), 114.3, 55.4. ^{19}F NMR (376 MHz, CDCl_3): δ -143.8 (dd, $J_F = 22.9, 8.1$ Hz, 2F), -156.8 (t, $J_F = 20.9$ Hz, 1F), -162.8 (td, $J_F = 22.9, 8.1$ Hz, 2F).

2,3,4,5,6-Pentafluoro-3'-methylbiphenyl (3j).¹ White solid, Mp: 44-45 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.36 (t, $J = 7.4$ Hz, 1H), 7.26 (d, $J = 7.6$ Hz, 1H), 7.23-7.17 (m, 2H), 2.40 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 144.3 (dm, $J_F = 245.9$ Hz), 140.4 (dm, $J_F = 250.9$ Hz), 138.6, 137.9 (dm, $J_F = 247.8$ Hz), 130.8, 130.2, 128.7, 127.3, 126.4, 116.2 (td, $J_F = 17.3, 4.0$ Hz), 21.4. ^{19}F NMR (376 MHz, CDCl_3): δ -143.1 (dd, $J_F = 22.9, 8.2$ Hz, 2F), -156.0 (t, $J_F = 20.9$ Hz, 1F), -162.4 – -162.6 (m, 2F).

2'-Chloro-2,3,4,5,6-pentafluorobiphenyl (3k).³ Colorless liquid. ^1H NMR (400 MHz, CDCl_3): δ 7.54 (dd, $J = 7.8, 1.0$ Hz, 1H), 7.45-7.34 (m, 2H), 7.30 (dd, $J = 7.6, 1.6$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 144.4 (dm, $J_F = 246.8$ Hz), 141.4 (dm, $J_F = 252.9$ Hz), 137.8 (dm, $J_F = 249.6$ Hz), 134.6, 132.2, 131.1, 130.1, 127.1, 125.9, 113.7 (td, $J_F = 19.1, 4.1$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -139.9 (dd, $J_F = 23.0, 8.0$ Hz, 2F), -154.3 (t, $J_F = 20.8$ Hz, 1F), -162.3 (td, $J_F = 22.6, 8.3$ Hz, 2F).

Methyl 2',3',4',5',6'-pentafluorobiphenyl-2-carboxylate (3l).³ Pale yellow solid, Mp: 59-60 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.05 (d, *J* = 7.6 Hz, 1H), 7.53 (t, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.2 Hz, 1H), 7.22 (d, *J* = 7.6 Hz, 1H), 3.68 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 166.3, 144.1 (dm, *J*_F = 244.3 Hz), 140.8 (dm, *J*_F = 251.6 Hz), 137.6 (dm, *J*_F = 248.6 Hz), 132.6, 132.2, 131.3, 130.4, 129.8, 127.6, 116.0 (td, *J*_F = 19.0, 4.0 Hz), 52.3. ¹⁹F NMR (376 MHz, CDCl₃): δ -142.0 (dd, *J*_F = 22.7, 7.5 Hz, 2F), -156.0 (t, *J*_F = 20.8 Hz, 1F), -163.2 – -163.4 (m, 2F).

2,3,4,5,6-Pentafluoro-2'-nitro-1,1'-biphenyl (3m).⁵ Yellow solid, Mp: 83-84 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.13 (d, *J* = 8.0 Hz, 1H), 7.68 (t, *J* = 7.2 Hz, 1H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.36 (d, *J* = 7.6 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 148.6, 144.0 (dm, *J*_F = 250.4 Hz), 141.5 (dm, *J*_F = 254.5 Hz), 137.9 (dm, *J*_F = 252.3 Hz), 133.9, 133.2, 131.1, 125.6, 121.6, 112.9 (td, *J*_F = 18.6, 4.3 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -141.4 (dd, *J*_F = 21.9, 7.1 Hz, 2F), -153.8 (t, *J*_F = 20.8 Hz, 1F), -161.8 (td, *J*_F = 21.6, 7.0 Hz, 2F).

2,3,4,5,6-Pentafluoro-2'-methylbiphenyl (3n).² Colorless liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.41-7.33 (m, 2H), 7.31-7.26 (m, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 2.18 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 144.1 (dm, *J*_F = 246.6 Hz), 140.7 (dm, *J*_F = 252.2 Hz), 137.7 (dm, *J*_F = 248.8 Hz), 137.4, 130.6, 130.5, 129.7, 126.0, 125.9, 115.5 (td, *J*_F = 20.2, 4.2 Hz), 19.7. ¹⁹F NMR (376 MHz, CDCl₃): δ -140.6 (dd, *J*_F = 23.1, 8.2 Hz, 2F), -155.5 (t, *J*_F = 20.8 Hz, 1F), -162.3 (td, *J*_F = 23.1, 8.4 Hz, 2F).

2,3,4,5,6-Pentafluoro-2'-methoxybiphenyl (3o).² Colorless liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.44 (t, *J* = 8.6 Hz, 1H), 7.22 (d, *J* = 6.8 Hz, 1H), 7.07-7.00 (m, 2H), 3.80 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 157.3, 144.6 (dm, *J*_F = 245.6 Hz), 140.6 (dm, *J*_F = 251.2 Hz), 137.7 (dm, *J*_F = 248.2 Hz), 131.8, 131.2, 120.7, 115.4, 112.9 (td, *J*_F = 19.3, 4.0 Hz), 111.4, 55.7. ¹⁹F NMR (376 MHz, CDCl₃): δ -140.3 (dd, *J*_F = 22.9, 7.9 Hz, 2F), -156.3 (t, *J*_F = 20.8 Hz, 1F), -163.4 (td, *J*_F = 23.0, 8.1 Hz, 2F).

3-(Pentafluorophenyl)pyridine (3p).⁶ Pale yellow solid, Mp: 61-62 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.62 (s, 2H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.40-7.36 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 150.5, 150.4, 144.3 (dm, *J*_F = 245.7 Hz), 141.1 (dm, *J*_F = 253.2 Hz), 138.0 (dm, *J*_F = 250.6 Hz), 137.6, 123.7, 123.1, 112.7 (td, *J*_F = 17.0, 4.2 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -142.9 (dd, *J*_F = 22.2, 7.8 Hz, 2F), -153.5 (t, *J*_F = 20.9 Hz, 1F), -161.2 (td, *J*_F = 22.3, 8.1 Hz, 2F).

2-(Pentafluorophenyl)pyridine (3q).⁶ Pale yellow solid, Mp: 60-61 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.68 (d, *J* = 4.8 Hz, 1H), 7.77-7.72 (m, 1H), 7.39 (d, *J* = 8.0 Hz, 1H), 7.32-7.27 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 150.3, 147.0, 144.8 (dm, *J*_F = 249.0 Hz), 141.4 (dm, *J*_F = 253.5 Hz), 137.8 (dm, *J*_F = 250.3 Hz), 136.8, 126.0, 123.9, 115.6 (td, *J*_F = 16.6, 3.9 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -143.3 (dd, *J*_F = 22.7, 8.5 Hz, 2F), -154.0 (t, *J*_F = 20.8 Hz, 1F), -162.0 (td, *J*_F = 22.1, 8.2 Hz, 2F).

4-(Pentafluorophenyl)pyridine (3r).⁶ White solid, Mp: 92-93 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.77 (d, *J* = 5.0 Hz, 2H), 7.38 (d, *J* = 5.0 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 150.3, 144.1 (dm, *J*_F = 250.0 Hz), 141.3 (dm, *J*_F = 254.6 Hz), 138.0 (dm, *J*_F = 251.9 Hz), 134.7, 124.6, 113.3 (td, *J*_F = 16.6, 4.3 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -142.6 (dd, *J*_F = 22.0, 7.9 Hz, 2F), -152.8 (td, *J*_F = 21.0, 1.5 Hz, 1F), -160.9 – -161.1 (m, 2F).

3-(Pentafluorophenyl)thiophene (3s).⁶ Yellow liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.64 (dd, *J* = 2.8, 1.2 Hz, 1H), 7.44 (dd, *J* = 5.0, 3.0 Hz, 1H), 7.37-7.32 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 144.3 (dm, *J*_F = 247.1 Hz), 140.1 (dm, *J*_F = 252.3 Hz), 138.1 (dm, *J*_F = 248.7 Hz), 128.3 (t, *J*_F = 3.6 Hz), 127.2 (t, *J*_F = 4.0 Hz), 125.8, 125.7, 111.3 (td, *J*_F = 16.3, 4.2 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -142.0 (dd, *J*_F = 22.5, 7.8 Hz, 2F), -156.4 (t, *J*_F = 21.0 Hz, 1F), -162.5 (td, *J*_F = 22.6, 7.9 Hz, 2F).

1-(Pentafluorophenyl)naphthalene (3t).⁶ White solid, Mp: 91-92 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.95 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.56-7.40 (m, 5H). ¹³C NMR (100 MHz, CDCl₃): δ 144.7 (dm, *J*_F = 246.0 Hz), 141.1 (dm, *J*_F = 252.4 Hz),

137.9 (dm, $J_F = 250.9$ Hz), 133.8, 131.7, 130.2, 129.1, 128.7, 127.2, 126.5, 125.3, 124.6, 123.9, 114.5 (td, $J_F = 19.5, 3.8$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -139.4 (dd, $J_F = 23.0, 8.2$ Hz, 2F), -154.7 (t, $J_F = 20.8$ Hz, 1F), -162.0 (td, $J_F = 22.9, 8.3$ Hz, 2F).

Methyl 2',3',4',5',6'-pentafluorobiphenyl-4-carboxylate (3u).² White solid, Mp: 112-113 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.04 (d, $J = 8.4$ Hz, 2H), 7.40 (d, $J = 8.4$, 2H), 3.85 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 166.5, 144.3 (dm, $J_F = 247.4$ Hz), 141.0 (dm, $J_F = 253.5$ Hz), 138.1 (dm, $J_F = 251.5$ Hz), 137.9, 131.1, 130.4, 130.0, 115.2 (td, $J_F = 16.9, 3.9$ Hz), 52.4. ^{19}F NMR (376 MHz, CDCl_3): δ -143.0 (dd, $J_F = 22.5, 8.1$ Hz, 2F), -154.4 (t, $J_F = 20.8$ Hz, 1F), -161.9 (td, $J_F = 21.4, 7.6$ Hz, 2F).

2,3,4,5,6-Pentafluoro-3',5'-dimethylbiphenyl (3v).³ White solid, Mp: 82-83 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.10 (s, 1H), 7.02 (s, 2H), 2.38 (s, 6H). ^{13}C NMR (100 MHz, CDCl_3): δ 144.3 (dm, $J_F = 245.7$ Hz), 140.3 (dm, $J_F = 245.1$ Hz), 138.4, 137.9 (dm, $J_F = 248.2$ Hz), 131.1, 127.9, 126.2, 116.4 (td, $J_F = 17.7, 4.3$ Hz), 21.3. ^{19}F NMR (376 MHz, CDCl_3): δ -143.0 (dd, $J_F = 23.0, 8.3$ Hz, 2F), -156.3 (t, $J_F = 20.9$ Hz, 1F), -162.6 (td, $J_F = 22.5, 8.2$ Hz, 2F).

2,6-Difluoro-4'-nitrobiphenyl (5a).⁷ Pale yellow solid, Mp: 117-118 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.22 (d, $J = 8.4$ Hz, 2H), 7.57 (d, $J = 8.4$ Hz, 2H), 7.35-7.24 (m, 1H), 6.95 (t, $J = 8.0$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 159.8 (dd, $J_F = 250.4, 6.5$ Hz), 147.6, 136.0, 131.4 (t, $J_F = 2.2$ Hz), 130.5 (t, $J_F = 10.4$ Hz), 123.5, 116.4 (t, $J_F = 18.1$ Hz), 112.0 (dd, $J_F = 19.5, 6.6$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -114.1 (s, 2F).

2,6-Difluoro-4'-methoxybiphenyl (5b).³ White solid, Mp: 57-58 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.40 (d, $J = 8.8$ Hz, 2H), 7.25-7.17 (m, 1H), 7.00-6.91 (m, 4H), 3.82 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 160.2 (dd, $J_F = 247.8, 7.2$ Hz), 159.5, 131.6 (t, $J_F = 1.9$ Hz), 128.4 (t, $J_F = 10.4$ Hz), 121.3, 118.2 (t, $J_F = 18.6$ Hz), 113.8, 111.6 (dd, $J_F = 19.3, 7.4$ Hz), 55.3. ^{19}F NMR (376 MHz, CDCl_3): δ -114.7 (s, 2F).

2,6-Difluoro-3'-nitro-1,1'-biphenyl (5c).⁸ Pale yellow solid, Mp: 119-120 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.36 (s, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 7.6 Hz, 1H), 7.64 (t, *J* = 8.0 Hz, 1H), 7.41-7.33 (m, 1H), 7.08-6.99 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 159.9 (dd, *J*_F = 248.5, 6.5 Hz), 148.3, 136.4 (t, *J*_F = 2.0 Hz), 130.9, 130.3 (t, *J*_F = 10.4 Hz), 129.3, 125.4 (t, *J*_F = 2.2 Hz), 123.2, 116.2 (t, *J*_F = 18.0 Hz), 112.0 (dd, *J*_F = 19.3, 6.6 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -114.6 (s, 2F).

2,6-Difluoro-2'-nitro-1,1'-biphenyl (5d).⁹ Pale yellow solid, Mp: 108-109 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.07 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.65-7.60 (m, 1H), 7.54-7.49 (m, 1H), 7.40 (d, *J* = 7.6 Hz, 1H), 7.32-7.24 (m, 1H), 6.95-6.88 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 159.6 (dd, *J*_F = 248.5, 6.6 Hz), 149.0, 133.2, 133.0, 130.2 (t, *J*_F = 10.2 Hz), 129.7, 124.9, 124.4, 115.0 (t, *J*_F = 19.9 Hz), 111.6 (dd, *J*_F = 19.0, 6.3 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -113.5 (s, 2F).

2,6-Difluoro-2'-methoxybiphenyl (5e).³ Colorless liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.41-7.35 (m, 1H), 7.29-7.22 (m, 2H), 7.04-6.97 (m, 2H), 6.96-6.90 (m, 2H), 3.77 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 160.6 (dd, *J*_F = 248.2, 7.5 Hz), 157.4, 131.9, 130.1, 128.9 (t, *J*_F = 10.2 Hz), 120.4, 118.4, 115.4 (t, *J*_F = 20.7 Hz), 111.2 (dd, *J*_F = 19.1, 7.0 Hz), 111.1, 55.7. ¹⁹F NMR (376 MHz, CDCl₃): δ -112.1 (s, 2F).

2,3,6-Trifluoro-4'-nitro-biphenyl (5f). Pale yellow solid, Mp: 125-126 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.33 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H), 7.28-7.19 (m, 1H), 7.03-6.96 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 155.0 (ddd, *J*_F = 247.0, 4.3, 2.8 Hz), 148.0, 147.7 (dm, *J*_F = 248.2 Hz), 147.5 (dm, *J*_F = 244.6 Hz), 135.1 (d, *J*_F = 1.9 Hz), 131.4 (t, *J*_F = 2.2 Hz), 123.6, 118.2 (dd, *J*_F = 20.1, 14.5 Hz), 117.4 (qd, *J*_F = 10.0, 1.5 Hz), 111.5 (ddd, *J*_F = 25.1, 6.6, 4.3 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -119.3 (dd, *J*_F = 15.2, 1.9 Hz, 1F), -137.3 (dd, *J*_F = 21.1, 1.9 Hz, 1F), -141.0 (dd, *J*_F = 21.0, 15.4 Hz, 1F). HRMS calcd for C₁₂H₆F₃NO₂⁺ [M⁺]: 253.0351, found 253.0348.

2',3',6'-Trifluorobiphenyl-4-carbonitrile (5g).³ White solid, Mp: 152-153 °C. ¹H

NMR (400 MHz, CDCl₃): δ 7.67 (d, J = 8.4 Hz, 2H), 7.50 (d, J = 8.0 Hz, 2H), 7.18-7.07 (m, 1H), 6.92-6.85 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 155.0 (ddd, J_F = 245.2, 4.4, 2.8 Hz), 147.7 (dm, J_F = 249.9 Hz), 147.5 (dm, J_F = 244.4 Hz), 133.2 (d, J_F = 1.8 Hz), 132.2, 131.1 (t, J_F = 2.2 Hz), 118.5 (dd, J_F = 20.2, 14.3 Hz), 118.4, 117.2 (qd, J_F = 9.7, 1.4 Hz), 112.7, 111.4 (ddd, J_F = 24.9, 6.7, 4.3 Hz). ¹⁹F NMR (376 MHz, CDCl₃): δ -119.5 (dd, J_F = 15.2, 2.0 Hz, 1F), -137.5 (dd, J_F = 21.1, 2.0 Hz, 1F), -141.1 (dd, J_F = 21.1, 15.2 Hz, 1F).

2,3,6-Trifluoro-4'-methoxybiphenyl (5h).³ White solid, Mp: 54-55 °C. ¹H NMR (400 MHz, CDCl₃): δ 7.39 (d, J = 8.4 Hz, 2H), 7.09-7.02 (m, 1H), 6.99 (d, J = 8.8 Hz, 2H), 6.90-6.84 (m, 1H), 3.82 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 159.9, 155.4 (ddd, J_F = 244.2, 4.7, 2.6 Hz), 147.9 (dq, J_F = 249.0, 7.3 Hz), 147.6 (ddd, J_F = 244.3, 13.8, 3.6 Hz), 131.5, 120.5 (d, J_F = 1.7 Hz), 120.0 (dd, J_F = 20.6, 15.1 Hz), 115.1 (qd, J_F = 9.8, 1.0 Hz), 114.0, 110.8 (ddd, J_F = 25.7, 6.8, 4.2 Hz), 55.3. ¹⁹F NMR (376 MHz, CDCl₃): δ -120.0 (dd, J_F = 15.0, 3.8 Hz, 1F), -138.2 (ddd, J_F = 21.7, 3.9, 1.2 Hz, 1F), -142.1 (dd, J_F = 21.5, 15.0 Hz, 1F).

2,3,6-Trifluoro-2'-methoxybiphenyl (5i). Colorless liquid. ¹H NMR (400 MHz, CDCl₃): δ 7.43-7.36 (m, 1H), 7.27-7.23 (m, 1H), 7.14-6.98 (m, 3H), 6.89-6.82 (m, 1H), 3.78 (s, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 157.2, 155.8 (ddd, J_F = 244.6, 5.2, 2.5 Hz), 148.3 (dq, J_F = 249.7, 7.7 Hz), 147.3 (ddd, J_F = 243.9, 13.7, 3.6 Hz), 131.8, 130.6, 120.5, 117.4 (d, J_F = 1.7 Hz), 117.3 (dd, J_F = 22.7, 17.1 Hz), 115.8 (qd, J_F = 9.7, 1.0 Hz), 111.3, 110.4 (ddd, J_F = 25.3, 6.7, 4.2 Hz), 55.7. ¹⁹F NMR (376 MHz, CDCl₃): δ -117.4 (dd, J_F = 14.8, 3.0 Hz, 1F), -134.7 (dd, J_F = 21.8, 2.9 Hz, 1F), -142.9 (dd, J_F = 21.8, 14.8 Hz, 1F). HRMS calcd for C₁₃H₉F₃O⁺ [M⁺]: 238.0605, found 238.0610.

3-(2',3',6'-Trifluorophenyl)pyridine (5j). Pale yellow solid, Mp: 39-40 °C. ¹H NMR (400 MHz, CDCl₃): δ 8.73 (s, 1H), 8.66 (dd, J = 5.0, 1.4 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.42 (dd, J = 8.0, 4.8 Hz, 1H), 7.23-7.14 (m, 1H), 7.01-6.94 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 155.3 (ddd, J_F = 244.8, 4.6, 2.8 Hz), 150.7 (t, J_F = 2.6 Hz),

149.8, 148.0 (dq, $J_F = 249.8, 7.2$ Hz), 147.6 (ddd, $J_F = 244.1, 13.3, 3.7$ Hz), 137.6 (t, $J_F = 2.0$ Hz), 124.9, 123.4, 116.9 (dd, $J_F = 19.9, 15.7$ Hz), 116.8 (qd, $J_F = 9.7, 1.4$ Hz), 111.3 (ddd, $J_F = 25.0, 6.6, 4.2$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -119.8 (dd, $J_F = 15.5, 2.6$ Hz, 1F), -137.8 (d, $J_F = 21.2$ Hz, 1F), -141.4 (dd, $J_F = 21.2, 15.3$ Hz, 1F). HRMS calcd for $\text{C}_{11}\text{H}_6\text{F}_3\text{N}^+$ [M^+]: 209.0452, found 209.0453.

2,3,5,6-Tetrafluoro-4'-nitro-1,1'-biphenyl (5k).¹⁰ Pale yellow solid, Mp: 179-180 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.36 (d, $J = 8.4$ Hz, 2H), 7.68 (d, $J = 8.4$ Hz, 2H), 7.23-7.12 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 148.3, 146.4 (dm, $J_F = 245.4$ Hz), 143.6 (dm, $J_F = 245.1$ Hz), 134.0, 131.3, 123.8, 119.3 (t, $J_F = 16.1$ Hz), 106.5 (t, $J_F = 22.5$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -137.9 (dd, $J_F = 21.8, 12.8$ Hz, 2F), -143.2 (dd, $J_F = 21.8, 12.8$ Hz, 2F).

2',3',5',6'-Tetrafluorobiphenyl-4-carbonitrile (5l).¹¹ White solid, Mp: 172-173 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.80 (d, $J = 8.4$ Hz, 2H), 7.60 (d, $J = 8.0$ Hz, 2H), 7.20-7.11 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3): δ 146.4 (dm, $J_F = 245.2$ Hz), 143.6 (dm, $J_F = 244.9$ Hz), 132.4, 132.2 (t, $J_F = 2.2$ Hz), 131.1 (t, $J_F = 2.3$ Hz), 119.6 (t, $J_F = 16.1$ Hz), 118.2, 113.3, 106.3 (t, $J_F = 22.5$ Hz). ^{19}F NMR (376 MHz, CDCl_3): δ -138.0 (dd, $J_F = 22.0, 13.0$ Hz, 2F), -143.5 (dd, $J_F = 22.0, 13.0$ Hz, 2F).

2,3,5,6-Tetrafluoro-4'-methylbiphenyl (5m).⁴ White solid, Mp: 89-90 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.35 (d, $J = 8.0$ Hz, 2H), 7.29 (d, $J = 8.0$ Hz, 2H), 7.07-6.97 (m, 1H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 146.3 (dm, $J_F = 244.7$ Hz), 143.8 (dm, $J_F = 245.2$ Hz), 139.3, 130.0 (t, $J_F = 2.0$ Hz), 129.4, 124.5 (t, $J_F = 2.2$ Hz), 121.6 (t, $J_F = 16.6$ Hz), 104.6 (t, $J_F = 22.6$ Hz), 21.4. ^{19}F NMR (376 MHz, CDCl_3): δ -139.4 (dd, $J_F = 22.4, 12.8$ Hz, 2F), -144.0 (dd, $J_F = 22.4, 12.8$ Hz, 2F).

2,3,5,6-Tetrafluoro-4'-methoxybiphenyl (5n).³ White solid, Mp: 107-108 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.39 (d, $J = 8.8$ Hz, 2H), 7.04-6.97 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 160.3, 146.3 (dm, $J_F = 244.7$ Hz), 143.9 (dm, $J_F = 248.1$ Hz), 131.5 (t, $J_F = 2.2$ Hz), 121.3 (t, $J_F = 16.5$ Hz), 119.6 (t, $J_F = 2.2$ Hz), 114.2, 104.3 (t,

$J_F = 22.7$ Hz), 55.3. ^{19}F NMR (376 MHz, CDCl_3): δ -139.5 (dd, $J_F = 22.4, 12.6$ Hz, 2F), -144.3 (dd, $J_F = 22.4, 12.7$ Hz, 2F).

2,3,5,6-Tetrafluoro-4-methyl-4'-nitrobiphenyl (5o).³ Pale yellow solid, Mp: 140-141 °C. ^1H NMR (400 MHz, CDCl_3): δ 8.24 (d, $J = 8.8$ Hz, 2H), 7.57 (d, $J = 8.4$ Hz, 2H), 2.26 (s, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 148.1, 145.5 (dm, $J_F = 244.0$ Hz), 143.5 (dm, $J_F = 246.5$ Hz), 134.5, 131.4 (t, $J_F = 2.3$ Hz), 123.8, 117.1 (t, $J_F = 19.1$ Hz), 115.8 (t, $J_F = 16.1$ Hz), 7.8. ^{19}F NMR (376 MHz, CDCl_3): δ -142.8 (dd, $J_F = 21.9, 12.6$ Hz, 2F), -145.1 (dd, $J_F = 21.7, 12.4$ Hz, 2F).

2,3,5,6-Tetrafluoro-4'-methoxy-4-methylbiphenyl (5p).¹² White solid, Mp: 117-118 °C. ^1H NMR (400 MHz, CDCl_3): δ 7.37 (d, $J = 8.8$ Hz, 2H), 6.98 (d, $J = 8.8$ Hz, 2H), 3.83 (s, 3H), 2.28 (t, $J = 1.8$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 160.1, 145.4 (dm, $J_F = 242.2$ Hz), 143.7 (dm, $J_F = 243.3$ Hz), 131.5 (t, $J_F = 2.1$ Hz), 119.9 (t, $J_F = 2.1$ Hz), 117.8 (t, $J_F = 16.6$ Hz), 114.6 (t, $J_F = 19.1$ Hz), 114.1, 55.3, 7.5. ^{19}F NMR (376 MHz, CDCl_3): δ -144.5 (dd, $J_F = 22.4, 12.6$ Hz, 2F), -146.0 (dd, $J_F = 22.4, 12.6$ Hz, 2F).

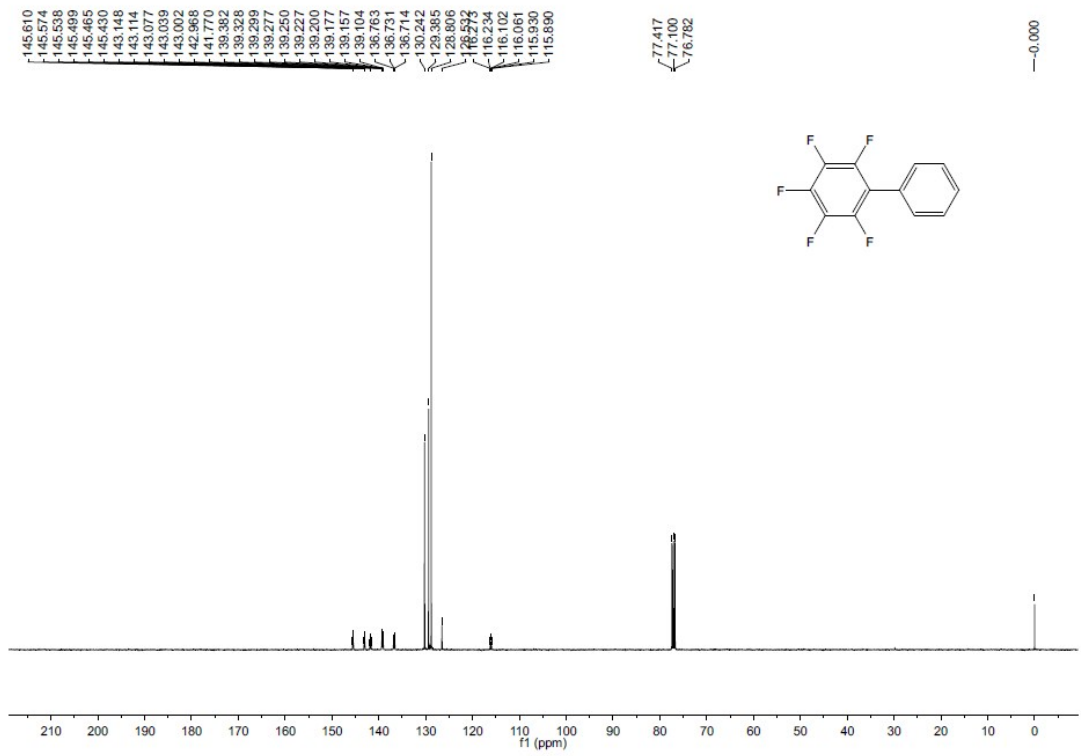
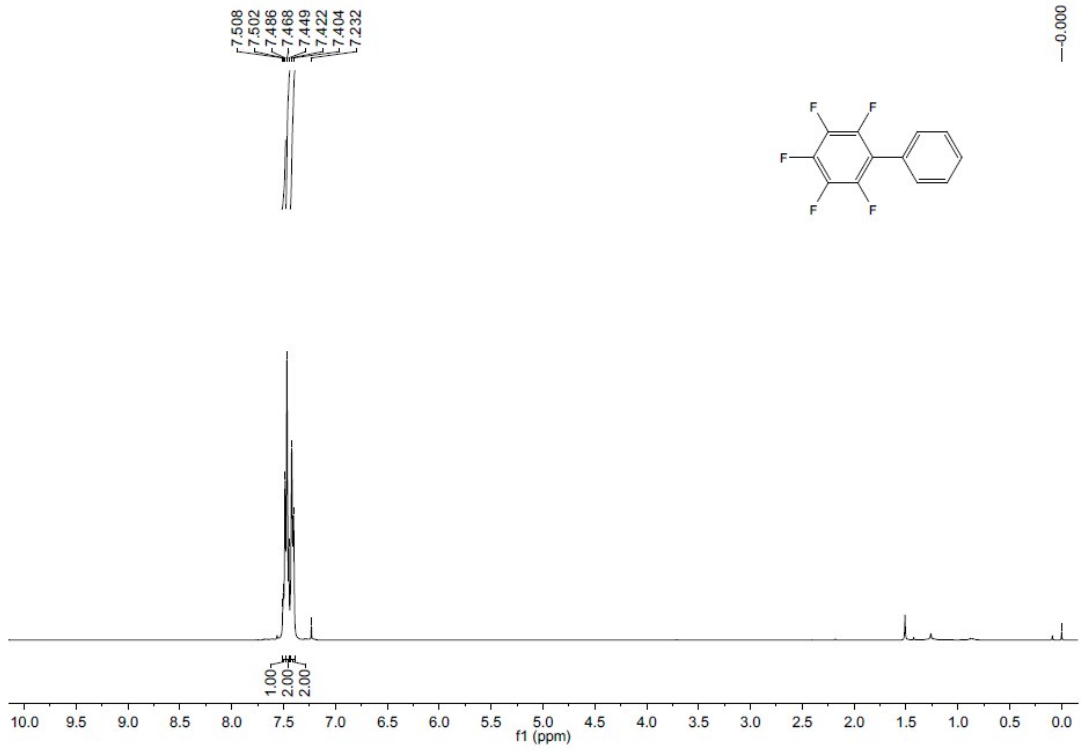
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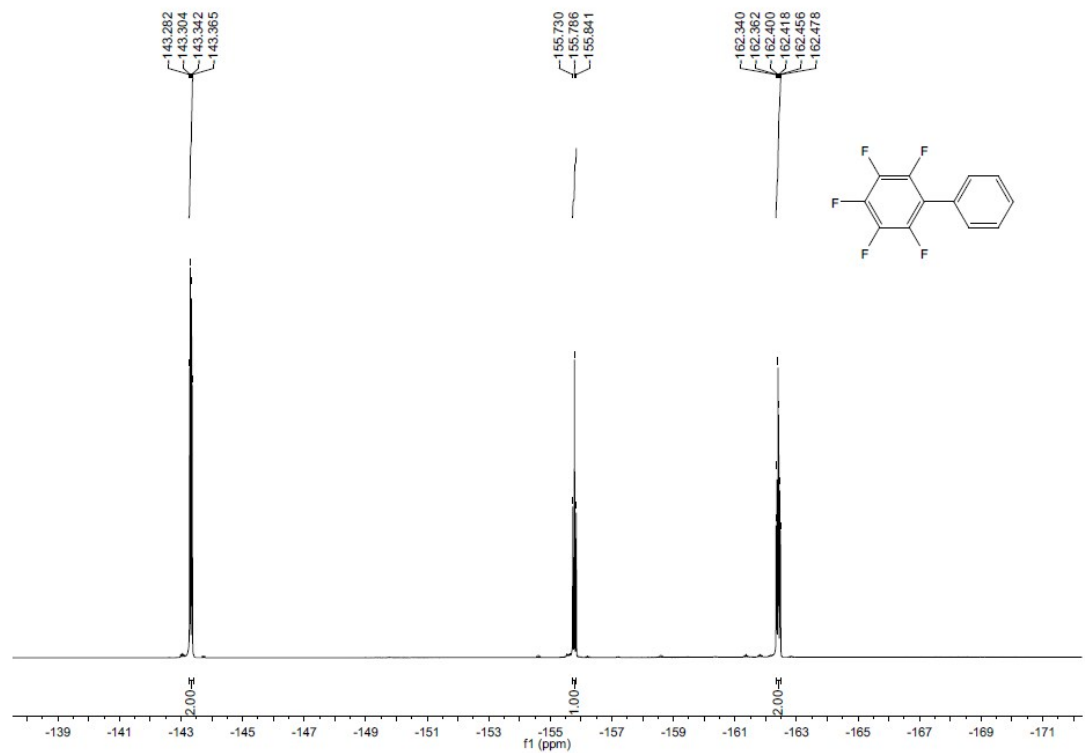
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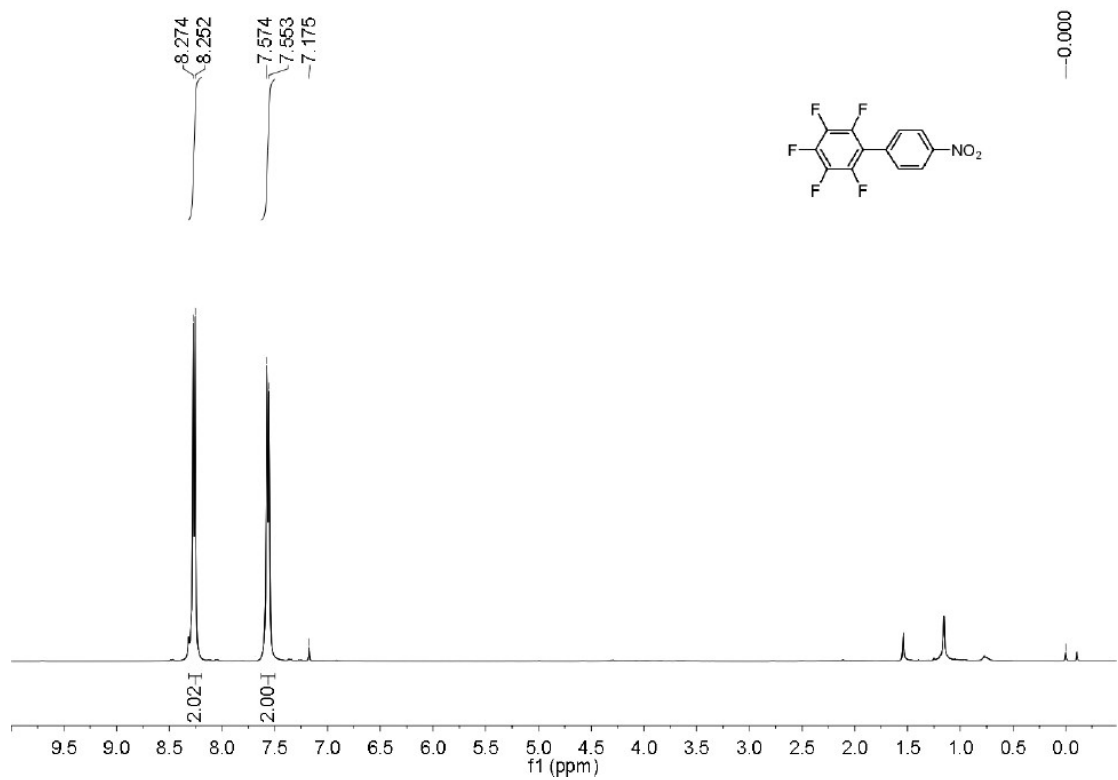
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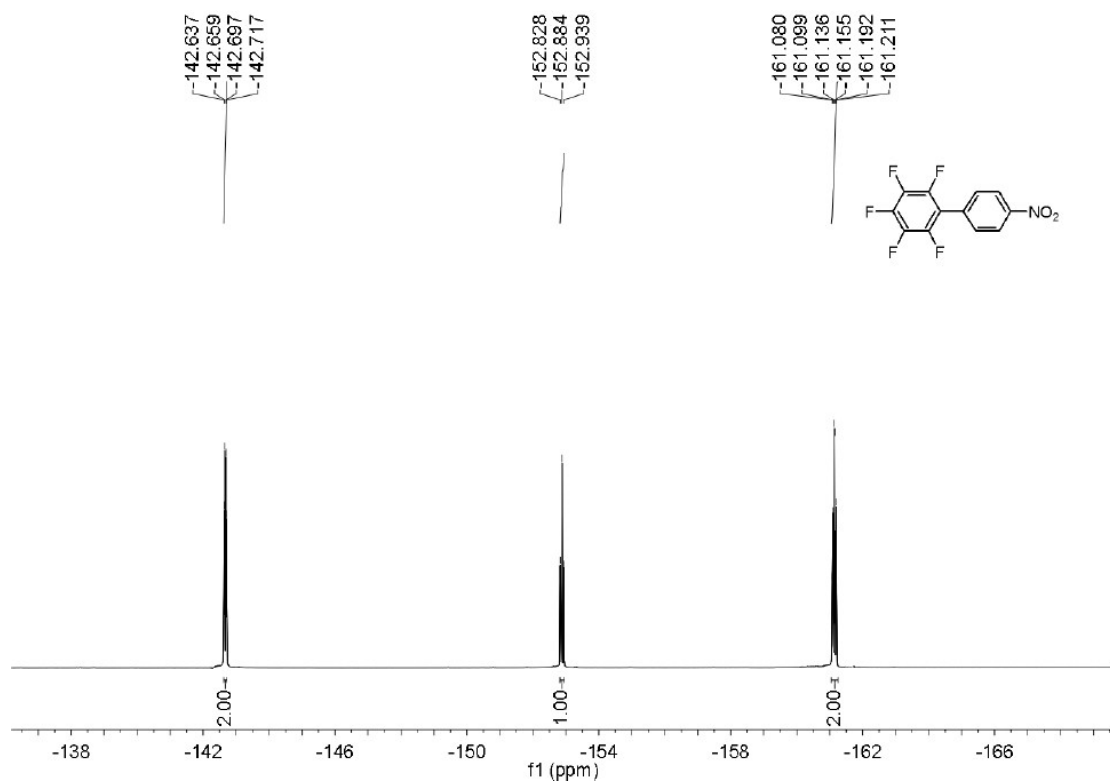
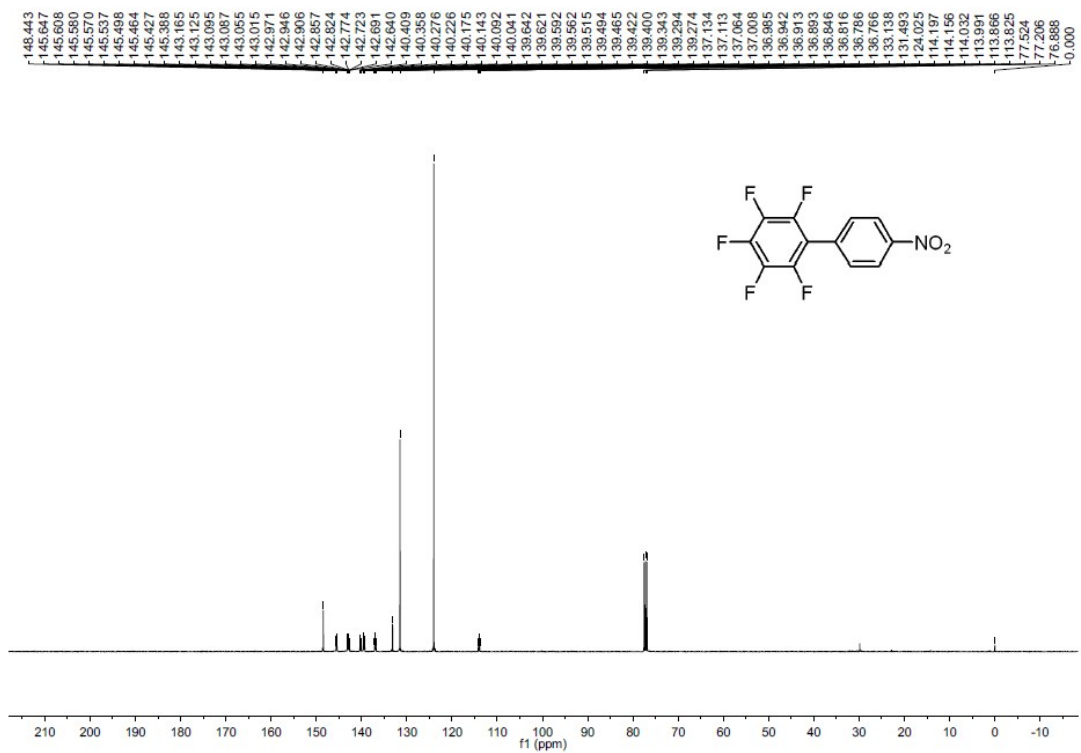
**Copies of ^1H NMR, ^{13}C NMR and ^{19}F NMR Spectra of Compounds
3a-v and 5a-p**



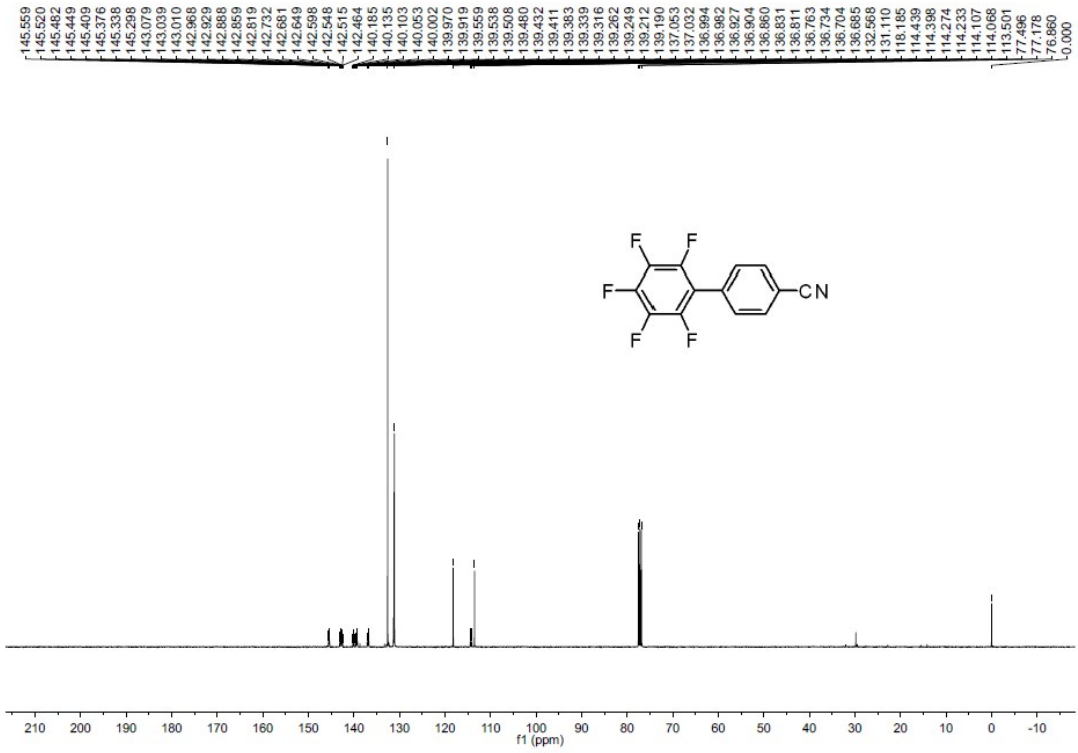
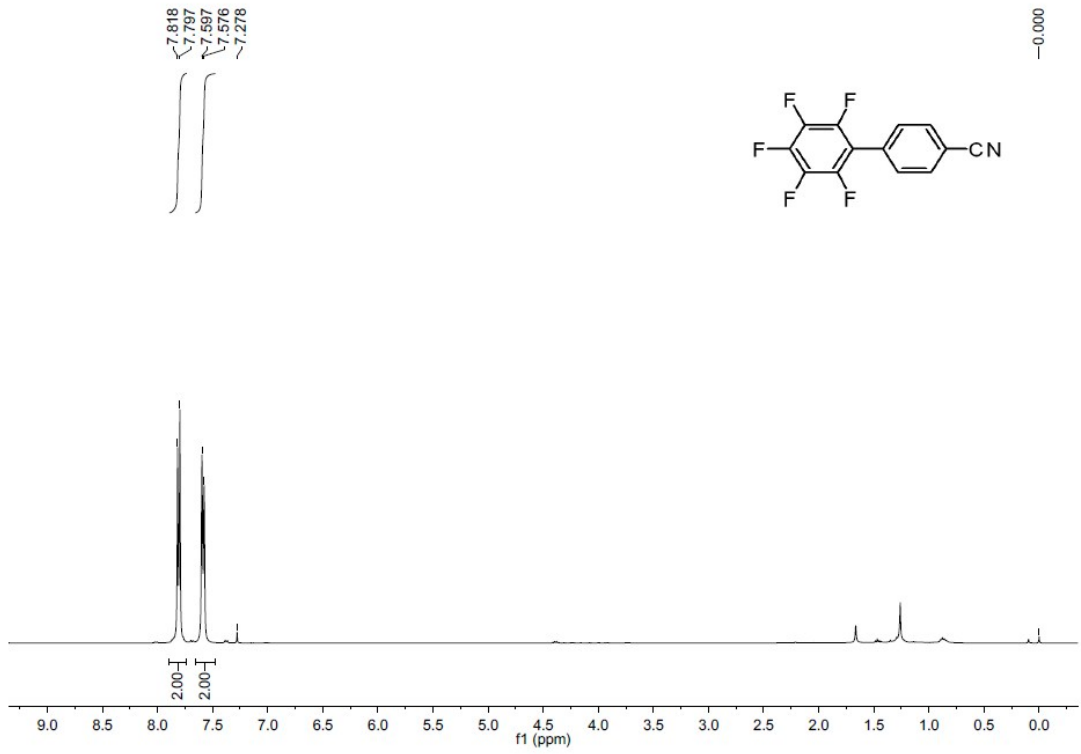


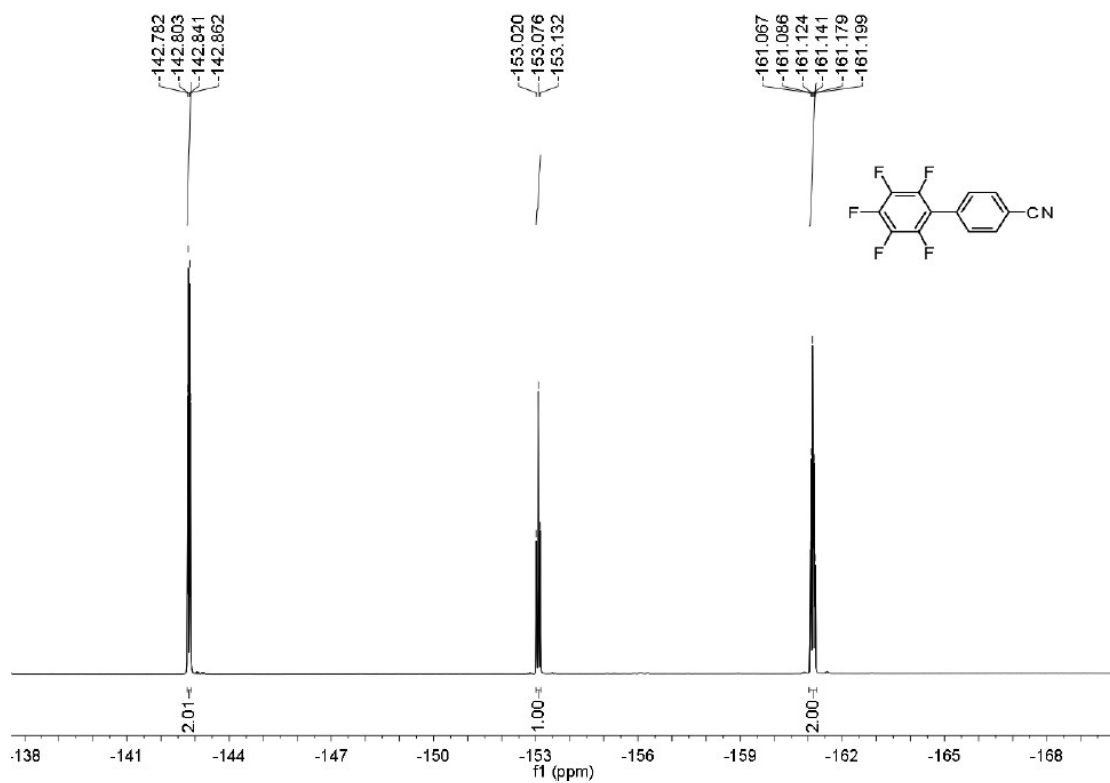
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3a**



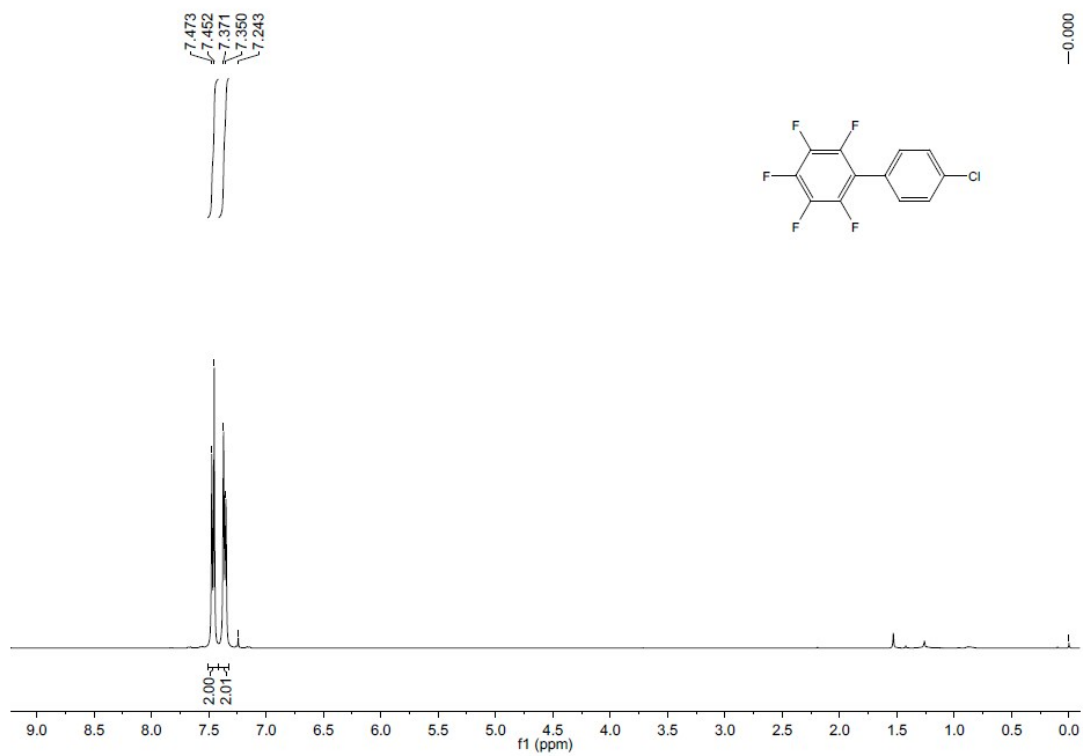


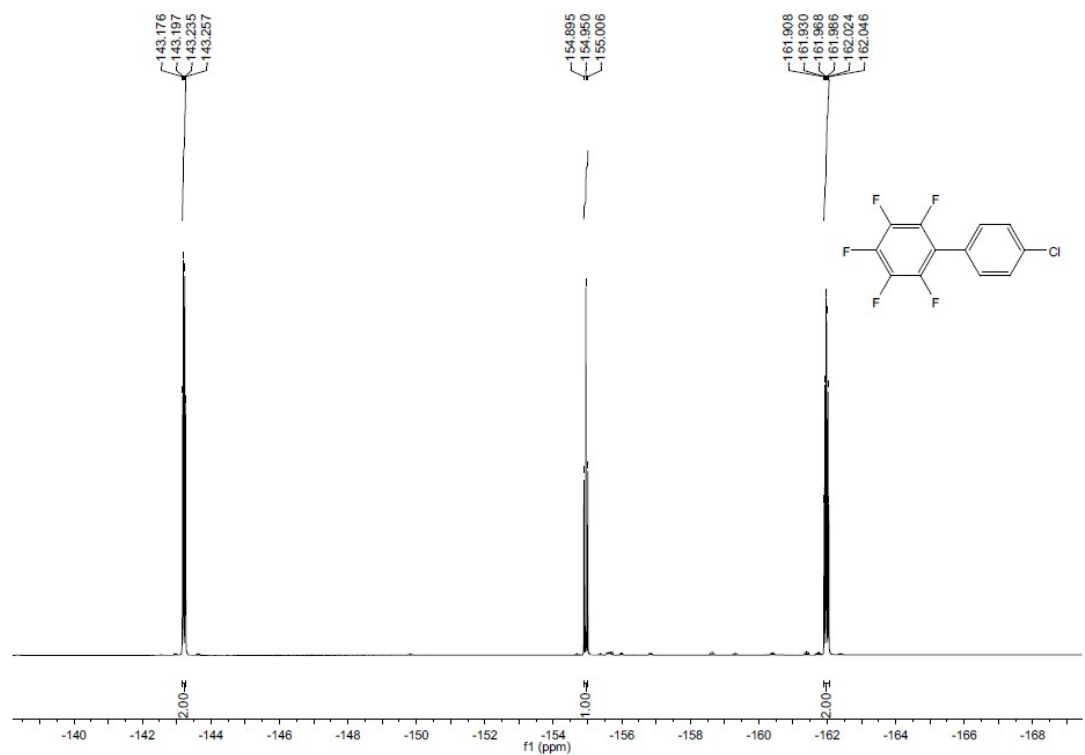
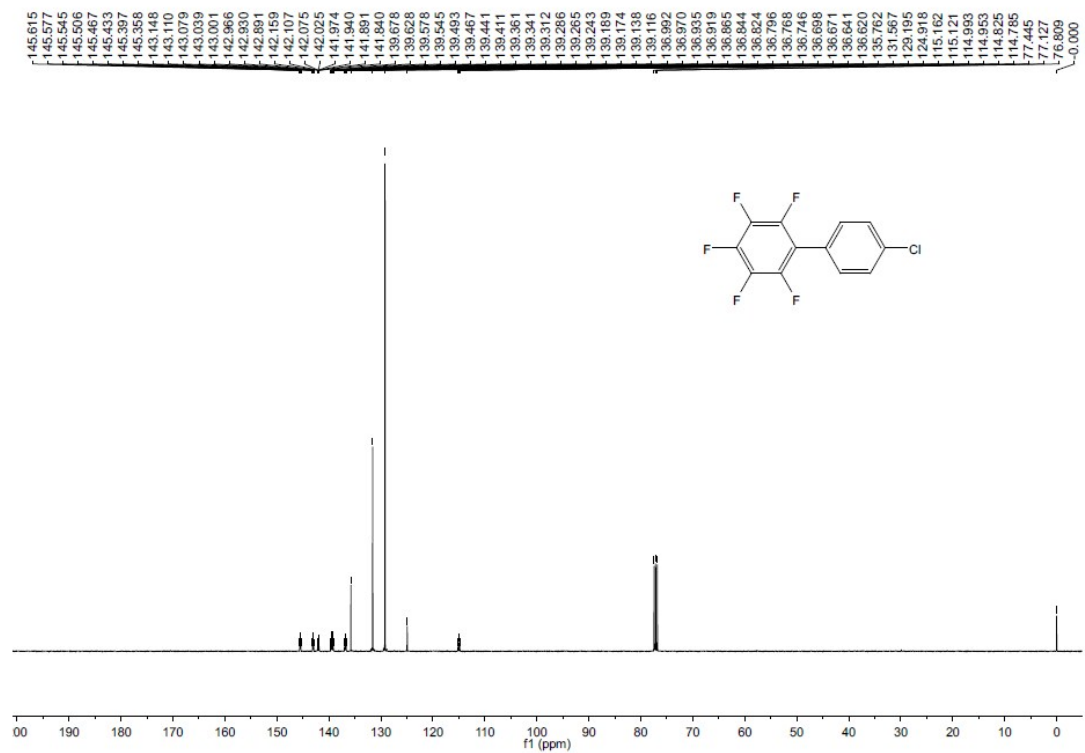
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3b**



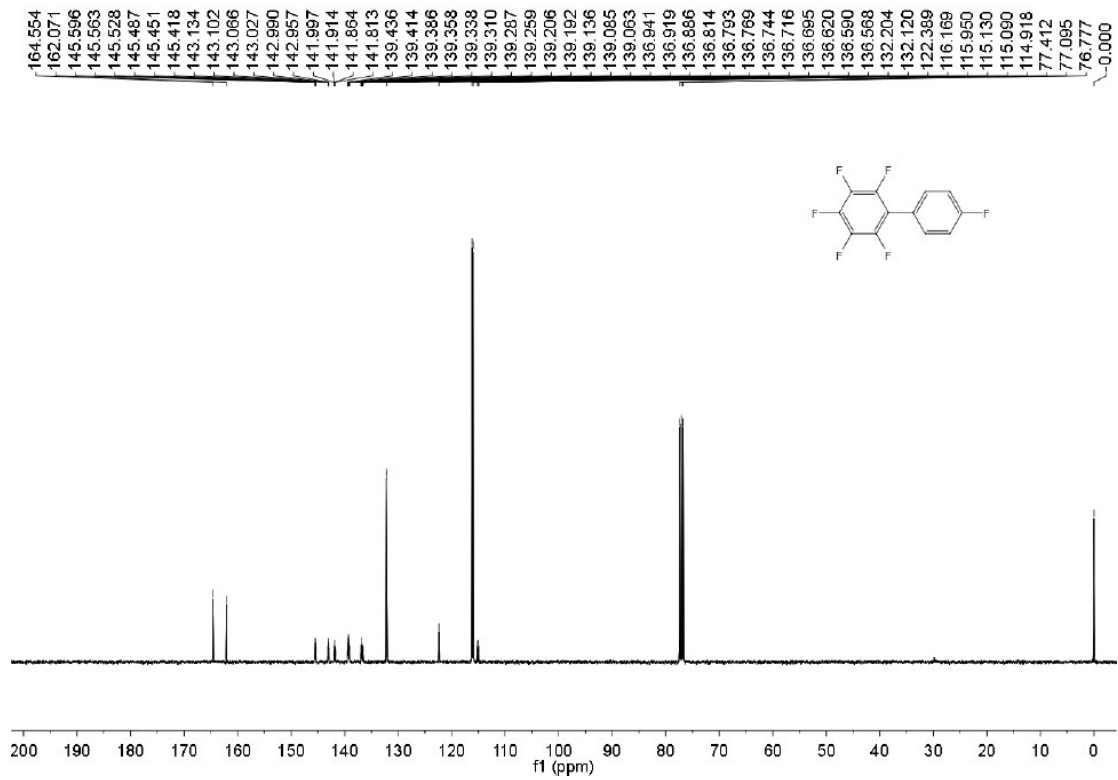
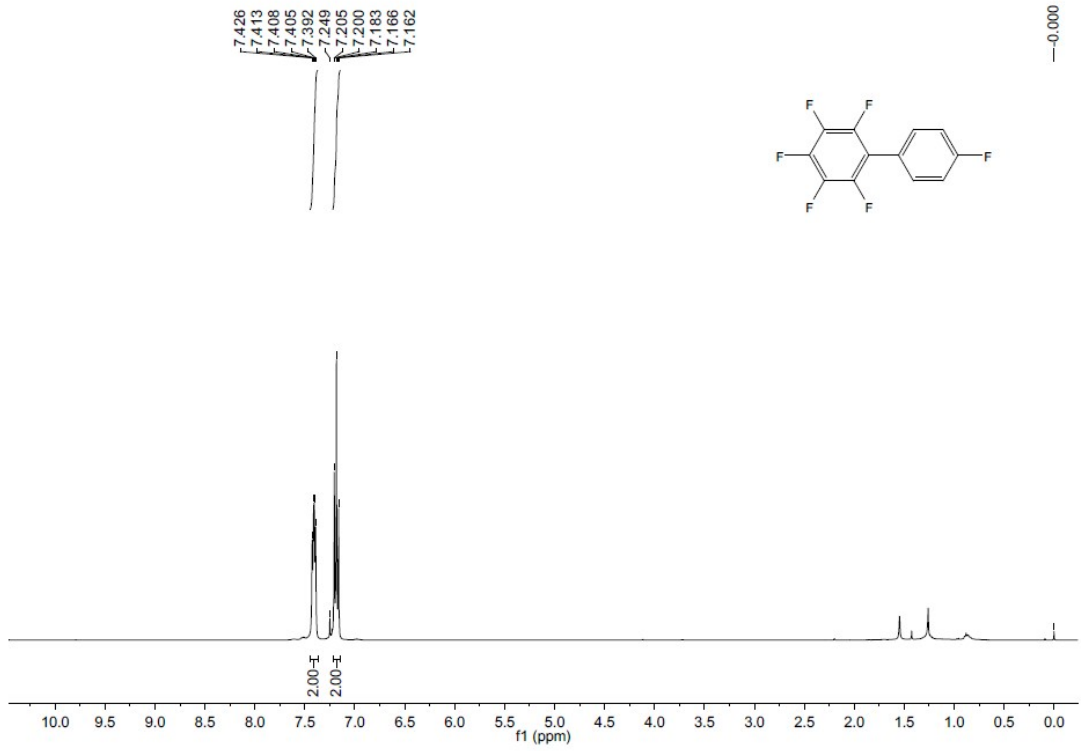


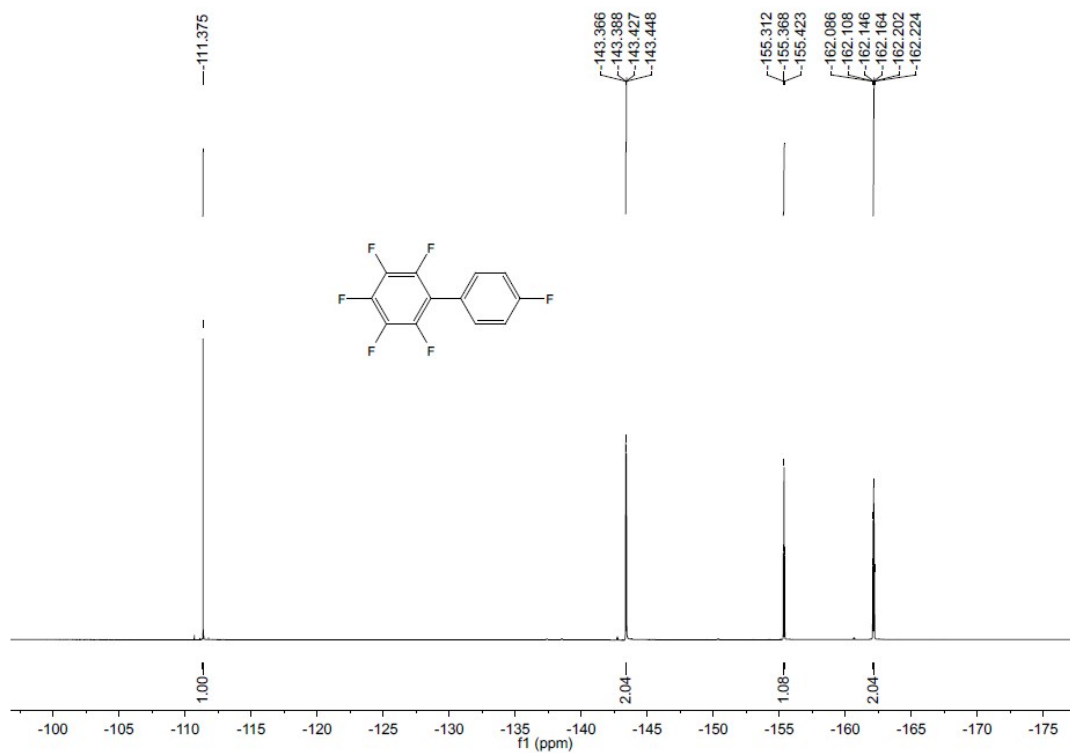
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3c**



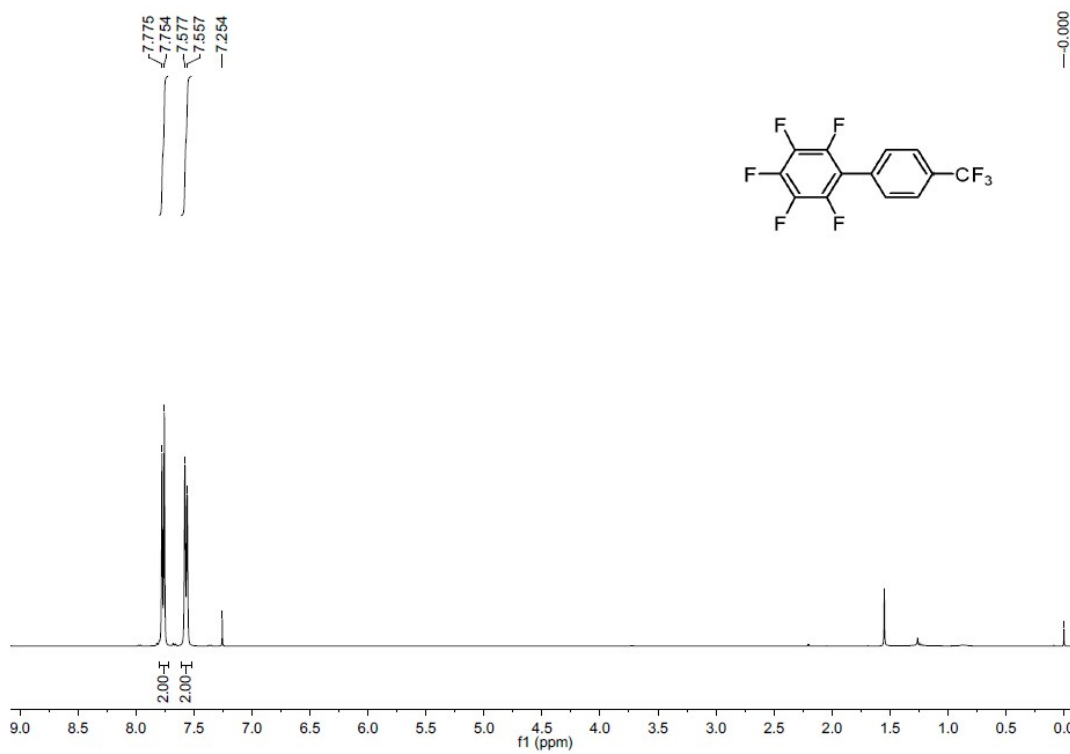


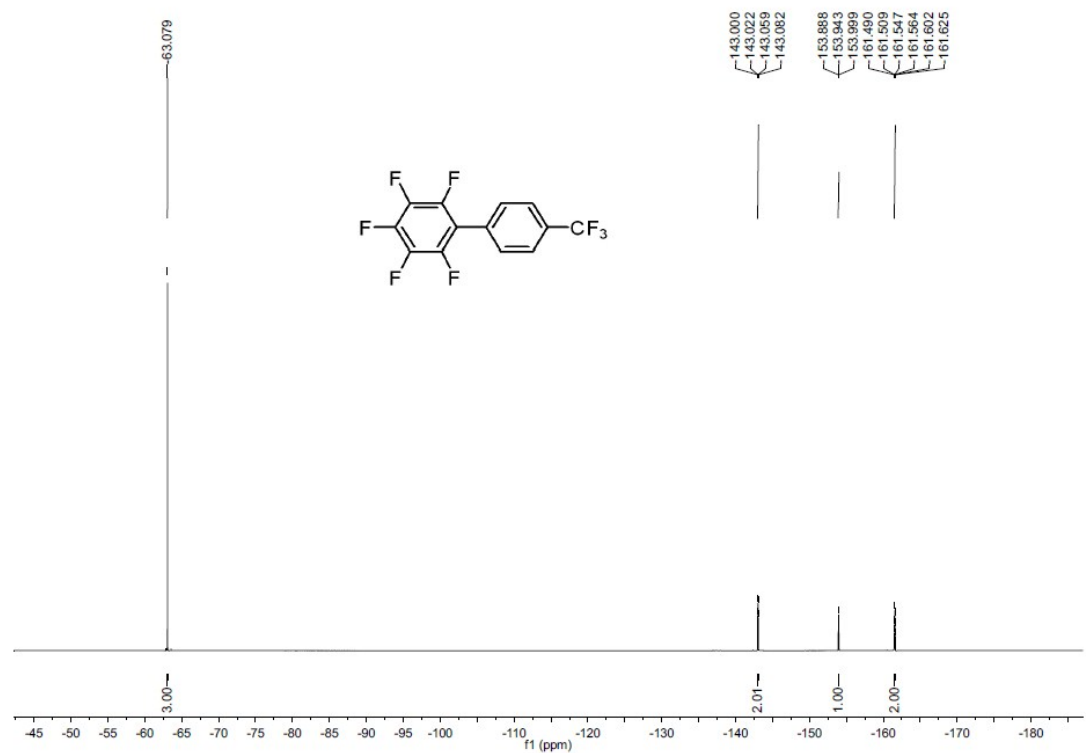
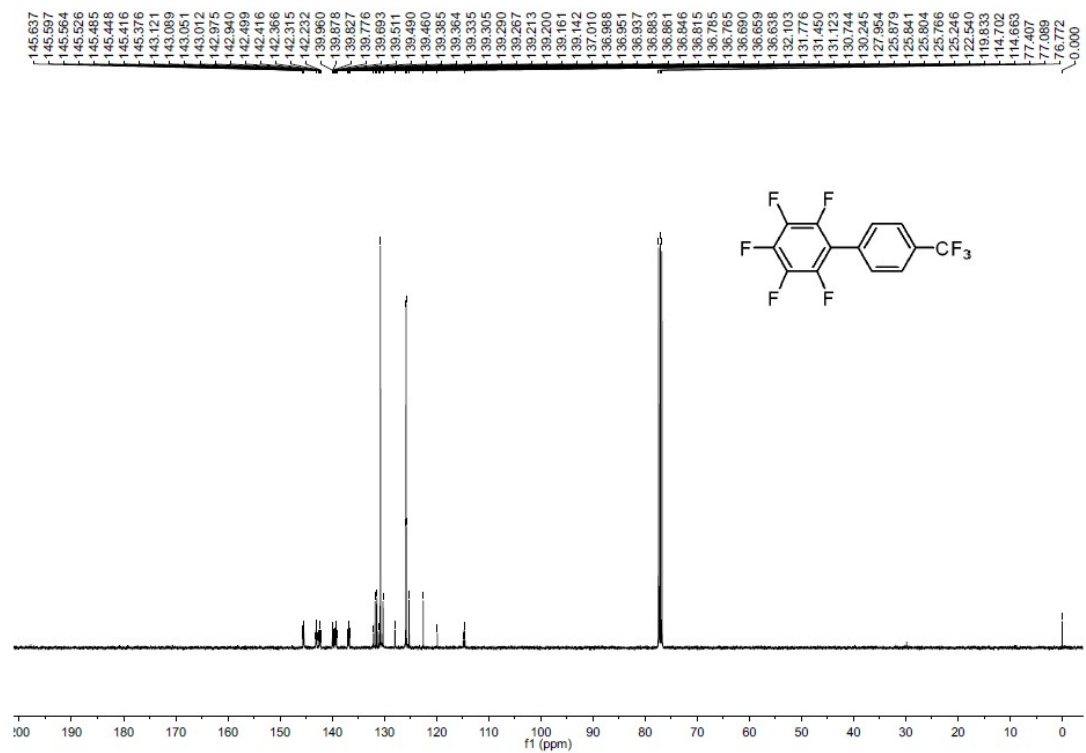
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3d**



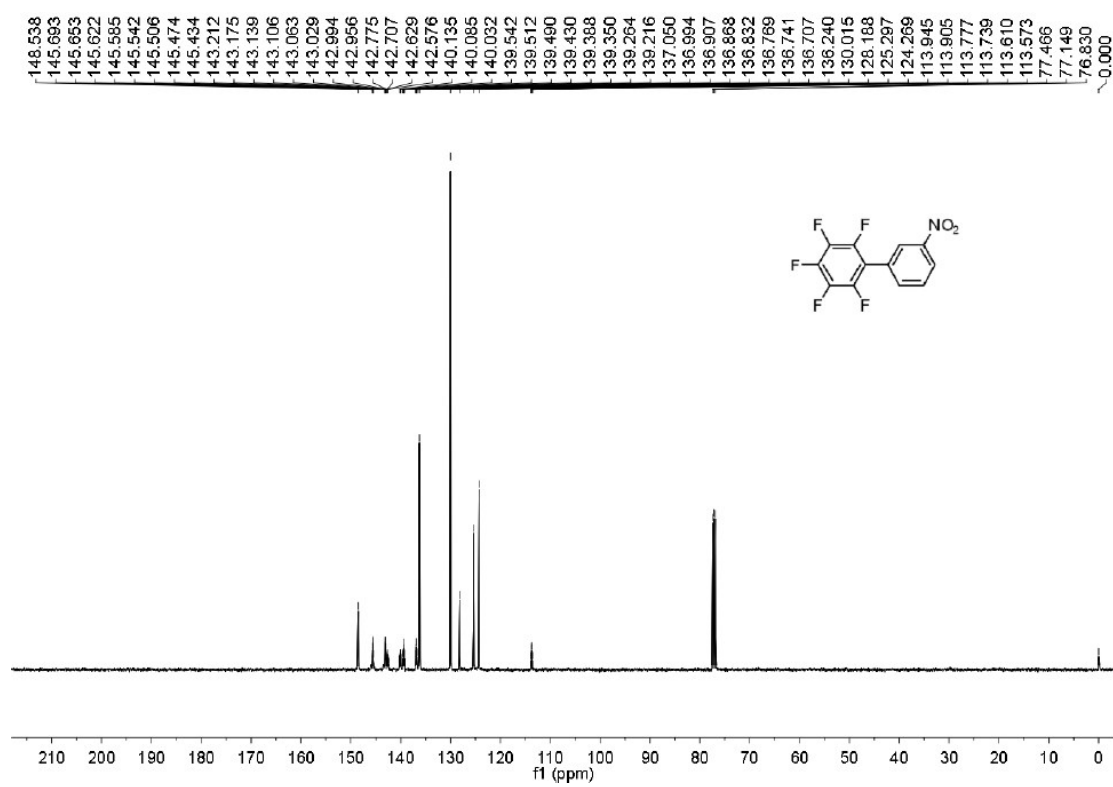
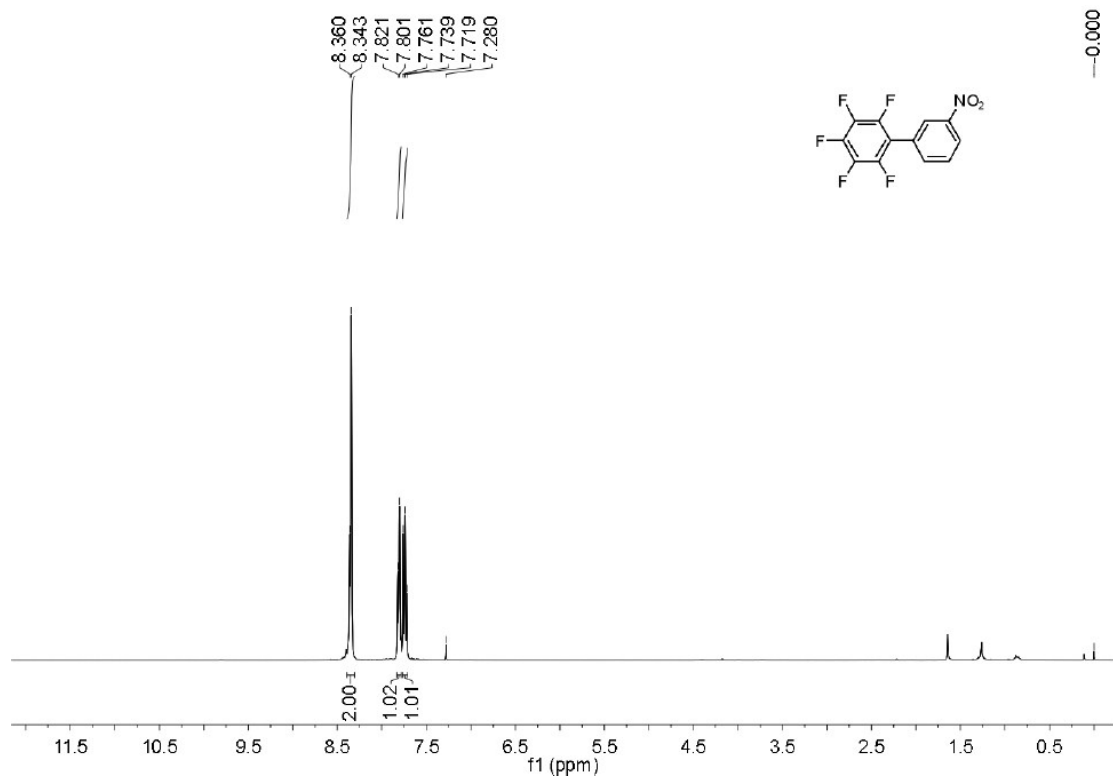


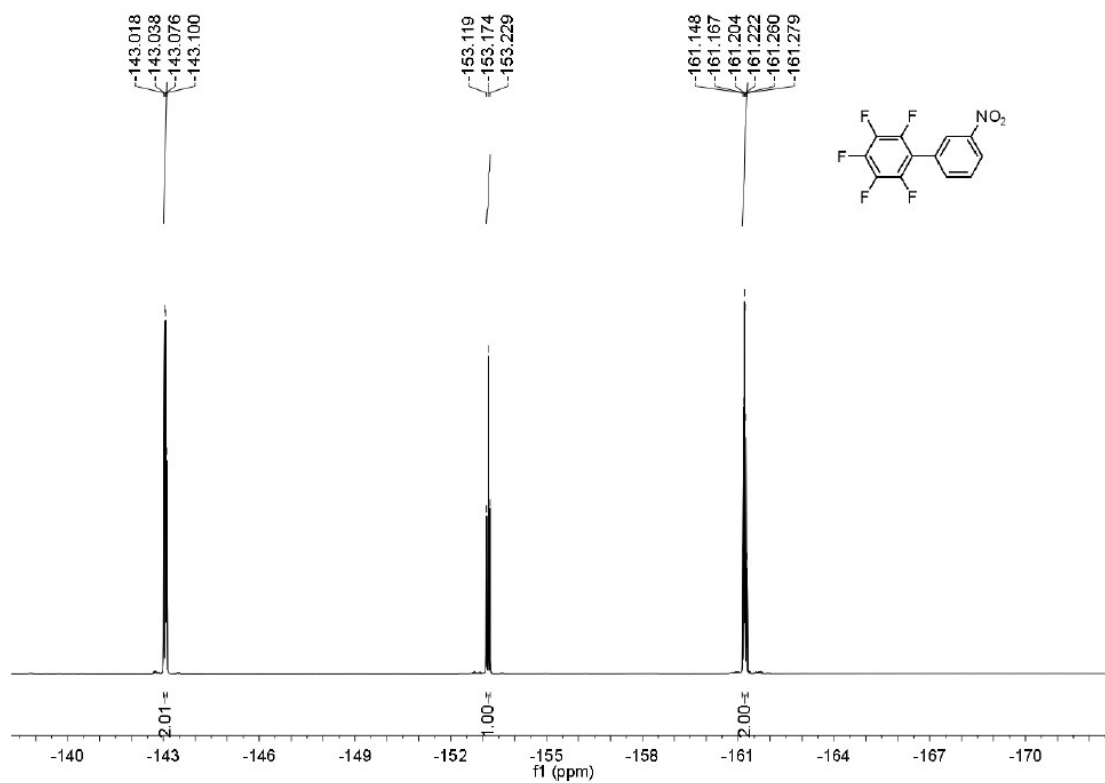
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3e**



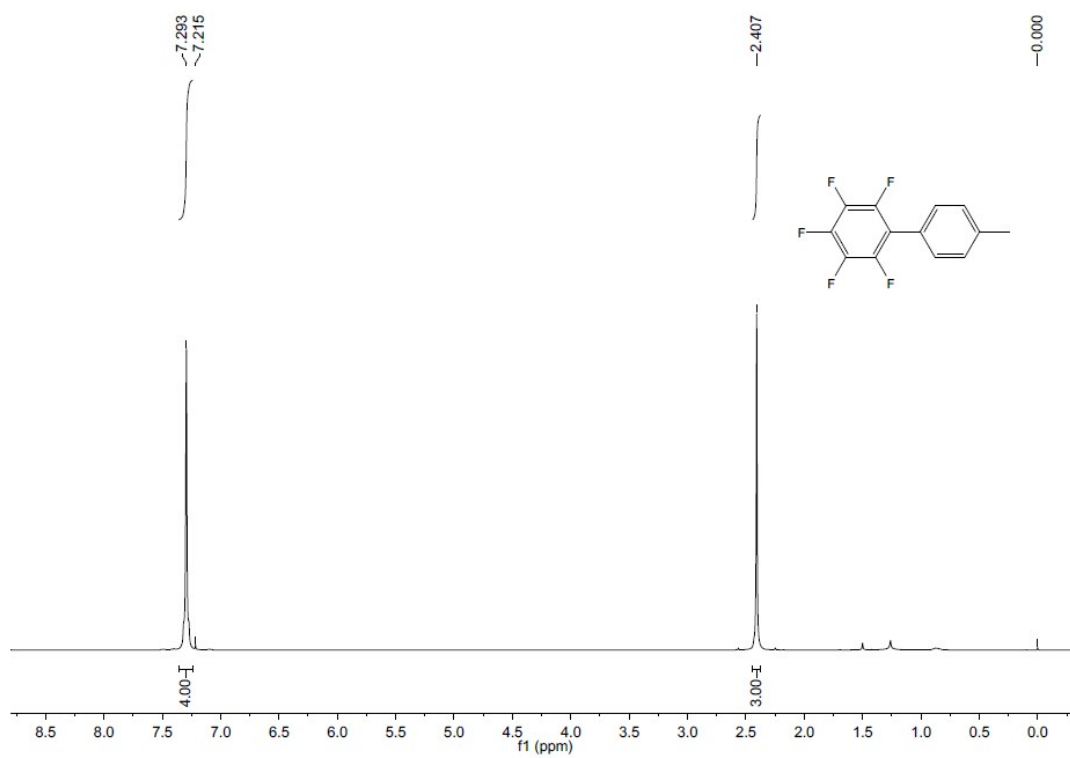


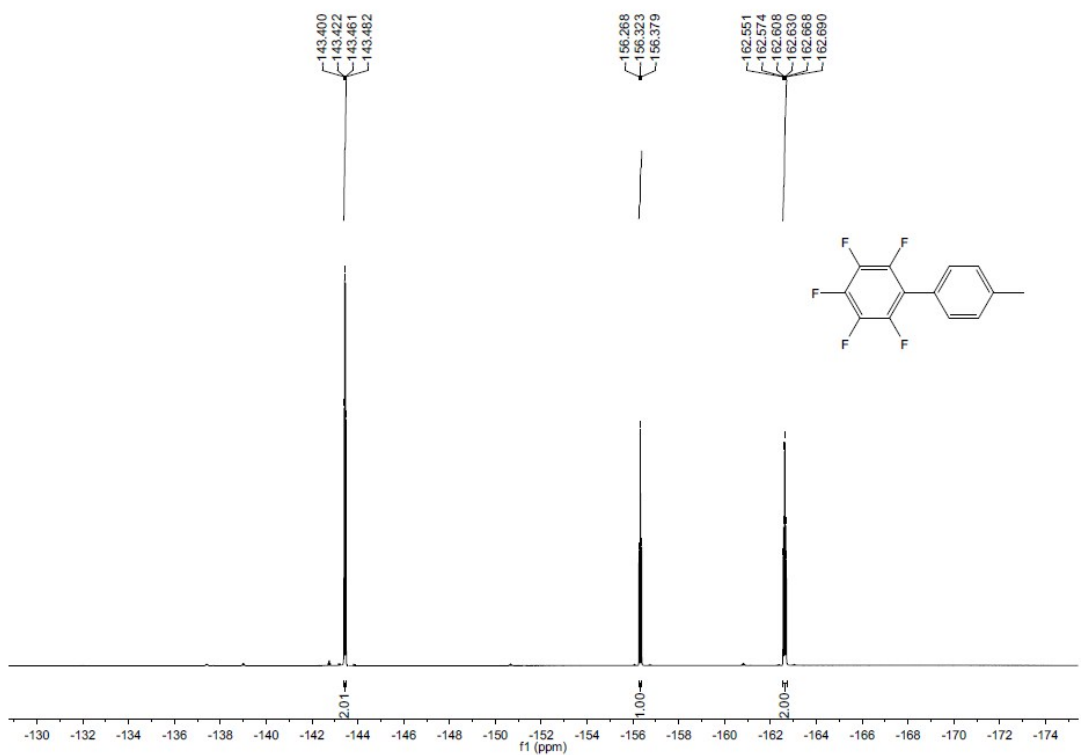
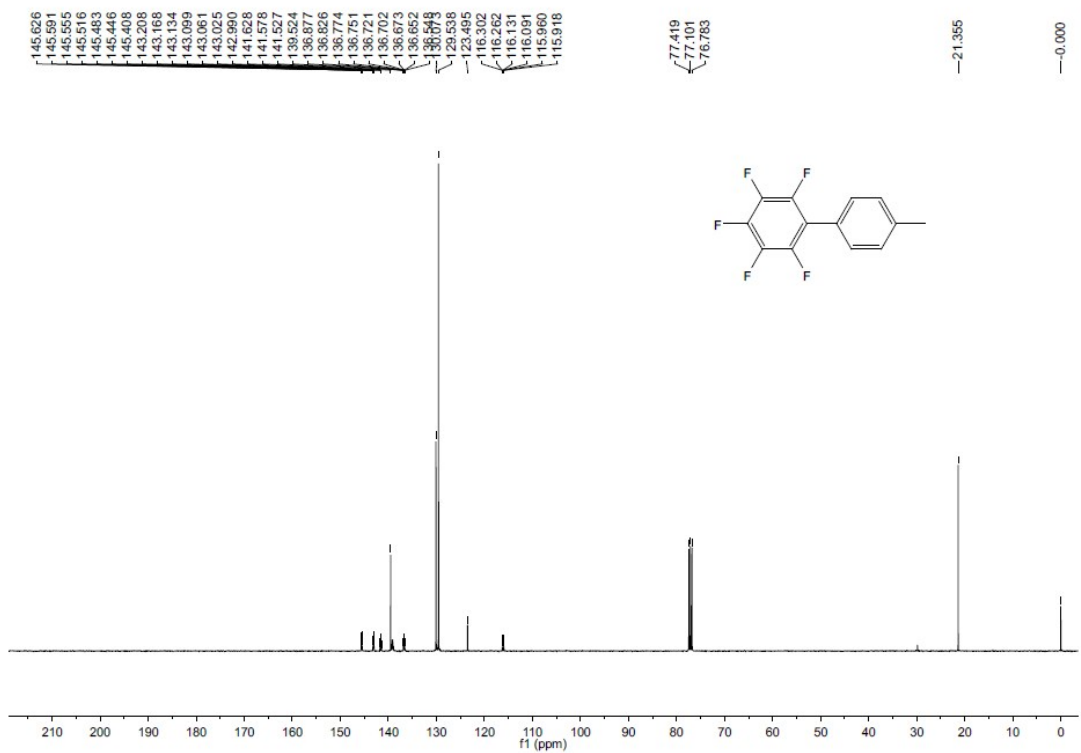
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3f**



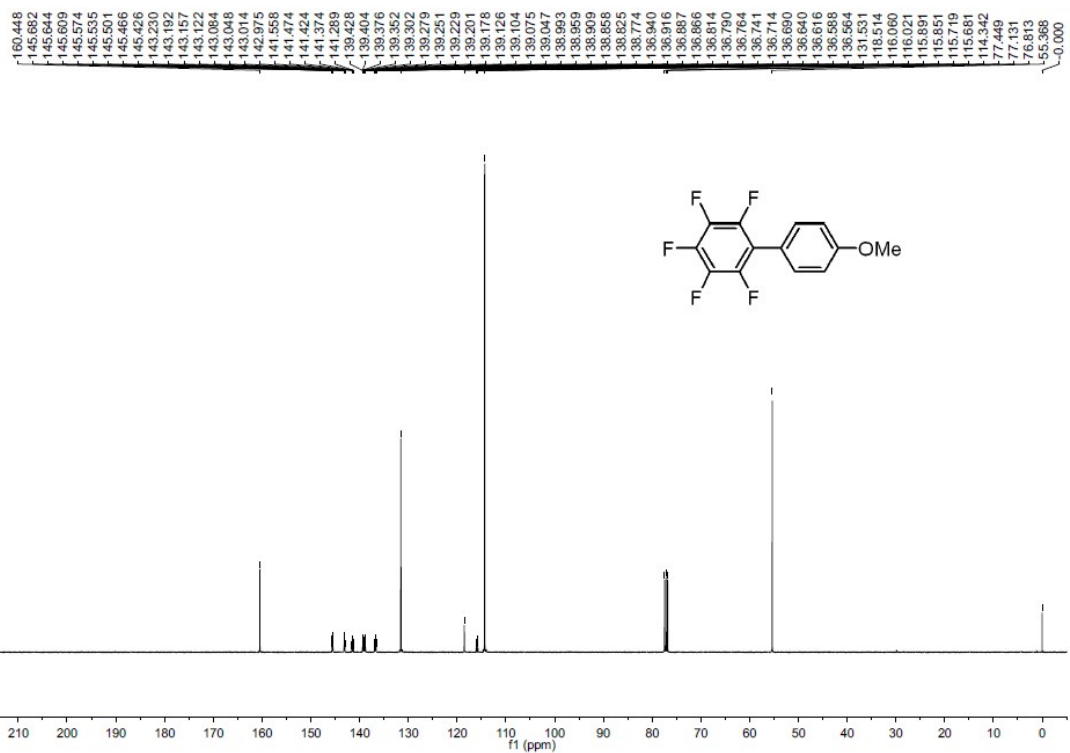
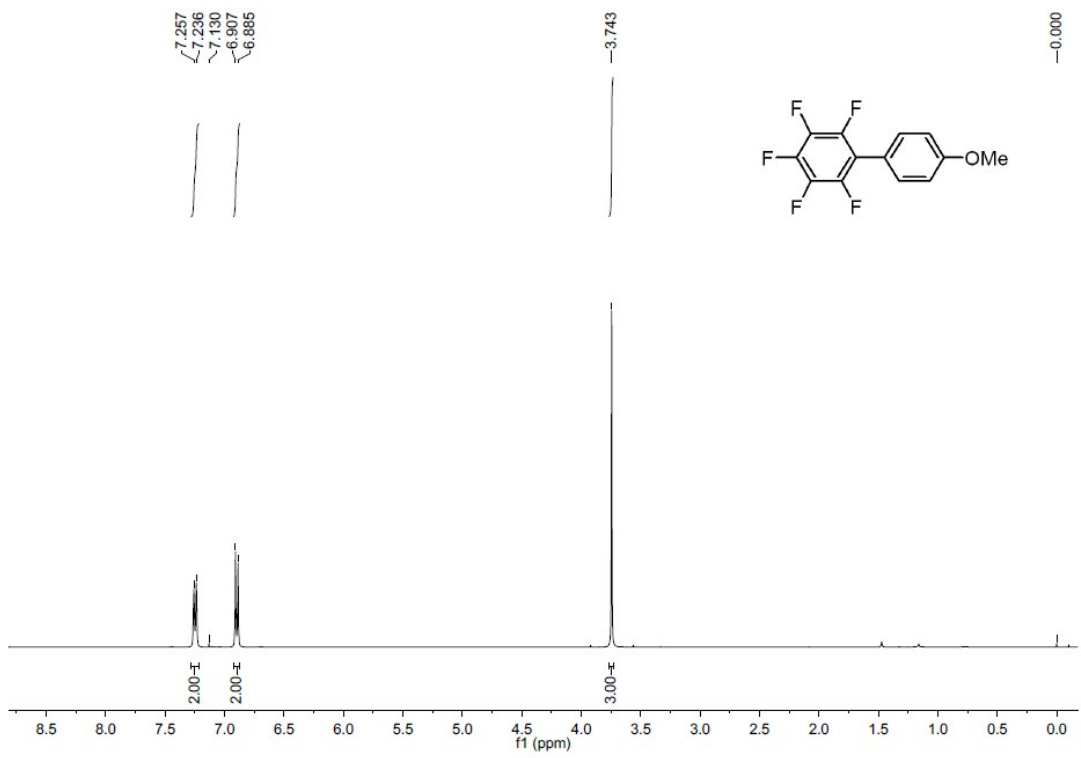


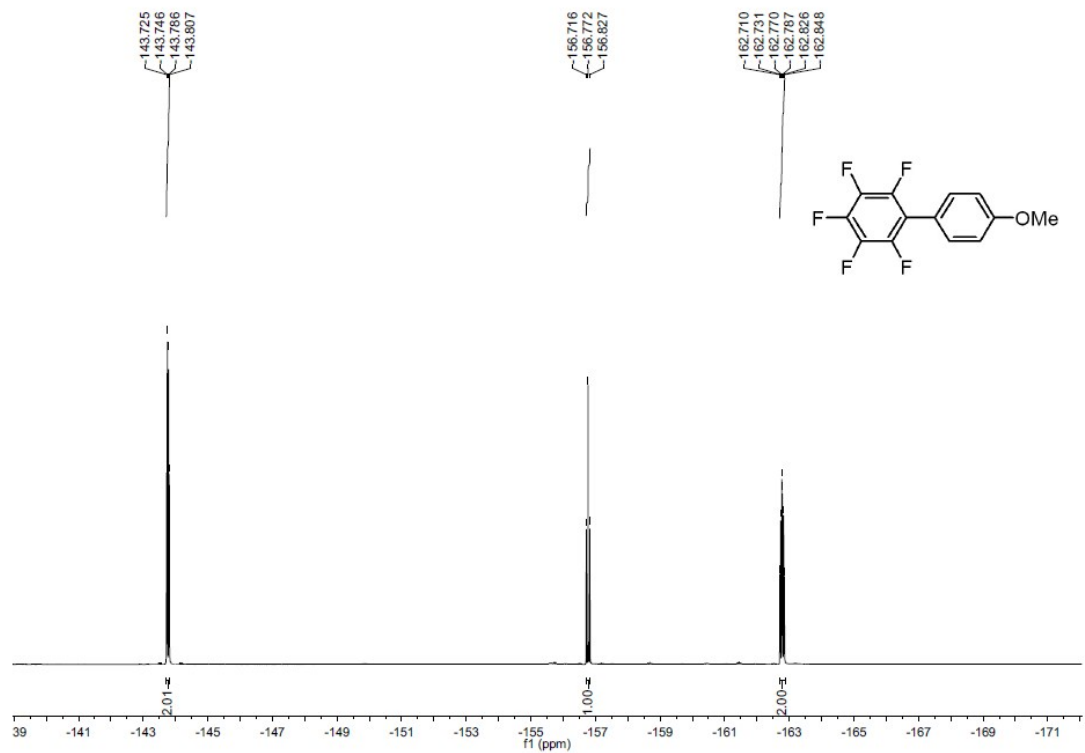
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3g**



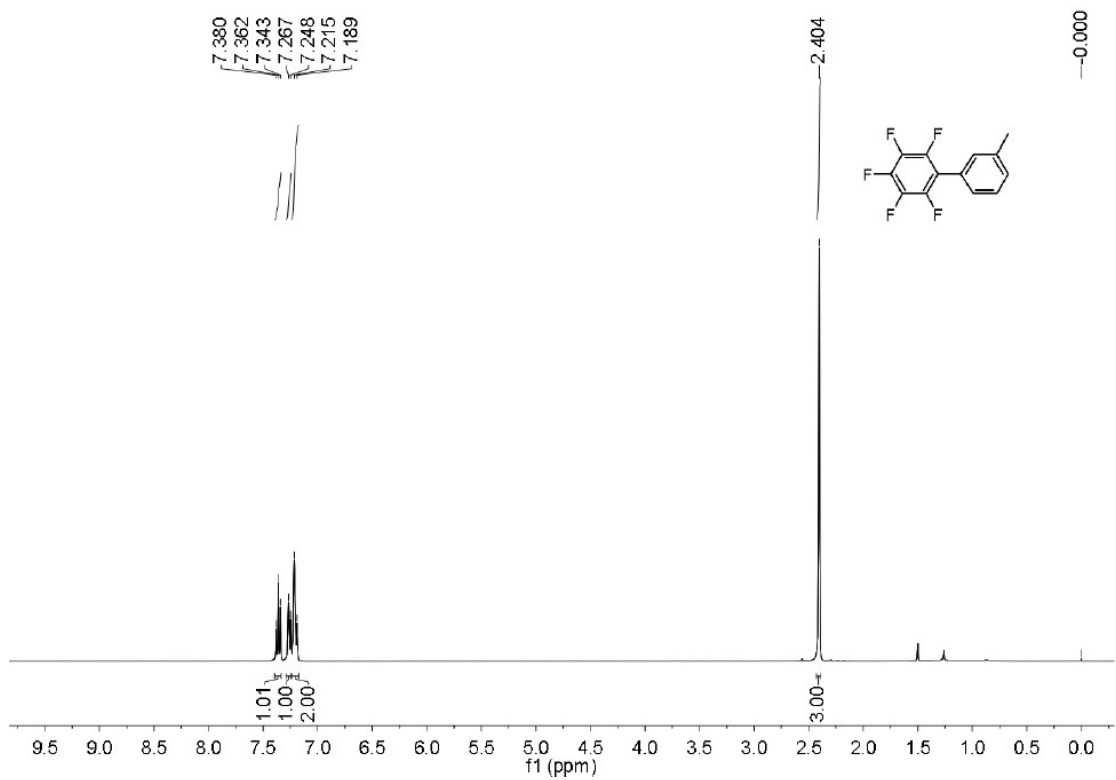


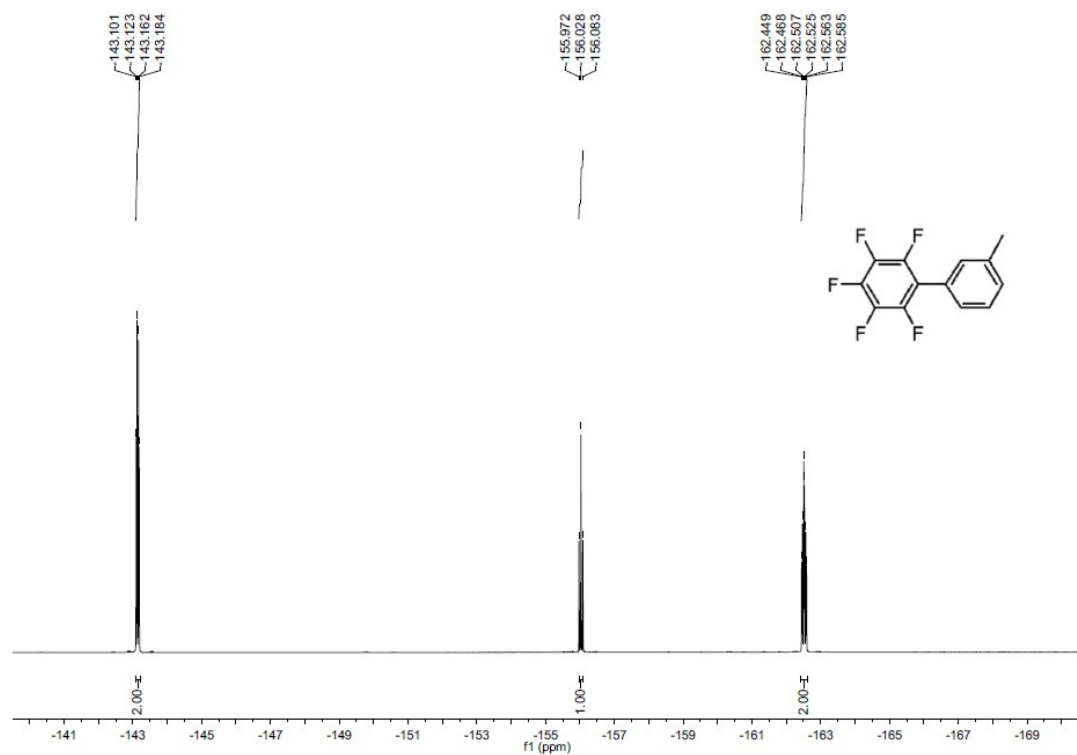
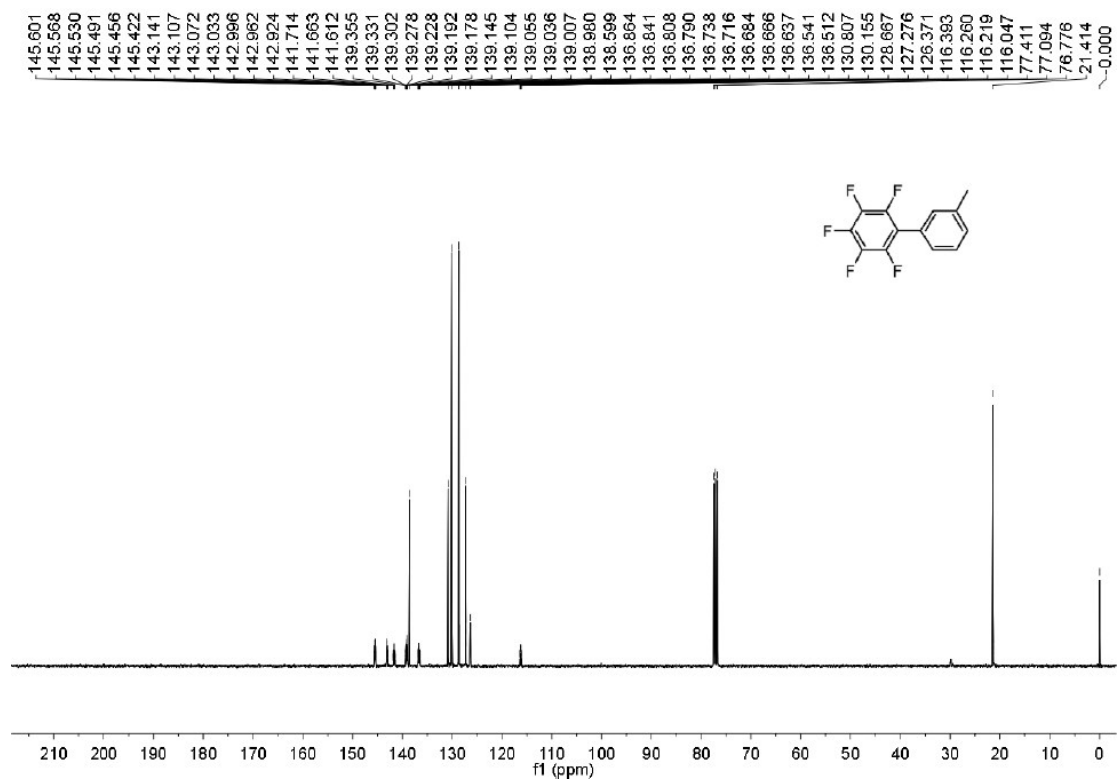
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3h**



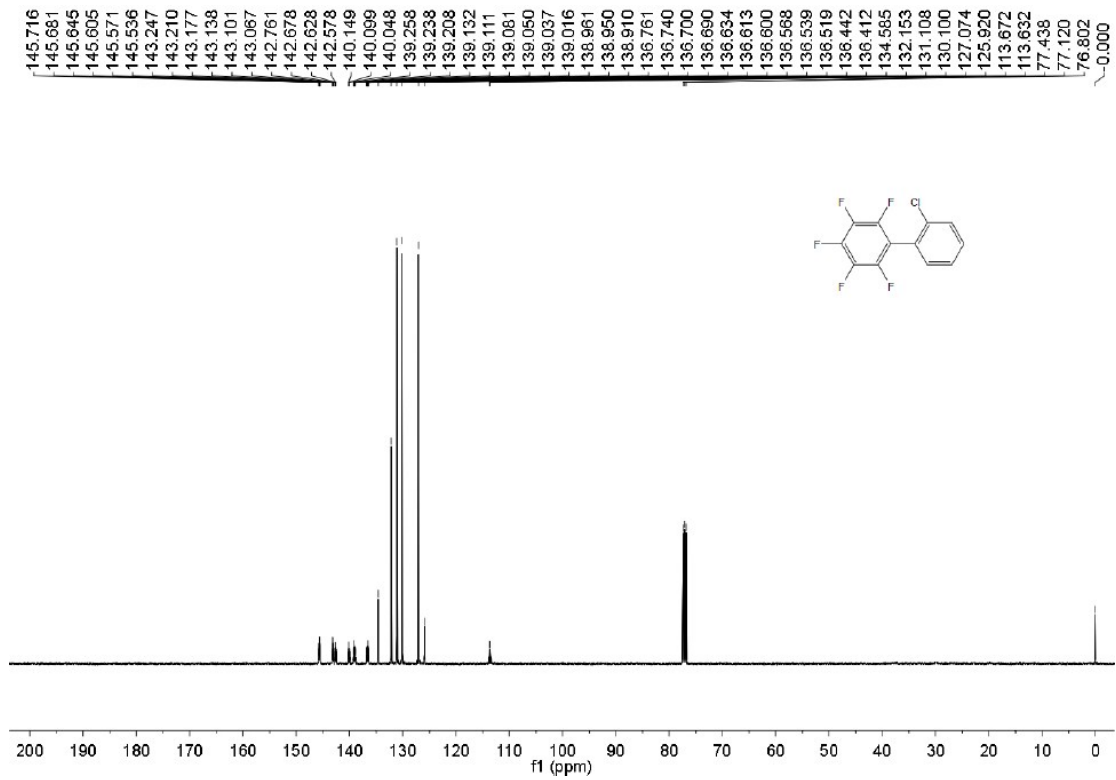
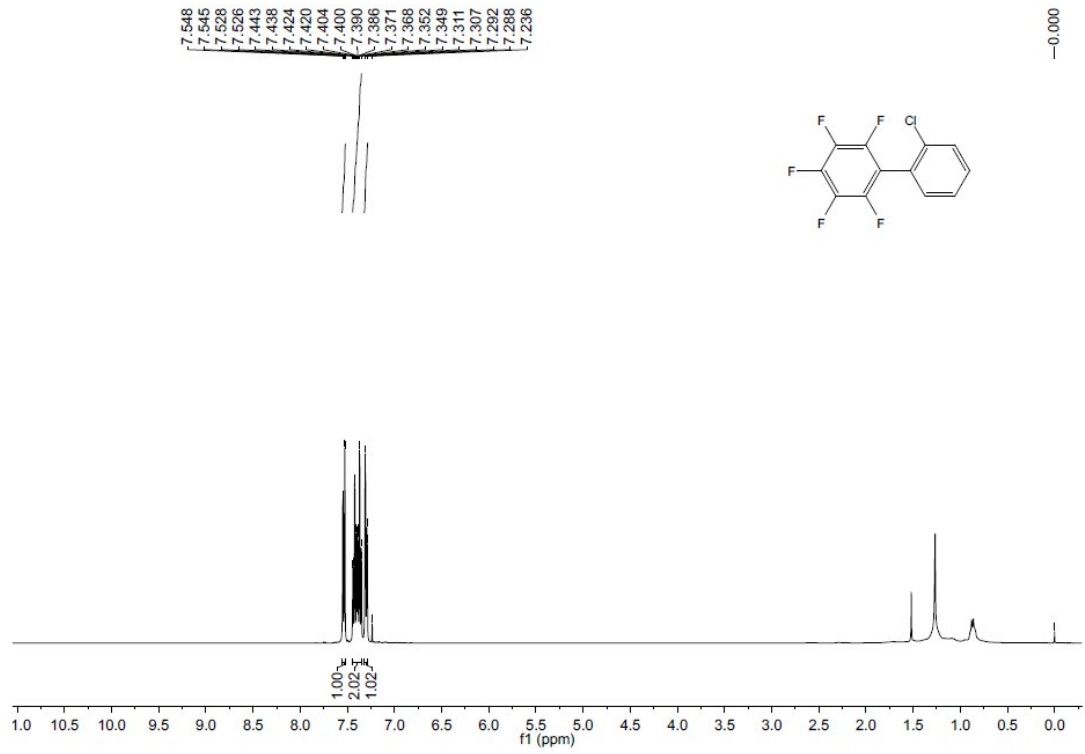


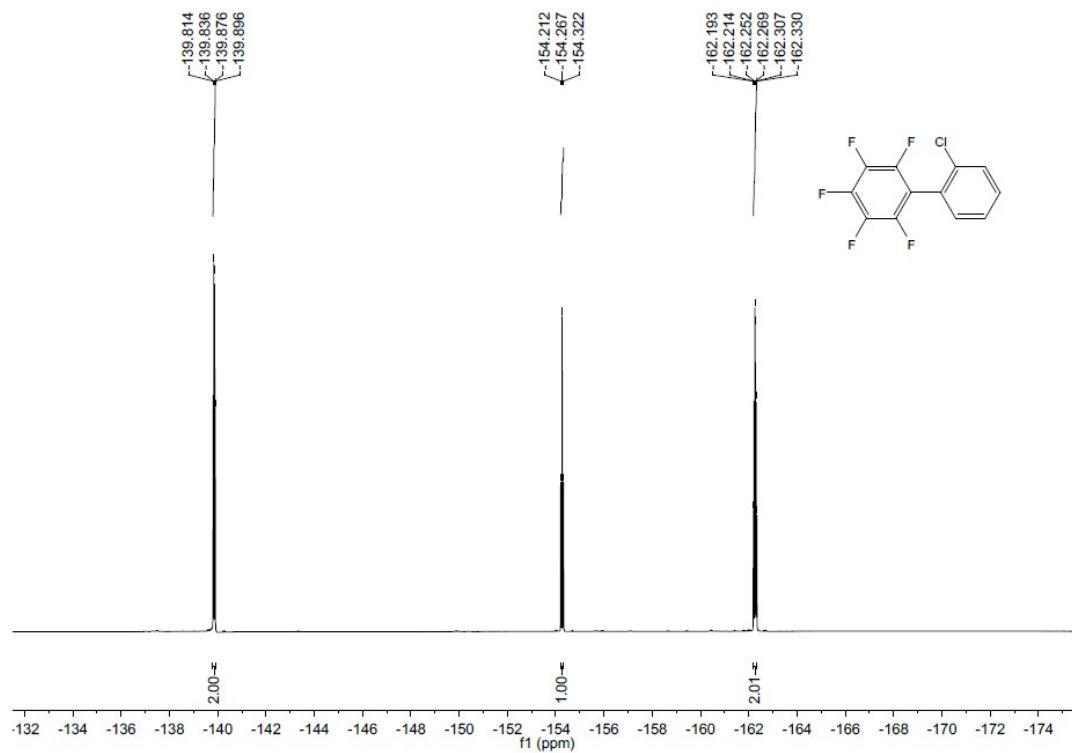
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3i**



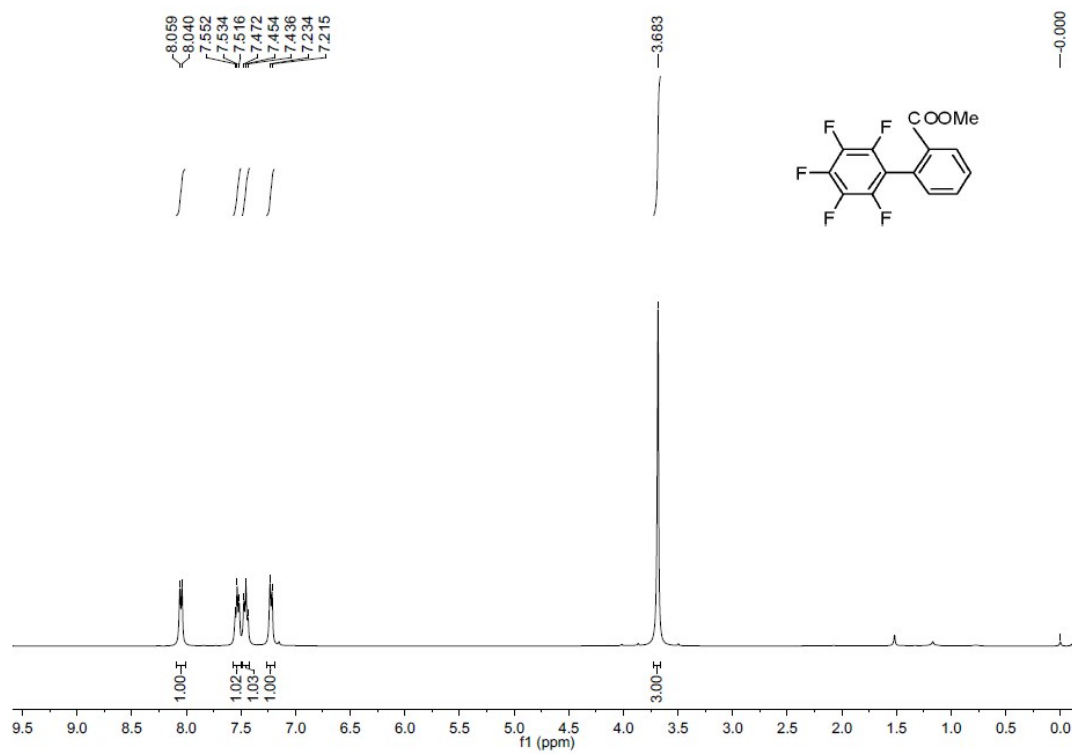


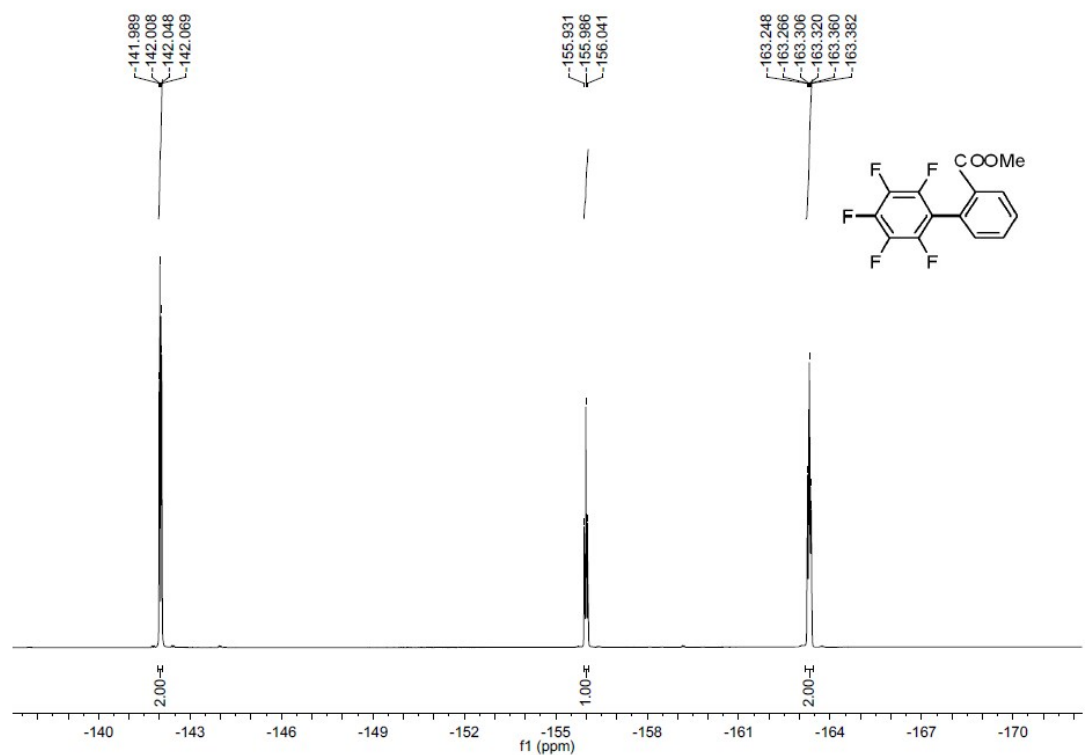
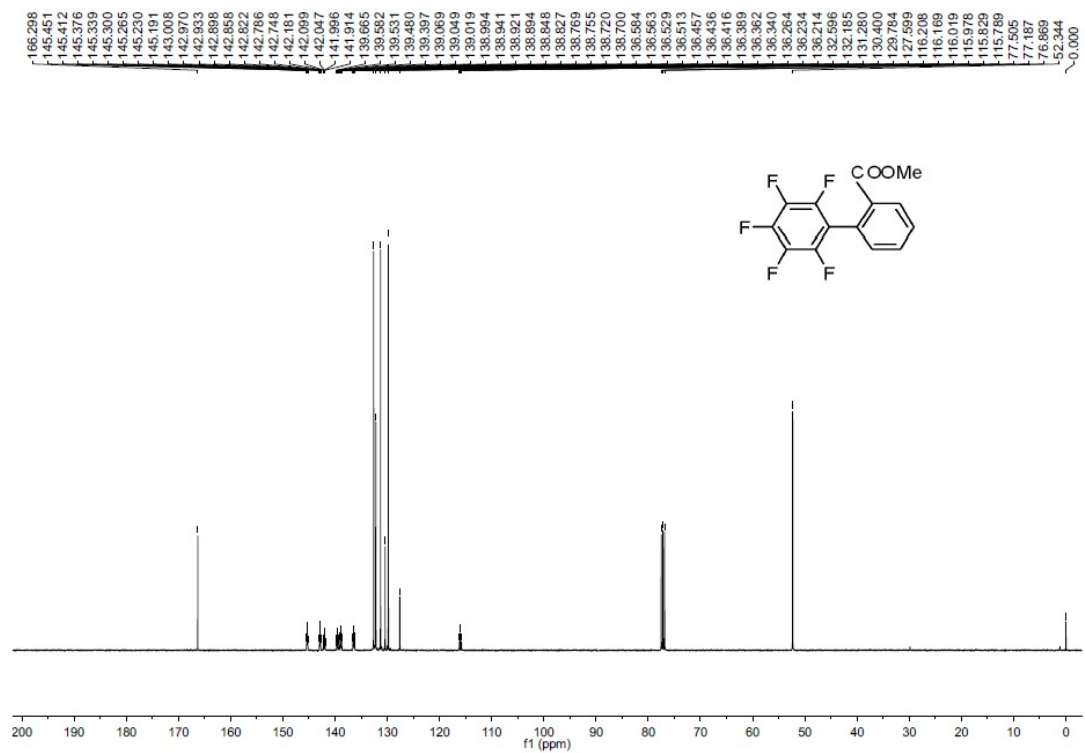
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3j**



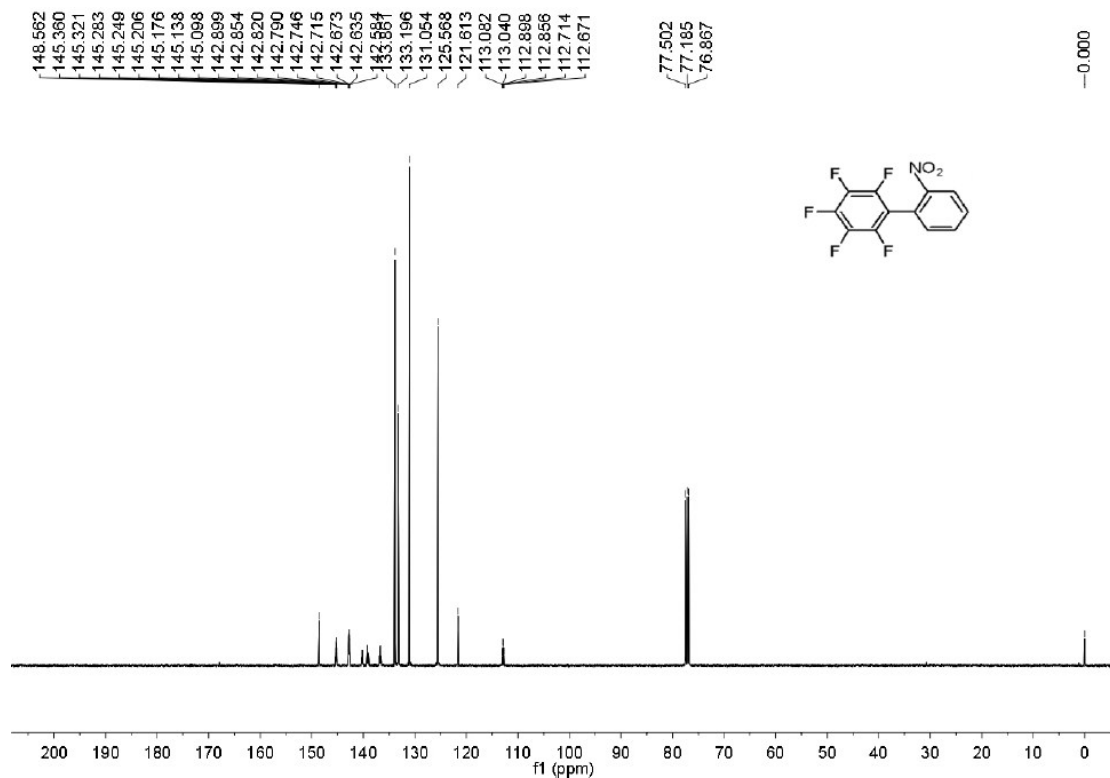
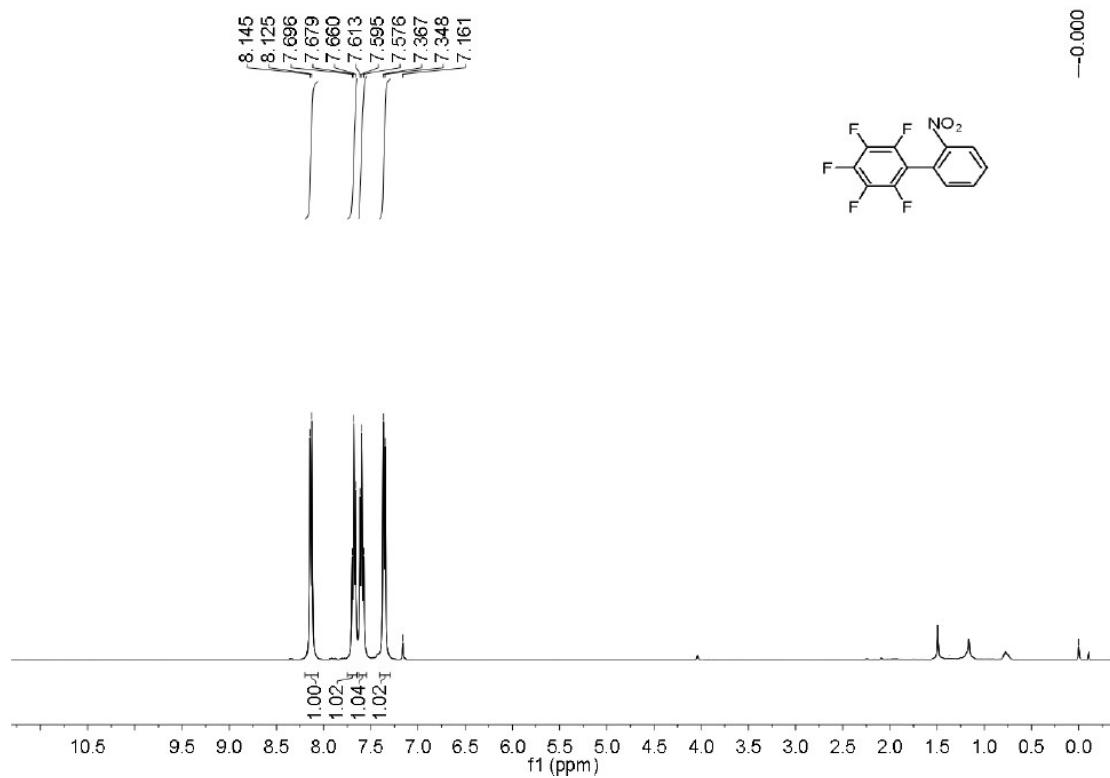


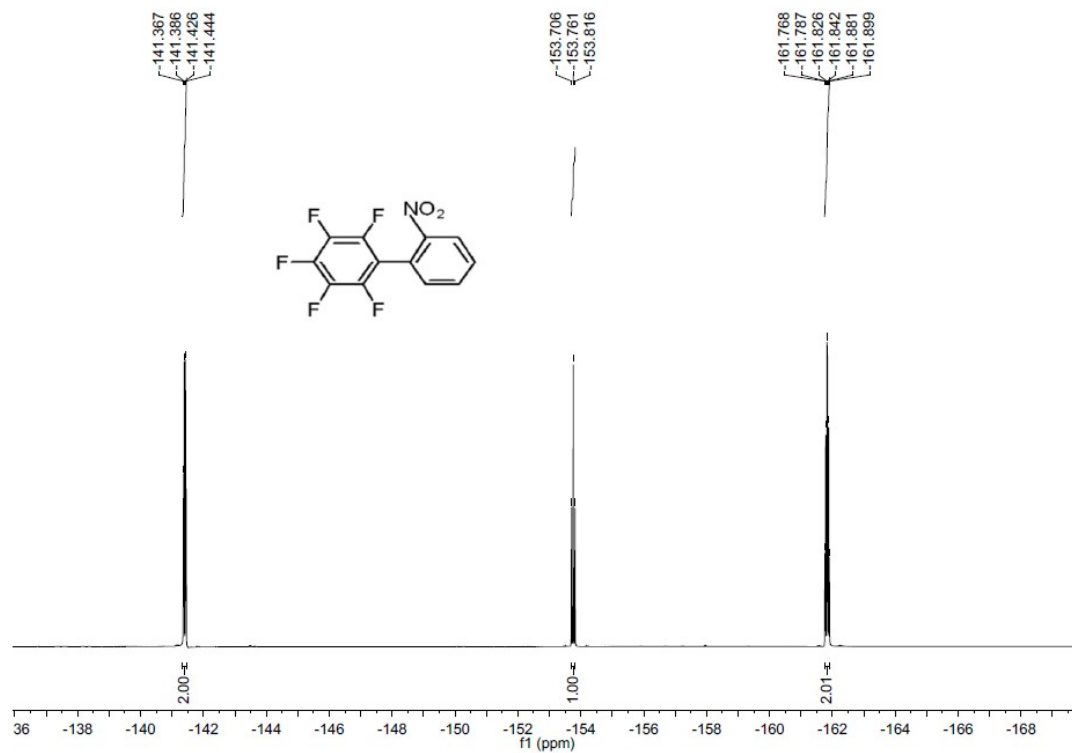
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3k**



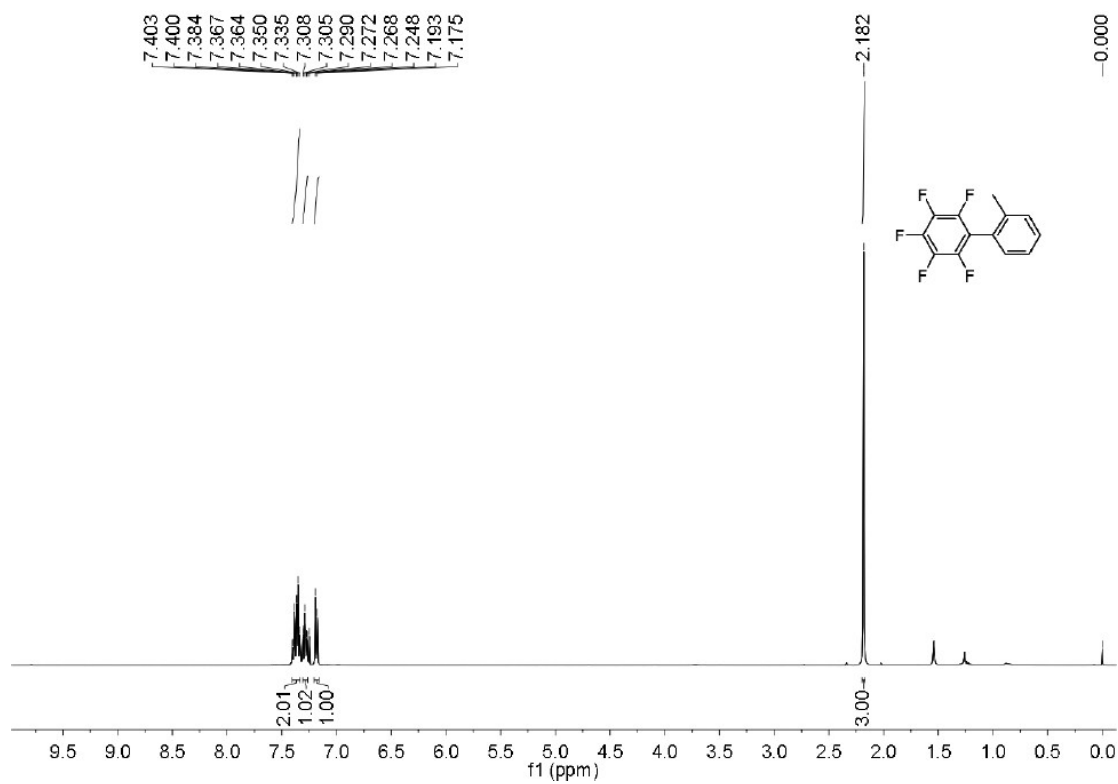


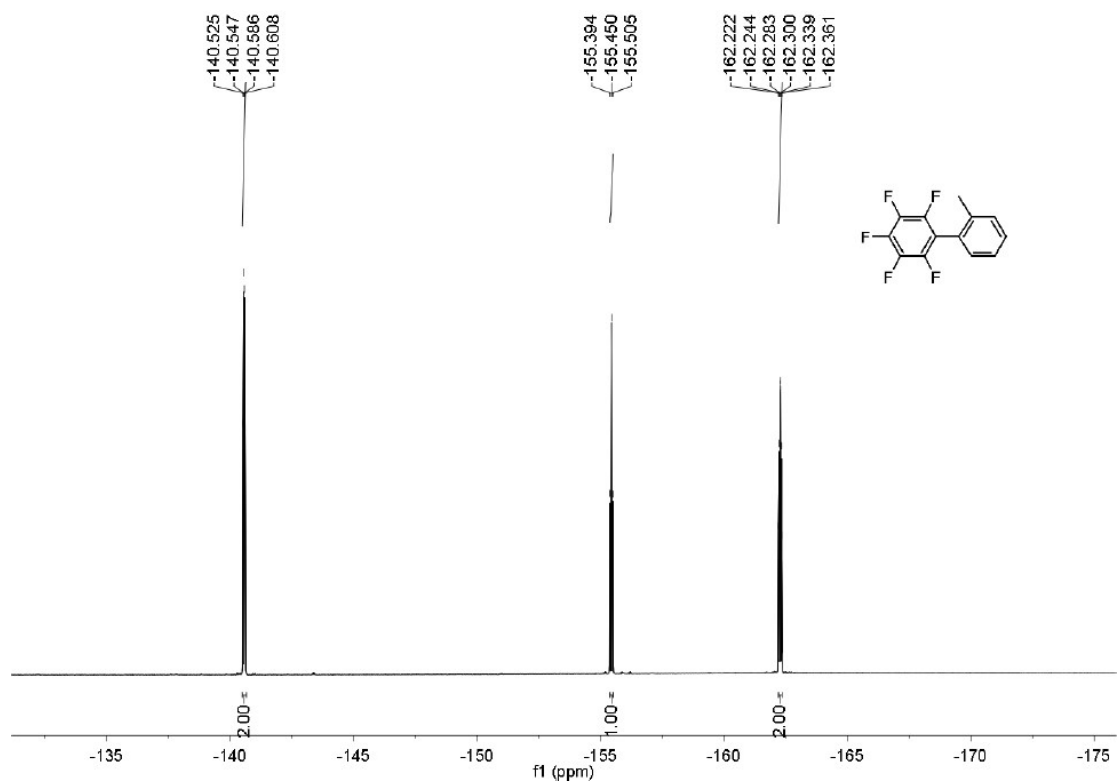
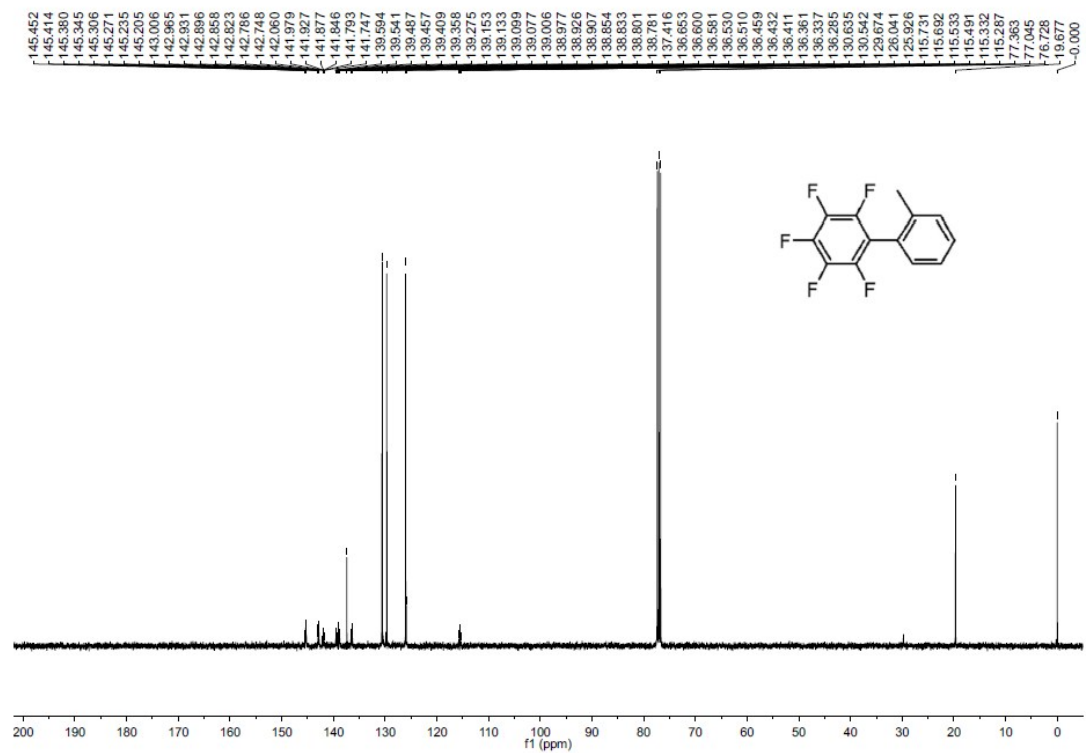
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **31**



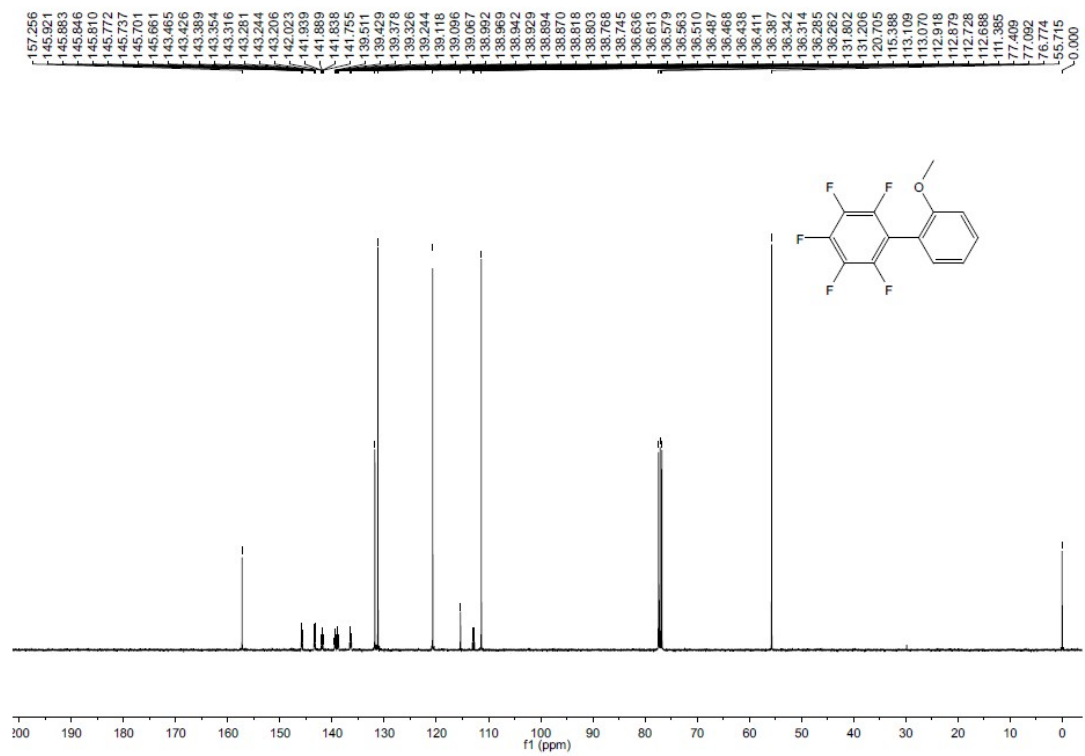
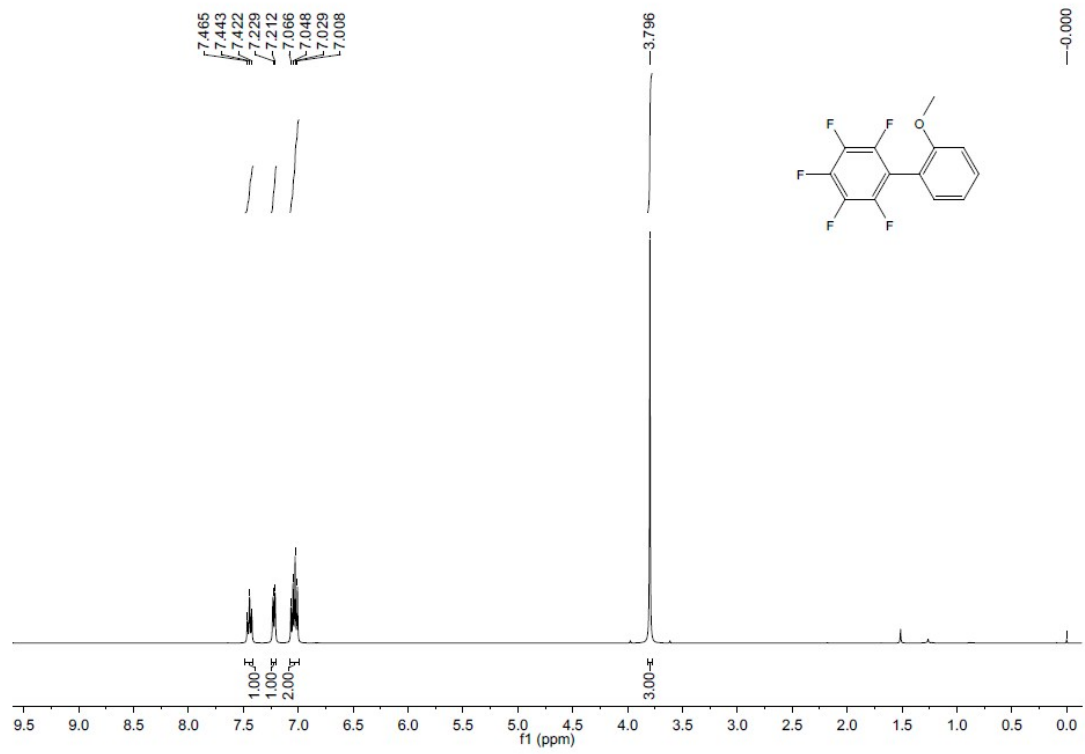


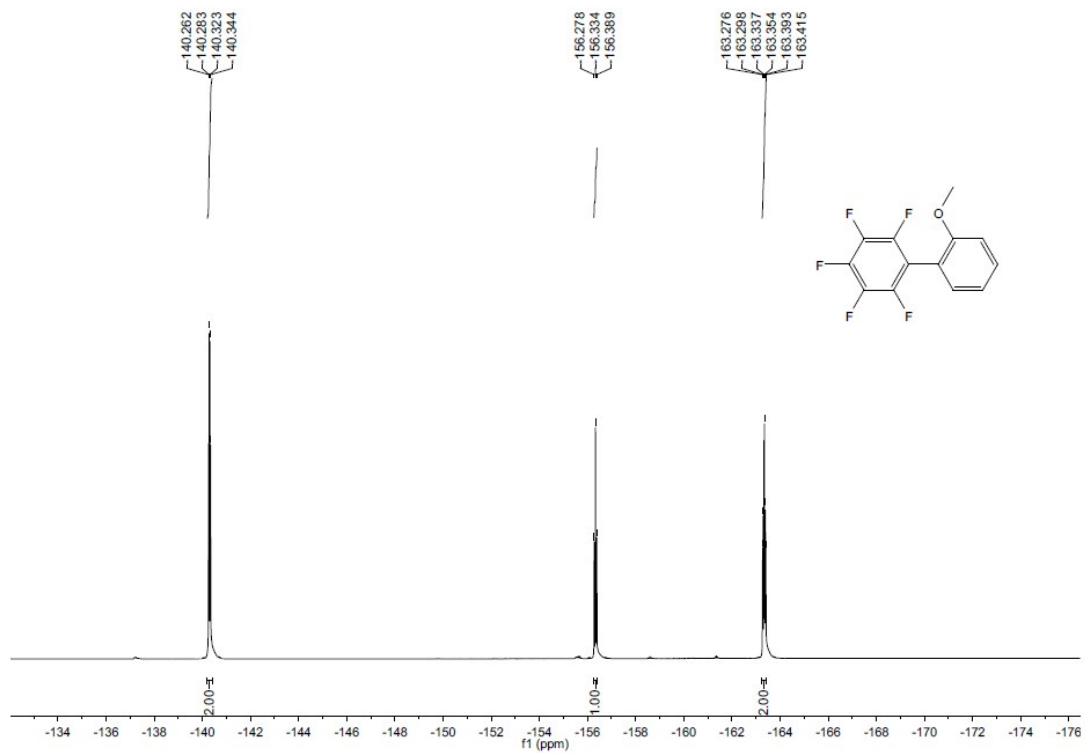
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3m**



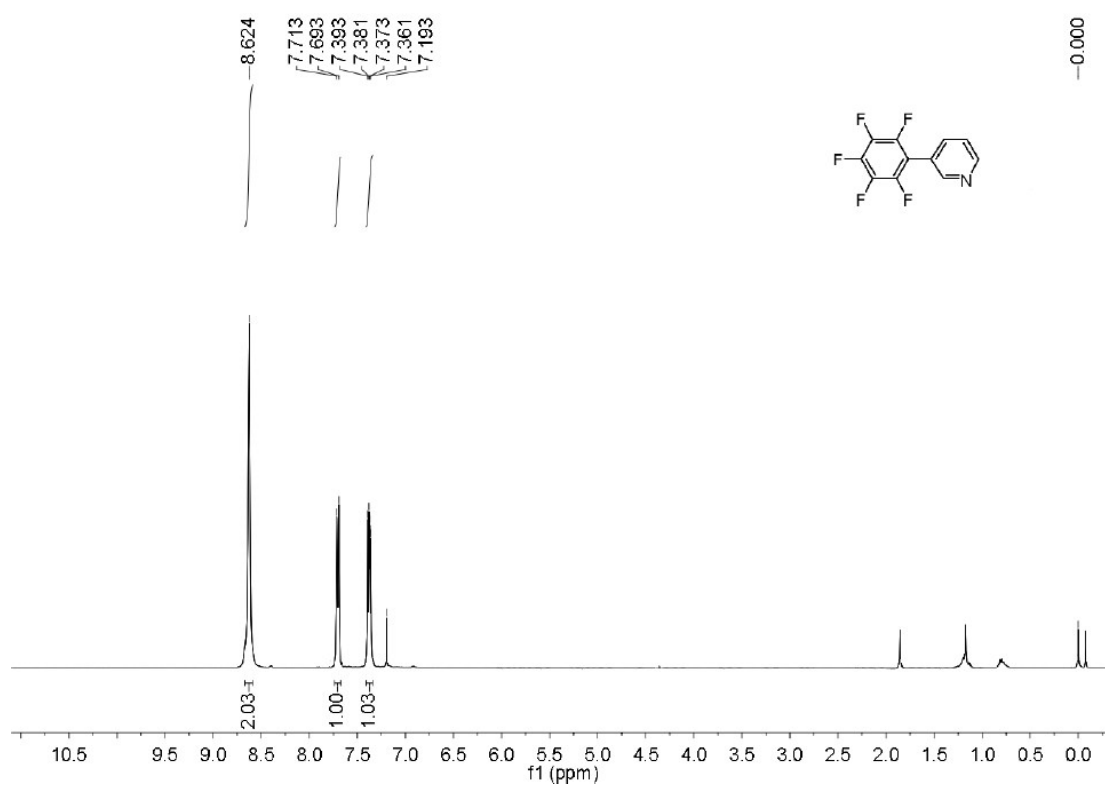


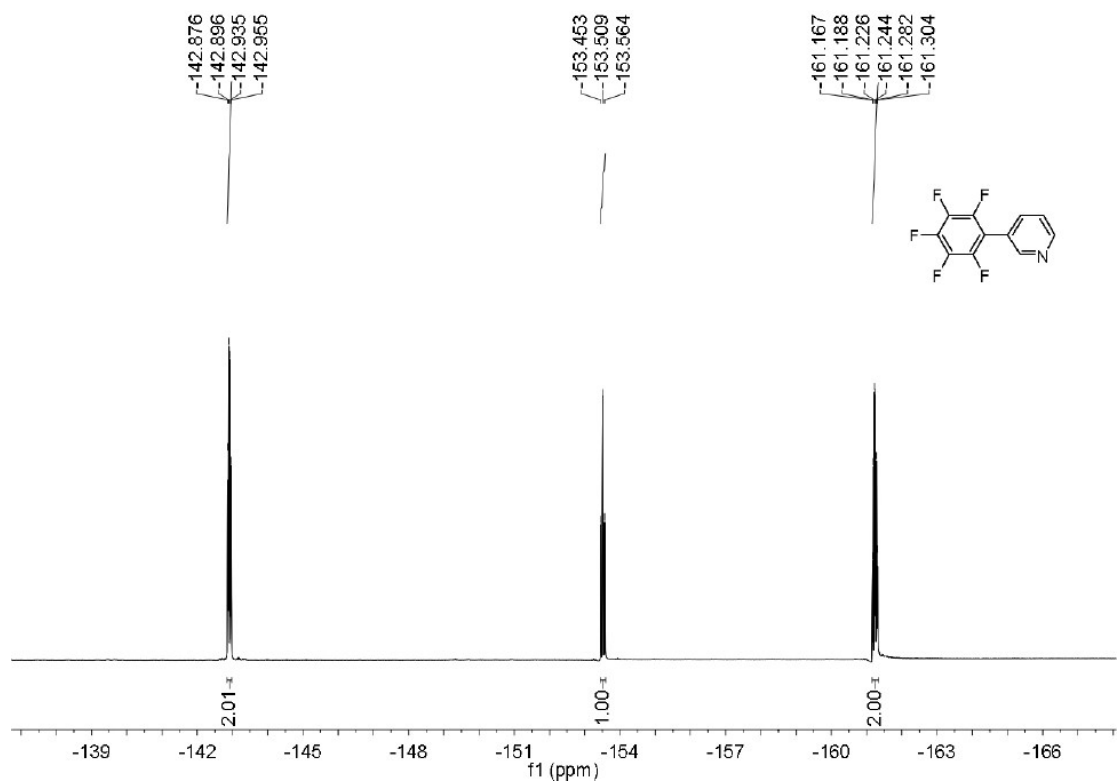
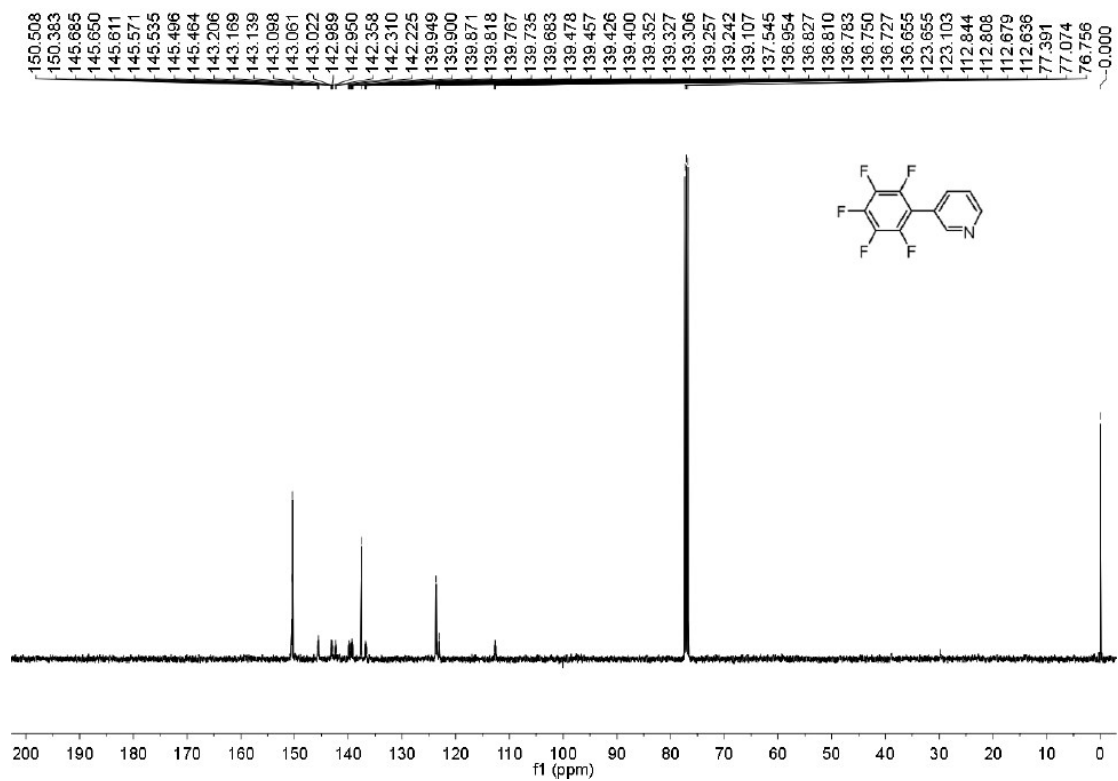
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3n**



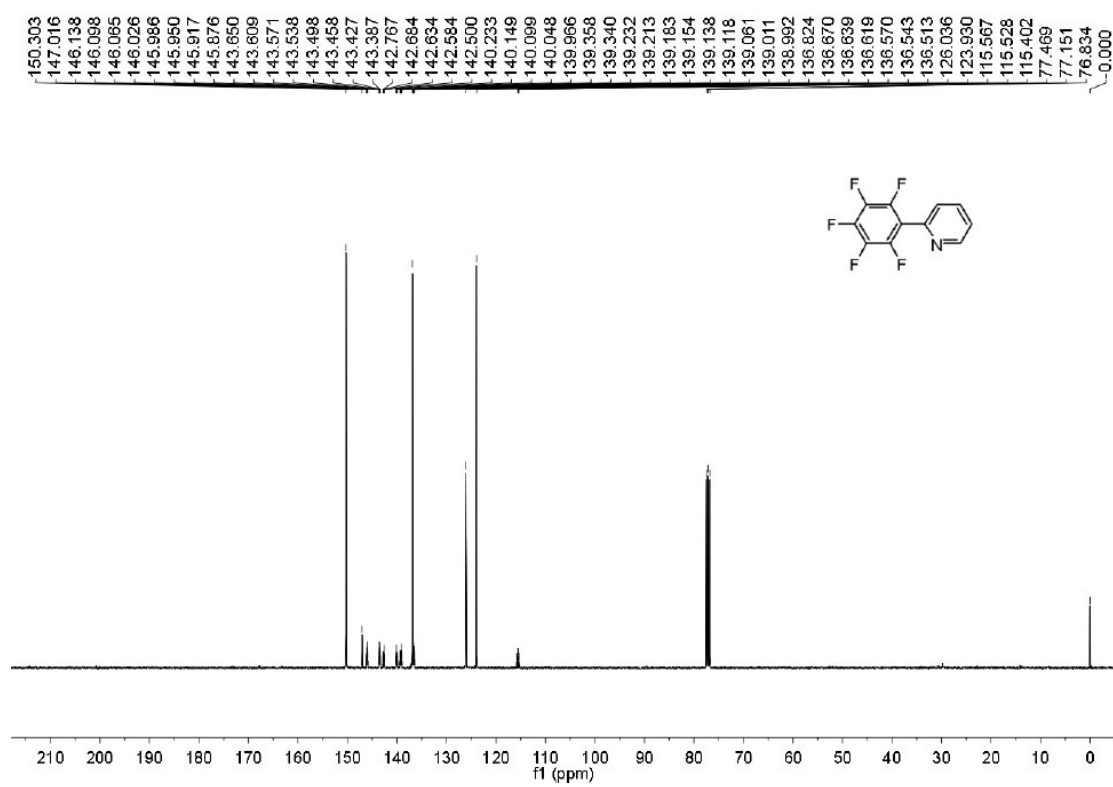
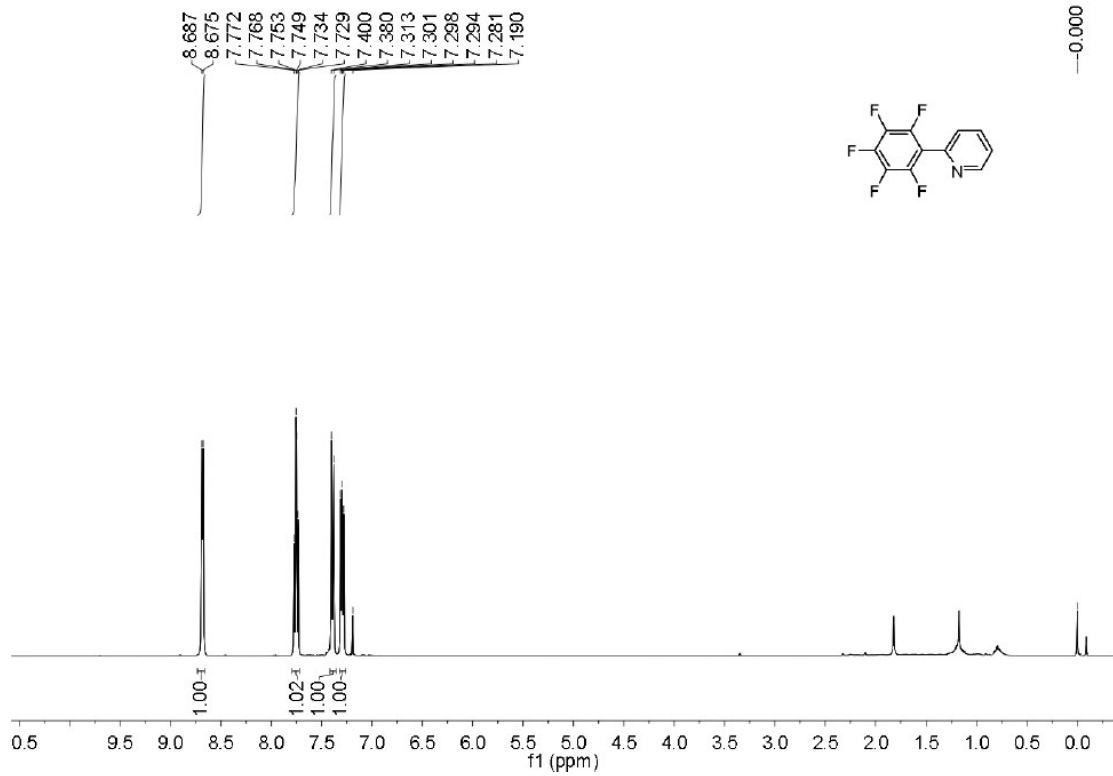


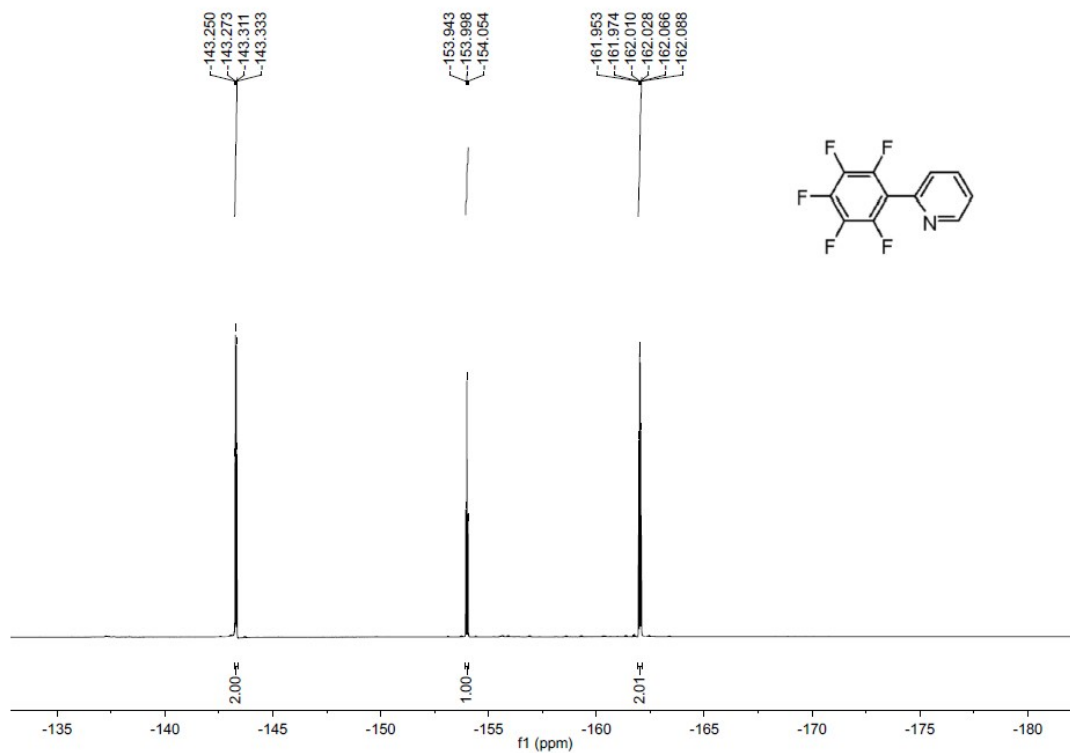
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **30**



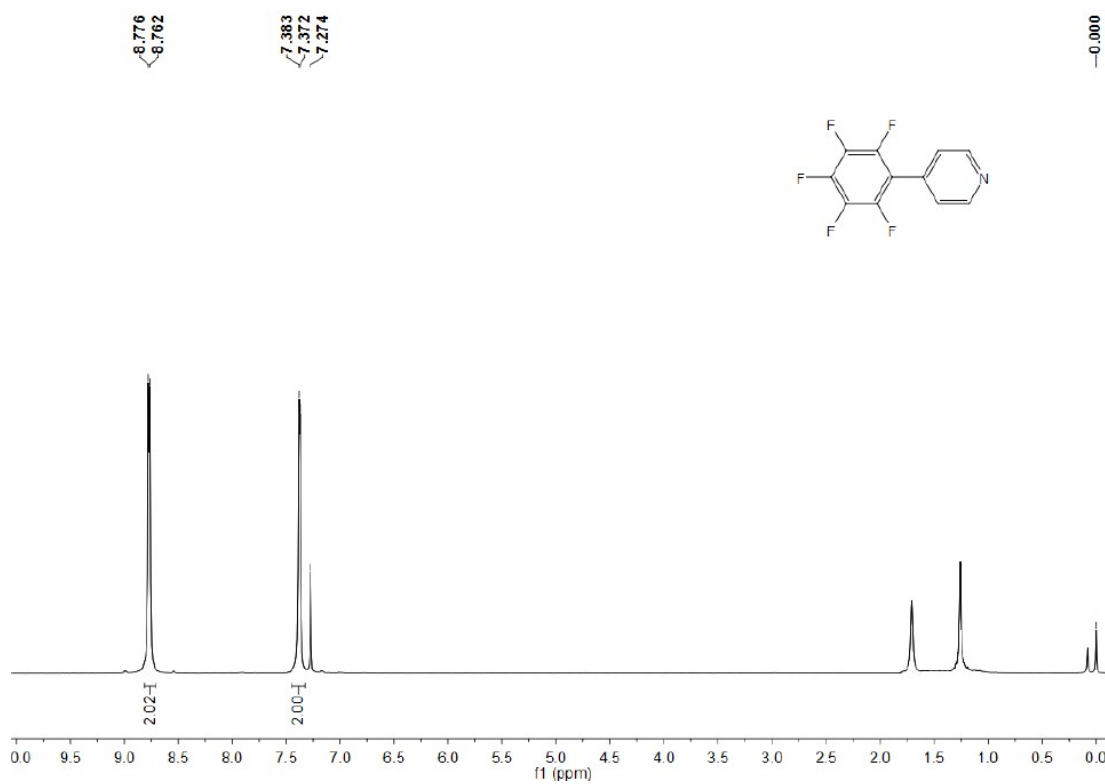


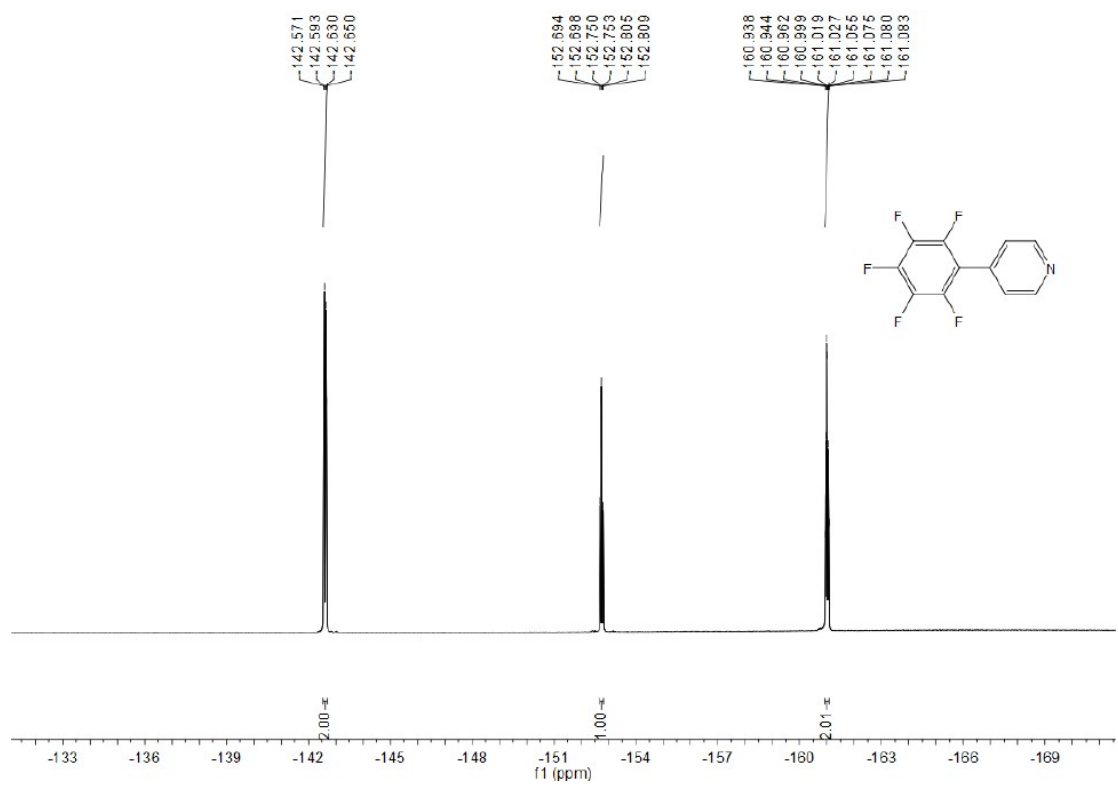
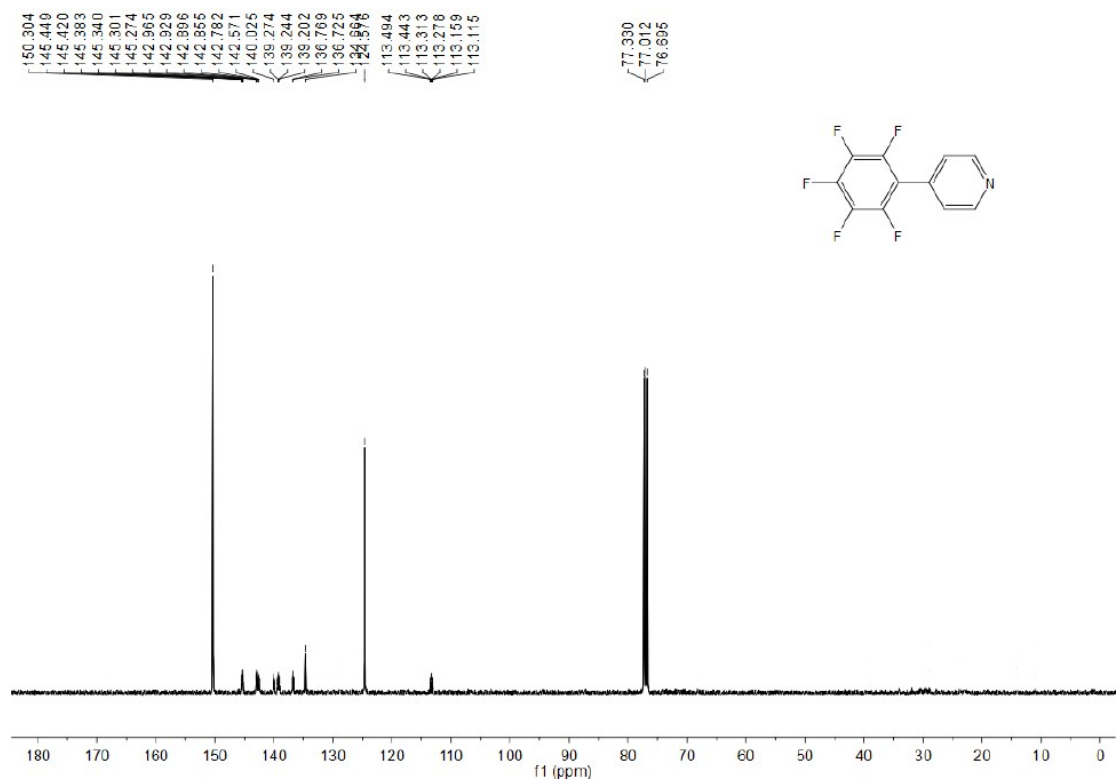
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3p**



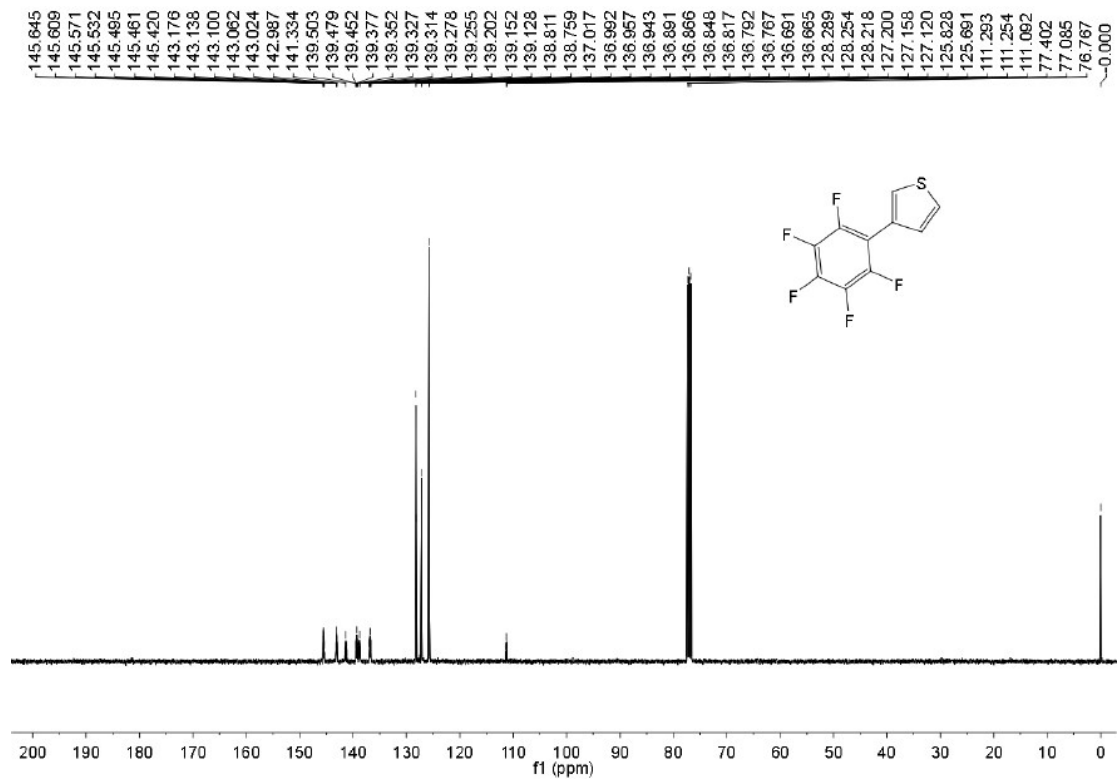
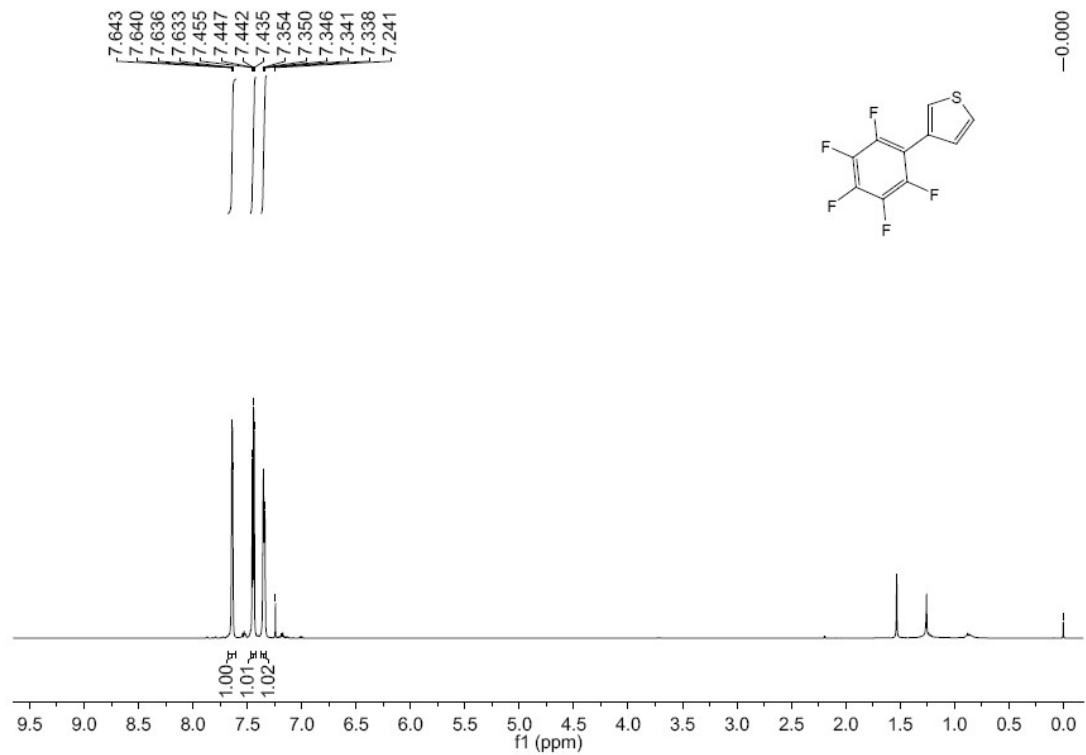


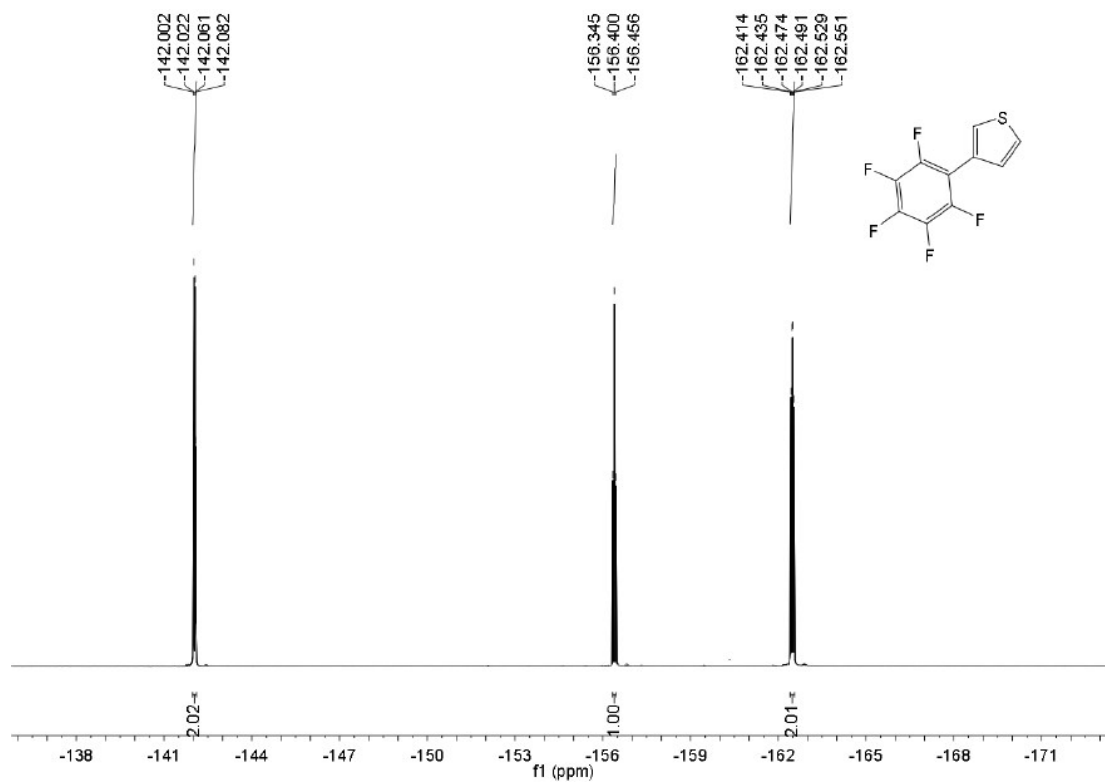
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3q**



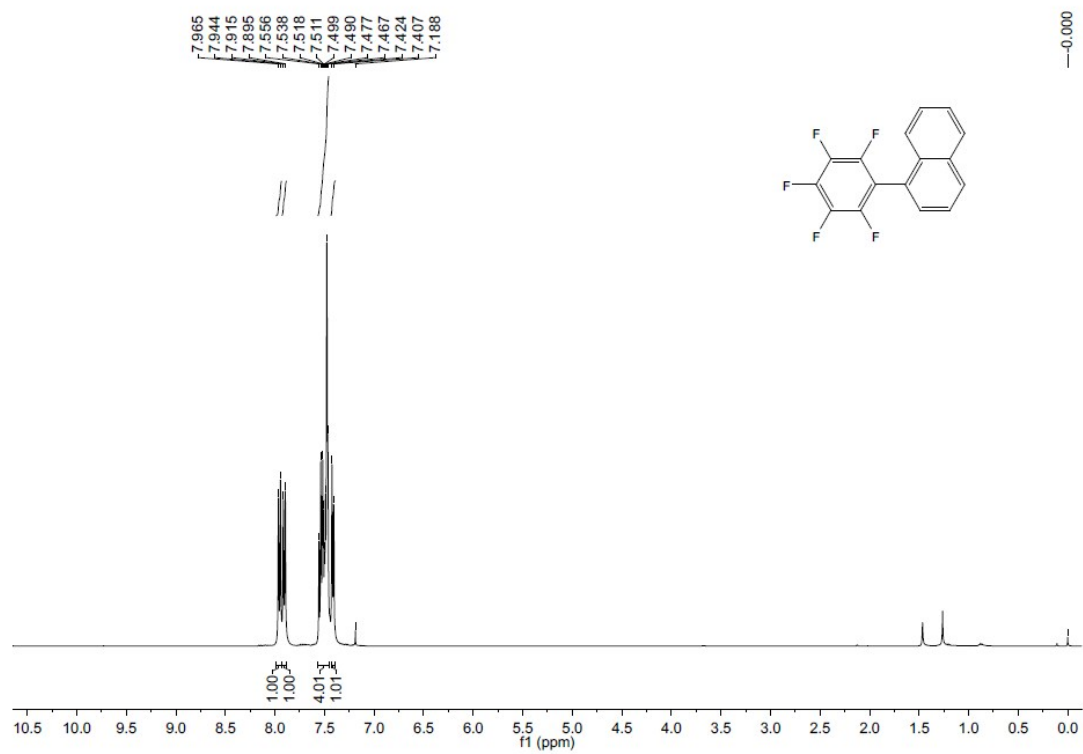


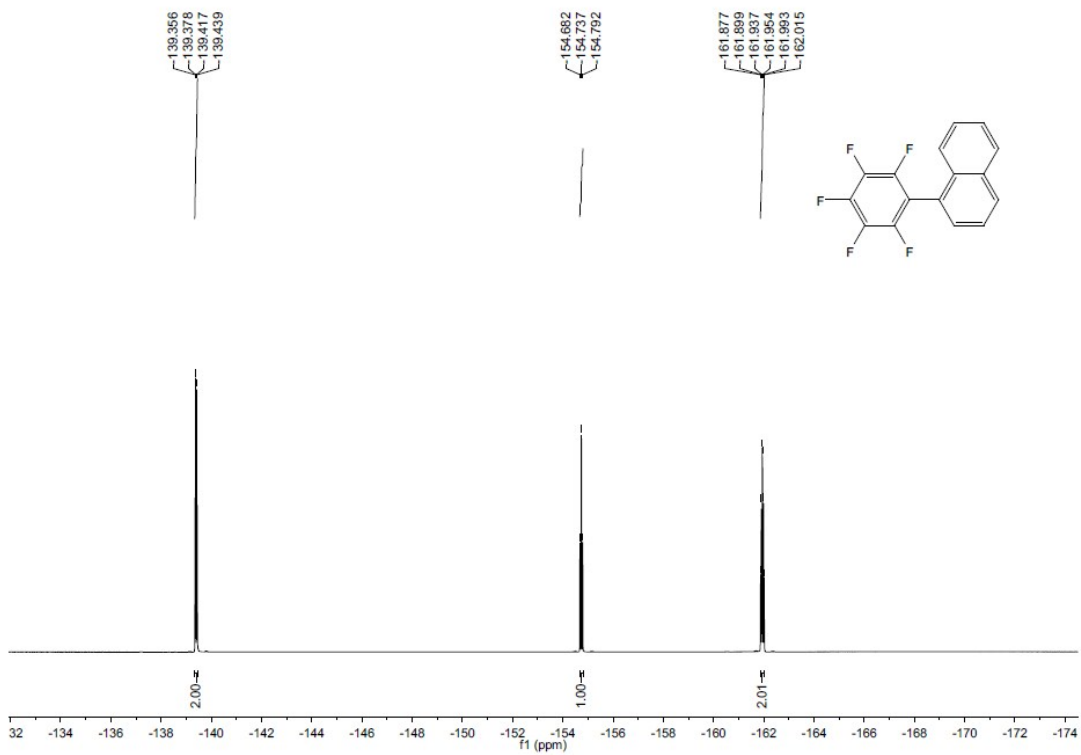
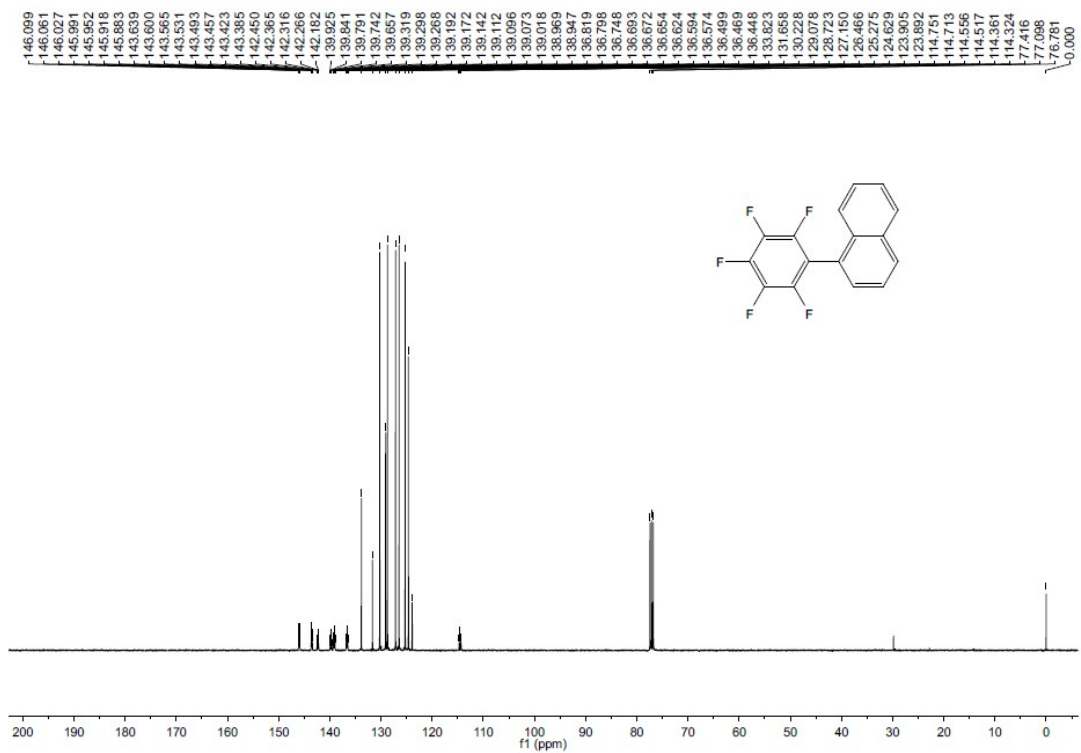
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3r**



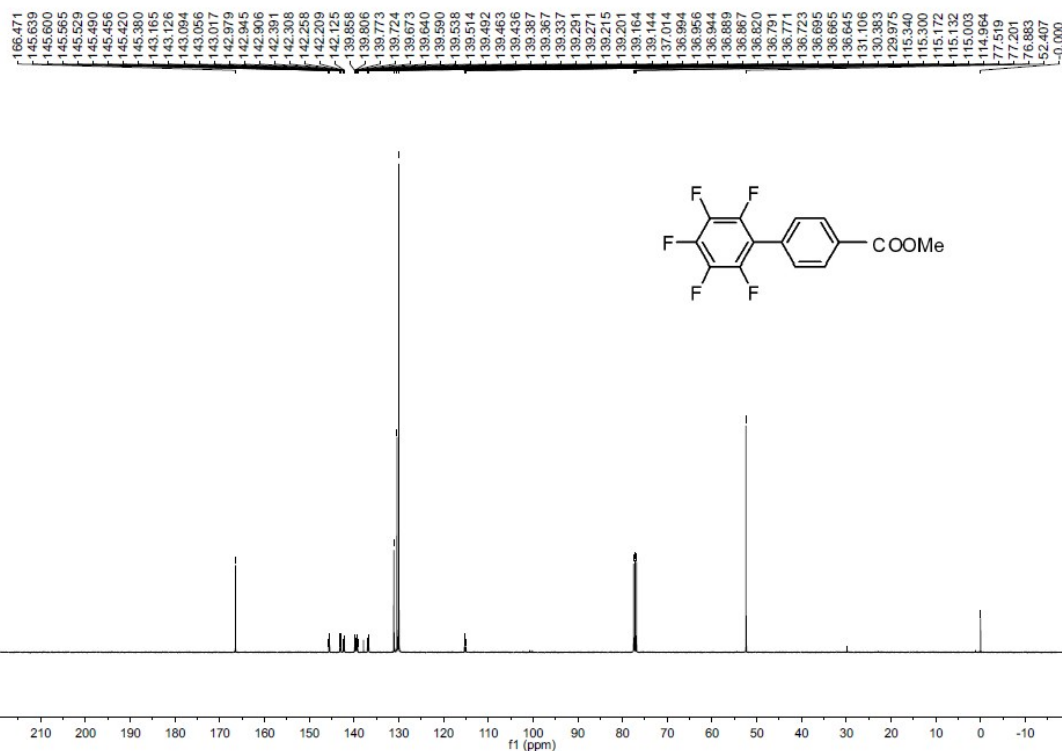
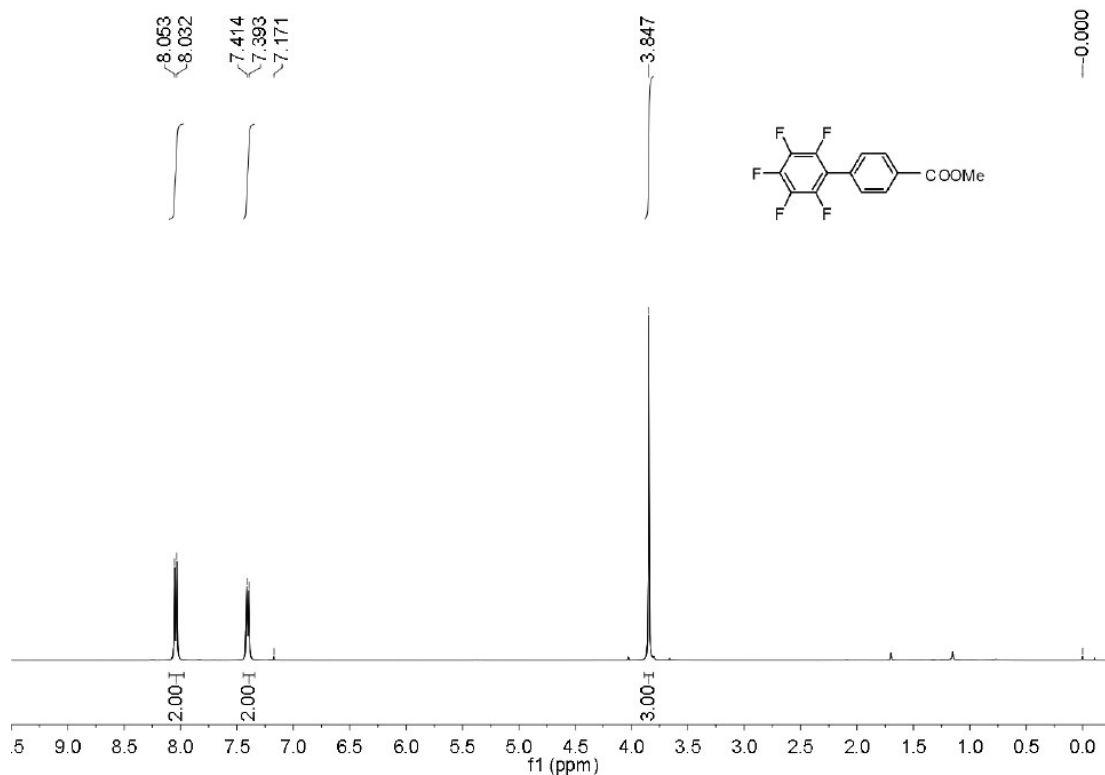


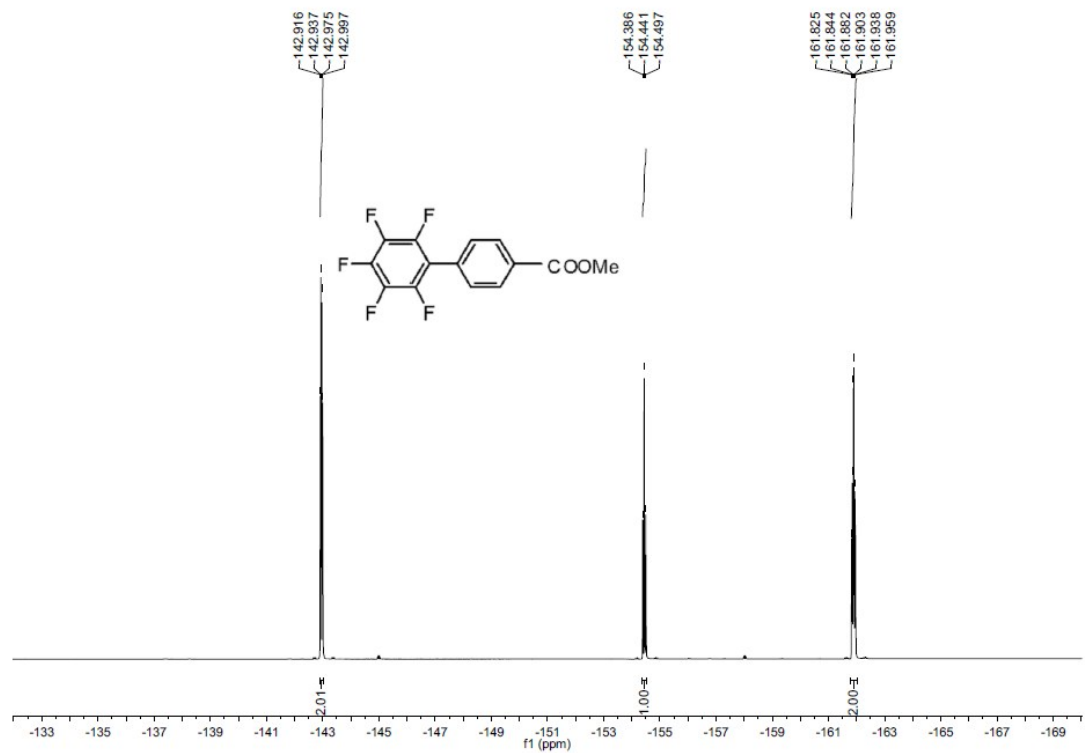
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3s**



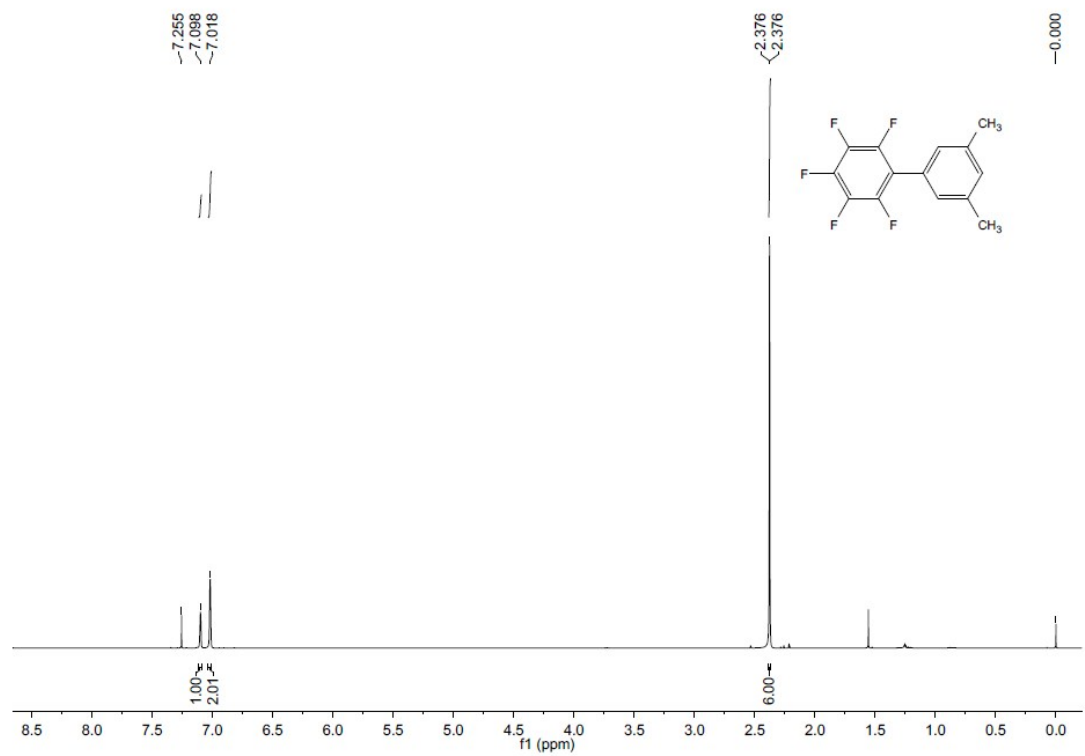


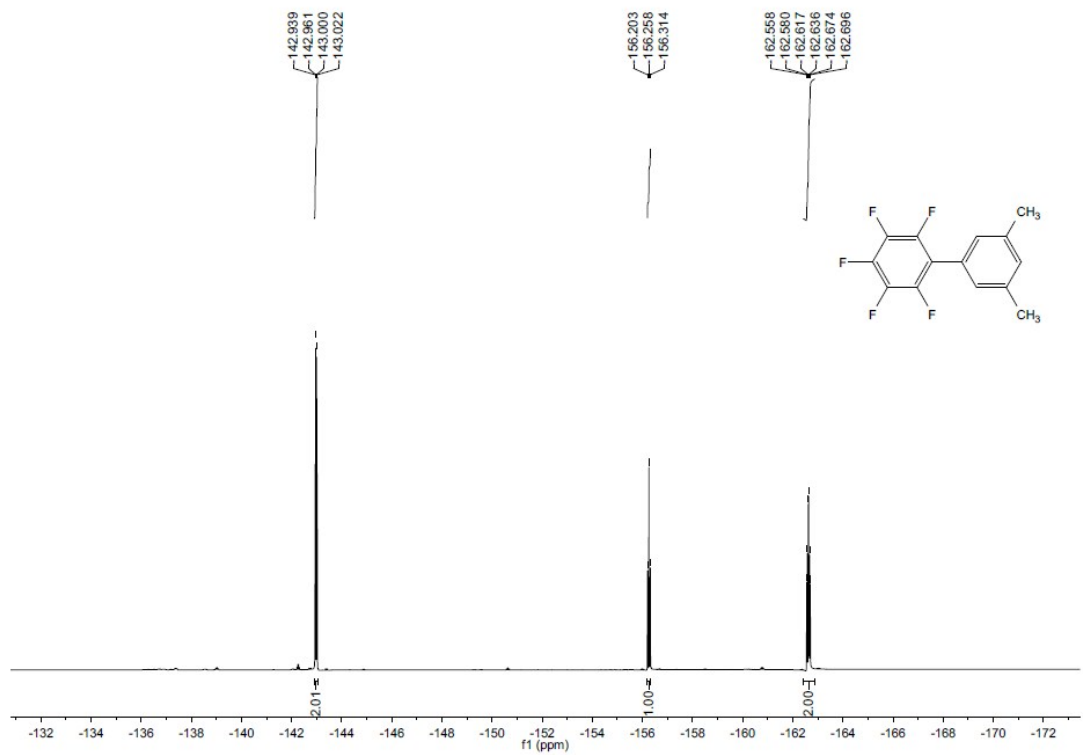
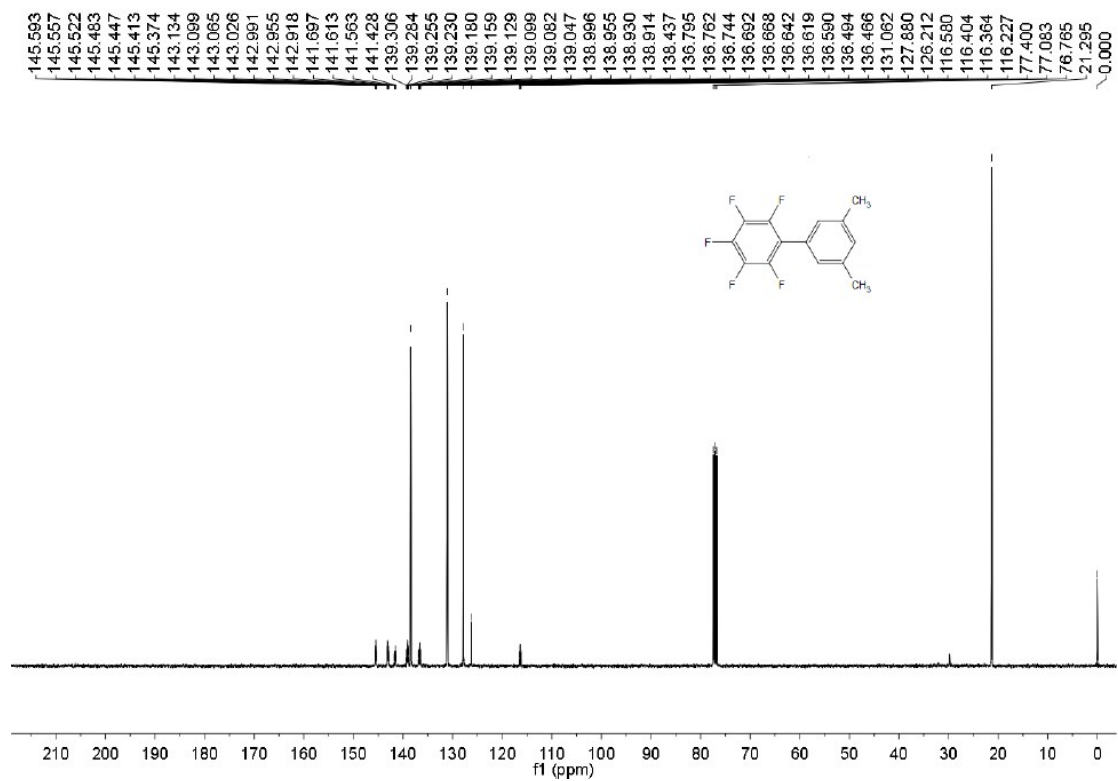
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **3t**



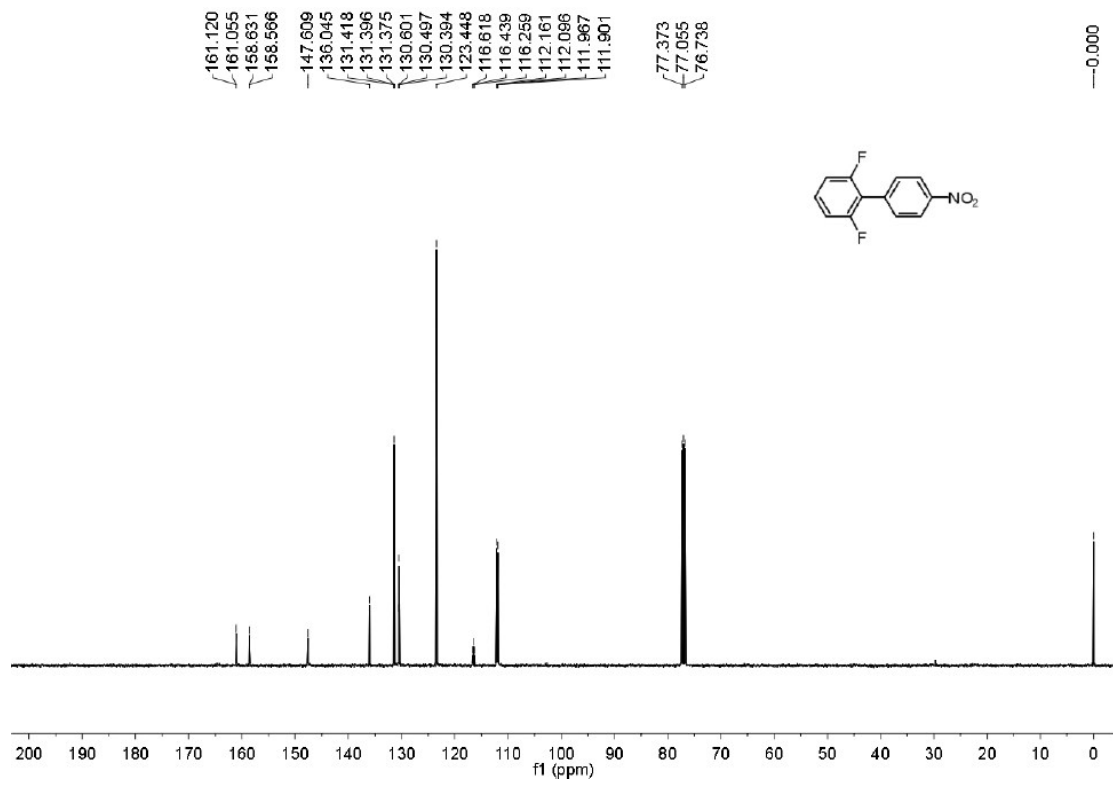
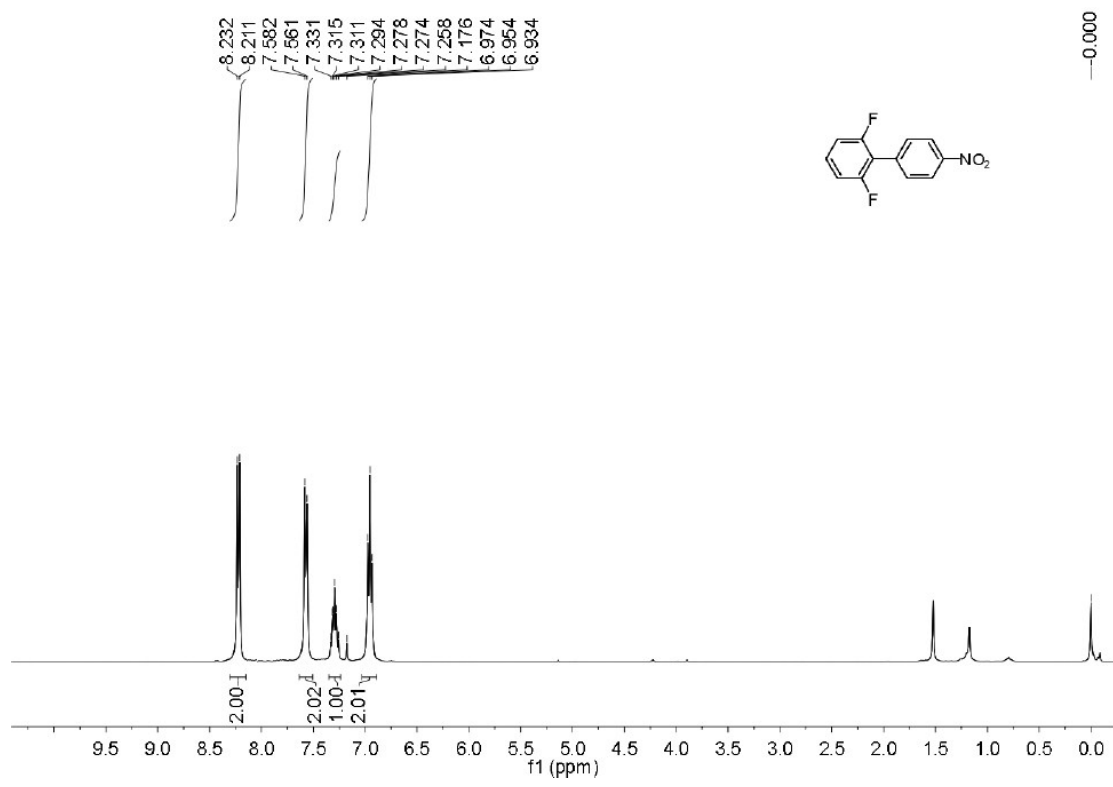


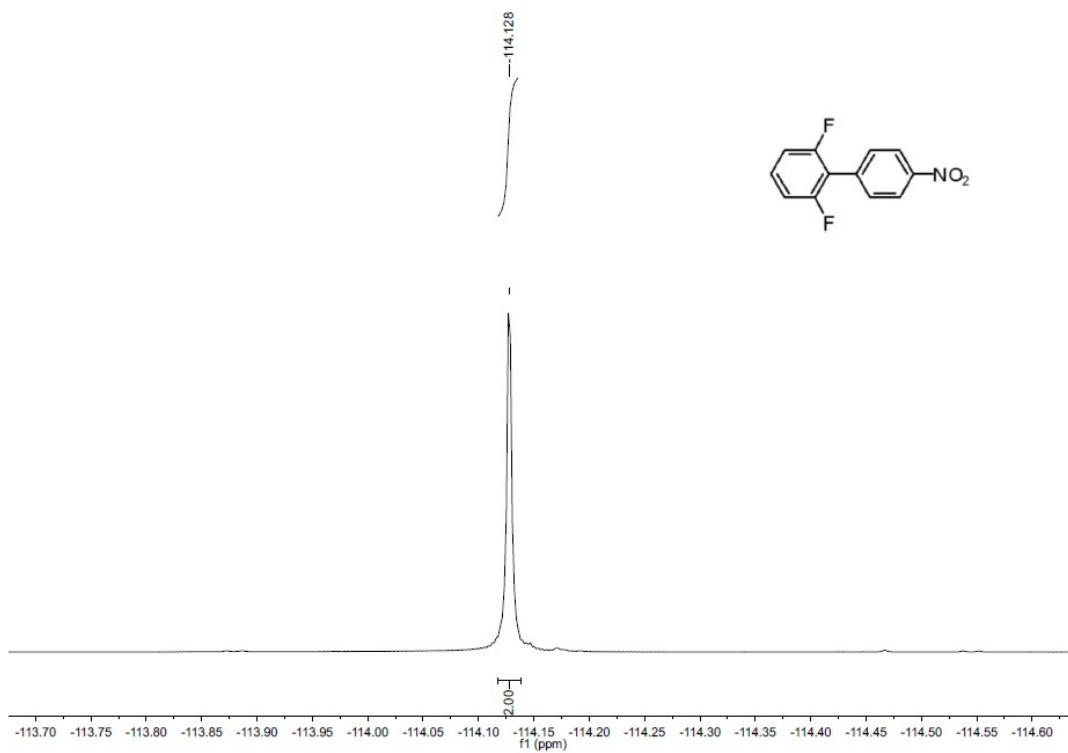
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3u**



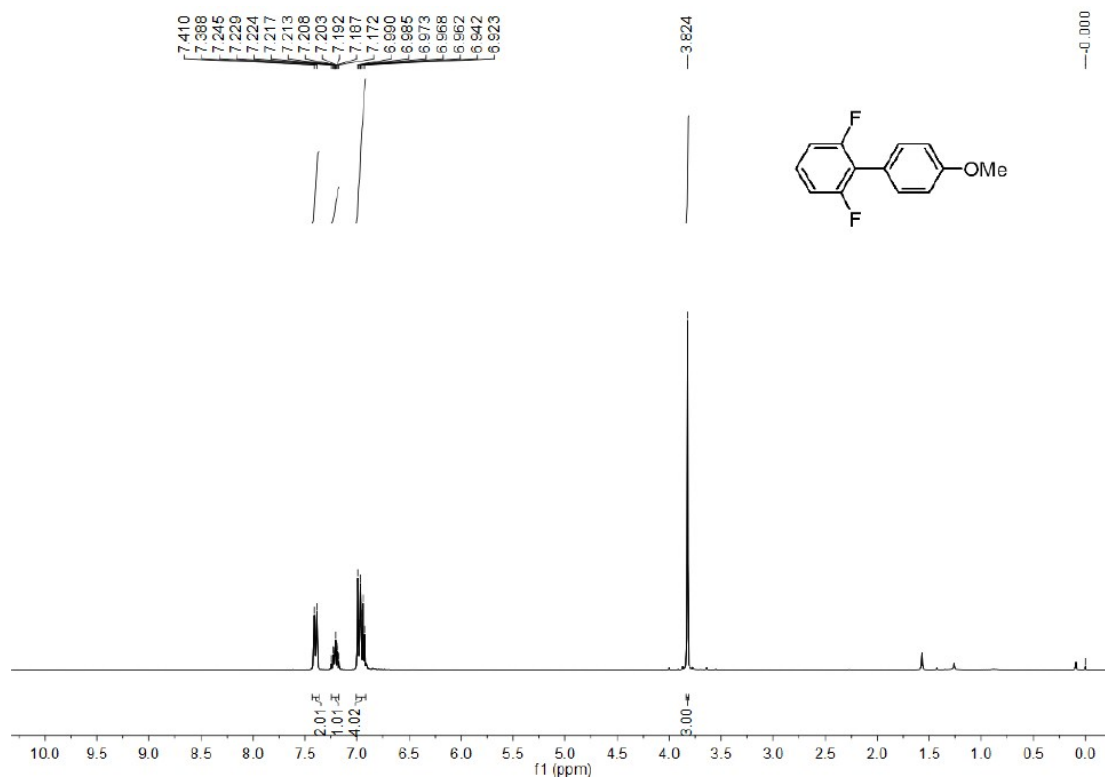


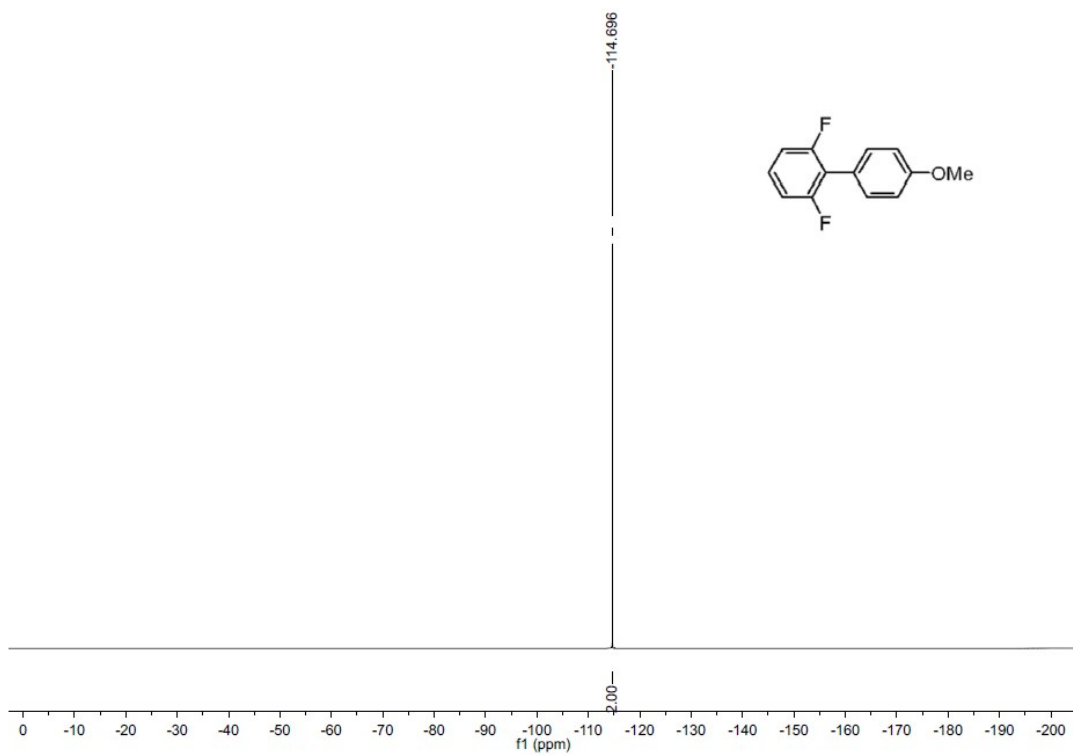
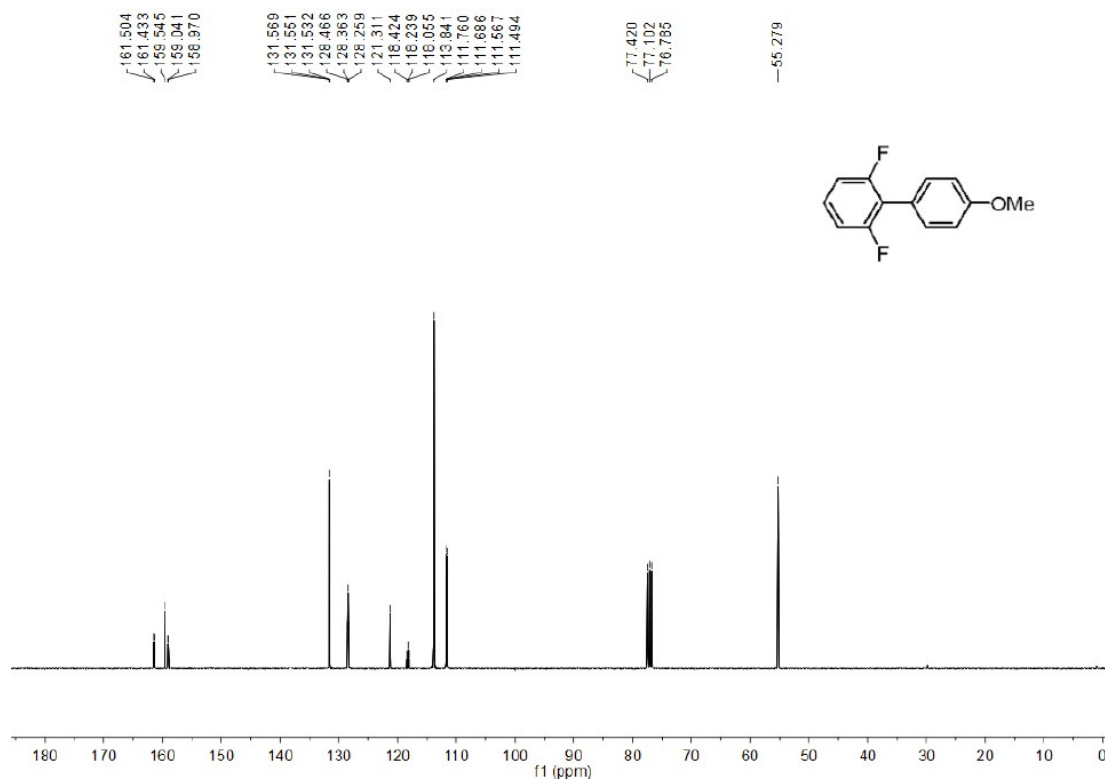
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **3v**



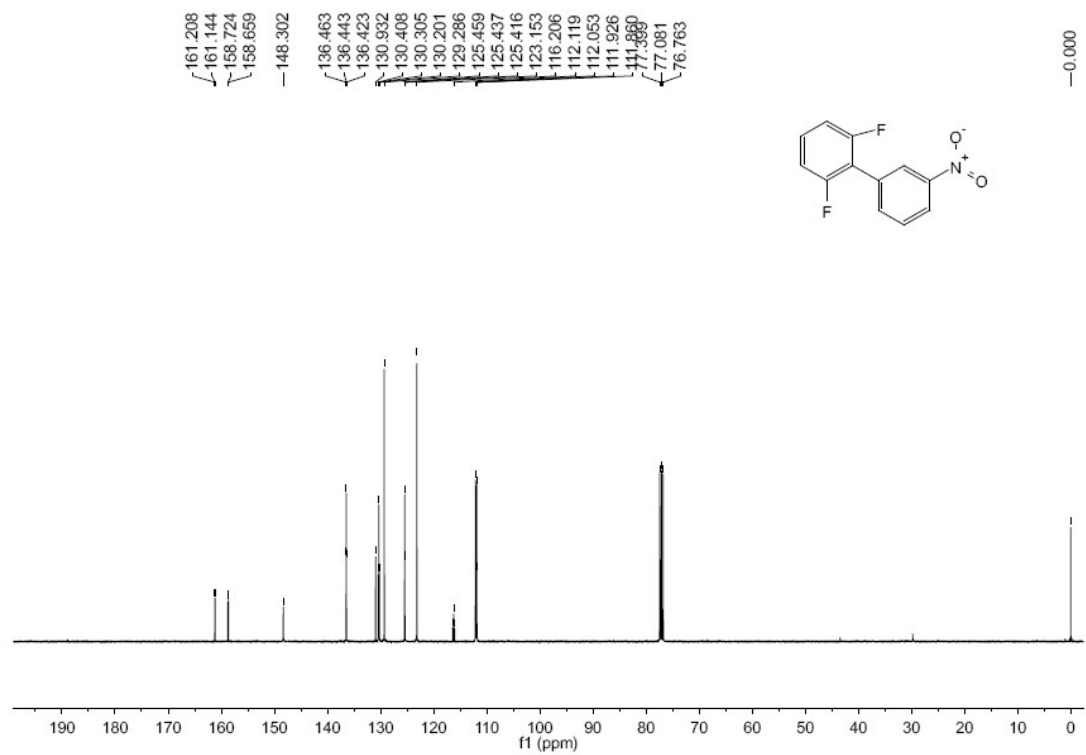
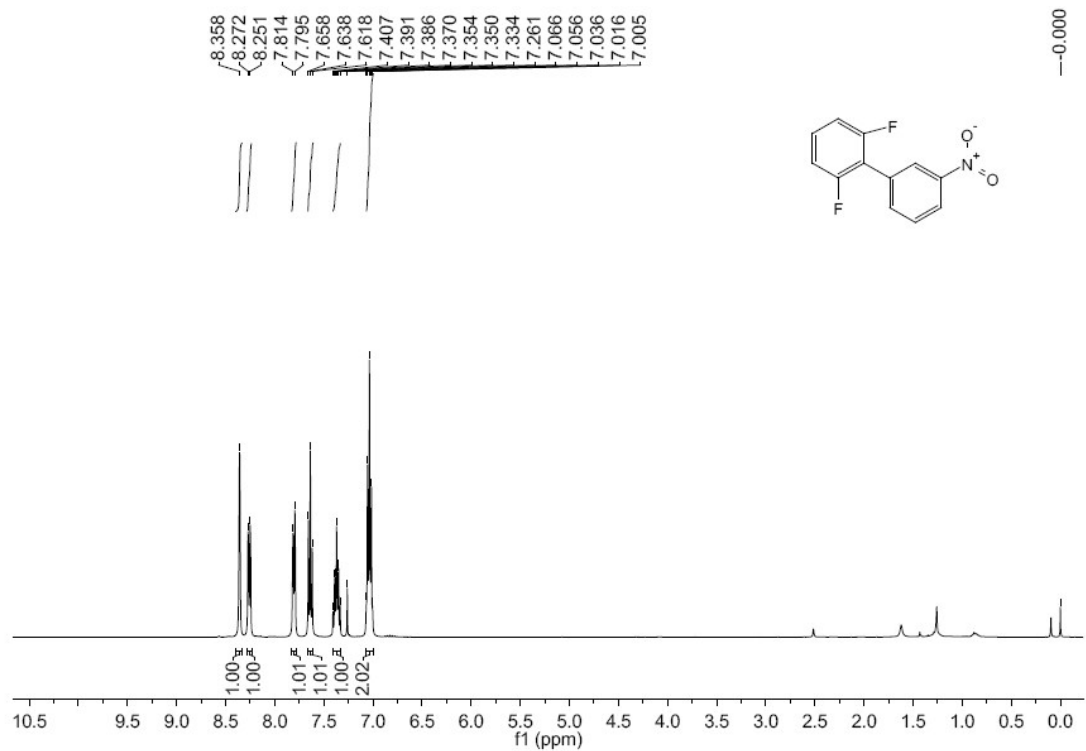


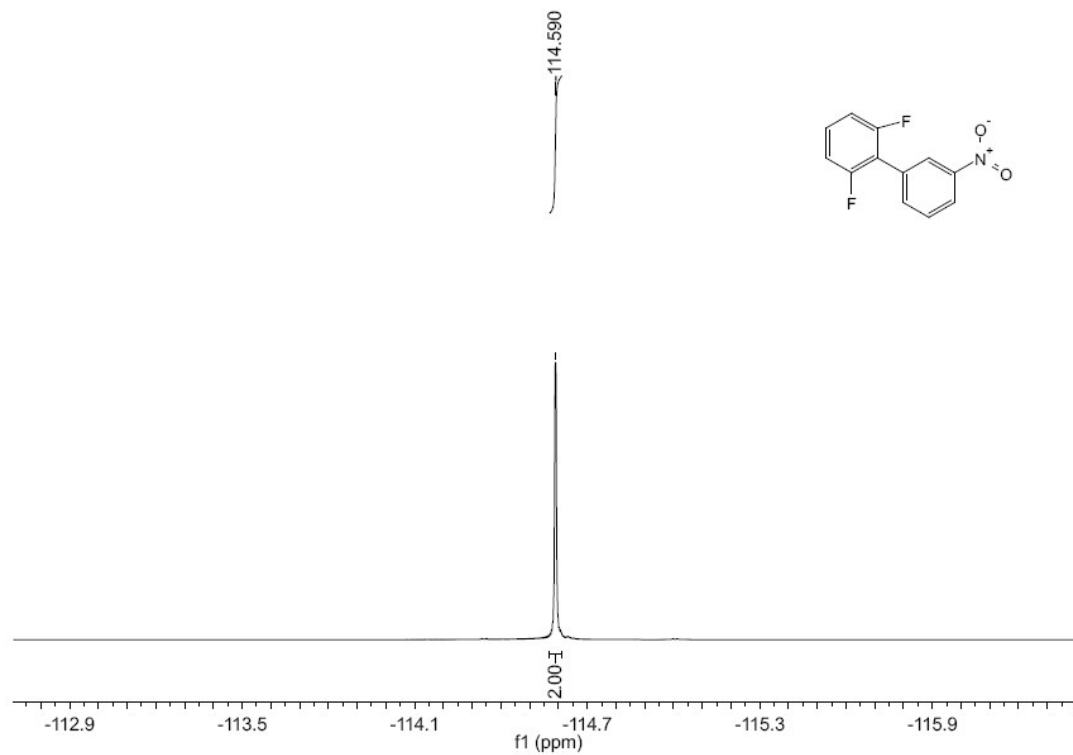
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5a**



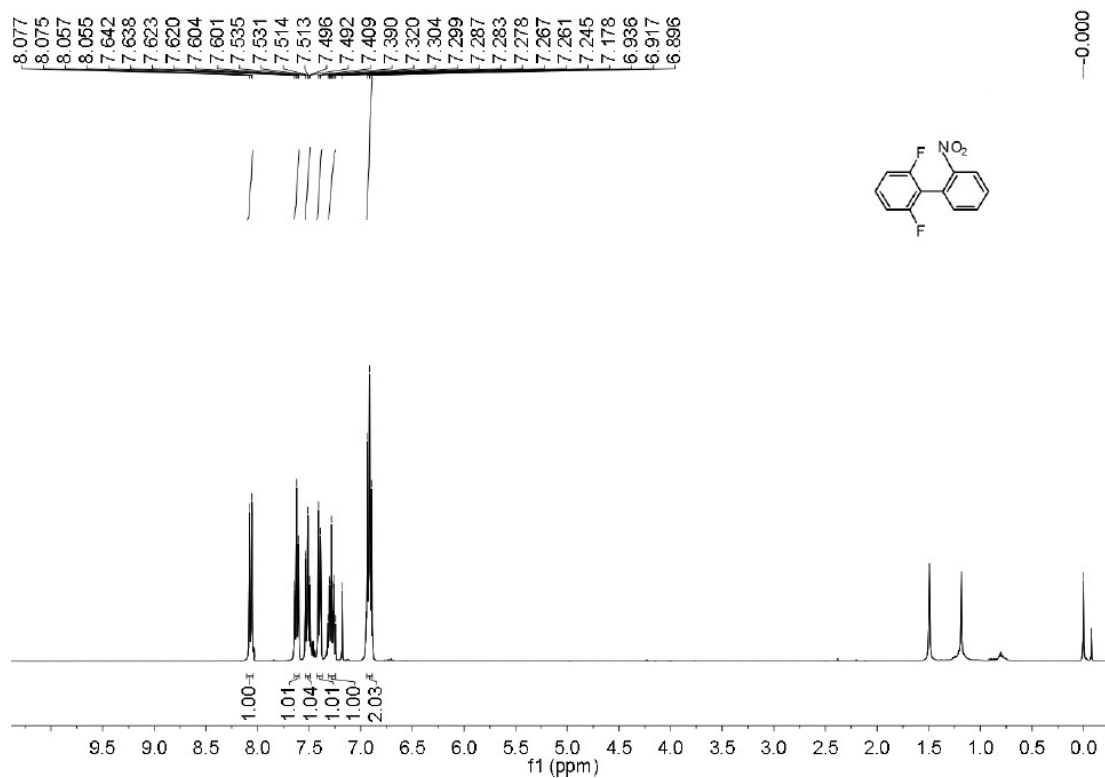


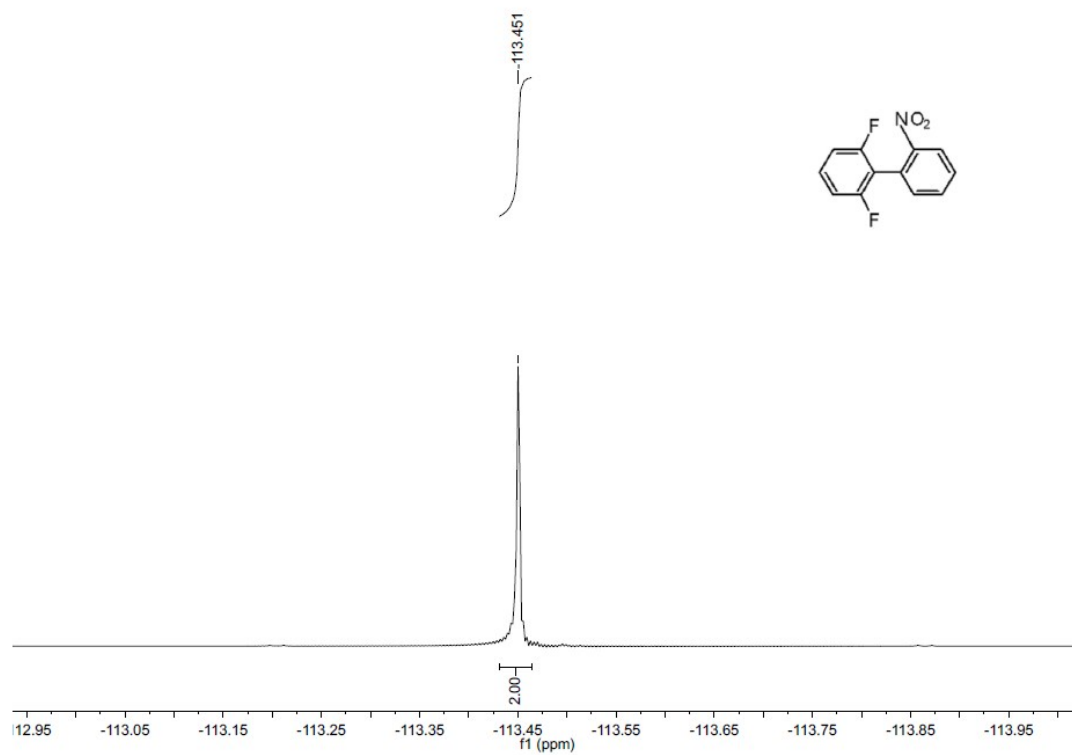
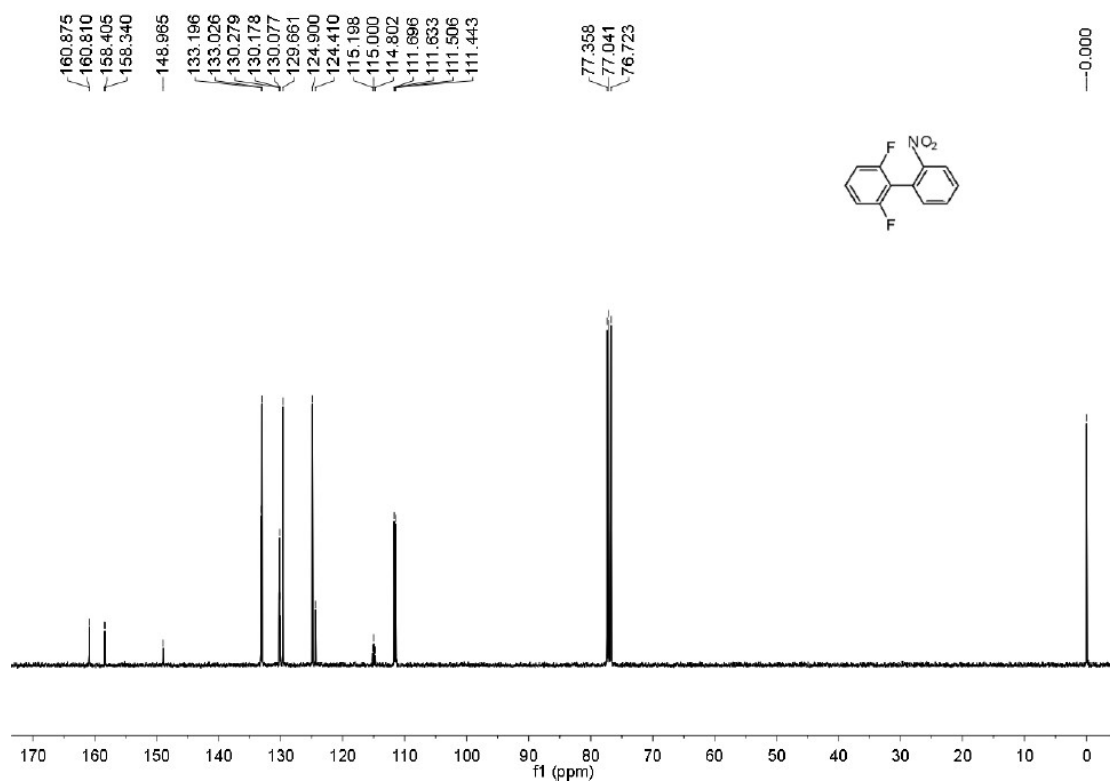
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5b**



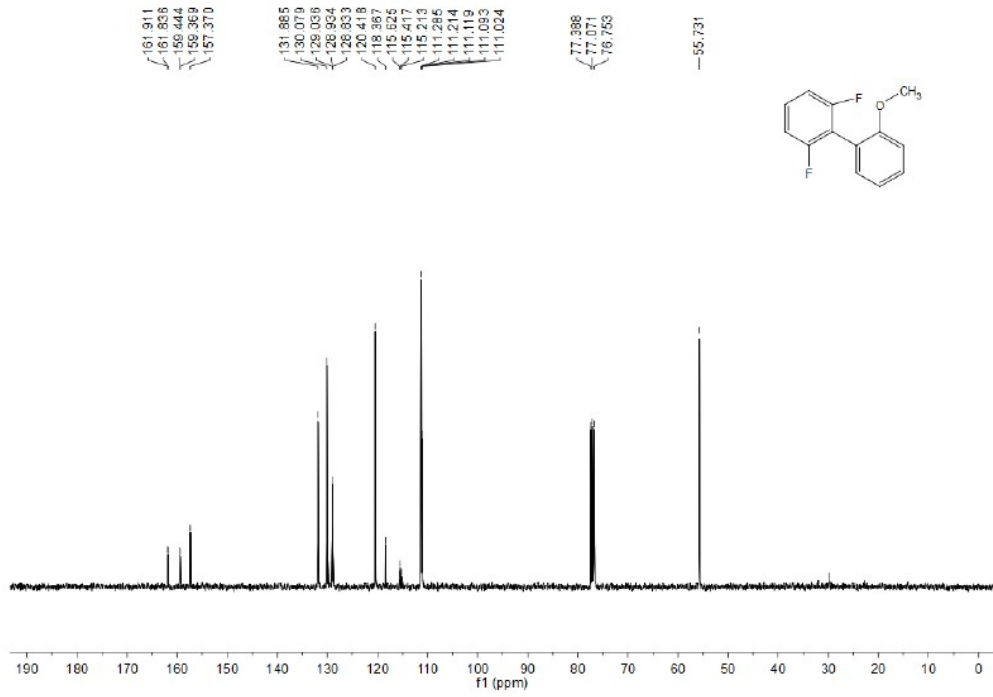
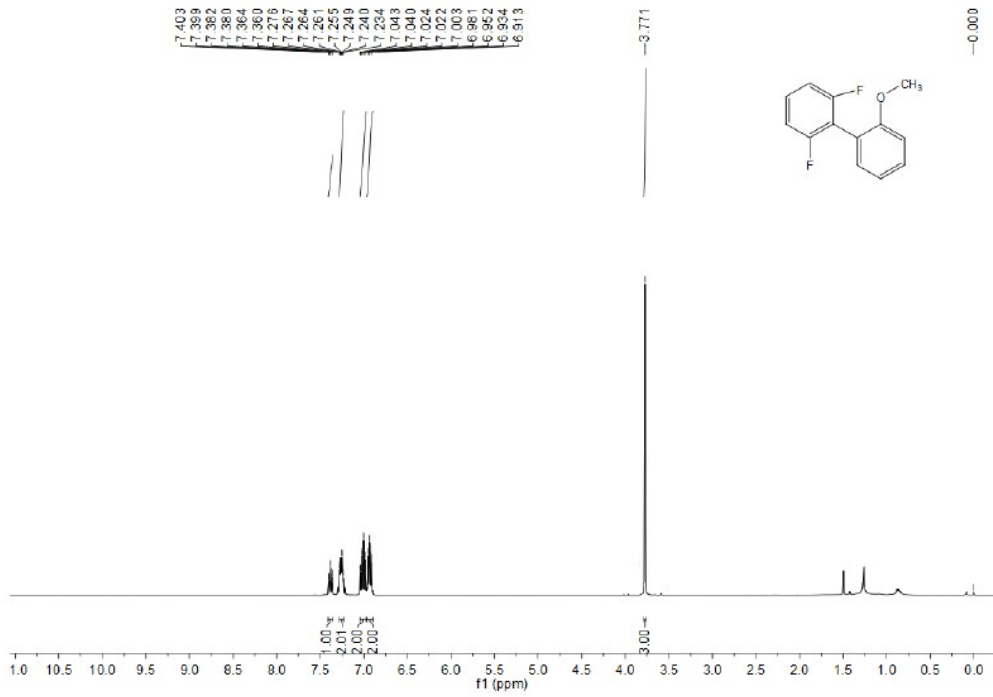


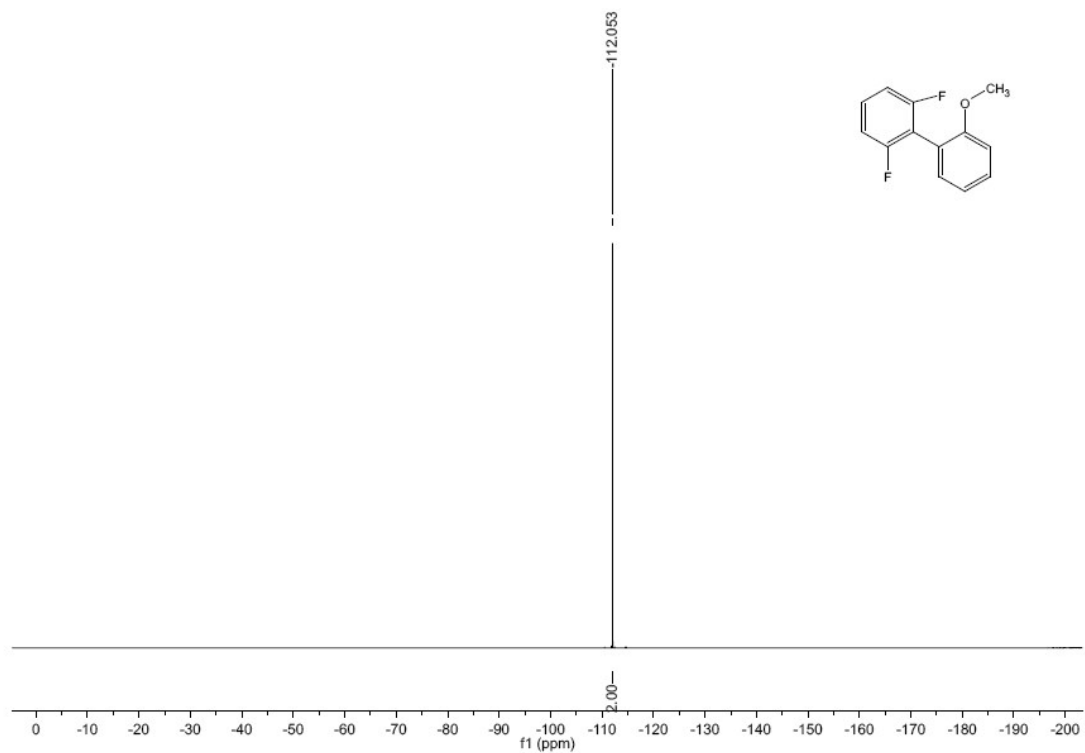
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5c**



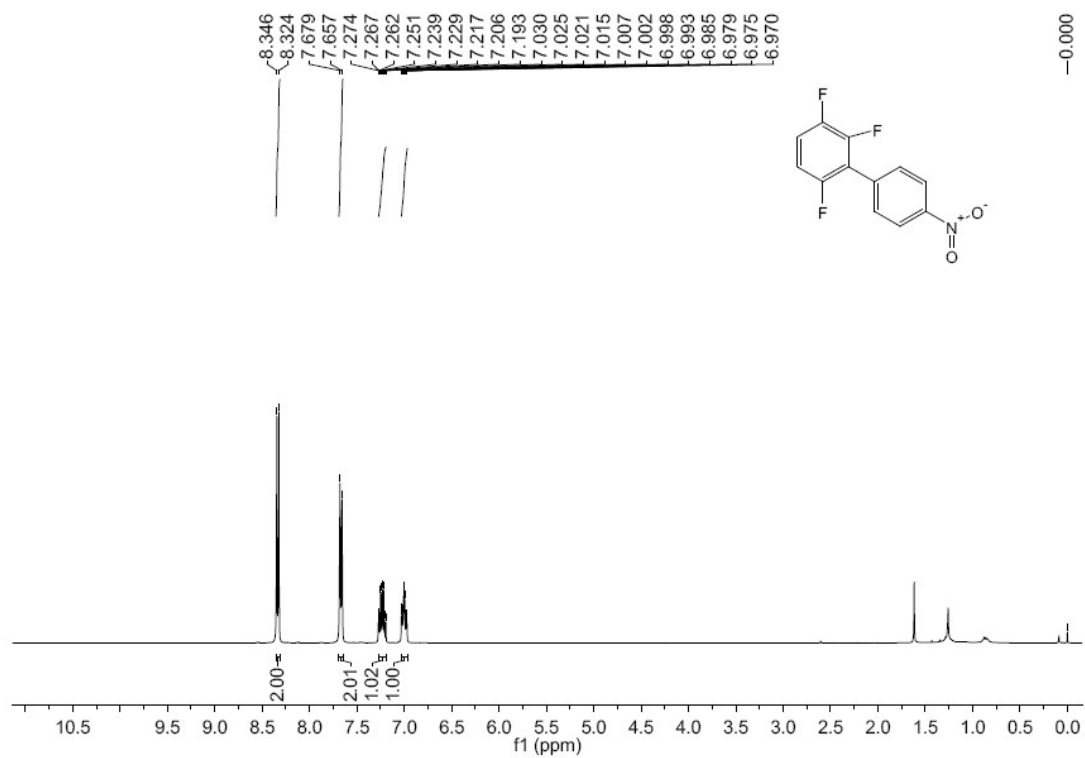


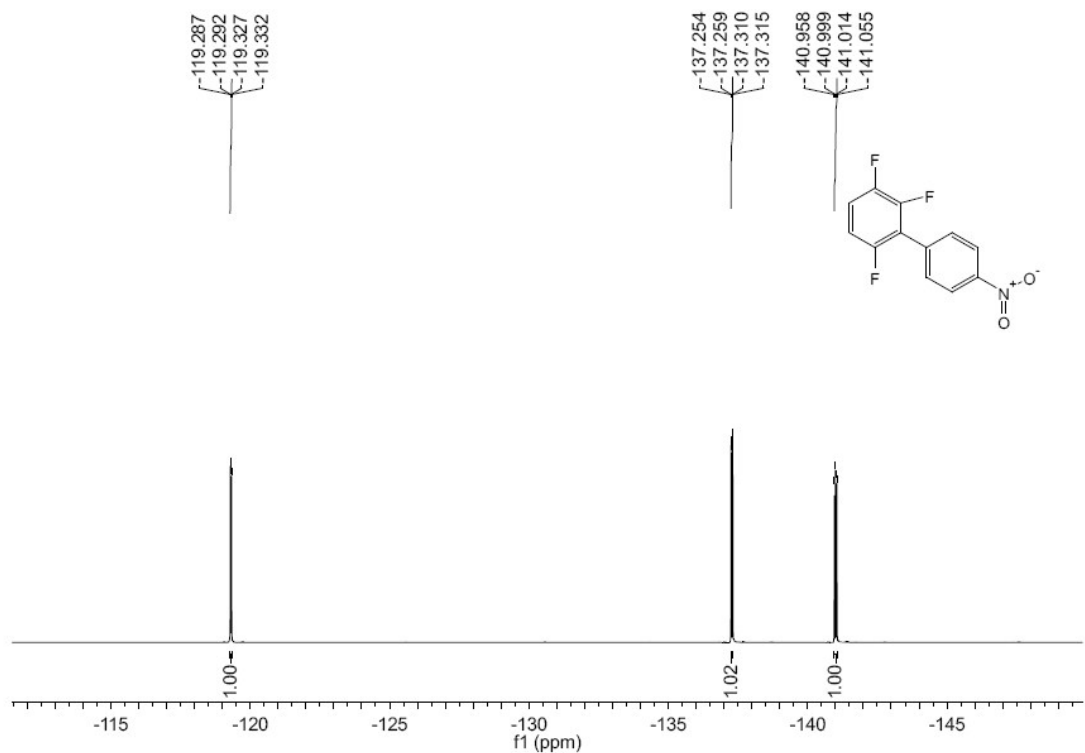
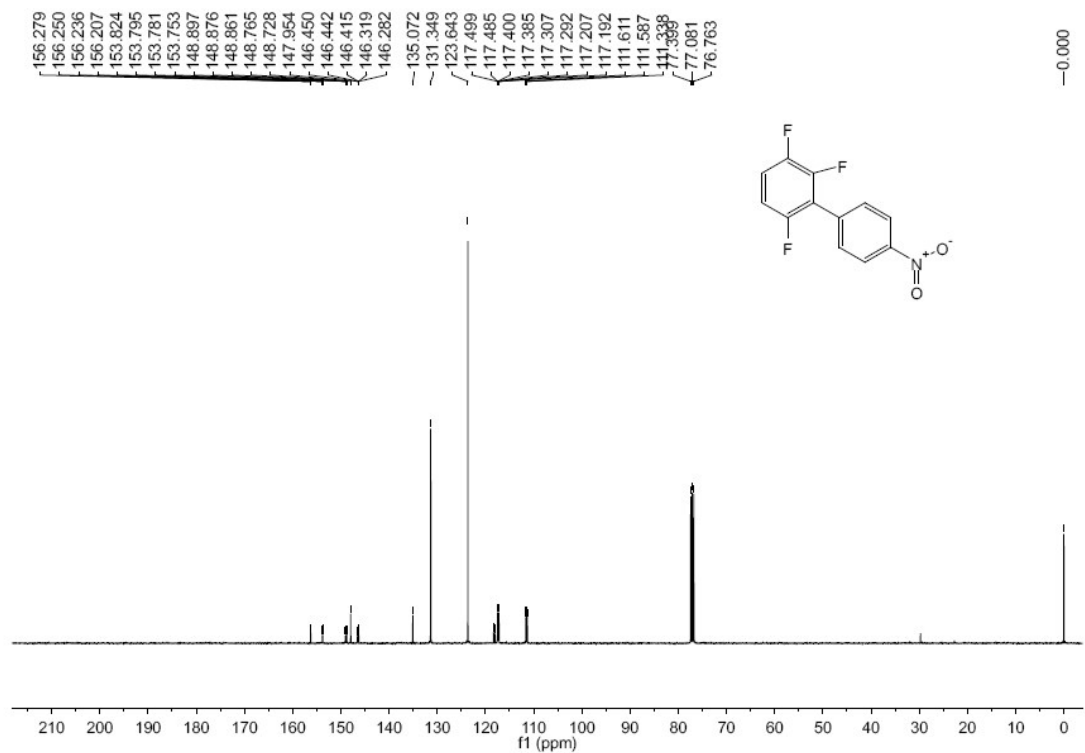
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5d**



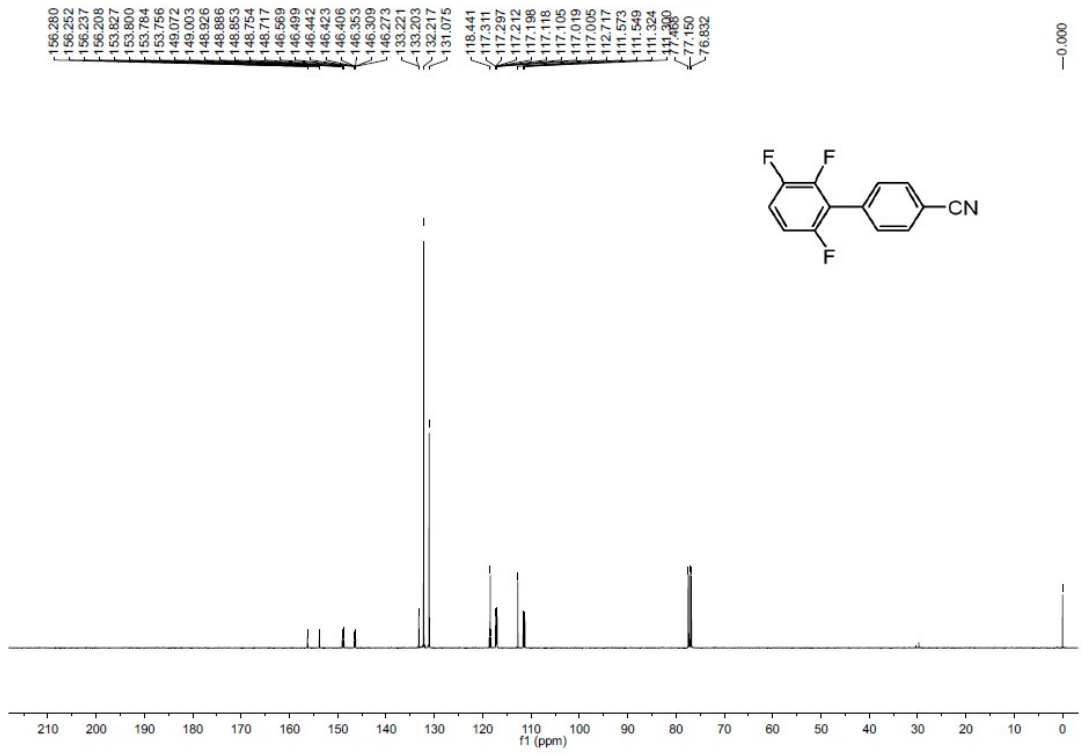
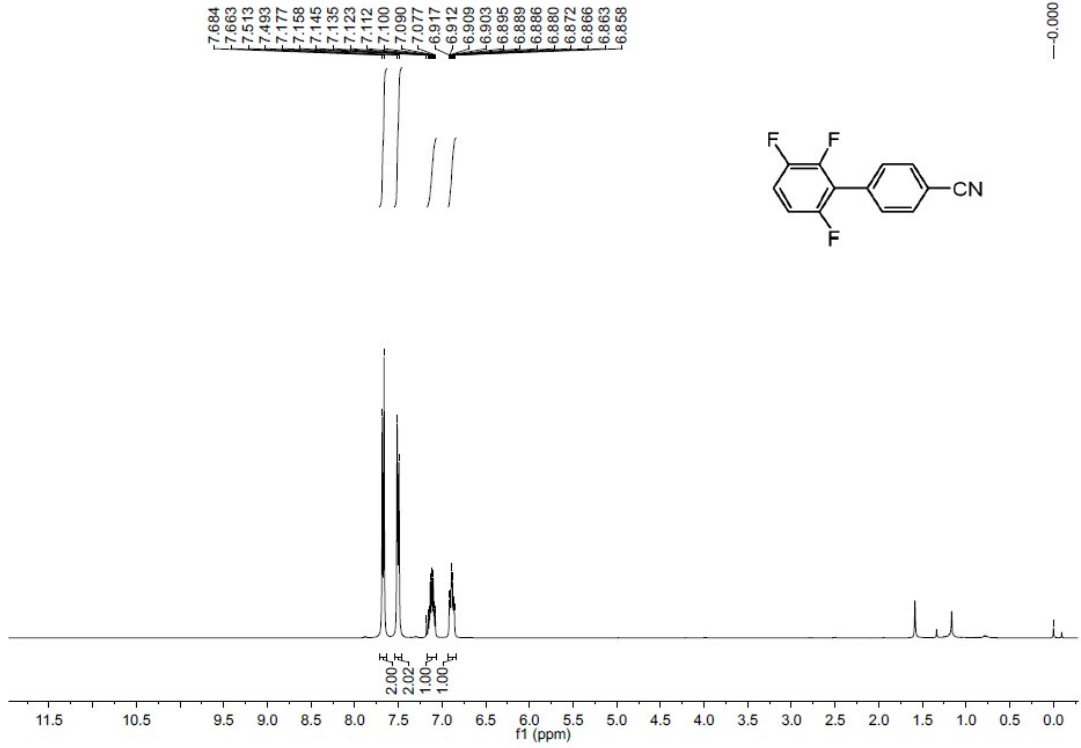


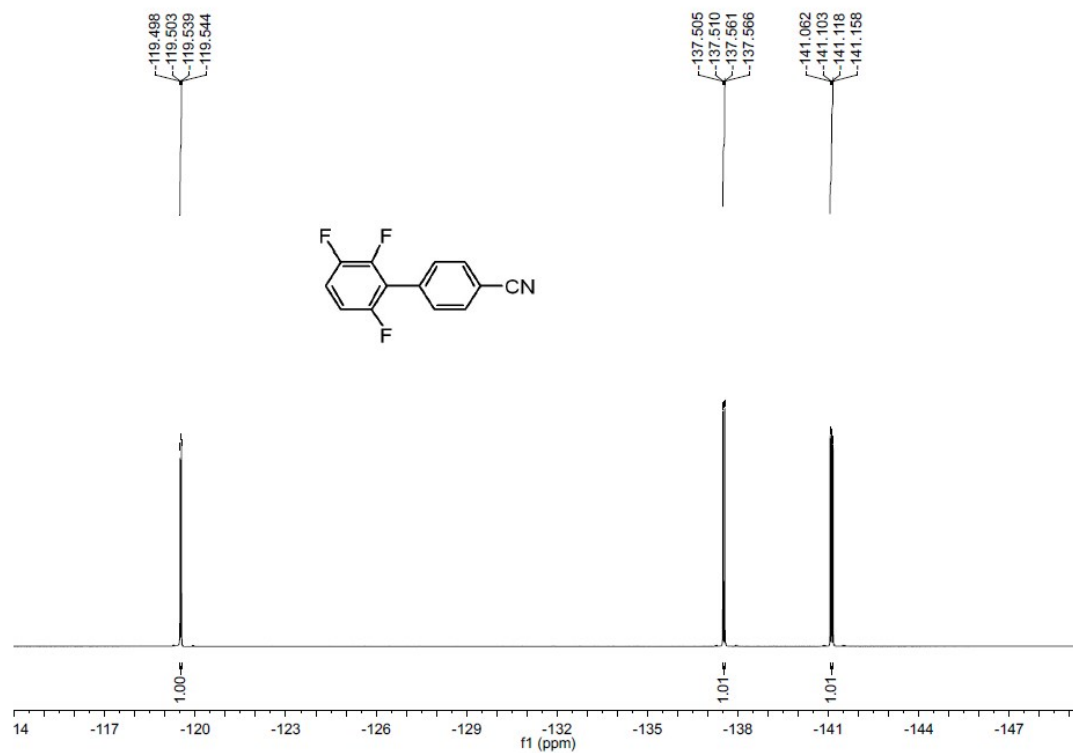
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5e**



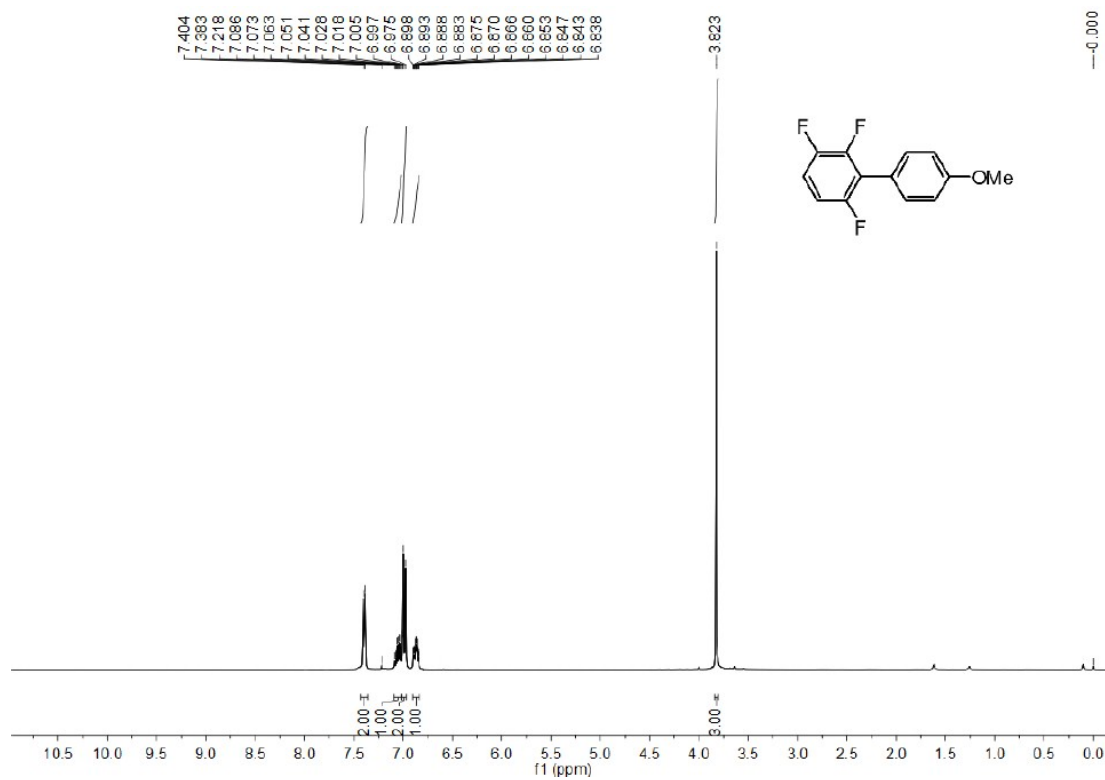


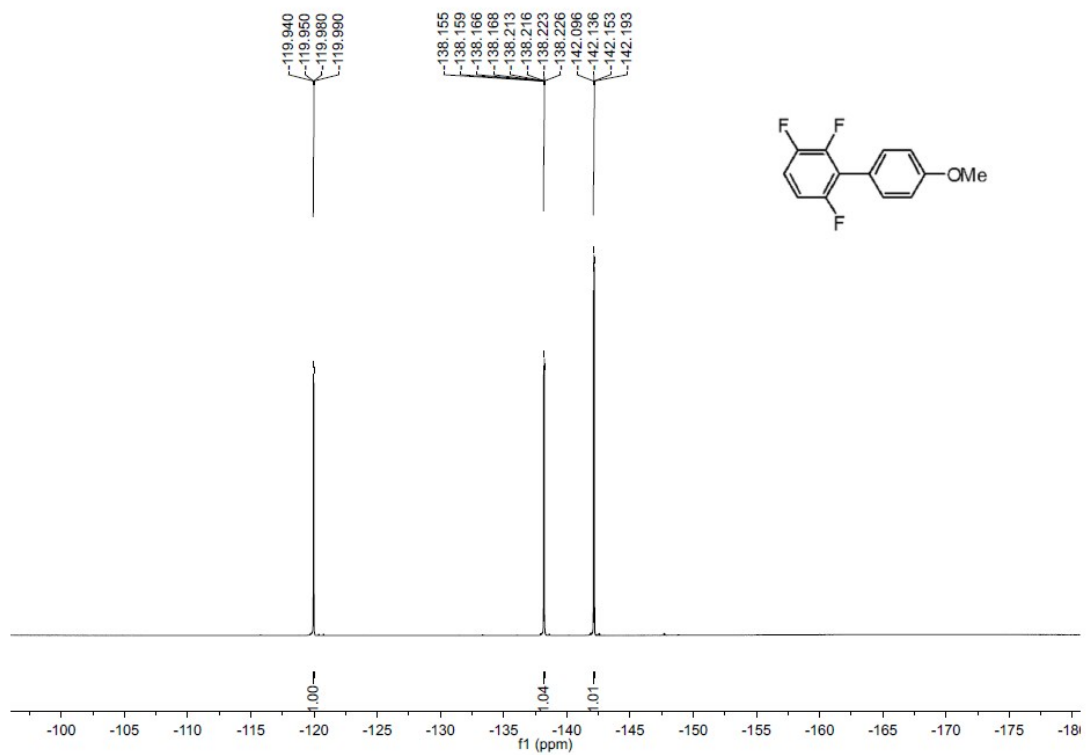
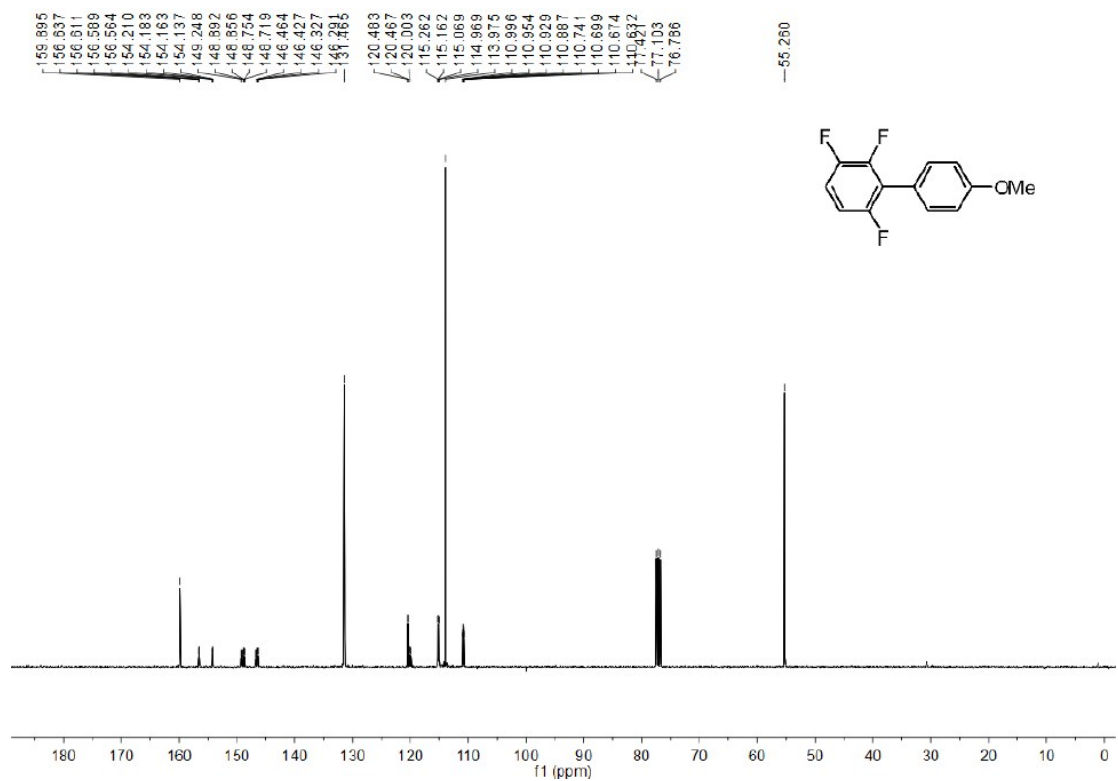
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5f**



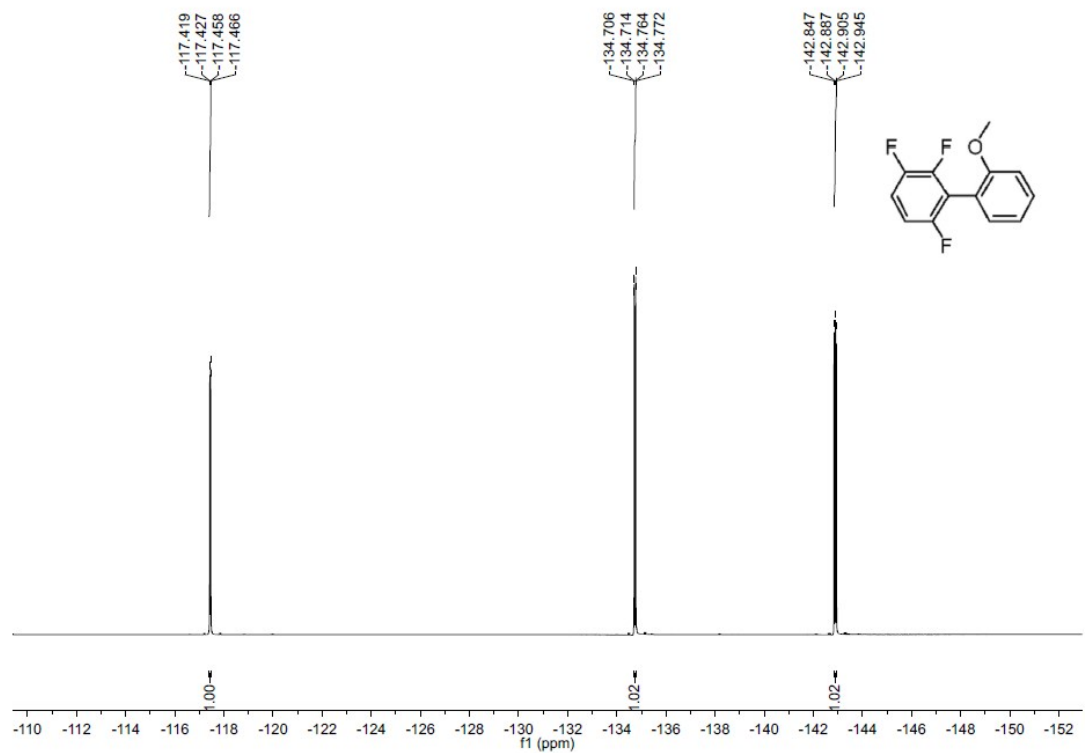


^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5g**

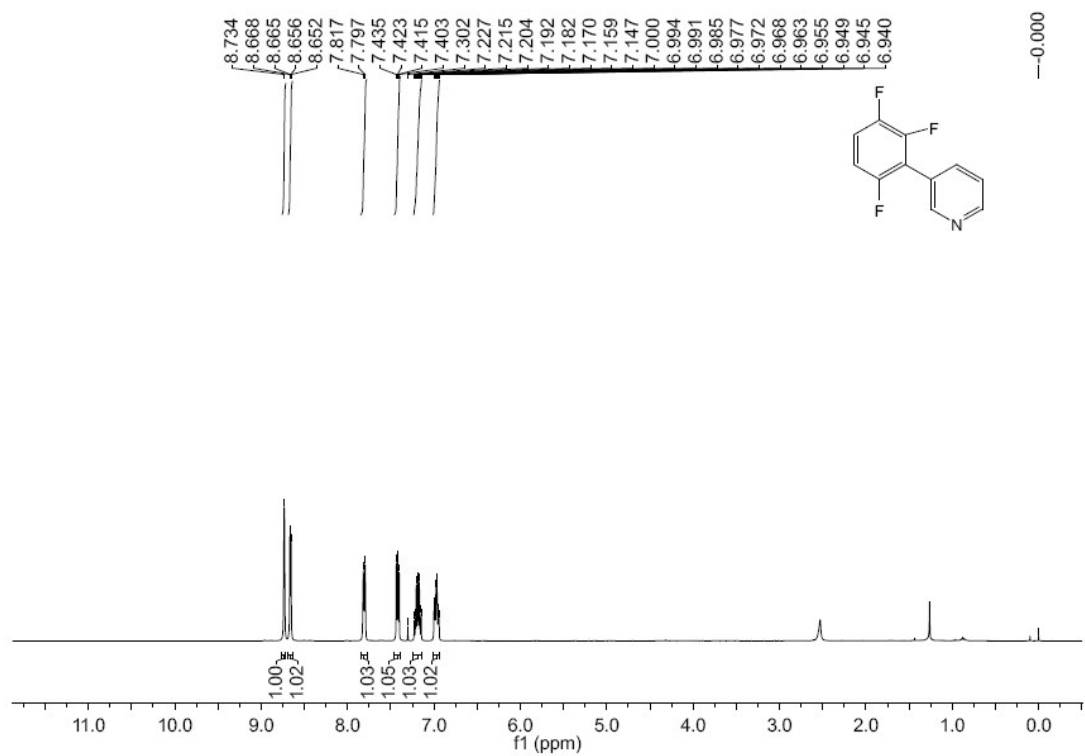


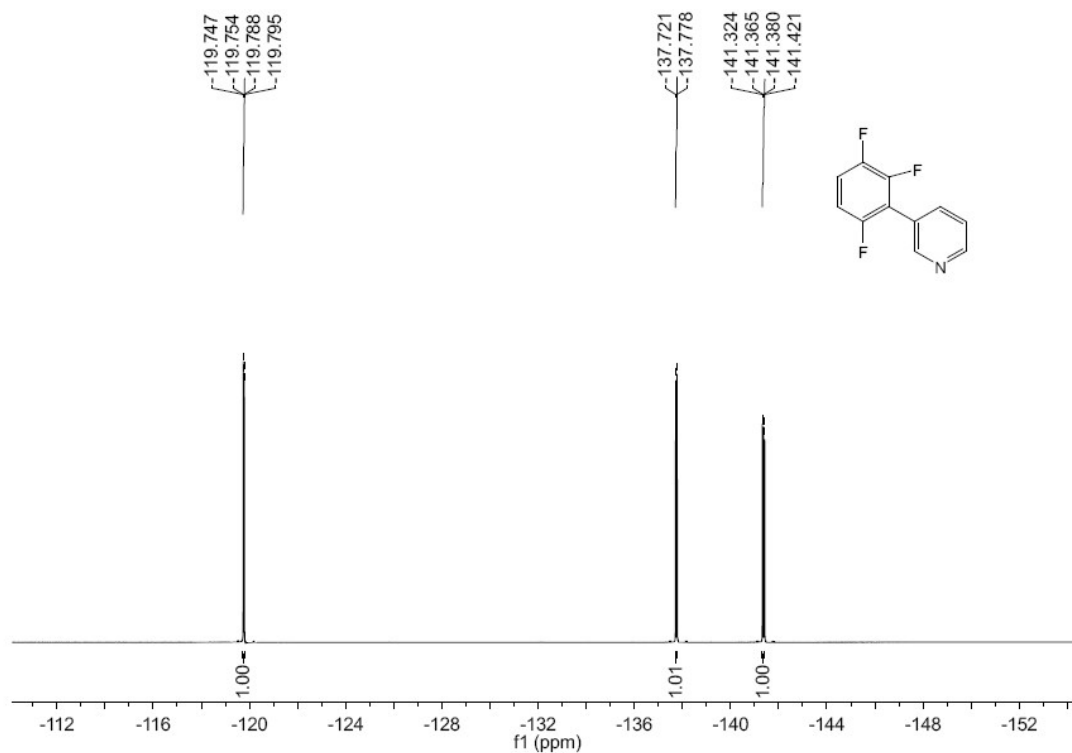
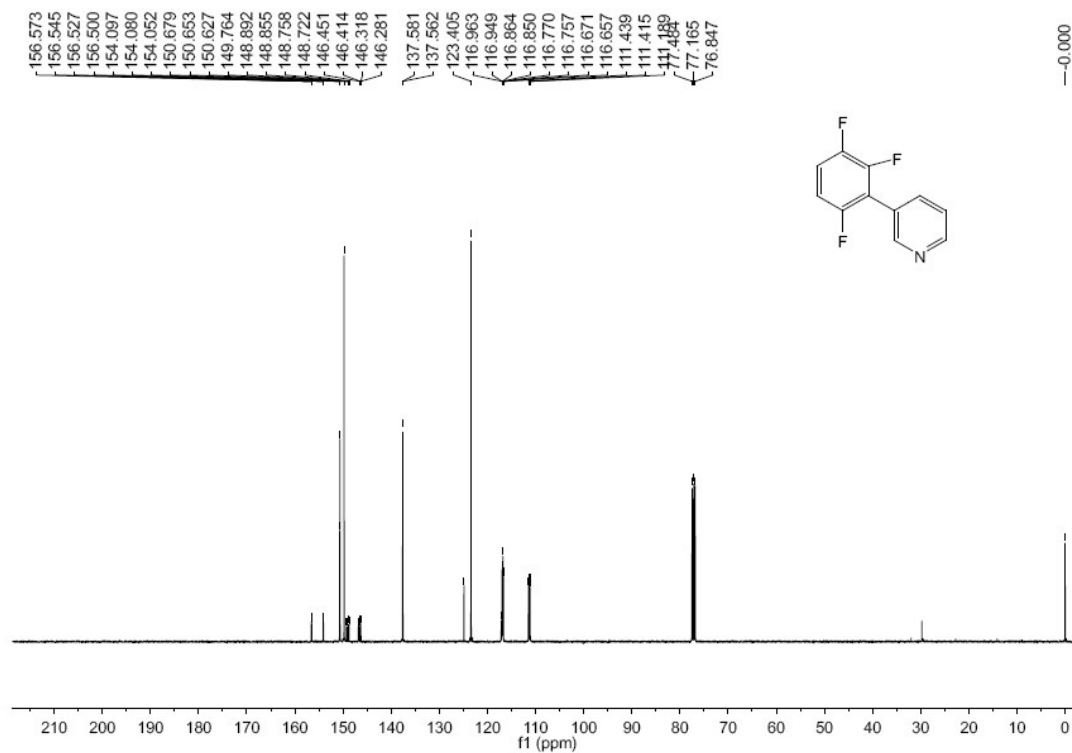


¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5h**

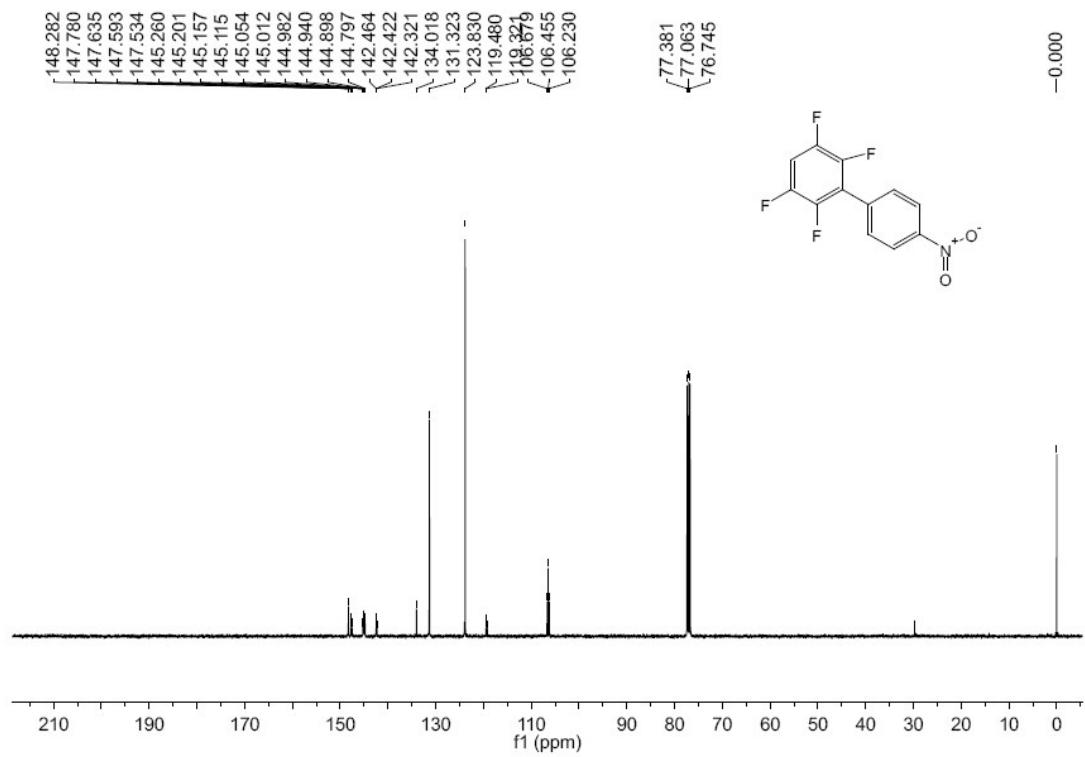
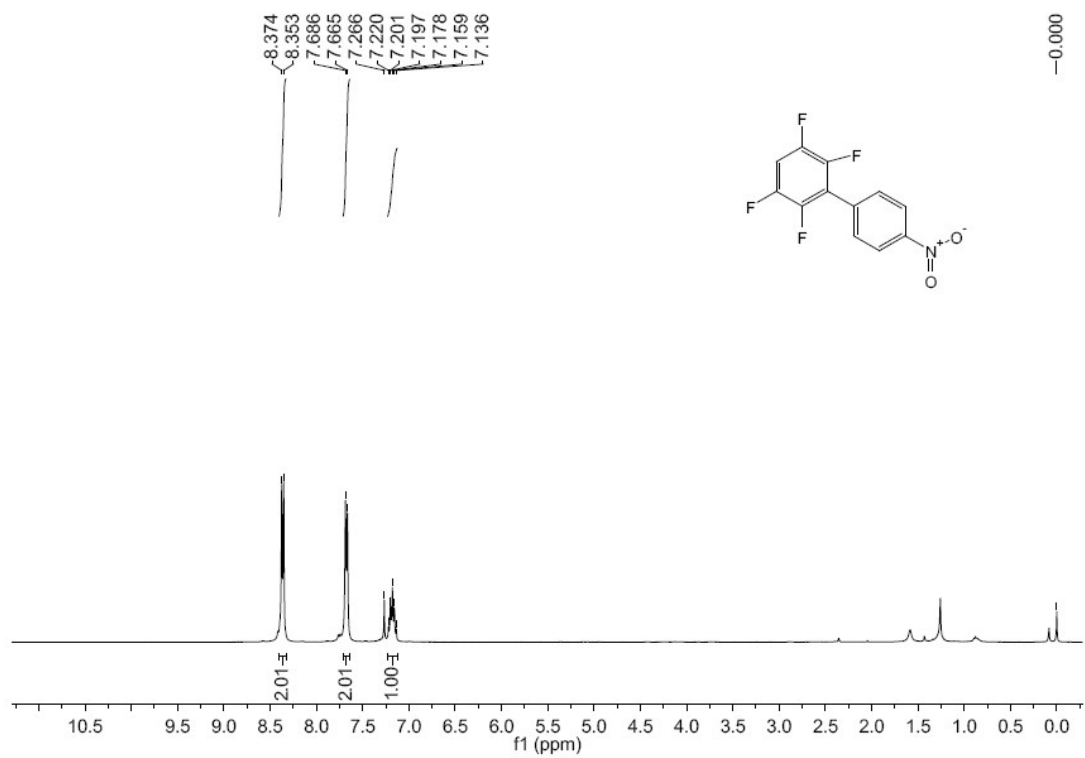


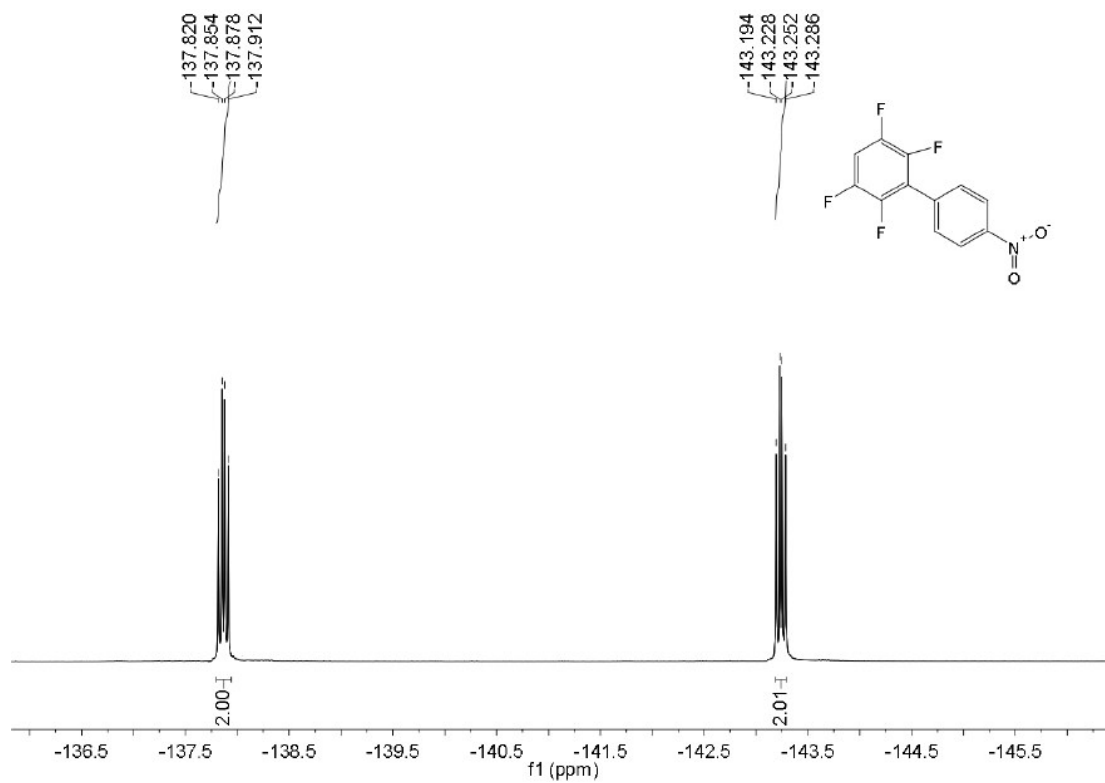
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5i**



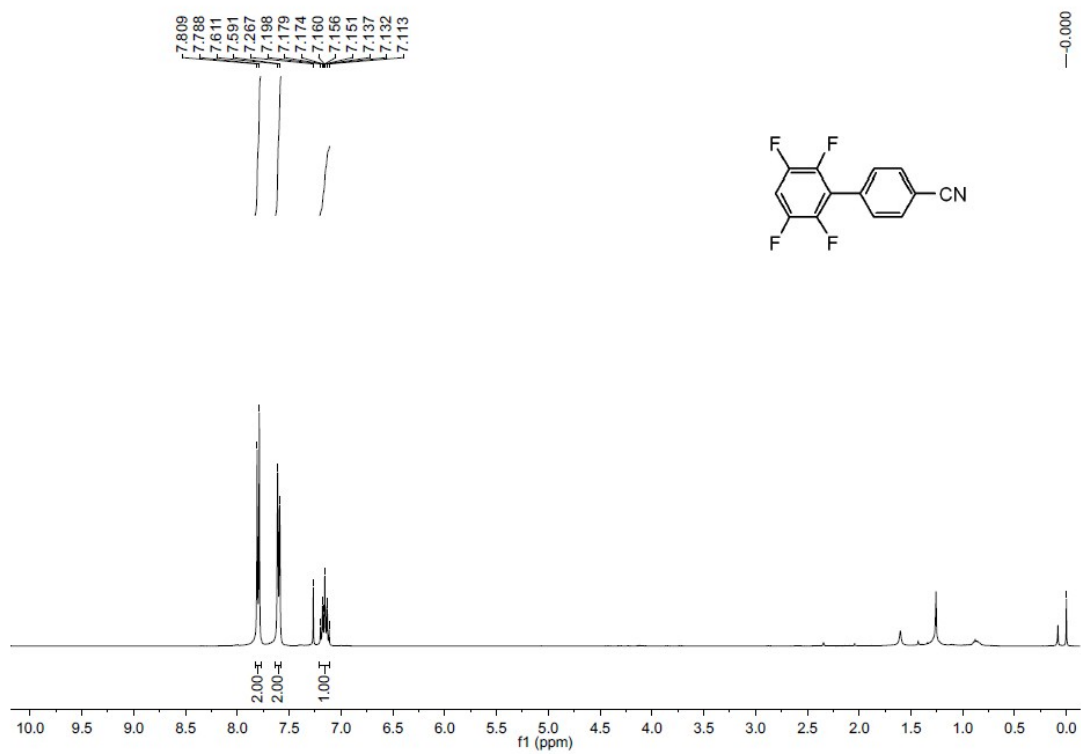


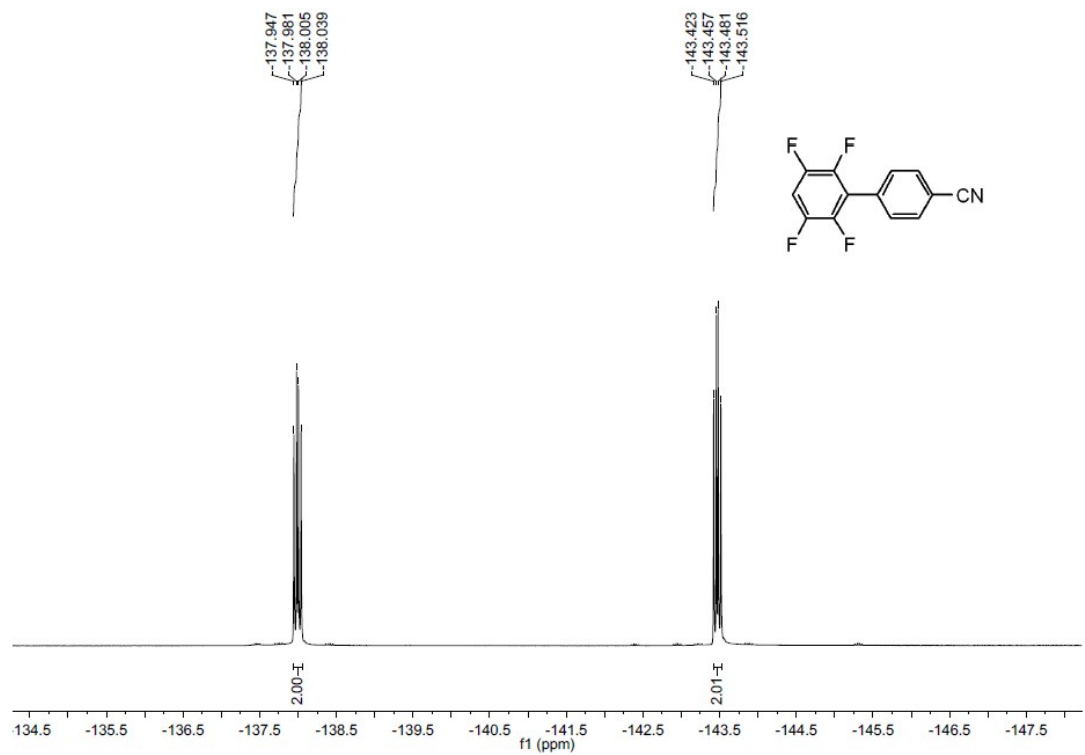
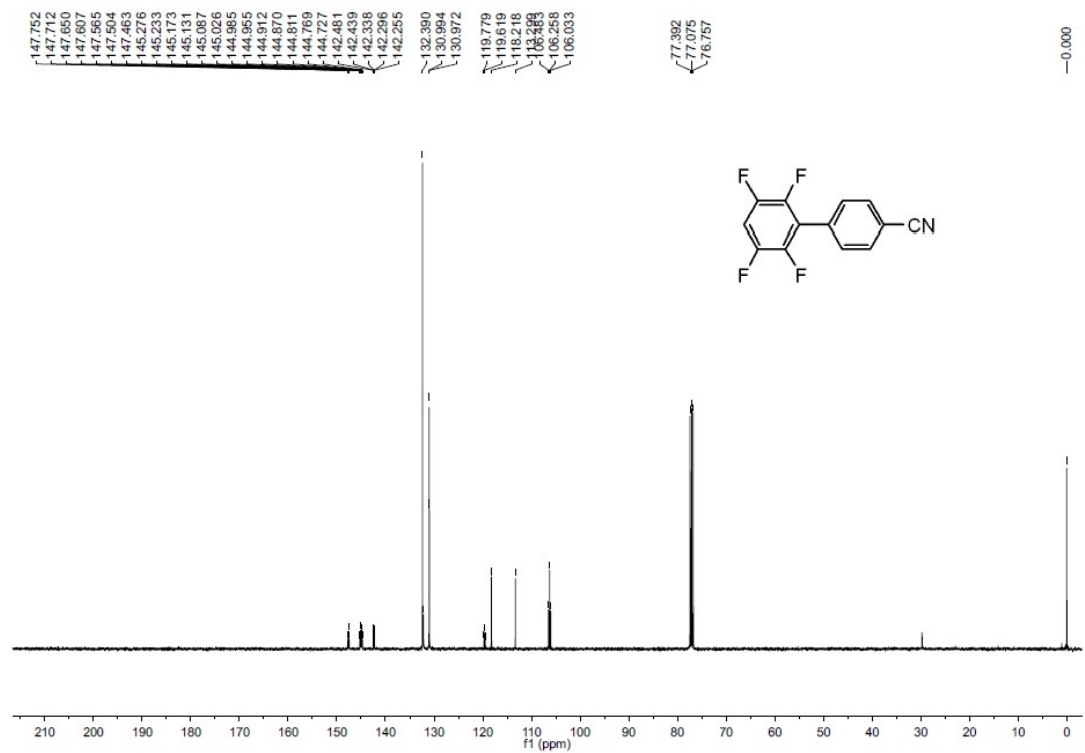
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5j**



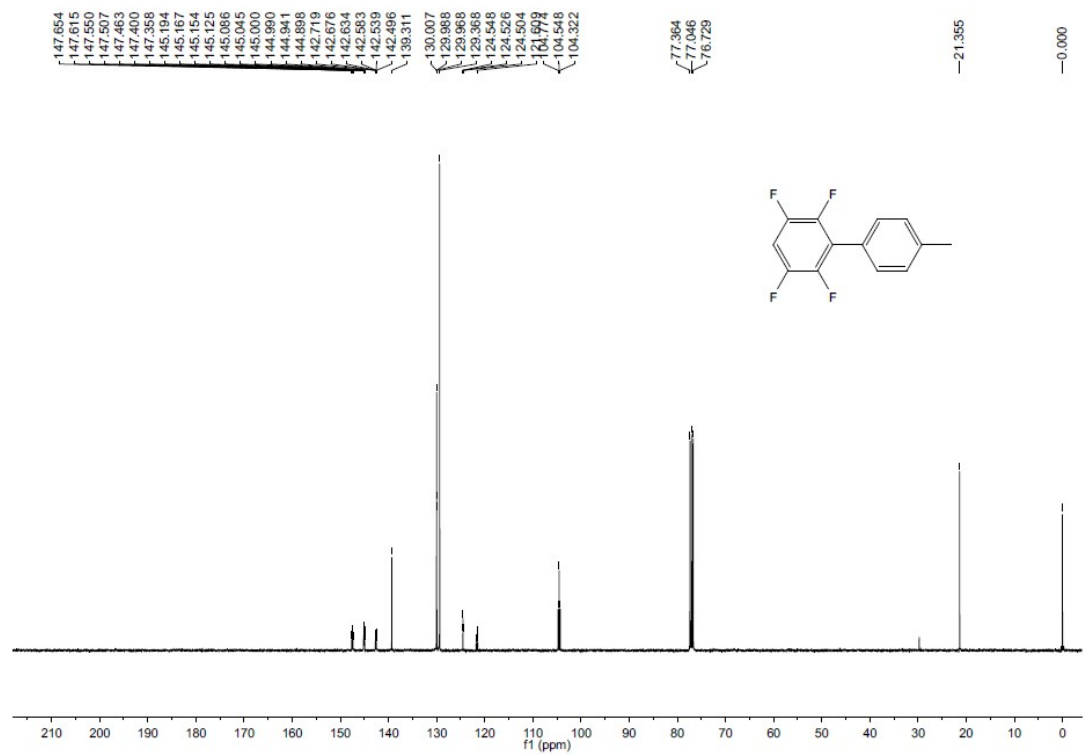
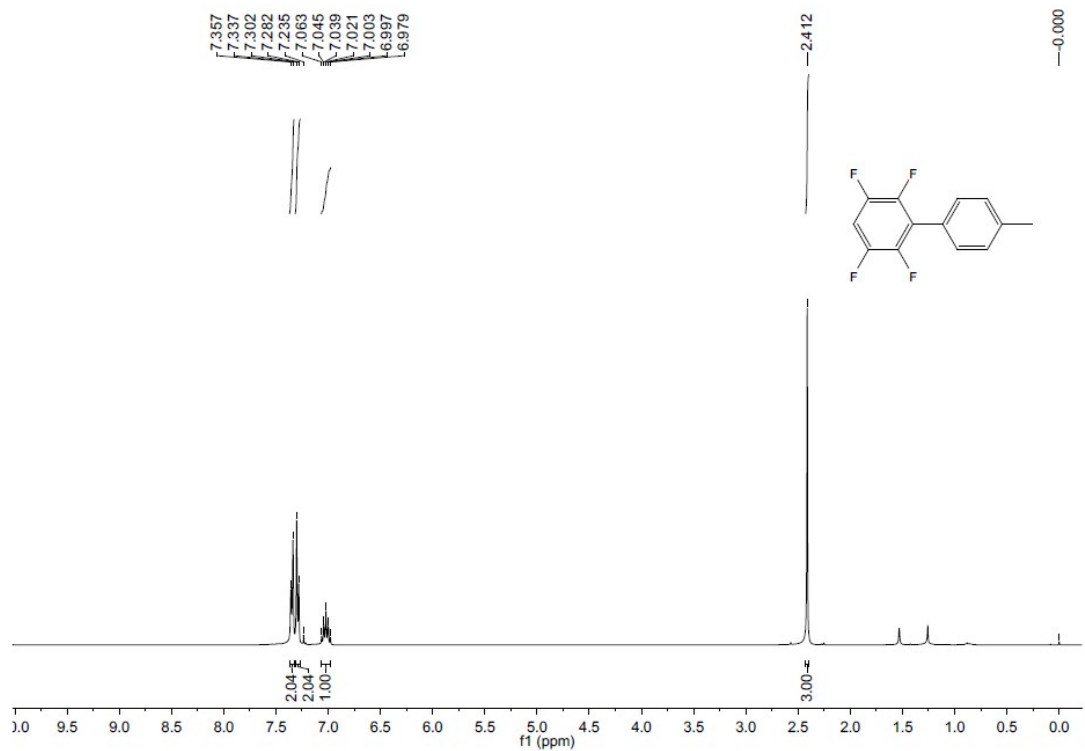


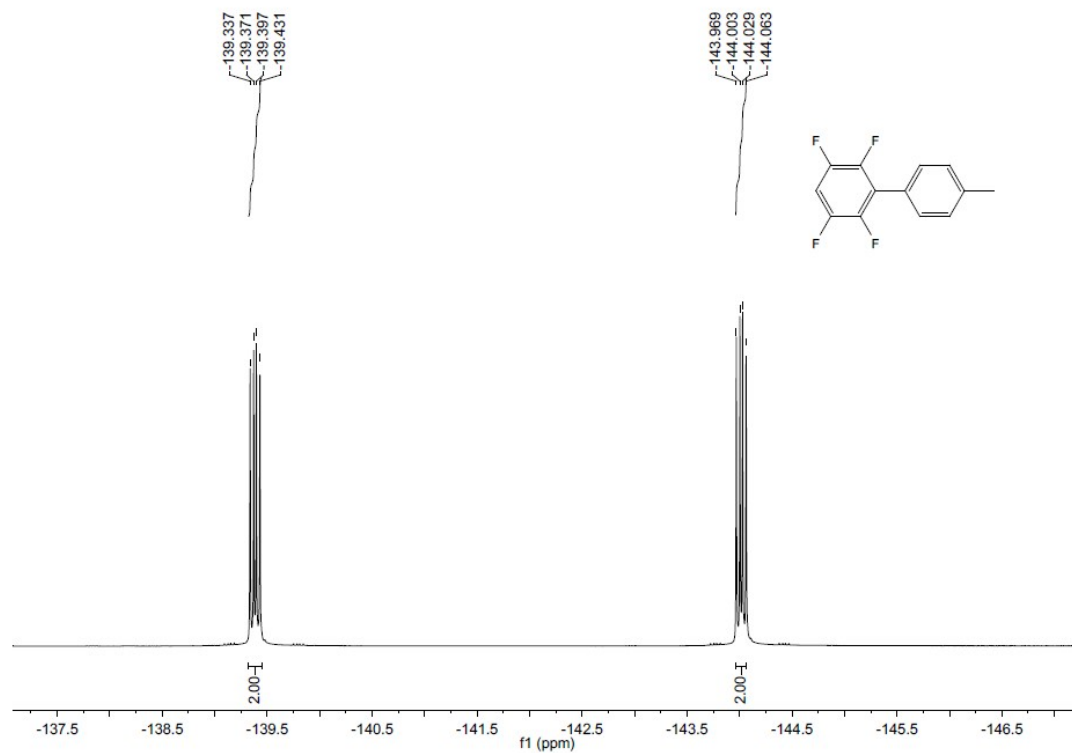
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5k**



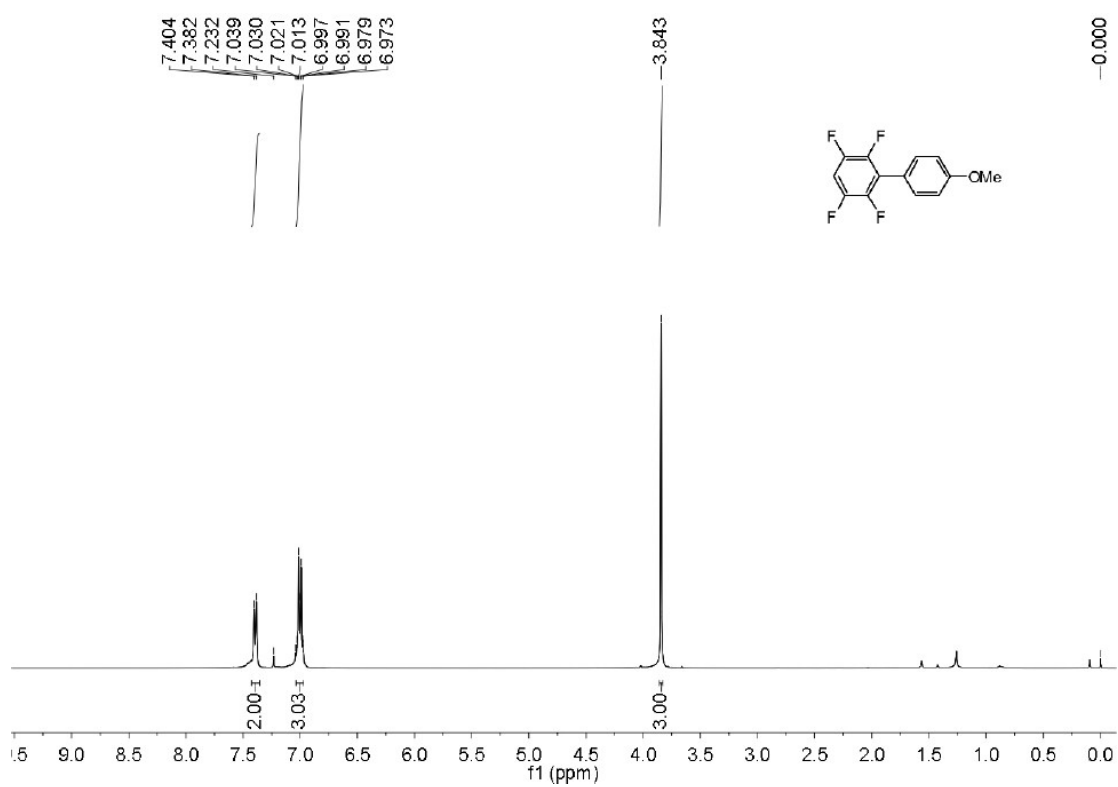


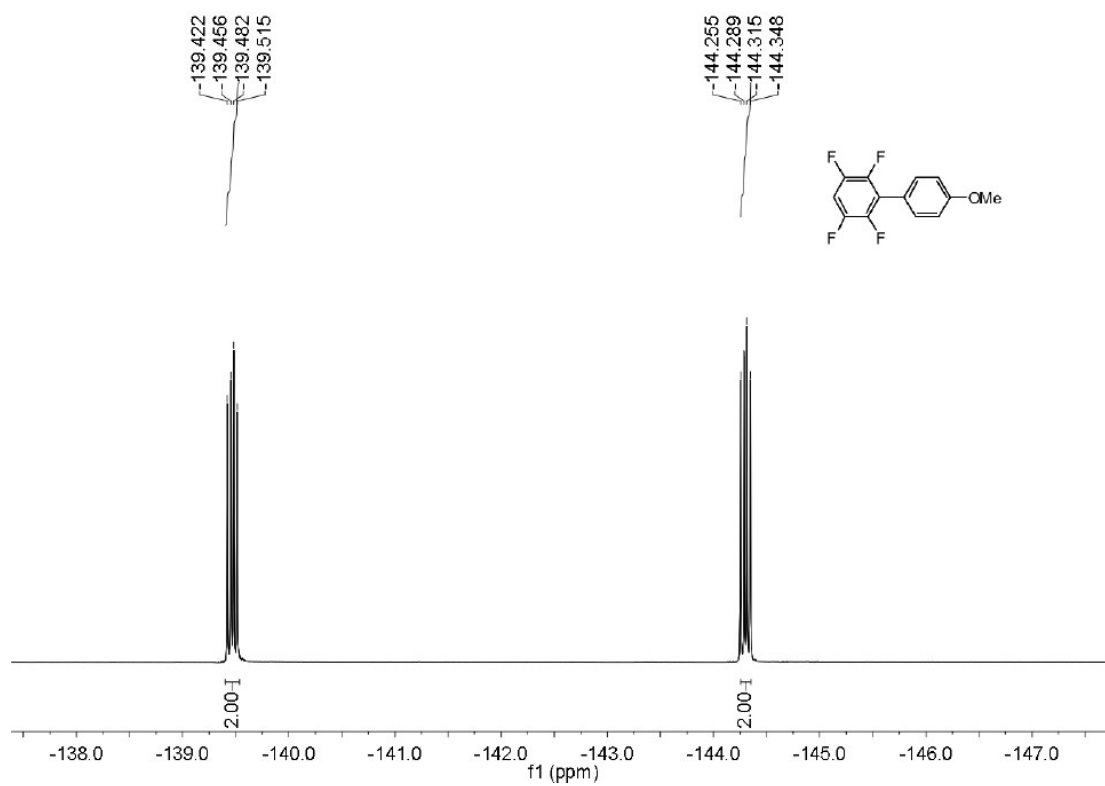
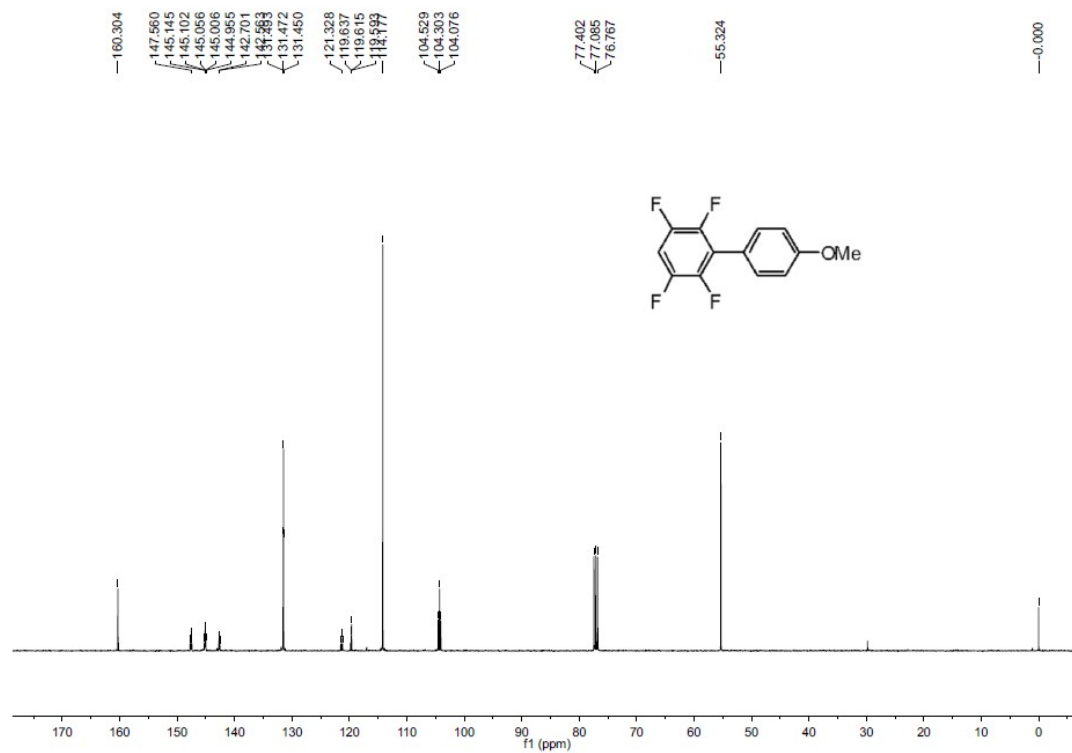
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **51**



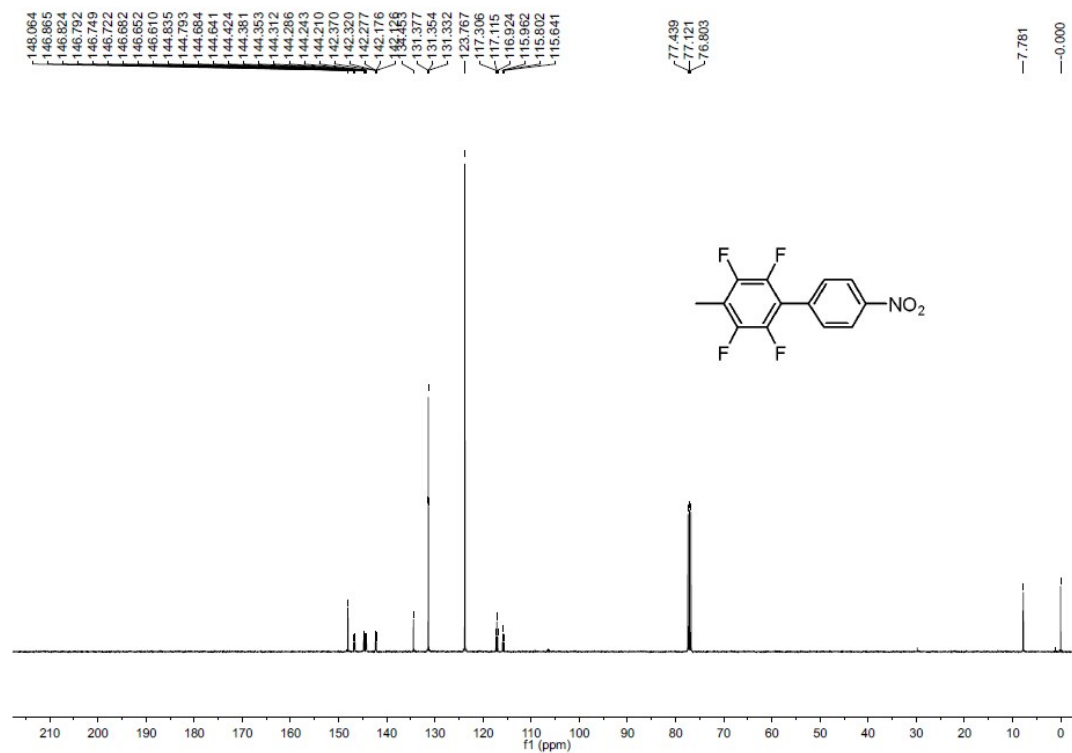
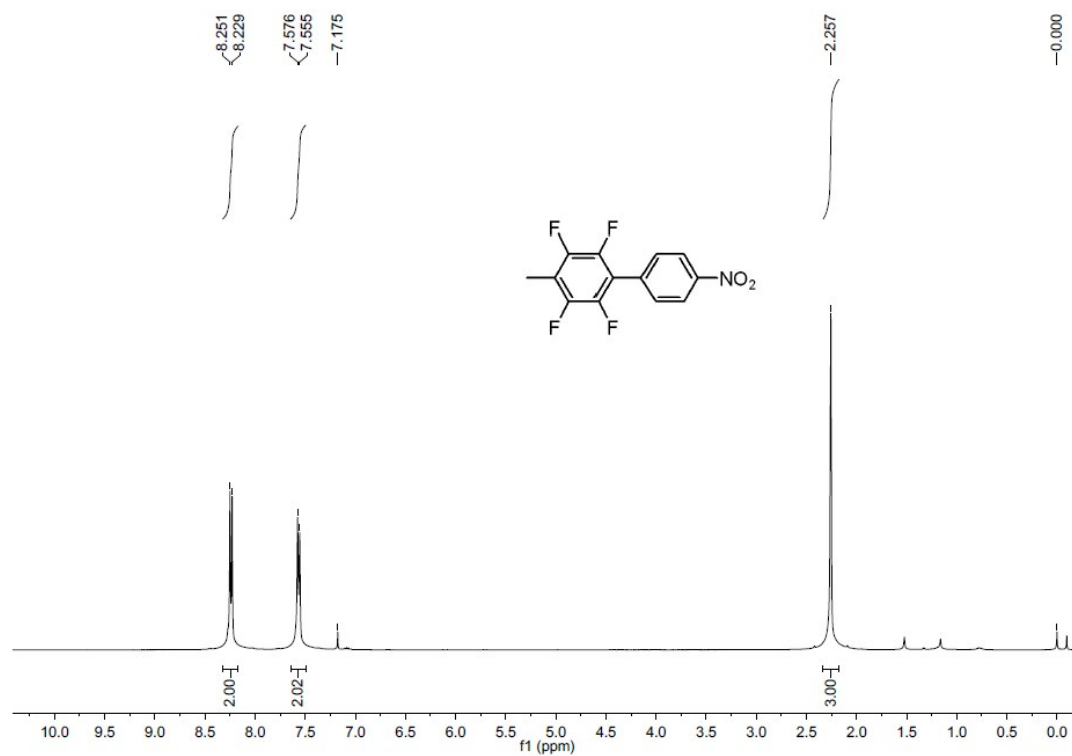


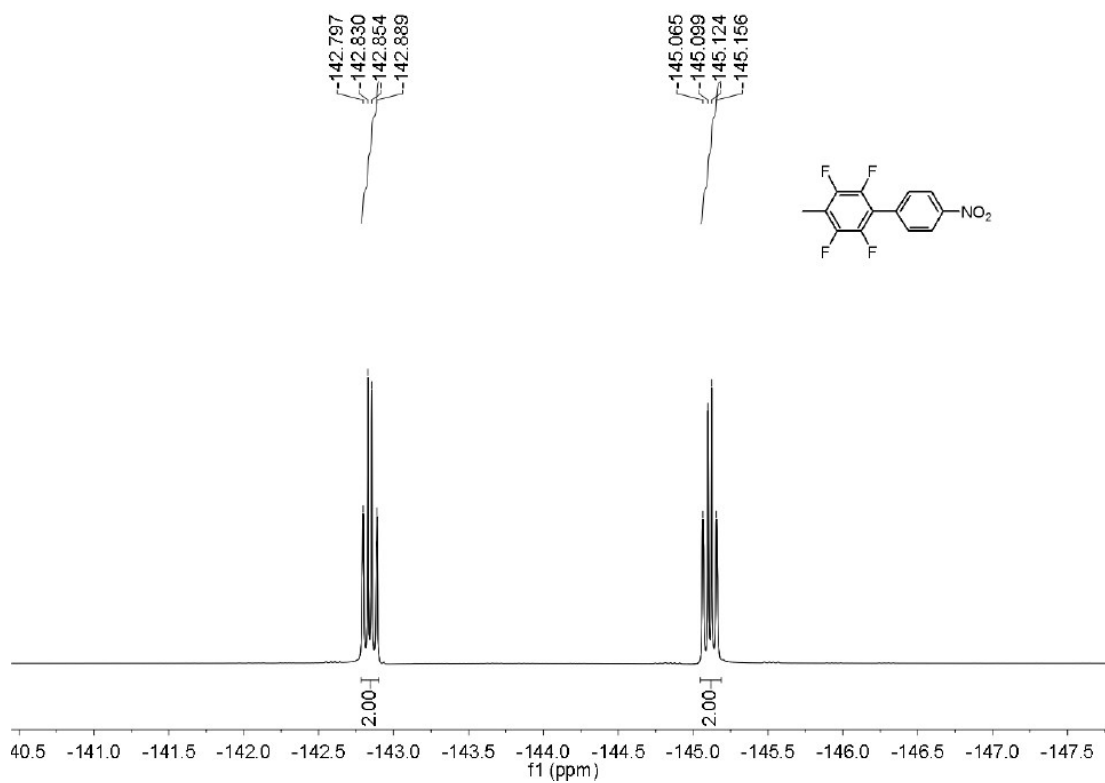
¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **5m**



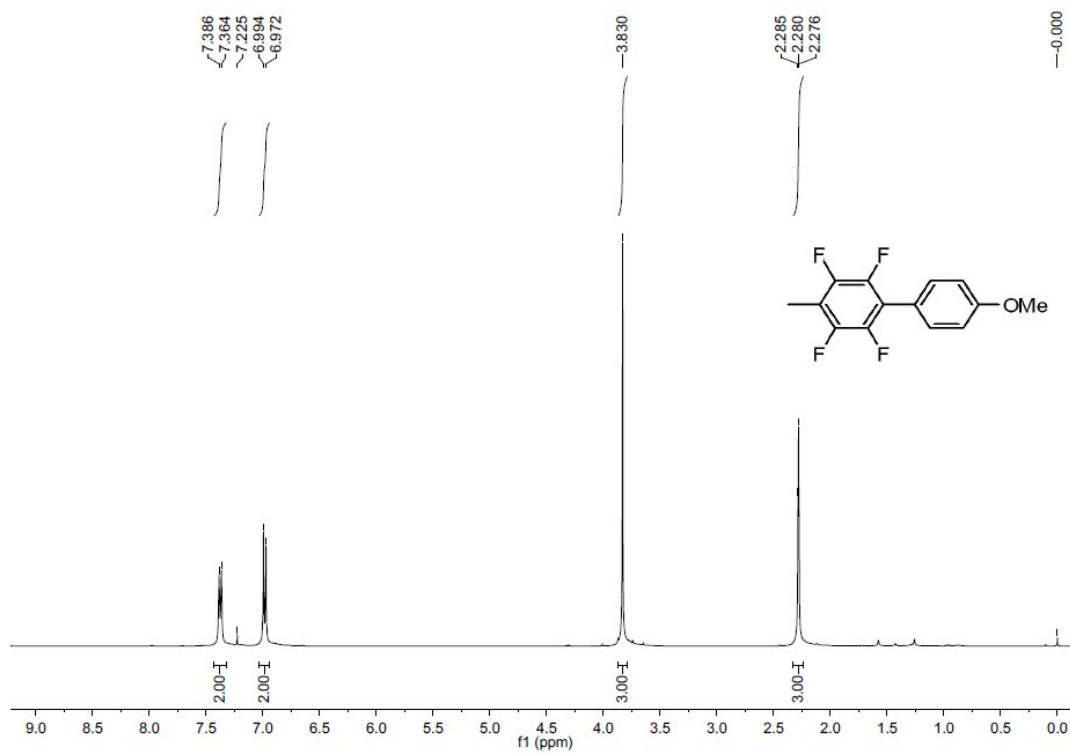


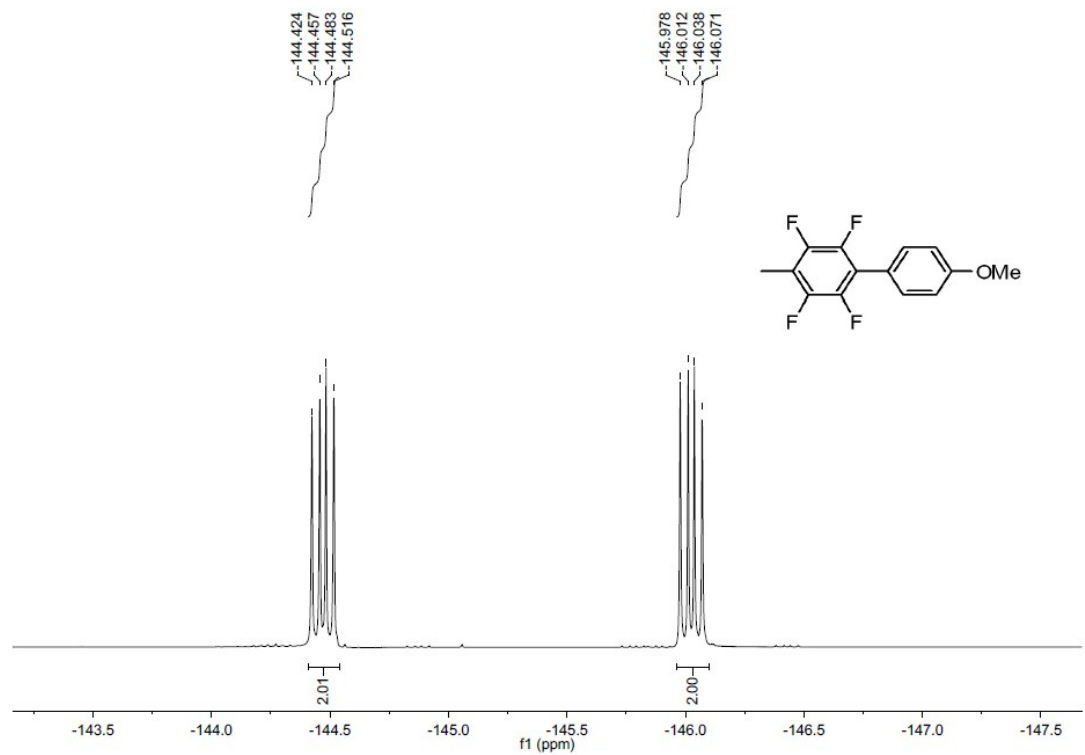
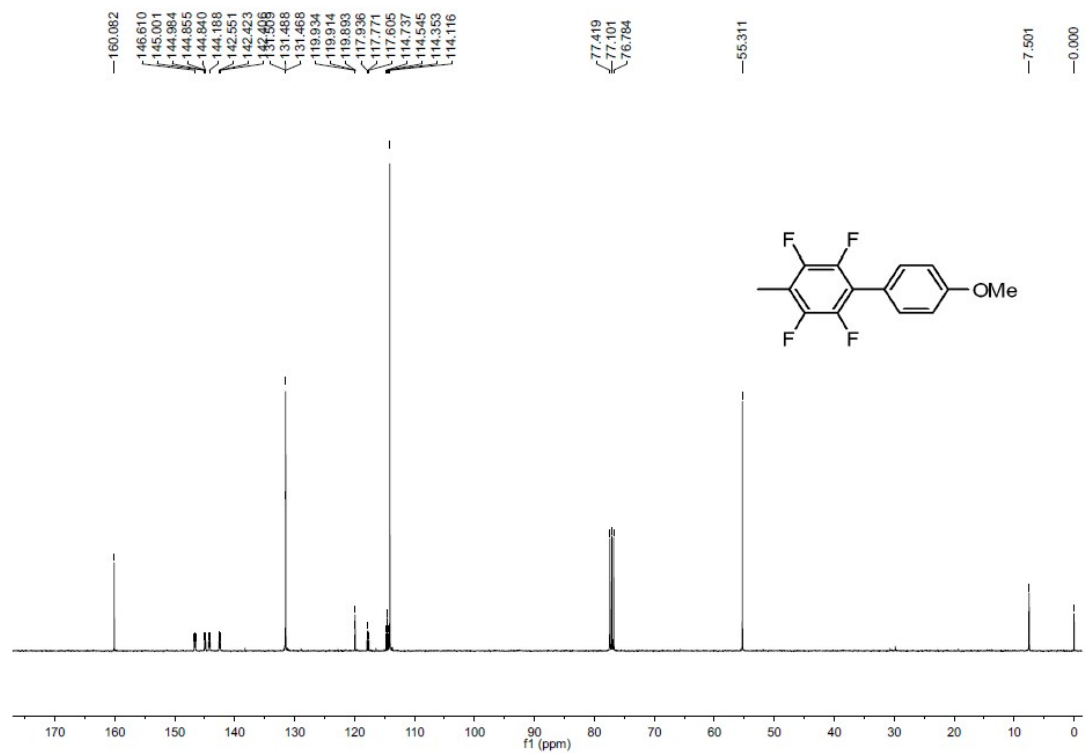
^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5n**





^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5b**





^1H NMR, ^{13}C NMR and ^{19}F NMR spectra of **5p**