Supporting Information

AgF-Mediated Fluorinative Homocoupling of *gem*-Difluoroalkenes

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Table of Contents

1. General information ……S2
2. Preparation of starting materials ……S2
3. Characterization data of the starting materials ……..……S2
4. General experimental procedure ……..S6
5. Characterization data of the products ……..……S7
6. References ……..……S14
7. Copies of ^{19}F NMR, ^{1}H NMR, and ^{13}C NMR Spectra ……..…….……...S15

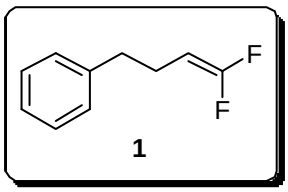
1. General information:

All reactions were carried out in oven-dried sealed tube under nitrogen atmosphere. Commercial reagents and superdry solvents (water < 0.005%, with molecular sieves) were used as received. Product purification was performed using 300-400 mesh silica gel column chromatography. The NMR spectra for ^{1}H, ^{19}F, ^{13}C were recorded with AM300, vx300, MR400 spectrometer. Chemical shifts (δ) were given in ppm and spin-spin coupling constants (*J*) were given in Hz. The residual proton signal of $CDCl_3$ (δ = 7.26 ppm) in the ^{1}H NMR spectra, the carbon signal of $CDCl_3$ (δ = 77.0 ppm) in the ^{13}C NMR spectra, and the signal of $CFCl_3$ (δ = 0.0 ppm) in the ^{19}F NMR spectra were used as internal references. IR spectra were recorded with a Nicolet 380FT-IR spectrometer. High-resolution MS spectra were recorded with a tandem TOF spectrometer. The *syn*/*anti* ratio of products 4a-4k was determined according to ^{19}F NMR spectra of the corresponding crude products. The chemical shift of the *anti* isomer is normally about 2.8 ppm downfield from the *syn* isomer according to product 4m (*anti* configuration, -62.82 ppm; *syn* configuration, -65.58 ppm), the *syn* isomer of which was confirmed by X-ray crystallography.

2. Preparation of starting materials:

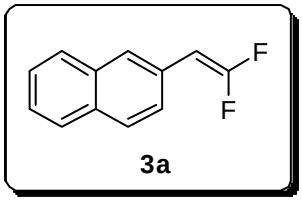
Substrates **1**, **3a-3m** were prepared according to our reported procedure,[1] and the new method for the preparation of **4m** will be published separately.

3. Characterization data of the starting materials:



(4,4-Difluorobut-3-en-1-yl)benzene (1)[1]

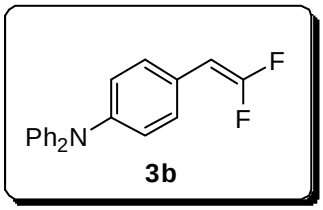
Colorless oil. **^{1}H NMR** ($CDCl_3$, 400 MHz): δ 7.17−7.32 (m, 5H), 4.16 (dtd, *J* = 25.6 Hz, 7.8 Hz, 2.4 Hz, 1H), 2.69 (t, *J* = 7.8 Hz, 2H), 2.30 (m, 2H). **^{19}F NMR** ($CDCl_3$, 376 MHz): δ -89.04 (d, *J* = 46.4 Hz, 1F), -91.07 (dd, *J* = 46.3 Hz, 24.4 Hz, 1F). **MS** (EI, m/z): 168 (M^+, 17.7), 91 (100.0), 65 (12.6).



2-(2,2-difluorovinyl)naphthalene (3a)[1]

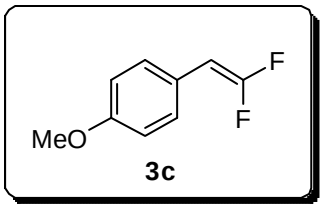
White solid. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.85–7.72 (m, 4H), 7.53–7.40 (m, 3H), 5.43 (dd, *J* = 26.2, 3.9 Hz, 1H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -81.91 (dd, *J* = 30.8, 26.2 Hz, 1F), -83.63 (dd, *J* = 30.8, 3.9 Hz, 1F). **MS** (EI, m/z): 190 (M^+, 100.0), 170

(24.8), 139 (23.6), 95 (8.1).



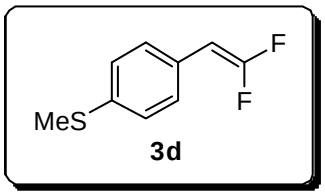
4-(2,2-difluorovinyl)-N,N-diphenylaniline (3b)

White solid. Yield: 64%. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30–7.21 (m, 6H), 7.14–7.03 (m, 8H), 5.24 (dd, *J* = 26.5, 3.8 Hz, 1H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -83.49 (dd, *J* = 34.5, 26.5 Hz, 1F), -85.57 (dd, *J* = 34.5, 3.8 Hz, 1F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 155.93 (dd, *J* = 297.4, 287.4 Hz), 147.44 (s), 146.63 (t, *J* = 2.3 Hz), 129.25 (s), 128.36 (dd, *J* = 6.4, 3.6 Hz), 124.36 (s), 124.12 (t, *J* = 6.1 Hz), 123.58 (s), 122.98 (s), 81.69 (dd, *J* = 29.1, 13.9 Hz). **MS** (EI, m/z, %): 307 (M^+, 100.00), 308 (21.49), 84 (10.86), 77 (8.40). **HRMS** (EI): Calcd. For $C_{20}H_{15}NF_2$: 307.1173; Found: 307.1169. **IR** (film): 3060.7, 3035.8, 2921.8, 2850.4, 1940.7, 1728.1, 1592.6, 1494.2, 1328.7, 1166.6, 938.2, 847.1, 753.6, 695.8, 578.2 cm^{-1}.



1-(2,2-difluorovinyl)-4-methoxybenzene (3c)[1]

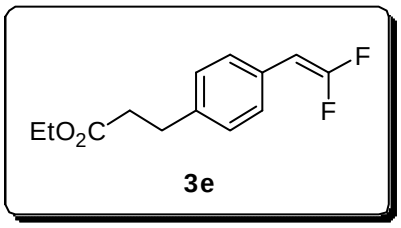
Colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.7 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 5.20 (dd, *J* = 26.4, 3.9 Hz, 1H), 3.79 (s, 3H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -84.68 (dd, *J* = 36.8, 26.4 Hz, 1F), -86.48 (dd, *J* = 36.8, 3.7 Hz, 1F). **MS** (EI, m/z, %): 170 (M^+, 100.00), 127 (80.40), 77 (17.03), 51 (9.95).



(4-(2,2-difluorovinyl)phenyl)(methyl)sulfane (3d)

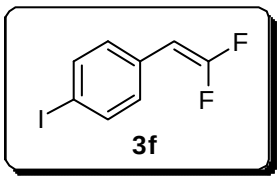
White solid. Yield: 86%. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.25–7.17 (m, 4H), 5.21 (dd, *J* = 26.3, 3.7 Hz, 1H), 2.46 (s, 3H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -82.30 (dd, *J* = 32.2, 26.3 Hz, 1F), -84.47 (dd, *J* = 32.2, 3.4 Hz, 1F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 156.09 (dd, *J* = 298.0, 288.1 Hz), 137.24 (t, *J* = 2.3 Hz), 127.87 (dd, *J* = 6.4, 3.6 Hz), 127.06–126.92 (m), 126.61 (s), 81.90–81.44 (m), 15.61 (s). **MS** (EI, m/z, %): 186 (M^+, 100.00), 171 (50.54), 127 (27.17), 121 (8.56). **HRMS** (EI): Calcd. For $C_9H_8F_2S$: 186.0315; Found: 186.0312. **IR** (film): 3030.3, 2986.1, 2922.1, 1731.3, 1599.0,

1496.3, 1406.8, 1351.1, 1248.2, 1168.2, 1095.1, 938.1, 837.6, 510.5 cm^{-1}.



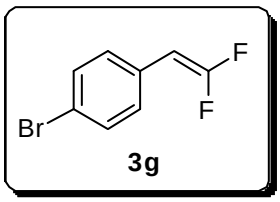
ethyl 3-(4-(2,2-difluorovinyl)phenyl)propanoate (3e)

Colorless oil. Yield: 75%. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.24 (d, *J* = 8.2 Hz, 2H), 7.16 (d, *J* = 8.2 Hz, 2H), 5.22 (dd, *J* = 26.4, 3.8 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 2.92 (t, *J* = 7.8 Hz, 2H), 2.60 (t, *J* = 7.8 Hz, 2H), 1.22 (t, *J* = 7.1 Hz, 3H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -82.90 (dd, *J* = 32.8, 26.4 Hz, 1F), -84.91 (dd, *J* = 32.8, 3.5 Hz, 1F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 172.70 (s), 156.14 (dd, *J* = 297.8, 287.7 Hz), 139.46 (t, *J* = 2.0 Hz), 128.60 (s), 128.30–128.15 (m), 127.65 (dd, *J* = 6.3, 3.5 Hz), 81.83 (dd, *J* = 29.0, 13.7 Hz), 60.38 (s), 35.69 (s), 30.55 (s), 14.11 (s). **MS** (EI, m/z, %): 240 (M^+, 33.14), 166 (100.00), 153 (64.93), 169 (35.70). **HRMS** (EI): Calcd. For $C_{13}H_{14}O_2F_2$: 240.0962; Found: 240.0960. **IR** (film): 3417.6, 2983.6, 2255.9, 1732.0, 1516.7, 1446.2, 1350.4, 1249.1, 1167.5, 1039.3, 939.8, 911.1, 853.6, 734.3 cm^{-1}



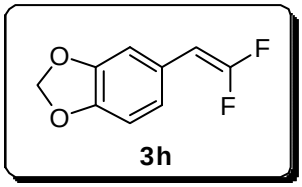
1-(2,2-difluorovinyl)-4-iodobenzene (3f) [2]

Colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.64 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.4 Hz, 2H), 5.19 (dd, *J* = 25.9, 3.5 Hz, 1H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -80.89 (dd, *J* = 28.4, 26.2 Hz, 1F), -82.78 (dd, *J* = 28.4, 3.4 Hz, 1F). **MS** (EI, m/z, %): 266 (M^+, 98.64), 184 (100.00), 183 (91.82), 119 (59.9), 205 (44.3).



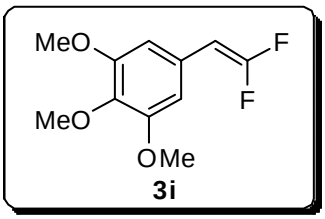
1-bromo-4-(2,2-difluorovinyl)benzene (3g) [1]

Colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.45 (d, *J* = 8.5 Hz, 2H), 7.18 (d, *J* = 8.5 Hz, 2H), 5.22 (dd, *J* = 25.9, 3.6 Hz, 1H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -81.27 (dd, *J* = 29.2, 26.0 Hz, 1F), -83.09 (dd, *J* = 29.2, 3.5 Hz, 1F). **MS** (EI, m/z, %): 218 (M^+, 20.20), 127 (100.00), 155 (70.25), 77 (27.70).



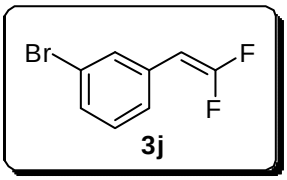
5-(2,2-difluorovinyl)benzo[d][1,3]dioxole (3h) [1]

Colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.86 (d, *J* = 0.4 Hz, 1H), 6.78–6.69 (m, 2H), 5.93 (s, 2H), 5.17 (dd, *J* = 25.9, 3.9 Hz, 1H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -83.93 (dd, *J* = 35.9, 25.9 Hz, 1F), -86.15 (dd, *J* = 35.9, 3.8 Hz, 1F). **MS** (EI, m/z, %): 184 (M^+, 100.00), 183 (85.34), 125 (17.02), 107 (4.66).



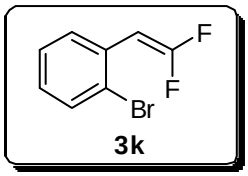
5-(2,2-difluorovinyl)-1,2,3-trimethoxybenzene (3i)

White solid. Yield: 89%. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 6.55 (s, 2H), 5.21 (dd, *J* = 25.8, 3.9 Hz, 1H), 3.85 (s, 6H), 3.85 (s, 3H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -82.95 (dd, *J* = 33.3, 25.8 Hz, 1F), -84.78 (dd, *J* = 33.3, 3.8 Hz, 1F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 155.90 (dd, *J* = 297.5, 287.8 Hz), 153.16 (d, *J* = 0.6 Hz), 136.97 (t, *J* = 2.4 Hz), 125.72 (dd, *J* = 7.0, 6.0 Hz), 104.65 (dd, *J* = 6.7, 3.5 Hz), 82.19 (dd, *J* = 29.6, 13.2 Hz), 60.73 (s), 55.88 (s). **MS** (EI, m/z, %): 230 (M^+, 100.00), 215 (84.36), 187 (36.57), 157 (20.10). **HRMS** (EI): Calcd. For $C_{11}H_{12}O_3F_2$: 230.0755; Found: 230.0751. **IR** (film): 3396.0, 2941.6, 2842.0, 1731.4, 1584.4, 1510.9, 1455.8, 1421.6, 1358.9, 1325.4, 1251.8, 1130.3, 901.9 712.8 cm^{-1}.



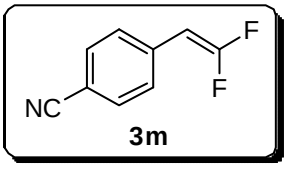
1-bromo-3-(2,2-difluorovinyl)benzene (3j) [3]

Colorless oil. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.47 (s, 1H), 7.36 (d, *J* = 7.6 Hz, 1H), 7.26–7.15 (m, 2H), 5.28–5.15 (m, 1H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -80.46 (dd, *J* = 27.5, 26.6 Hz, 1F), -82.41 (dd, *J* = 27.5, 3.0 Hz, 1F). **MS** (EI, m/z, %): 218 (M^+, 13.47), 220 (12.53), 119 (9.71), 51 (34.1).



1-bromo-2-(2,2-difluorovinyl)benzene (3k) [4]

Colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.30 (t, *J* = 7.6 Hz, 1H), 7.15–7.06 (m, 1H), 5.69 (dd, *J* = 25.4, 3.7 Hz, 1H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -81.79 (dd, *J* = 26.3, 3.6 Hz, 1F), -83.05 (dd, *J* = 26.3 Hz, 25.9 Hz, 1F). **MS** (EI, m/z, %): 218 (M^+, 98.82), 220 (100.00), 119 (85.85), 139 (47.95), 84 (18.29).



4-(2,2-difluorovinyl)benzonitrile (3m) [5]

White solid. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 2H), 5.35 (dd, *J* = 25.6, 3.4 Hz, 1H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -77.83 (dd, *J* = 25.6, 20.6 Hz, 1F), -79.49 (dd, *J* = 20.6, 3.3 Hz, 1F). **MS** (EI, m/z, %): 165 (M^+, 100), 145 (10.79), 166 (10.49), 138 (8.12).

4. General experimental procedure:

General procedure for the homocoupling reaction:

In the glovebox, 310 mg of AgF (3.0 mmol, 3.0 equiv.) and 50 mg of molecular sieves were added to a dried sealed tube equipped with a stir bar. Solvent THF (2.5 mL) and pyridine (2.5 mL) were injected sequentially, and then 1.0 mmol of *gem*-difluoroalkene (1.0 equiv.) was added. The tube was sealed with Teflon stopcock and moved into oil bath (80 ^{o}C) outside the glovebox. The reaction mixture was stirred at 80 ^{o}C under the protection from light for 6 h. After that, the reaction mixture was cooled to room temperature and then filtered through celite with ether (20 mL). Thereafter, the solvent were removed via rotary evaporation and purified by silica gel flash chromatography with hexane and EtOAc.

The preparation of 5a/5b from 4m:

The reaction was conducted following the general procedure of homocoupling reaction on 0.1 mmol scale. The product was confirmed by comparing the GC-MS signal and ^{19}F signal with authentic sample of **4m**, and the yield was determined by ^{19}F NMR spectra with 1,3,5-trifluorobenzene as an internal standard.

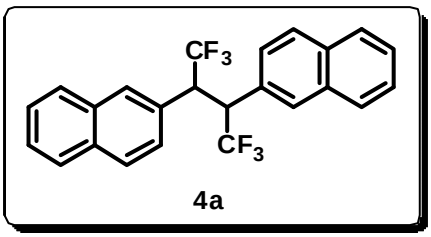
The radical capture experiment (preparation of 7 from 3a):

The reaction was conducted following the general homocoupling procdure with additional TEMPO (2,2,6,6-Tetramethyl-1-oxylpiperidine, 3.0 equiv.).

General procedure for the reaction of 3b with different "F^-" sources:

The reactions were conducted similar to the general homocoupling procedure on 0.2 mmol scale without molecular sieves. "F^-" sources **A** (CsF, 3.0 equiv.), **B** (AgF, 3.0 equiv.), and **C** (CsF, 3.0 equiv.; $AgBF_4$, 3.0 equiv.) were used separately, and solvent DMSO (2.0 mL) was used instead of THF-pyridine. The yield was determined via ^{19}F NMR spectra with 1,3,5-trifluorobenzene as an internal standard.

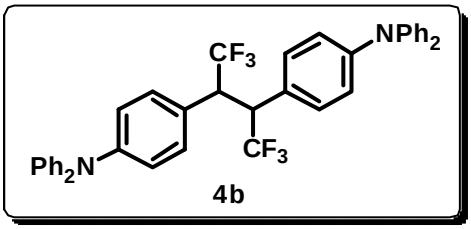
5. Characterization data of the products:



2,2'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)dinaphthalene (4a)

4a-*syn*: Colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.74 (d, *J* = 7.5 Hz, 2H), 7.66 (d, *J* = 7.5 Hz, 2H), 7.58 (d, *J* = 8.6 Hz, 2H), 7.53 (s, 2H), 7.48–7.41 (m, 2H), 6.99 (d, *J* = 8.6 Hz, 2H), 4.36–4.33 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -65.59 (d, *J* = 8.8 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 132.91 (s), 132.74 (s), 130.36 (s), 128.52 (s), 127.96 (s), 127.78 (s), 127.51 (s), 127.04 (s), 126.61 (s), 126.30 (s), 124.75 (s), 50.36 (q, *J* = 28.1 Hz). **MS** (EI, m/z, %): 418 (M^+, 5.04), 209 (100.00), 189 (13.57), 210 (13.36), 139 (6.04). **HRMS** (EI): Calcd. For $C_{24}H_{16}F_6$: 418.1156; Found: 418.1154. **IR** (film): 3060.2, 2924.9, 1601.5, 1510.1, 1254.4, 1158.6, 1115.6, 815.6, 749.7, 478.0 cm^{-1}.

4a-*anti*: White solid. **MP**: 248-250 ^{o}C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.92–7.87 (m, 8H), 7.58–7.52 (m, 6H), 4.30–4.25 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -62.84 (d, *J* = 7.7 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 133.22 (s), 133.17 (s), 130.14 (s), 129.15 (s), 128.58 (s), 128.00 (s), 127.72 (s), 126.67 (s), 126.54 (s), 125.69 (s), 125.63 (q, *J* = 281.9 Hz), 51.95 (q, *J* = 26.5 Hz). **MS** (EI, m/z, %): 418 (M^+, 4.22), 209 (100.00), 159 (24.33), 189 (17.56), 190 (3.61). **HRMS** (EI): Calcd. For $C_{24}H_{16}F_6$: 418.1156; Found: 418.1160. **IR** (film): 3415.8, 2848.9, 2256.2, 2129.0, 1651.8, 1315.0, 1276.4, 1178.2, 1094.6, 1026.0, 1000.7, 821.8, 762.0 cm^{-1}.

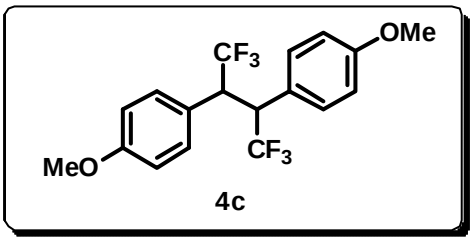


4,4'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(N,N-diphenylaniline) (4b)

4b-*syn*: White solid. **MP**: 152-153 ^{o}C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.22 (t, *J* = 7.9 Hz, 8H), 7.06–7.00 (m, 12H), 6.89 (d, *J* = 8.7 Hz, 4H), 6.80 (d, *J* = 8.5 Hz, 4H), 4.01–3.89 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -66.09 (d, *J* = 9.0 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 147.83 (s), 147.36 (s), 130.90 (s), 129.34 (s), 127.59 (q, *J* = 280.8 Hz), 124.90 (s), 124.80 (s), 124.57 (s), 123.22 (s), 49.73 (q, *J* = 28.2 Hz). **MS** (ESI, m/z, %): 653 ($M + H^+$). **HRMS** (ESI): Calcd. For $C_{40}H_{31}N_2F_6^+$: 653.23859; Found: 653.2380. **IR** (film): 3036.7, 1613.0, 1592.8, 1511.0, 1493.2, 1385.4, 1332.1, 1283.1, 1252.3, 1127.5, 1094.5, 827.3, 751.7, 695.9, 501.2 cm^{-1}.

4b-*anti*: White solid. **MP**: 145-147 ^{o}C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30–7.21 (m,

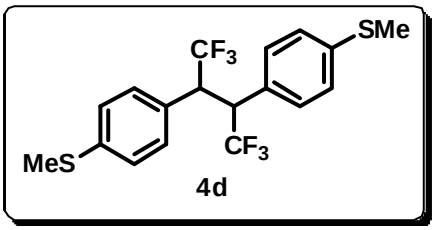
12H), 7.13–7.03 (m, 16H), 3.94–3.83 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.25 (d, *J* = 6.3 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 148.10 (s), 147.43 (s), 129.76 (s), 129.32 (s), 127.13 (q, *J* = 280.8 Hz), 126.13 (s), 124.69 (s), 123.22 (s), 122.92 (s), 51.22 (q, *J* = 26.3 Hz). **MS** (ESI, m/z, %): 653 (M + H^+). **HRMS** (ESI): Calcd. For $C_{40}H_{31}N_2F_6^+$: 653.23859; Found: 653.2394. **IR** (film): 3035.8, 1592.6, 1512.9, 1494.4, 1336.8, 1273.3, 1192.7, 1174.1, 1096.8, 1028.2, 900.3, 09.7, 747.7, 695.3, 498.4 cm^{-1}.



4,4'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(methoxybenzene) (4c)

4c-*syn*: Colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.84 (d, *J* = 8.5 Hz, 4H), 6.72 (d, *J* = 8.9 Hz, 4H), 4.01–3.94 (m, 2H), 3.76 (s, 6H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -66.33 (d, *J* = 9.3 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 159.48 (s), 131.47 (s), 126.20 (q, *J* = 280.7 Hz), 123.05 (s), 113.47 (s), 55.12 (s), 49.33 (q, *J* = 27.5 Hz). **MS** (EI, m/z, %): 378 (M^+, 2.79), 189 (100.00), 190 (10.30), 127 (1.04), 96 (0.87). **HRMS** (EI): Calcd. For $C_{18}H_{16}O_2F_6$: 378.1054; Found: 378.1055. **IR** (film): 3036.9, 2961.3, 2913.1, 1615.3, 1587.9, 1517.7, 1465.5, 1367.8, 1254.5, 1186.8, 1133.2, 1109.4, 1030.7, 814.0, 692.1, 532.7 cm^{-1}.

4c-*anti*: White solid. **MP**: 162-164 ^oC. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.6 Hz, 4H), 6.93 (d, *J* = 8.8 Hz, 4H), 3.89 (M, 2H), 3.83 (s, 6H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.52 (d, *J* = 8.0 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 159.71 (s), 130.13 (s), 125.71 (q, *J* = 281.5 Hz), 124.82 (s), 114.08 (s), 55.20 (s), 51.05 (q, *J* = 26.3 Hz). **MS** (EI, m/z, %): 378 (M^+, 2.03), 189 (100.00), 190 (9.79), 249 (4.25), 237 (2.54), 239 (2.23). **HRMS** (EI): Calcd. For $C_{18}H_{16}O_2F_6$: 378.1054; Found: 378.1052. **IR** (film): 3015.4, 3013.3, 2978.7, 2846.2, 1619.3, 1519.8, 1470.8, 1339.0, 1302.7, 1273.1, 1232.5, 1178.3, 1090.4, 1028.0, 851.2, 808.7, 641.5 cm^{-1}.

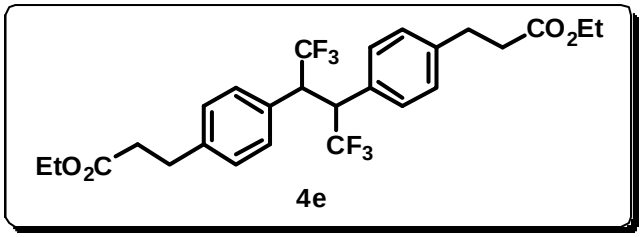


((1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(4,1-phenylene))bis(methylsulfane)(4d)

4d-*syn*: White solid. **MP**: 70-72 ^oC. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.06 (d, *J* = 8.5 Hz, 4H), 6.84 (d, *J* = 8.1 Hz, 4H), 4.03–3.94 (m, 2H), 2.44 (s, 6H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -66.04 (d, *J* = 9.0 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 139.39 (s), 130.60 (s), 127.29 (s), 125.94 (q, *J* = 280.8 Hz), 125.61 (s), 49.57 (q), 15.22 (s). **MS** (EI, m/z, %): 410 (M^+, 6.98), 205 (100.00), 155 (14.12), 247 (3.59). **HRMS** (EI): Calcd. For $C_{18}H_{16}F_6S_2$: 410.0598; Found: 410.0594. **IR** (film): 3033.7, 2922.1, 1601.6,

1499.6, 1439.8, 1326.0, 1256.7, 1197.7, 1092.8, 10181, 975.2, 903.2, 818.1, 753.5, 661.6, 539.2 cm^{-1}.

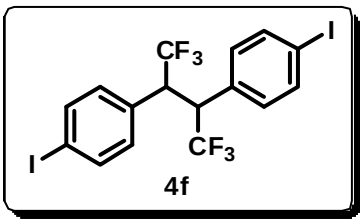
4d-*anti*: White solid. **MP**: 156-158 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.31–7.24 (m, 8H), 3.95–3.85 (m, 2H), 2.50 (s, 6H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.24 (d, *J* = 7.4 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 139.60 (s), 129.38 (s), 129.10 (s), 126.27 (s), 125.48 (q, *J* = 281.6 Hz), 51.24 (q, *J* = 26.3 Hz), 15.33 (s). **MS** (EI, m/z, %): 410 (M^+, 2.8), 205 (100.00), 155 (17.72), 206 (11.82), 307 (9.46), 165 (6.94). **HRMS** (EI): Calcd. For $C_{18}H_{16}F_6S_2$: 410.0598; Found: 410.0600. **IR** (film): 1910.0, 1595.5, 1492.1, 1410.6, 1325.9, 1259.9, 1109.0, 1076.3, 1010.8, 914.7, 845.6, 821.2, 725.7, 703.5, 691.5, 607.2, 507.6 cm^{-1}.



Diethyl-3,3'-((1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(4,1-phenylene))dipropanoate (4e)

Mixture of *syn* & *anti* (*syn*/*anti* = 1.00:0.13): Colorless oil. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 7.8 Hz, 0.13×4H), 7.22 (t, *J* = 7.8 Hz, 0.13×4H), 6.98 (d, *J* = 7.9 Hz, 4H), 6.80 (d, *J* = 7.9 Hz, 4H), 4.09 (q, *J* = 7.1 Hz, 1.13×4H), 4.02–3.89 (m, 1.13×2H), 2.95 (t, *J* = 7.8 Hz, 0.13×4H), 2.86 (t, *J* = 7.8 Hz, 4H), 2.62 (t, 0.13×4H), 2.54 (t, *J* = 7.8 Hz, 4H), 1.19 (t, *J* = 7.1 Hz, 1.13×6H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.31 (d, *J* = 7.5 Hz, 0.13×6F), -65.99 (d, *J* = 9.1 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 172.72 (s), 172.62 (s), 141.14 (s), 140.86 (s), 130.25 (s), 129.09 (s), 129.02 (s), 128.65 (s), 127.99 (s), 126.02 (q, *J* = 280.8 Hz), 60.39 (s), 51.38 (q, *J* = 27.5 Hz), 49.79 (q, *J* = 27.9 Hz), 35.61 (s), 35.59 (s), 30.57 (s), 30.44 (s), 14.12 (s), 14.08 (s). **MS** (EI, m/z, %): 518 (M^+, 1.72), 185 (100.00), 259 (36.42), 186 (18.03), 71 (14.21), 117 (8.39). **HRMS** (EI): Calcd. For $C_{26}H_{28}O_4F_6$: 518.1892; Found: 518.1891. **IR** (film): 3438.9, 2983.6, 1732.6, 1615.9, 1518.8, 1427.4, 1373.9, 1256.7, 1163.1, 1125.1, 1040.1, 942.5, 822.4, 735.1, 667.3 cm^{-1}.

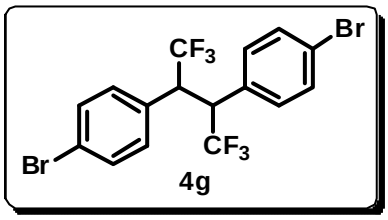
4e-*anti*: White solid. **MP**: 101-103 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.31 (d, *J* = 8.1 Hz, 4H), 7.24 (d, *J* = 8.1 Hz, 4H), 4.12 (q, *J* = 7.1 Hz, 4H), 3.98–3.87 (m, 2H), 2.97 (q, *J* = 7.8 Hz, 4H), 2.64 (t, *J* = 7.8 Hz, 4H), 1.21 (t, *J* = 7.1 Hz, 6H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.30 (d, *J* = 7.9 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 172.74 (s), 141.14 (s), 130.63 (d, *J* = 10.8 Hz), 129.10 (s), 128.65 (s), 125.56 (q, *J* = 281.3 Hz), 60.42 (s), 51.39 (q, *J* = 26.6 Hz), 35.62 (s), 30.58 (s), 14.10 (s). **MS** (EI, m/z, %): 518 (M^+, 1.59), 185 (100.00), 259 (53.29), 186 (19.14), 285 (17.22), 205 (10.9). **HRMS** (EI): Calcd. For $C_{26}H_{28}O_4F_6$: 518.1892; Found: 518.1895. **IR** (film): 2991.3, 2942.7, 1723.1, 1519.1, 1446.0, 1372.4, 1247.2, 1227.0, 1161.6, 1107.8, 1037.1, 945.6, 856.6, 810.5, 669.7, 643.9 cm^{-1}.



4,4'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(iodobenzene) (4f)

4f-*syn*: White solid. **MP**: 156-158 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.56 (d, *J* = 8.4 Hz, 4H), 6.67 (d, *J* = 8.4 Hz, 4H), 4.01–3.94 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -65.92 (d, *J* = 8.9 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 137.54 (s), 131.94 (s), 130.31 (s), 125.56 (q, *J* = 280.9 Hz), 95.00 (s), 49.57 (q, *J* = 28.2 Hz). **MS** (EI, m/z, %): 570 (M^+, 5.65), 285 (100.00), 235 (27.93), 205 (17.88), 158 (11.91). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6I_2$: 569.8776; Found: 569.8777. **IR** (film): 3091.2, 1909.7, 1589.5, 1488.2, 1412.5, 1357.5, 1251.2, 1159.4, 1134.8, 1064.9, 1009.4, 904.9, 815.8, 742.7, 657.2, 527.1 cm^{-1}.

4f-*anti*: White solid. **MP**: 190-192 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 8.3 Hz, 4H), 7.13 (d, *J* = 8.3 Hz, 4H), 3.93–3.83 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.13 (d, *J* = 7.4 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 138.04 (s), 131.98 (d, *J* = 13.9 Hz), 130.73 (s), 125.10 (q, *J* = 281.9 Hz), 94.91 (s), 51.26 (q, *J* = 26.7 Hz). **MS** (EI, m/z, %): 570 (M^+, 4.83), 285 (100.00), 235 (26.27), 205 (18.13), 71 (12.83). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6I_2$: 569.8776; Found: 569.8773. **IR** (film): 2947.6, 1906.2, 1589.7, 1488.3, 1408.2, 1333.1, 1242.6, 1162.3, 1108.5, 1007.1, 841.9, 722.6, 670.5, 646.9, 610.0 cm^{-1}.

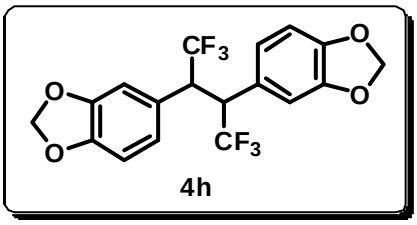


4,4'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(bromobenzene) (4g)

4g-*syn*: White solid. **MP**: 141–143 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.36 (d, *J* = 8.3 Hz, 4H), 6.80 (d, *J* = 8.3 Hz, 4H), 4.03–3.97 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -66.01 (d, *J* = 8.9 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 131.77 (s), 131.59 (s), 129.68 (s), 125.60 (q, *J* = 280.8 Hz), 123.19 (s), 49.48 (q, *J* = 28.2 Hz). **MS** (EI, m/z, %): 474 (M^+, 1.32), 237 (100.00), 239 (98.59), 187 (29.11). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6Br_2$: 473.9053; Found: 473.9050. **IR** (film): 3388.3, 2920.6, 2850.3, 1595.1, 1491.8, 1461.6, 1409.9, 1365.6, 1245.1, 1154.0, 1107.6, 1011.2, 845.5, 801.4, 726.2, 507.9 cm^{-1}.

4g-*anti*: White solid. **MP**: 196–198 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.3 Hz, 4H), 7.26 (d, *J* = 8.3 Hz, 4H), 3.96–3.86 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.17 (d, *J* = 7.7 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 132.11 (s), 131.40 (s), 130.57 (s), 125.13 (q, *J* = 281.5 Hz), 123.21 (s), 51.19 (q, *J* = 26.9 Hz).

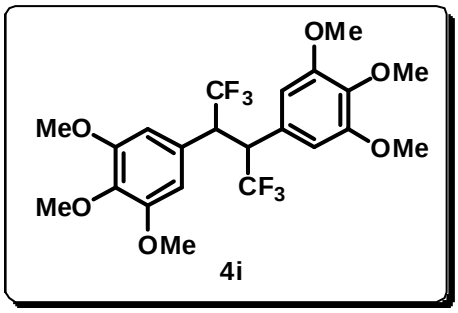
MS (EI, m/z, %): 474 (M^+, 1.61), 237 (100.00), 239 (94.87), 187 (26.64), 189 (26.06). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6Br_2$: 473.9053; Found: 473.9051. **IR** (film): 3054.1, 2961.7, 2926.7, 2853.9, 2305.6, 1906.8, 1595.9, 1493.0, 1411.4, 1307.0, 1244.2, 1166.2, 1104.2, 947.1, 804.1, 725.6, 630.0, 514.8 cm^{-1}.



4h

5,5'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(benzo[d][1,3]dioxole) (4h)

Mixture of *syn* & *anti* (*syn*/*anti* = 1.5:1.0): White solid. **MP**: 61–63 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 6.86 (s, 2H), 6.83–6.80 (m, 4H), 6.65 (d, *J* = 8.0 Hz, 1.5×2H), 6.47 (d, *J* = 8.0 Hz, 1.5×2H), 6.43 (s, 1.5×2H), 6.00 (s, 4H), 5.92 (s, 1.5×4H), 3.93–3.85 (m, 1.5×2H), 3.84–3.78 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.57 (d, *J* = 7.3 Hz, 6F), -66.15 (d, *J* = 8.9 Hz, 1.5×6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 147.96 (s), 147.65 (s), 147.47 (s), 130.07 (s), 127.28 (s), 126.12 (s), 124.58 (t, *J* = 271.8 Hz), 124.49 (t, *J* = 281.1 Hz), 124.15 (s), 123.06 (s), 110.04 (s), 108.76 (s), 108.41 (s), 108.00 (s), 101.38 (s), 101.23 (s), 51.99–51.01 (m), 49.90 (q, *J* = 28.0 Hz). **MS** (EI, m/z, %): 406 (M^+, 3.39), 203 (100.00), 204 (10.32), 153 (8.53). **HRMS** (EI): Calcd. For $C_{18}H_{12}O_4F_6$: 406.0640; Found: 406.0644. **IR** (film): 3497.3, 2916.6, 2786.0, 1732.3, 1612.1, 1507.0, 1490.4, 1378.1, 1256.3, 1161.4, 1094.7, 925.6, 814.2, 680.8, 558.0 cm^{-1}.



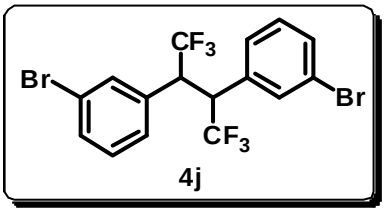
4i

5,5'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(1,2,3-trimethoxybenzene) (4i)

4i-*syn*: White solid. **MP**: 143–145 °C. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 6.13 (s, 4H), 3.99–3.90 (m, 2H), 3.81 (s, 6H), 3.67 (s, 12H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -66.22 (d, *J* = 9.3 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 154.34 (s), 139.87 (s), 127.83 (s), 126.41 (t, *J* = 280.6 Hz), 109.49 (s), 62.51 (s), 57.72 (s), 51.68 (q, *J* = 27.6 Hz). **MS** (EI, m/z, %): 498 (M^+, 2.26), 249 (100.00), 250 (14.02), 216 (3.08). **HRMS** (EI): Calcd. For $C_{22}H_{24}O_6F_6$: 498.1477; Found: 498.1479. **IR** (film): 3009.9, 2967.2, 2843.1, 1954.8, 1596.3, 1508.6, 1464.8, 1427.9, 1256.1, 1185.7, 1119.3, 1001.1, 830.2, 716.4, 638.1, 529.1 cm^{-1}.

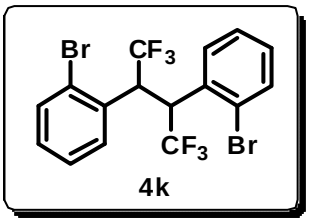
4i-*anti*: White solid. **MP**: 214–216 °C. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 6.56 (s, 4H), 3.90–3.78 (m, 20H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -63.38 (d, *J* = 7.2 Hz, 6F). **^{13}C**

NMR (101 MHz, $CDCl_3$): δ 154.97 (s), 140.12 (s), 129.68 (s), 128.61 (t, *J* = 273.7 Hz), 108.10 (s), 62.65 (s), 57.95 (s), 54.10–53.28 (m). **MS** (EI, m/z, %): 498 (M^+, 1.84), 249 (100.00), 250 (13.5), 228 (5.34), 277 (5.19). **HRMS** (EI): Calcd. For $C_{22}H_{24}O_6F_6$: 498.1477; Found: 498.1474. **IR** (film): 3010.6, 2963.9, 2944.4, 2844.2, 1593.0, 1512.9, 1469.5, 1426.9, 1325.8, 1249.2, 1182.0, 1131.8, 1093.5, 1001.4, 817.8, 712.0, 640.9 cm^{-1}.



3,3'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(bromobenzene) (4j)

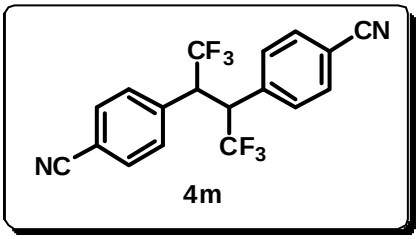
4j-*syn*: White solid. **MP**: 60–62 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.5 Hz, 2H), 7.09 (dd, *J* = 15.1, 7.0 Hz, 4H), 6.87 (d, *J* = 7.5 Hz, 2H), 4.04–3.95 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -65.86 (d, *J* = 8.9 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 133.09 (s), 132.68 (d, *J* = 15.2 Hz), 131.96 (s), 129.76 (s), 128.79 (s), 125.53 (q, *J* = 281.4 Hz), 122.26 (s), 49.69 (q, *J* = 28.2 Hz). **MS** (EI, m/z, %): 474 (M^+, 3.35), 476 (6.02), 478 (2.94), 237 (100.00), 239 (95.71), 187 (31.82), 189 (30.83). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6Br_2$: 473.9053; Found: 473.9054. **IR** (film): 2918.3, 2849.5, 1596.1, 1571.1, 1478.4, 1433.5, 1254.4, 1168.8, 1123.0, 936.9, 782.9, 708.3, 668.0, 610.9 cm^{-1}.
4j-*anti*: White solid. **MP**: 149-151 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.57–7.53 (m, 4H), 7.36–7.25 (m, 4H), 3.97–3.85 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.04 (d, *J* = 7.5 Hz, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 134.54–134.27 (m), 132.19 (s), 131.92 (s), 130.36 (s), 127.68 (s), 125.09 (q, *J* = 281.8 Hz), 122.79 (s), 51.28 (q, *J* = 26.5 Hz). **MS** (EI, m/z, %): 474 (M^+, 2.94), 476 (5.10), 478 (1.89), 237 (100.00), 239 (96.33), 187 (32.96), 189 (29.08). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6Br_2$: 473.9053; Found: 473.9048. **IR** (film): 2923.2, 1595.9, 1571.6, 1477.5, 1432.5, 1411.7, 1332.3, 1289.0, 1239.9, 1179.1, 1160.8, 1092.2, 788.5, 706.9, 678.5, 601.8, 498.3 cm^{-1}.



2,2'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)bis(bromobenzene) (4k)

Mixture of *syn* & *anti* (*syn*/*anti* = 1.03:1.00): White solid. **MP**: 61-63 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.68 (dd, *J* = 8.1, 1.0 Hz, 1.03×2H), 7.63 (d, *J* = 7.8 Hz, 1.03 ×2H), 7.43–7.39 (m, 4H), 7.34 (d, *J* = 7.5 Hz, 1.03×2H), 7.25 (td, *J* = 7.7, 1.5 Hz, 1.03×2H), 7.16 (td, *J* = 7.9, 1.1 Hz, 2H), 7.00 (td, *J* = 8.0, 1.5 Hz, 2H), 5.00–4.96 (m, 1.03×2H), 4.95–4.91 (m, 2H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -63.44 (d, *J* = 7.3 Hz,

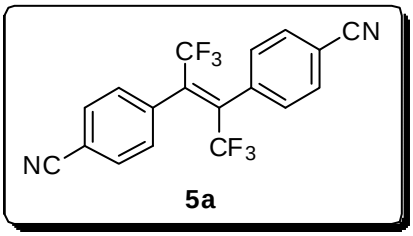
6F), -65.43 – -65.51 (m, 1.03×6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 133.45 (s), 132.98 (s), 132.07 (s), 131.94 (s), 130.29 (s), 129.91 (s), 129.60–129.46 (m), 129.22 (s), 127.74 (s), 127.53 (s), 126.17 (s), 125.71 (q, *J* = 281.0 Hz), 125.21 (q, *J* = 281.9 Hz), 48.89 (q, *J* = 27.1 Hz), 48.60–47.68 (m). **MS** (EI, m/z, %): 474 (M^+, 1.86), 476 (3.47), 478 (1.51), 237 (100.00), 239 (95.73), 158 (52.13). **HRMS** (EI): Calcd. For $C_{16}H_{10}F_6Br_2$: 473.9053; Found: 473.9049. **IR** (film): 3066.6, 2972.9, 1592.1, 1570.8, 1475.2, 1431.1, 1379.4, 1252.3, 1189.6, 1150.1, 1025.0, 951.7, 757.5, 732.3 cm^{-1}.



4m

4,4'-(1,1,1,4,4,4-hexafluorobutane-2,3-diyl)dibenzonitrile (4m)

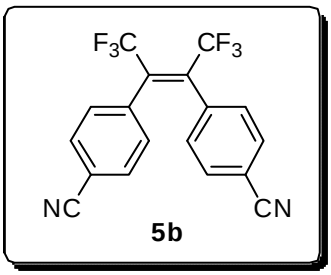
Mixture of *syn* & *anti* (*syn*/*anti* = 1.21:1.00, the ***syn*** configuration was determined via X-ray diffraction and was further confirmed by **^{19}F NMR**): White solid. **^{19}F NMR** (376 MHz, $CDCl_3$): δ -62.82 (d, *J* = 7.4 Hz, 6F), -65.58 (d, *J* = 8.7 Hz, 1.21×6F). **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.73 (d, *J* = 8.5 Hz, 4H), 7.50–7.54 (m, 2.21×4H), 7.04 (d, *J* = 8.3 Hz, 1.21×4H), 4.12 (dt, J = 9.4, 6.9 Hz, 1.21×2H), 4.03 (dt, *J* = 7.7, 5.2 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 136.98 (s), 135.49 (s), 132.73 (s), 132.22 (s), 130.75 (s), 129.77 (s), 125.18 (q, *J* = 281.2 Hz), 124.74 (q, *J* = 281.8 Hz), 117.89 (s), 117.65 (s), 113.48 (s), 113.35 (s), 51.56 (q, *J* = 27.4 Hz), 49.97 (q, *J* = 29.2 Hz). **MS** (ESI, m/z, %): 368 (M^+, 19.80), 134 (100.00), 184 (64.70). **HRMS** (EI): Calcd. For $C_{18}H_{10}N_2F_6$: 368.0748; Found: 368.0744. **IR** (film): 2972.2, 2924.9, 2234.6, 1611.5, 1508.4, 1425.5, 1348.2, 1298.0, 1254.9, 1165.1, 1112.6, 849.0, 693.4 cm^{-1}.



5a

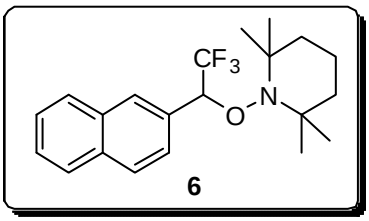
(E)-4,4'-(perfluorobut-2-ene-2,3-diyl)dibenzonitrile (5a)

White solid. **MP**: 257-259 °C. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.79 (d, *J* = 8.1 Hz, 4H), 7.51 (d, *J* = 8.1 Hz, 4H). **^{19}F NMR** (282 MHz, $CDCl_3$): δ -57.61 (s, 6F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 137.91–136.88 (m), 135.21 (s), 132.19 (s), 129.56 (s), 121.28 (q, *J* = 277.5 Hz), 117.83 (s), 113.96 (s). **MS** (EI, m/z, %): 366 (M^+, 60.32), 277 (100.00), 228 (87.9), 297 (85.11), 260 (49.20). **HRMS** (EI): Calcd. For $C_{18}H_8N_2F_6$: 366.0592; Found: 366.0596. **IR** (film): 3098.2, 3067.7, 2234.9, 1610.2, 1507.5, 1403.8, 1266.8, 1238.1, 1178.7, 1146.7, 888.7, 872.1, 825.5, 660.0, 559.7 479.3 cm^{-1}.



(Z)-4,4'-(perfluorobut-2-ene-2,3-diyl)dibenzonitrile (5b)

White solid. **MP**: 199-201 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.1 Hz, 4H), 7.14 (d, *J* = 8.1 Hz, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -57.80 (s). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 138.81–137.76 (m), 136.40 (s), 132.22 (s), 129.73 (s), 120.80 (q, *J* = 277.3 Hz), 117.46 (s), 113.47 (s). **MS** (EI, m/z, %): 366 (M^+, 58.78), 277 (100.00), 228 (84.94), 297 (83.66), 278 (20.06), 148 (18.91). **HRMS** (EI): Calcd. For $C_{18}H_8N_2F_6$: 366.0592; Found: 366.0588. **IR** (film): 2229.2, 1654.7, 1608.5, 1501.6, 1404.9, 1323.2, 1228.8, 1165.1, 1135.6, 847.9, 820.3, 676.9 cm^{-1}.



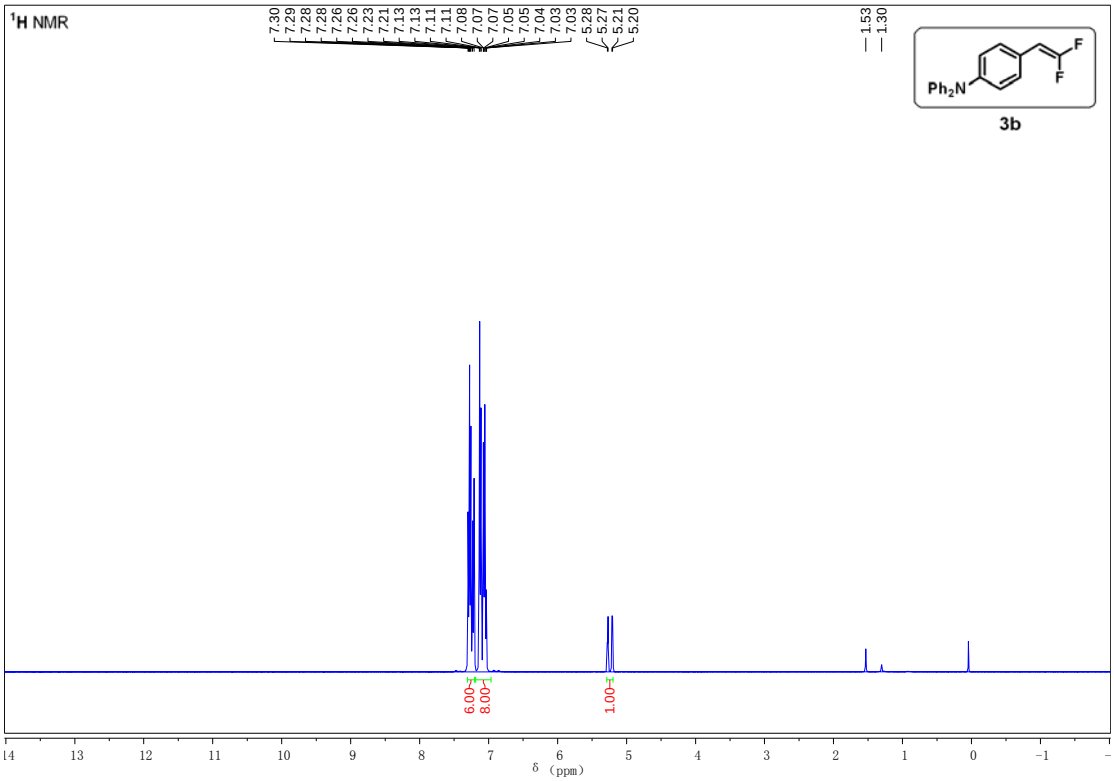
2,2,6,6-tetramethyl-1-(2,2,2-trifluoro-1-(naphthalen-2-yl)ethoxy)piperidine (6)

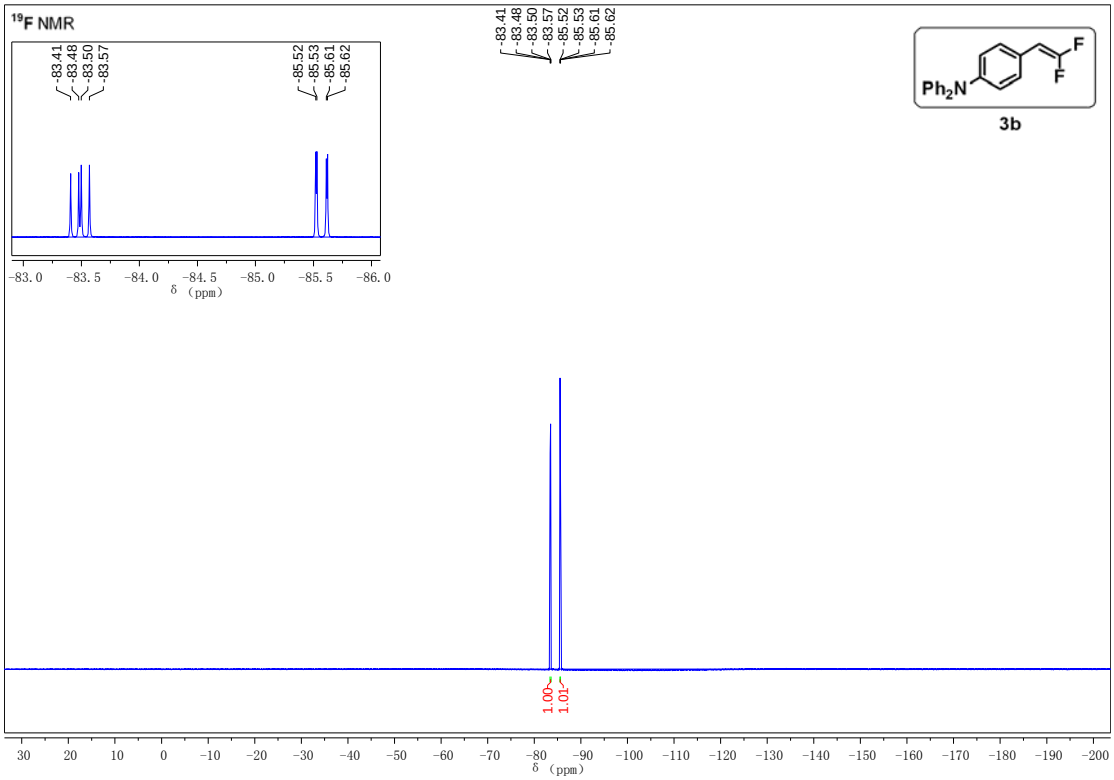
White solid. **MP**: 83–85 °C. **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.91–7.81 (m, 4H), 7.58 (d, *J* = 8.6 Hz, 1H), 7.51 (dd, *J* = 6.0, 3.4 Hz, 2H), 5.34–5.20 (m, 1H), 1.51–1.04 (m, 15H), 0.53 (s, 3H). **^{19}F NMR** (376 MHz, $CDCl_3$): δ -71.36 (d, *J* = 6.8 Hz, 3F). **^{13}C NMR** (101 MHz, $CDCl_3$): δ 133.49 (s), 132.73 (s), 132.26 (s), 129.12 (s), 128.20 (s), 127.88 (s), 127.65 (s), 126.53 (s), 126.20 (s), 126.09 (s), 125.79 (s), 122.96 (s), 84.89 (q, *J* = 28.6 Hz), 40.71 (s), 33.99 (s), 33.30 (s), 20.23 (s), 16.90 (s). **MS** (ESI, m/z, %): 366 ($M+H^+$, 100.00). **HRMS** (ESI): Calcd. For $C_{21}H_{27}F_3NO^+$: 366.20393; Found: 366.20502. **IR** (film): 3061.5, 3008.1, 2932.9, 1603.1, 1510.1, 1464.7, 1364.4, 1253.2, 1167.9, 1129.5, 1059.1, 1016.8, 878.7, 818.0, 746.8, 478.3 cm^{-1}.

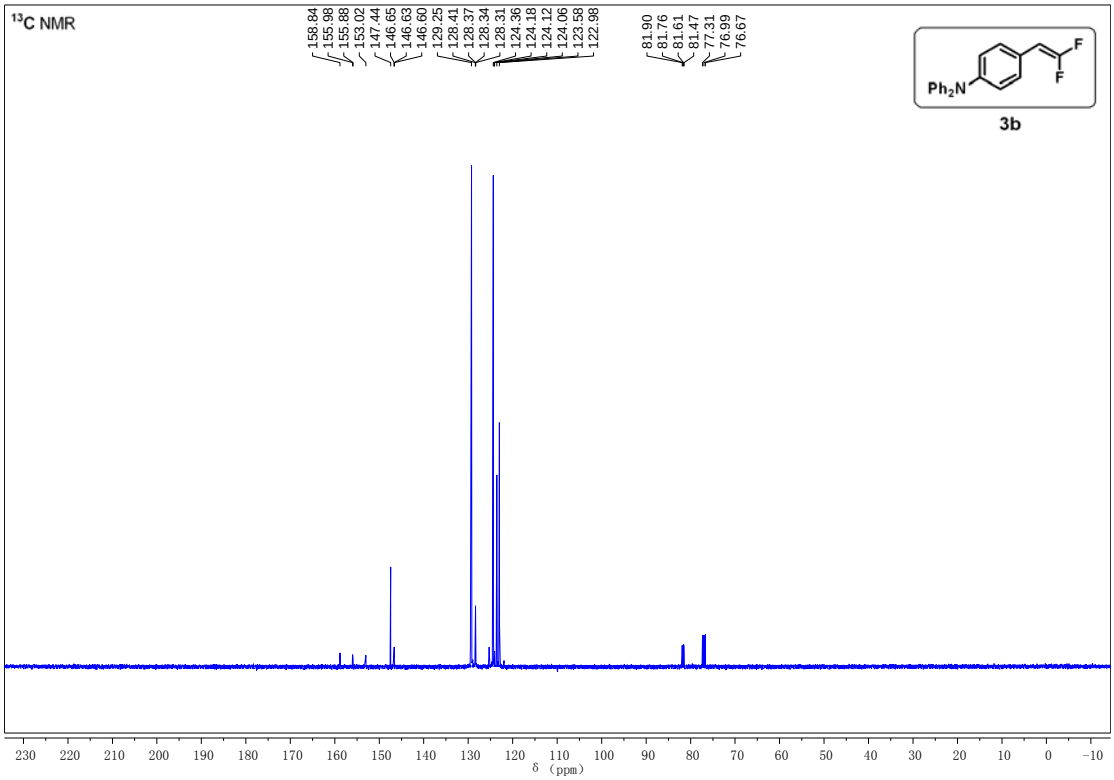
6. References:

1. Zhao, Y.-C.; Huang, W.-Z.; Zhu, L.; Hu, J.-B. *Org. Lett.* **2010**, *12*, 1444.
2. Nenajdenko, V. G.; Varseev, G. N.; Korotchenko, V. N.; Shastin, A. V.; Balenkova, E. S. *J. Fluorine Chem.* **2003**, *124*, 115.
3. Fujita, T.; Ichitsuka, T.; Fuchibe, K.; Ichikawa, J. *Chem. Lett.* **2011**, *40*, 986.
4. Bhadury, P. S.; Palit, M.; Sharma, M.; Raza, S. K.; Jaiswal, D. K. *J. Fluorine Chem.* **2002**, *116*, 75.
5. Gøgsig, T. M.; Søbjerg, L. S.; Lindhardt, A. T.; Jensen, K. L.; Skrydstrup, T. *J. Org. Chem.* **2008**, *73*, 3404.

7. Copies of ^{19}F, ^{1}H and ^{13}C NMR Spectra:



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128.31
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124.18
124.12
124.06
123.58
122.98
81.90
81.76
81.61
81.47
77.31
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Ph2N
F
F
3b
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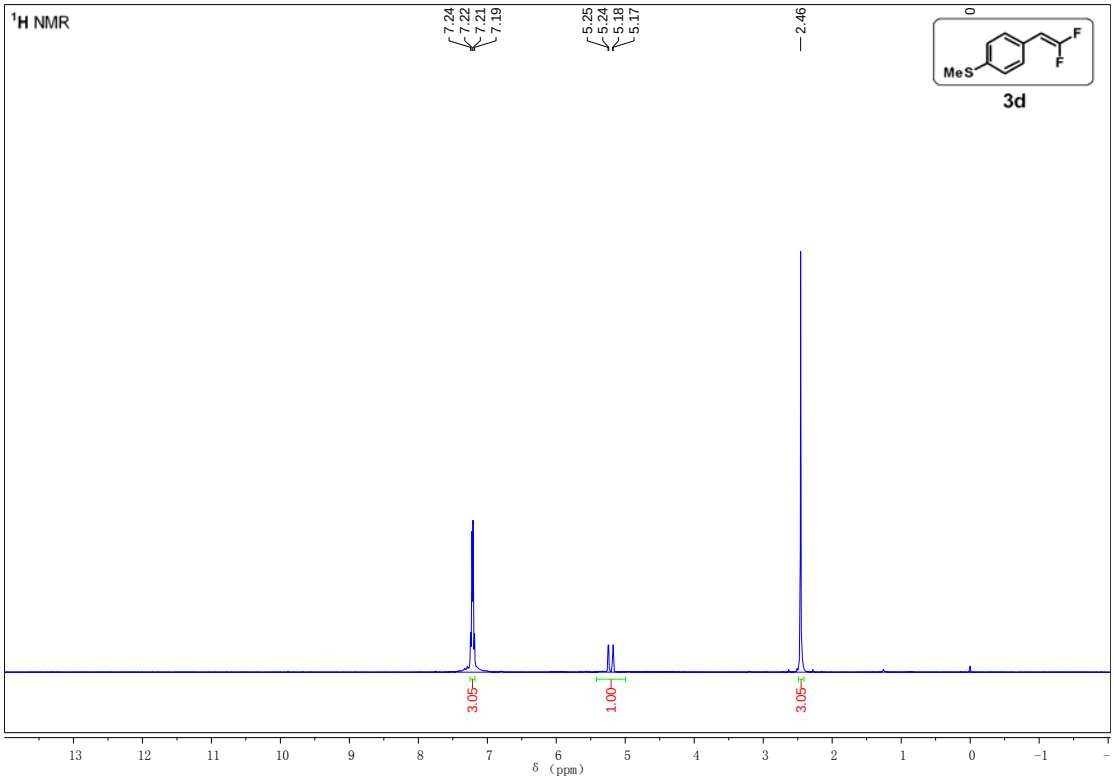
δ (ppm)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F
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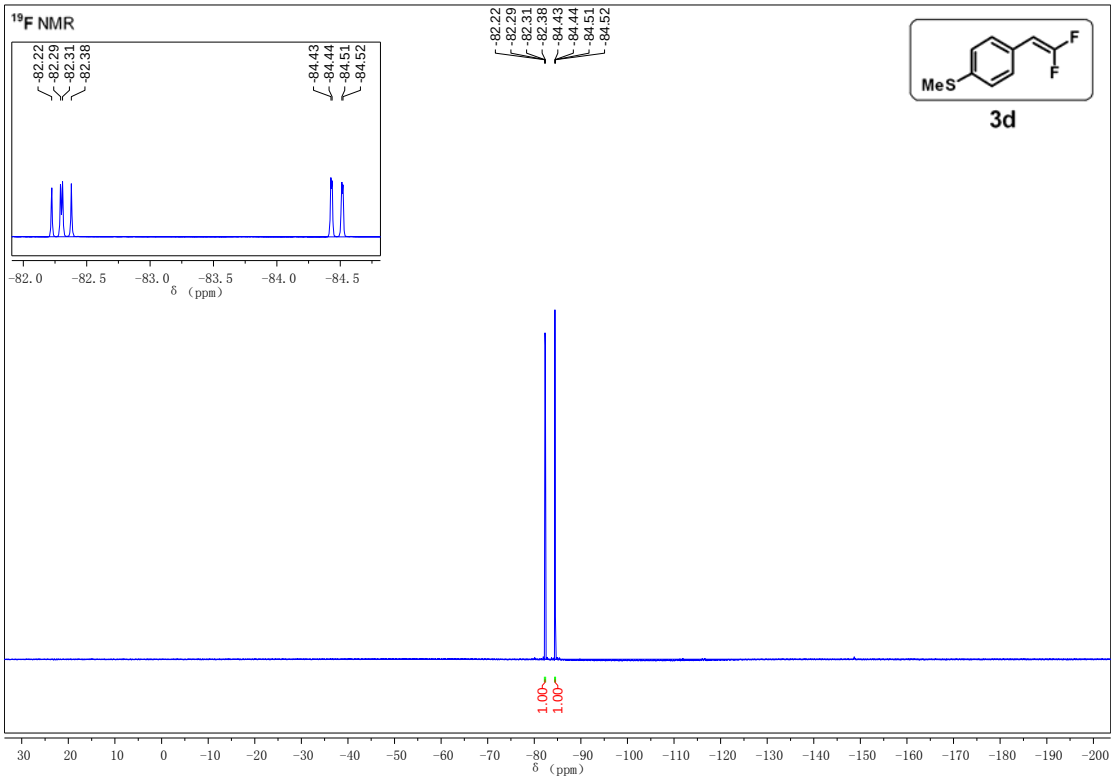
3.05
13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1
δ (ppm)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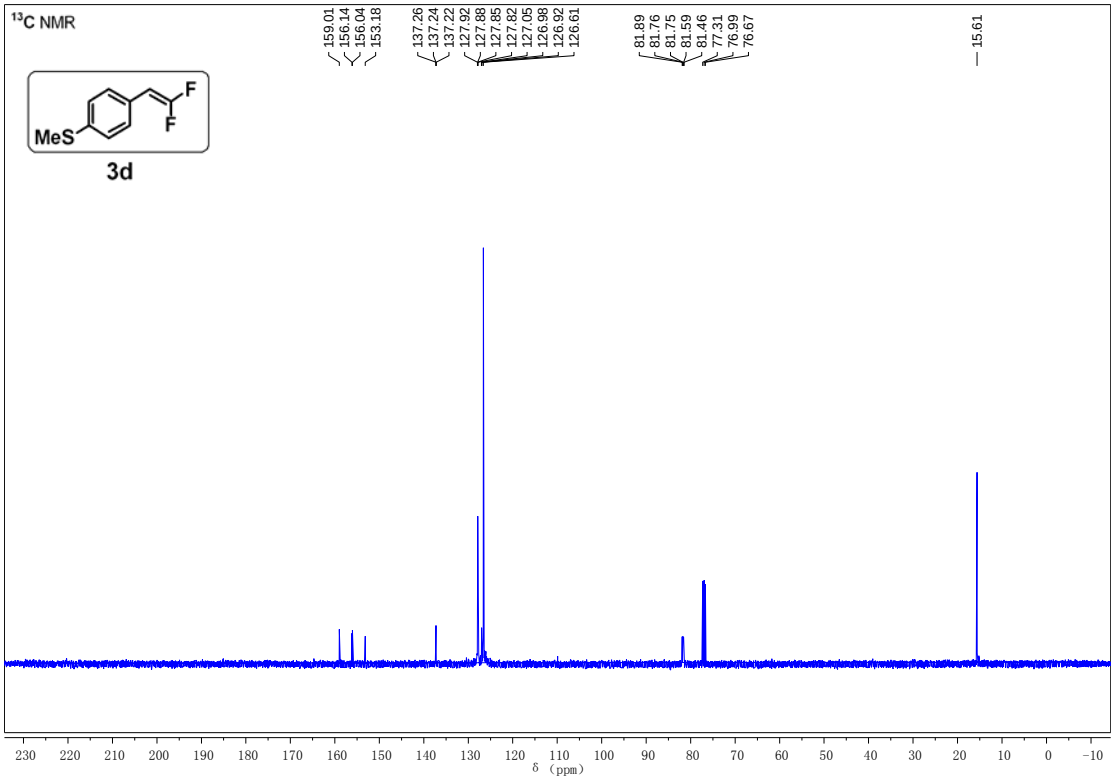
δ (ppm)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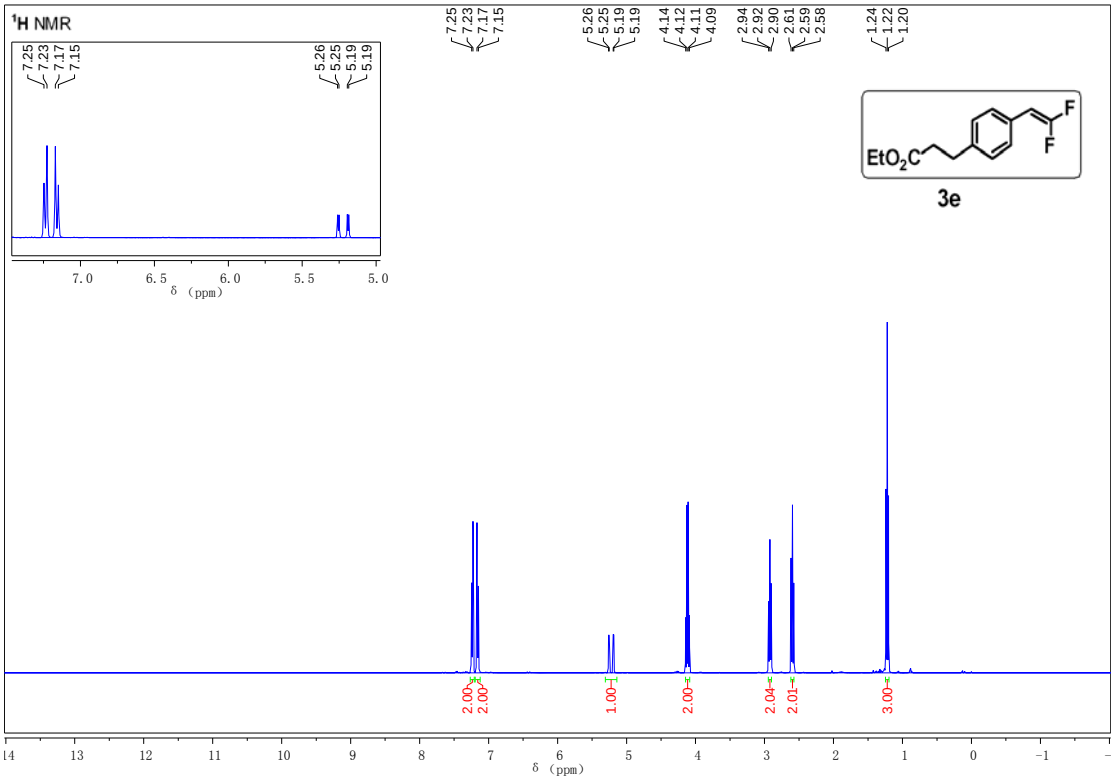
δ (ppm)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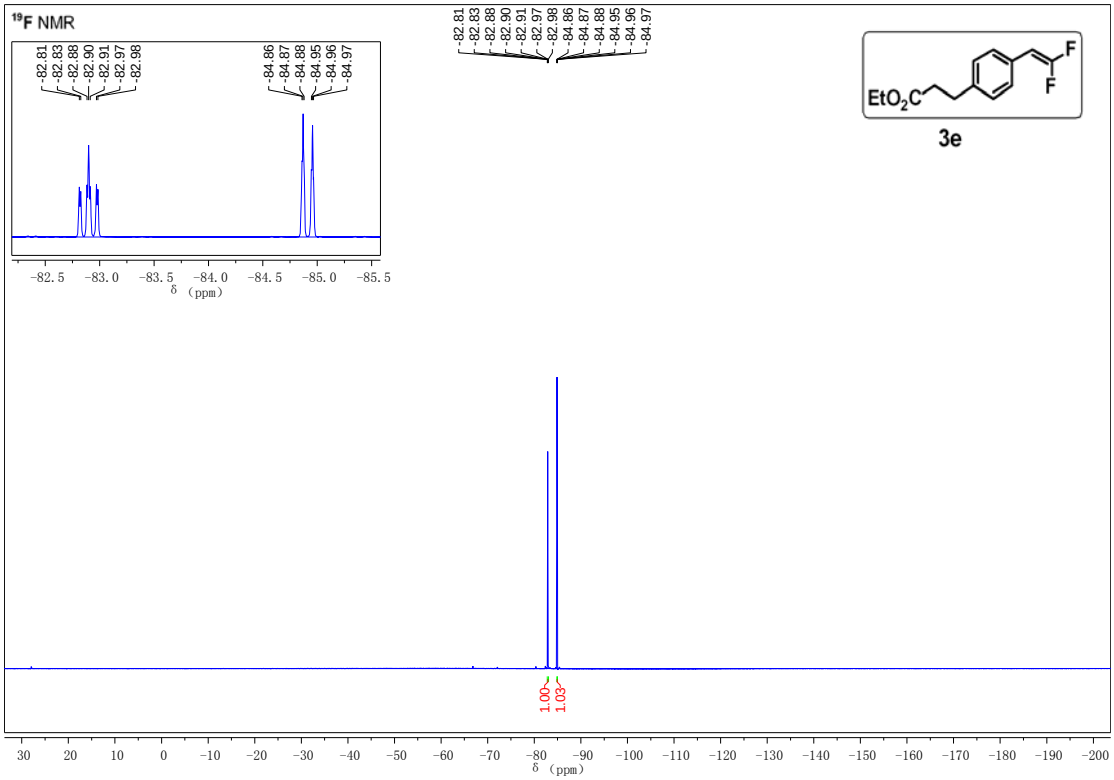
δ (ppm)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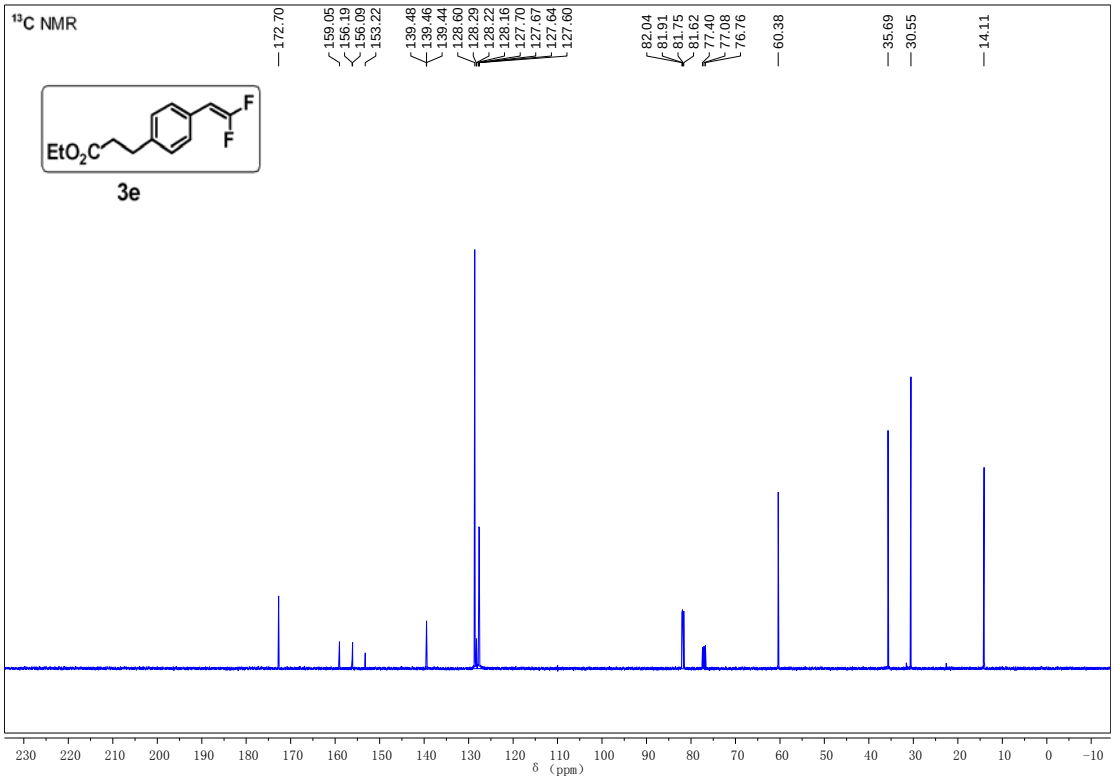
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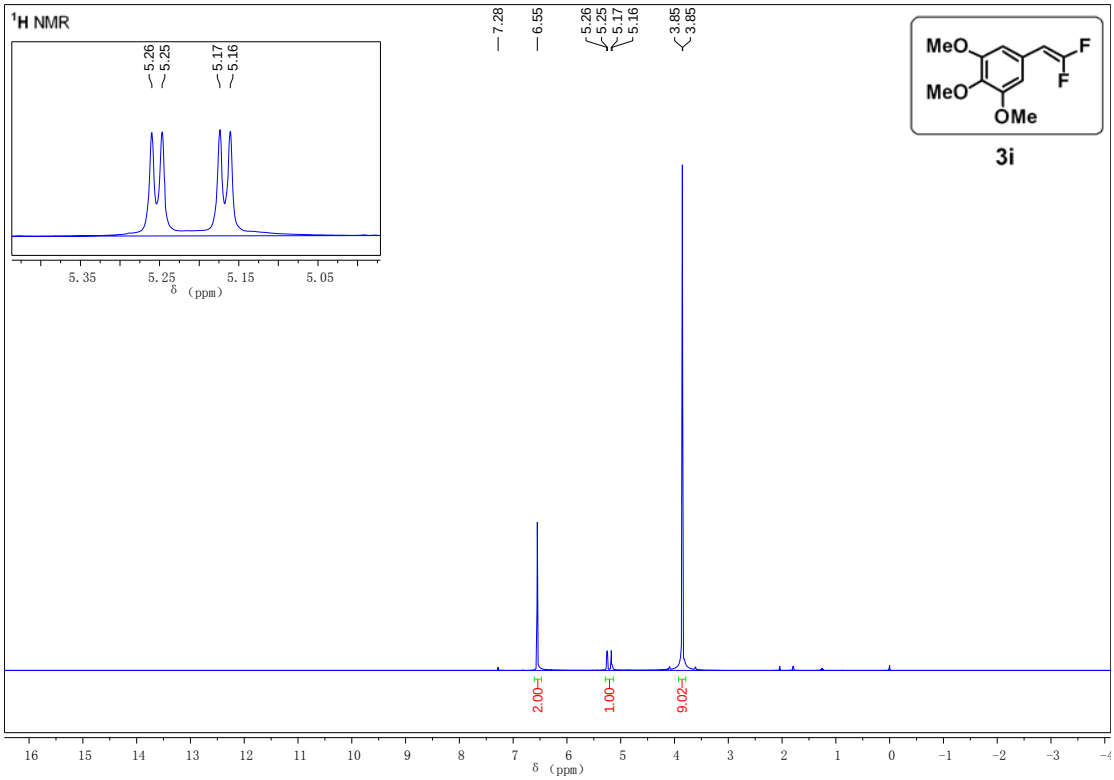
δ (ppm)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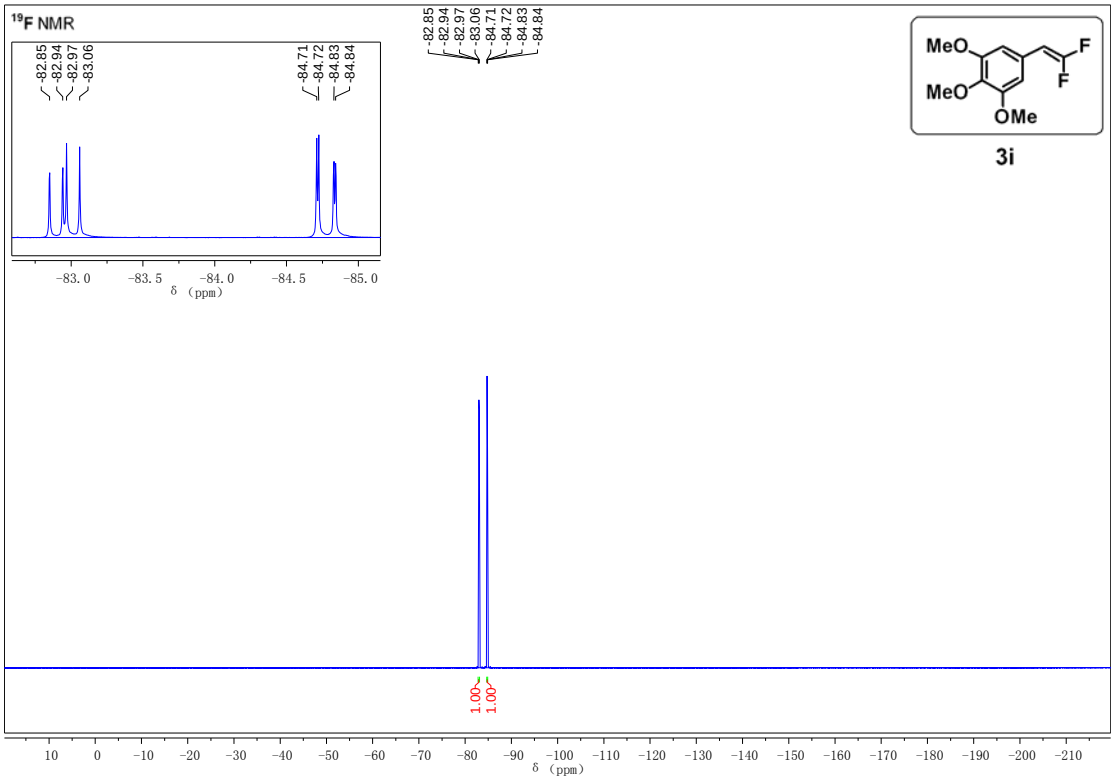
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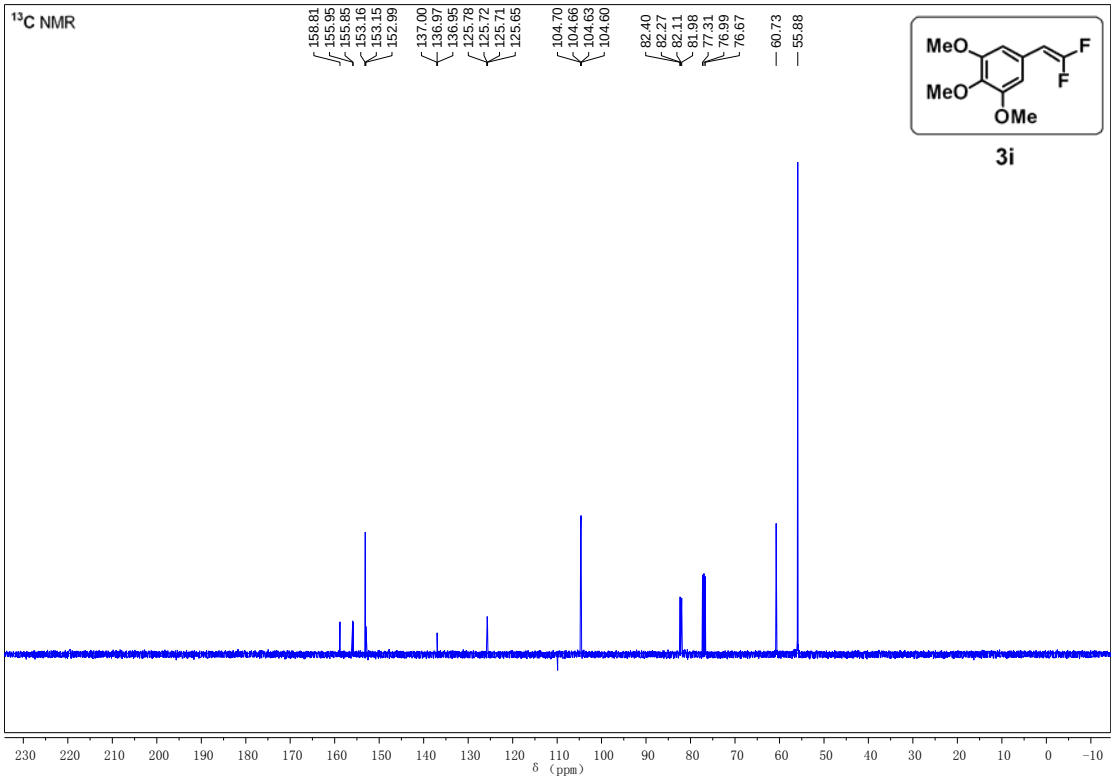
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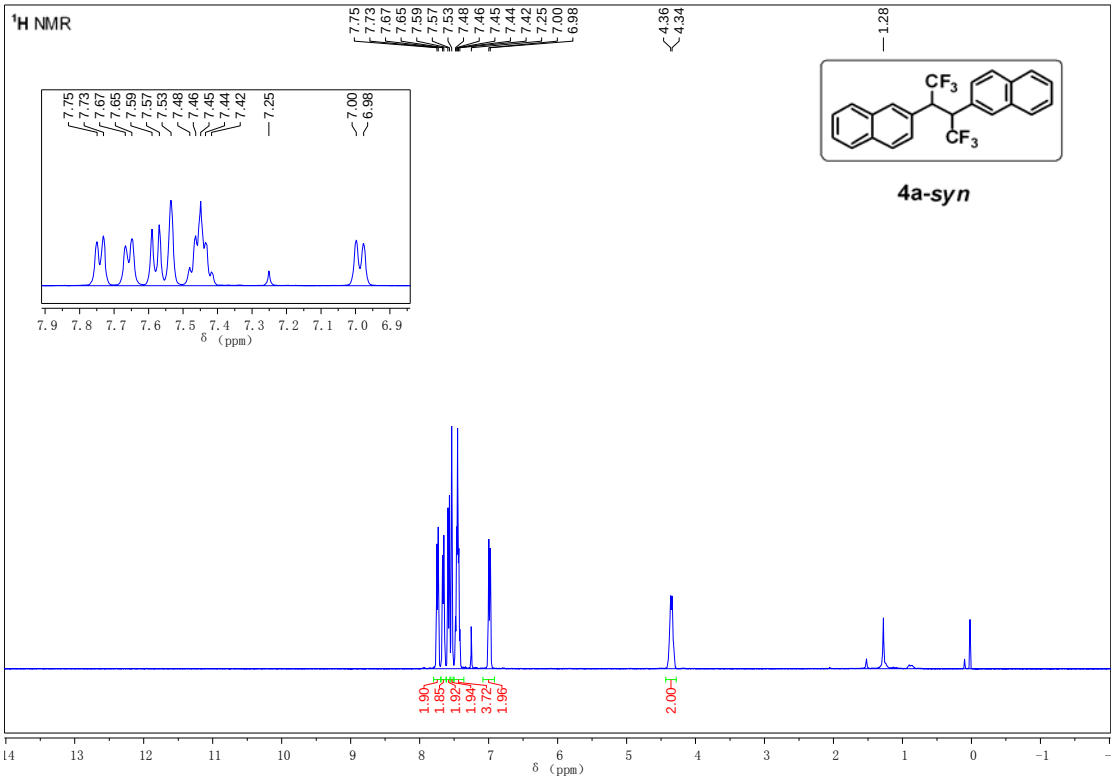
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7.25
7.00
6.98
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4.34
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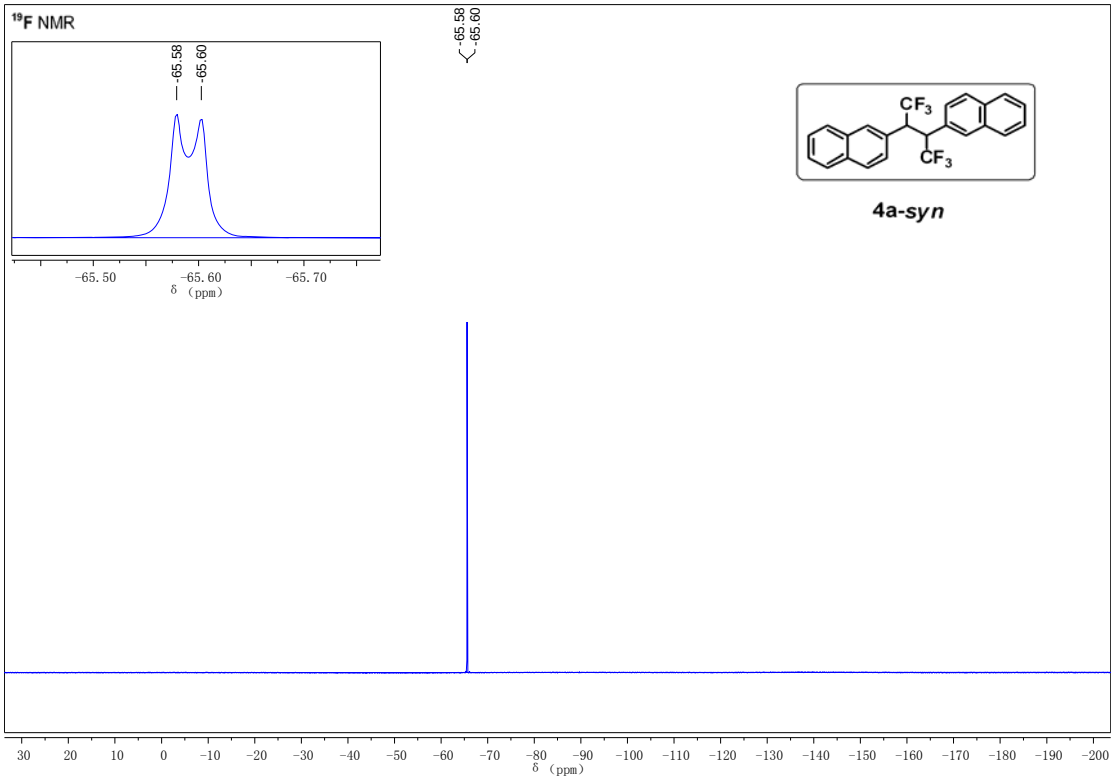
2.00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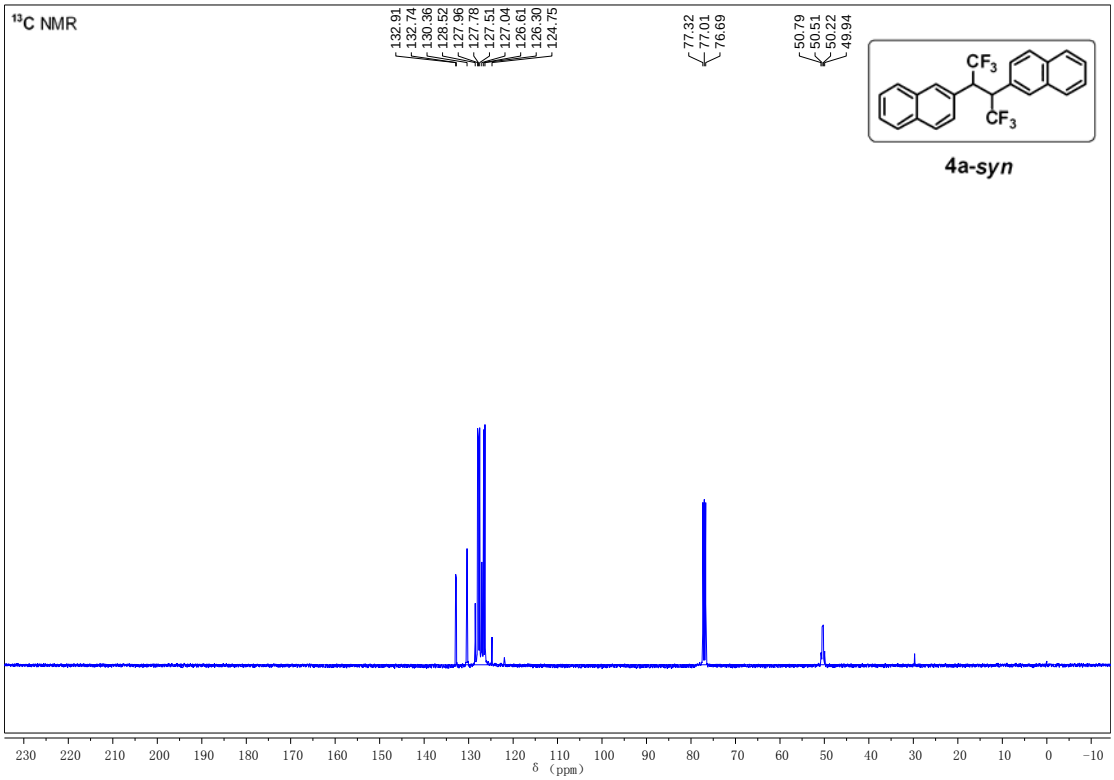
δ (ppm)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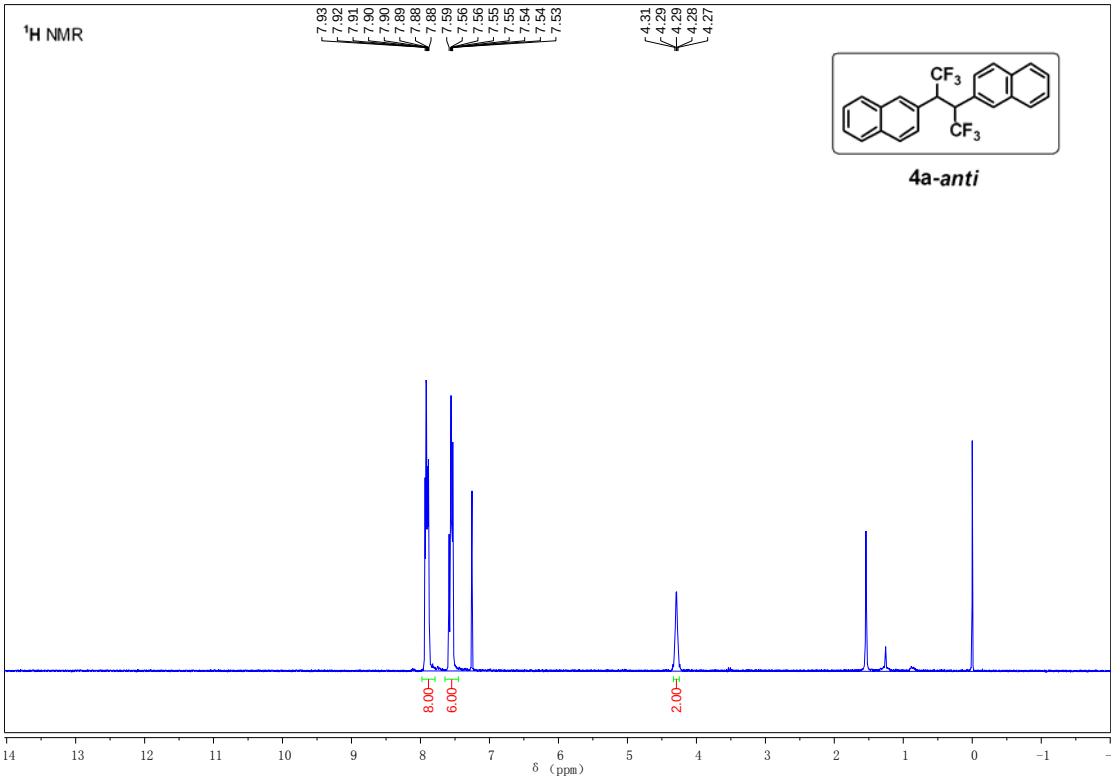
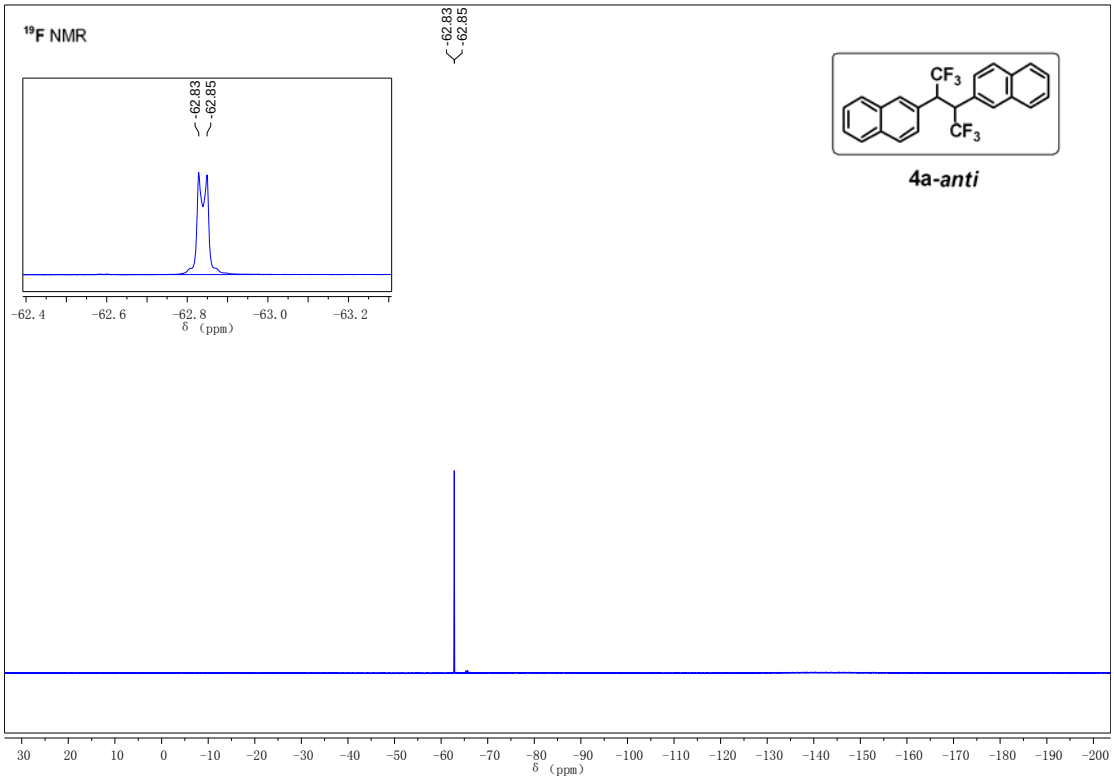
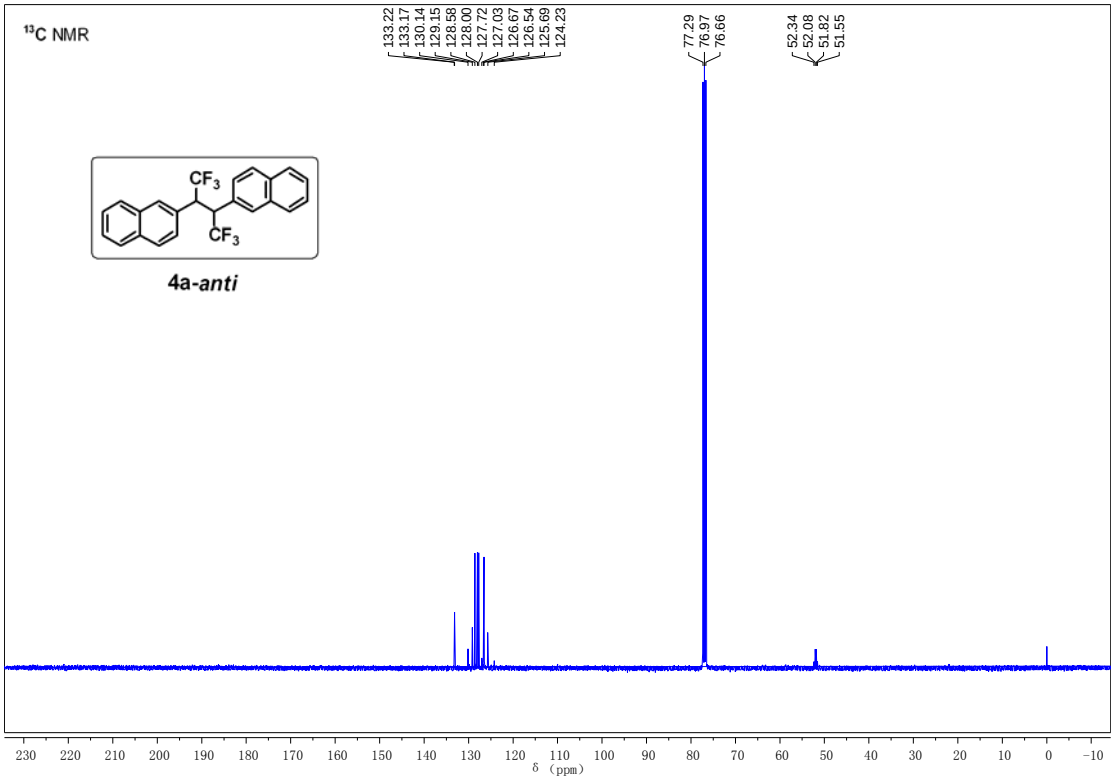
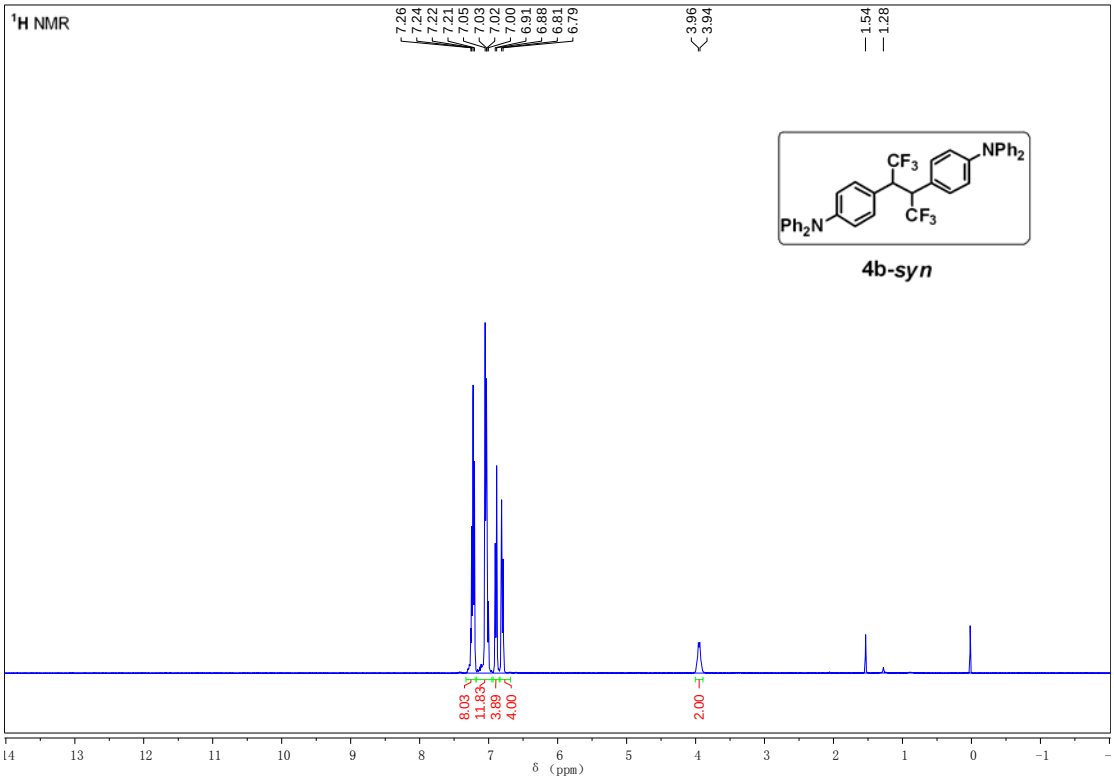
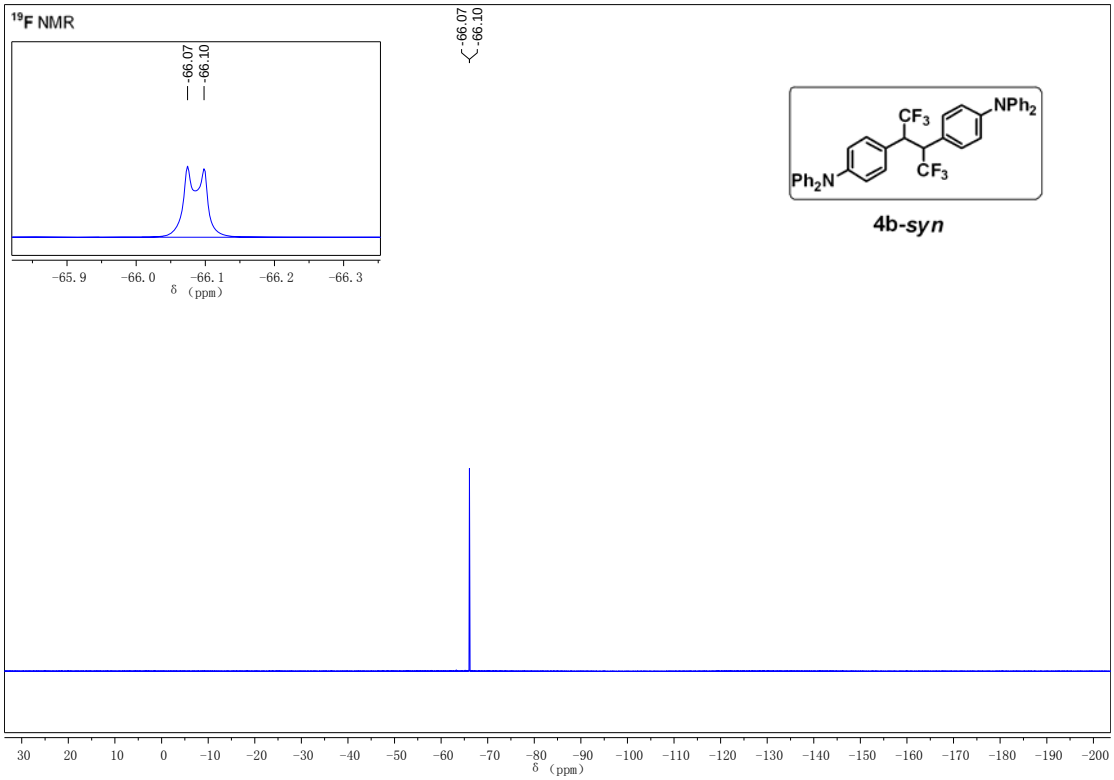
CF3
CF3
4a-syn
δ (ppm)

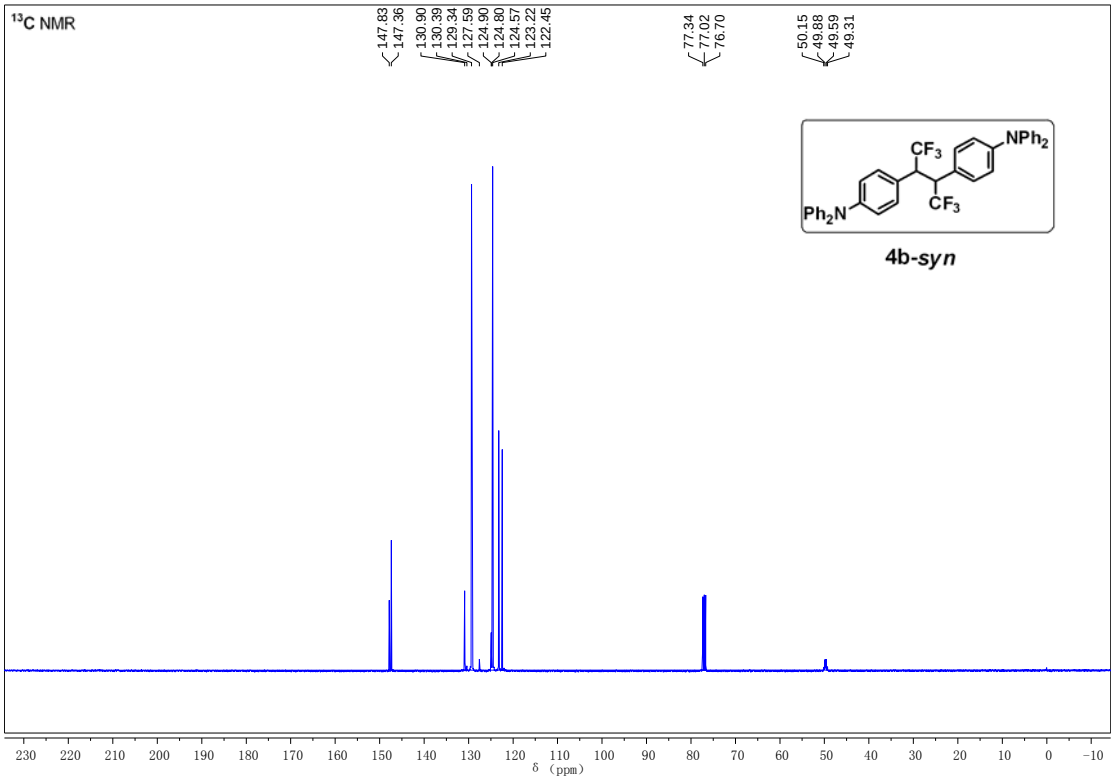

1H NMR
7.93
7.92
7.91
7.90
7.90
7.89
7.88
7.88
7.59
7.56
7.56
7.55
7.55
7.54
7.54
7.53
4.31
4.29
4.29
4.28
4.27
CF3
CF3
4a-anti
8.00
6.00
2.00
δ (ppm)

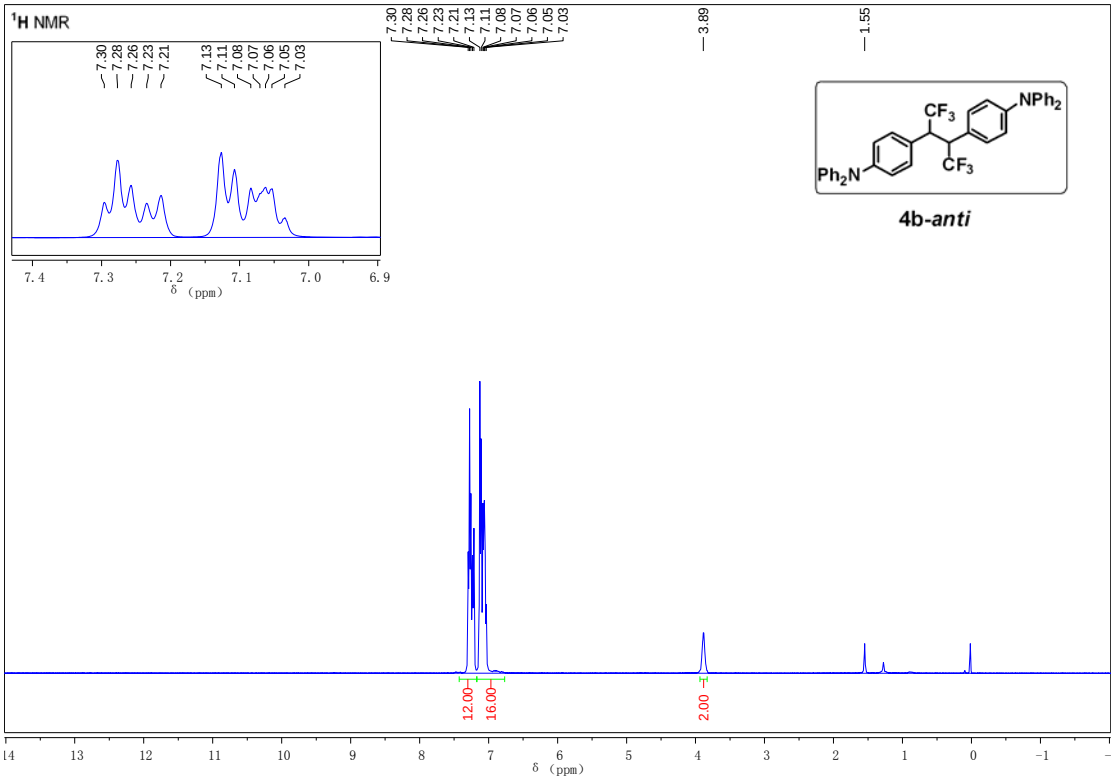

19F NMR
-62.83
-62.85
CF3
CF3
4a-anti
δ (ppm)

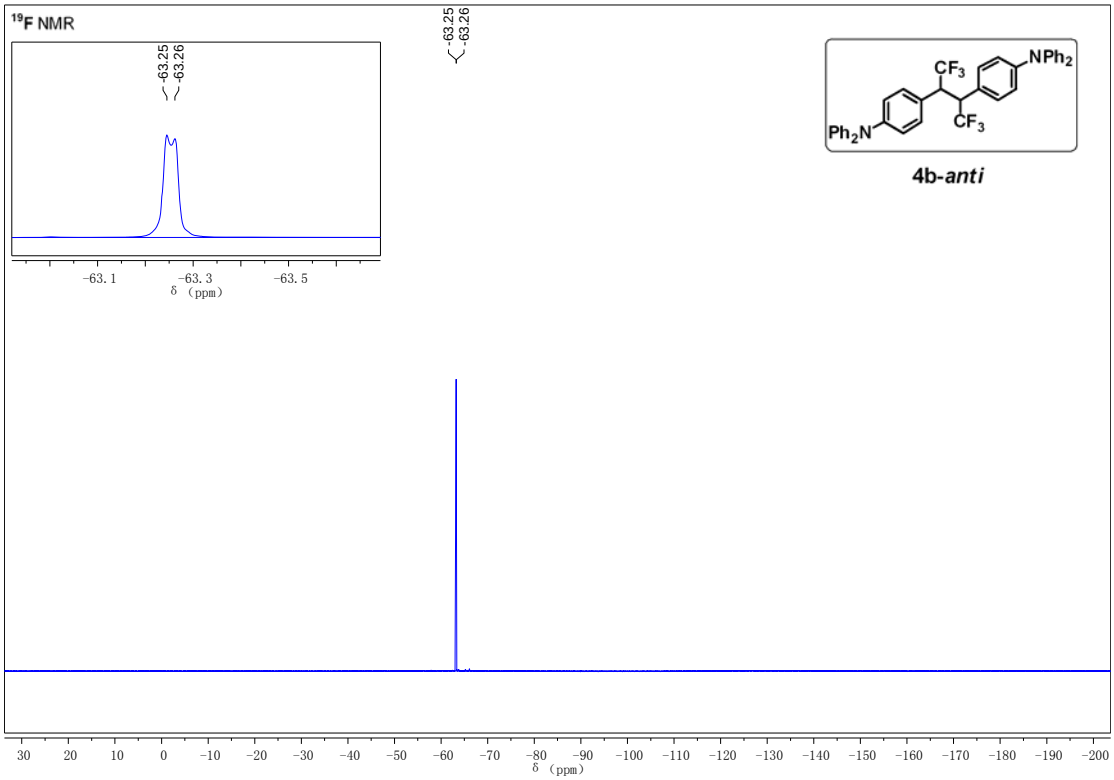

13C NMR
133.22
133.17
130.14
129.15
128.58
128.00
127.72
127.03
126.67
126.54
125.69
124.23
77.29
76.97
76.66
52.34
52.08
51.82
51.55
CF3
CF3
4a-anti
δ (ppm)

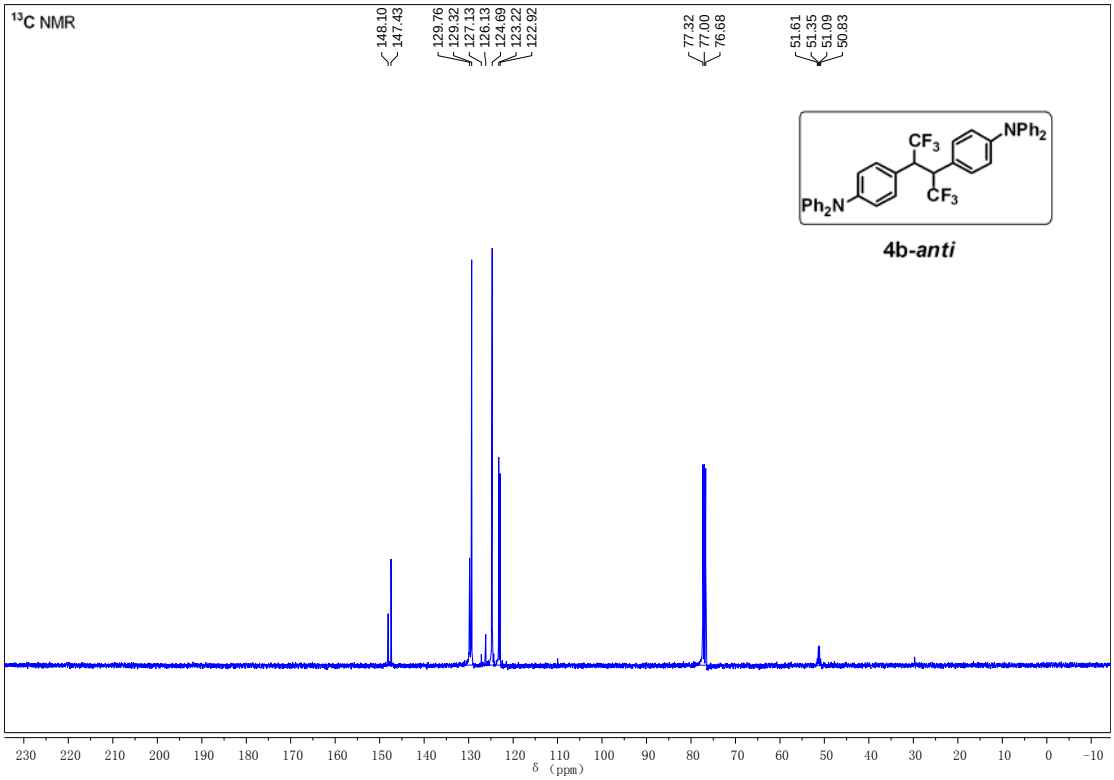

1H NMR
7.26
7.24
7.22
7.21
7.05
7.03
7.02
7.00
6.91
6.88
6.81
6.79
3.96
3.94
1.54
1.28
CF3
NPh2
Ph2N
CF3
4b-syn
8.03
11.83
3.89
4.00
2.00
δ (ppm)

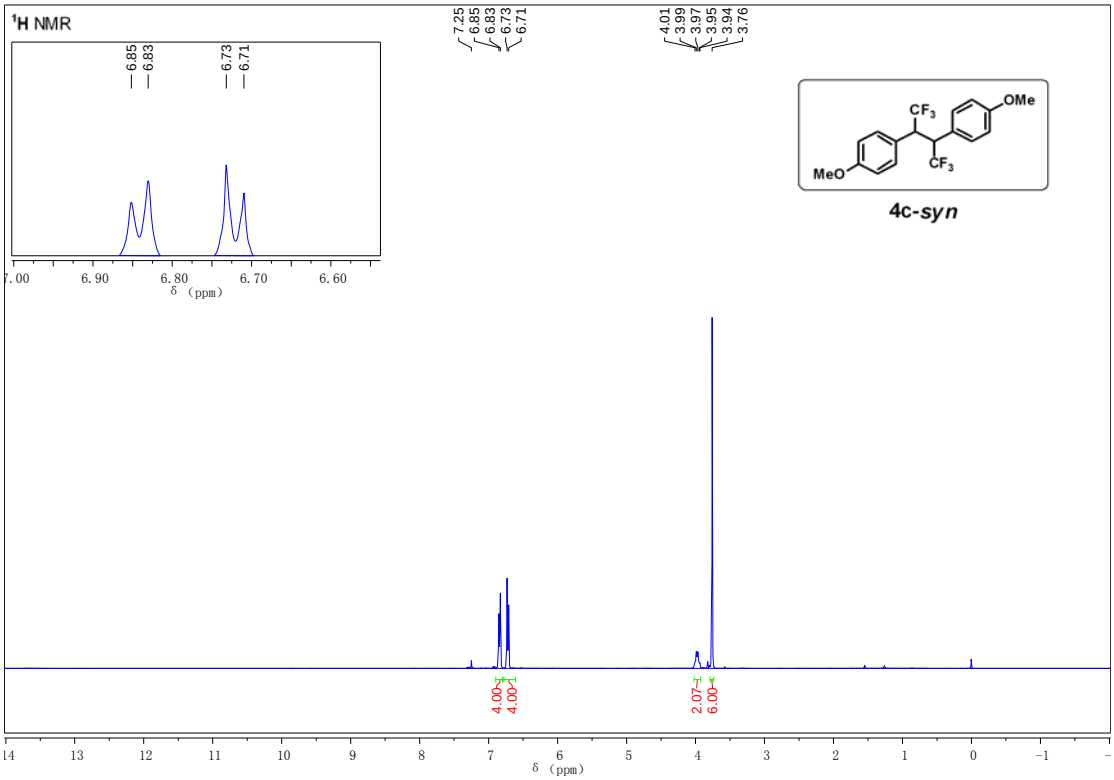

19F NMR
-66.07
-66.10
-66.07
-66.10
CF3
NPh2
Ph2N
CF3
4b-syn
δ (ppm)

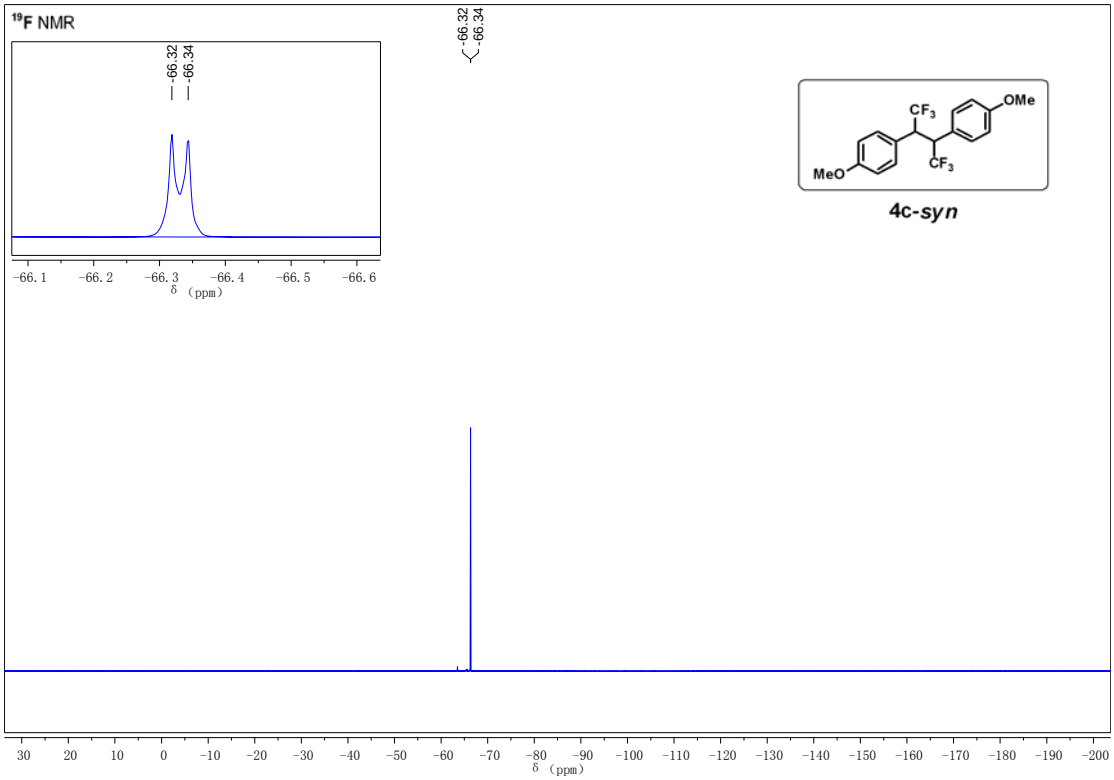

13C NMR
147.83
147.36
130.90
130.39
129.34
127.59
124.90
124.80
124.57
123.22
122.45
77.34
77.02
76.70
50.15
49.88
49.59
49.31
CF3
NPh2
Ph2N
CF3
4b-syn
δ (ppm)

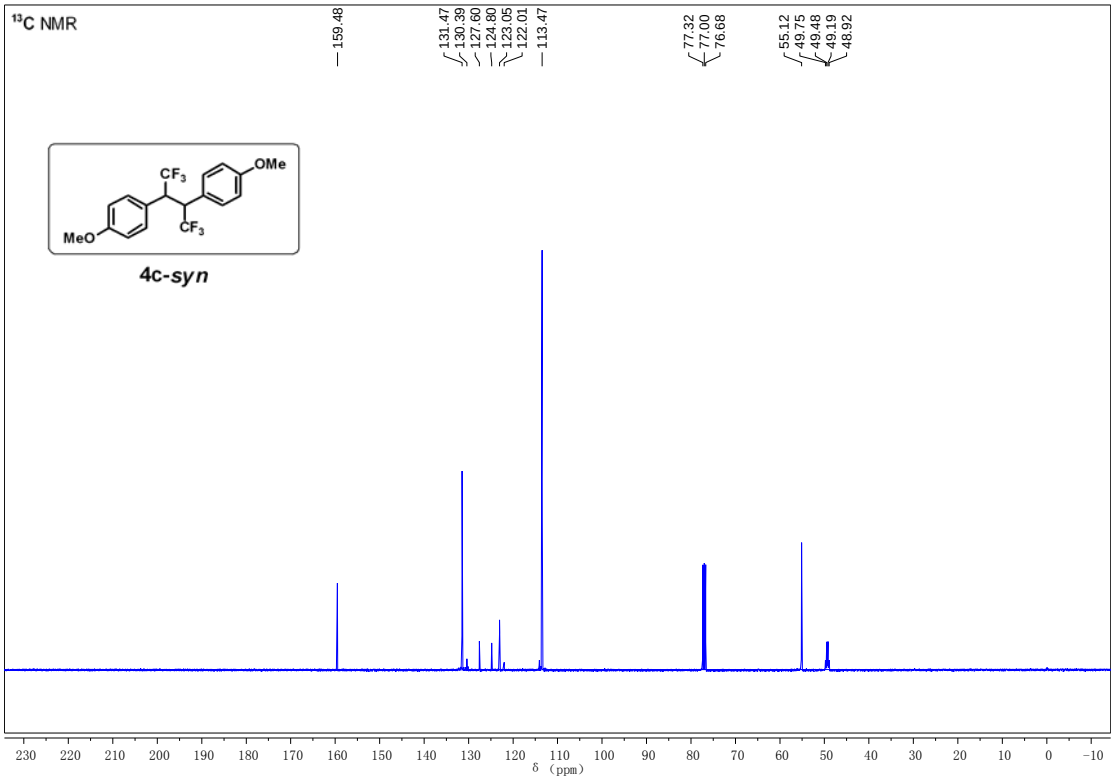

1H NMR
7.30
7.28
7.26
7.23
7.21
7.13
7.11
7.08
7.07
7.06
7.05
7.03
3.89
1.55
CF3
NPh2
Ph2N
CF3
4b-anti
12.00
16.00
2.00
δ (ppm)

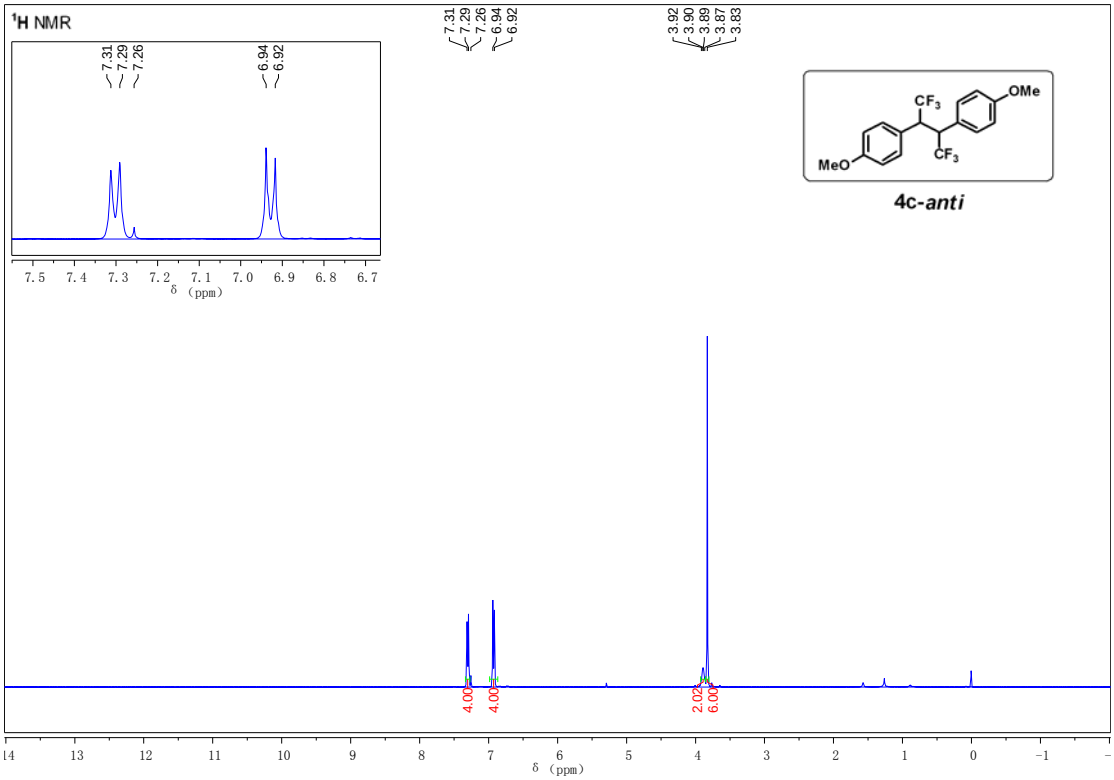

19F NMR
-63.25
-63.26
-63.1
-63.3
-63.5
δ (ppm)
CF3
NPh2
Ph2N
CF3
4b-anti
30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200
δ (ppm)

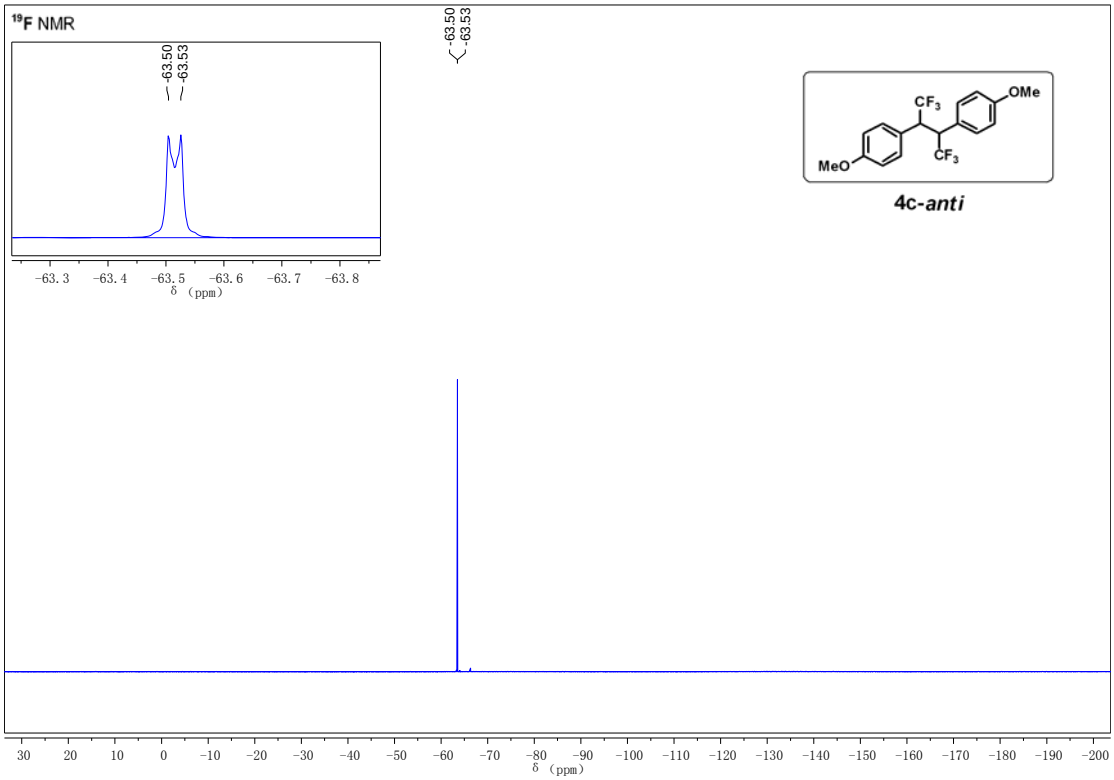

13C NMR
148.10
147.43
129.76
129.32
127.13
126.13
124.69
123.22
122.92
77.32
77.00
76.68
51.61
51.35
51.09
50.83
CF3
NPh2
Ph2N
CF3
4b-anti
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
δ (ppm)

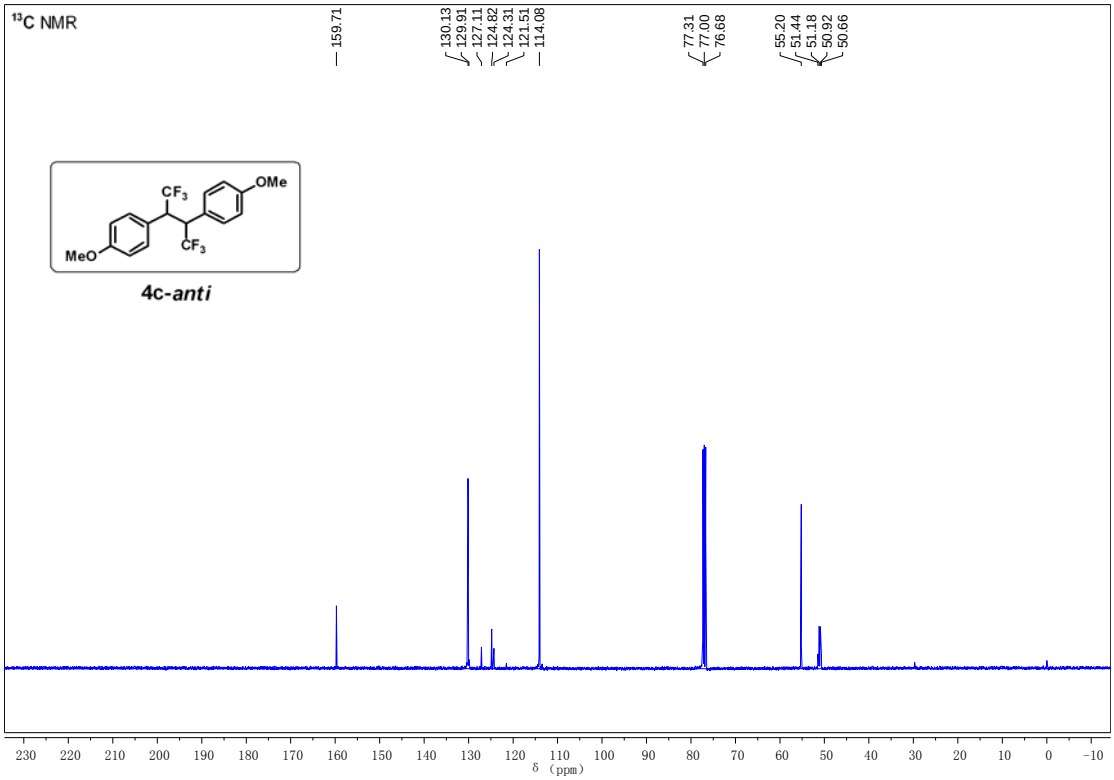

1H NMR
7.25
6.85
6.83
6.73
6.71
4.01
3.99
3.97
3.95
3.94
3.76
6.85
6.83
6.73
6.71
δ (ppm)
4.00
4.00
2.07
6.00
δ (ppm)
CF3
OMe
MeO
CF3
4c-syn

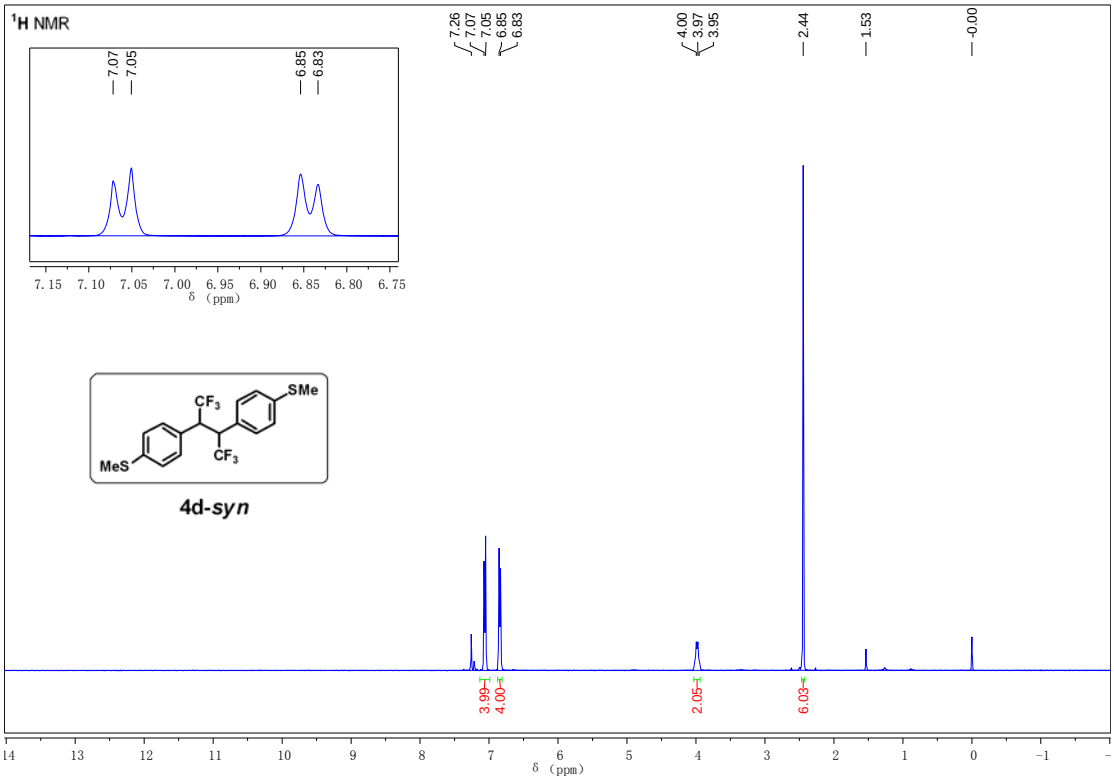

19F NMR
-66.32
-66.34
-66.32
-66.34
δ (ppm)
δ (ppm)
CF3
OMe
MeO
CF3
4c-syn

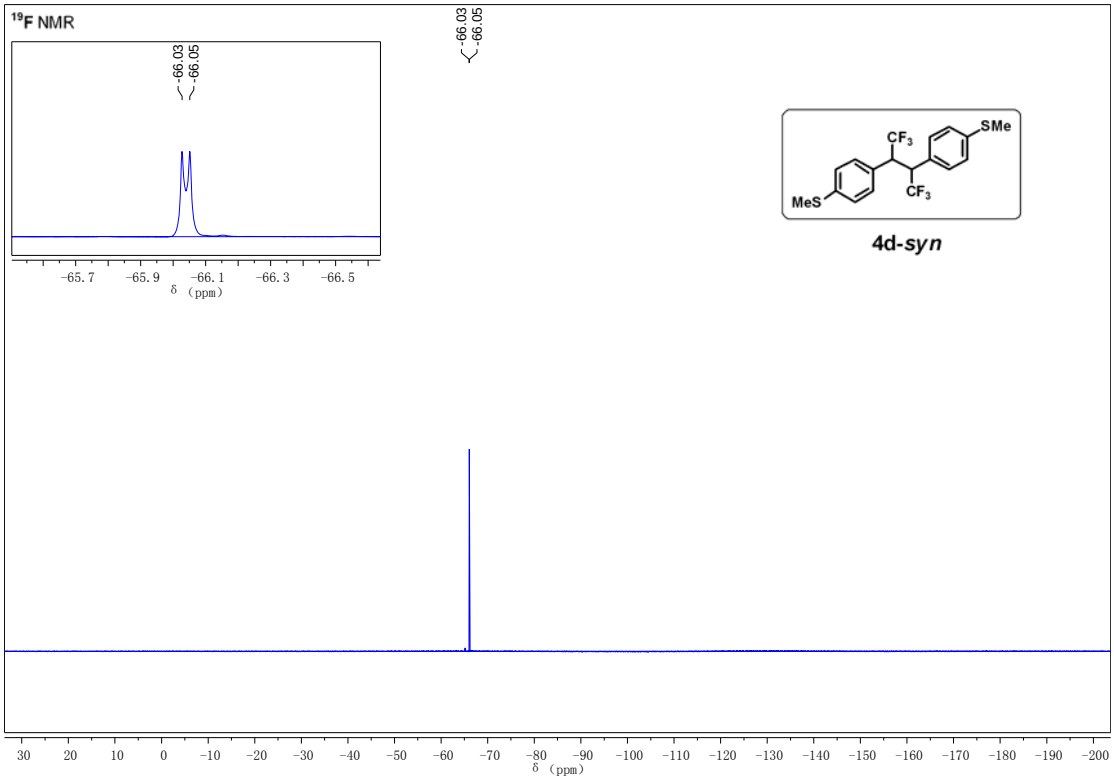

13C NMR
159.48
131.47
130.39
127.60
124.80
123.05
122.01
113.47
77.32
77.00
76.68
55.12
49.75
49.48
49.19
48.92
CF3
OMe
MeO
CF3
4c-syn
δ (ppm)

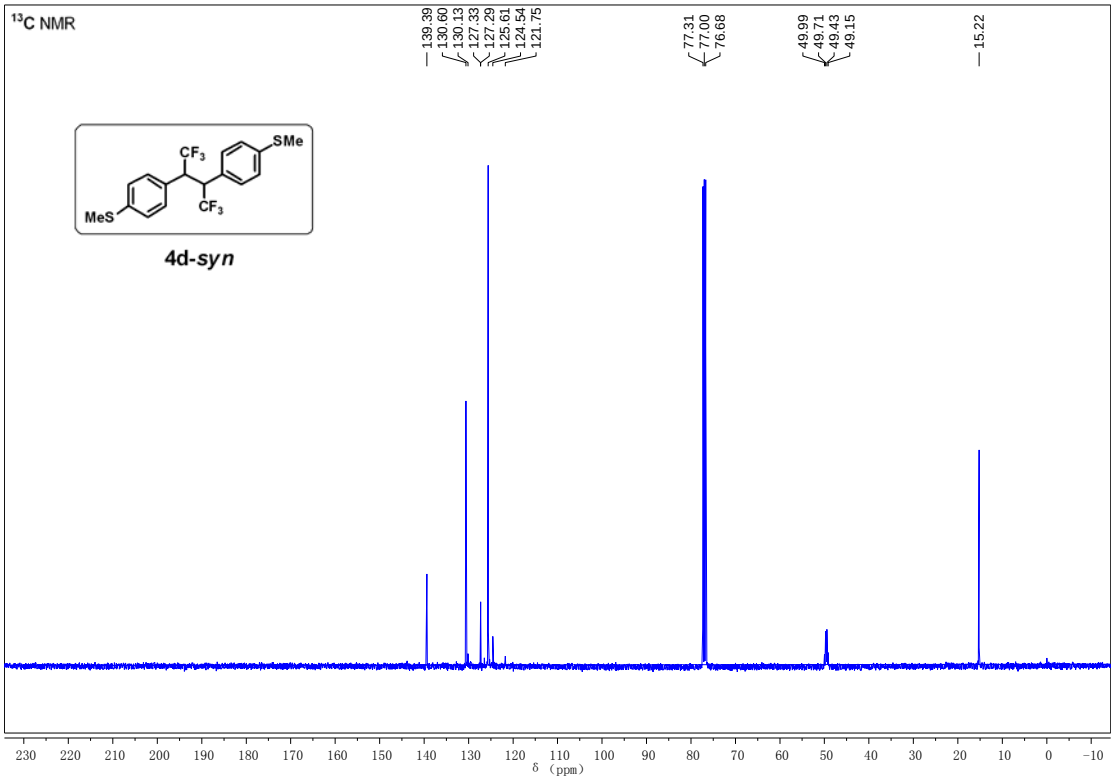

1H NMR
7.31
7.29
7.26
6.94
6.92
3.92
3.90
3.89
3.87
3.83
CF3
OMe
MeO
CF3
4c-anti
4.00
4.00
2.02
6.00
δ (ppm)

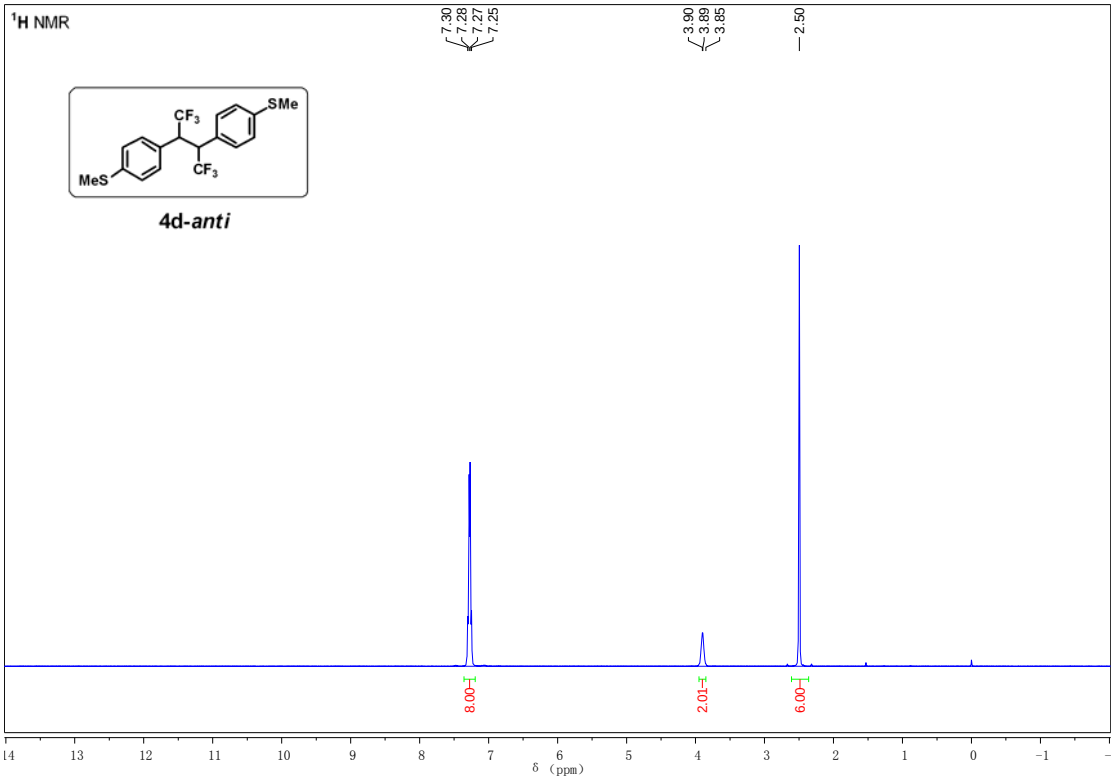

19F NMR
-63.50
-63.53
-63.3 -63.4 -63.5 -63.6 -63.7 -63.8
δ (ppm)
CF3
OMe
MeO
CF3
4c-anti
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
δ (ppm)

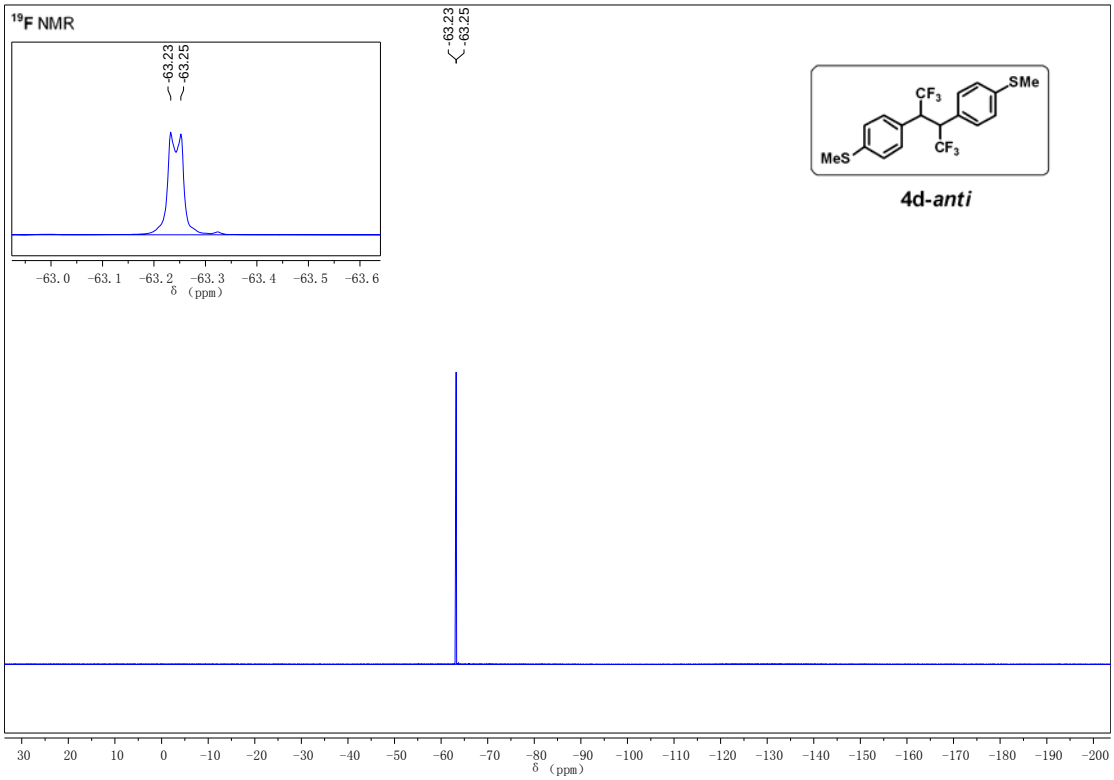

13C NMR
159.71
130.13
129.91
127.11
124.82
124.31
121.51
114.08
77.31
77.00
76.68
55.20
51.44
51.18
50.92
50.66
CF3
OMe
MeO
CF3
4c-anti
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
δ (ppm)

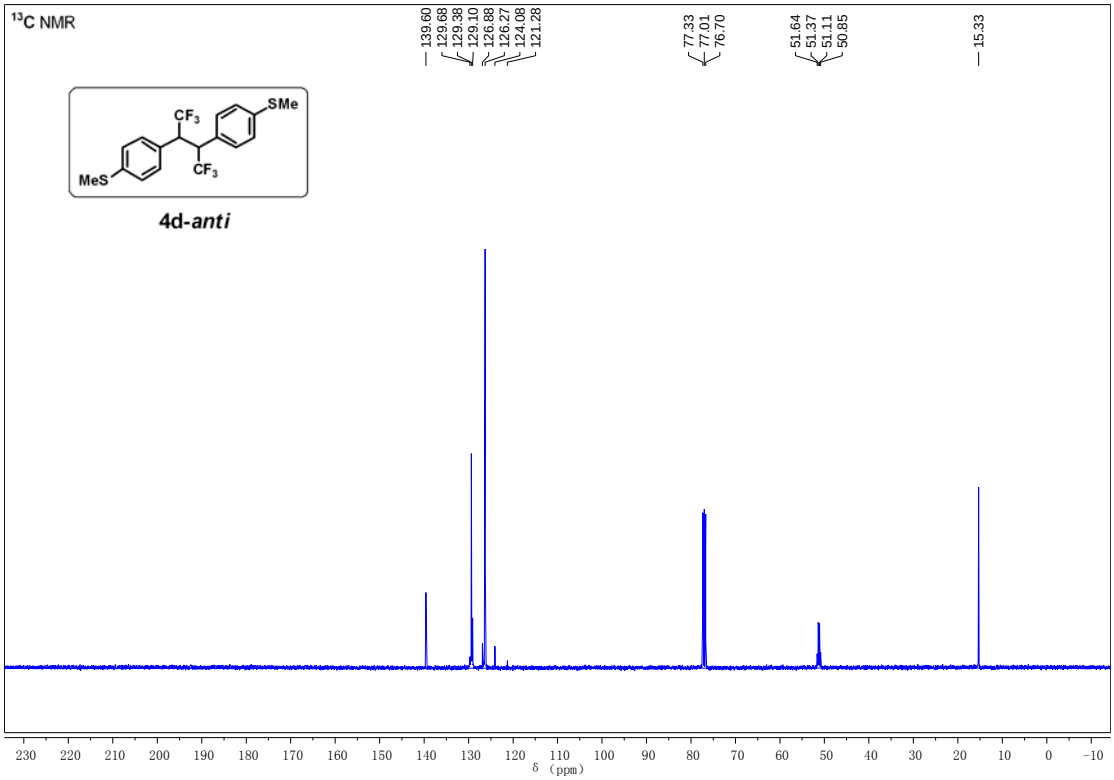

1H NMR
7.26
7.07
7.05
6.85
6.83
4.00
3.97
3.95
2.44
1.53
-0.00
7.07
7.05
6.85
6.83
7.15 7.10 7.05 7.00 6.95 6.90 6.85 6.80 6.75
δ (ppm)
CF3
SMe
MeS
CF3
4d-syn
3.99
4.00
2.05
6.03
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
δ (ppm)

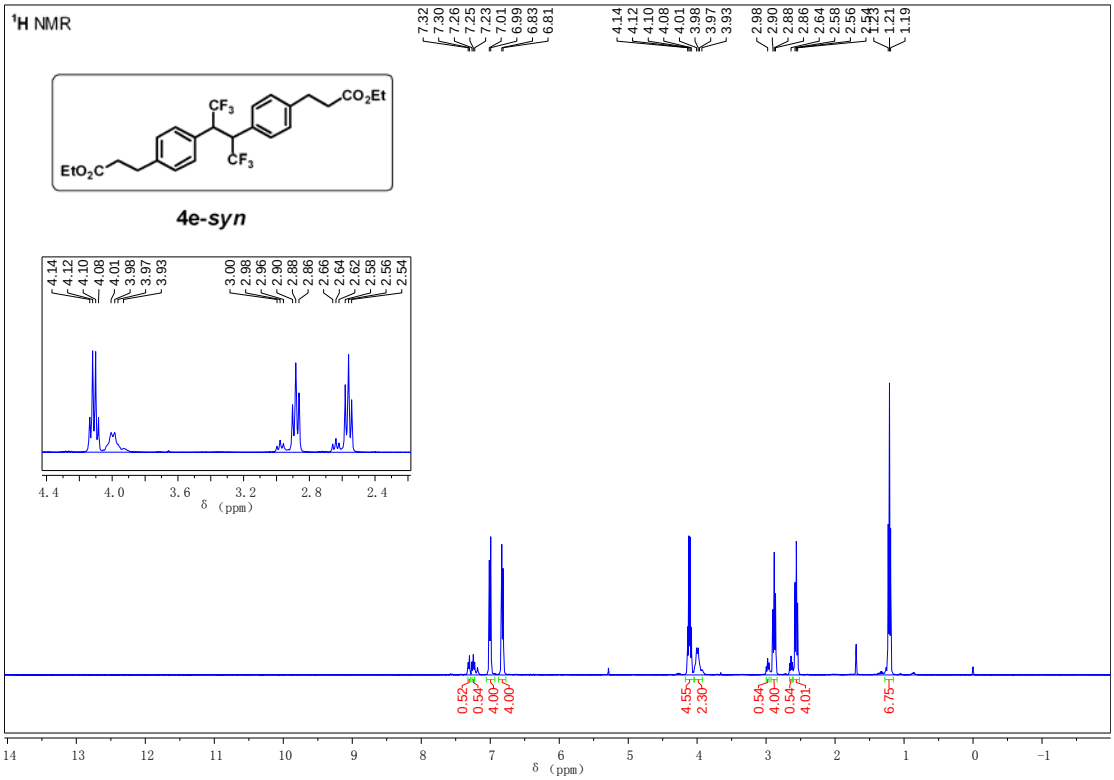

19F NMR
-66.03
-66.05
-66.03
-66.05
-65.7 -65.9 -66.1 -66.3 -66.5
δ (ppm)
CF3
SMe
MeS
CF3
4d-syn
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
δ (ppm)

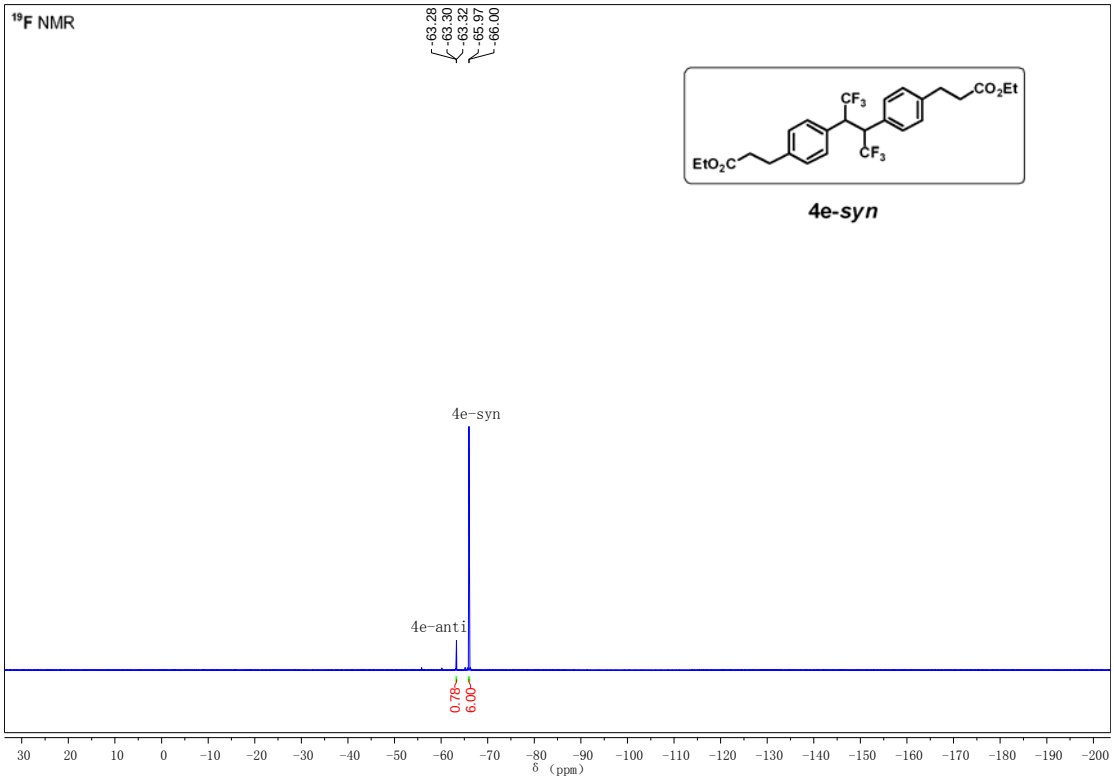

13C NMR
139.39
130.60
130.13
127.33
127.29
125.61
124.54
121.75
77.31
77.00
76.68
49.99
49.71
49.43
49.15
15.22
SMe
CF3
CF3
MeS
4d-syn
δ (ppm)

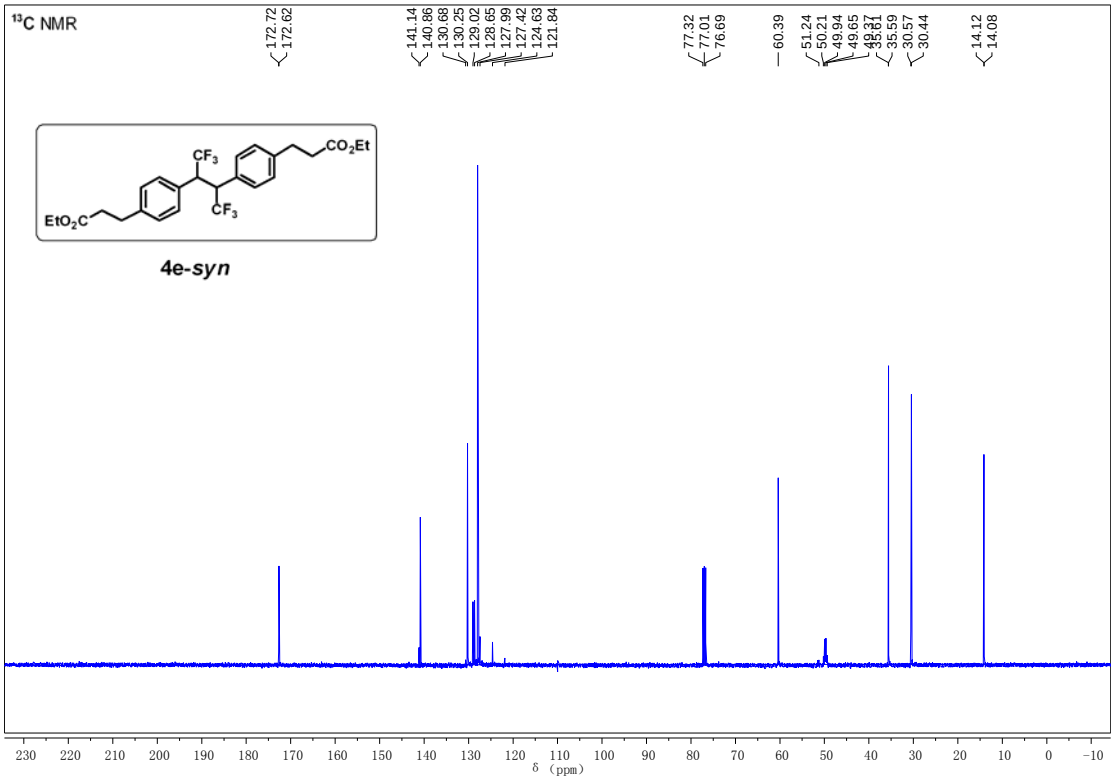

1H NMR
7.30
7.28
7.27
7.25
3.90
3.89
3.85
2.50
SMe
CF3
CF3
MeS
4d-anti
8.00
2.01
6.00
δ (ppm)

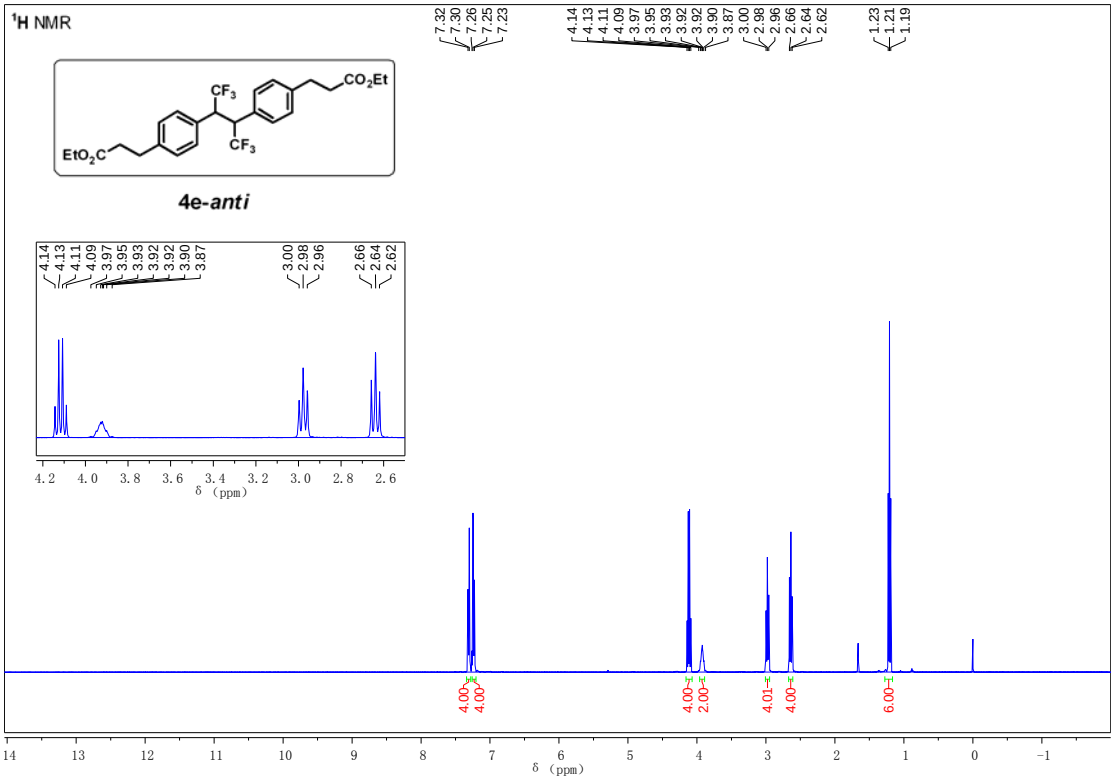

19F NMR
-63.23
-63.25
-63.0 -63.1 -63.2 -63.3 -63.4 -63.5 -63.6
δ (ppm)
CF3
SMe
MeS
CF3
4d-anti
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
δ (ppm)

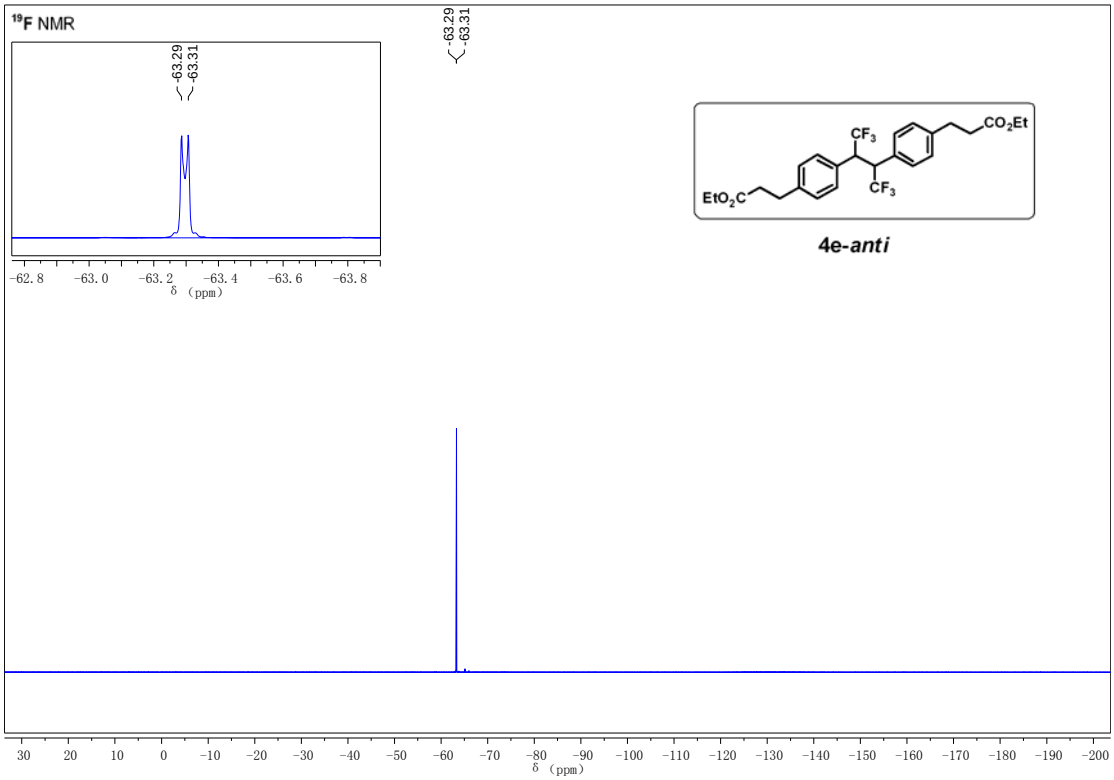

13C NMR
139.60
129.68
129.38
129.10
126.88
126.27
124.08
121.28
77.33
77.01
76.70
51.64
51.37
51.11
50.85
15.33
CF3
SMe
MeS
CF3
4d-anti
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
δ (ppm)

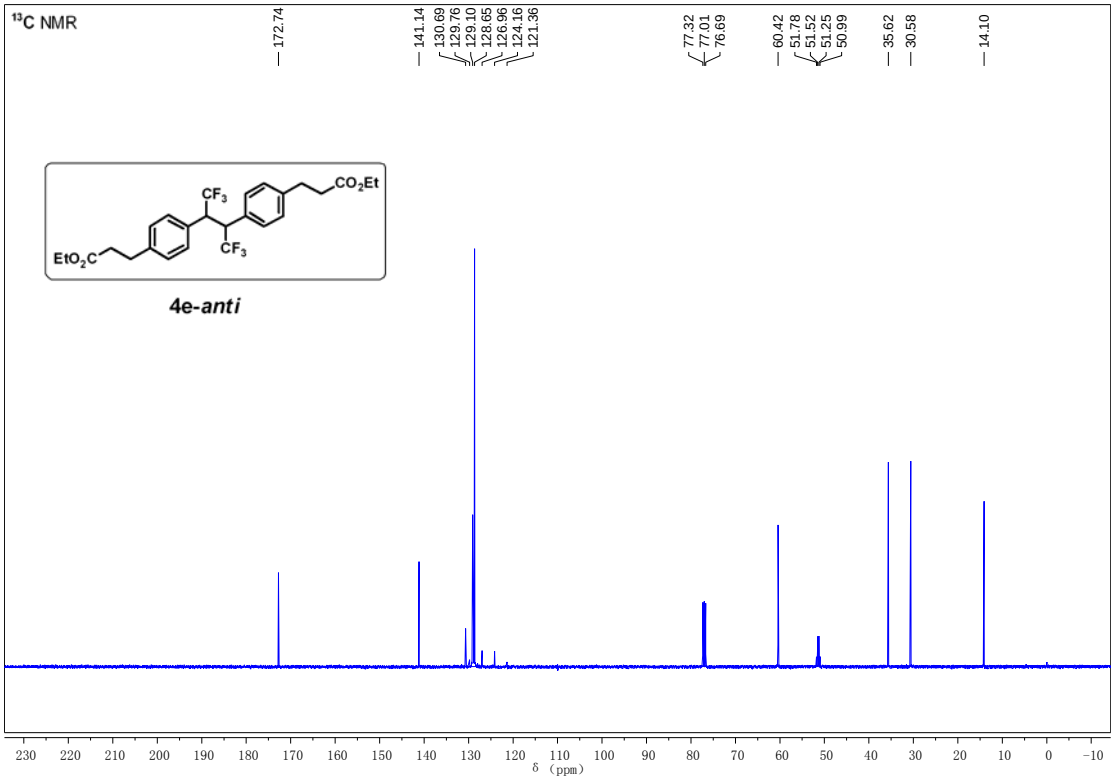

1H NMR
4e-syn
δ (ppm)

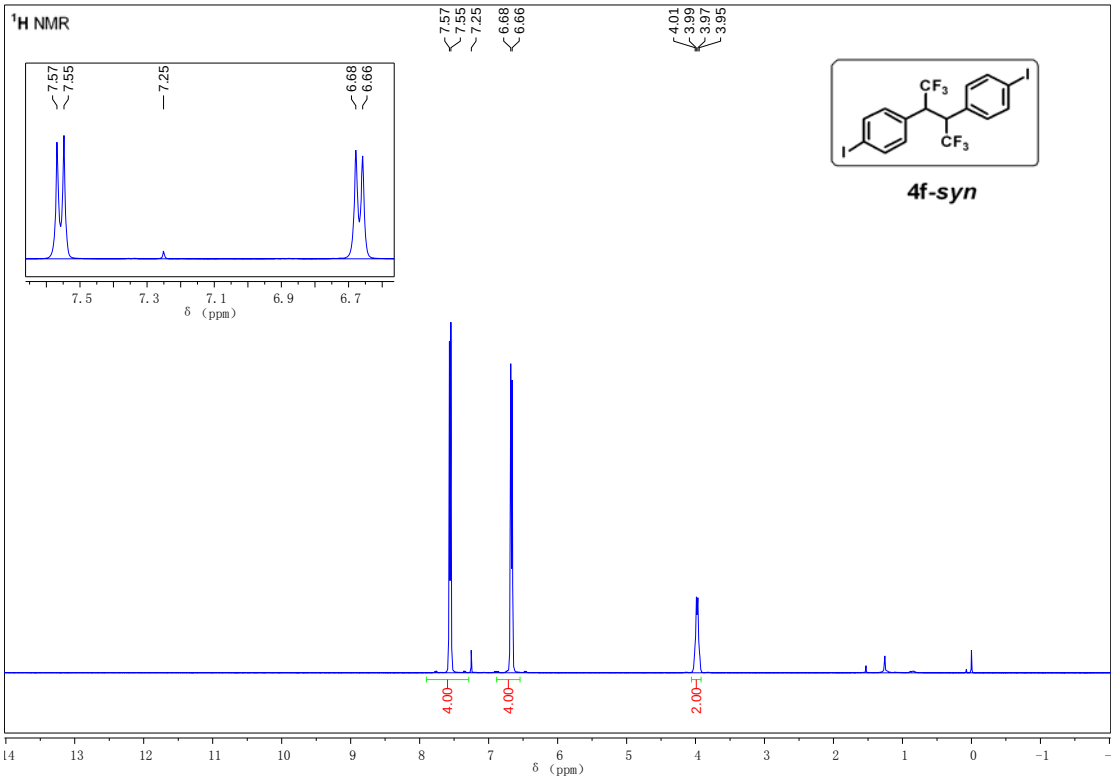

19F NMR
4e-syn
4e-syn
4e-anti
δ (ppm)

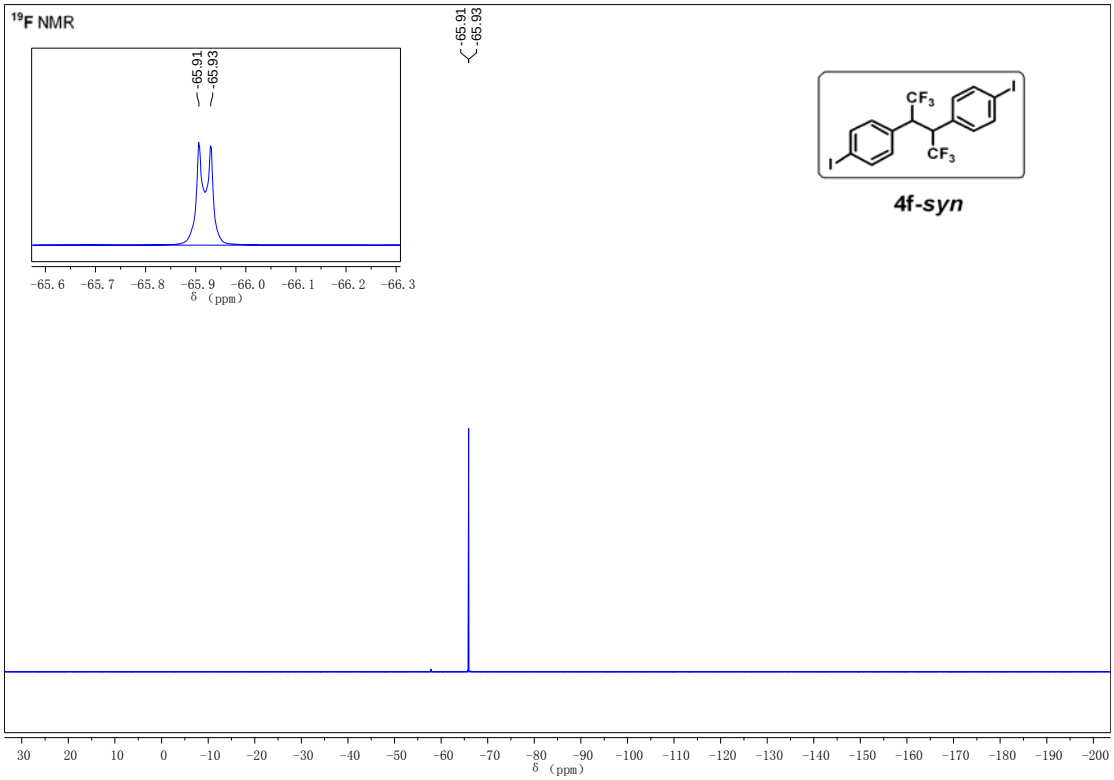

13C NMR
172.72
172.62
141.14
140.86
130.68
130.25
129.02
128.65
127.99
127.42
124.63
121.84
77.32
77.01
76.69
60.39
51.24
50.21
49.94
49.65
49.37
35.81
35.59
30.57
30.44
14.12
14.08
CF3
CO2Et
EtO2C
CF3
4e-syn
δ (ppm)


1H NMR
7.32
7.30
7.26
7.25
7.23
4.14
4.13
4.11
4.09
3.97
3.95
3.93
3.92
3.92
3.90
3.87
3.00
2.98
2.96
2.66
2.64
2.62
1.23
1.21
1.19
CF3
CO2Et
EtO2C
CF3
4e-anti
δ (ppm)
4.00
4.00
4.00
2.00
4.01
4.00
6.00


19F NMR
-63.29
-63.31
-62.8
-63.0
-63.2
-63.4
-63.6
-63.8
δ (ppm)
CF3
CF3
CO2Et
EtO2C
4e-anti
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
δ (ppm)


13C NMR
172.74
141.14
130.69
129.76
129.10
128.65
126.96
124.16
121.36
77.32
77.01
76.69
60.42
51.78
51.52
51.25
50.99
35.62
30.58
14.10
CF3
CF3
CO2Et
EtO2C
4e-anti
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
δ (ppm)


1H NMR
7.57
7.55
7.25
6.68
6.66
4.01
3.99
3.97
3.95
CF3
CF3
I
I
4f-syn
4.00
4.00
2.00
δ (ppm)


19F NMR
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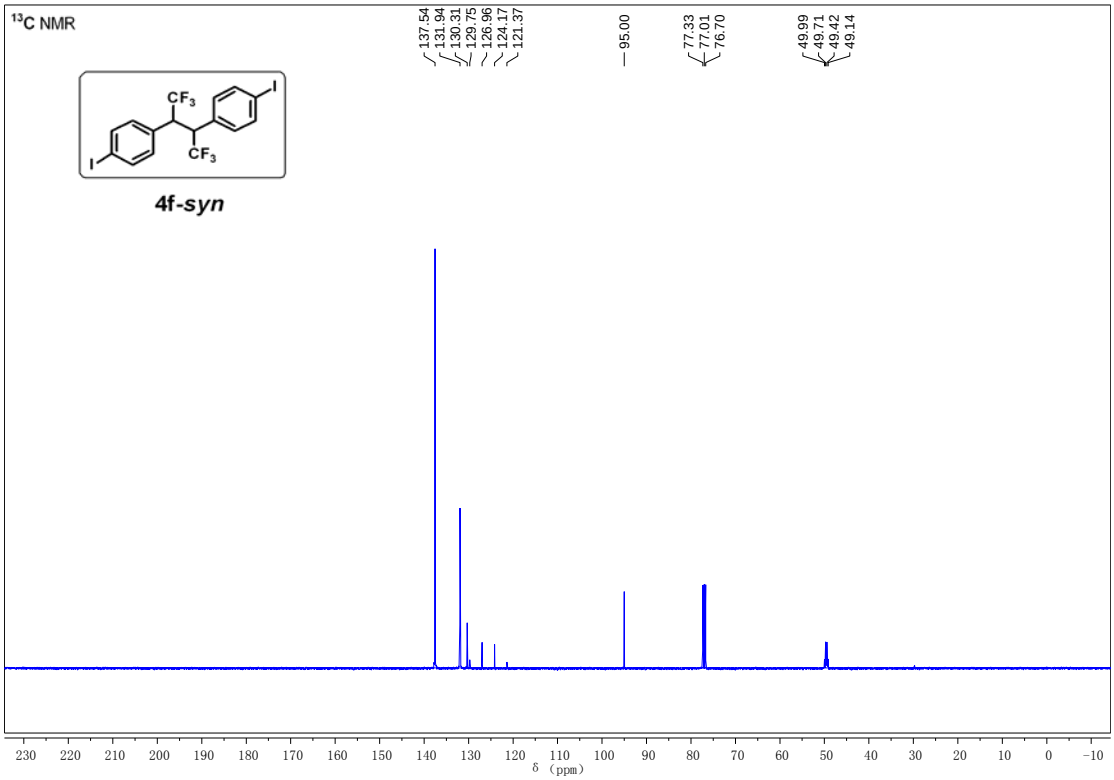
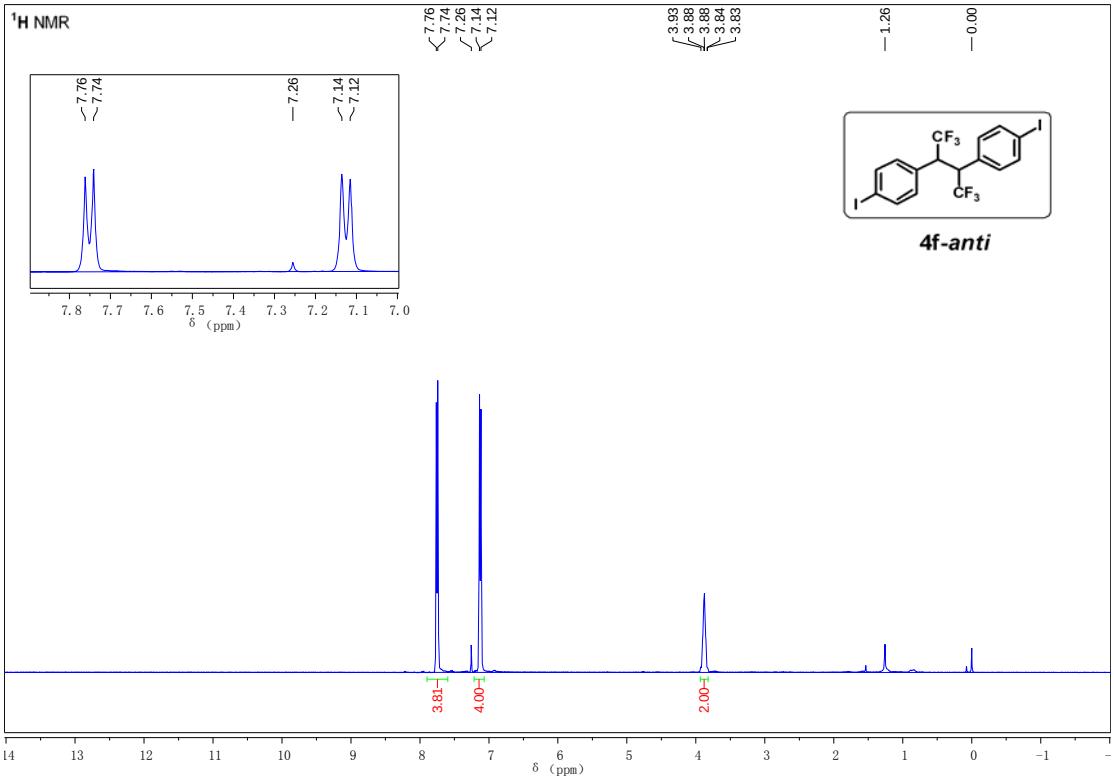
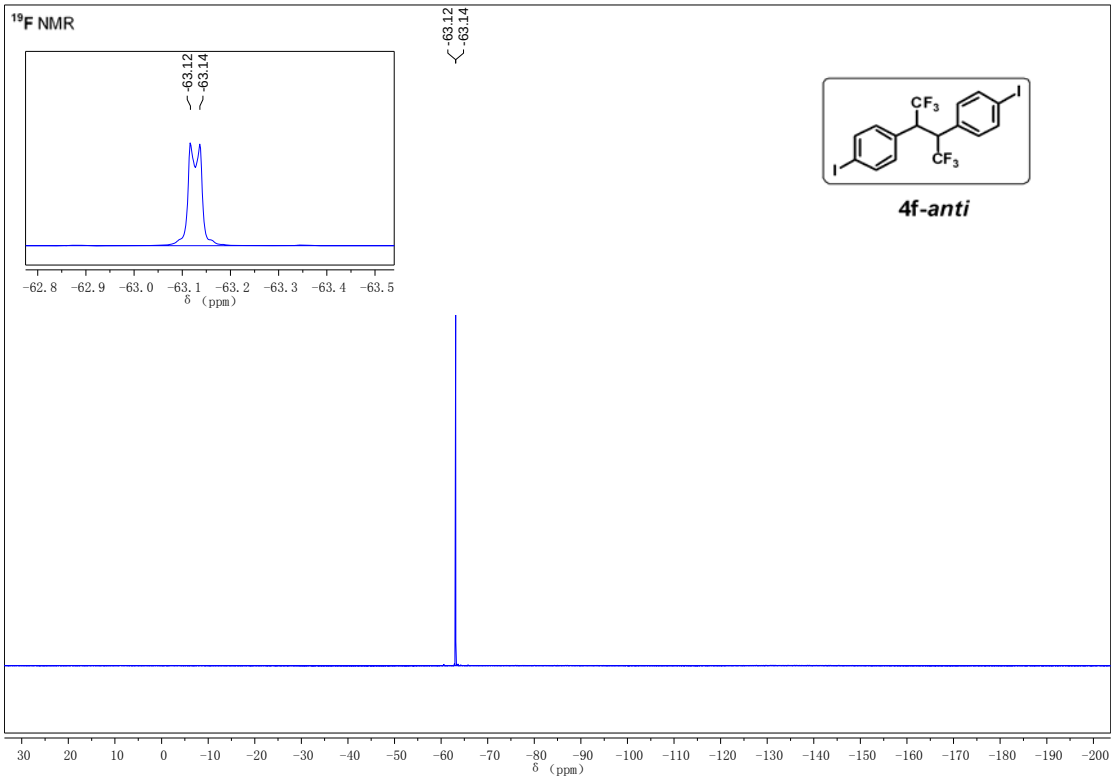
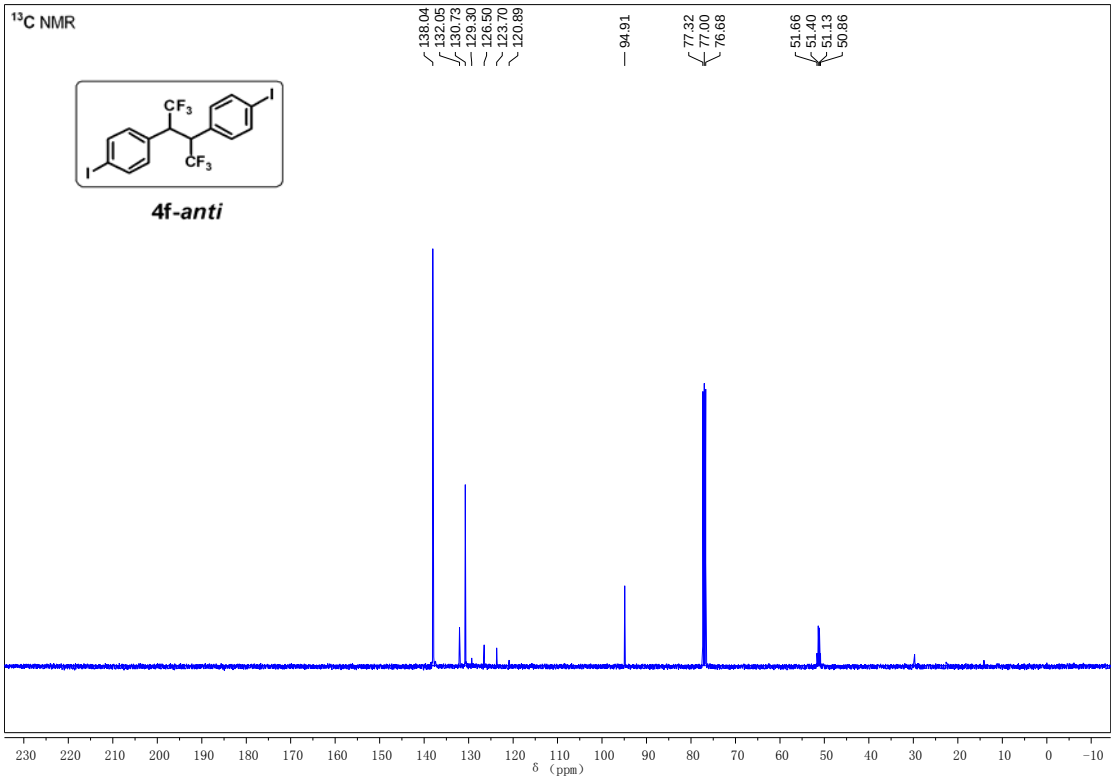
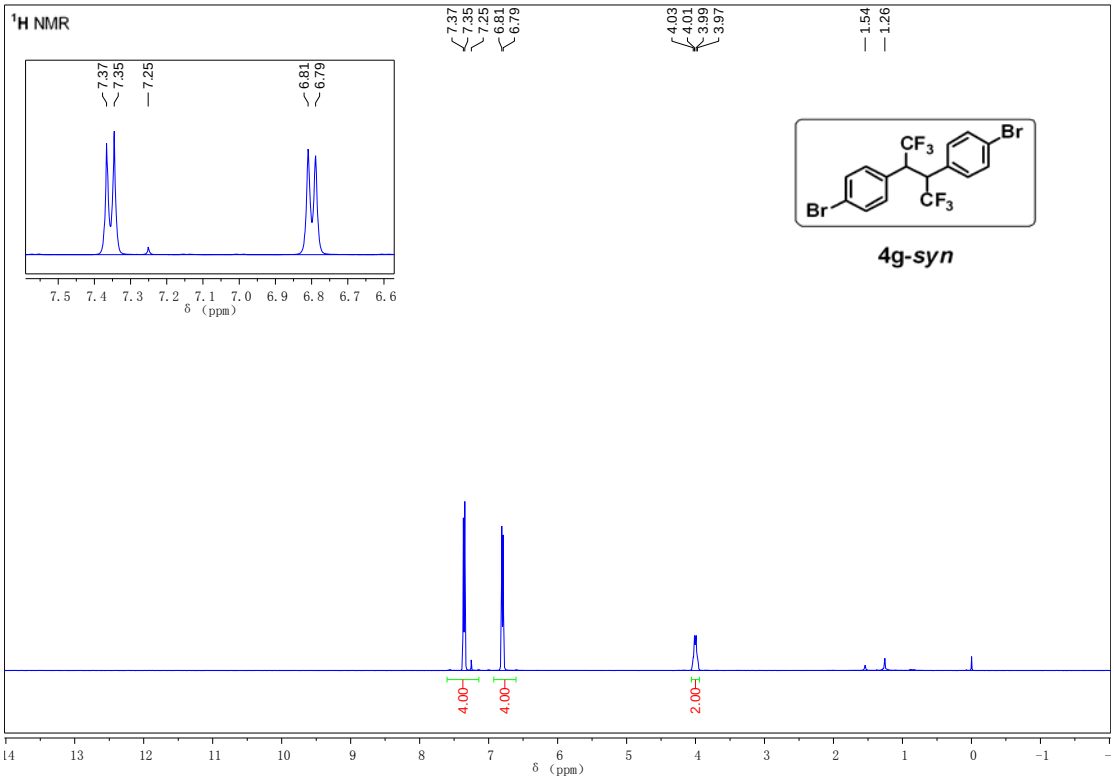
-65.93
CF3
CF3
I
I
4f-syn
δ (ppm)

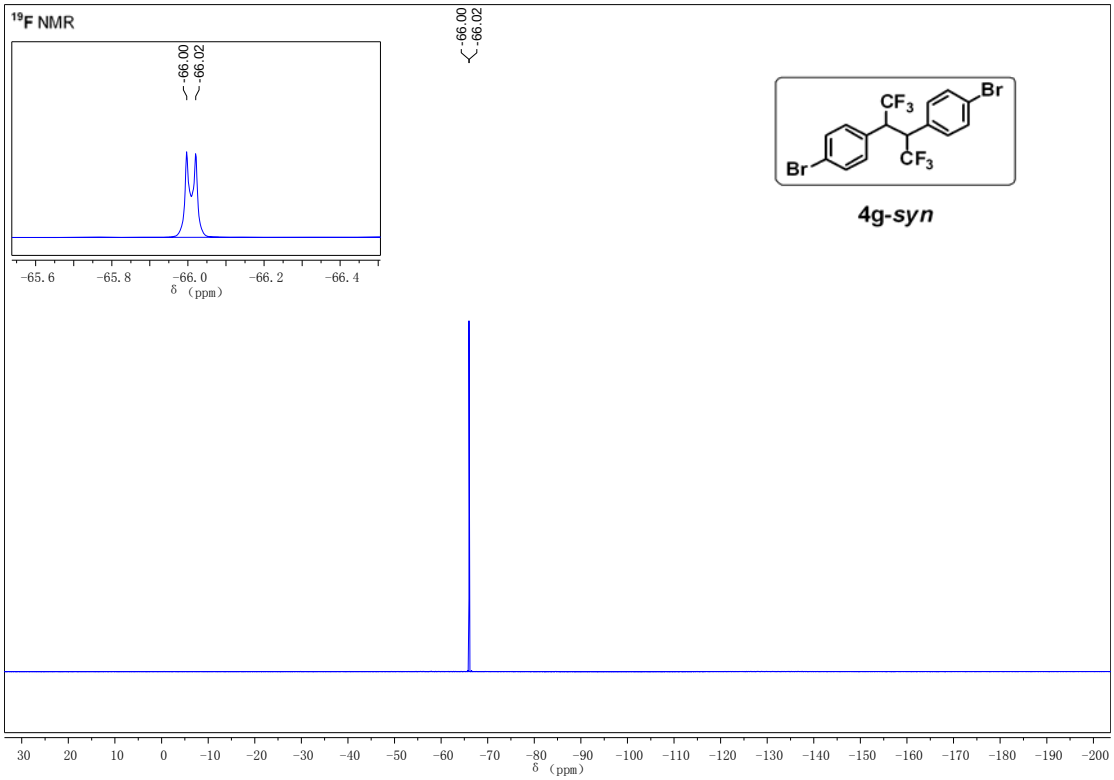

13C NMR
137.54
131.94
130.31
129.75
126.96
124.17
121.37
95.00
77.33
77.01
76.70
49.99
49.71
49.42
49.14
CF3
CF3
I
I
4f-syn
δ (ppm)

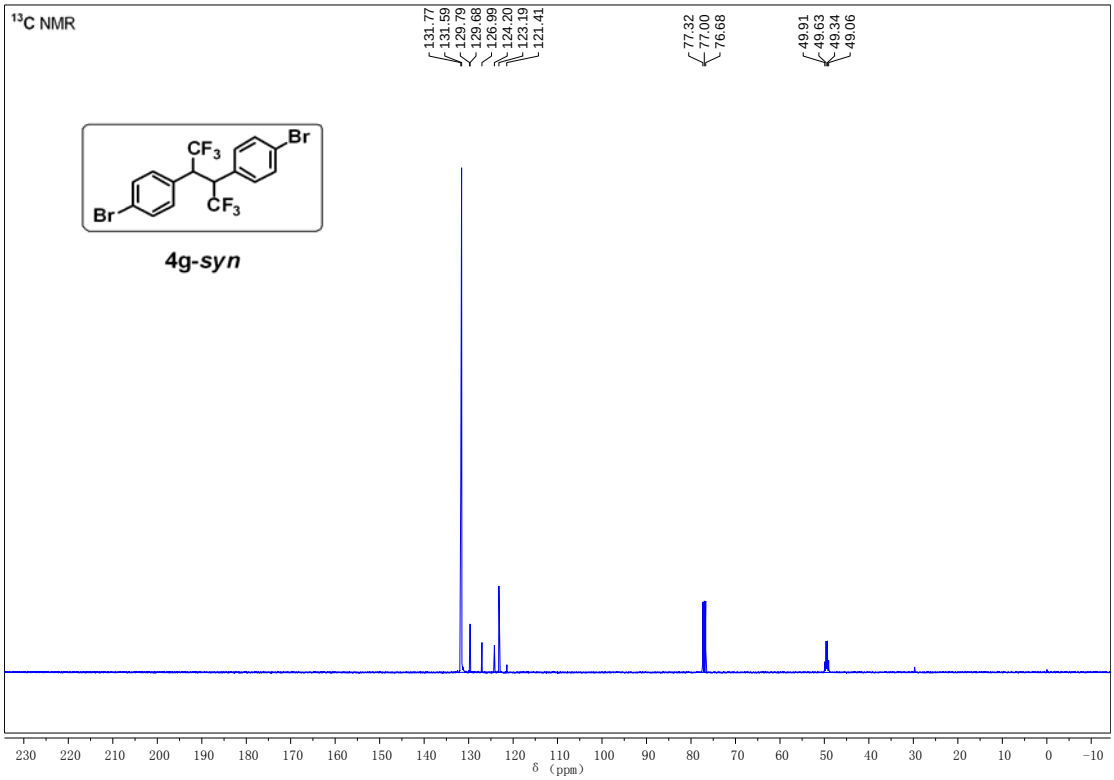

1H NMR
7.76
7.74
7.26
7.14
7.12
3.93
3.88
3.88
3.84
3.83
1.26
0.00
7.76
7.74
7.26
7.14
7.12
CF3
CF3
I
I
4f-anti
3.81
4.00
2.00
δ (ppm)

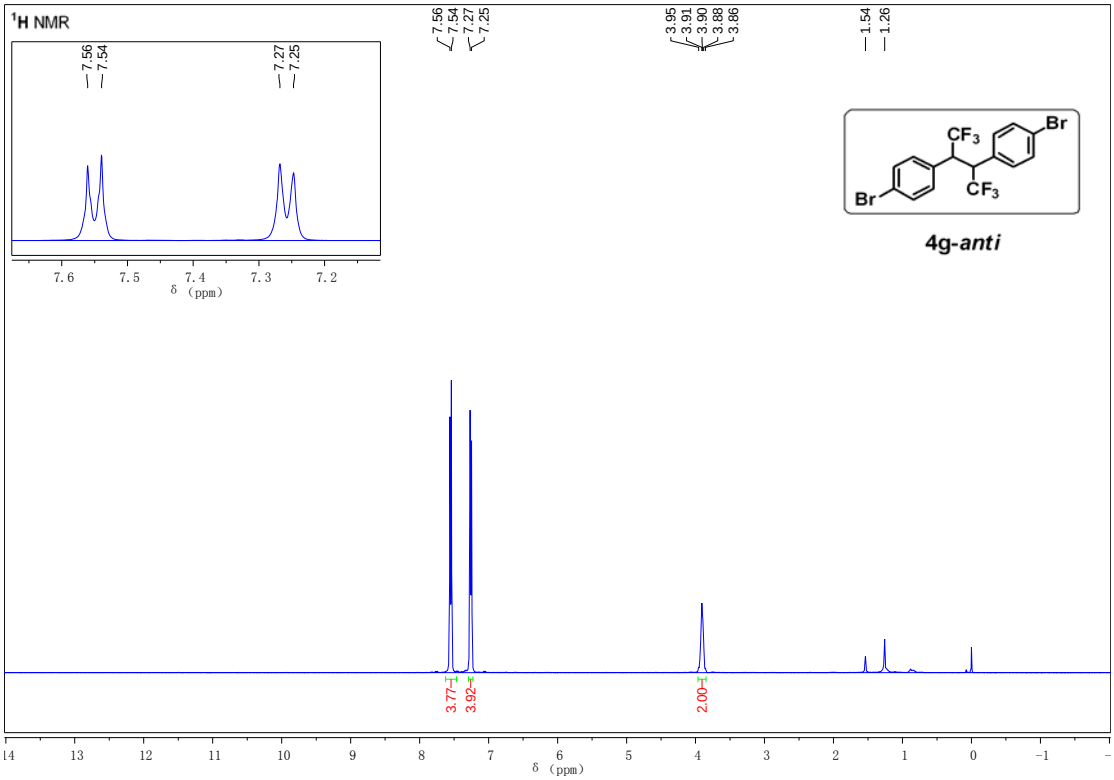

19F NMR
-63.12
-63.14
-62.8 -62.9 -63.0 -63.1 -63.2 -63.3 -63.4 -63.5
δ (ppm)
CF3
I
4f-anti
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
δ (ppm)

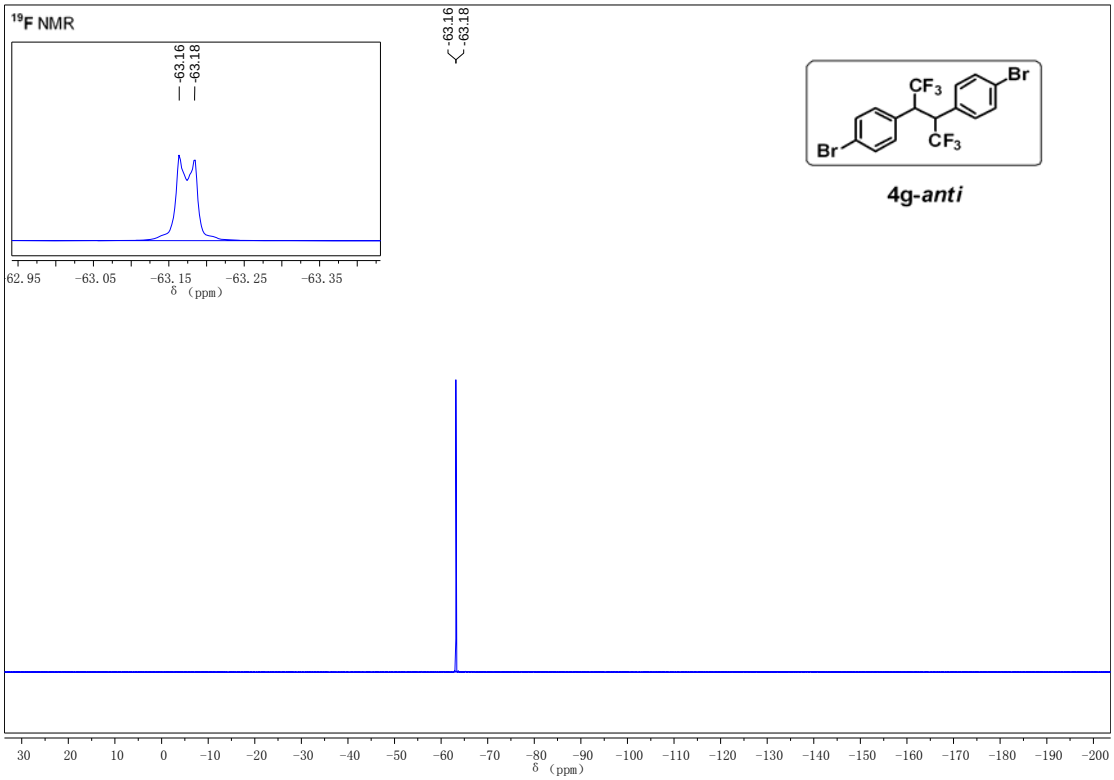

13C NMR
138.04
132.05
130.73
129.30
126.50
123.70
120.89
94.91
77.32
77.00
76.68
51.66
51.40
51.13
50.86
CF3
I
4f-anti
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
δ (ppm)

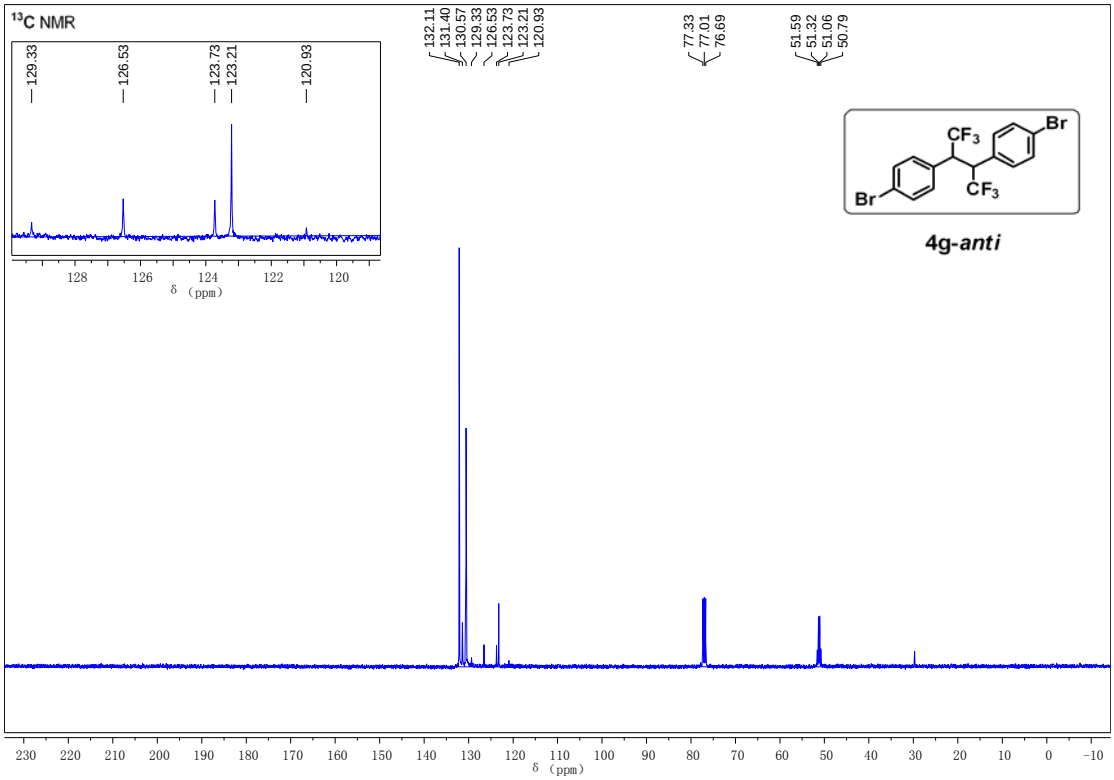

1H NMR
7.37
7.35
7.25
6.81
6.79
4.03
4.01
3.99
3.97
1.54
1.26
CF3
Br
Br
CF3
4g-syn
4.00
4.00
2.00
δ (ppm)

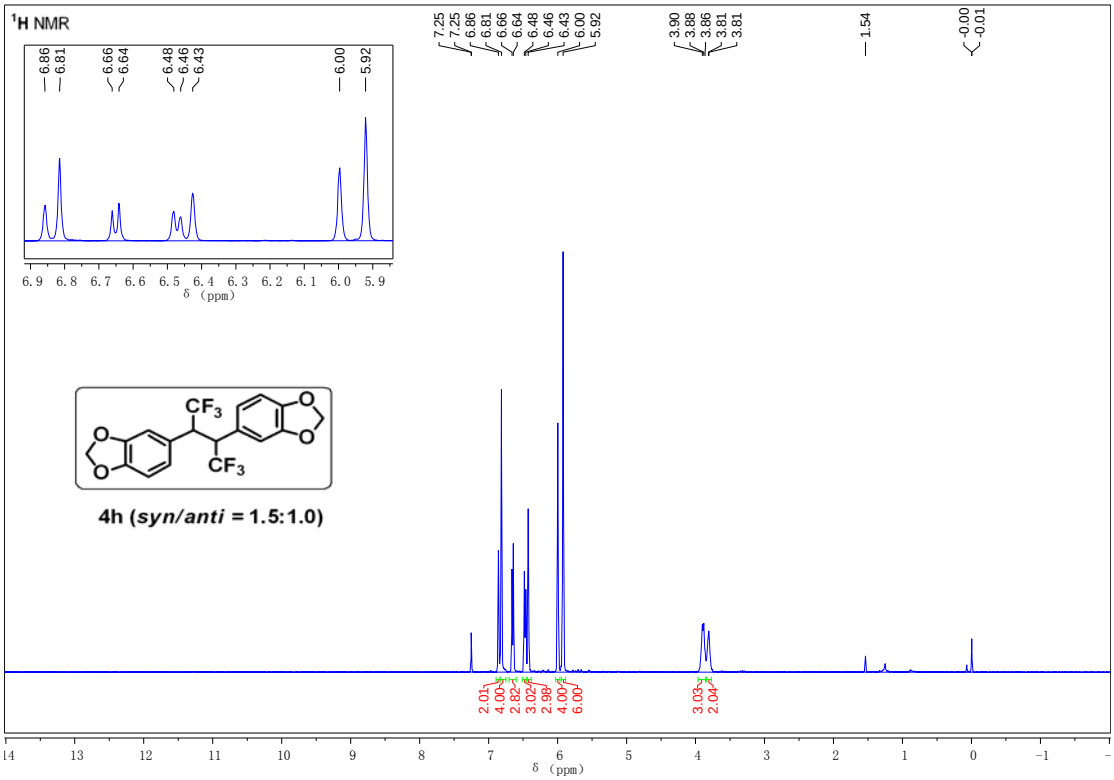

19F NMR
-66.00
-66.02
CF3
Br
Br
CF3
4g-syn
δ (ppm)

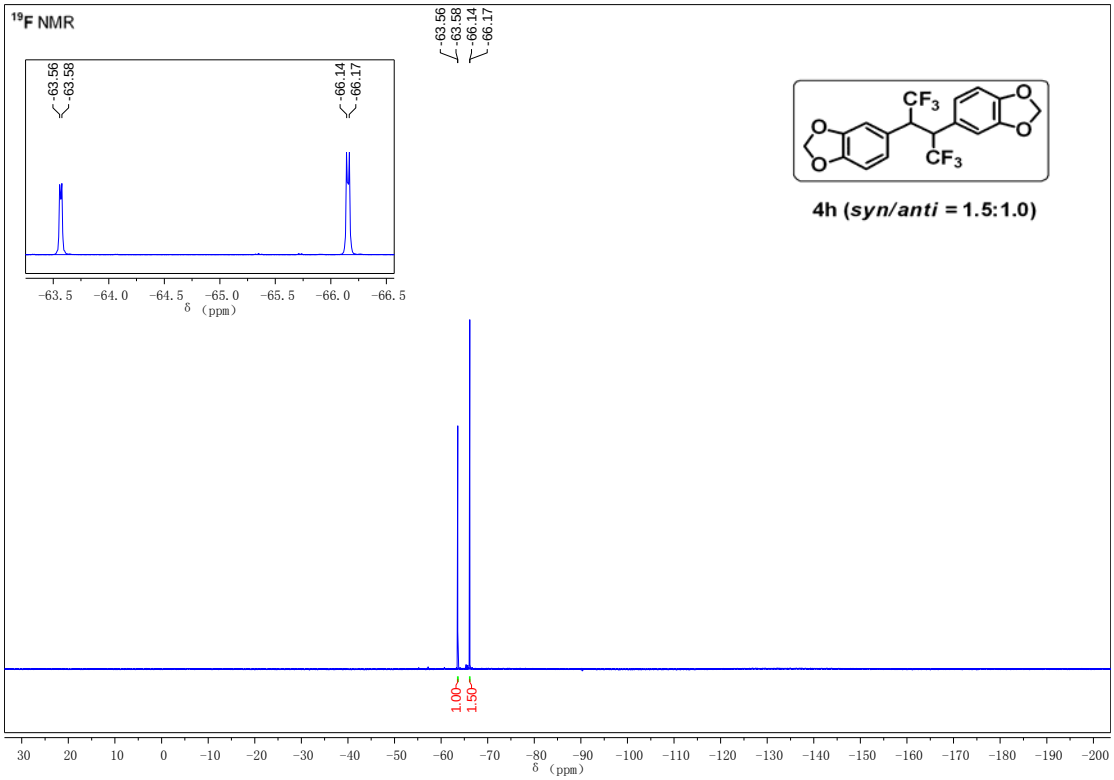

13C NMR
131.77
131.59
129.79
129.68
126.99
124.20
123.19
121.41
77.32
77.00
76.68
49.91
49.63
49.34
49.06
CF3
Br
CF3
Br
4g-syn
δ (ppm)

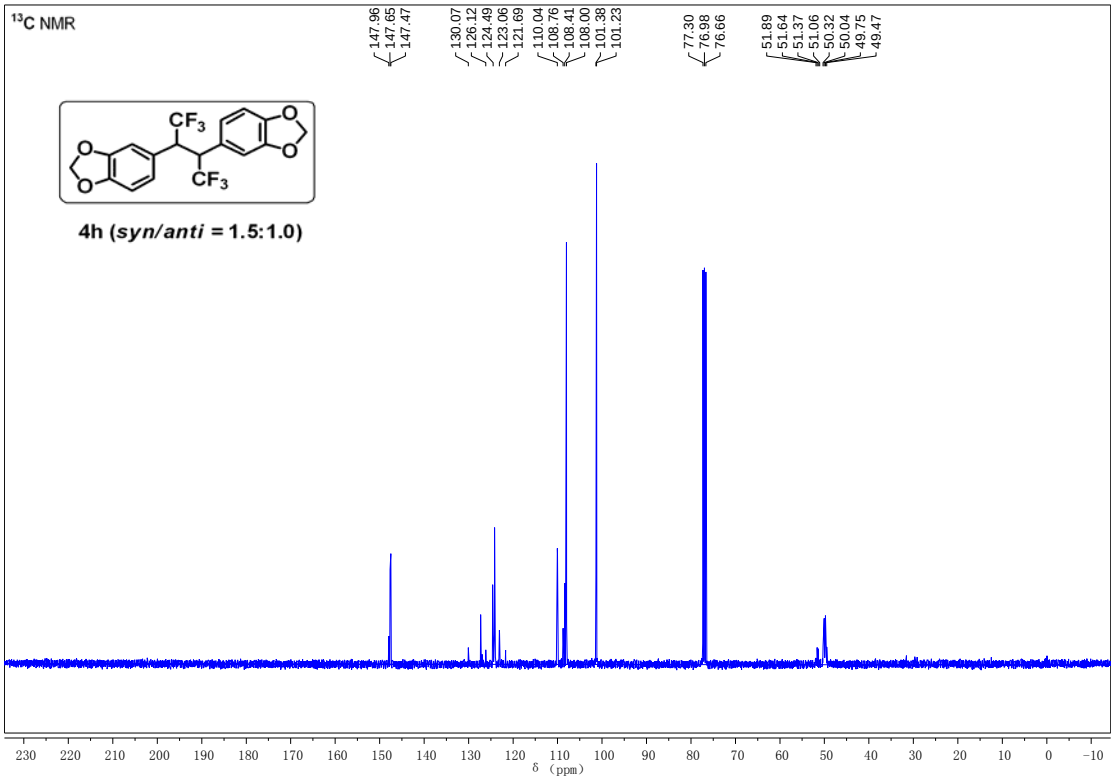

1H NMR
7.56
7.54
7.27
7.25
3.95
3.91
3.90
3.88
3.86
1.54
1.26
CF3
Br
CF3
Br
4g-anti
3.77
3.92
2.00
δ (ppm)

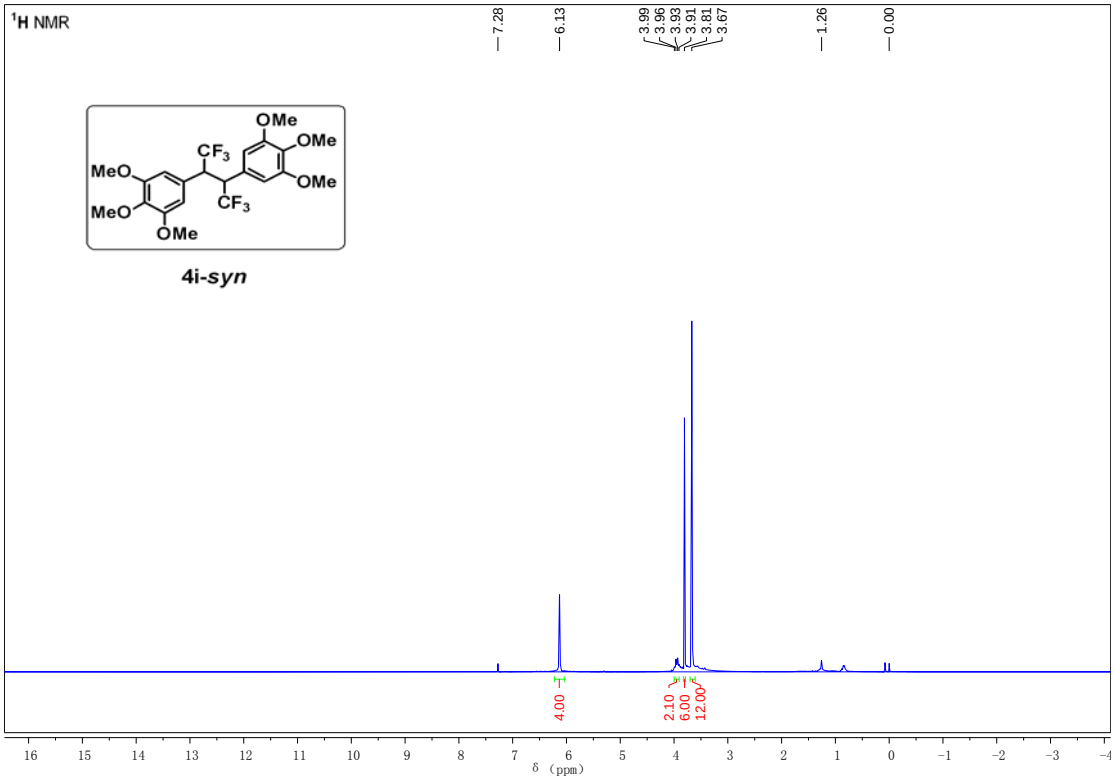

19F NMR
-63.16
-63.18
CF3
Br
Br
CF3
4g-anti
δ (ppm)

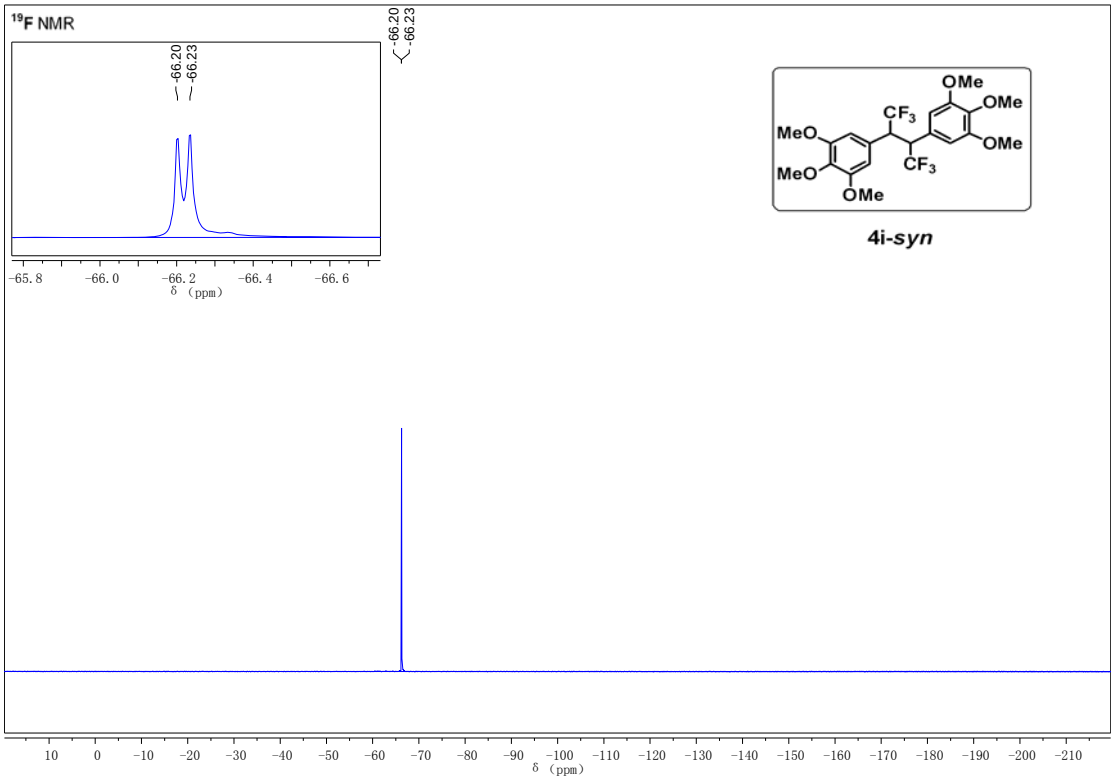

13C NMR
129.33
126.53
123.73
123.21
120.93
132.11
131.40
130.57
129.33
126.53
123.73
123.21
120.93
77.33
77.01
76.69
51.59
51.32
51.06
50.79
CF3
Br
Br
CF3
4g-anti
δ (ppm)

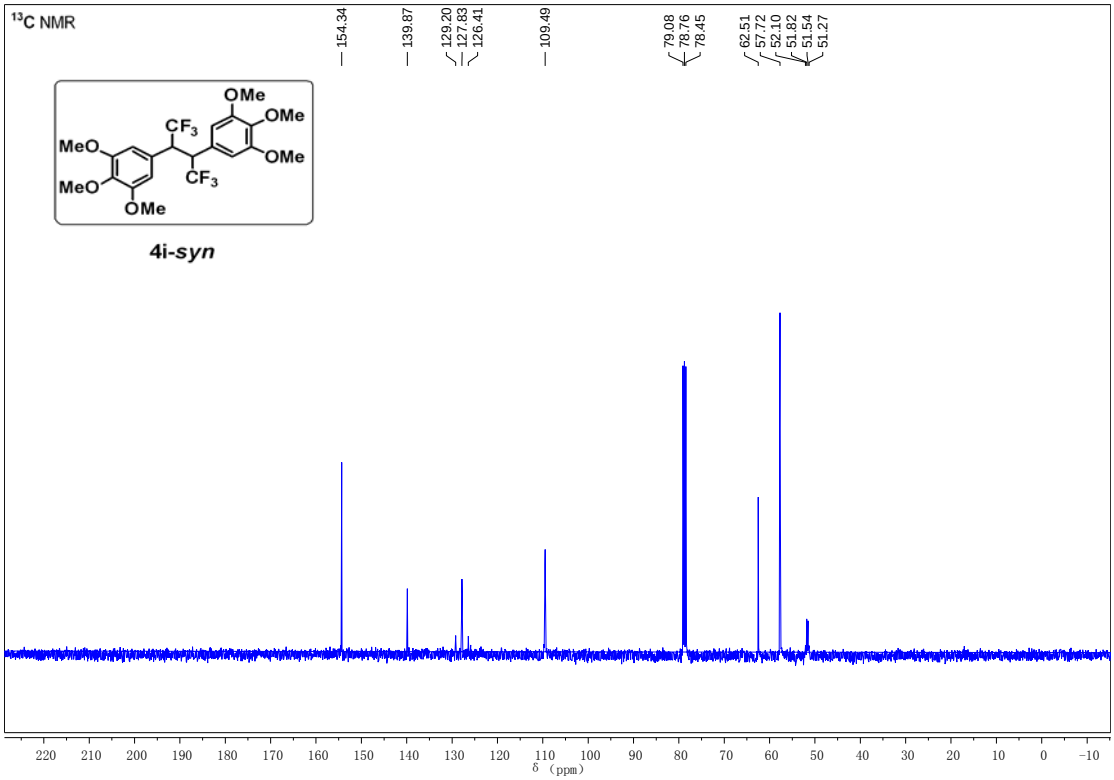

1H NMR
7.25
7.25
6.86
6.81
6.66
6.64
6.48
6.46
6.43
6.00
5.92
3.90
3.88
3.86
3.81
3.81
1.54
-0.00
-0.01
6.86
6.81
6.66
6.64
6.48
6.46
6.43
6.00
5.92
6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1 6.0 5.9
δ (ppm)
CF3
CF3
4h (syn/anti = 1.5:1.0)
2.01
4.00
2.82
3.02
2.99
4.00
6.00
3.03
2.04
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1
δ (ppm)

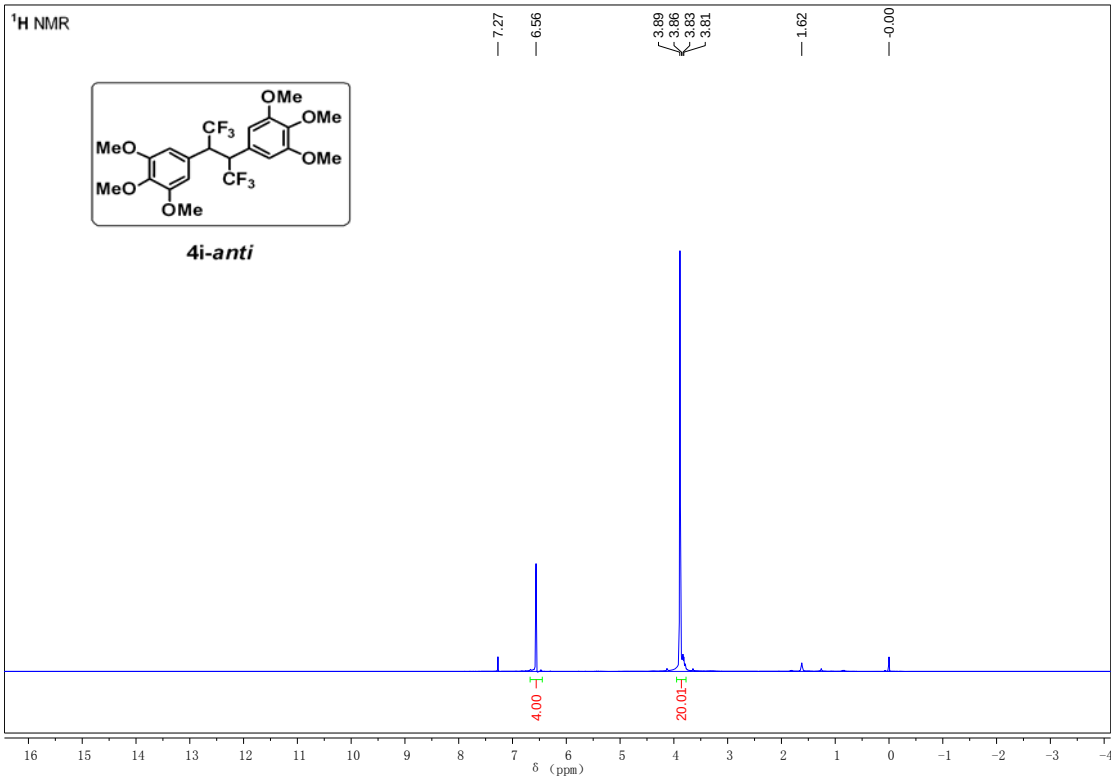

19F NMR
-63.56
-63.58
-66.14
-66.17
-63.56
-63.58
-66.14
-66.17
−63.5 −64.0 −64.5 −65.0 −65.5 −66.0 −66.5
δ (ppm)
CF3
CF3
4h (syn/anti = 1.5:1.0)
1.00
1.50
30 20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200
δ (ppm)

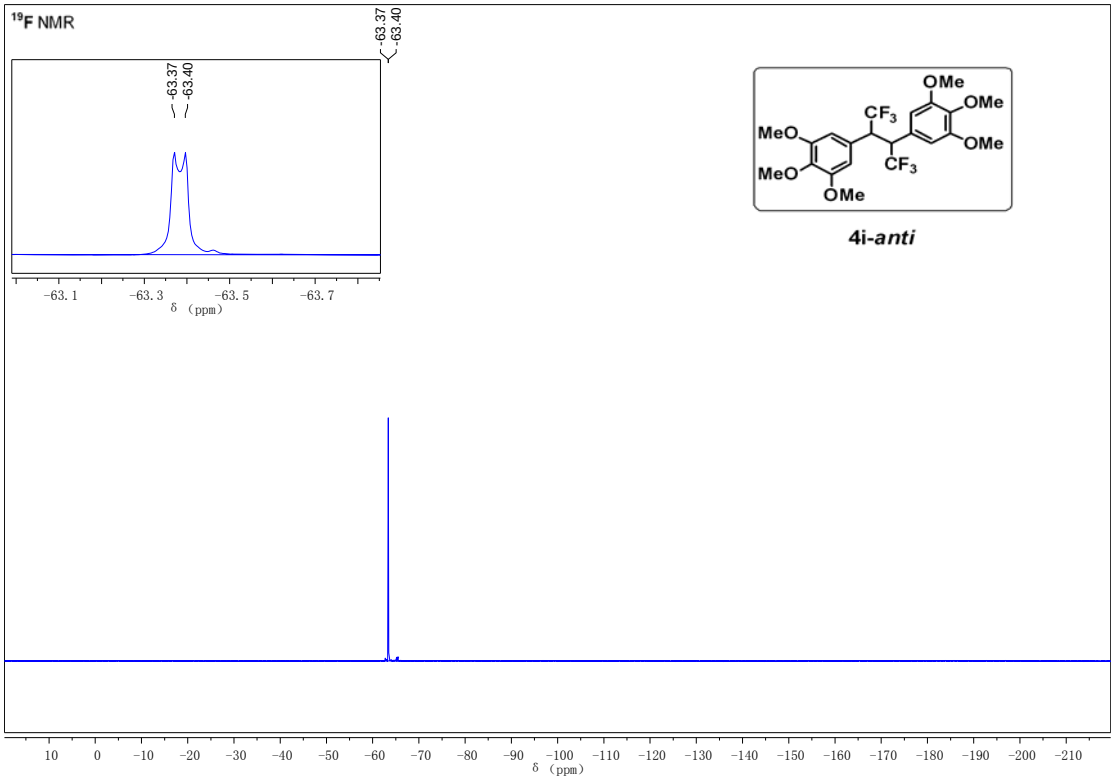

13C NMR
147.96
147.65
147.47
130.07
126.12
124.49
123.06
121.69
110.04
108.76
108.41
108.00
101.38
101.23
77.30
76.98
76.66
51.89
51.64
51.37
51.06
50.32
50.04
49.75
49.47
CF3
CF3
4h (syn/anti = 1.5:1.0)
δ (ppm)

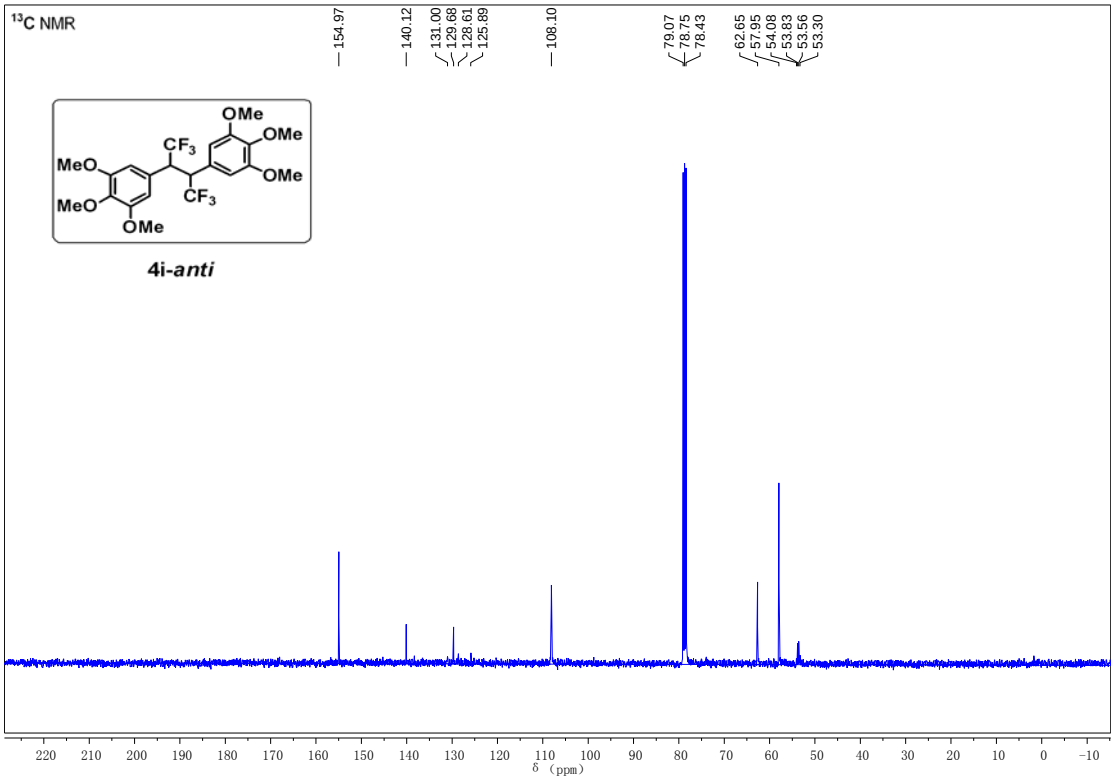

1H NMR
7.28
6.13
3.99
3.96
3.93
3.91
3.81
3.67
1.26
0.00
OMe
OMe
OMe
MeO
MeO
OMe
CF3
CF3
4i-syn
4.00
2.10
6.00
12.00
δ (ppm)

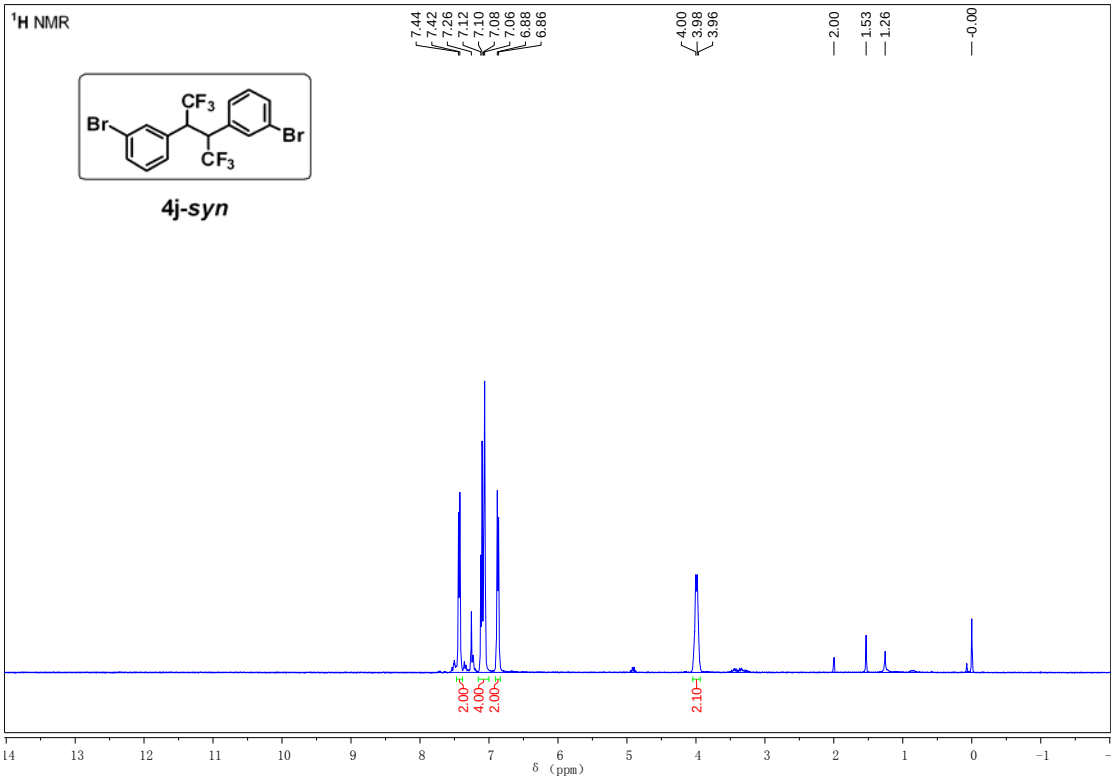

19F NMR
-66.20
-66.23
-65.8
-66.0
-66.2
-66.4
-66.6
δ (ppm)
OMe
OMe
OMe
CF3
CF3
MeO
MeO
OMe
4i-syn
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
δ (ppm)

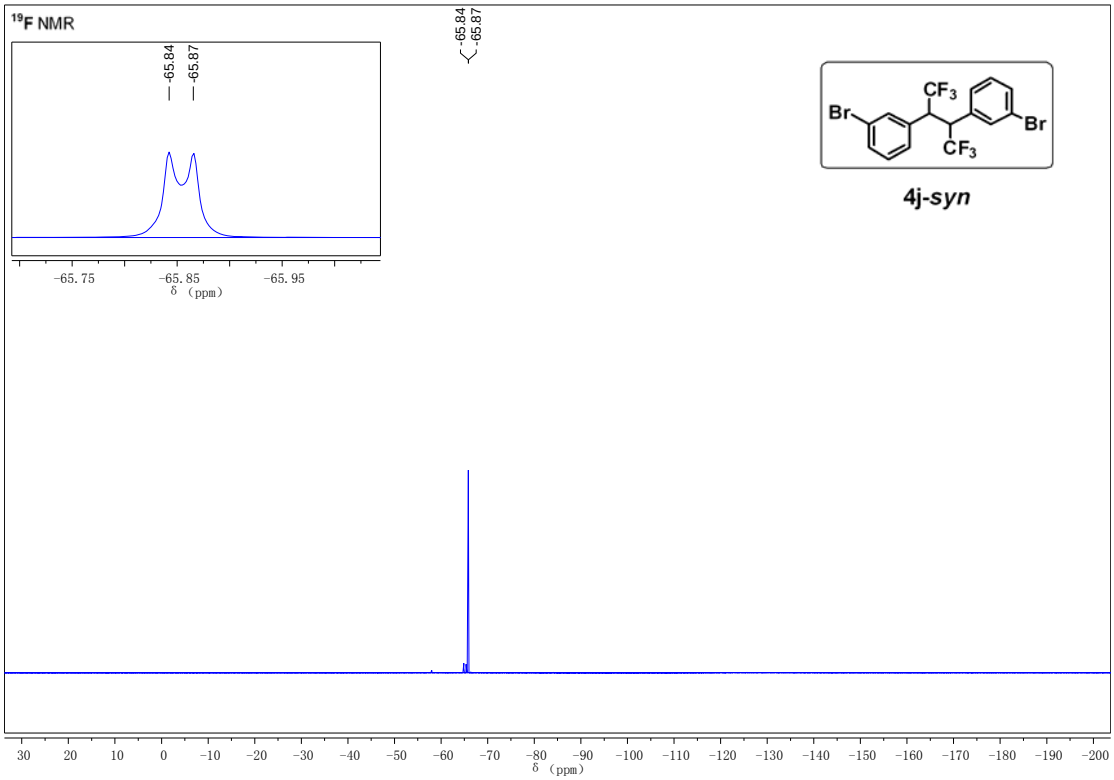

13C NMR
154.34
139.87
129.20
127.83
126.41
109.49
79.08
78.76
78.45
62.51
57.72
52.10
51.82
51.54
51.27
OMe
OMe
OMe
CF3
CF3
MeO
MeO
OMe
4i-syn
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
δ (ppm)

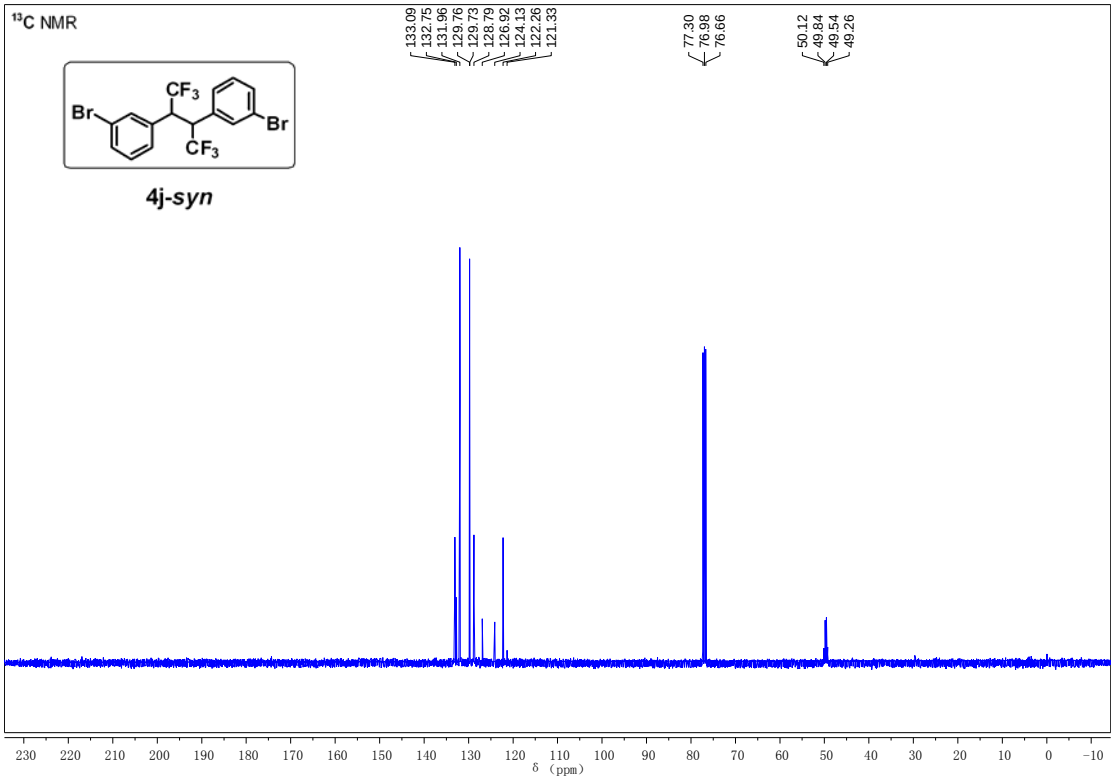

1H NMR
7.27
6.56
3.89
3.86
3.83
3.81
1.62
-0.00
OMe
OMe
OMe
CF3
CF3
MeO
MeO
OMe
4i-anti
4.00
20.01
δ (ppm)

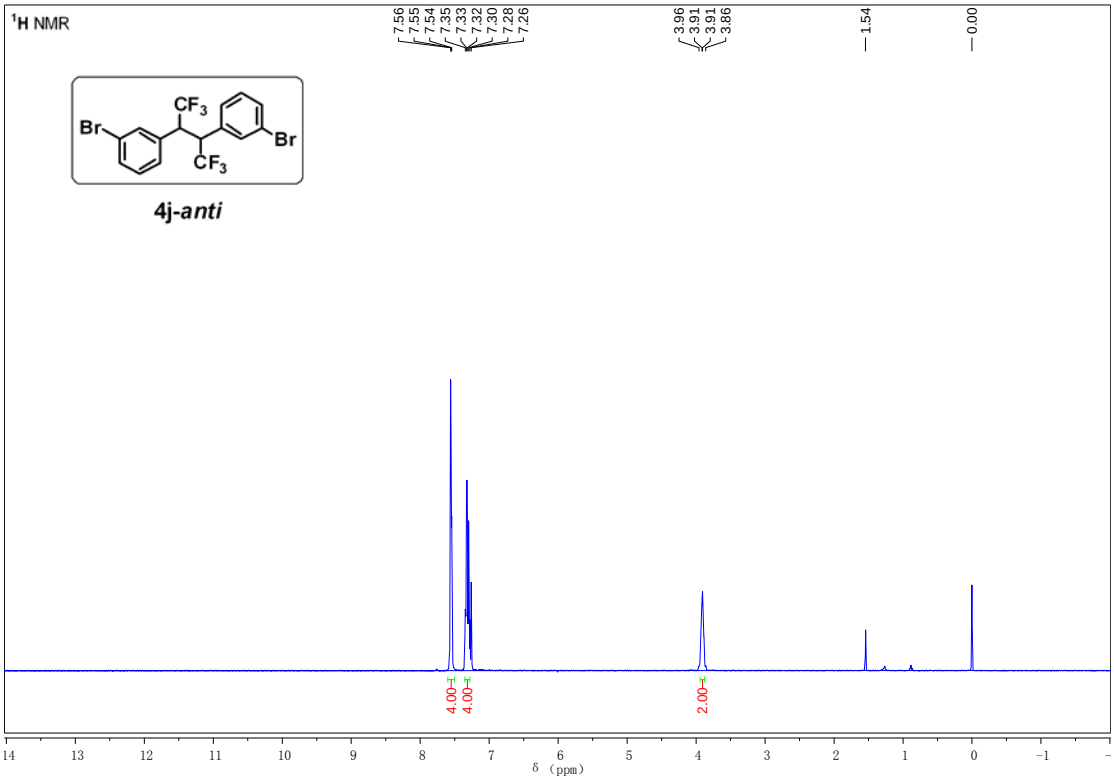

19F NMR
-63.37
-63.40
-63.37
-63.40
δ (ppm)
OMe
OMe
OMe
CF3
CF3
MeO
MeO
OMe
4i-anti
δ (ppm)

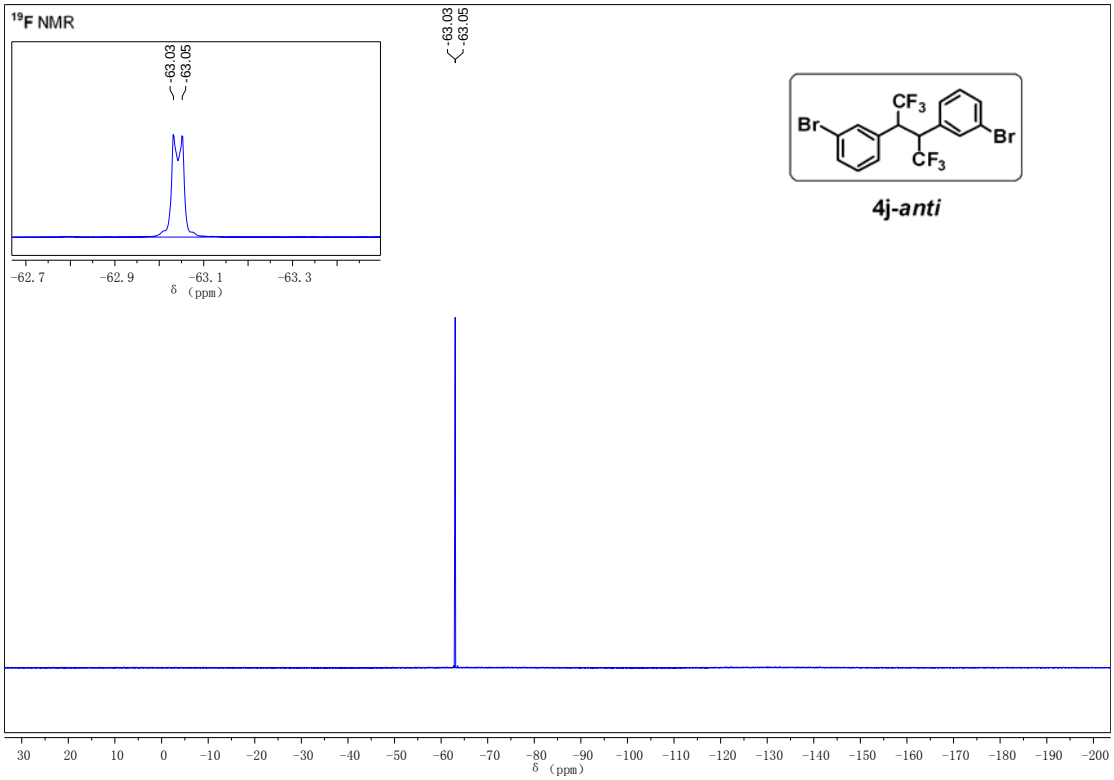

13C NMR
154.97
140.12
131.00
129.68
128.61
125.89
108.10
79.07
78.75
78.43
62.65
57.95
54.08
53.83
53.56
53.30
OMe
OMe
OMe
CF3
CF3
MeO
MeO
OMe
4i-anti
δ (ppm)

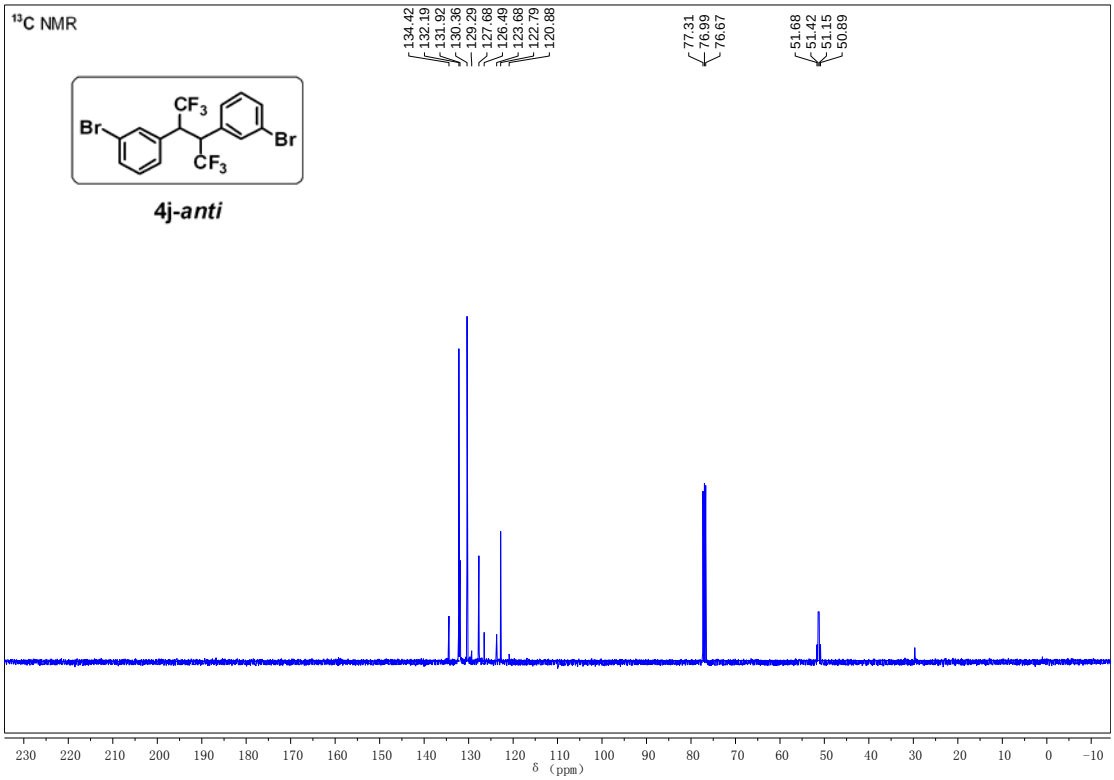

1H NMR
7.44
7.42
7.26
7.12
7.10
7.08
7.06
6.88
6.86
4.00
3.98
3.96
2.00
1.53
1.26
0.00
CF3
CF3
Br
Br
4j-syn
2.00
4.00
2.00
2.10
δ (ppm)

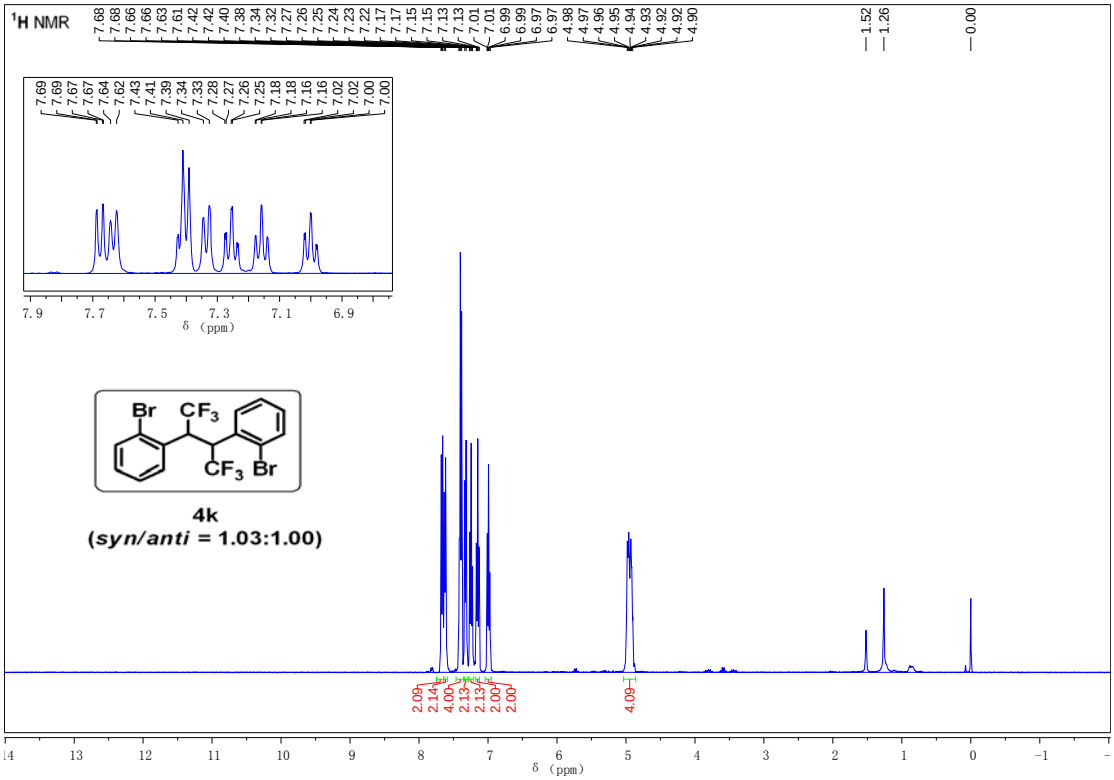

19F NMR
-65.84
-65.87
-65.75
-65.85
-65.95
δ (ppm)
Br
CF3
CF3
Br
4j-syn

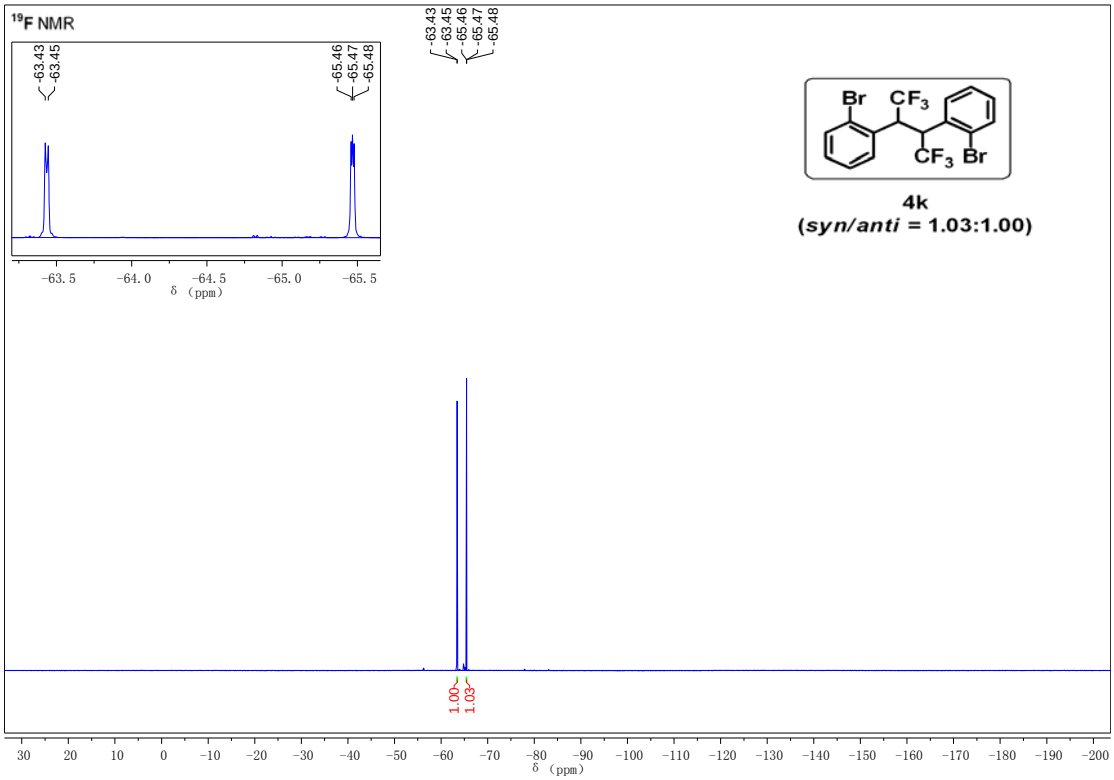

13C NMR
133.09
132.75
131.96
129.76
129.73
128.79
126.92
124.13
122.26
121.33
77.30
76.98
76.66
50.12
49.84
49.54
49.26
Br
CF3
CF3
Br
4j-syn
δ (ppm)

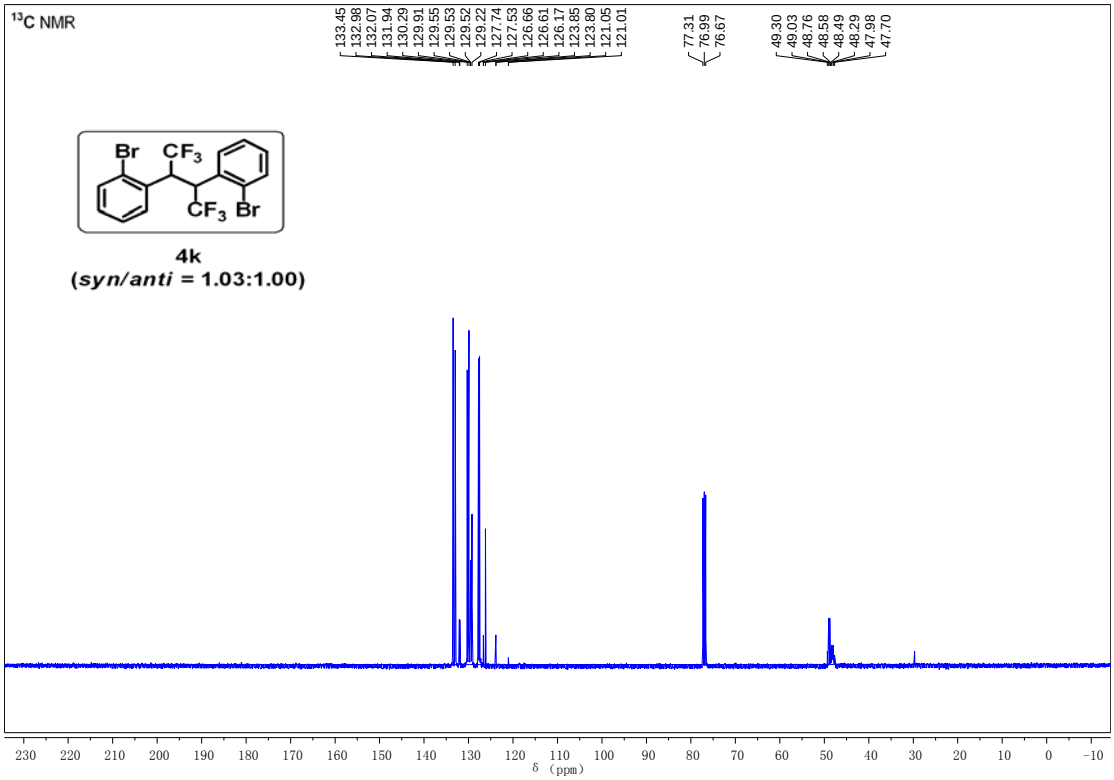

1H NMR
7.56
7.55
7.54
7.35
7.33
7.32
7.30
7.28
7.26
3.96
3.91
3.91
3.86
1.54
0.00
Br
CF3
CF3
Br
4j-anti
4.00
4.00
2.00
δ (ppm)

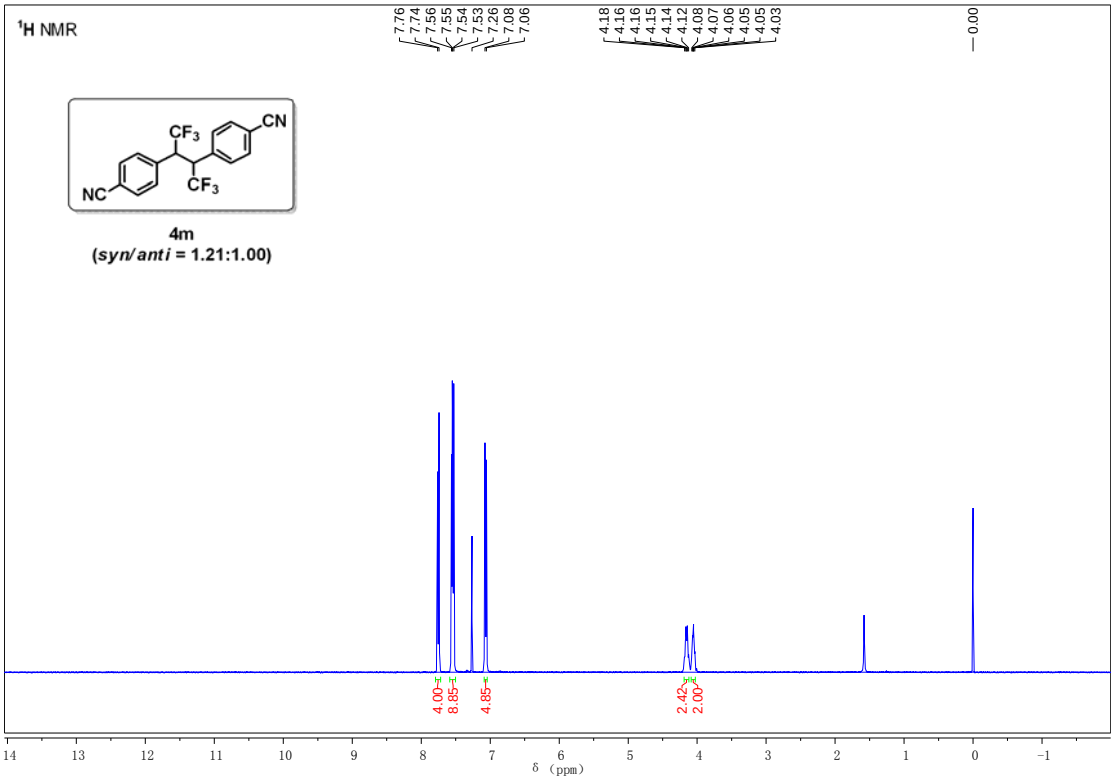

19F NMR
-63.03
-63.05
-62.7
-62.9
-63.1
-63.3
δ (ppm)
Br
CF3
CF3
Br
4j-anti
δ (ppm)

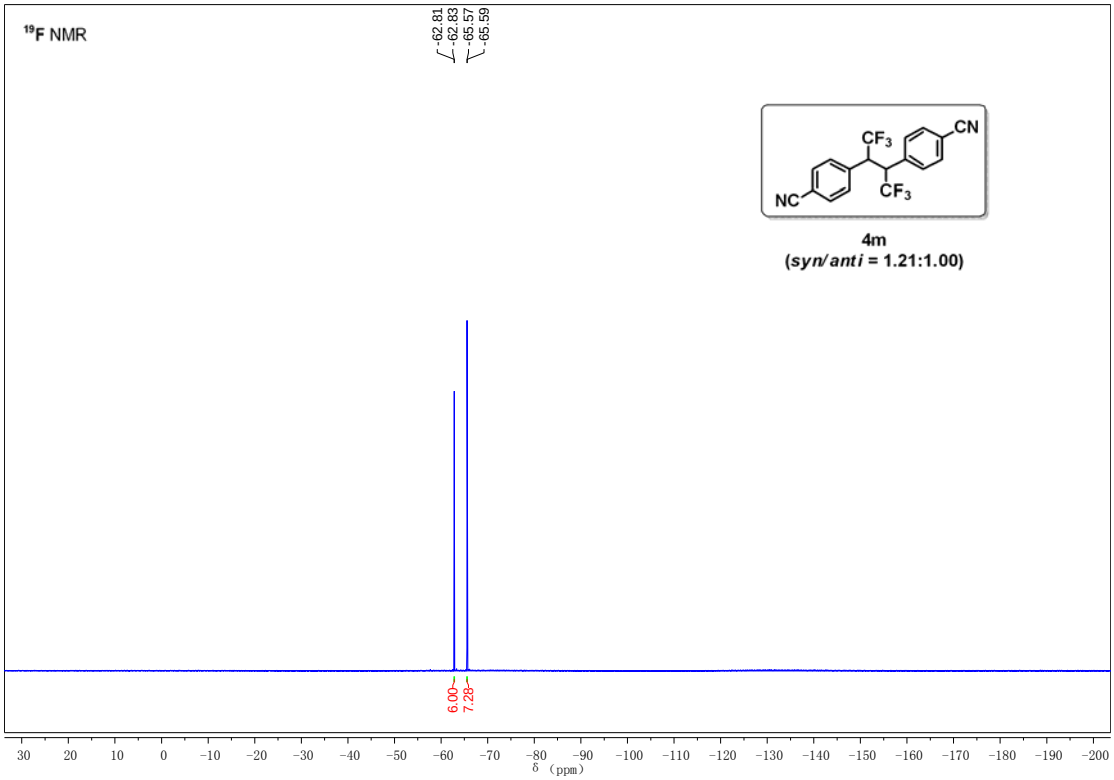

13C NMR
134.42
132.19
131.92
130.36
129.29
127.68
126.49
123.68
122.79
120.88
77.31
76.99
76.67
51.68
51.42
51.15
50.89
Br
CF3
CF3
Br
4j-anti
δ (ppm)

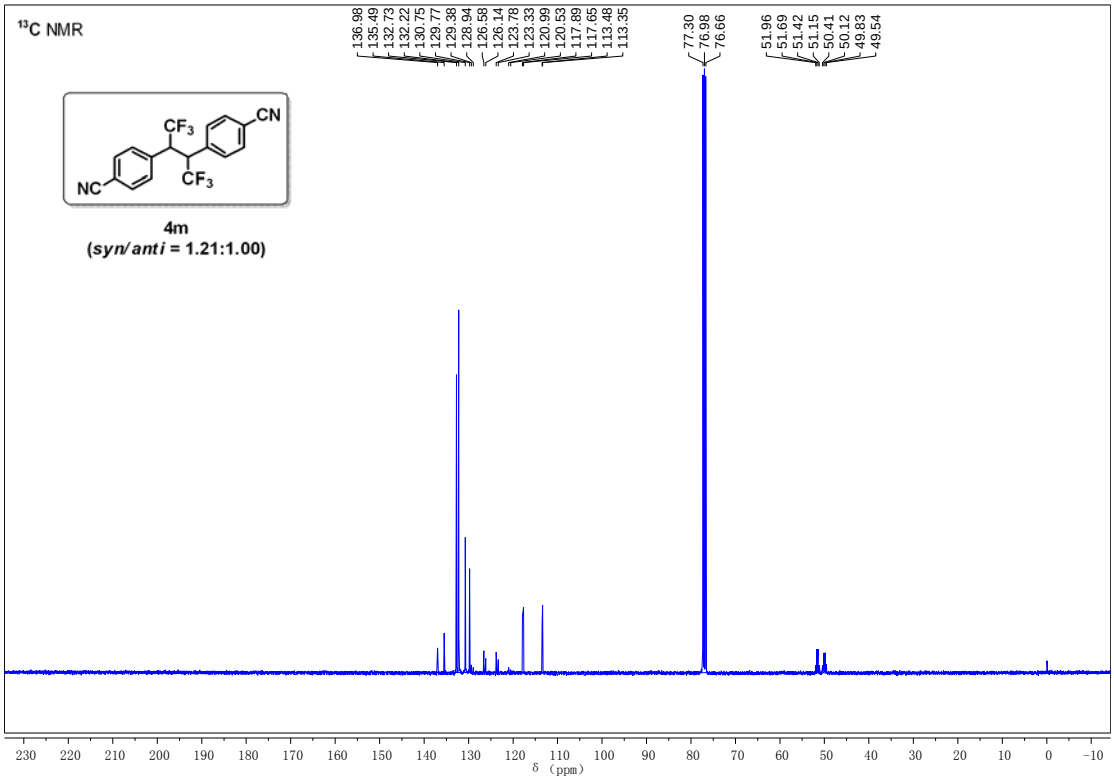

1H NMR
7.68
7.68
7.66
7.66
7.63
7.61
7.42
7.42
7.38
7.34
7.32
7.27
7.26
7.25
7.24
7.23
7.22
7.17
7.17
7.15
7.15
7.13
7.13
7.01
7.01
6.99
6.99
6.97
6.97
4.98
4.97
4.96
4.95
4.94
4.93
4.92
4.92
4.90
1.52
1.26
0.00
7.69
7.69
7.67
7.67
7.67
7.64
7.62
7.43
7.41
7.39
7.34
7.33
7.28
7.27
7.26
7.25
7.18
7.18
7.16
7.16
7.02
7.02
7.00
7.00
δ (ppm)
Br
CF3
CF3
Br
4k
(syn/anti = 1.03:1.00)
2.09
2.14
4.00
2.13
2.13
2.00
2.00
4.09
δ (ppm)

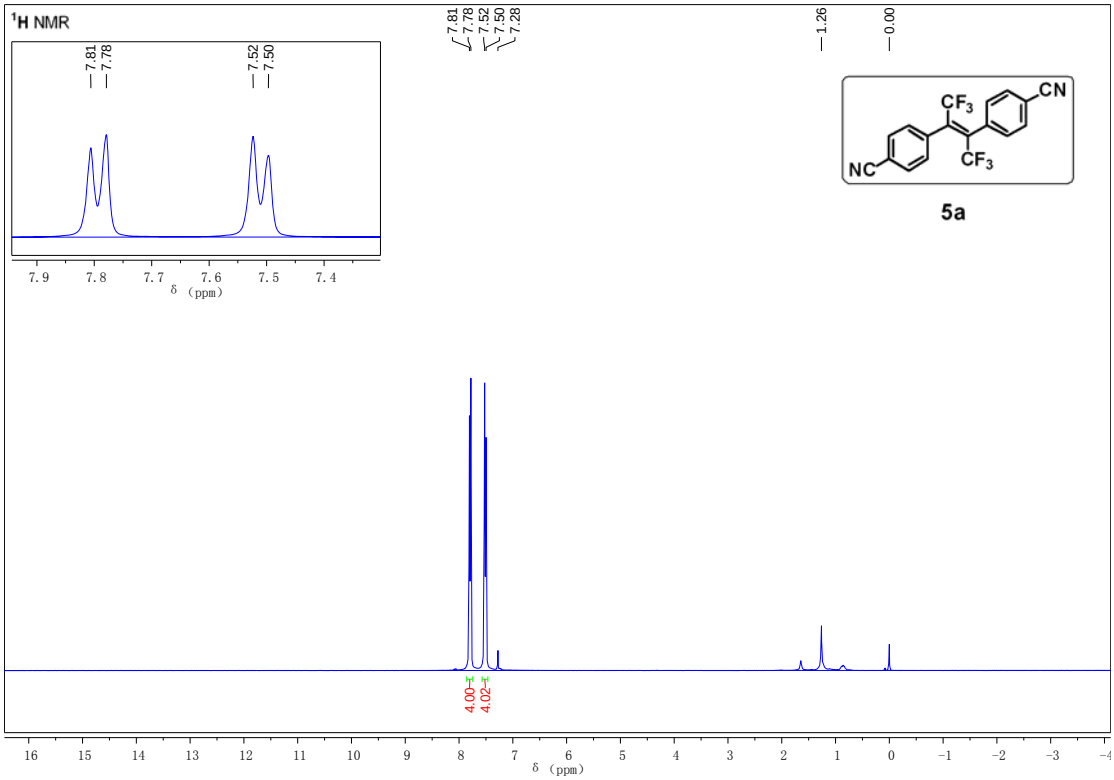

19F NMR
-63.43
-63.45
-65.46
-65.47
-65.48
Br
CF3
CF3
Br
4k
(syn/anti = 1.03:1.00)
1.00
1.03
δ (ppm)

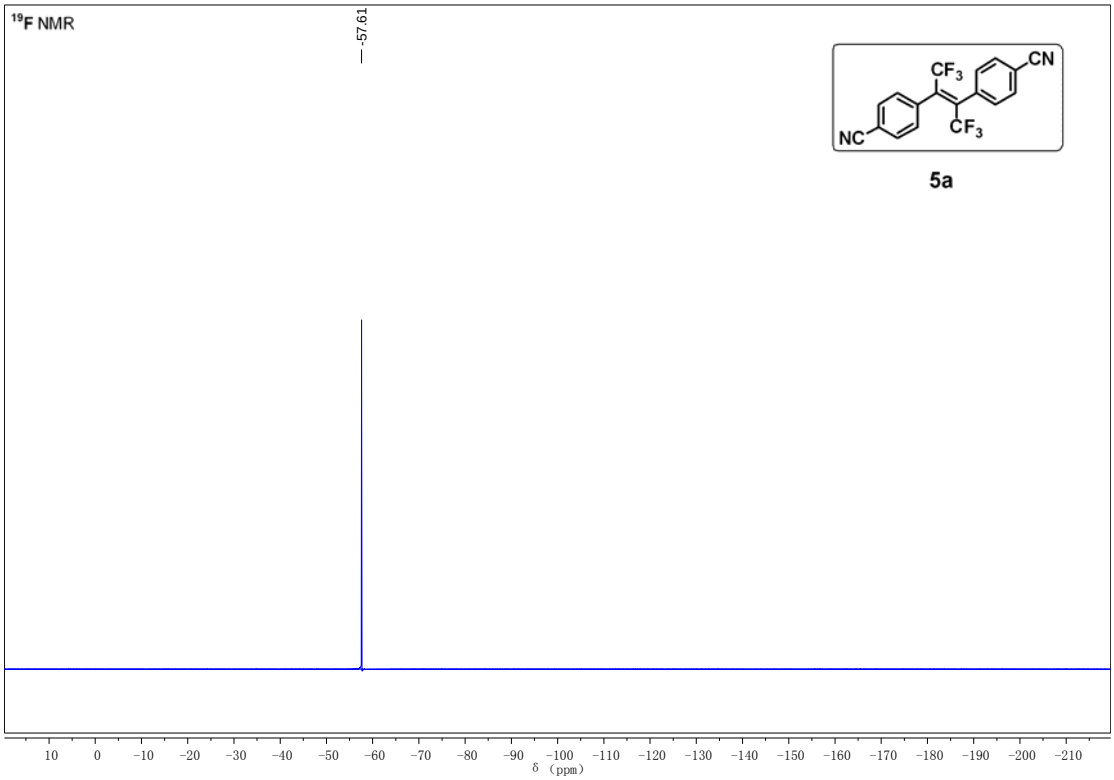

13C NMR
133.45
132.98
132.07
131.94
130.29
129.91
129.55
129.53
129.52
129.22
127.74
127.53
126.66
126.61
126.17
123.85
123.80
121.05
121.01
77.31
76.99
76.67
49.30
49.03
48.76
48.58
48.49
48.29
47.98
47.70
Br
CF3
CF3
Br
4k
(syn/anti = 1.03:1.00)
δ (ppm)

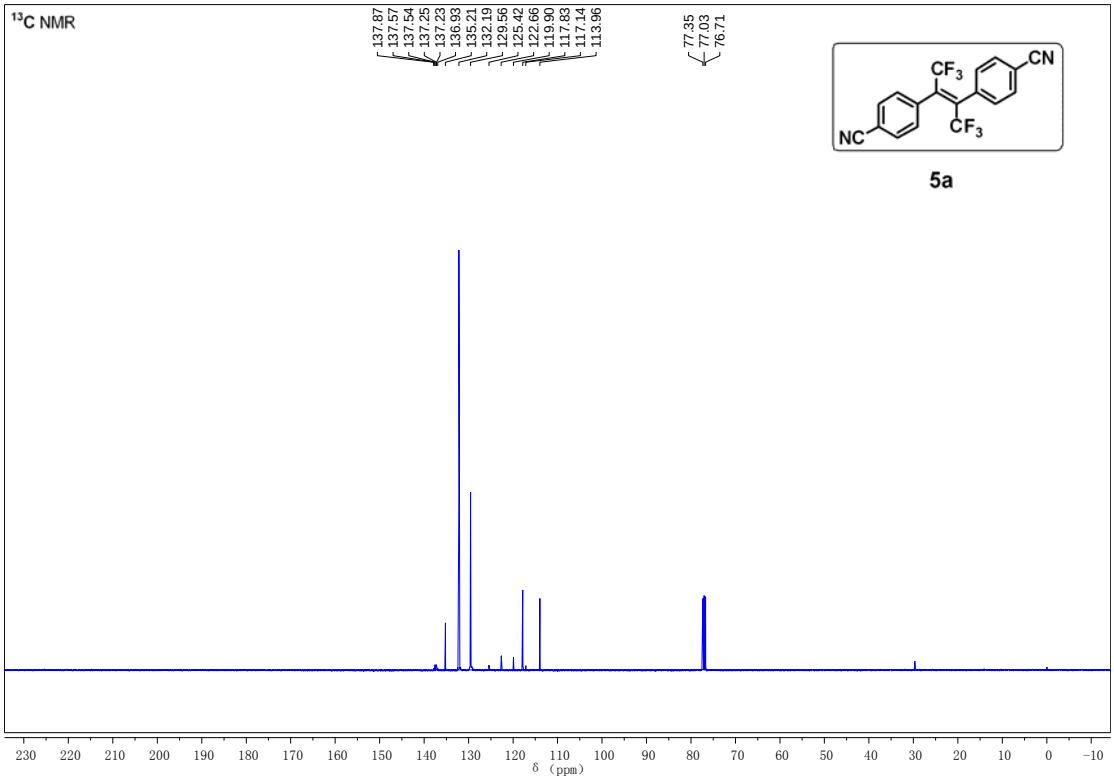

¹H NMR
7.76
7.74
7.56
7.55
7.54
7.53
7.26
7.08
7.06
4.18
4.16
4.16
4.15
4.14
4.12
4.08
4.07
4.06
4.05
4.05
4.03
0.00
CF3
CN
NC
CF3
4m
(syn/anti = 1.21:1.00)
4.00
8.85
4.85
2.42
2.00
δ (ppm)

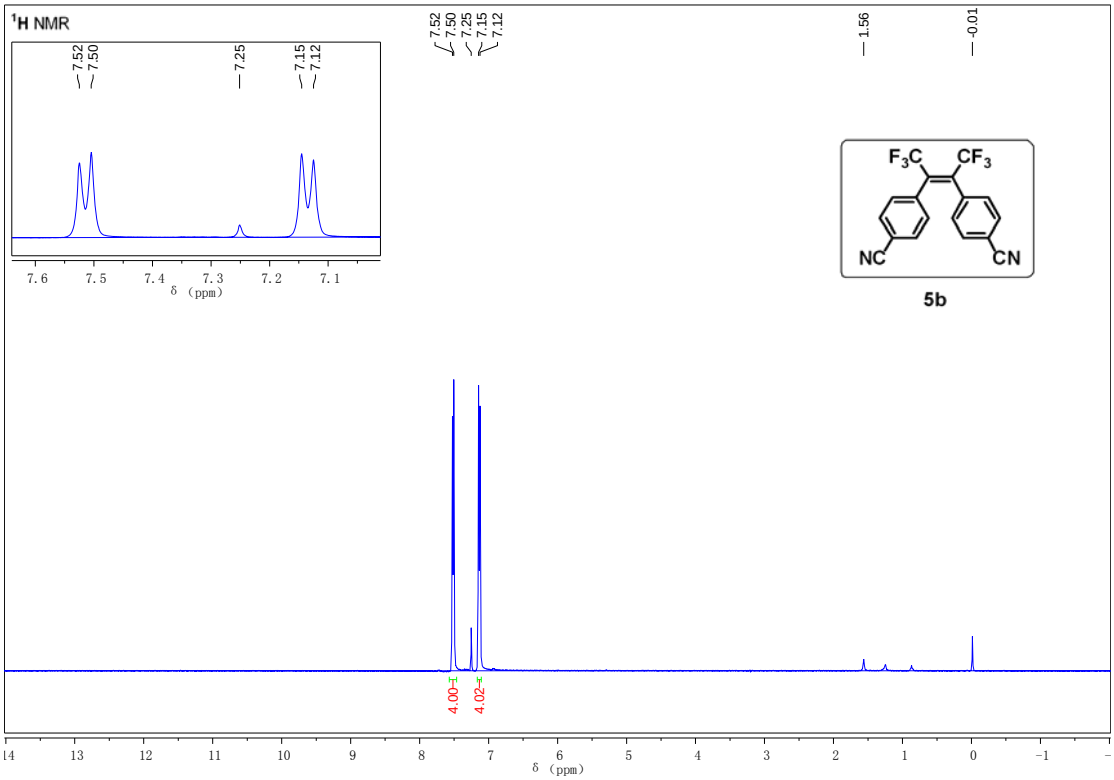

¹⁹F NMR
-62.81
-62.83
-65.57
-65.59
CF3
CN
NC
CF3
4m
(syn/anti = 1.21:1.00)
6.00
7.28
δ (ppm)

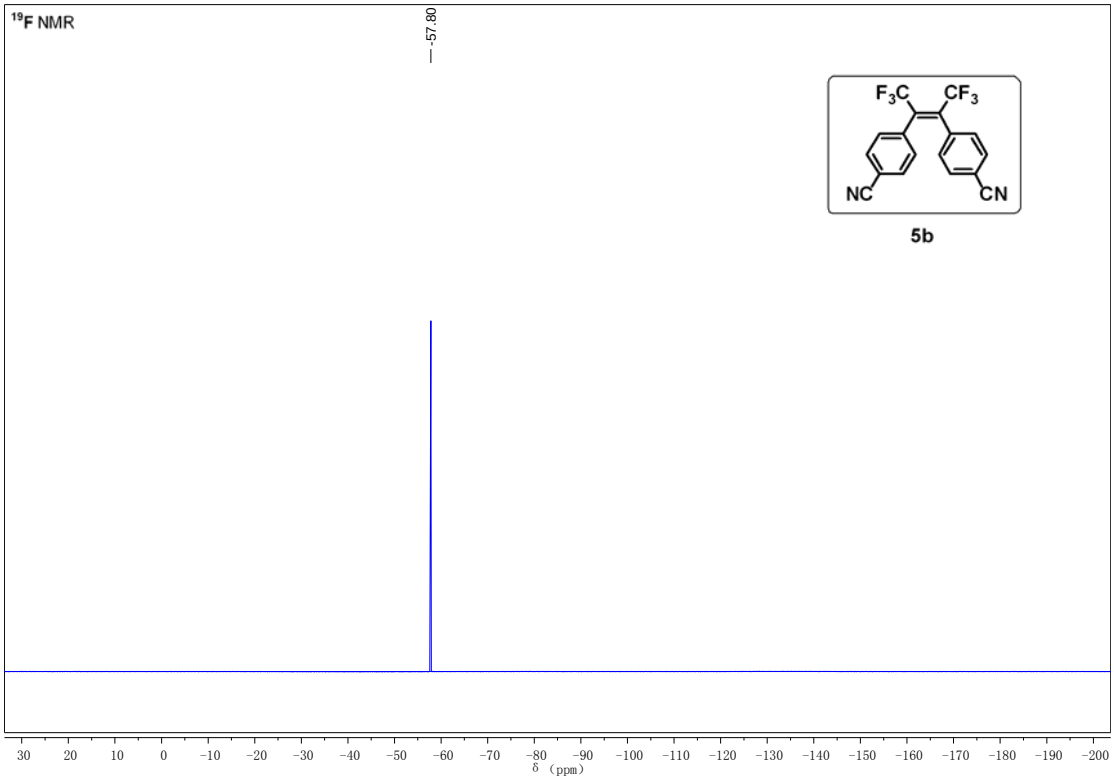

13C NMR
136.98
135.49
132.73
132.22
130.75
129.77
129.38
128.94
126.58
126.14
123.78
123.33
120.99
120.53
117.89
117.65
113.48
113.35
77.30
76.98
76.66
51.96
51.69
51.42
51.15
50.41
50.12
49.83
49.54
CF3
CN
NC
CF3
4m
(syn/anti = 1.21:1.00)
δ (ppm)

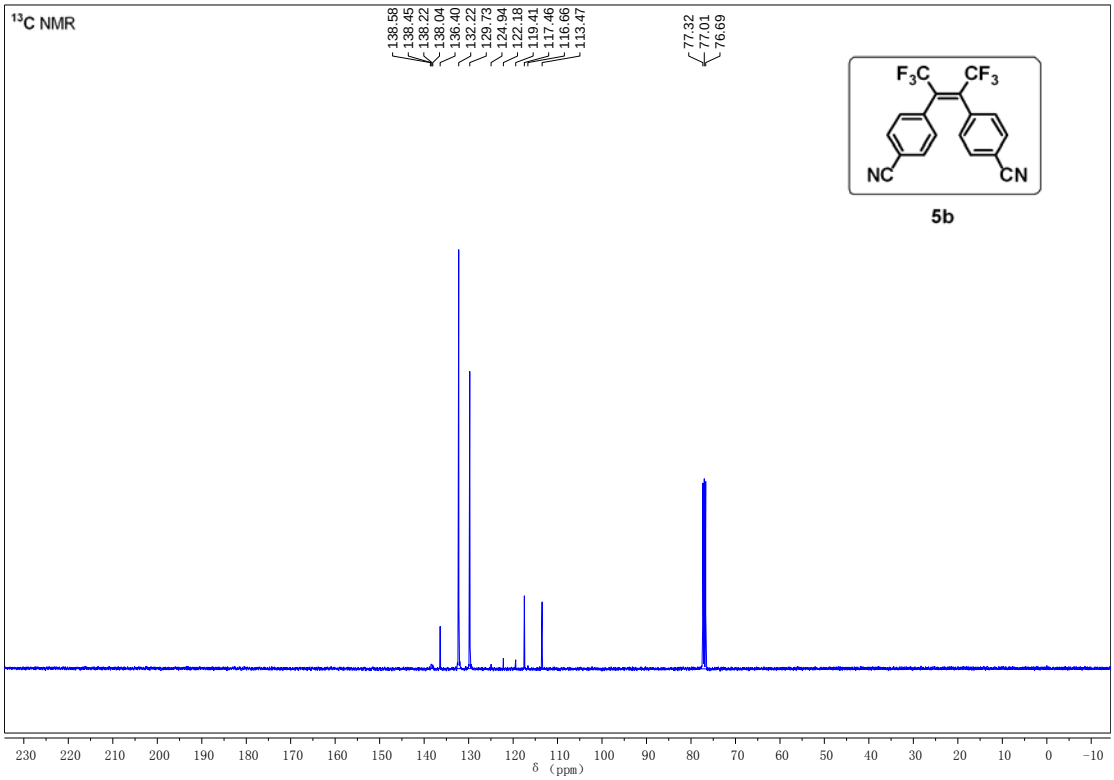

1H NMR
7.81
7.78
7.52
7.50
7.28
1.26
0.00
7.81
7.78
7.52
7.50
CF3
CN
NC
CF3
5a
4.00
4.02
δ (ppm)

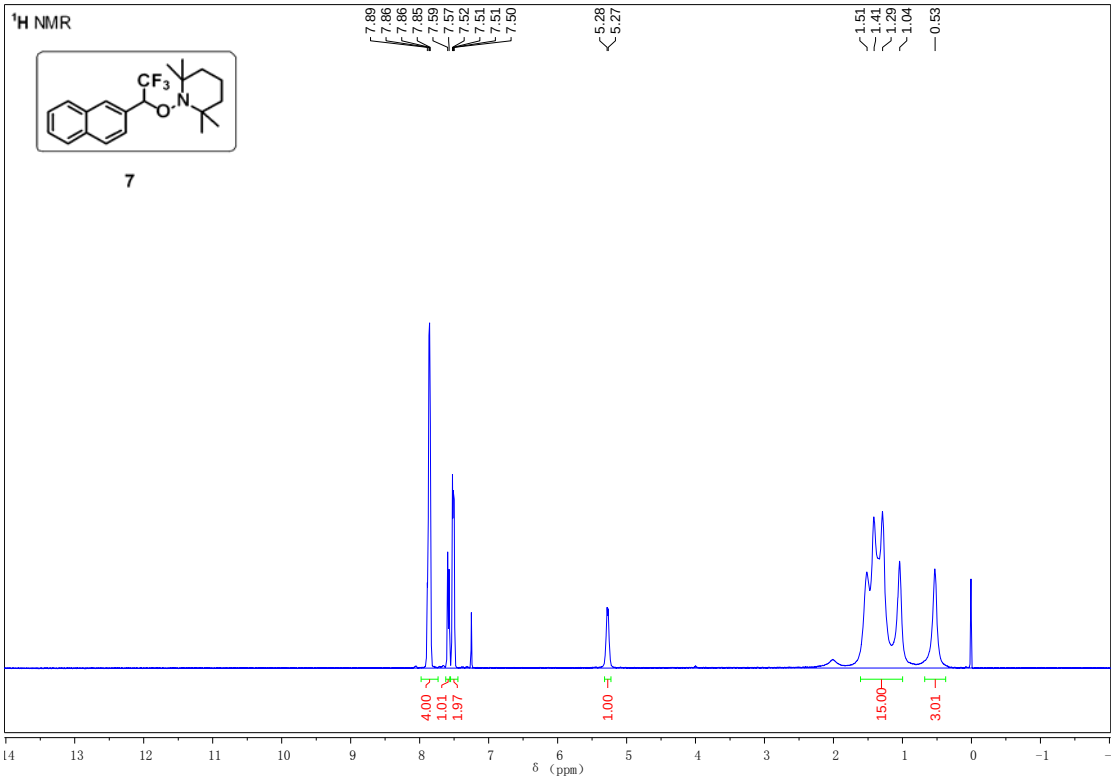

19F NMR
-57.61
CF3
CN
NC
CF3
5a
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
δ (ppm)

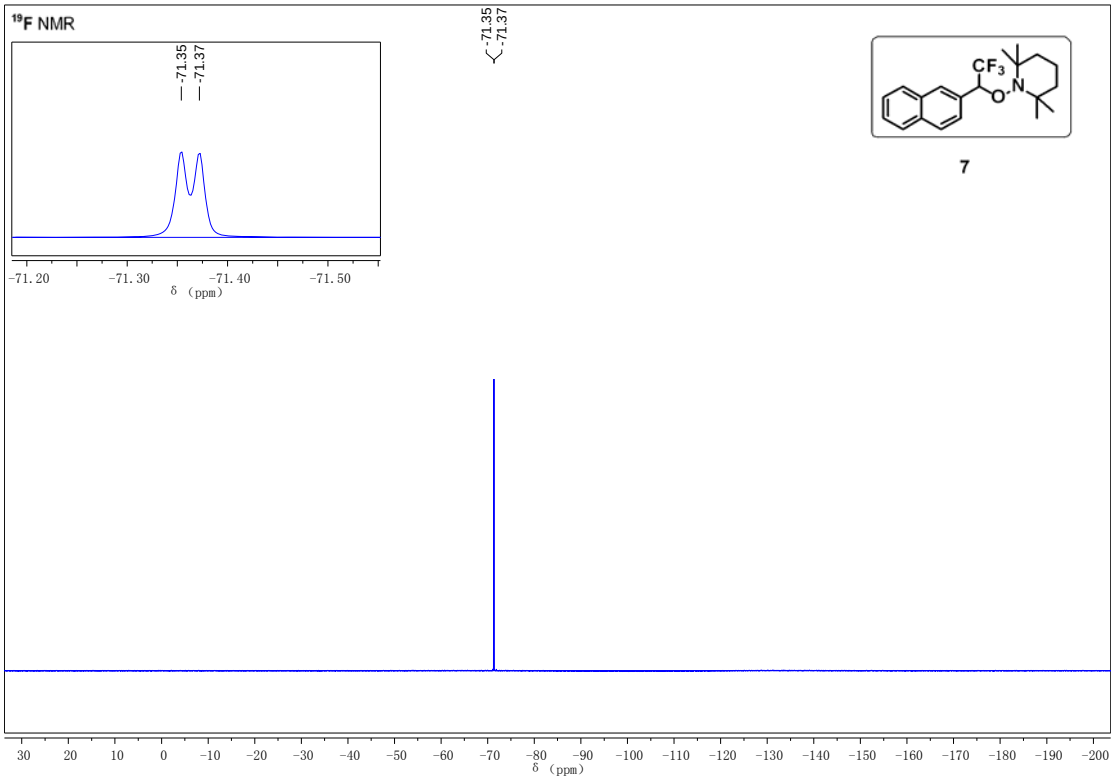

13C NMR
137.87
137.57
137.54
137.25
137.23
136.93
135.21
132.19
129.56
125.42
122.66
119.90
117.83
117.14
113.96
77.35
77.03
76.71
CF3
CN
NC
CF3
5a
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
δ (ppm)

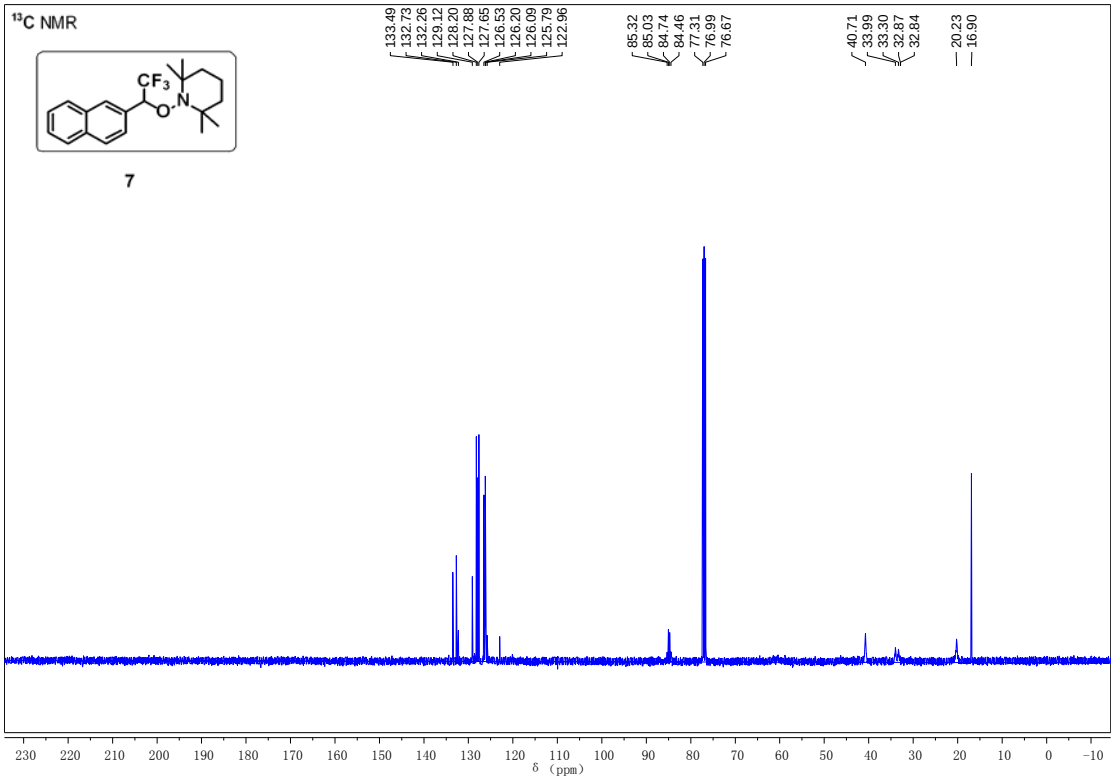

1H NMR
7.52
7.50
7.25
7.15
7.12
1.56
-0.01
F3C
CF3
NC
CN
5b
4.00
4.02
δ (ppm)


19F NMR
-57.80
F3C
CF3
NC
CN
5b
δ (ppm)


13C NMR
138.58
138.45
138.22
138.04
136.40
132.22
129.73
124.94
122.18
119.41
117.46
116.66
113.47
77.32
77.01
76.69
F3C
CF3
NC
CN
5b
δ (ppm)


1H NMR
7.89
7.86
7.86
7.85
7.59
7.57
7.52
7.51
7.51
7.50
5.28
5.27
1.51
1.41
1.29
1.04
0.53
CF3
O
N
7
4.00
1.01
1.97
1.00
15.00
3.01
δ (ppm)


19F NMR
-71.35
-71.37
-71.20
-71.30
-71.40
-71.50
δ (ppm)
CF3
O
N
7
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
δ (ppm)


13C NMR
133.49
132.73
132.26
129.12
128.20
127.88
127.65
126.53
126.20
126.09
125.79
122.96
85.32
85.03
84.74
84.46
77.31
76.99
76.67
40.71
33.99
33.30
32.87
32.84
20.23
16.90
CF3
O
N
7
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
δ (ppm)