Supplementary Information

Assembly of unsymmetrical 1,3,5-triarylbenzenes via tandem reaction of β-arylethenesulfonyl fluorides and α-cyano-β-methylenones

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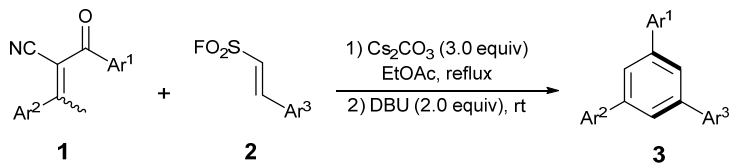
1. General information.

Unless otherwise indicated, all reactions were conducted under air atmosphere in oven-dried glassware with magnetic stirring bar. All other chemicals were obtained from commercial supplies and used as received without any further purification. 2-Arylethenesulfonyl fluorides,[1] α-cyano-β-methylenones[2] were prepared according to literature procedures. Column chromatograph was performed with silica gel (200~300 mesh) and analytical TLC on silica gel 60-F_{254}. ^{1}H, ^{13}C, ^{19}F NMR spectras were recorded on a Bruker AVANCE III spectrometer (400 MHz, 100 MHz and 376 MHz, respectively), Chemical shifts are reported parts per million (ppm) referenced to $CDCl_3$ (δ 7.26 ppm), tetramethylsilane (TMS, δ 0.00 ppm) for ^{1}H, ^{13}C and ^{19}F NMR. Melting points of the products were measured on a micro melting point apparatus (SGW X-4) and uncorrected. High-resolution mass spectra (HRMS) were obtained on a Q Exactive mass spectrometry and a LTQ Orbitrap XL mass spectrometry equipped with an EI or ESI source from Thermo Scientific. X-Ray diffraction study for product **3w′** was carried out on Bruker D8 VENTURE photon II diffractometer with Iμs 3.0 microfocus X-ray source using APEX III program.

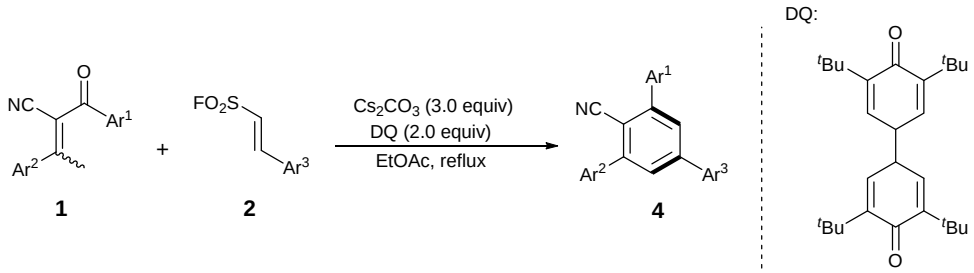
2. General procedure for the synthesis of 1,3,5-triarylbenzenes



A 25 mL Schlenk tube equipped with a stir bar was charged with β-arylenone **1** (0.30 mmol), 2-arylethenesulfonyl fluoride **2** (0.45 mmol), and Cs_2CO_3 (0.90

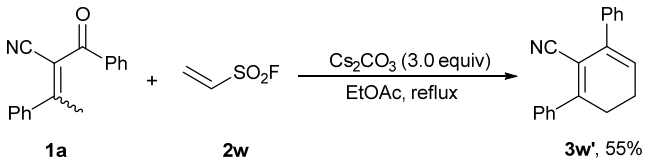
mmol), ethyl acetate (3 mL) was added in turn to the Schlenk tube. The reaction mixture was stirred at 78 °C for 2 h until complete consumption of the starting material (monitored by TLC). Then DBU was added to the reaction mixture and stirred for 2 h at room temperature. When the reaction was finished, the reaction mixture was diluted by dichloromethane, and then concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (silica gel, PE/EtOAc (v : v) = 300:1) to afford the desired product **3**.

3. General procedure for the synthesis of 1,3,5-triarylbenzonitriles

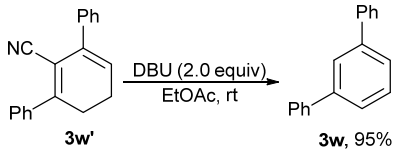


A 25 mL Schlenk tube equipped with a stir bar was charged with β-arylenones **1** (0.30 mmol), 2-arylethenesulfonyl fluorides **2** (0.45 mmol), DQ (0.60 mmol) and Cs_2CO_3 (0.90 mmol), ethyl acetate (3 mL) was added in turn to the Schlenk tube. The reaction mixture was stirred at 78 °C for 5 h until complete consumption of the starting material (monitored by TLC). When the reaction was finished, the reaction mixture was diluted by dichloromethane, and then concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (silica gel, PE/EtOAc (v : v) = 20:1) to afford the desired product **4**.

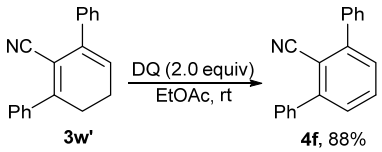
4. General procedure for control experiments.



A 25 mL Schlenk tube equipped with a stir bar was charged with enone **1a** (0.30 mmol), and Cs_2CO_3 (0.90 mmol), ethyl acetate (3 mL) and vinyl sulfonyl fluoride **2w** (0.45 mmol) were added in turn to the Schlenk tube. The reaction mixture was stirred at 78 °C for 12 h until complete consumption of the starting material (monitored by TLC). When the reaction was finished, the reaction mixture was diluted by dichloromethane, and then concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (silica gel, PE/EtOAc (v : v) = 20:1) to afford the desired product **3w′** (55%, 42.4 mg).



A 25 mL Schlenk tube equipped with a stir bar was charged with **3w′** (31.0 mg, 0.12 mmol), ethyl acetate (1 mL) and DBU (37.0 mg, 0.24 mmol) was added in turn to the Schlenk tube. When the reaction was finished, the reaction mixture was diluted by dichloromethane, and then concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (silica gel, PE/EtOAc (v : v) = 300:1) to afford the desired product **3w**.



A 25 mL Schlenk tube equipped with a stir bar was charged with **3w′** (31.0 mg, 0.12 mmol) and DQ (98.0 mg, 0.24 mmol), ethyl acetate (1 mL) was added in turn to the Schlenk tube. When the reaction was finished, the reaction mixture was diluted by dichloromethane, and then concentrated under reduced pressure. The

residue was purified by column chromatography on silica gel (silica gel, PE/EtOAc (v : v) = 20:1) to afford the desired product **4f**.

5. Single-crystal X-ray structure analysis

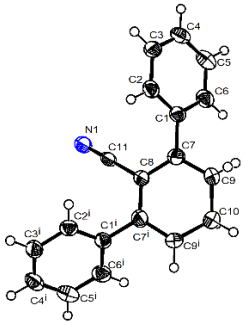


Figure S1. Crystal structure of **3w′**

data-3w′

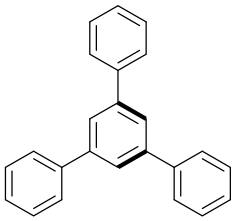
Table S1. Crystal data and structure refinement for **3w′**

Identification code	**3w′**	
Empirical formula	$C_{19}H_{14}N$	
Formula weight	256.31	
Temperature	173.0 K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$Pmn2_1$	
Unit cell dimensions	a = 14.1719(5) Å	a= 90°.
	b = 8.9226(3) Å	b= 90°.
	c = 5.6638(2) Å	g = 90°.
Volume	716.19(4) $Å^3$	
Z	2	
Density (calculated)	1.189 Mg/m^3	
Absorption coefficient	0.528 mm^{-1}	
F(000)	270	
Crystal size	0.16 x 0.13 x 0.12 mm^3	
Theta range for data collection	4.956 to 72.166°.	
Index ranges	-17<=h<=14, -11<=k<=11, -6<=l<=6	
Reflections collected	8521	
Independent reflections	1417 [R(int) = 0.0456]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7536 and 0.6855	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1417 / 67 / 114	
Goodness-of-fit on F^2	1.083	
Final R indices [I>2sigma(I)]	R1 = 0.0337, wR2 = 0.0925	
R indices (all data)	R1 = 0.0353, wR2 = 0.0936	

Absolute structure parameter	0.5
Extinction coefficient	0.008(3)
Largest diff. peak and hole	0.158 and -0.137 e.Å^{-3}

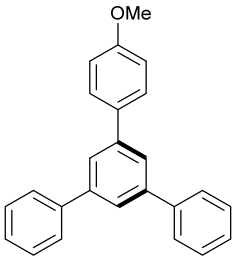
The CCDC number of product **3w′** is 2118497.

6. Characterization data of products



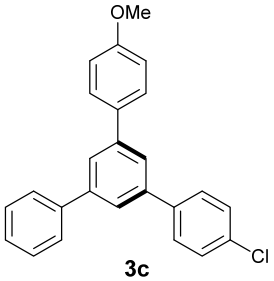
3a

5'-phenyl-1,1':3',1''-terphenyl (known compound)[3] White solid, 51.4 mg, 56% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 (s, 3H), 7.73 – 7.66 (m, 6H), 7.52 – 7.44 (m, 6H), 7.43 – 7.34 (m, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.5, 141.3, 129.0, 127.7, 127.5, 125.3.



3b

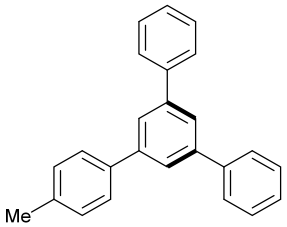
4-methoxy-5'-phenyl-1,1':3',1''-terphenyl (known compound)[3] White solid, 47.4 mg, 47% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (s, 3H), 7.72 – 7.67 (m, 4H), 7.66 – 7.61 (m, 2H), 7.51 – 7.44 (m, 4H), 7.42 – 7.35 (m, 2H), 7.04 – 6.98 (m, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 159.5, 142.5, 142.1., 141.4, 133.8, 129.0, 128.5, 127.6, 127.5, 124.9, 124.8, 114.4, 55.5.



3c

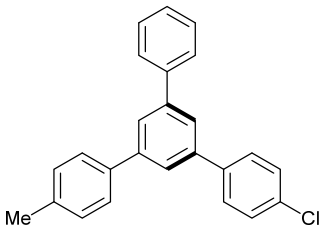
4-chloro-4''-methoxy-5'-phenyl-1,1':3',1''-terphenyl (known compound)[3] White solid, 46.7 mg, 42% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.75 (s, 1H), 7.71 – 7.65 (m, 4H), 7.62 (d, J = 8.4 Hz, 4H), 7.52 – 7.38 (m, 5H), 7.04 – 6.98 (m, 2H), 3.87 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 159.6, 142.6, 142.2, 141.2,

139.8, 133.8, 133.6, 129.1, 129.0, 128.7, 128.5, 127.8, 127.5, 125.2, 124.7, 124.5, 114.5, 55.5.



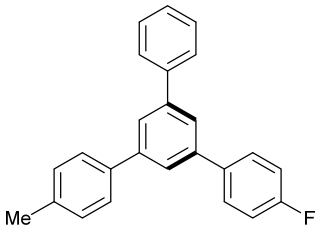
3d

4-methyl-5'-phenyl-1,1':3',1''-terphenyl (known compound) [3] White solid, 50.9 mg, 53% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 – 7.74 (m, 3H), 7.72 – 7.66 (m, 4H), 7.60 (d, J = 8.0 Hz, 2H), 7.51 – 7.44 (m, 4H), 7.41 – 7.35 (m, 2H), 7.29 (d, J = 8.0 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.5, 142.4, 141.4, 138.4, 137.5, 129.7, 129.0, 127.7, 127.5, 127.3, 125.1, 125.0, 21.3.



3e

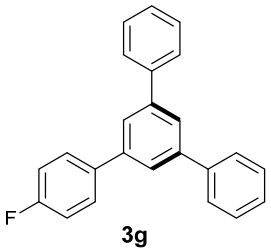
4-chloro-4''-methyl-5'-phenyl-1,1':3',1''-terphenyl White solid, 65.9 mg, 62% yield. m.p. 160.3-161.0 ℃; IR (cm^{-1}): 3030, 2922, 2854, 1595, 1549, 1492, 1091, 813, 761, 698; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.78 (s, 1H), 7.74 – 7.65 (m, 4H), 7.65 – 7.55 (m, 4H), 7.51 – 7.37 (m, 5H), 7.29 (d, J = 7.6 Hz, 2H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.6, 142.5, 141.2, 139.8, 138.2, 137.6, 133.8, 129.7, 129.1, 129.0, 128.7, 127.8, 127.5, 127.3, 125.4, 124.9, 124.8, 21.3; HRMS (EI) m/z calcd for $C_{25}H_{19}Cl$ $[M]^+$ 354.1175, found 354.1168.



3f

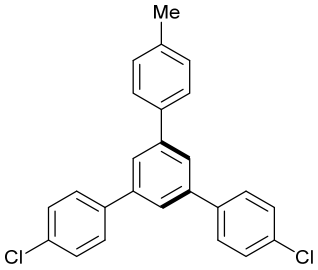
4-fluoro-4''-methyl-5'-phenyl-1,1':3',1''-terphenyl White solid, 49.7 mg, 49% yield. m.p. 127.3-128.4 ℃; IR (cm^{-1}): 3049, 2954, 2854, 1603, 1511, 1230, 1158, 913, 743, 692; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 (s, 1H), 7.77 – 7.71 (m, 4H), 7.70 – 7.65 (m, 2H), 7.63 (d, J = 8.0 Hz, 2H), 7.52 (t, J = 7.6 Hz, 2H), 7.46 – 7.39 (m, 1H), 7.33 (d, J = 7.8 Hz, 2H), 7.24 – 7.15 (m, 2H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 162.7 (d, J = 245.0 Hz), 142.5, 142.4, 141.4, 141.2, 138.2, 137.6, 137.4 (d, J = 3.0 Hz), 129.7, 129.1, 129.0, 127.7, 127.5, 127.3, 125.1,

124.9 (d, J = 5.0 Hz), 115.8 (d, J = 22.0 Hz), 21.3; ^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.9; HRMS (EI) m/z calcd for $C_{25}H_{19}F$ $[M]^+$ 338.1471, found 338.1462.



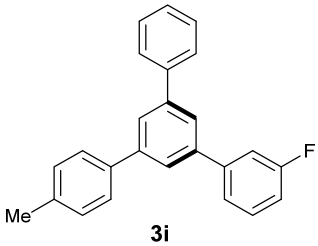
3g

4-fluoro-5'-phenyl-1,1':3',1''-terphenyl (known compound) [3] White solid, 48.7 mg, 50% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.80 – 7.74 (m, 1H), 7.74 – 7.70 (m, 2H), 7.70 – 7.66 (m, 4H), 7.66 – 7.59 (m, 2H), 7.51 – 7.42 (m, 4H), 7.42 – 7.34 (m, 2H), 7.20 – 7.09 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 162.8 (d, J = 245.0 Hz), 142.6, 141.5, 141.2, 137.4 (d, J = 3.0 Hz), 129.1, 129.0, 127.8, 127.5, 125.3, 125.2, 115.9 (d, J = 21.0 Hz); ^{19}F NMR (376 MHz, Chloroform-*d*) δ -115.3.



3h

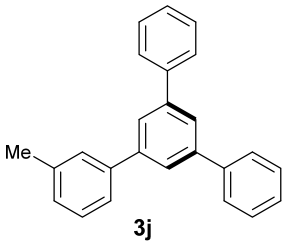
4,4''-dichloro-5'-(p-tolyl)-1,1':3',1''-terphenyl White solid, 53.7 mg, 46% yield. m.p. 239.5-240.2 ℃; IR (cm^{-1}): 3535, 2392, 2280, 1598, 1513, 1492, 1441, 1092, 1012, 811; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 – 7.67 (m, 2H), 7.64 (s, 1H), 7.62 – 7.52 (m, 6H), 7.43 (d, J = 8.2 Hz, 4H), 7.28 (d, J = 8.0 Hz, 2H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.8, 141.4, 139.6, 138.0, 137.8, 133.9, 129.8, 129.2, 128.7, 127.3, 125.2, 124.6, 21.3; HRMS (EI) m/z calcd for $C_{25}H_{18}Cl_2$ $[M]^+$ 388.0786, found 388.0779.



3i

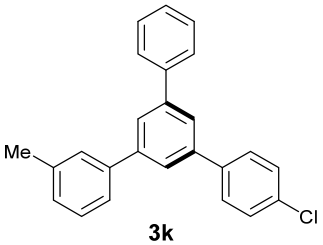
3-fluoro-4''-methyl-5'-phenyl-1,1':3',1''-terphenyl White solid, 50.7 mg, 50% yield. m.p. 135.0-136.4 ℃; IR (cm^{-1}): 3056, 3035, 2923, 2851, 2367, 1584, 1514, 1179, 756, 697; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 – 7.77 (m, 1H), 7.76 –

7.71 (m, 2H), 7.71 – 7.64 (m, 2H), 7.63 – 7.55 (m, 2H), 7.52 – 7.45 (m, 3H), 7.44 – 7.35 (m, 3H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.13 – 7.01 (m, 1H), 2.42 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.4 (d, *J* = 244.0 Hz), 143.6 (d, *J* = 8.0 Hz), 142.6, 142.5, 141.1, 138.1, 137.7, 130.4 (d, *J* = 8.0 Hz), 129.8, 129.0, 127.8, 127.5, 127.3, 125.7, 125.0, 124.9, 123.1 (d, *J* = 2.0 Hz), 114.44 (d, *J* = 21.0 Hz), 114.38 (d, *J* = 22.0 Hz), 21.3; ^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.0; HRMS (EI) m/z calcd for $C_{25}H_{19}F$ $[M]^+$ 338.1471, found 338.1462.



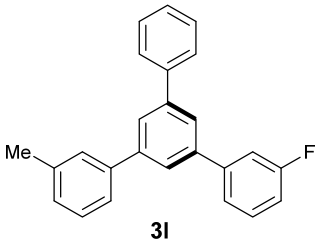
3j

3-methyl-5'-phenyl-1,1':3',1''-terphenyl (known compound)[4] White solid, 49.0 mg, 51% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 – 7.74 (m, 3H), 7.72 – 7.66 (m, 4H), 7.52 – 7.43 (m, 6H), 7.41 – 7.32 (m, 3H), 7.21 – 7.17 (m, 1H), 2.44 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.6, 142.4, 141.3, 141.2, 138.6, 129.0, 128.9, 128.4, 128.3, 127.7, 127.5, 125.3, 125.2, 124.6, 21.7.



3k

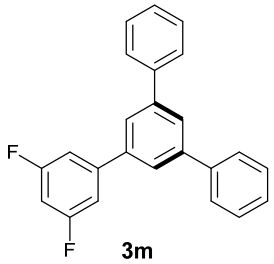
4''-chloro-3-methyl-5'-phenyl-1,1':3',1''-terphenyl White solid, 55.3 mg, 52% yield. m.p. 156.0-157.1 ℃; IR (cm^{-1}): 3034, 2924, 2854, 1596, 1494, 1092, 878, 826, 760, 699; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 – 7.68 (m, 1H), 7.64 (s, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.43 – 7.36 (m, 5H), 7.36 – 7.26 (m, 3H), 7.15 – 7.10 (m, 1H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.8, 142.6, 141.2, 141.1, 139.8, 138.6, 133.8, 129.1, 129.0, 128.9, 128.7, 128.5, 128.3, 127.8, 127.5, 125.6, 125.1, 125.0, 124.6, 21.7; HRMS (EI) m/z calcd for $C_{25}H_{19}Cl$ $[M]^+$ 354.1175, found 354.1168.



3l

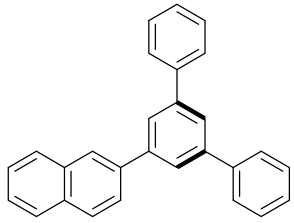
3-fluoro-3''-methyl-5'-phenyl-1,1':3',1''-terphenyl White solid, 51.7 mg, 51% yield. m.p. 138.5-139.1 ℃; IR (cm^{-1}): 3056, 2922, 1610, 1582, 1408, 1265, 1178,

866, 752, 696; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (s, 1H), 7.77 (s, 2H), 7.71 (d, J = 7.5 Hz, 2H), 7.54 – 7.47 (m, 5H), 7.47 – 7.36 (m, 4H), 7.23 (d, J = 7.4 Hz, 1H), 7.10 (t, J = 7.6 Hz, 1H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.4 (d, J = 244.0 Hz), 143.6(d, J = 8.0 Hz), 142.8, 142.6, 141.2, 141.1, 141.0, 138.7, 130.4 (d, J = 8.0 Hz), 129.0, 128.9, 128.6, 128.3, 127.8, 127.5, 125.9, 125.2, 125.1, 124.6, 123.1 (d, J = 3.0 Hz), 114.5 (d, J = 21.0 Hz), 114.4 (d, J = 22.0 Hz), 21.7; ^{19}F NMR (376 MHz, Chloroform-*d*) δ -113.0; HRMS (EI) m/z calcd for $C_{25}H_{19}F$ $[M]^+$ 338.1471, found 338.1462.



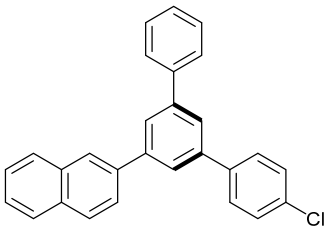
3m

3,5-difluoro-5'-phenyl-1,1':3',1''-terphenyl (known compound) [5] White solid, 45.1 mg, 44% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (s, 1H), 7.75 – 7.70 (m, 2H), 7.70 – 7.65 (m, 4H), 7.49 (t, J = 7.6 Hz, 4H), 7.44 – 7.36 (m, 2H), 7.24 – 7.16 (m, 2H), 6.90 – 6.74 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.5 (dd, J = 246.0 Hz), 144.6 (t, J = 9.0 Hz), 142.8, 140.8, 140.2, 129.1, 127.9, 127.5, 126.4, 125.0, 110.3 (dd, J = 18.0 Hz), 102.9 (t, J = 25.0 Hz); ^{19}F NMR (376 MHz, Chloroform-*d*) δ -109.5.



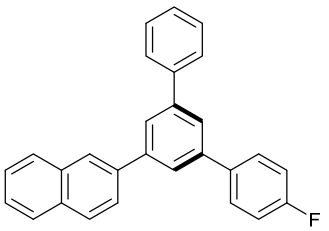
3n

2-([1,1':3',1''-terphenyl]-5'-yl)naphthalene (known compound) [6] White solid, 67.3 mg, 63% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.96 – 7.89 (m, 4H), 7.89 – 7.79 (m, 3H),7.75 – 7.68 (m, 4H), 7.54 – 7.45 (m, 6H), 7.43 – 7.34 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.6, 142.4, 141.3, 138.6, 133.8, 132.9, 129.0, 128.7, 128.4, 127.8, 127.7, 127.5, 126.5, 126.2, 125.8, 125.6, 125.4.



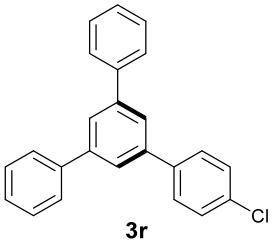
3o

2-(4-chloro-[1,1':3',1''-terphenyl]-5'-yl)naphthalene White solid, 60.9 mg, 52% yield. m.p. 188.2-189.0 ℃; IR (cm^{-1}): 3053, 2956, 2853, 1594, 1492, 1092, 885, 816, 760, 701; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.99 – 7.91 (m, 3H), 7.91 – 7.81 (m, 3H), 7.77 (s, 1H), 7.74 – 7.68 (m, 2H), 7.68 – 7.62 (m, 2H), 7.57 – 7.50 (m, 3H), 7.50 – 7.43 (m, 3H), 7.43 – 7.38 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.8, 142.6, 141.4, 141.1, 139.7, 138.4, 133.9, 133.8, 132.9, 129.2, 129.1, 128.8, 128.4, 127.8, 127.5, 126.6, 126.3, 126.2, 125.9, 125.7, 125.3, 125.2; HRMS (EI) m/z calcd for $C_{28}H_{19}Cl$ $[M]^+$ 390.1175, found 390.1170.



3p

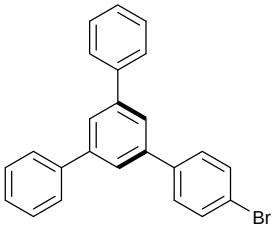
2-(4-fluoro-[1,1':3',1''-terphenyl]-5'-yl)naphthalene White solid, 50.5 mg, 45% yield. m.p. 193.4-194.2 ℃; IR (cm^{-1}): 3057, 2926, 2392, 1600, 1518, 1446, 1229, 1159, 834, 753; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.97 – 7.86 (m, 4H), 7.86 – 7.79 (m, 2H), 7.79 – 7.63 (m, 5H), 7.56 – 7.44 (m, 4H), 7.44 – 7.35 (m, 1H), 7.21 – 7.12 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 162.8 (d, J = 245.0 Hz), 142.7, 142.5, 141.6, 141.2, 138.5, 137.4 (d, J = 3.0 Hz), 133.8, 132.9, 129.1, 129.0, 128.7, 128.4, 127.84, 127.81, 127.5, 126.6, 126.3, 126.2, 125.8, 125.6, 125.4, 125.3, 115.9 (d, J = 21.0 Hz); ^{19}F NMR (376 MHz, Chloroform-*d*) δ -115.2; HRMS (EI) m/z calcd for $C_{28}H_{19}F$ $[M]^+$ 374.1471, found 374.1463.



3r

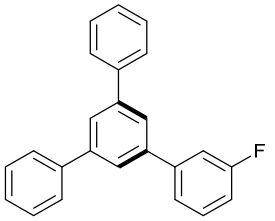
4-chloro-5'-phenyl-1,1':3',1''-terphenyl (known compound) [3] White solid, 58.2 mg, 57% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 – 7.77 (m, 1H), 7.73 (d, J = 1.6 Hz, 2H), 7.71 – 7.66 (m, 4H), 7.65 – 7.59 (m, 2H), 7.53 – 7.47 (m, 3H),

7.47 – 7.42 (m, 3H), 7.42 – 7.35 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.7, 141.2, 141.1, 139.7, 133.8, 129.2, 129.0, 128.7, 127.8, 127.5, 125.6, 125.1.



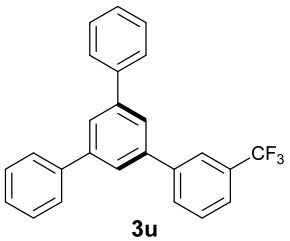
3s

4-bromo-5'-phenyl-1,1':3',1''-terphenyl (known compound) [6] White solid, 46.2 mg, 40% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 – 7.77 (m, 1H), 7.73 (d, *J* = 1.6 Hz, 2H), 7.71 – 7.64 (m, 4H), 7.63 – 7.53 (m, 4H), 7.52 – 7.44 (m, 4H), 7.43 – 7.34 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.7, 141.3, 141.1, 140.2, 132.1, 129.1, 129.0, 127.8, 127.5, 125.7, 125.0, 122.0.



3t

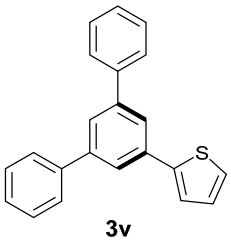
3-fluoro-5'-phenyl-1,1':3',1''-terphenyl (known compound) [6] White solid, 55.4 mg, 57% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 – 7.78 (m, 1H), 7.78 – 7.73 (m, 2H), 7.72 – 7.66 (m, 4H), 7.52 – 7.46 (m, 5H), 7.45 – 7.36 (m, 4H), 7.15 – 7.02 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.4 (d, *J* = 245.0 Hz), 143.6 (d, *J* = 8.0 Hz), 142.7, 141.2, 141.1, 141.1, 130.5 (d, *J* = 9.0 Hz), 129.0, 127.8, 127.5, 125.9, 125.2, 123.1 (d, *J* = 2.0 Hz), 114.5 (d, *J* = 21.0 Hz), 114.4 (d, *J* = 21.0 Hz); ^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.9.



3u

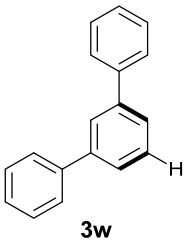
5'-phenyl-3-(trifluoromethyl)-1,1':3',1''-terphenyl White solid, 44.9 mg, 40% yield. m.p. 199.3-200.6 ℃; IR (cm^{-1}): 3035, 2924, 2853, 2377, 1596, 1497, 1321, 1126, 756, 698; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 (s, 1H), 7.87 (d, *J* = 7.6 Hz, 1H), 7.84 – 7.80 (m, 1H), 7.77 (d, *J* = 1.6 Hz, 2H), 7.72 – 7.67 (m, 4H), 7.67 – 7.56 (m, 2H), 7.52 – 7.46 (m, 4H), 7.44 – 7.36 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 142.8, 142.1, 141.1, 141.0, 131.4 (q, *J* = 32.0 Hz), 130.8, 129.5, 129.1, 127.9, 127.5, 126.1, 125.3, 124.3 (d, *J* = 271.0 Hz), 124.5 – 124.2 (m), 122.8;

^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.5; HRMS (EI) m/z calcd for $C_{25}H_{17}F_3$ $[M]^+$ 374.1282, found 374.1273.



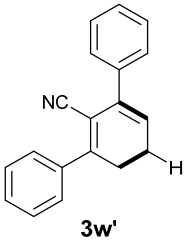
3v

2-([1,1':3',1''-terphenyl]-5'-yl)thiophene White solid, 40.0 mg, 43% yield; m.p. 180.2-181.0 ℃; IR (cm^{-1}): 3057, 2924, 1595, 1489, 1313, 819, 759, 698. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 – 7.79 (m, 2H), 7.76 – 7.67 (m, 5H), .7.55 – 7.47 (m, 4H), 7.47 – 7.39 (m, 3H), 7.34 (d, *J* = 5.04 Hz, 1H), 7.18 – 7.10 (m, 1H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 144.4, 142.7, 141.0, 135.5, 129.0, 128.2, 127.8, 127.5, 125.6, 125.3, 124.0, 123.7. HRMS (EI) m/z calcd for $C_{22}H_{16}S$ $[M]^+$ 312.0973, found 312.0966.



3w

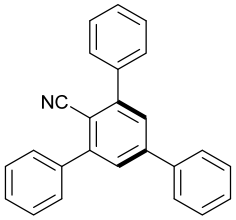
1,1':3',1''-terphenyl (known compound) [7] White solid, 34.5 mg, 50% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.81 (s, 1H), 7.66 (s, 2H), 7.64 (s, 2H), 7.60 – 7.55 (m, 2H), 7.54 – 7.50 (m, 1H), 7.49 – 7.43 (m, 4H), 7.40 – 7.34 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 141.9, 141.3, 129.3, 128.9, 127.6, 127.4, 126.3, 126.2.



3w'

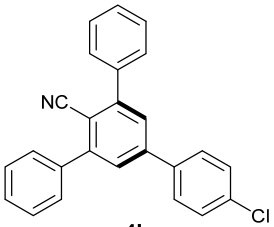
4',5'-dihydro-[1,1':3',1''-terphenyl]-2'-carbonitrile White solid, 42.4 mg, 55% yield. m.p. 74.5-75.3 ℃; IR (cm^{-1}): 3057, 2919, 2850, 2397, 1598, 1552, 1492, 1443, 761, 698; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61 – 7.56 (m, 2H), 7.48 – 7.41 (m, 3H), 7.43 – 7.33 (m, 5H), 6.12 (t, *J* = 4.8 Hz, 1H), 2.81 – 2.73 (m, 2H), 2.52 – 2.44 (m, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 155.8, 139.1, 138.5, 136.0, 129.6,

128.7, 128.4, 128.1, 127.92, 127.90, 126.0, 118.0, 108.8, 29.7, 22.5; HRMS (ESI) m/z calcd for $C_{19}H_{15}NNa^+$ $[M+Na]^+$ 280.1097, found 280.1097.



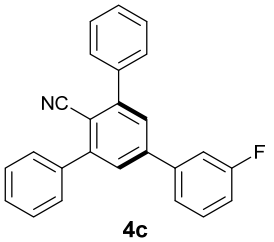
4a

5'-phenyl-[1,1':3',1''-terphenyl]-2'-carbonitrile (known compound) [8] White solid, 49.7 mg, 50% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 – 7.67 (m, 3H), 7.67 – 7.60 (m, 5H),7.56 – 7.52 (m, 1H), 7.52 – 7.48 (m, 5H), 7.48 – 7.45 (m, 2H), 7.45 – 7.40 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 147.5, 145.3, 139.2, 138.9, 129.3, 129.2, 128.9, 128.8, 127.7, 127.5, 118.2, 109.2.



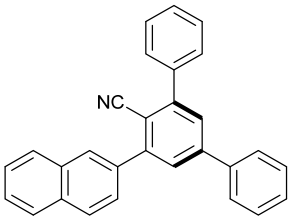
4b

5'-(4-chlorophenyl)-[1,1':3',1''-terphenyl]-2'-carbonitrile (known compound) [9] White solid, 62.5 mg, 57% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 (s, 4H), 7.63 – 7.60 (m, 3H), 7.60 – 7.57 (m, 1H), 7.55 – 7.50 (m, 3H), 7.50 – 7.48 (m, 2H), 7.48 – 7.42 (m, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 147.7, 144.0, 138.7, 137.6, 135.2, 129.5, 129.2, 129.0, 128.9, 128.7, 127.4, 118.0, 109.5.



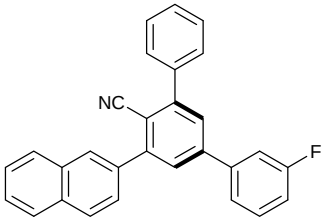
4c

5'-(3-fluorophenyl)-[1,1':3',1''-terphenyl]-2'-carbonitrile (known compound) [10] White solid, 52.4 mg, 50% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 – 7.60 (m, 6H), 7.56 – 7.47 (m, 6H), 7.47 – 7.42 (m, 2H), 7.37 (d, J = 9.2 Hz, 1H), 7.18 – 7.07 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.4 (d, J = 246.0 Hz), 147.7, 143.9, 141.4 (d, J = 8.0 Hz), 138.6, 130.8 (d, J = 8.0 Hz), 129.2, 129.0, 128.9, 127.6, 123.2 (d, J = 3.0 Hz), 118.0, 115.8 (d, J = 21.0 Hz), 114.5 (d, J = 22.0 Hz), 109.8.; ^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.1.



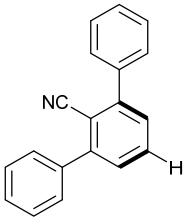
4d

5'-(naphthalen-2-yl)-[1,1':3',1''-terphenyl]-4'-carbonitrile (known compound) [8] White solid, 56.1 mg, 49% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.12 (s, 1H), 7.99 (d, J = 8.8 Hz, 1H), 7.96 – 7.88 (m, 2H), 7.81 – 7.74 (m, 2H), 7.73 – 7.63 (m, 5H), 7.58 – 7.51 (m, 4H), 7.51 – 7.39 (m, 4H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 147.6, 145.3, 139.2, 133.3, 129.3, 129.2, 128.9, 128.8, 128.7, 128.6, 127.9, 127.7, 127.5, 126.9, 118.3, 109.4.



4e

3-fluoro-5'-(naphthalen-2-yl)-[1,1':3',1''-terphenyl]-4'-carbonitrile White solid, 57.5 mg, 48% yield. m.p. 255.2-256.7 ℃; IR (cm^{-1}): 3058, 2852, 1592, 1493, 1266, 1157, 1125, 853, 748, 699; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.11 (s, 1H), 7.99 (d, J = 8.8 Hz, 1H), 7.96 – 7.88 (m, 2H), 7.79 – 7.72 (m, 2H), 7.71 – 7.61 (m, 3H), 7.59 – 7.51 (m, 4H), 7.51 – 7.43 (m, 3H), 7.43 – 7.36 (m, 1H), 7.19 – 7.07 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 163.4 (d, J = 245.0 Hz), 147.8, 147.7, 143.9, 141.4 (d, J = 7.0 Hz), 138.6, 136.0, 133.34, 133.30, 130.9 (d, J = 8.0 Hz), 129.2, 129.0, 128.9, 128.7, 128.6, 128.5, 127.9, 127.8, 127.7, 127.0, 126.8, 126.7, 123.2 (d, J = 3.03 Hz), 118.1, 115.8 (d, J = 21.0 Hz), 114.5 (d, J = 23.0 Hz), 110.0; ^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.0; HRMS (ESI) m/z calcd for $C_{29}H_{18}FNNa^{+}$ $[M+Na]^{+}$ 422.1315, found 422.1314.



4f

[1,1':3',1''-terphenyl]-2'-carbonitrile (known compound) [8] White solid, 34.4 mg, 45% yield; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.69 – 7.64 (m, 1H), 7.62 – 7.55

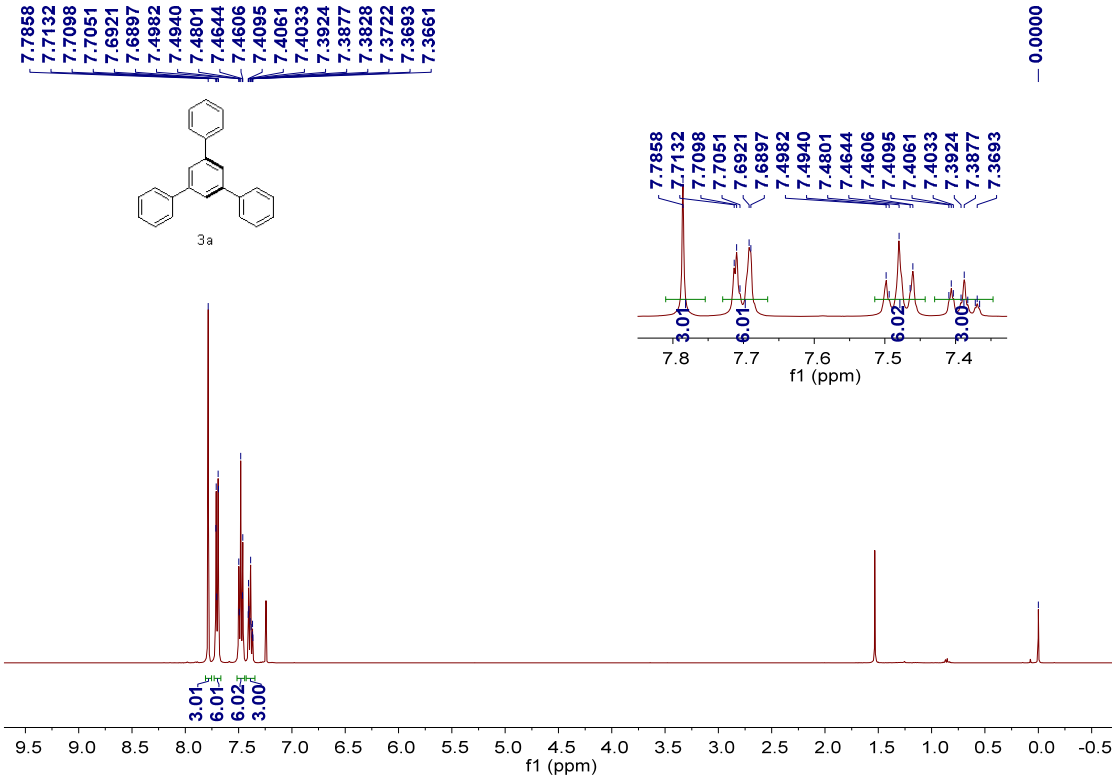
(m, 4H), 7.54 – 7.51 (m, 1H), 7.51 – 7.49 (m, 2H), 7.49 – 7.4511 (m, 4H), 7.45 – 7.42 (m, 1H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 147.1, 138.8, 132.4, 129.2, 129.0, 128.8, 128.7.

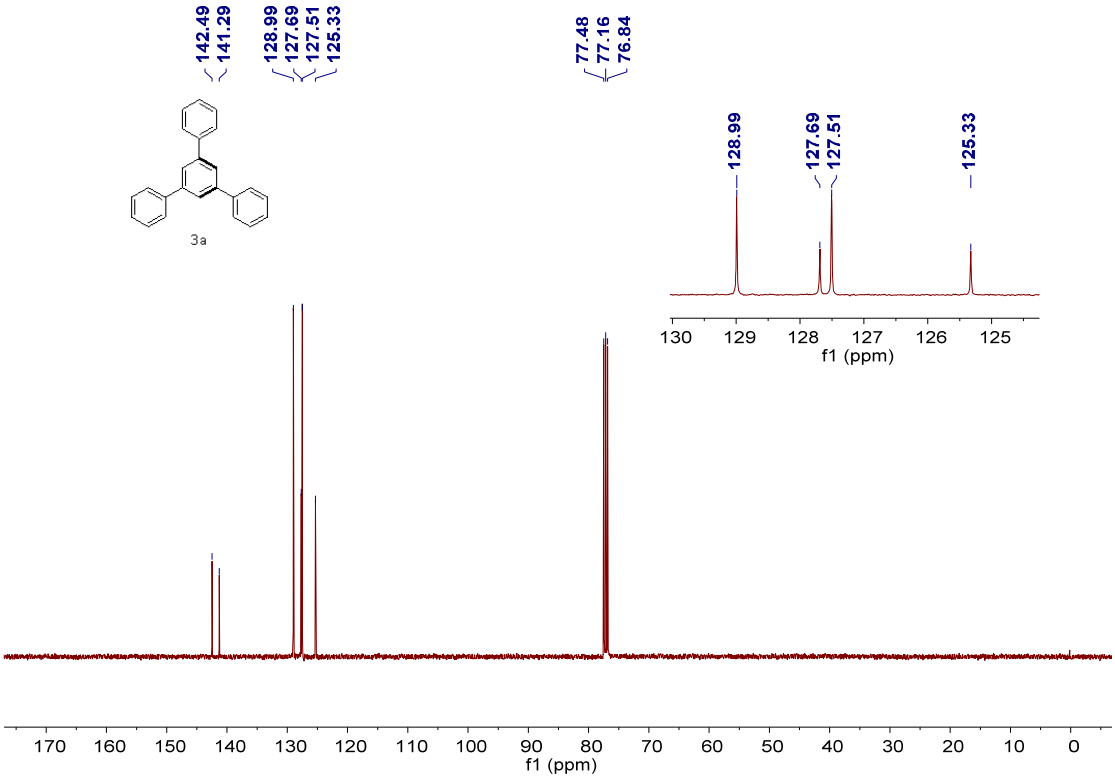
References.

1. G.-F. Zha, Q. Zheng, J. Leng, P. Wu, H.-L. Qin, K. B. Sharpless, Palladium-Catalyzed Fluorosulfonylvinylation of Organic Iodides. *Angew Chem Int Ed Engl.,* 2017, **56**, 4849-4852.
2. Y.-T. Gu, P.-F. Hu, C.-J. Ni, X.-F. Tong, Phosphine-Catalyzed Addition/Cycloaddition Domino Reactions of beta'-Acetoxy Allenoate: Highly Stereoselective Access to 2-Oxabicyclo[3.3.1]nonane and Cyclopenta[a]pyrrolizine. *J Am Chem Soc.,* 2015, **137**, 6400-6406.
3. C.-L. Zhang, S. Ye, N-Heterocyclic Carbene-Catalyzed Construction of 1,3,5-Trisubstituted Benzenes from Bromoenals and α-Cyano-β-methylenones, *Org. Lett.,* 2016, **18**, 6408 - 6411.
4. C.-L. Zhang, Z.-F. Zhang, Z.-H. Xia, Y.-F. Han, S. Ye, DBU-Mediated Construction of 1,3,5-Trisubstituted Benzenes via Annulation of α,β-Unsaturated Carboxylic Acids and α-Cyano-β-methylenones, *J. Org. Chem.,* 2018, **83**, 12507 - 12513.
5. J.-Y. Pan, R.-F. He, J.-H. Tan, Y.-N. Li, *PCT Int. Appl.* 2018095391, 31 May 2018.
6. F.-Y. Wang, J.-H. Shen, G.-L. Cheng, X.-L. Cui, Practical access to 1,3,5-triarylbenzenes from chalcones and DMSO, *RSC Advances.,* 2015, **5**, 73180 - 73183.
7. L.-F. Liu, Y.-H. Zhang, B.-W. Xin, Synthesis of Biaryls and Polyaryls by Ligand-Free Suzuki Reaction in Aqueous Phase, *J. Org. Chem.,* 2006, **71**, 3994 - 3997.
8. Q.-F. Jia, J. Wang, Synthesis of Biaryls and Polyaryls by Ligand-Free Suzuki Reaction in Aqueous Phase, *Org. Lett.,* 2016, **18**, 2212 - 2215.
9. C.-L. Zhang, Z.-H. Gao, Z.-Q. Liang, S. Ye, N-Heterocyclic Carbene-Catalyzed Synthesis of Multi- Substituted Benzenes from Enals and α-Cyano-β-methylenones, *Adv. Synth. Catal.,* 2016, **358**, 2862 - 2866.
10. M. K. R. Singam, A. Nagireddy and S. R. Maddi, Base-mediated benzannulation of α-cyanocrotonates with ynones: facile synthesis of benzonitriles and fluorenes, *Green Chem.,* 2020, **22**, 2370 - 2374.

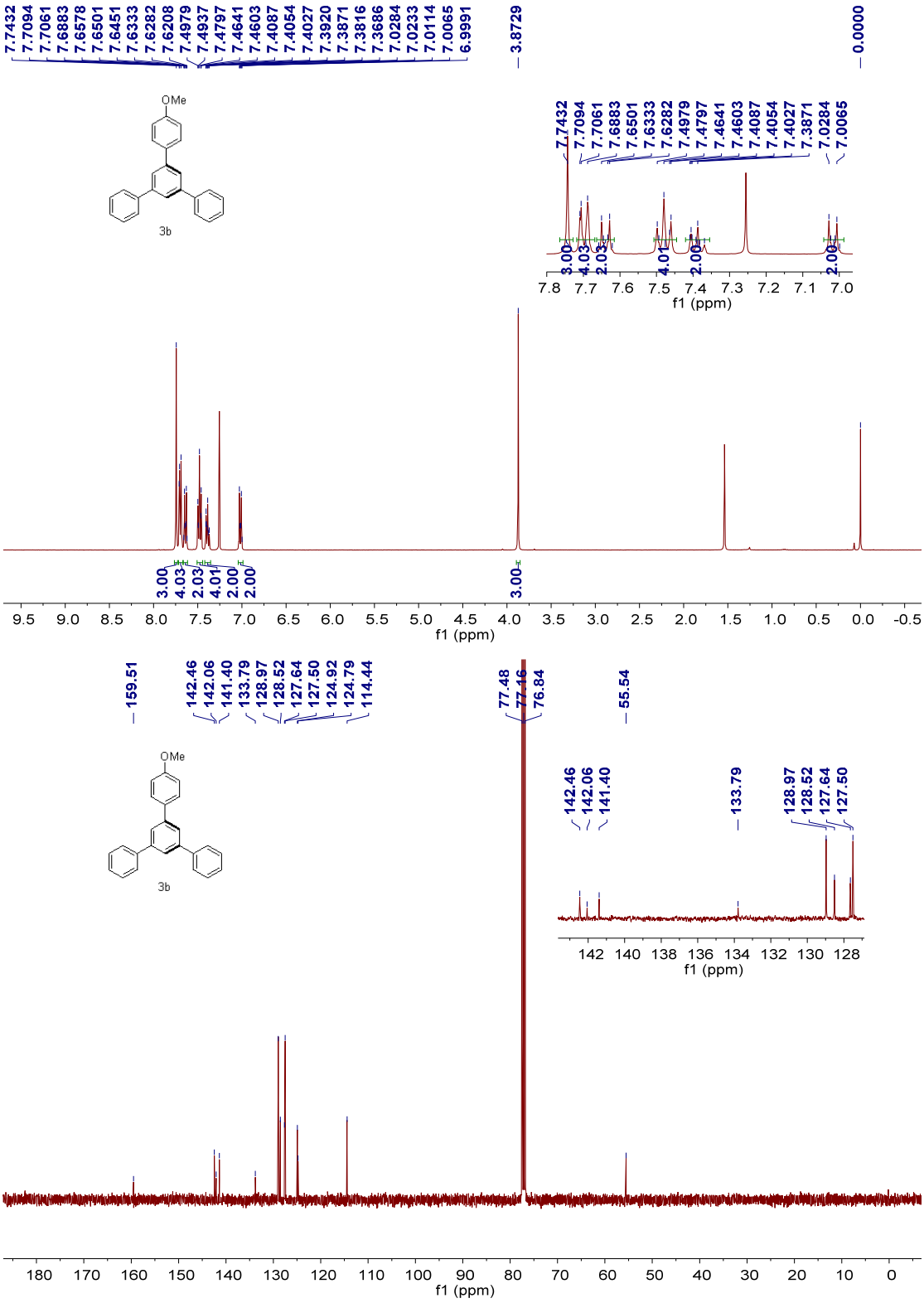
7. Copies of NMR spectra

3a





3b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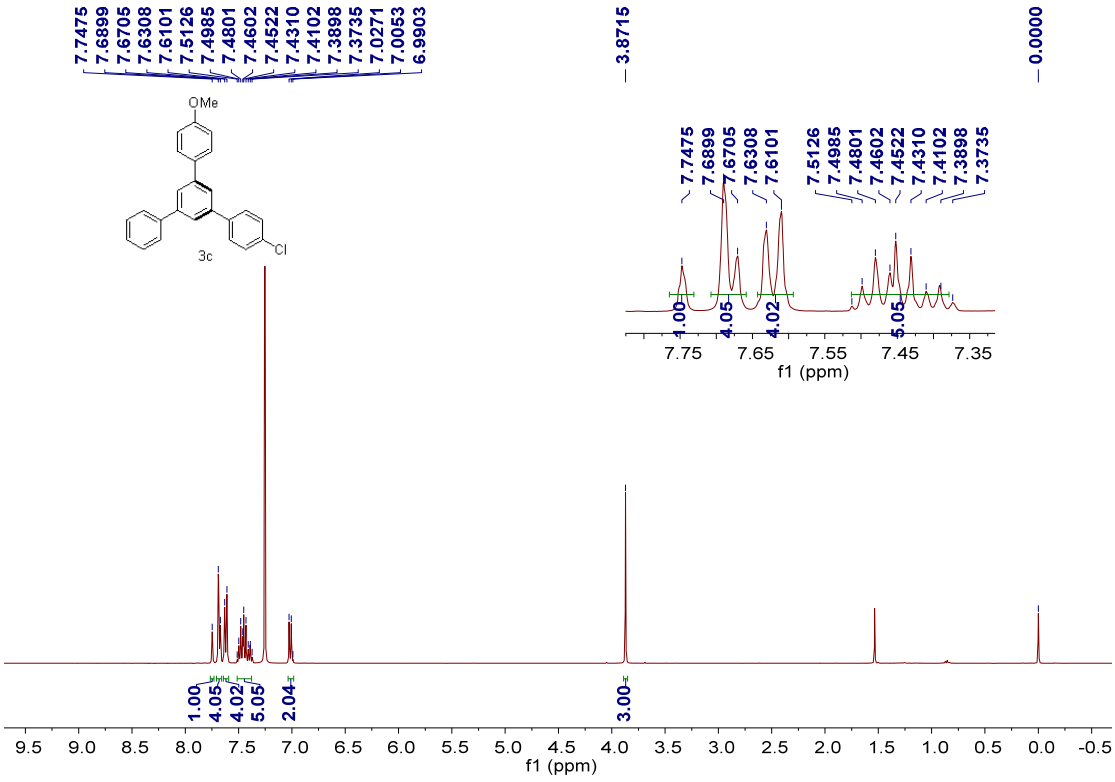
3.8729
0.0000
OMe
3b
3.00
4.03
2.03
4.01
2.00
2.00
3.00
f1 (ppm)
159.51
142.46
142.06
141.40
133.79
128.97
128.52
127.64
127.50
124.92
124.79
114.44
77.48
77.16
76.84
55.54
f1 (ppm)

3c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7.6705
7.6308
7.6101
7.5126
7.4985
7.4801
7.4602
7.4522
7.4310
7.4102
7.3898
7.3735
7.0271
7.0053
6.9903
3.8715
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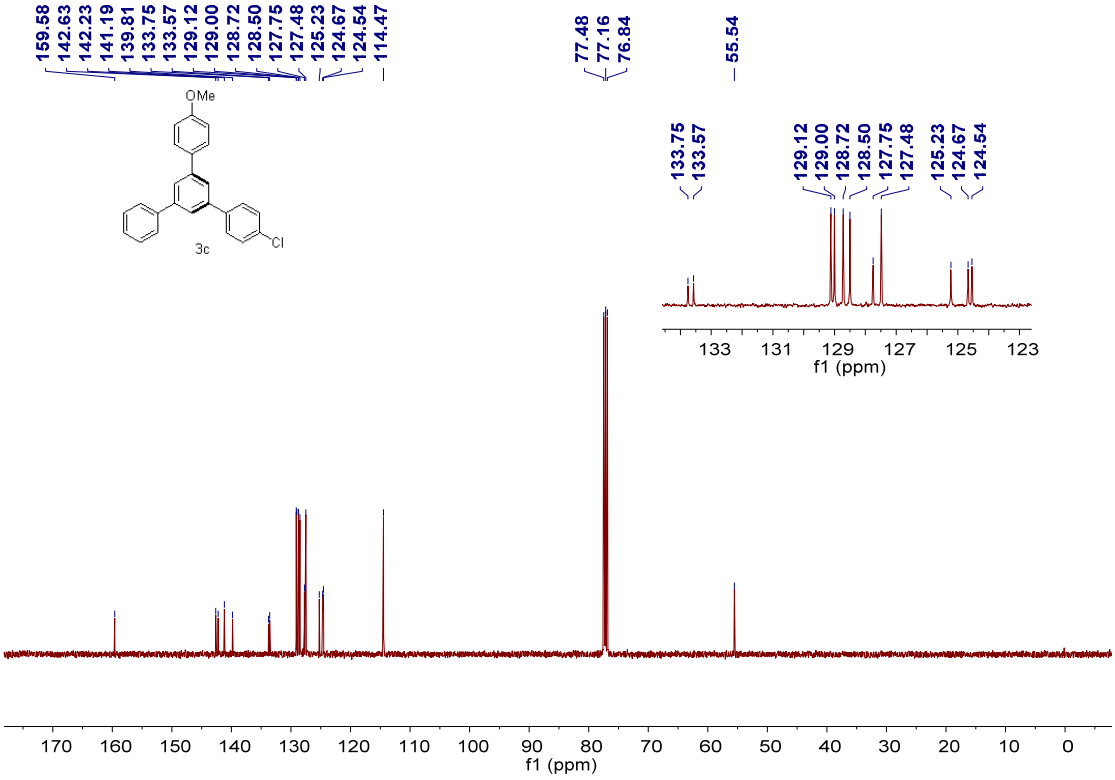
OMe
Cl
3c
1.00
4.05
4.02
5.05
2.04
3.00
f1 (ppm)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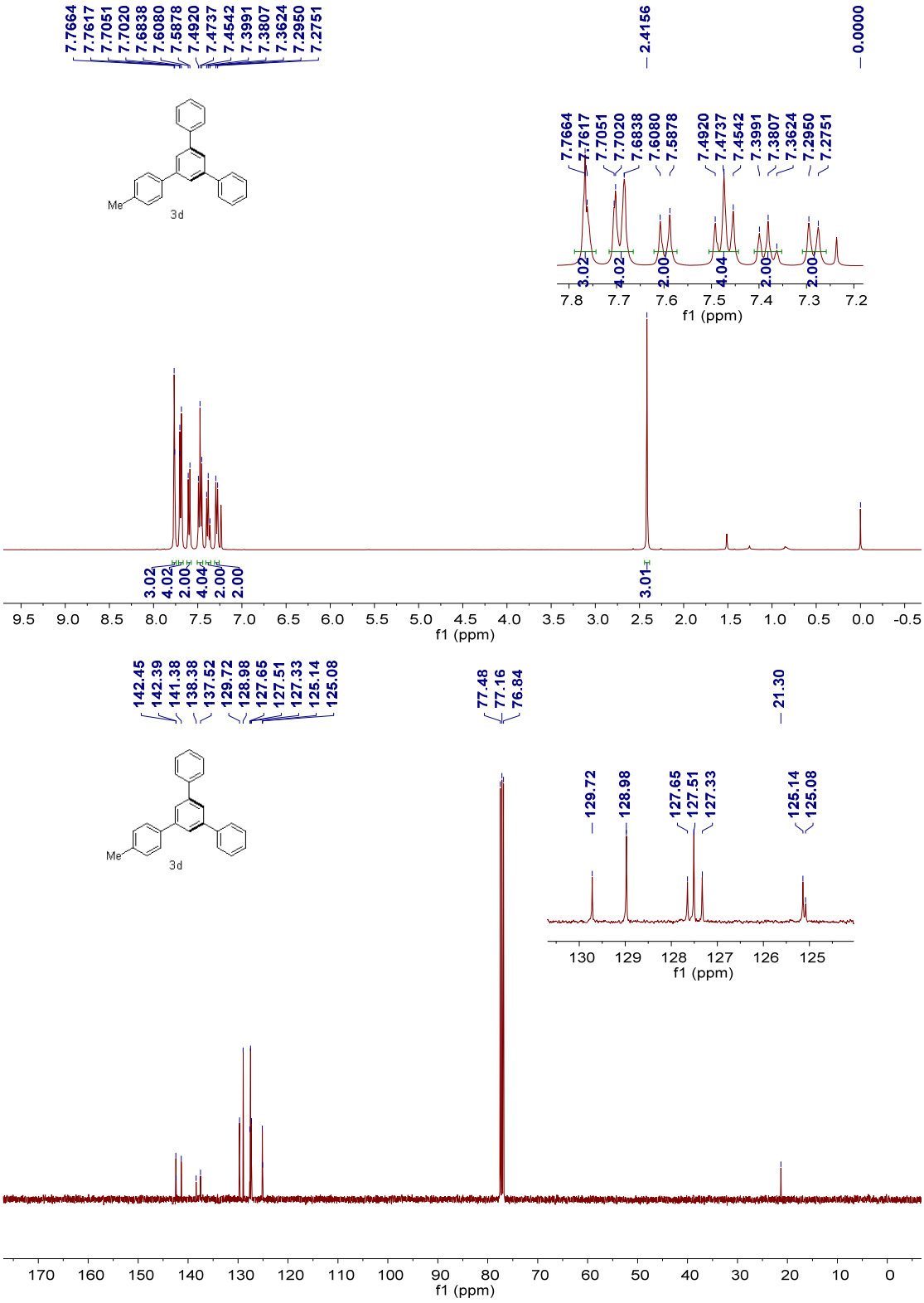
142.23
141.19
139.81
133.75
133.57
129.12
129.00
128.72
128.50
127.75
127.48
125.23
124.67
124.54
114.47
77.48
77.16
76.84
55.54
OMe
Cl
3c
f1 (ppm)

3d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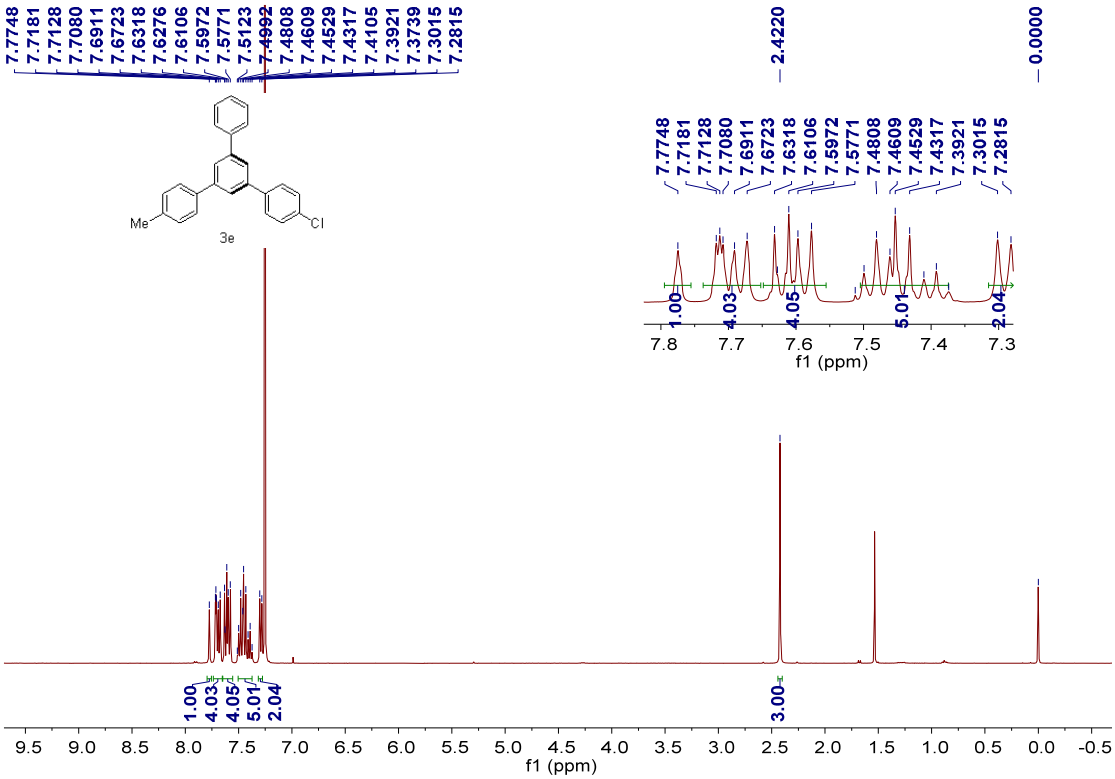
7.7617
7.7051
7.7020
7.6838
7.6080
7.5878
7.4920
7.4737
7.4542
7.3991
7.3807
7.3624
7.2950
7.2751
2.4156
0.0000
Me
3d
3.02
4.02
2.00
4.04
2.00
2.00
3.01
f1 (ppm)
142.45
142.39
141.38
138.38
137.52
129.72
128.98
127.65
127.51
127.33
125.14
125.08
77.48
77.16
76.84
21.30
f1 (ppm)

3e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7.6911
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7.6318
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7.6106
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7.5771
7.5123
7.4992
7.4808
7.4609
7.4529
7.4317
7.4105
7.3921
7.3739
7.3015
7.2815
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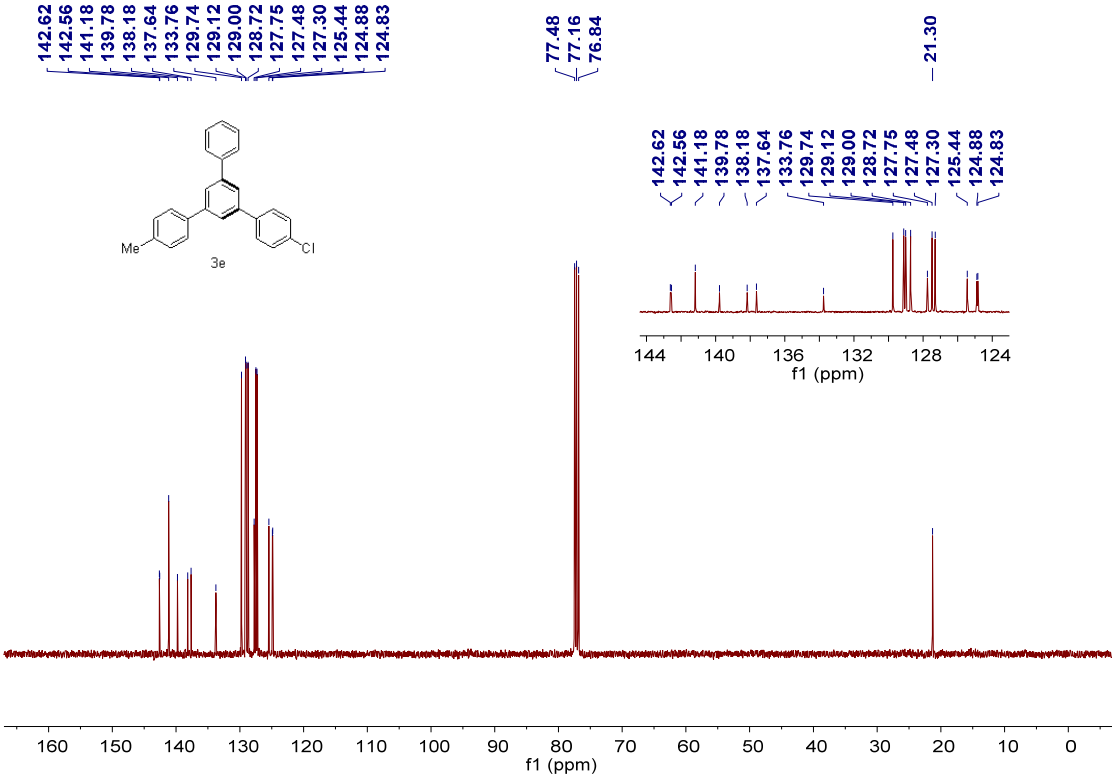
Me
Cl
3e
1.00
4.03
4.05
5.01
2.04
3.00
f1 (ppm)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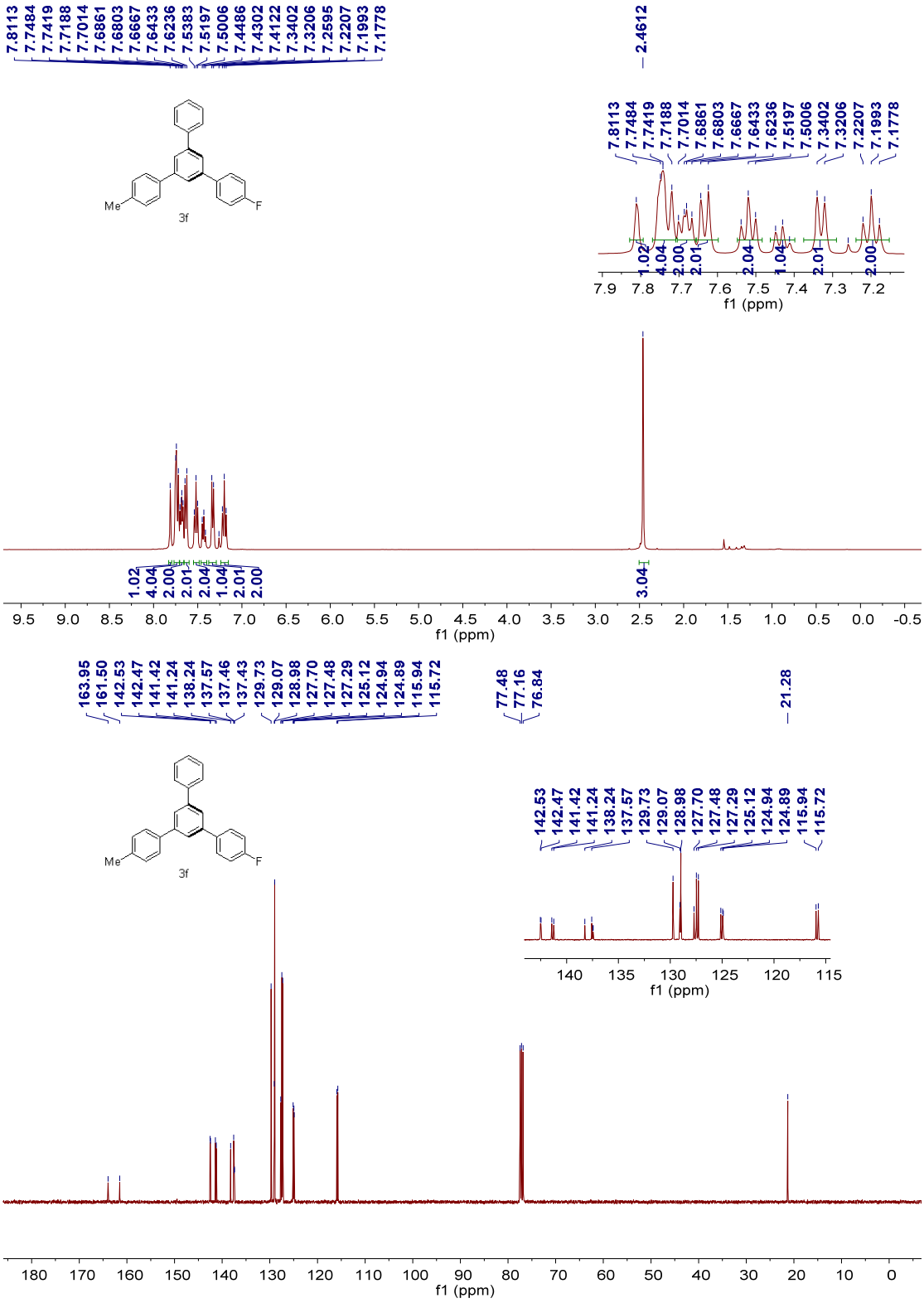
142.56
141.18
139.78
138.18
137.64
133.76
129.74
129.12
129.00
128.72
127.75
127.48
127.30
125.44
124.88
124.83
77.48
77.16
76.84
21.30
Me
Cl
3e
f1 (ppm)

3f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7.7419
7.7188
7.7014
7.6861
7.6803
7.6667
7.6433
7.6236
7.5383
7.5197
7.5006
7.4486
7.4302
7.4122
7.3402
7.3206
7.2595
7.2207
7.1993
7.1778
2.4612
Me
F
3f
1.02
4.04
2.00
2.01
2.04
1.04
2.01
2.00
3.04
f1 (ppm)
163.95
161.50
142.53
142.47
141.42
141.24
138.24
137.57
137.46
137.43
129.73
129.07
128.98
127.70
127.48
127.29
125.12
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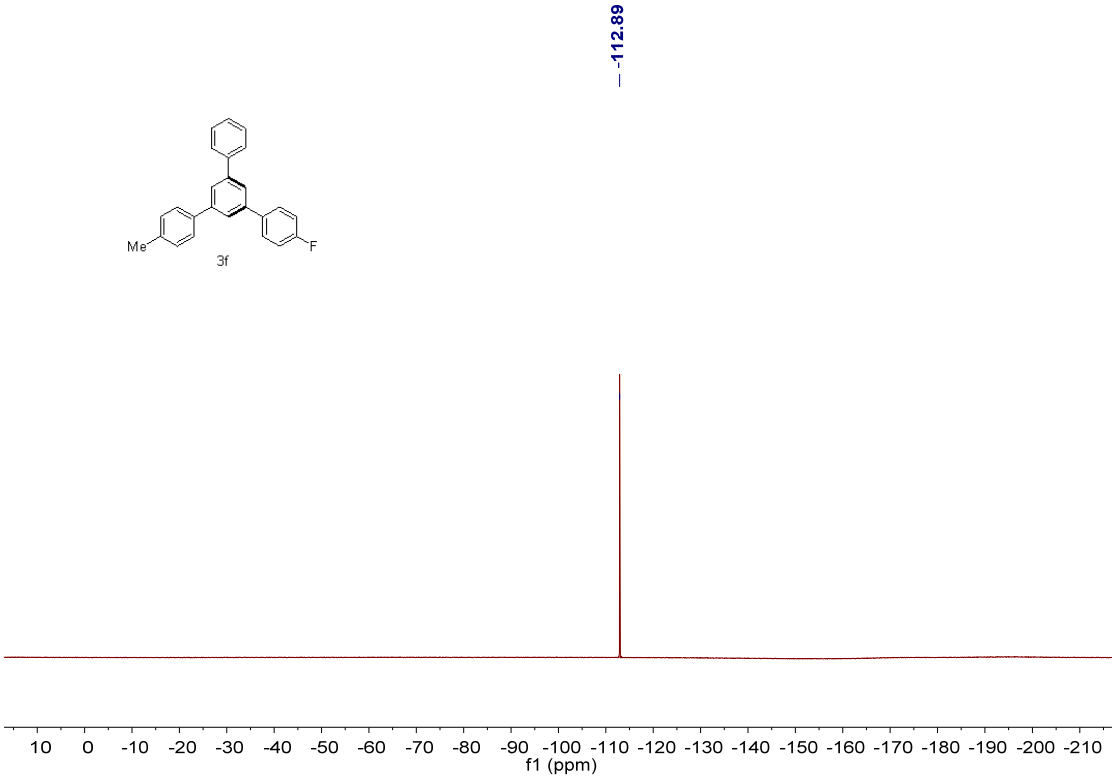
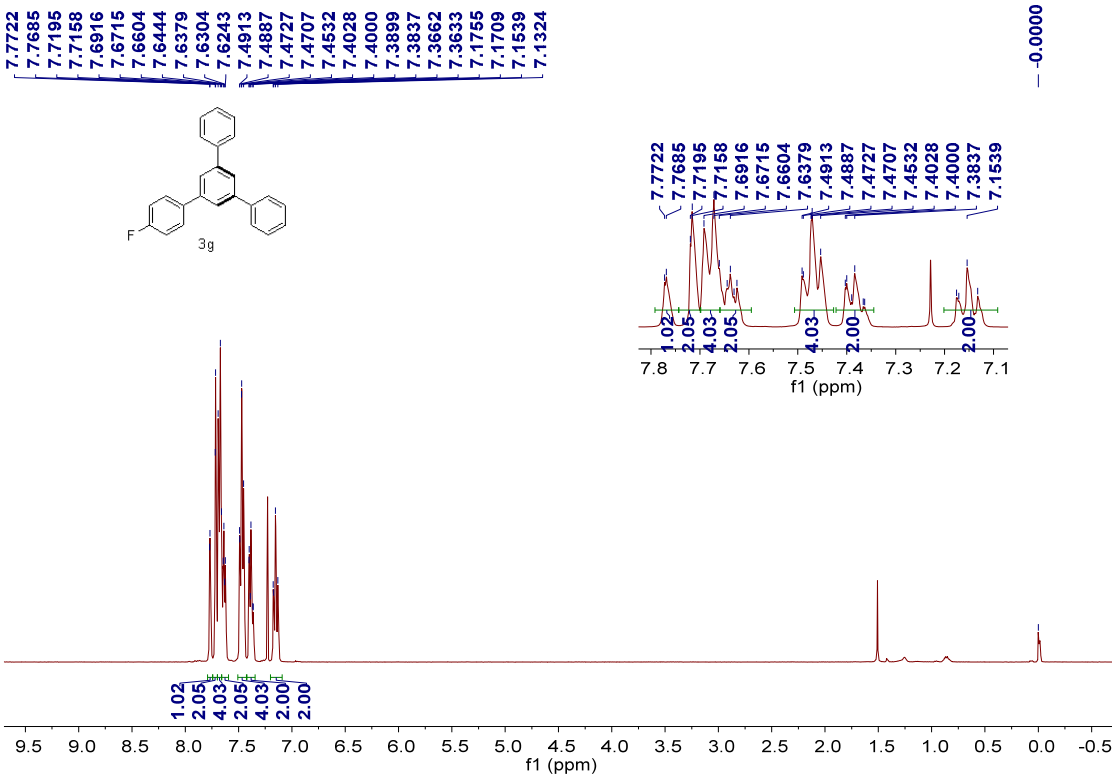
115.94
115.72
77.48
77.16
76.84
21.28


-112.89
Me
F
3f
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3g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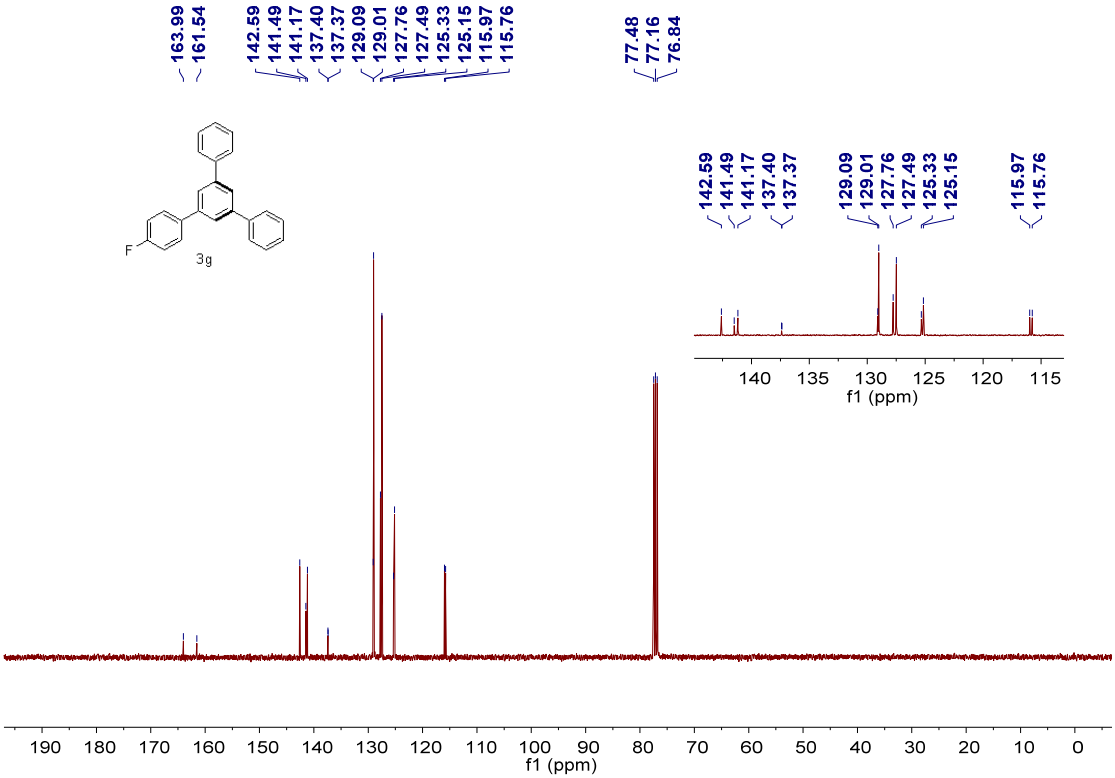
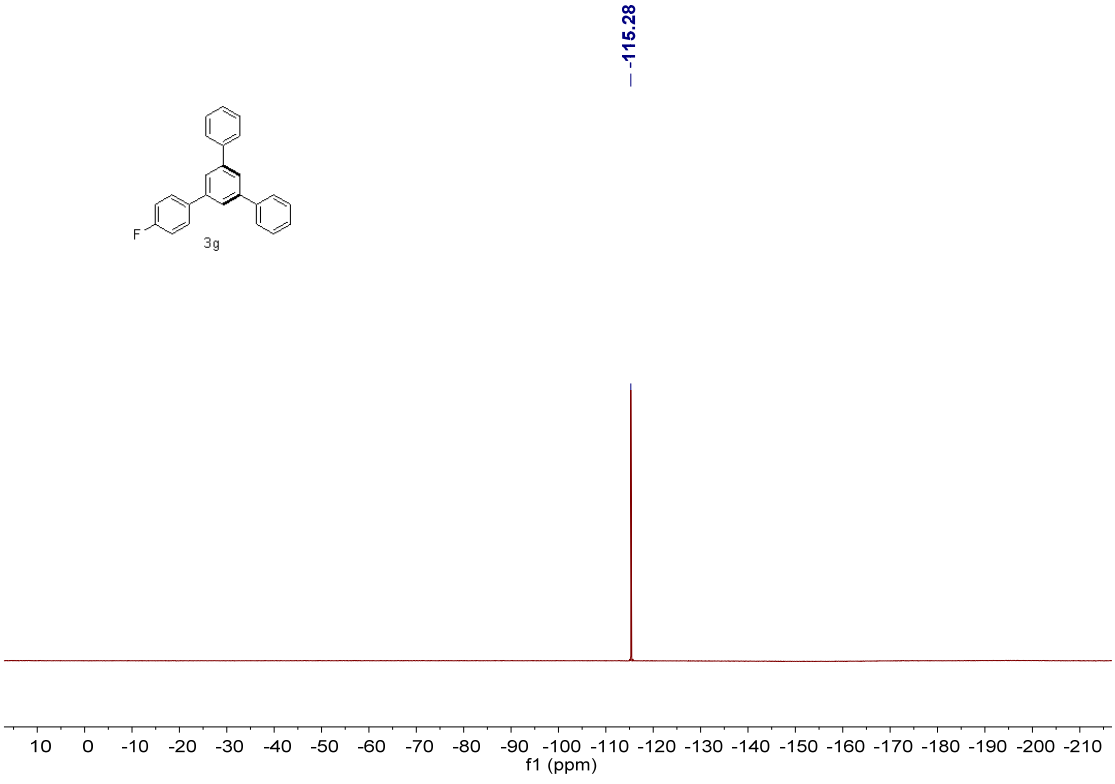
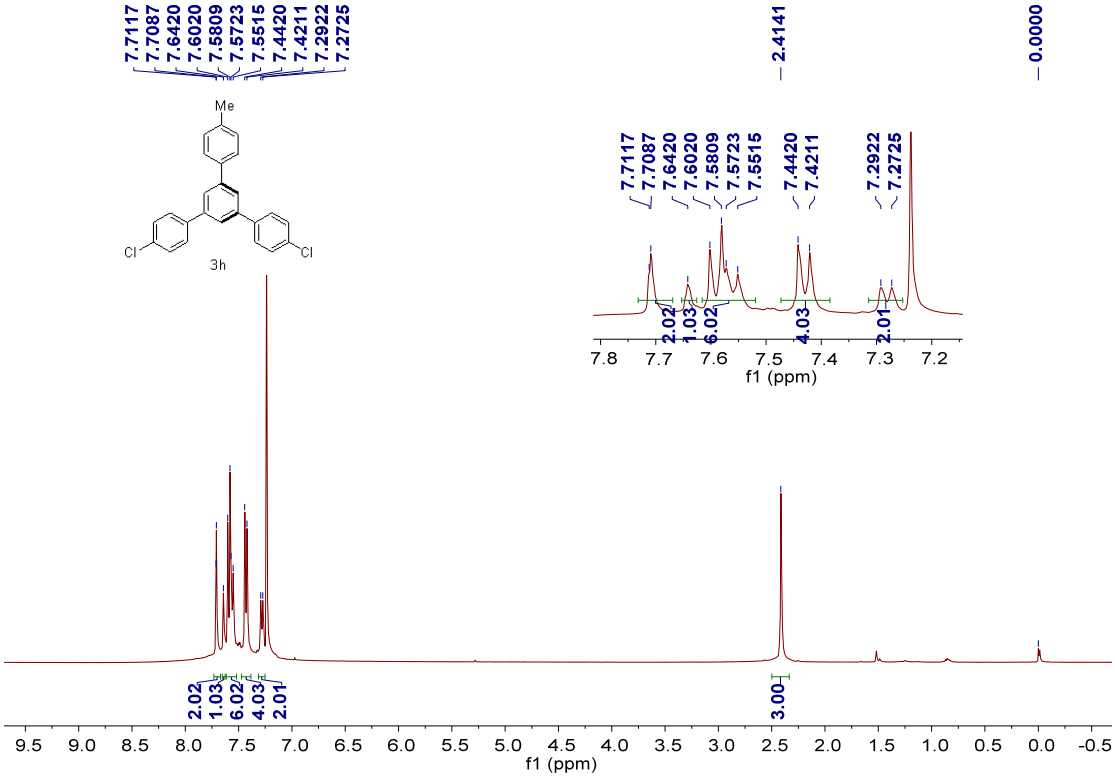
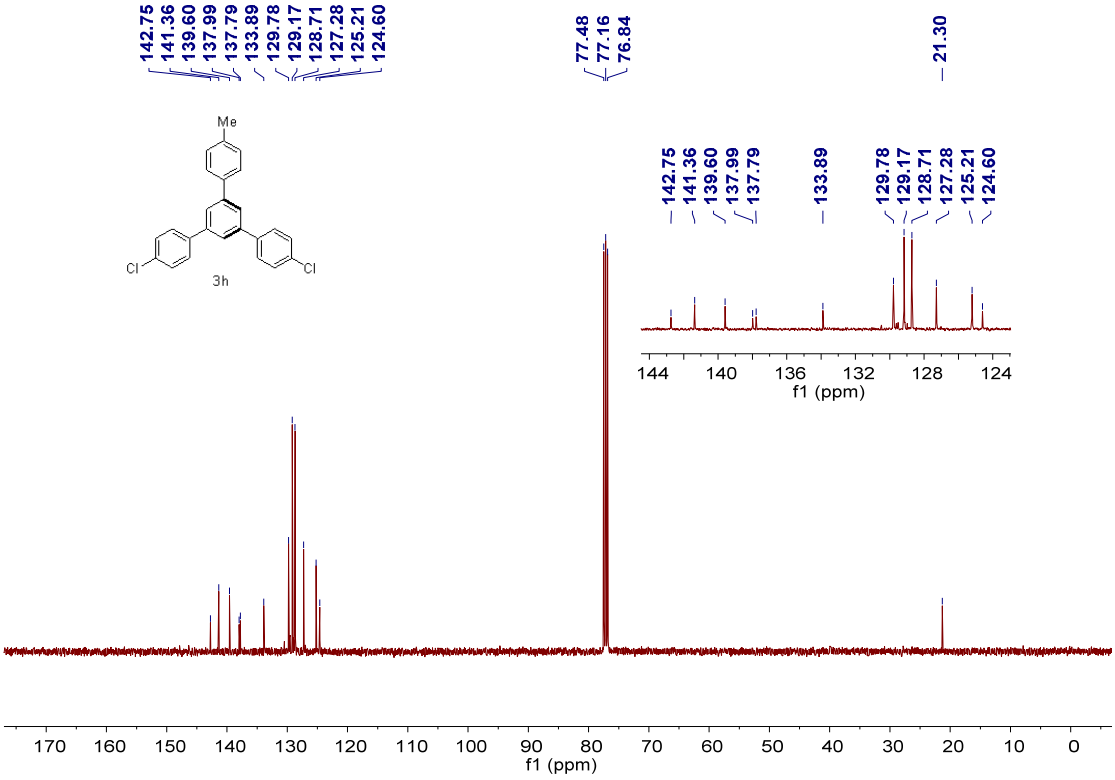
7.7195
7.7158
7.6916
7.6715
7.6604
7.6444
7.6379
7.6304
7.6243
7.4913
7.4887
7.4727
7.4707
7.4532
7.4028
7.4000
7.3899
7.3837
7.3662
7.3633
7.1755
7.1709
7.1539
7.1324
-0.0000
F
3g
1.02
2.05
4.03
2.05
4.03
2.00
2.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


163.99
161.54
142.59
141.49
141.17
137.40
137.37
129.09
129.01
127.76
127.49
125.33
125.15
115.97
115.76
77.48
77.16
76.84
F
3g
140 135 130 125 120 115
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

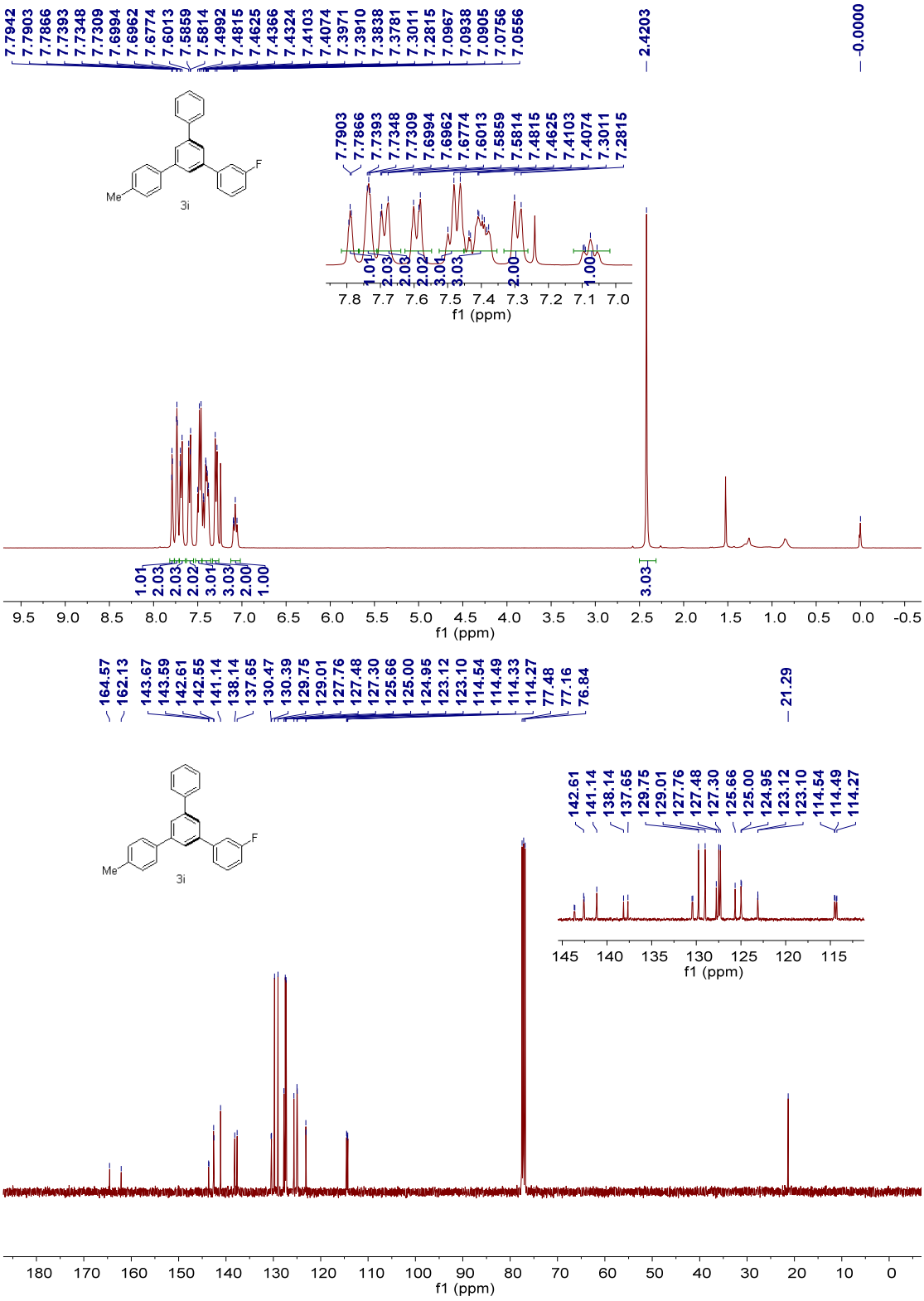

-115.28
F
3g
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3h


7.7117
7.7087
7.6420
7.6020
7.5809
7.5723
7.5515
7.4420
7.4211
7.2922
7.2725
2.4141
0.0000
Me
Cl
Cl
3h
2.02
1.03
6.02
4.03
2.01
3.00
f1 (ppm)


142.75
141.36
139.60
137.99
137.79
133.89
129.78
129.17
128.71
127.28
125.21
124.60
77.48
77.16
76.84
21.30
Me
Cl
Cl
3h
f1 (ppm)

3i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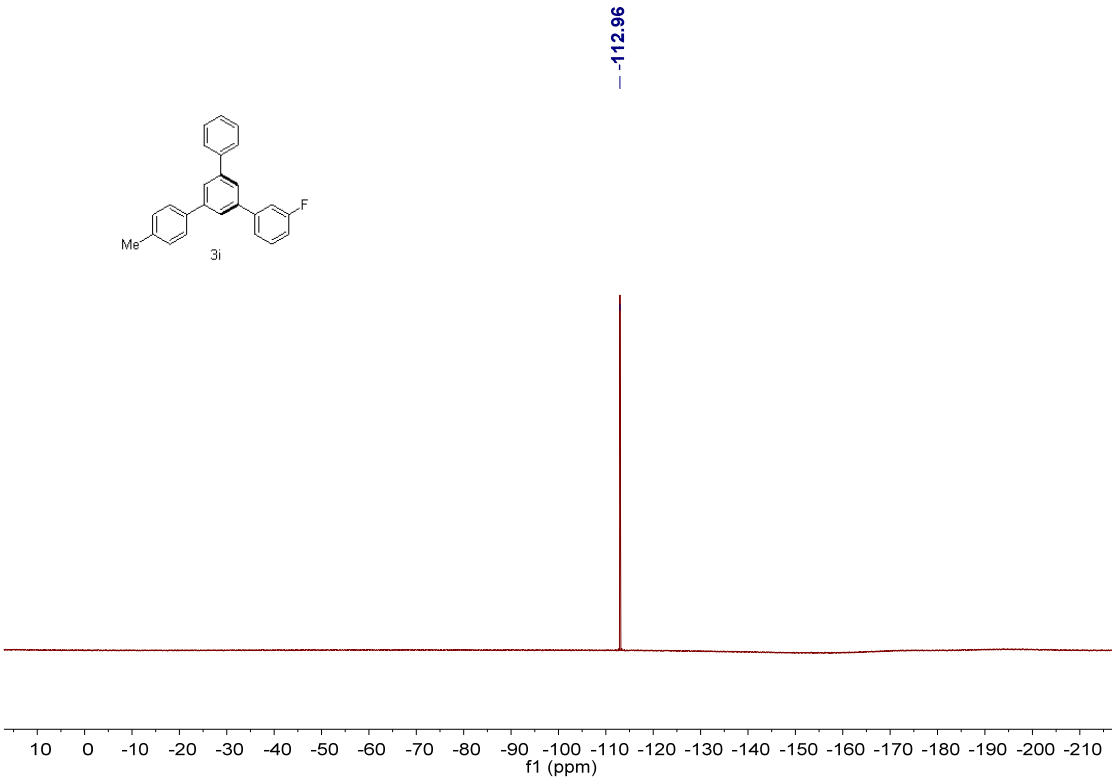
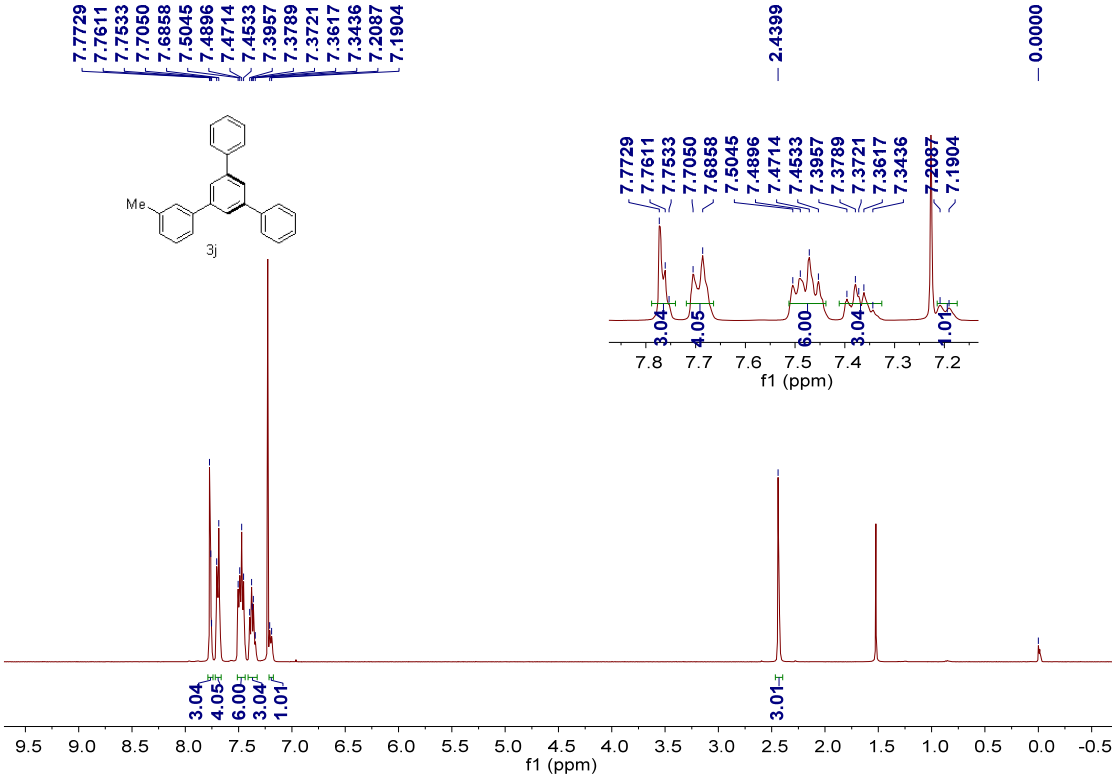
7.7866
7.7393
7.7348
7.7309
7.6994
7.6962
7.6774
7.6013
7.5859
7.5814
7.4992
7.4815
7.4625
7.4366
7.4324
7.4103
7.4074
7.3971
7.3910
7.3838
7.3781
7.3011
7.2815
7.0967
7.0938
7.0905
7.0756
7.0556
2.4203
-0.0000
Me
F
3i
1.01
2.03
2.03
2.02
3.01
3.03
2.00
1.00
3.03
f1 (ppm)
164.57
162.13
143.67
143.59
142.61
142.55
141.14
138.14
137.65
130.47
130.39
129.75
129.01
127.76
127.48
127.30
125.66
125.00
124.95
123.12
123.10
114.54
114.49
114.33
114.27
77.48
77.16
76.84
21.29
f1 (ppm)


-112.96
Me
F
3i
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3j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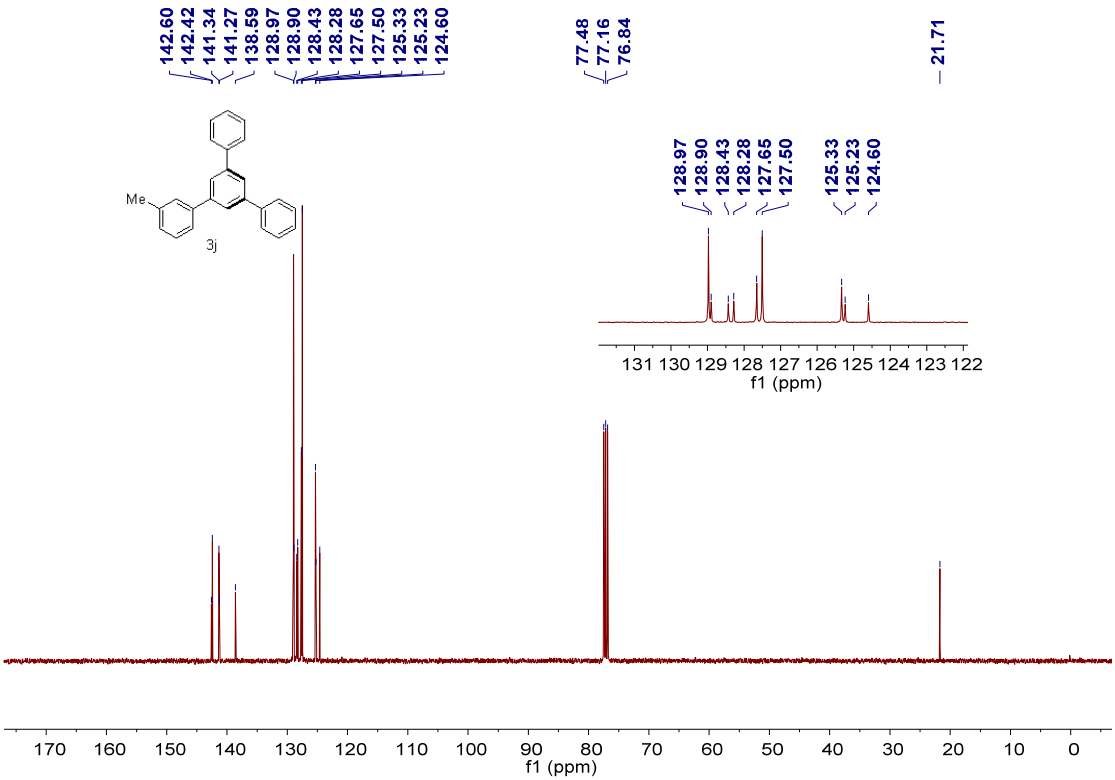
7.6858
7.5045
7.4896
7.4714
7.4533
7.3957
7.3789
7.3721
7.3617
7.3436
7.2087
7.1904
2.4399
0.0000
Me
3j
3.04
4.05
6.00
3.04
1.01
3.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
7.8 7.7 7.6 7.5 7.4 7.3 7.2
f1 (ppm)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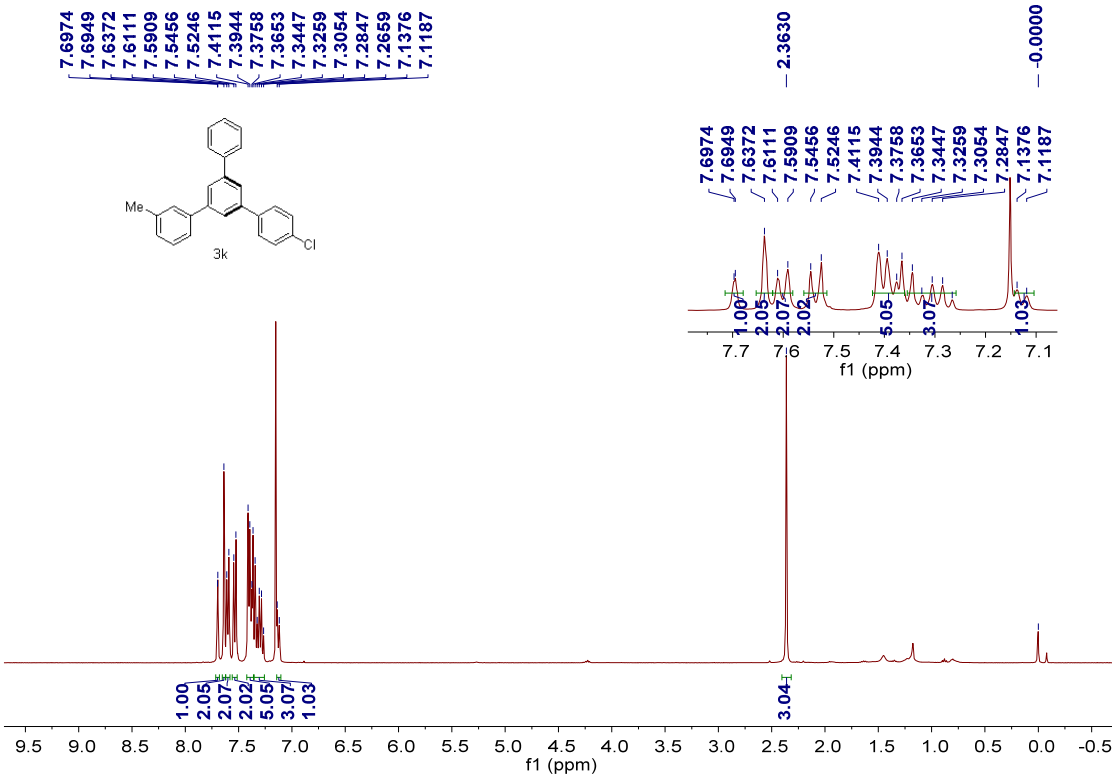
142.42
141.34
141.27
138.59
128.97
128.90
128.43
128.28
127.65
127.50
125.33
125.23
124.60
77.48
77.16
76.84
21.71
Me
3j
128.97
128.90
128.43
128.28
127.65
127.50
125.33
125.23
124.60
131 130 129 128 127 126 125 124 123 122
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3k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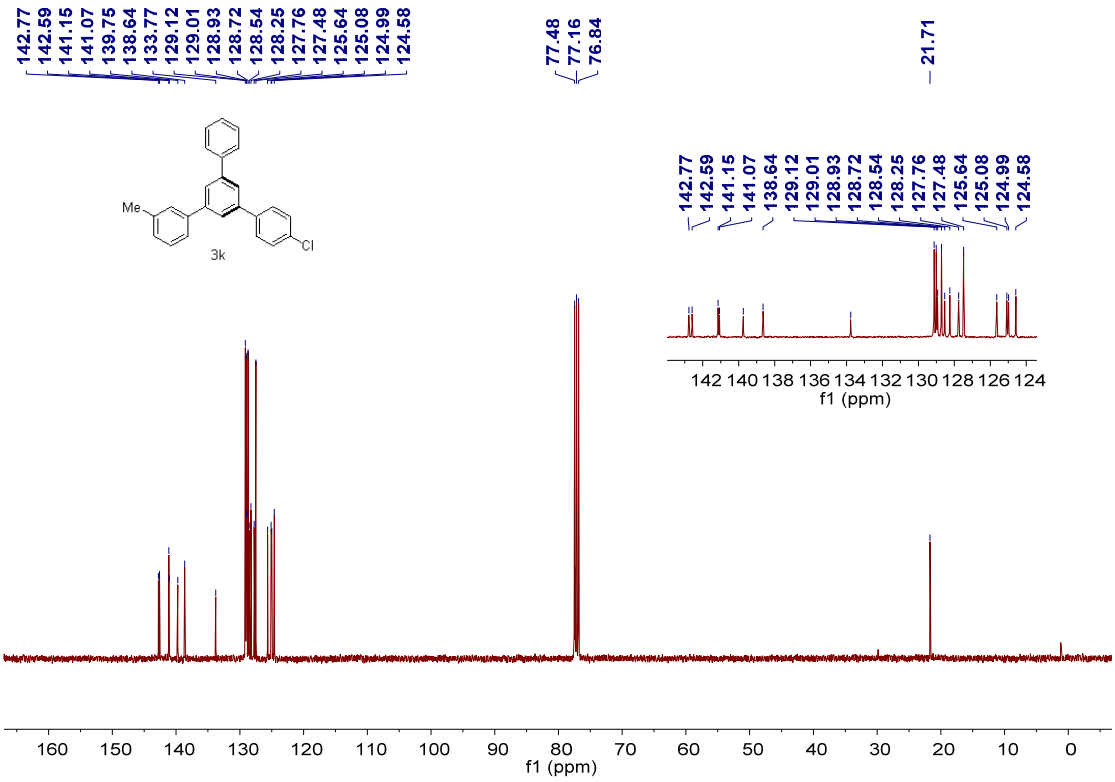
7.5456
7.5246
7.4115
7.3944
7.3758
7.3653
7.3447
7.3259
7.3054
7.2847
7.2659
7.1376
7.1187
2.3630
0.0000
Me
Cl
3k
1.00
2.05
2.07
2.02
5.05
3.07
1.03
3.04
7.7 7.6 7.5 7.4 7.3 7.2 7.1
f1 (ppm)
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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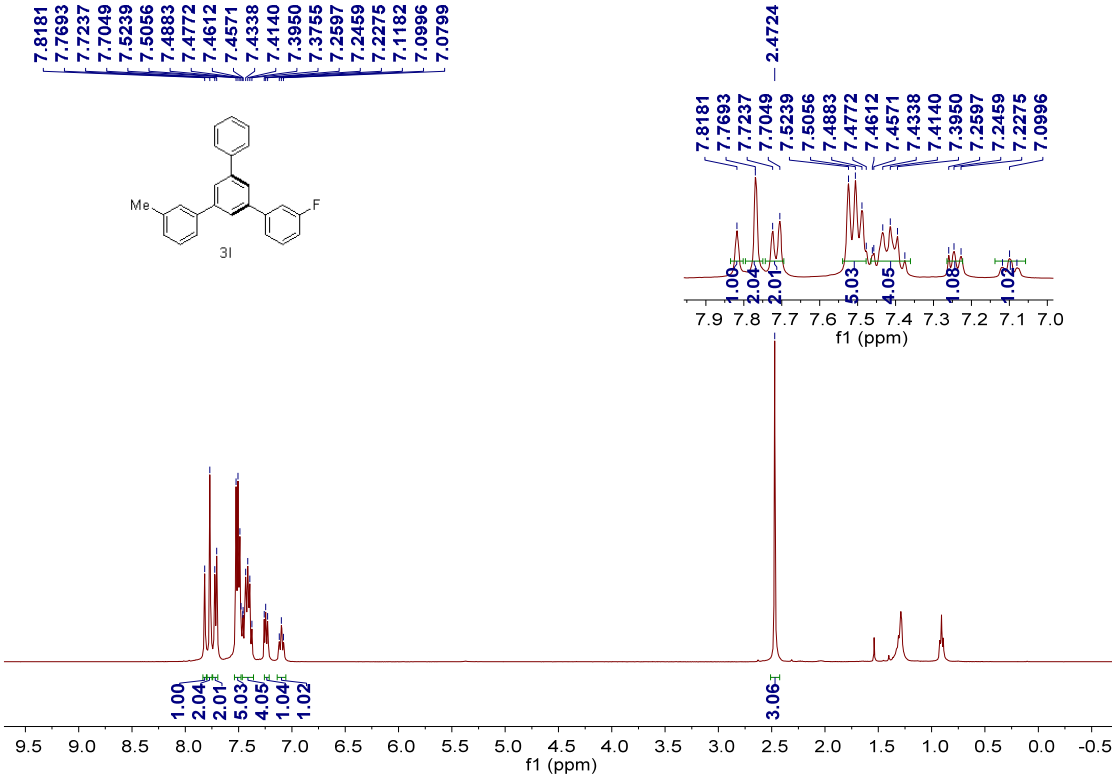
138.64
133.77
129.12
129.01
128.93
128.72
128.54
128.25
127.76
127.48
125.64
125.08
124.99
124.58
77.48
77.16
76.84
21.71
Me
Cl
3k
f1 (ppm)

3l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7.7237
7.7049
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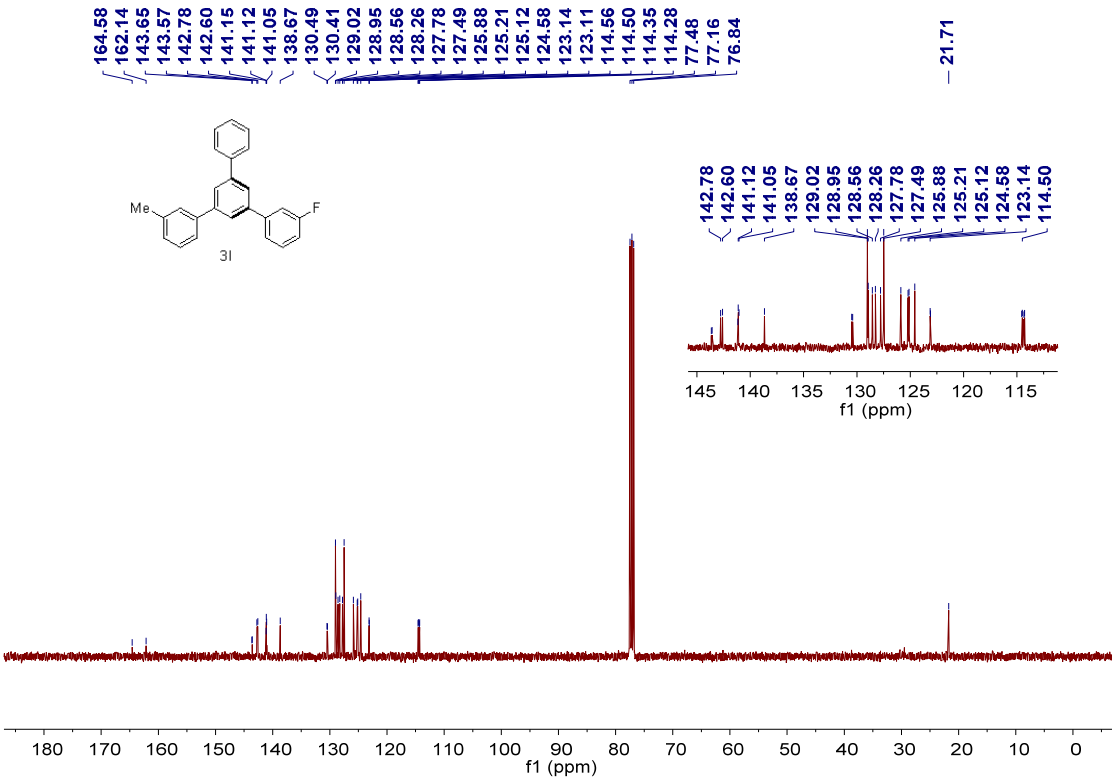
7.5056
7.4883
7.4772
7.4612
7.4571
7.4338
7.4140
7.3950
7.3755
7.2597
7.2459
7.2275
7.1182
7.0996
7.0799
2.4724
Me
F
3l
1.00
2.04
2.01
5.03
4.05
1.04
1.02
3.06
f1 (ppm)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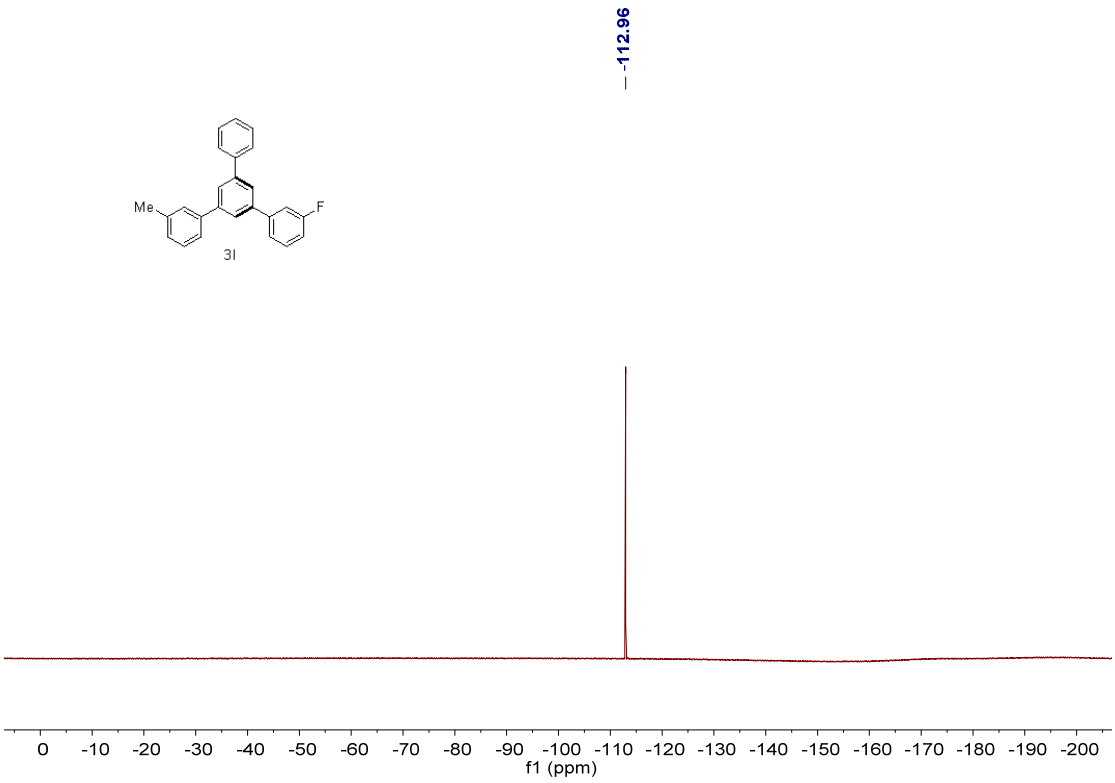
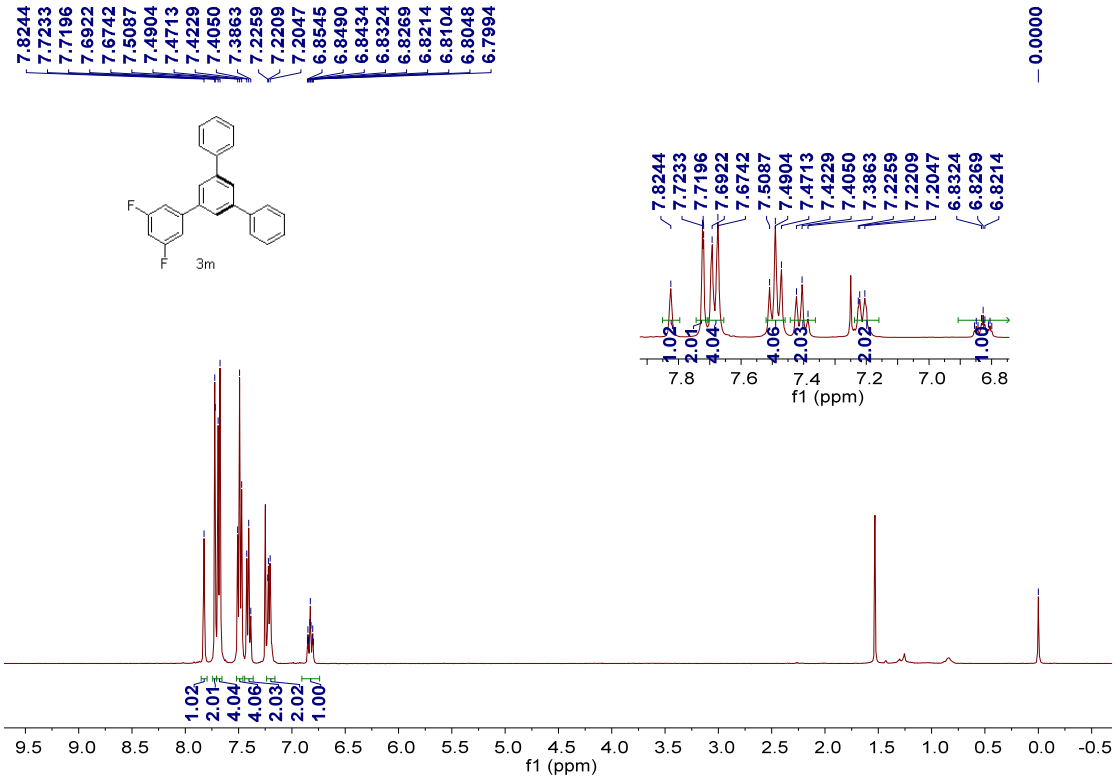
141.12
141.05
138.67
130.49
130.41
129.02
128.95
128.56
128.26
127.78
127.49
125.88
125.21
125.12
124.58
123.14
123.11
114.56
114.50
114.35
114.28
77.48
77.16
76.84
21.71
Me
F
3l
142.78
142.60
141.12
141.05
138.67
129.02
128.95
128.56
128.26
127.78
127.49
125.88
125.21
125.12
124.58
123.14
114.50
145 140 135 130 125 120 115
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-112.96
Me
F
3l
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

3m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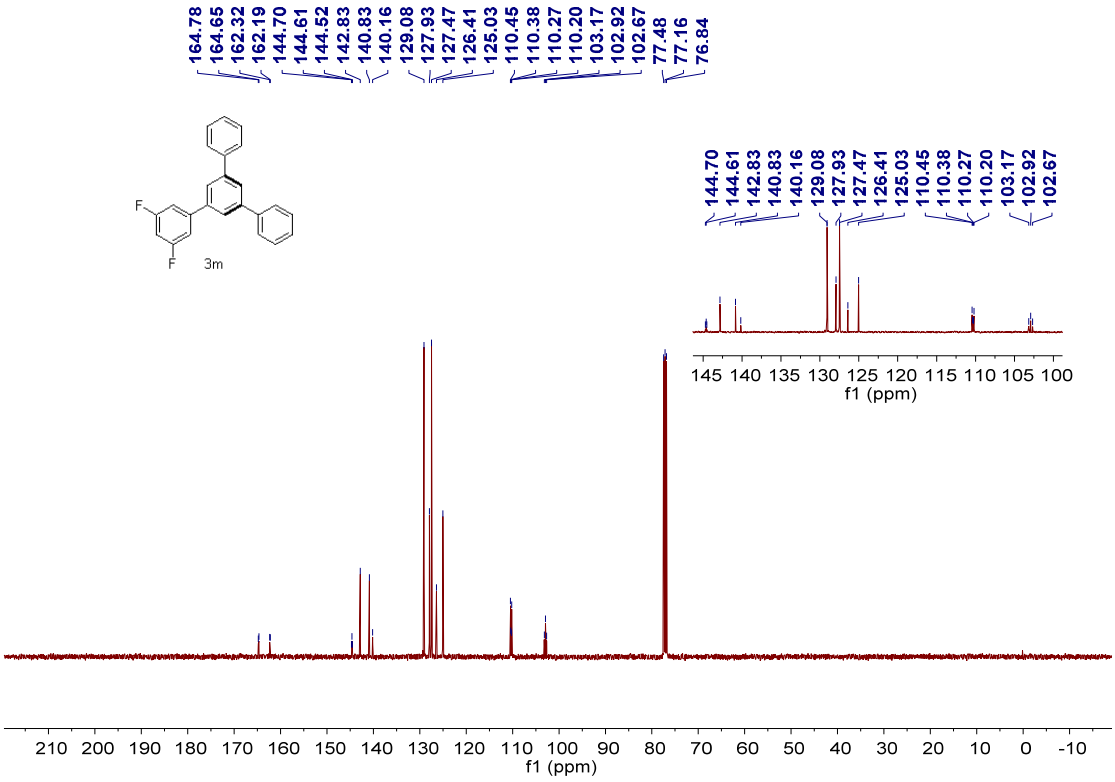
7.6922
7.6742
7.5087
7.4904
7.4713
7.4229
7.4050
7.3863
7.2259
7.2209
7.2047
6.8545
6.8490
6.8434
6.8324
6.8269
6.8214
6.8104
6.8048
6.7994
0.0000
F
F
3m
1.02
2.01
4.04
4.06
2.03
2.02
1.00
f1 (ppm)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162.19
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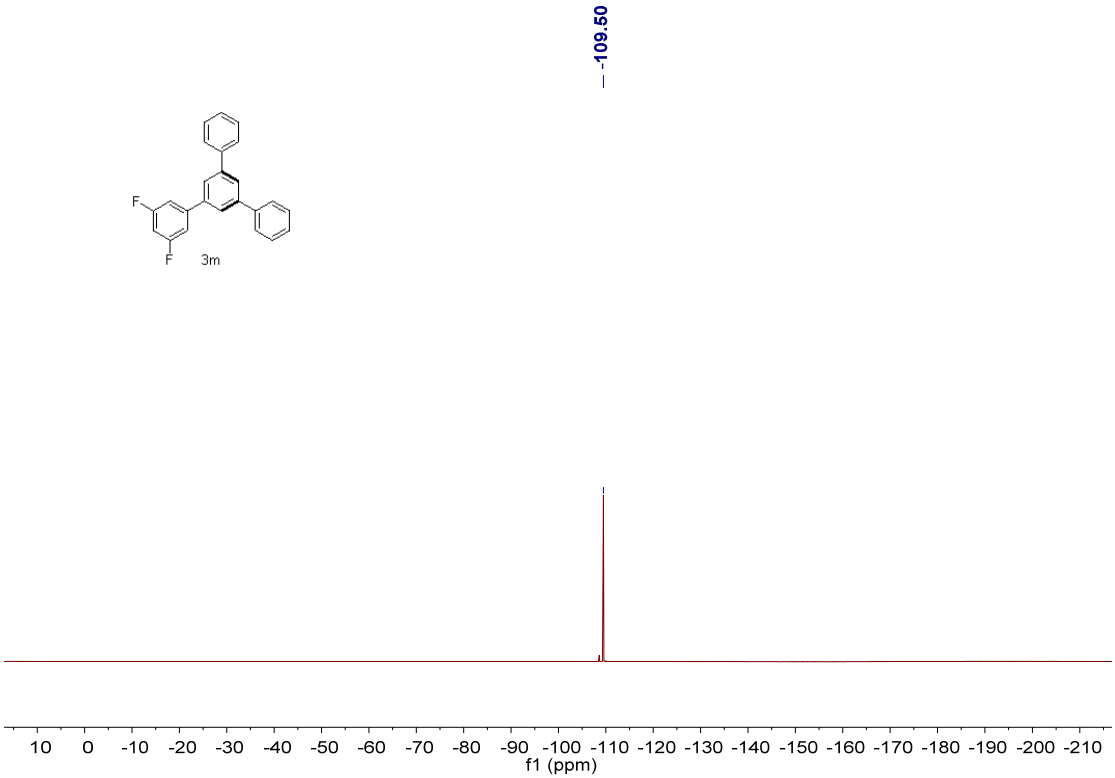
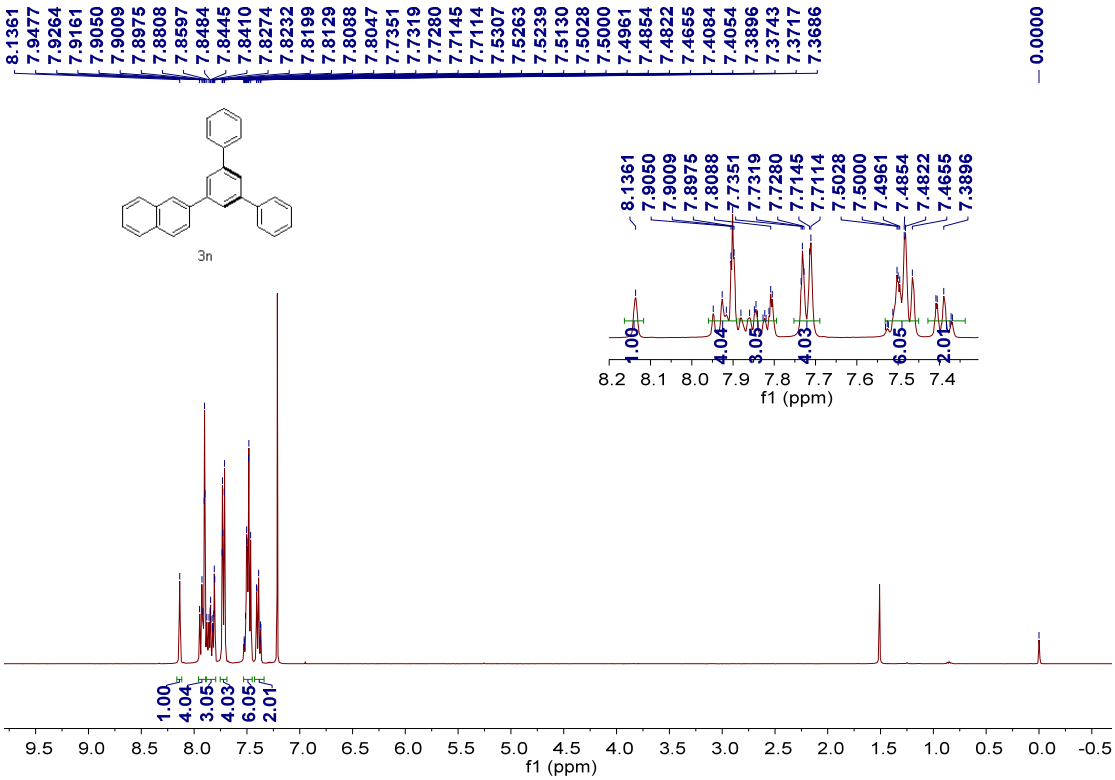
144.61
144.52
142.83
140.83
140.16
129.08
127.93
127.47
126.41
125.03
110.45
110.38
110.27
110.20
103.17
102.92
102.67
77.48
77.16
76.84
F
F
3m
f1 (ppm)


-109.50
F
F
3m
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

3n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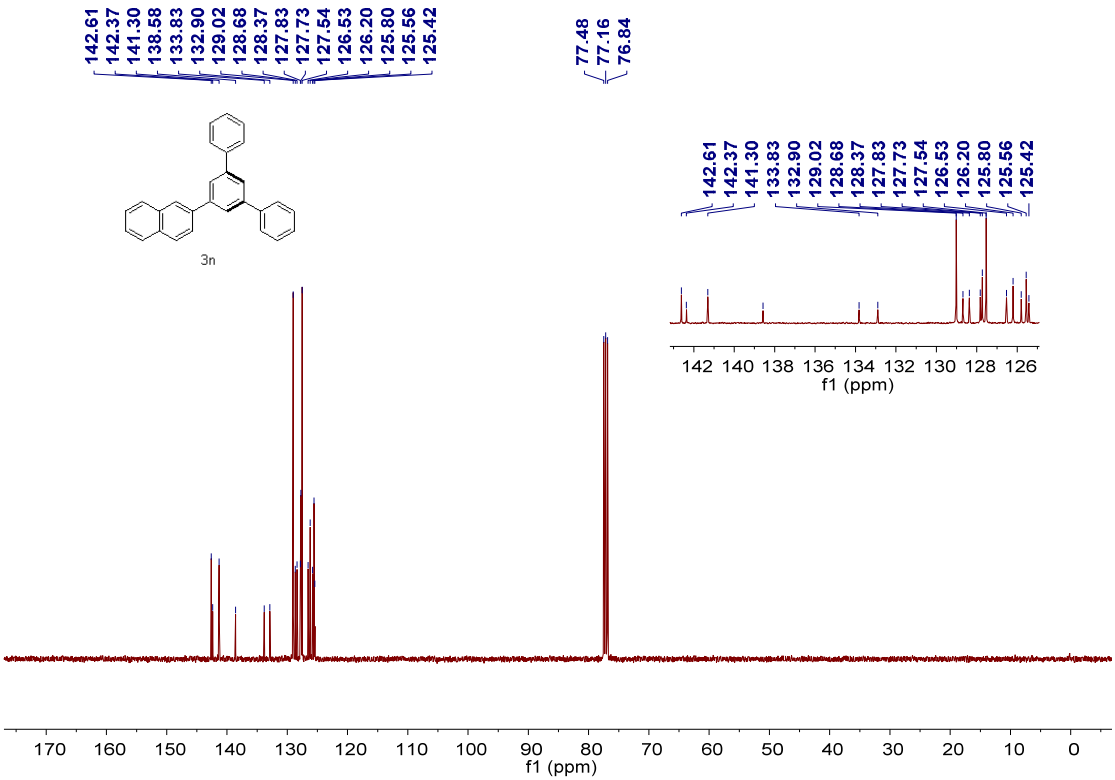
7.9050
7.9009
7.8975
7.8808
7.8597
7.8484
7.8445
7.8410
7.8274
7.8232
7.8199
7.8129
7.8088
7.8047
7.7351
7.7319
7.7280
7.7145
7.7114
7.5307
7.5263
7.5239
7.5130
7.5028
7.5000
7.4961
7.4854
7.4822
7.4655
7.4084
7.4054
7.3896
7.3743
7.3717
7.3686
0.0000
3n
1.00
4.04
3.05
4.03
6.05
2.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)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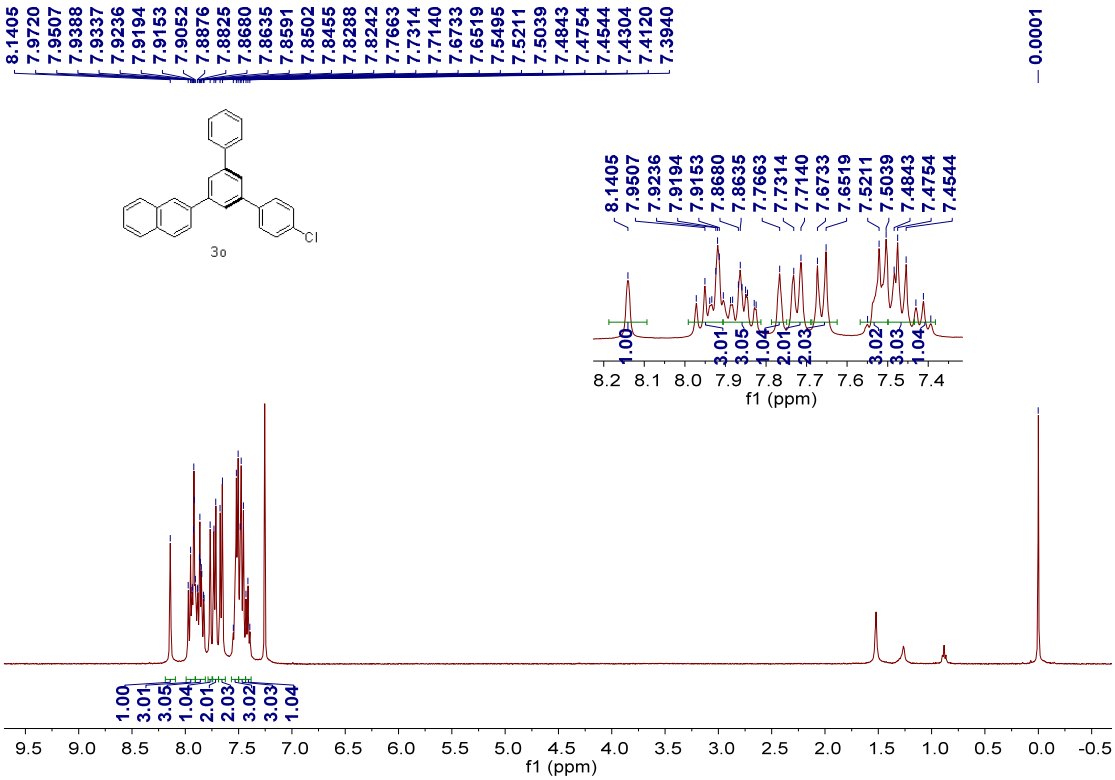
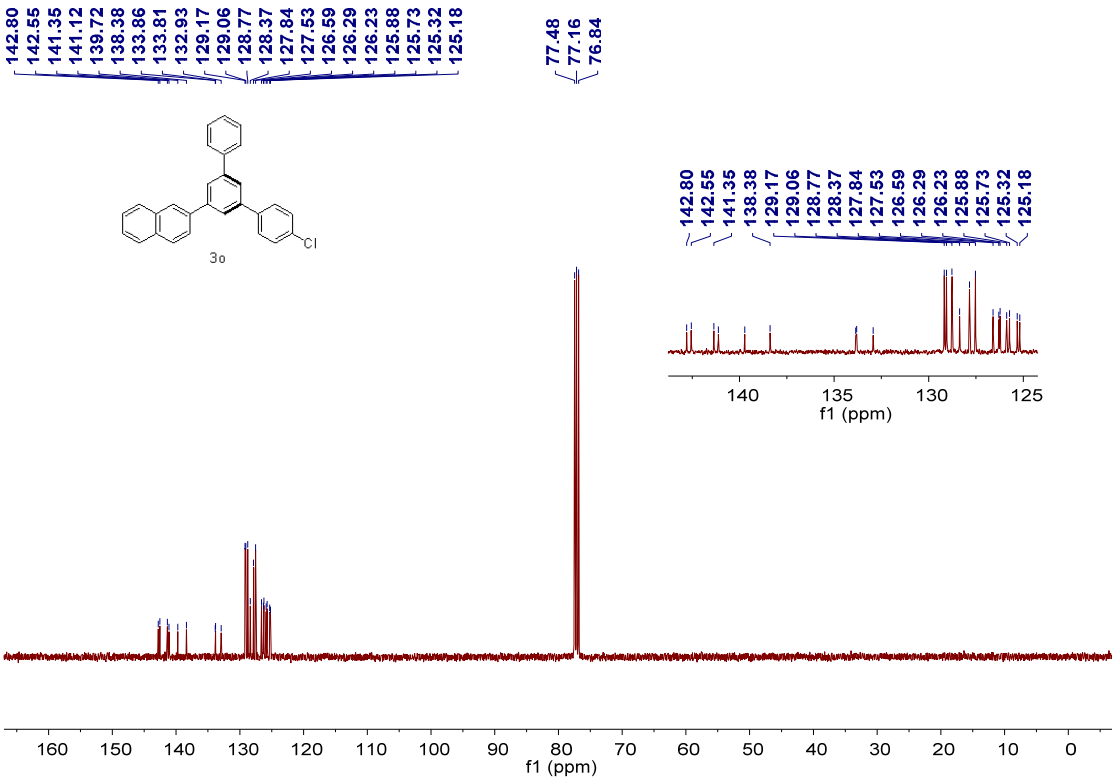
142.37
141.30
138.58
133.83
132.90
129.02
128.68
128.37
127.83
127.73
127.54
126.53
126.20
125.80
125.56
125.42
77.48
77.16
76.84
3n
142.61
142.37
141.30
133.83
132.90
129.02
128.68
128.37
127.83
127.73
127.54
126.53
126.20
125.80
125.56
125.42
142 140 138 136 134 132 130 128 126
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3o


8.1405
7.9720
7.9507
7.9388
7.9337
7.9236
7.9194
7.9153
7.9052
7.8876
7.8825
7.8680
7.8635
7.8591
7.8502
7.8455
7.8288
7.8242
7.7663
7.7314
7.7140
7.6733
7.6519
7.5495
7.5211
7.5039
7.4843
7.4754
7.4544
7.4304
7.4120
7.3940
0.0001
3o
Cl
8.1405
7.9507
7.9236
7.9194
7.9153
7.8680
7.8635
7.7663
7.7314
7.7140
7.6733
7.6519
7.5211
7.5039
7.4843
7.4754
7.4544
1.00
3.01
3.05
1.04
2.01
2.03
3.02
3.03
1.04
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4
f1 (ppm)
1.00
3.01
3.05
1.04
2.01
2.03
3.02
3.03
1.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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142.55
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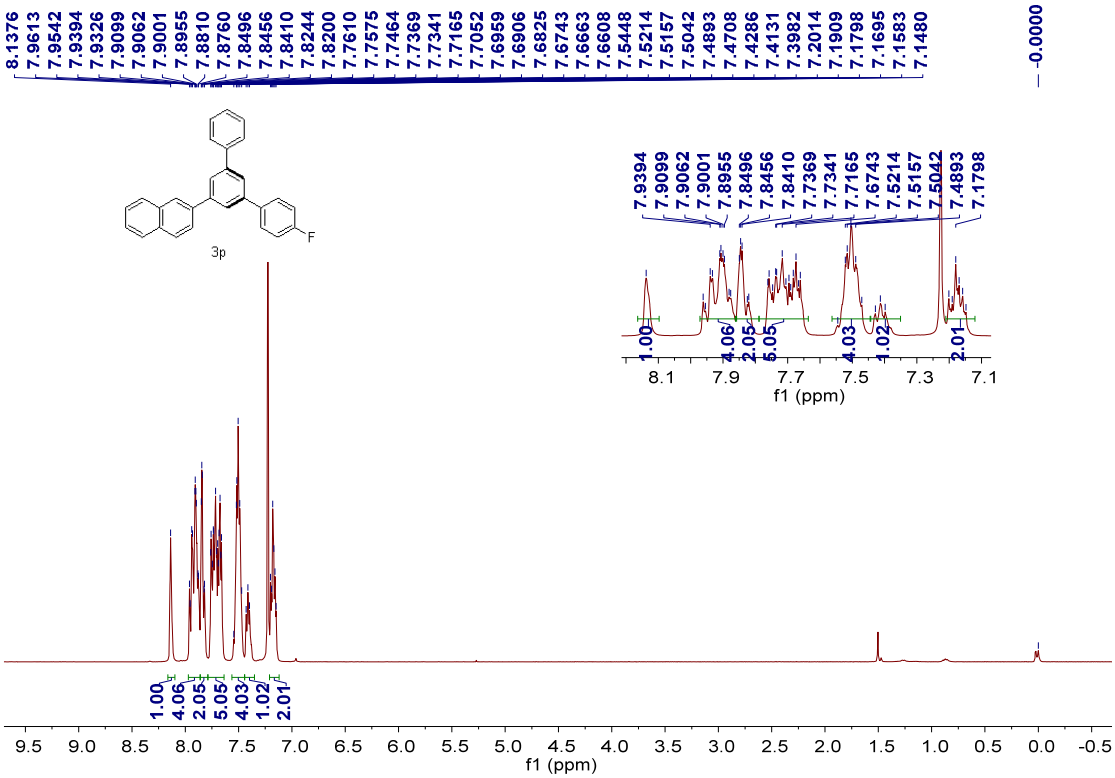
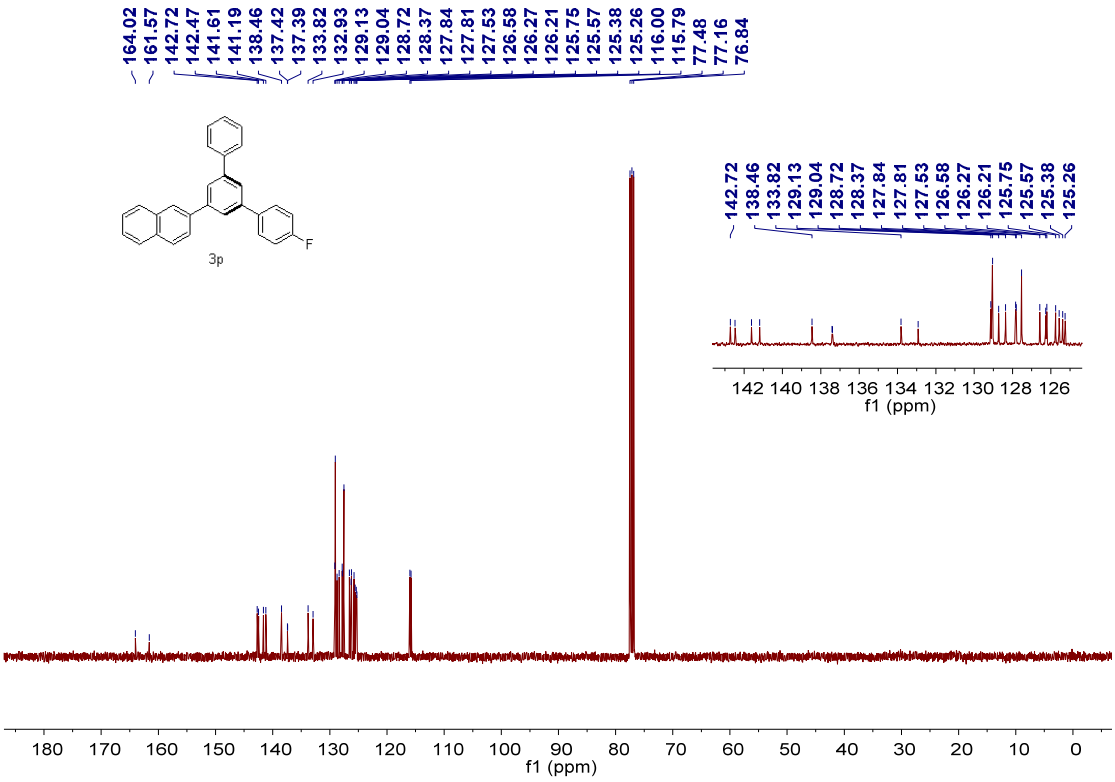
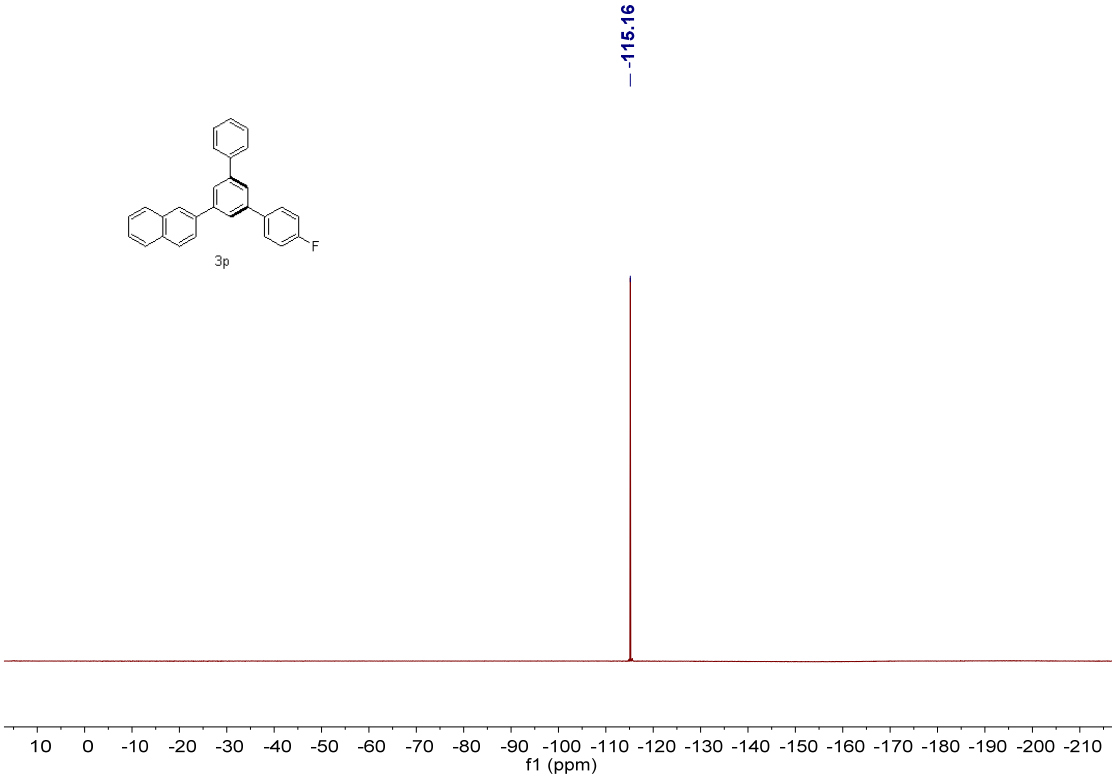
141.12
139.72
138.38
133.86
133.81
132.93
129.17
129.06
128.77
128.37
127.84
127.53
126.59
126.29
126.23
125.88
125.73
125.32
125.18
77.48
77.16
76.84
Cl
3o
f1 (ppm)

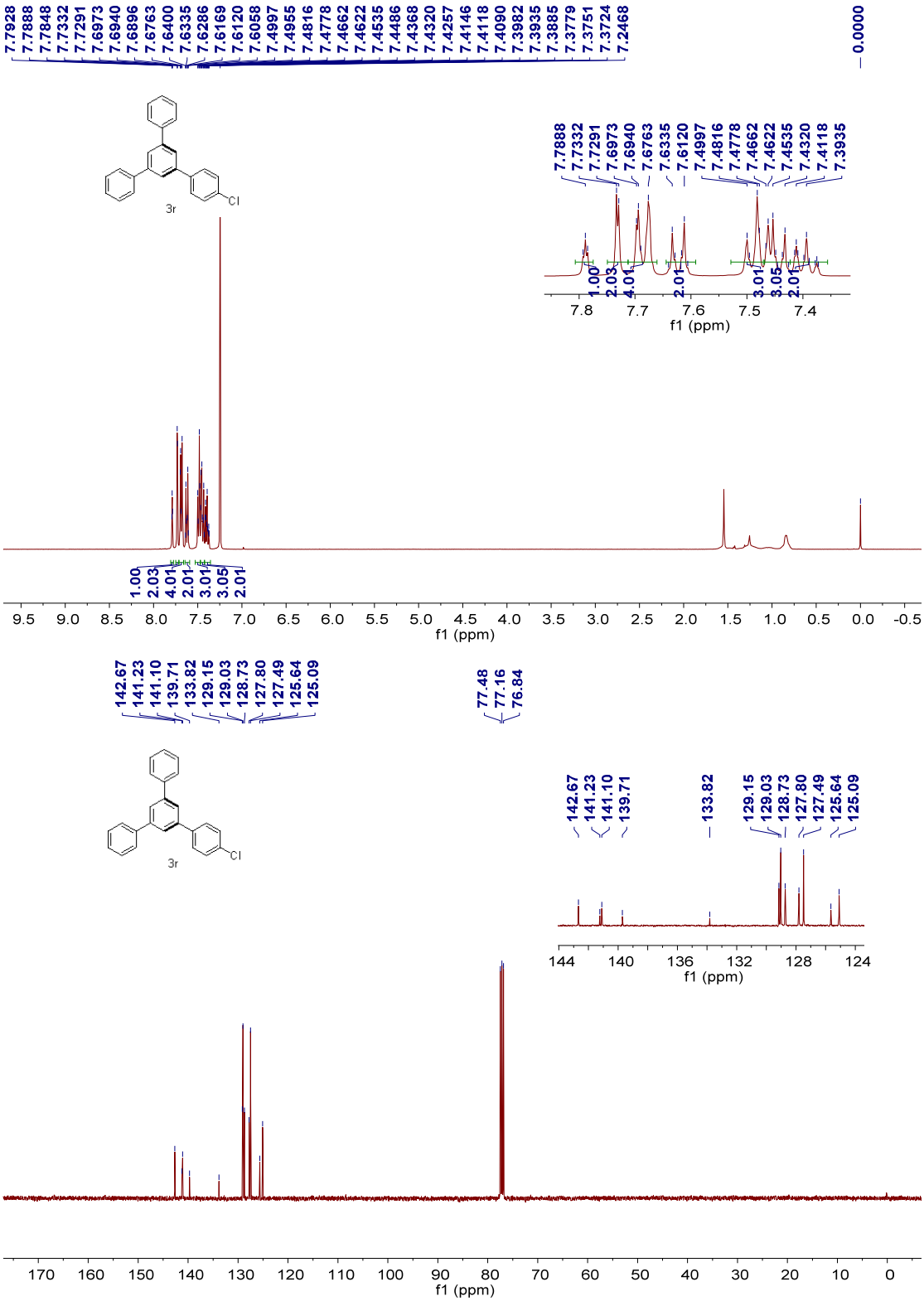
3p


8.1376
7.9613
7.9542
7.9394
7.9326
7.9099
7.9062
7.9001
7.8955
7.8810
7.8760
7.8496
7.8456
7.8410
7.8244
7.8200
7.7610
7.7575
7.7464
7.7369
7.7341
7.7165
7.7052
7.6959
7.6906
7.6825
7.6743
7.6663
7.6608
7.5448
7.5214
7.5157
7.5042
7.4893
7.4708
7.4286
7.4131
7.3982
7.2014
7.1909
7.1798
7.1695
7.1583
7.1480
-0.0000
F
3p
1.00
4.06
2.05
5.05
4.03
1.02
2.01
f1 (ppm)

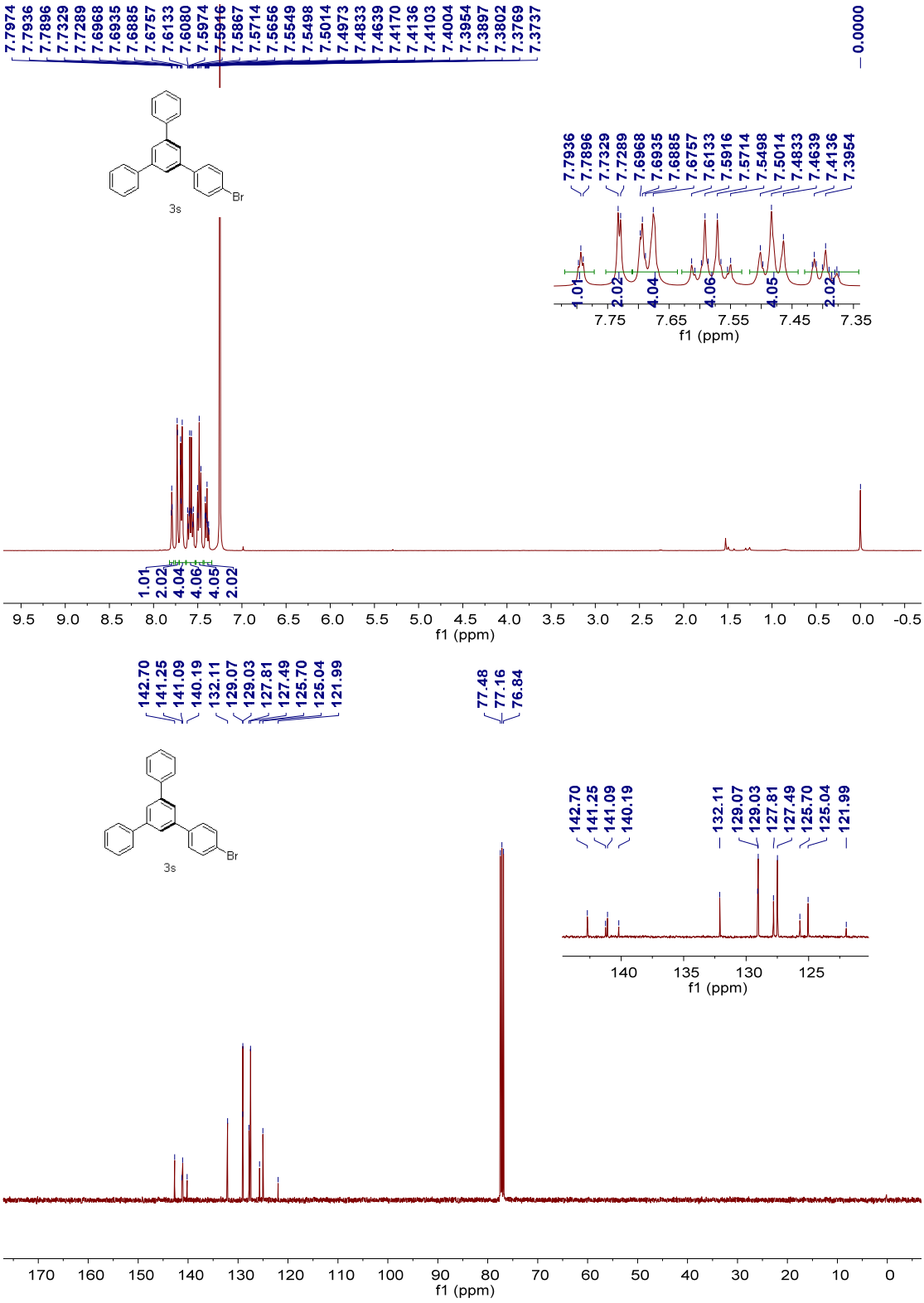

164.02
161.57
142.72
142.47
141.61
141.19
138.46
137.42
137.39
133.82
132.93
129.13
129.04
128.72
128.37
127.84
127.81
127.53
126.58
126.27
126.21
125.75
125.57
125.38
125.26
116.00
115.79
77.48
77.16
76.84
F
3p
142.72
138.46
133.82
129.13
129.04
128.72
128.37
127.84
127.81
127.53
126.58
126.27
126.21
125.75
125.57
125.38
125.26
142 140 138 136 134 132 130 128 126
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-115.16
F
3p
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

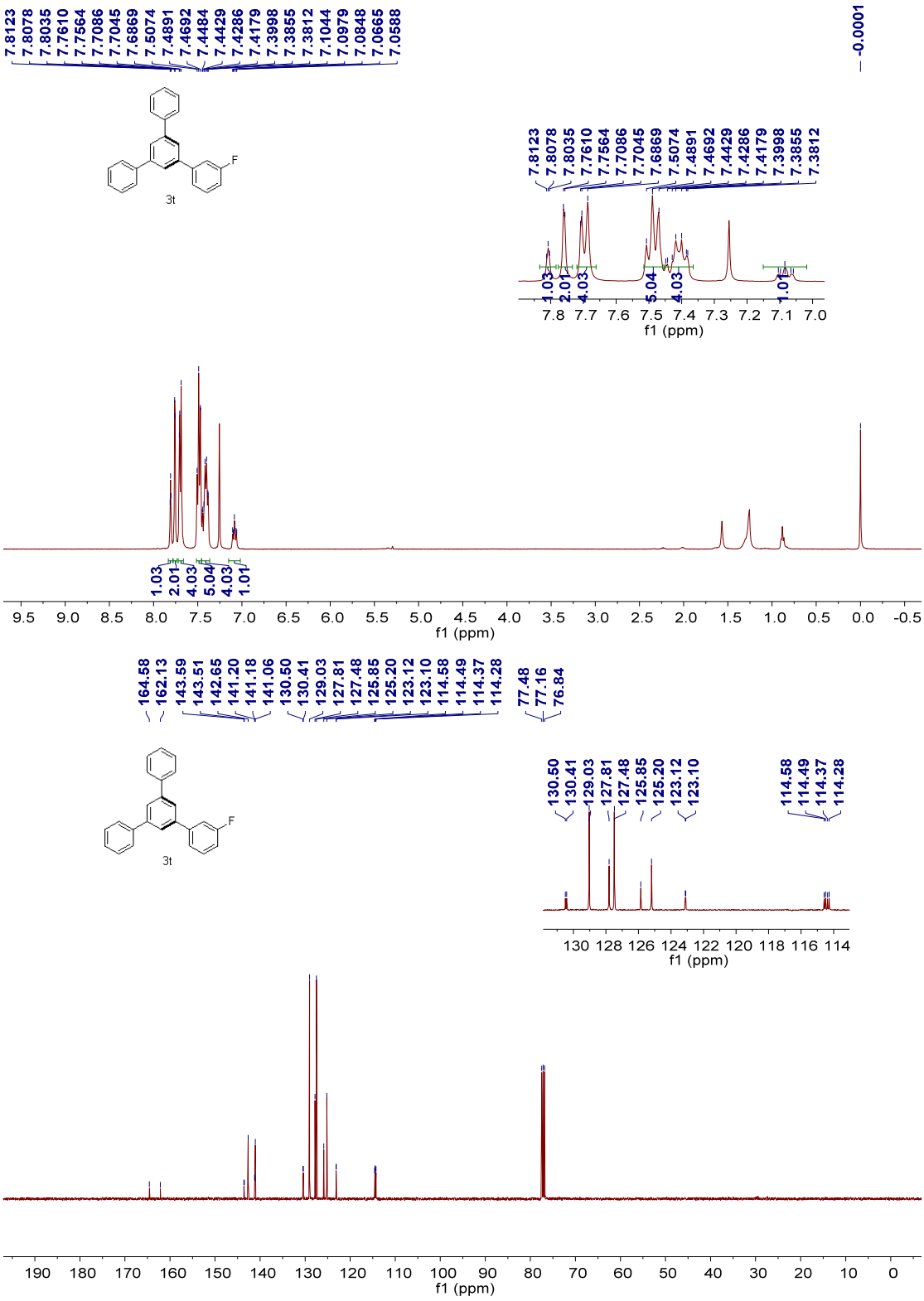
3r

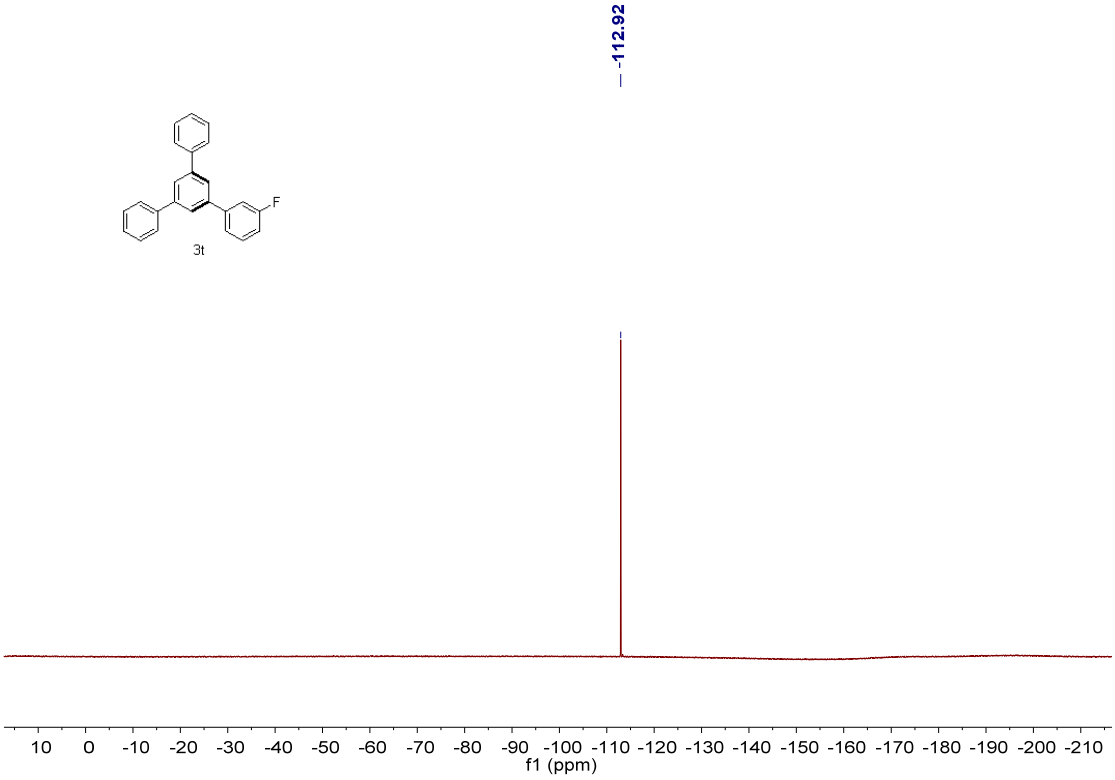

7.7928 7.7888 7.7848 7.7332 7.7291 7.6973 7.6940 7.6896 7.6763 7.6400 7.6335 7.6286 7.6169 7.6120 7.6058 7.4997 7.4955 7.4816 7.4778 7.4662 7.4622 7.4535 7.4486 7.4368 7.4320 7.4257 7.4146 7.4118 7.4090 7.3982 7.3935 7.3885 7.3779 7.3751 7.3724 7.2468
0.0000
3r
Cl
1.00 2.03 4.01 2.01 3.01 3.05 2.01
f1 (ppm)
142.67 141.23 141.10 139.71 133.82 129.15 129.03 128.73 127.80 127.49 125.64 125.09
77.48 77.16 76.84
f1 (ppm)

3s

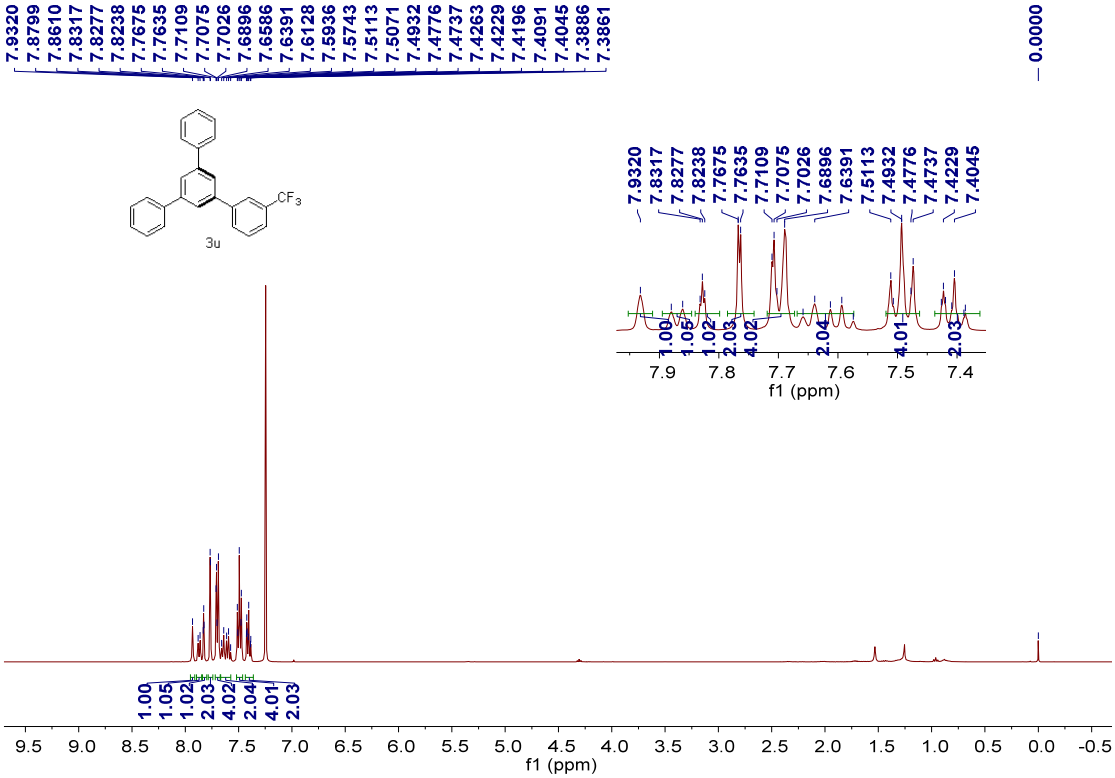

7.7974
7.7936
7.7896
7.7329
7.7289
7.6968
7.6935
7.6885
7.6757
7.6133
7.6080
7.5974
7.5916
7.5867
7.5714
7.5656
7.5549
7.5498
7.5014
7.4973
7.4833
7.4639
7.4170
7.4136
7.4103
7.4004
7.3954
7.3897
7.3802
7.3769
7.3737
0.0000
Br
3s
1.01
2.02
4.04
4.06
4.05
2.02
f1 (ppm)
142.70
141.25
141.09
140.19
132.11
129.07
129.03
127.81
127.49
125.70
125.04
121.99
77.48
77.16
76.84
f1 (ppm)

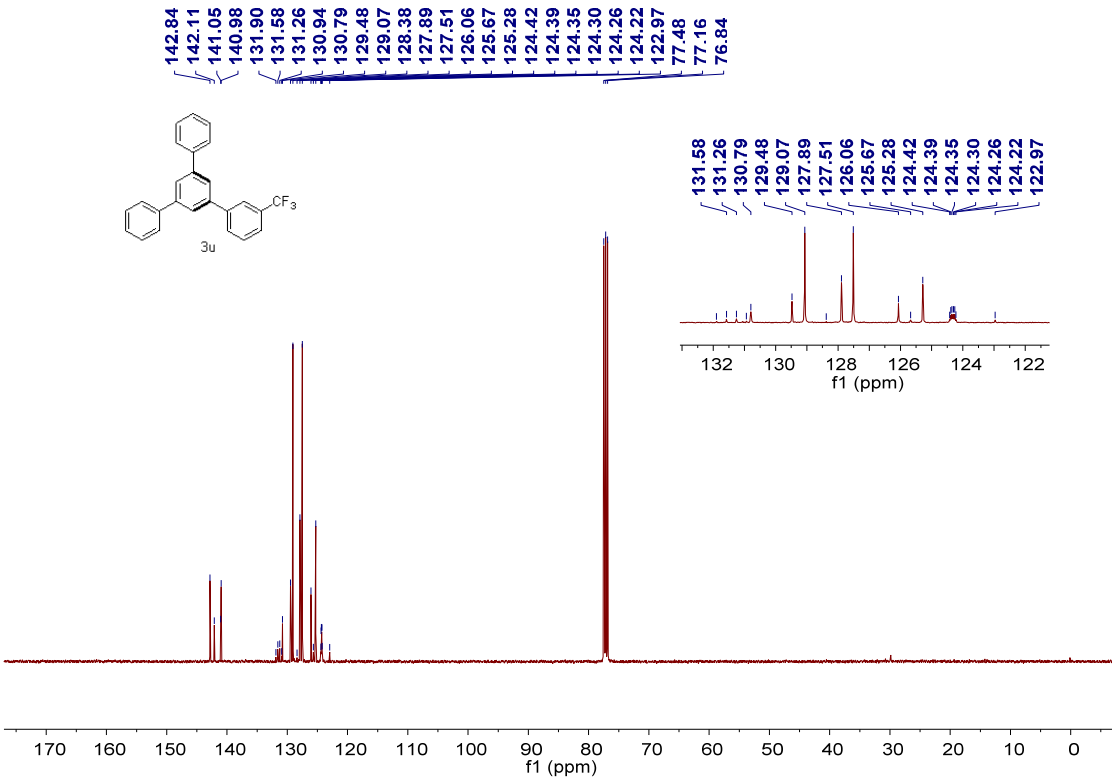
3t

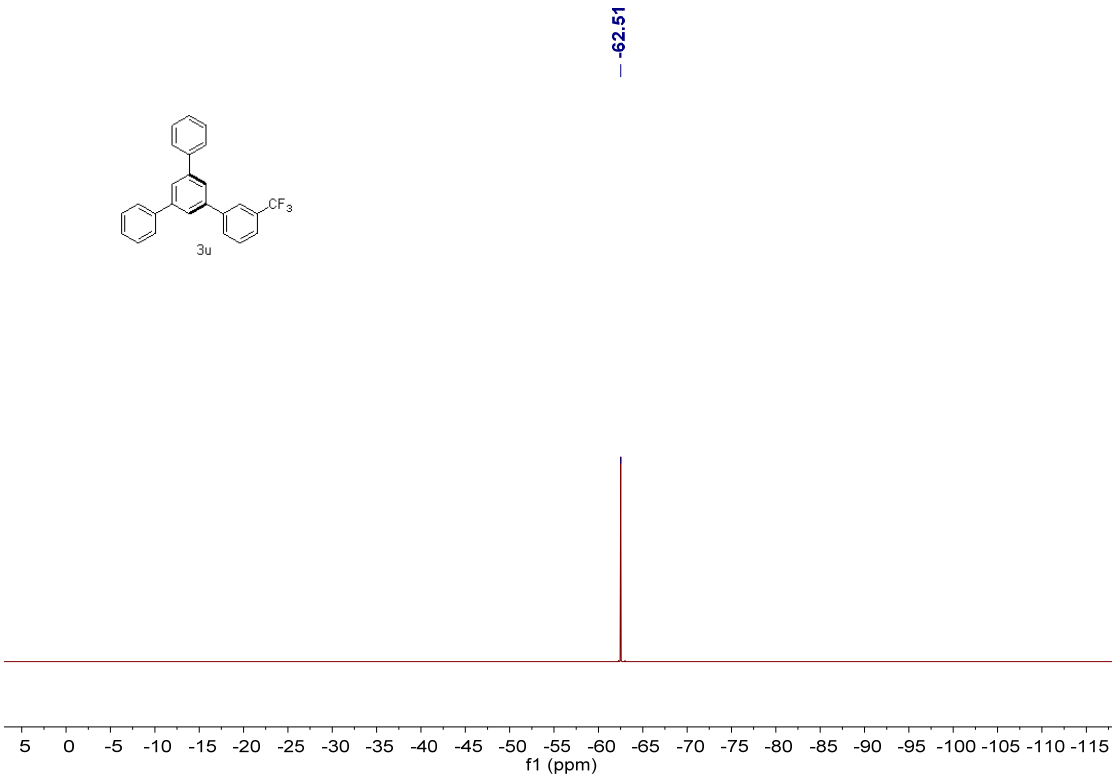

7.8123
7.8078
7.8035
7.7610
7.7564
7.7086
7.7045
7.6869
7.5074
7.4891
7.4692
7.4484
7.4429
7.4286
7.4179
7.3998
7.3855
7.3812
7.1044
7.0979
7.0848
7.0665
7.0588
-0.0001
F
3t
1.03
2.01
4.03
5.04
4.03
1.01
f1 (ppm)
164.58
162.13
143.59
143.51
142.65
141.20
141.18
141.06
130.50
130.41
129.03
127.81
127.48
125.85
125.20
123.12
123.10
114.58
114.49
114.37
114.28
77.48
77.16
76.84
f1 (ppm)


-112.92
F
3t
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

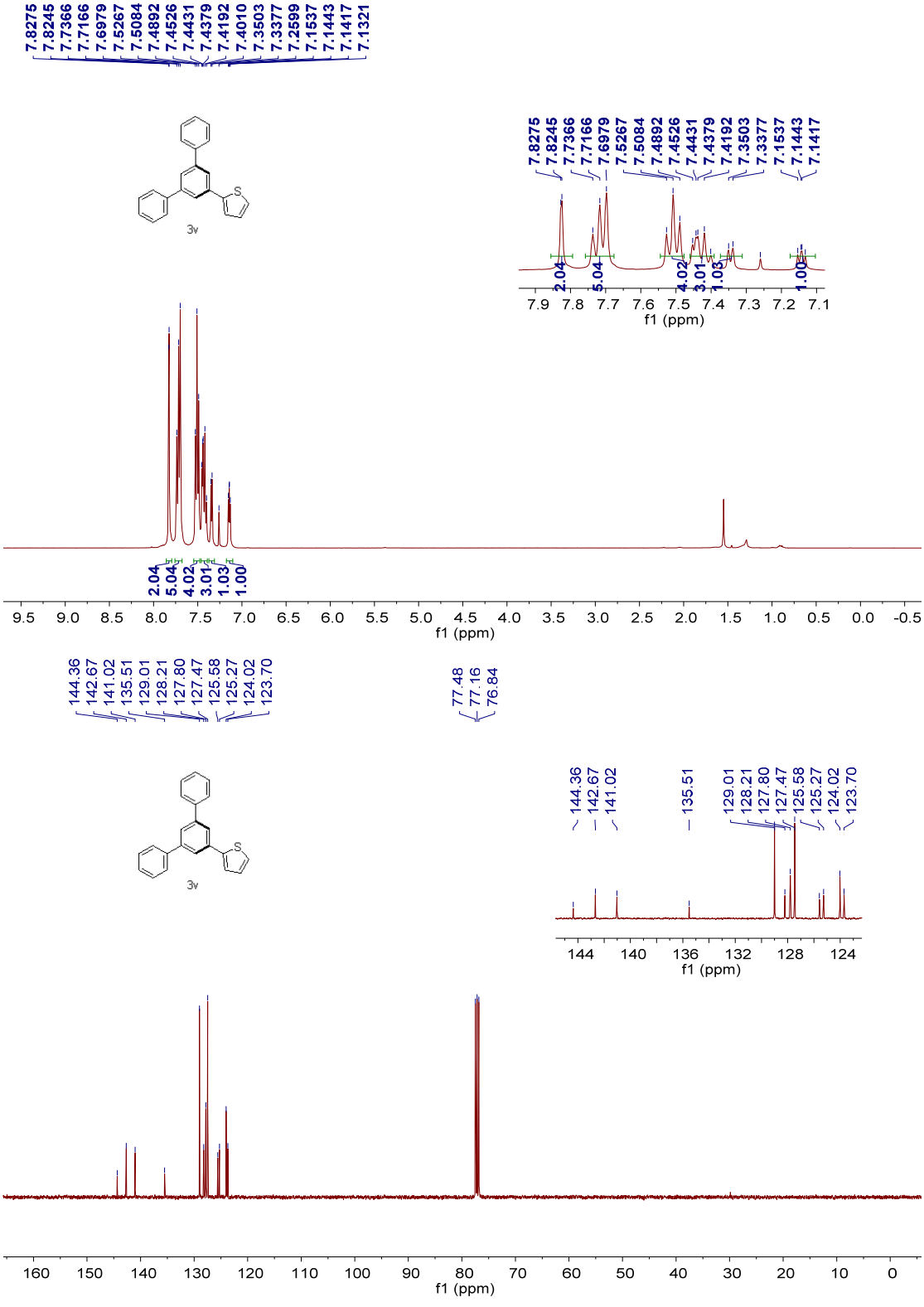
3u


7.9320
7.8799
7.8610
7.8317
7.8277
7.8238
7.7675
7.7635
7.7109
7.7075
7.7026
7.6896
7.6586
7.6391
7.6128
7.5936
7.5743
7.5113
7.5071
7.4932
7.4776
7.4737
7.4263
7.4229
7.4196
7.4091
7.4045
7.3886
7.3861
0.0000
CF3
3u
1.00 1.05 1.02 2.03 4.02 2.04 4.01 2.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

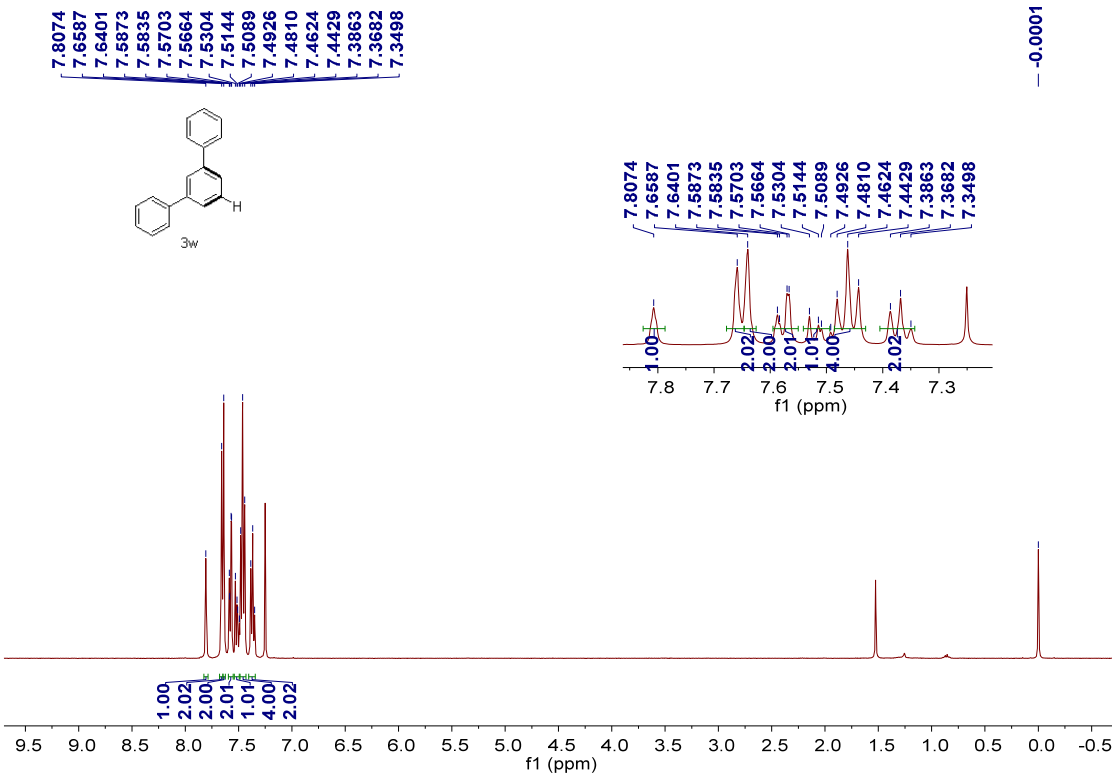

142.84
142.11
141.05
140.98
131.90
131.58
131.26
130.94
130.79
129.48
129.07
128.38
127.89
127.51
126.06
125.67
125.28
124.42
124.39
124.35
124.30
124.26
124.22
122.97
77.48
77.16
76.84
CF3
3u
f1 (ppm)

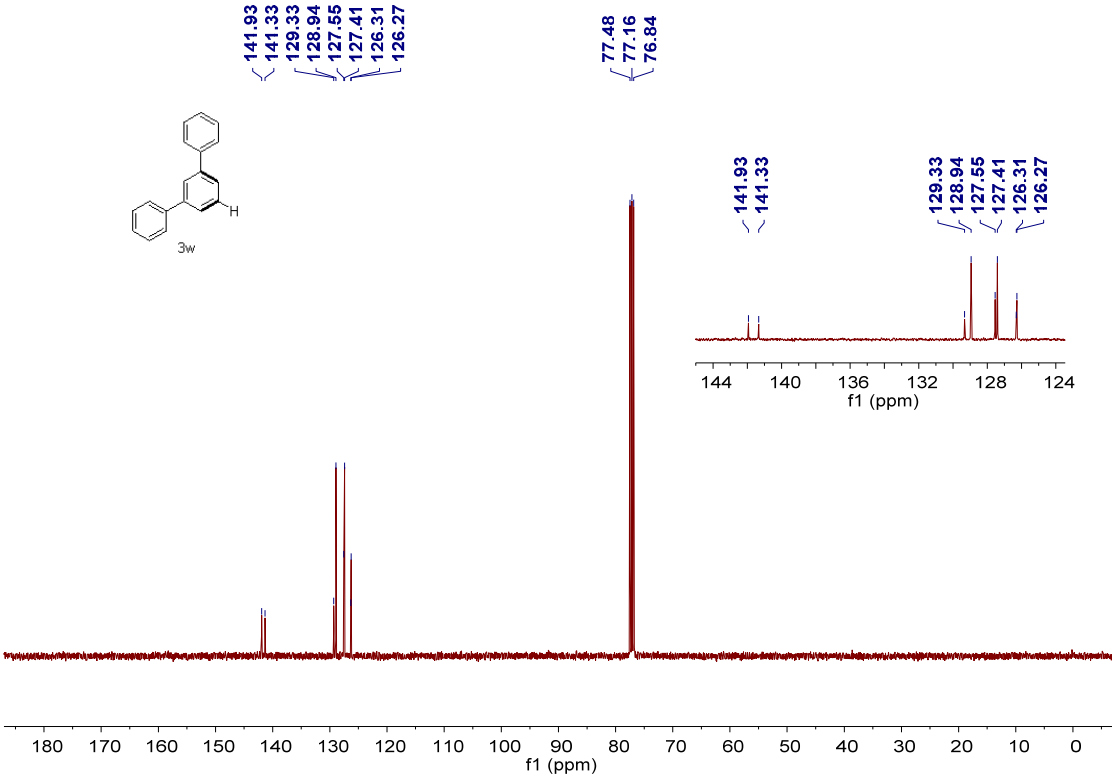

-62.51
CF3
3u
f1 (ppm)

3v

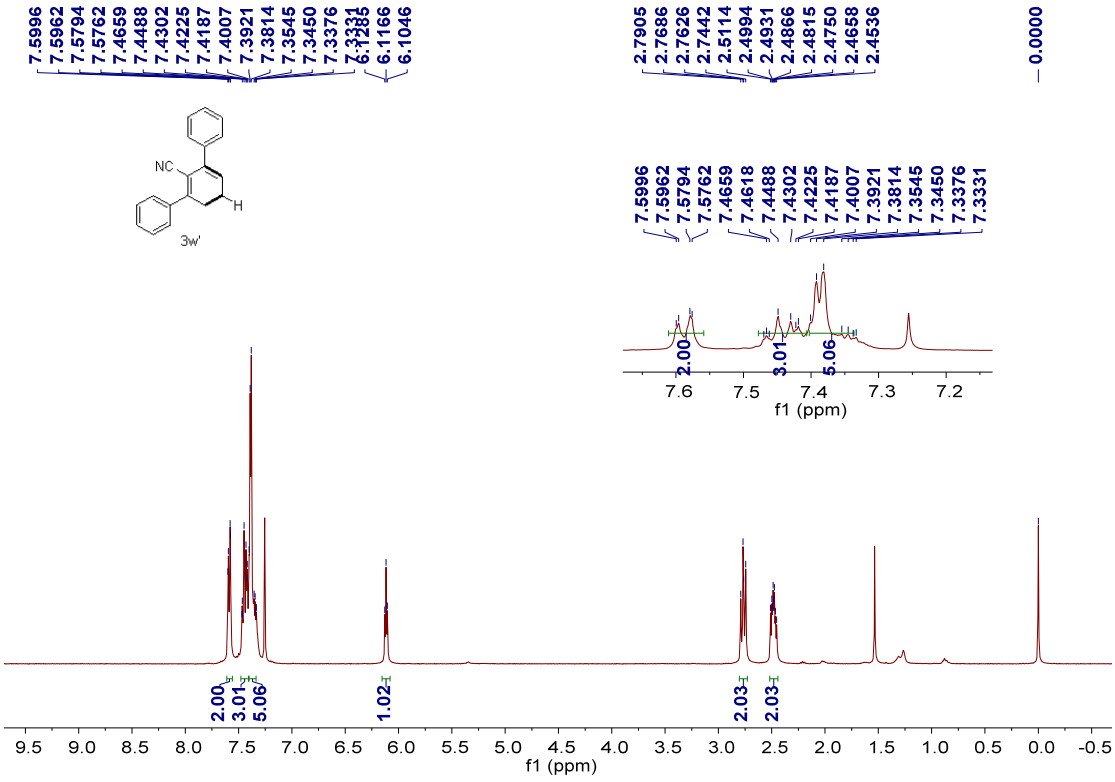

7.8275
7.8245
7.7366
7.7166
7.6979
7.5267
7.5084
7.4892
7.4526
7.4431
7.4379
7.4192
7.4010
7.3503
7.3377
7.2599
7.1537
7.1443
7.1417
7.1321
3v
2.04
5.04
4.02
3.01
1.03
1.00
f1 (ppm)
144.36
142.67
141.02
135.51
129.01
128.21
127.80
127.47
125.58
125.27
124.02
123.70
77.48
77.16
76.84
f1 (ppm)

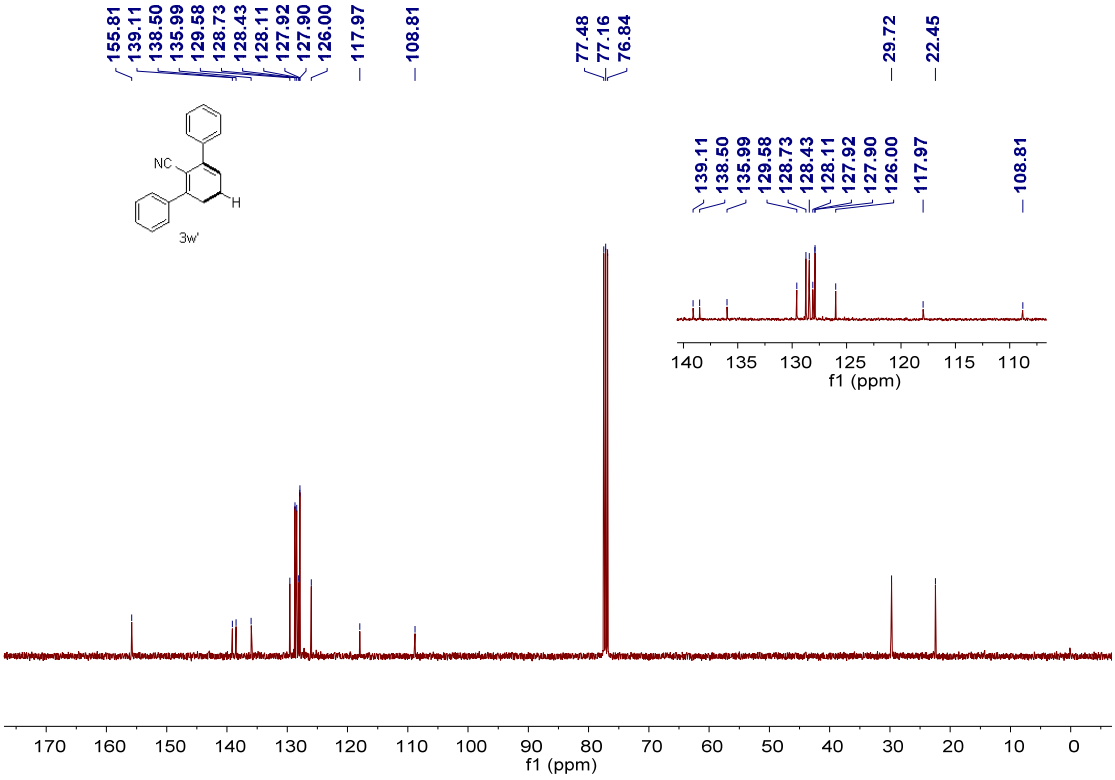
3w


7.8074
7.6587
7.6401
7.5873
7.5835
7.5703
7.5664
7.5304
7.5144
7.5089
7.4926
7.4810
7.4624
7.4429
7.3863
7.3682
7.3498
-0.0001
3w
1.00
2.02
2.00
2.01
1.01
4.00
2.02
f1 (ppm)

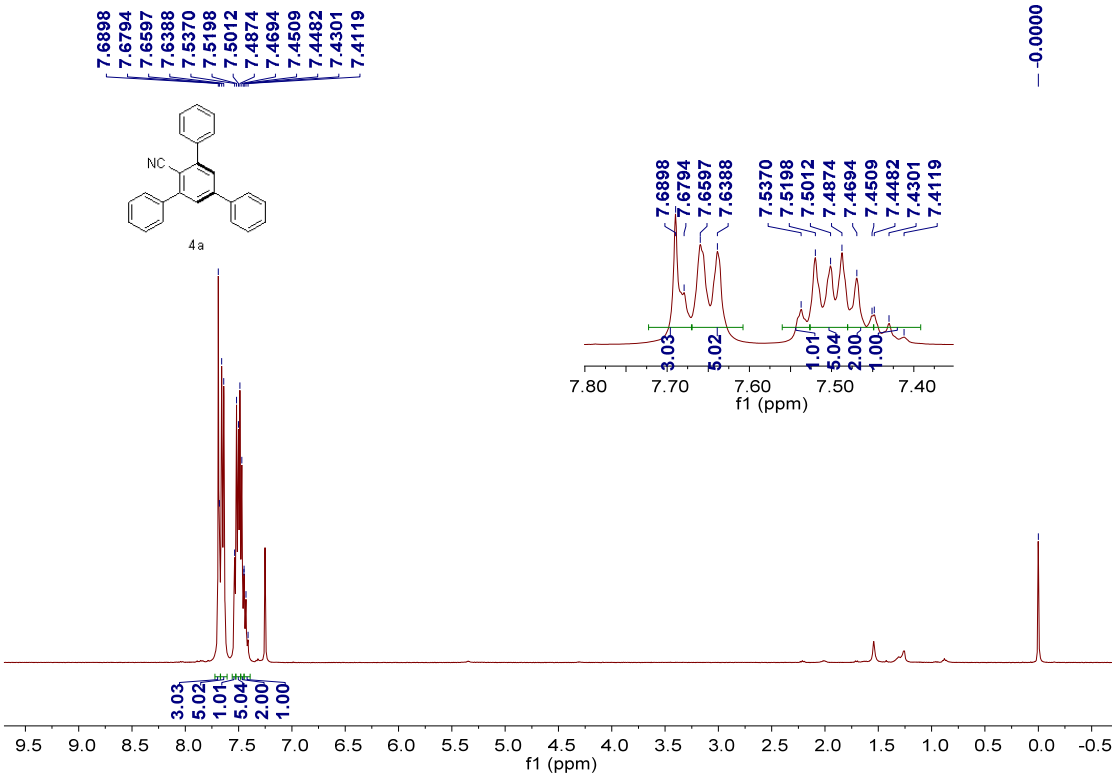

141.93
141.33
129.33
128.94
127.55
127.41
126.31
126.27
77.48
77.16
76.84
3w
f1 (ppm)

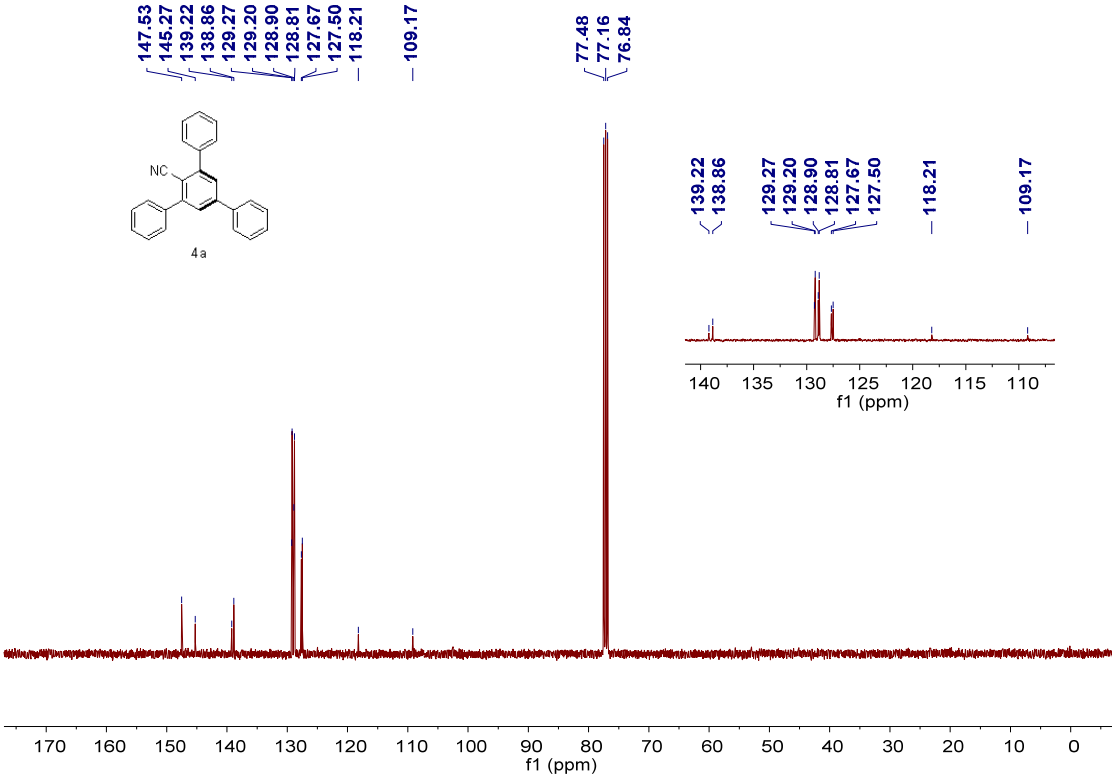
3w’


7.5996
7.5962
7.5794
7.5762
7.4659
7.4488
7.4302
7.4225
7.4187
7.4007
7.3921
7.3814
7.3545
7.3450
7.3376
7.3331
6.1166
6.1046
2.7905
2.7686
2.7626
2.7442
2.5114
2.4994
2.4931
2.4866
2.4815
2.4750
2.4658
2.4536
0.0000
NC
H
3w'
2.00
3.01
5.06
f1 (ppm)
1.02
2.03
2.03

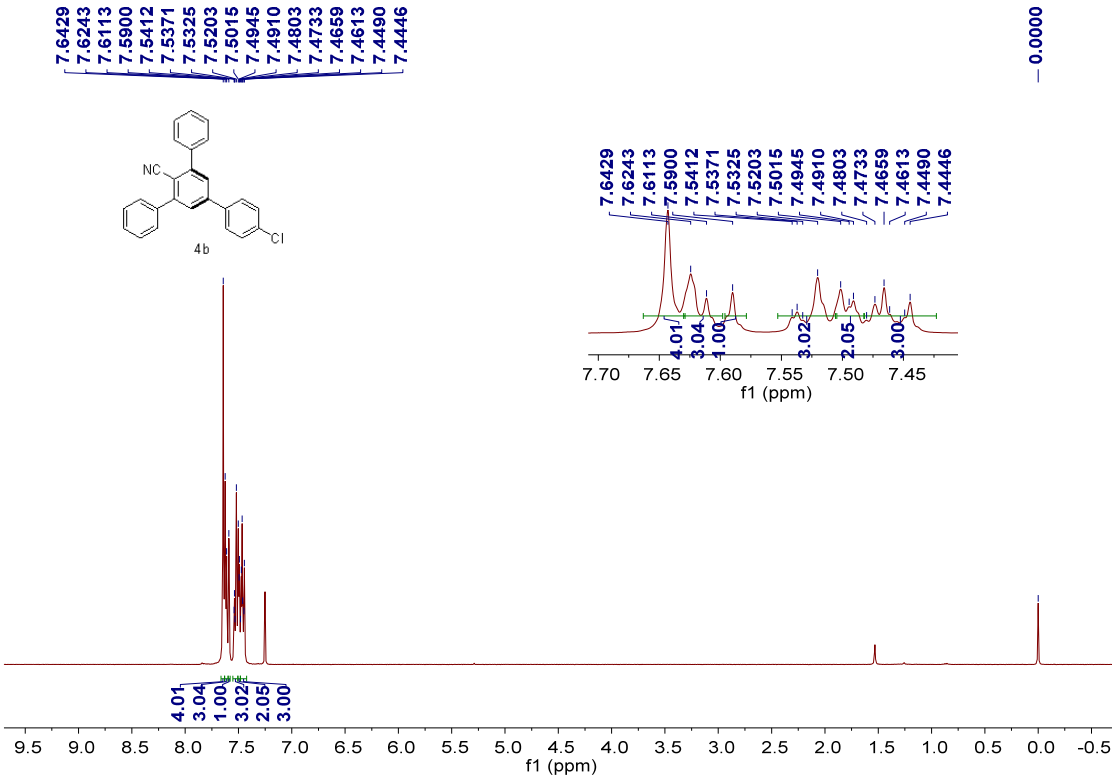

155.81
139.11
138.50
135.99
129.58
128.73
128.43
128.11
127.92
127.90
126.00
117.97
108.81
77.48
77.16
76.84
29.72
22.45
NC
H
3w'
f1 (ppm)

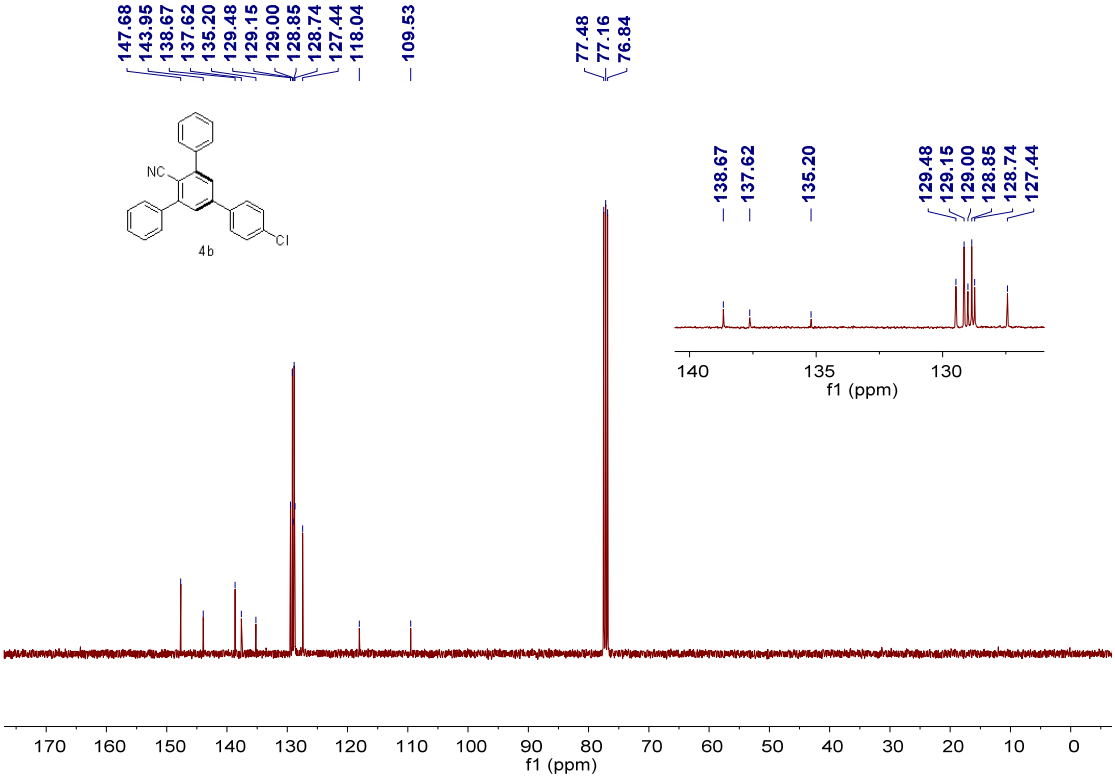
4a


7.6898
7.6794
7.6597
7.6388
7.5370
7.5198
7.5012
7.4874
7.4694
7.4509
7.4482
7.4301
7.4119
-0.0000
NC
4a
3.03
5.02
1.01
5.04
2.00
1.00
f1 (ppm)

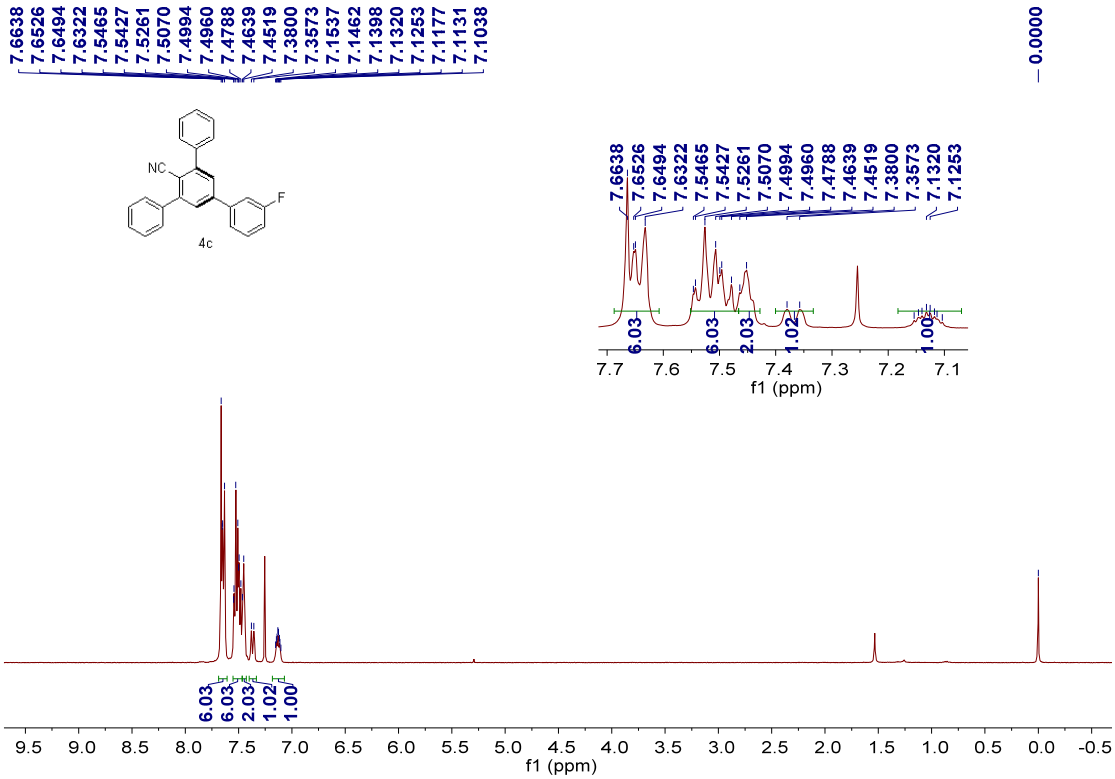

147.53
145.27
139.22
138.86
129.27
129.20
128.90
128.81
127.67
127.50
118.21
109.17
77.48
77.16
76.84
NC
4a
f1 (ppm)

4b


7.6429
7.6243
7.6113
7.5900
7.5412
7.5371
7.5325
7.5203
7.5015
7.4945
7.4910
7.4803
7.4733
7.4659
7.4613
7.4490
7.4446
0.0000
NC
Cl
4b
4.01
3.04
1.00
3.02
2.05
3.00
f1 (ppm)


147.68
143.95
138.67
137.62
135.20
129.48
129.15
129.00
128.85
128.74
127.44
118.04
109.53
77.48
77.16
76.84
NC
Cl
4b
f1 (ppm)

4c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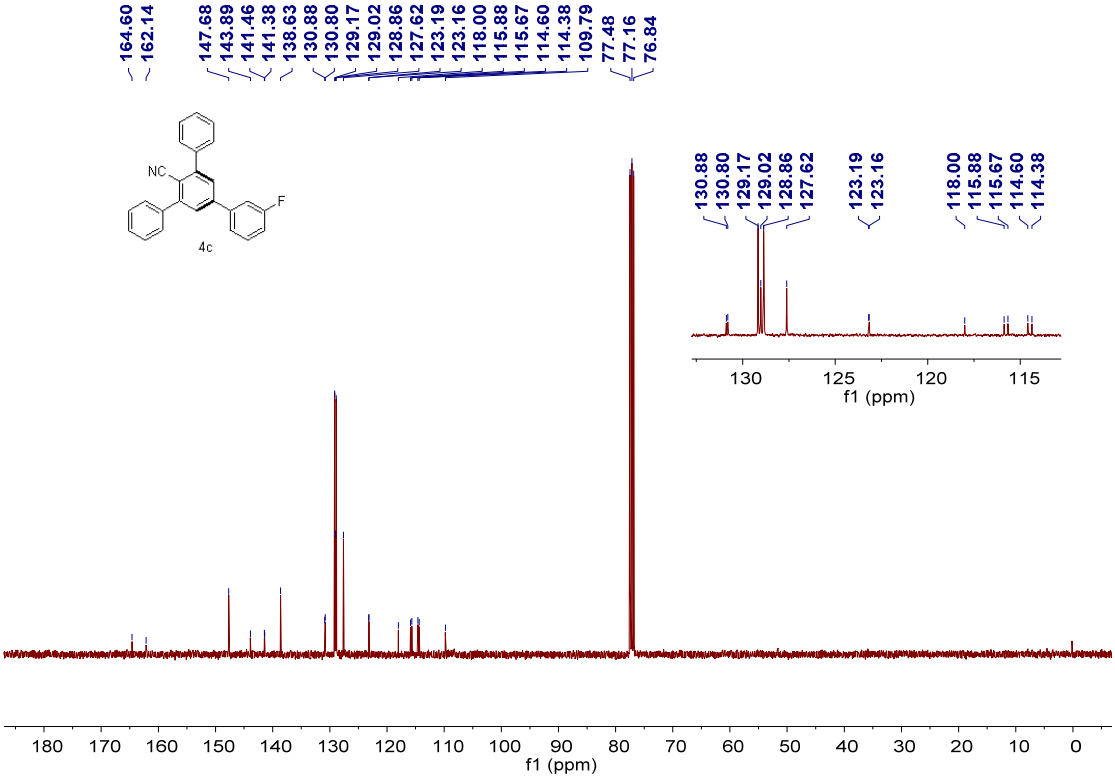
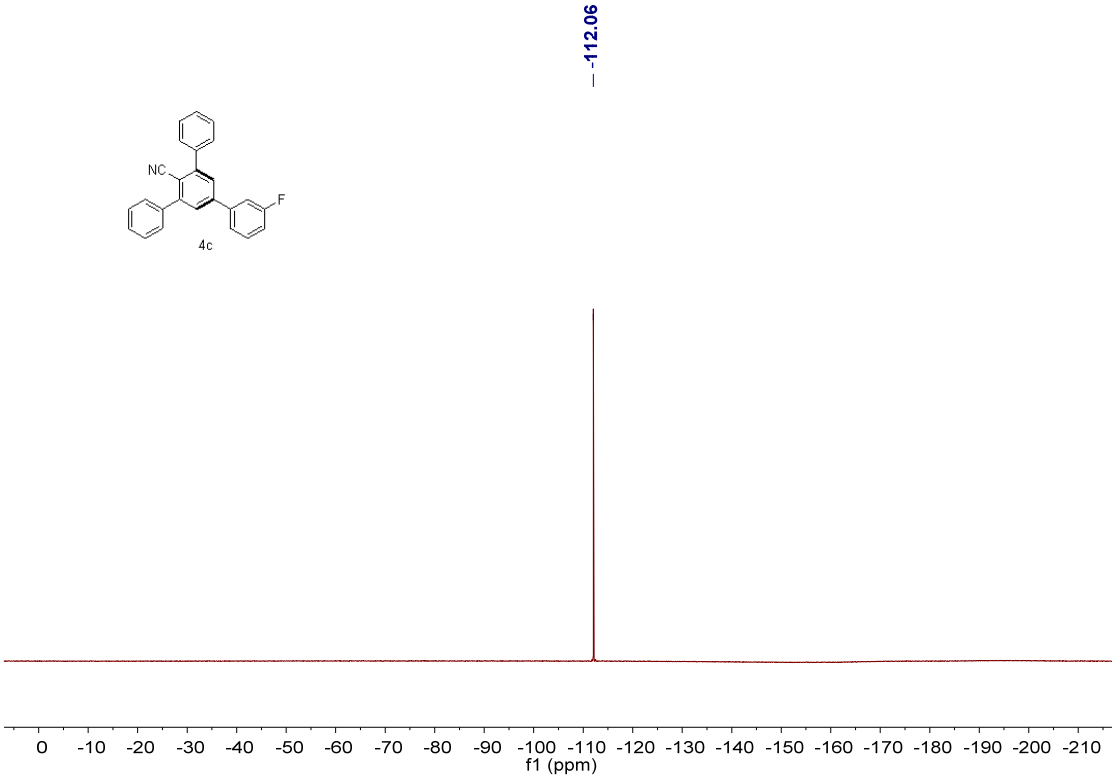
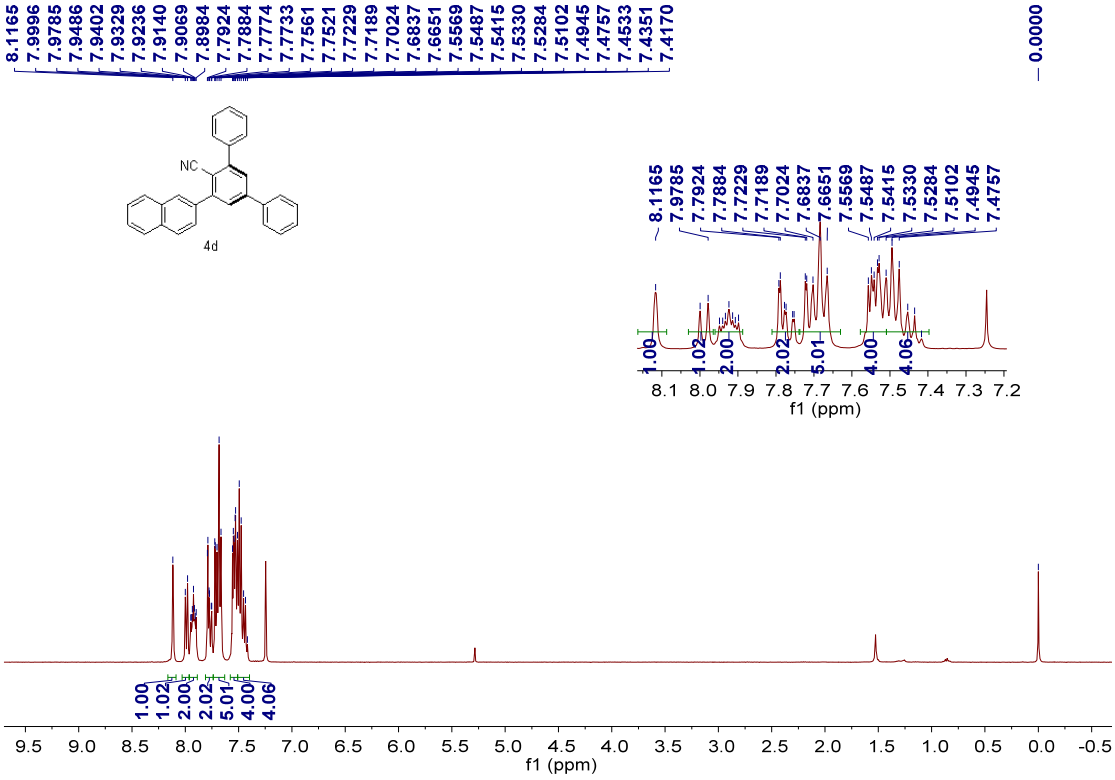
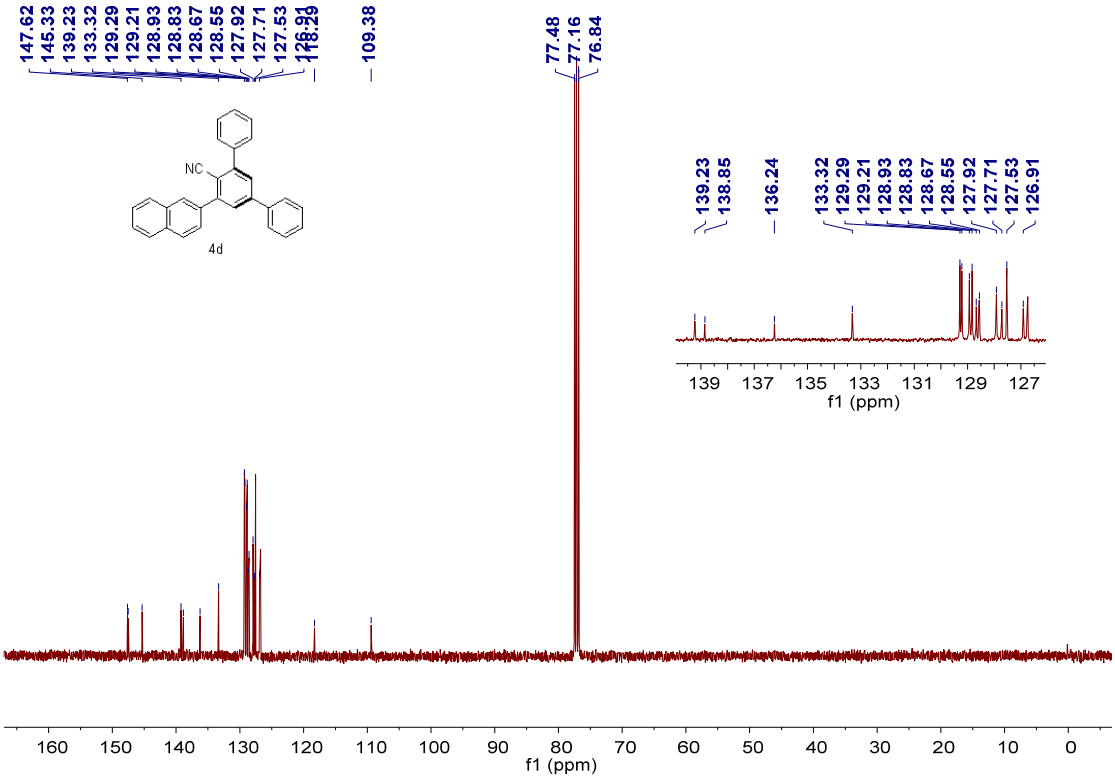
7.6526
7.6494
7.6322
7.5465
7.5427
7.5261
7.5070
7.4994
7.4960
7.4788
7.4639
7.4519
7.3800
7.3573
7.1537
7.1462
7.1398
7.1320
7.1253
7.1177
7.1131
7.1038
0.0000
NC
F
4c
6.03
6.03
2.03
1.02
1.00
f1 (ppm)


164.60
162.14
147.68
143.89
141.46
141.38
138.63
130.88
130.80
129.17
129.02
128.86
127.62
123.19
123.16
118.00
115.88
115.67
114.60
114.38
109.79
77.48
77.16
76.84
NC
F
4c
f1 (ppm)


-112.06
NC
F
4c
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

4d


8.1165
7.9996
7.9785
7.9486
7.9402
7.9329
7.9236
7.9140
7.9069
7.8984
7.7924
7.7884
7.7774
7.7733
7.7561
7.7521
7.7229
7.7189
7.7024
7.6837
7.6651
7.5569
7.5487
7.5415
7.5330
7.5284
7.5102
7.4945
7.4757
7.4533
7.4351
7.4170
0.0000
NC
4d
1.00 1.02 2.00 2.02 5.01 4.00 4.06
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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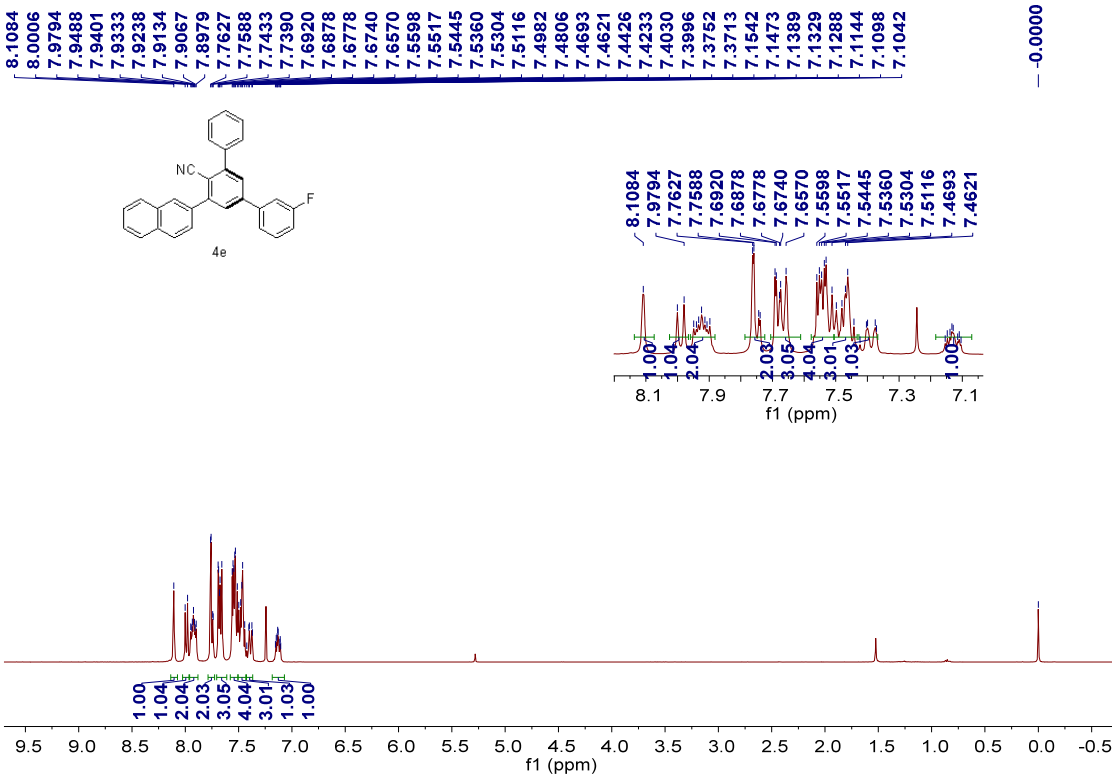
139.23
133.32
129.29
129.21
128.93
128.83
128.67
128.55
127.92
127.71
127.53
126.91
109.38
77.48
77.16
76.84
NC
4d
139.23
138.85
136.24
133.32
129.29
129.21
128.93
128.83
128.67
128.55
127.92
127.71
127.53
126.91
139 137 135 133 131 129 127
f1 (ppm)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4e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7.7433
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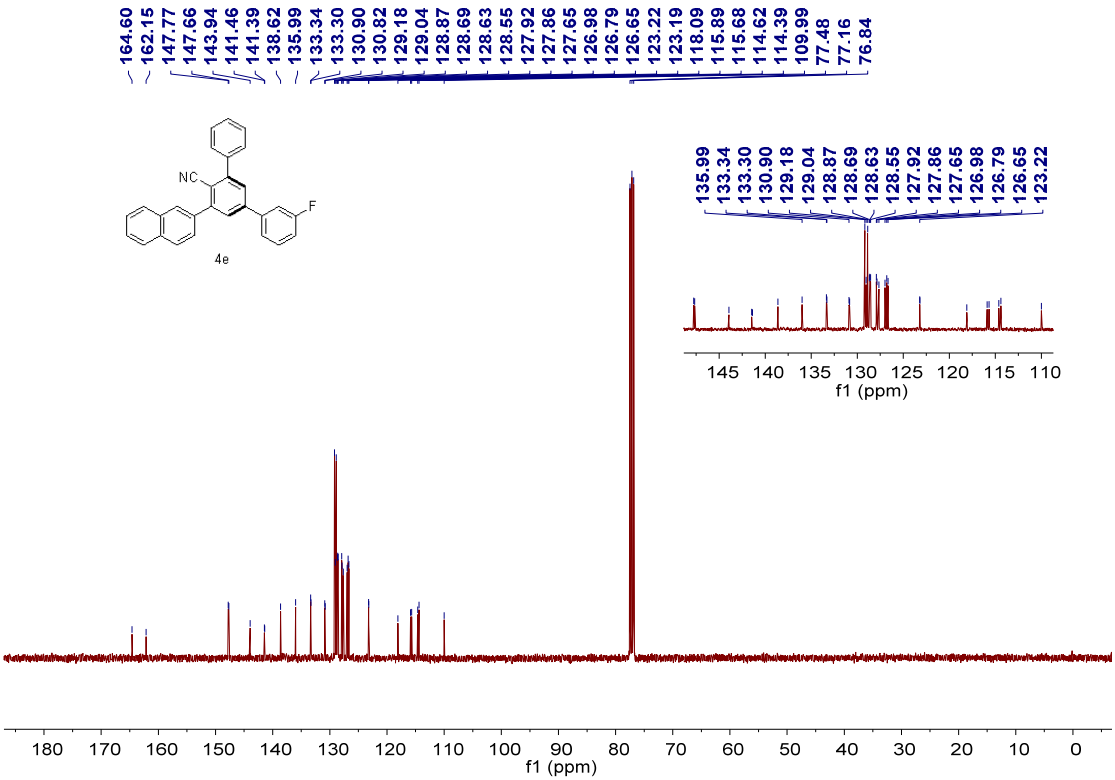
7.6920
7.6878
7.6778
7.6740
7.6570
7.5598
7.5517
7.5445
7.5360
7.5304
7.5116
7.4982
7.4806
7.4693
7.4621
7.4426
7.4233
7.4030
7.3996
7.3752
7.3713
7.1542
7.1473
7.1389
7.1329
7.1288
7.1144
7.1098
7.1042
-0.0000
NC
F
4e
8.1084
7.9794
7.7627
7.7588
7.6920
7.6878
7.6778
7.6740
7.6570
7.5598
7.5517
7.5445
7.5360
7.5304
7.5116
7.4693
7.4621
1.00
1.04
2.04
2.03
3.05
4.04
3.01
1.03
1.00
8.1 7.9 7.7 7.5 7.3 7.1
f1 (ppm)
1.00
1.04
2.04
2.03
3.05
4.04
3.01
1.03
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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143.94
141.46
141.39
138.62
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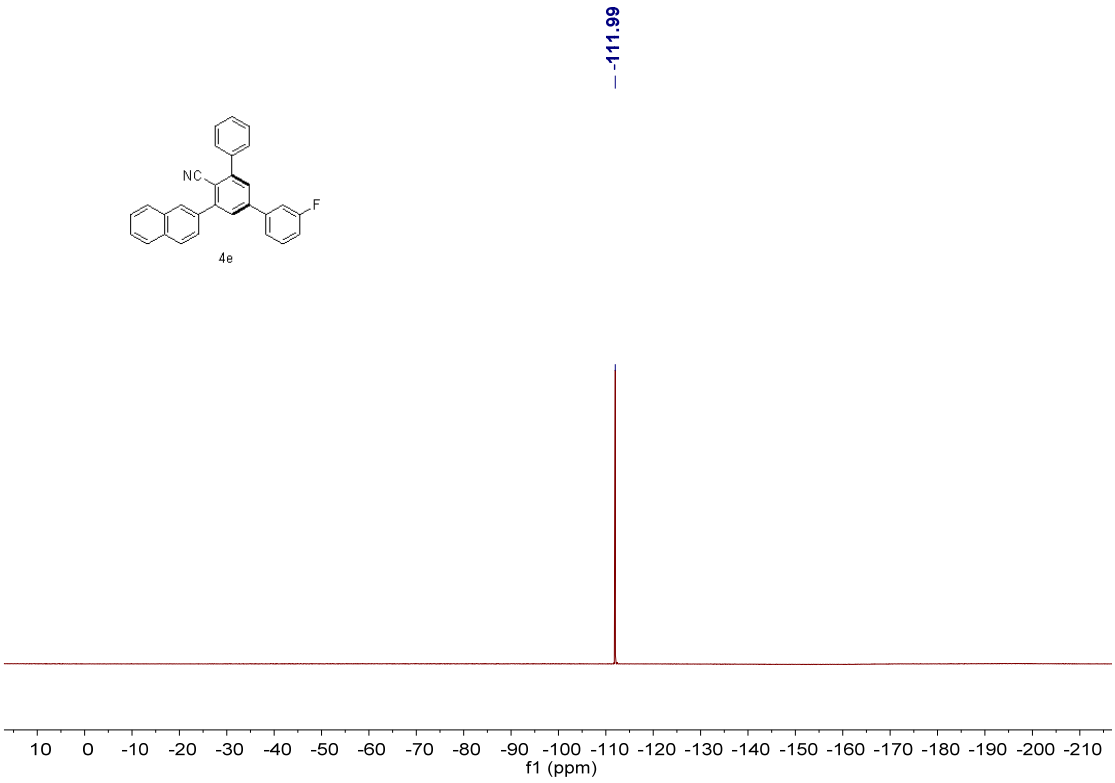
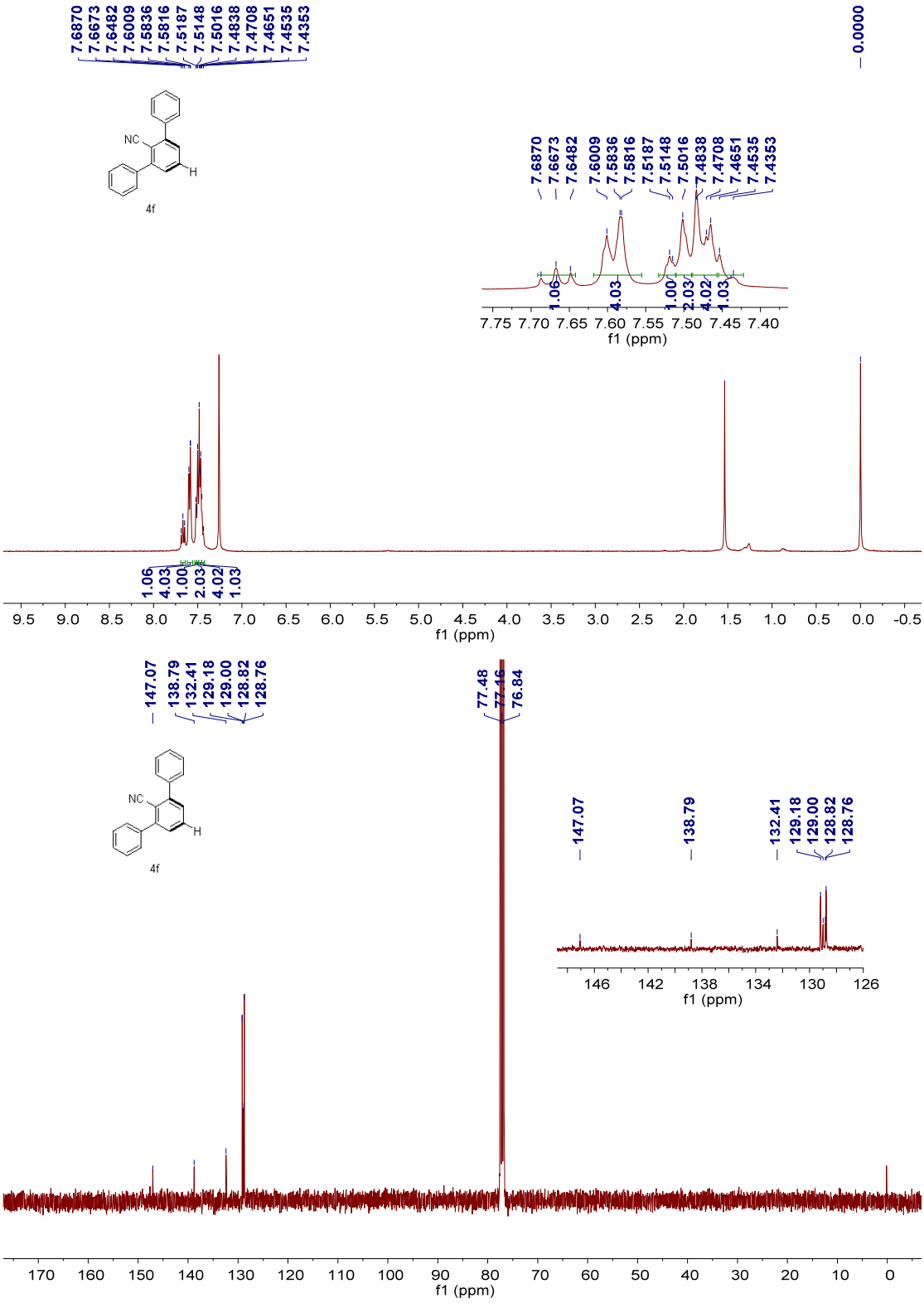
133.34
133.30
130.90
130.82
129.18
129.04
128.87
128.69
128.63
128.55
127.92
127.86
127.65
126.98
126.79
126.65
123.22
123.19
118.09
115.89
115.68
114.62
114.39
109.99
77.48
77.16
76.84
NC
F
4e
135.99
133.34
133.30
130.90
129.18
129.04
128.87
128.69
128.63
128.55
127.92
127.86
127.65
126.98
126.79
126.65
123.22
145 140 135 130 125 120 115 110
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


-111.99
NC
F
4e
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

4f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7.6482
7.6009
7.5836
7.5816
7.5187
7.5148
7.5016
7.4838
7.4708
7.4651
7.4535
7.4353
0.0000
NC
H
4f
1.06
4.03
1.00
2.03
4.02
1.03
f1 (ppm)
147.07
138.79
132.41
129.18
129.00
128.82
128.76
77.48
77.16
76.84
f1 (ppm)