Supporting Information

Iron-Catalyzed [2+2+2] Cyclotrimerization and Carbocyclization Reactions Using Trifluoromethyl Group Substituted Unsymmetrical Internal Alkynes

Maki Minakawa,* Tomoki Ishikawa, Junya Namioka, Souichirou Hirooka, Biao Zhou, and Motoi Kawatsura*

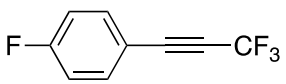
Department of Chemistry, College of Humanities & Sciences, Nihon University, Sakurajosui, Setagaya-ku, Tokyo 156-8550, Japan

minakawa@chs.nihon-u.ac.jp
kawatsur@chs.nihon-u.ac.jp

Contents of Supporting Information:

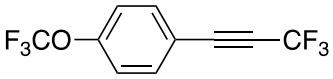
General and Materials: All manipulations were carried out under a nitrogen atmosphere. All the chemicals ($FeCl_2$, FeI_2, $FeBr_2$, $Fe(OTf)_2$, $FeCl_3$, DPPP, DPPE, DPPB, PPh_3, 1,6-diyne **4c**, 1,6-diyne **4d**, and other reagents include solvents) were purchased from common commercial sources, unless otherwise noted, and used without further purification. All trifluoromethylated alkynes[S1] **1a-m**, 1,6-diynes[S2] **4a**, **4b**, and **4e-g** were prepared according to the literatures. Thin layer chromatography (TLC) was performed on plates coated with 0.25 mm thick Silica Gel 60 F-254 (Merck Ltd.). Column chromatography was performed using neutral Silica Gel 60 N (Kanto Chemical co., Ltd.). The nuclear magnetic resonance (NMR) spectra were recorded on a JEOL EX-270 spectrometer (270 MHz for ^1H), JEOL JNM ECA-500 spectrometer (500 MHz for ^1H, 125 MHz for ^{13}C, and 470 MHz for ^{19}F) and Bruker Biospin AVANCE Ⅱ 600 (150 MHz for ^{13}C). Chemical shifts (δ) are given in ppm relative to $SiMe_4$ (0.00 ppm) in $CDCl_3$ for ^1H NMR spectra, $CDCl_3$ (77.0 ppm) or CD_3COCD_3 (29.8 ppm) for ^{13}C NMR, and C_6F_6 (0.00 ppm) in $CDCl_3$ for ^{19}F NMR. ^1H, ^{13}C, and ^{19}F NMR spectra were recorded in $CDCl_3$ or CD_3COCD_3 at 25 °C unless otherwise noted. The absolute values of the coupling constants are given in Hertz (Hz), regardless of their signs. Multiplicities are abbreviated as singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m). IR spectra were obtained using a JASCO FT/IR-4100 spectrophotometer. Melting points were determined using Yanaco micro melting point apparatus MP-500P and are uncorrected. ESI mass spectra were recorded on JEOL AccuTOFLC-plus JMS T100LP spectrometer.

Characterization of trifluoromethylated alkynes 1a-m: All alkynes **1a-m** were prepared according to Konno's procedure.[S1a] The spectral data for compounds are in agreement with previously reported values.[S1] New compounds **1b**, **1e**, and **1k** were characterized as follows:

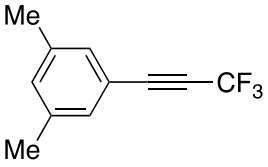


1-(4-fluorophenyl)-3,3,3-trifluoropropyne (1b): Colorless oil. ^1H NMR (500 MHz, $CDCl_3$): δ 7.09 (d, J = 8.6 Hz, 2H), 7.55 (dd, J = 5.7, 8.6 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 75.6 (q, J_{CF} = 52.8 Hz), 85.5 (q, J_{CF} = 6.8 Hz), 114.7, 114.8 (q, J_{CF} = 257.1 Hz), 116.2 (d, J_{CF} = 21.6 Hz), 134.7

(d, J_{CF} = 10.8 Hz), 164.1 (d, J_{CF} = 254.3 Hz). ^{19}F NMR (470 MHz, $CDCl_3$): δ 55.7-55.8 (m, 1F), 111.9 (s, 3F). IR (neat) 2930, 2258, 1603, 1509, 1316, 1240, 1156 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_9H_5F_4^+$ $[M+H]^+$ 189.0327, found 189.0307.

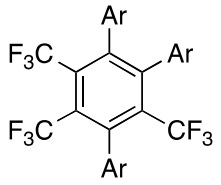


1-(4-trifluoromethoxyphenyl)-3,3,3-trifluoropropyne (1e): Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.23 (d, J = 8.3 Hz, 2H), 7.58 (d, J = 8.3 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 76.5 (q, J_{CF} = 53.2 Hz), 84.9 (q, J_{CF} = 6.4 Hz), 114.8 (q, J_{CF} = 257.1 Hz), 117.2, 120.4 (q, J_{CF} = 258.7 Hz), 121.0, 134.3, 151.0. ^{19}F NMR (470 MHz, $CDCl_3$): δ 104.2 (s, 3F), 111.8 (s, 3F). IR (neat) 2931, 2260, 1608, 1509, 1318, 1259, 1213, 1164, 1139 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{10}H_4F_6O$ [M] 254.0166, found 254.0166.



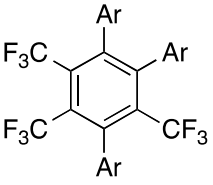
1-(3,5-dimethylphenyl)-3,3,3-trifluoropropyne (1k): Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.29 (s, 6H), 7.07 (s, 1H) 7.14 (s, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 20.9, 75.0 (q, J_{CF} = 52.2 Hz), 87.2 (q, J_{CF} = 6.4 Hz), 115.0 (q, J_{CF} = 256.7 Hz), 118.2, 130.1, 132.8, 138.4. ^{19}F NMR (470 MHz, $CDCl_3$): δ 112.33 (s, 3F). IR (neat) 2925, 2242, 1600, 1467, 1382, 1340, 1250, 1145 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{11}H_9F_3Na^+$ $[M+Na]^+$ 221.0549, found 221.0582.

General Procedure for the iron catalyzed [2+2+2] cyclotrimerization of trifluoromethylated alkynes 1a-m: A typical procedure is given for the reaction of **1a** (Table 1, entry 5). In a nitrogen-filled glove box, a solution of $FeCl_2$ (1.3 mg, 0.01 mmol), dppp (8.2 mg, 0.02 mmol) in anhydrous CH_3CN (0.33 mL) was stirred at room temperature for 10 min. Subsequently Zn dust (39.2 mg, 0.60 mmol), ZnI_2 (4.8 mg, 0.015 mmol) and **1a** (36.8 mg, 0.2 mmol) were added to the mixture at 80 ºC, and stirred for 36 h. The reaction mixture was filtered through Celite and Florisil. The combined organic layers were evaporated. The residue was chromatographed on silica gel (hexane/diethyl ether = 9/1) to give 32 mg (87%) of **2a** and **3a**. The ratio of the products was determined using ^{19}F NMR of the crude materials to be **2a**/**3a** = 95/5. The peak at 115 to 116 ppm in the ^{19}F NMR spectra is minor (symmetric) product.



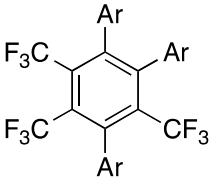
2a: Ar = 4-MeC_6H_4

Benzene derivative 2a[S3]**:** Isolated yield: 87% (Table 1, Entry 5). White solid. Mp 210-213 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.22 (s, 3H), 2.24 (s, 3H), 2.43 (s, 3H), 6.83-6.93 (m, 8H), 7.21-7.26 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.1, 21.2, 21.4, 122.7 (q, J_{CF} = 278.0 Hz), 122.8 (q, J_{CF} = 277.0 Hz), 123.0 (q, J_{CF} = 278.3 Hz), 127.5, 127.6, 128.0, 129.6, 129.7, 129.9 (q, J_{CF} = 30.7 Hz), 130.4, 131.8 (q, J_{CF} = 31.7 Hz), 132.5 (q, J_{CF} = 26.9 Hz), 133.1, 133.3, 133.6, 137.0, 137.2, 138.3, 141.9, 144.7, 144.8. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.7 (q, J = 17.3 Hz, 3F), 111.99 (q, J = 17.3 Hz, 3F), 113.7 (s, 3F). IR (KBr) 2999, 1515, 1412, 1341, 1175, 1036 cm^{-1}.



2b: Ar = 4-FC_6H_4

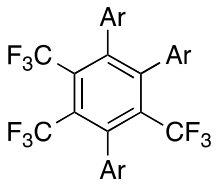
Benzene derivative 2b: Isolated yield: 96% (Table 2, Entry 1). White solid. Mp 168-171 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.80-6.88 (m, 4H), 6.88-6.98 (m, 4H), 7.09-7.17 (m, 2H), 7.30-7.38 (m, 2H). ^{13}C NMR (125 MHz, acetone-d6): δ 114.66, 114.70, 114.8, 114.9, 115.2, 115.4, 123.7 (q, J_{CF} = 276.7 Hz), 123.8 (q, J_{CF} = 276.3 Hz), 124.0 (q, J_{CF} = 277.9 Hz), 130.6 (q, J_{CF} = 30.8 Hz), 132.4 (q, J_{CF} = 31.2 Hz), 132.7 (d, J_{CF} = 8.4 Hz), 132.9 (d, J_{CF} = 8.4 Hz), 133.4 (q, J_{CF} = 27.6 Hz), 133.6 (d, J_{CF} = 8.4 Hz), 142.0, 145.1, 147.1, 162.9 (d, J_{CF} = 245.9 Hz), 163.0 (q, J_{CF} = 245.9 Hz), 163.9 (q, J_{CF} = 245.9 Hz). ^{19}F NMR (470 MHz, $CDCl_3$): δ 48.3-48.4 (m, 1F), 48.6-48.6 (m, 1F), 49.1-49.2 (m, 1F), 110.7 (q, J = 16.5 Hz, 3F), 111.9 (q, J = 16.5 Hz, 3F), 113.8 (s, 3F). IR (KBr) 3079, 1898, 1607, 1341, 1323, 1232 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{27}H_{11}F_{12}^-$ $[M–H]^-$ 563.0669, found 563.0669.



2c: Ar = 4-ClC_6H_4

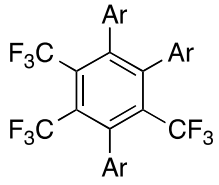
Benzene derivative 2c[S3]**:** Isolated yield: 96% (Table 2, Entry 2). White solid. Mp 290 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.88 (d, J = 8.2 Hz, 2H), 6.92 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 8.7 Hz, 2H), 7.15 (d, J = 8.2 Hz, 2H), 7.29 (d, J = 8.7 Hz, 2H), 7.42 (d, J = 8.2 Hz, 2H). ^{13}C NMR (150 MHz, acetone-d6): δ122.1 (q, J_{CF} = 276.1 Hz), 123.6 (q, J_{CF} = 274.9 Hz), 123.7 (q, J_{CF} = 274.9 Hz), 128.0, 128.1, 128.5, 130.6 (q, J_{CF} = 30.6 Hz), 132.3, 132.3 (q, J_{CF} = 30.8 Hz), 132.5, 133.2 (q, J_{CF} = 27.3 Hz), 133.2, 134.2, 134.4, 135.3, 135.5, 135.8, 136.1, 141.9, 144.6, 146.6. ^{19}F NMR (470 MHz,

$CDCl_3$): δ 110.7 (q, J = 17.3 Hz, 3F), 111.9 (q, J = 17.3 Hz, 3F), 113.8 (s, 3F). IR (KBr) 2964, 2105, 2078, 2022, 1597, 1495, 1414, 1238, 1174, 1093, 1031 cm^{-1}.



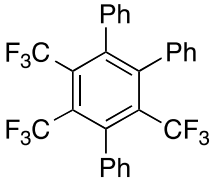
2d: Ar = 4-BrC_6H_4

Benzene derivative 2d: Isolated yield: 86% (Table 2, Entry 3). White solid. Mp 210-217 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.82 (d, J = 8.0 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 7.23 (d, J = 8.6 Hz, 2H), 7.30 (d, J = 6.3 Hz, 2H), 7.31 (d, J = 6.3 Hz, 2H), 7.57 (d, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz, acetone-d6): δ 122.5, 122.8, 123.63, 123.64 (q, J_{CF} = 276.7 Hz), 123.8 (q, J_{CF} = 276.7 Hz), 123.9 (q, J_{CF} = 278.3 Hz), 130.8 (q, J_{CF} = 31.2 Hz), 131.10, 131.14, 131.5, 132.5 (q, J_{CF} = 31.2 Hz), 132.7, 132.8, 133.2 (q, J_{CF} = 27.6 Hz), 133.5, 136.0, 136.3, 136.6, 142.0, 144.7, 146.6. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.8 (q, J = 16.5 Hz, 3F), 112.0 (q, J = 16.5 Hz, 3F), 113.9 (s, 3F). IR (KBr) 3439, 2965, 2317, 1717, 1590, 1491, 1412, 1240, 1174, 1012 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{27}H_{12}Br_3F_9$ [M] 743.8345, found 743.8321.



2e: Ar = 4-$CF_3OC_6H_4$

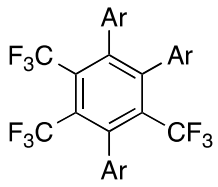
Benzene derivative 2e: Isolated yield: 80% (Table 2, Entry 4). White solid. Mp 155-160 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.91-7.00 (m, 8H), 7.27 (d, J = 8.0 Hz, 2H), 7.39 (d, J = 8.6 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 119.68, 119.73, 120.5 (q, J_{CF} = 257.9 Hz), 120.6 (q, J_{CF} = 269.9 Hz), 120.7 (q, J_{CF} = 256.7 Hz), 122.4 (q, J_{CF} = 277.1 Hz), 122.5 (q, J_{CF} = 277.1 Hz), 122.7 (q, J_{CF} = 279.9 Hz), 130.9 (q, J_{CF} = 22.8 Hz), 131.2, 131.3, 131.9, 132.4 (q, J_{CF} = 26.4 Hz), 132.9 (q, J_{CF} = 27.6 Hz), 133.9, 134.7, 134.8, 141.3, 143.9, 145.4, 148.9, 149.0, 149.1, 149.9. ^{19}F NMR (470 MHz, $CDCl_3$): δ 103.6 (s, 3F), 103.7 (s, 3F), 104.1 (s, 3F), 110.9 (q, J = 16.5 Hz, 3F), 112.1 (q, J = 16.5 Hz, 3F), 114.0 (s, 3F). IR (KBr) 3430, 3116, 1612, 1513, 1325, 1252, 1174, 1033 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{30}H_{11}F_{18}O_3^-$ $[M–H]^-$ 761.0421, found 761.0429.



2f

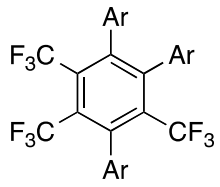
Benzene derivative 2f[S4]**:** Isolated yield: 79% (Table 2, Entry 5). White solid. Mp 218-221 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.98 (m, 4H), 7.09-7.12 (m, 6H), 7.38-7.45 (m, 5H). ^{13}C NMR (125

MHz, $CDCl_3$): δ 122.6 (q, J_{CF} = 278.0 Hz), 122.7 (q, J_{CF} = 277.3 Hz), 122.9 (q, J_{CF} = 278.6 Hz), 126.8, 126.9, 127.3, 127.4, 127.6, 128.5, 129.7, 129.8, 129.9, 130.0 (q, J_{CF} = 30.7 Hz), 131.8 (q, J_{CF} = 30.7 Hz), 132.3 (q, J_{CF} = 27.9 Hz), 135.9, 136.2, 136.4, 141.9, 144.6, 146.4. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.7 (q, J = 17.3 Hz, 3F), 112.0 (q, J = 17.3 Hz, 3F), 113.8 (s, 3F). IR (KBr) 3061, 2103, 2075, 1602, 1495, 1233, 1182, 1060 cm^{-1}.



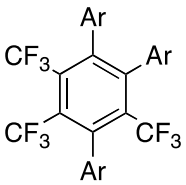
2g: Ar = 4-PhC_6H_4

Benzene derivative 2g[S3]**:** Isolated yield: 62% (Table 2, Entry 6). White solid. Mp >300 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.07 (d, J = 8.2 Hz, 2H), 7.10 (d, J = 8.2 Hz, 2H), 7.27-7.31 (m, 2H), 7.35-7.38 (m, 9H), 7.45-7.49 (m, 8H), 7.67-7.70 (m, 4H). ^{13}C NMR (150 MHz, $CDCl_3$): 122.7 (q, J_{CF} = 277.3 Hz), 122.8 (q, J_{CF} = 277.3 Hz), 125.5, 125.6, 125.94, 126.98, 127.0, 127.2, 127.4, 127.5, 127.6, 128.69, 128.70, 128.8, 130.0 (q, J_{CF} = 278.9 Hz), 130.2, 130.3, 130.4 (q, J_{CF} = 30.8 Hz), 130.9, 131.9 (q, J_{CF} = 30.8 Hz), 132.5 (q, J_{CF} = 27.1 Hz), 134.8, 135.2, 135.4, 140.15, 140.21, 140.23, 140.27, 140.31, 141.2, 141.8, 144.4, 146.3. ^{19}F NMR (470 MHz, $CDCl_3$ internal standard; C_6F_6): δ 110.60 (q, J = 17.3 Hz, 3F), 111.84 (q, J = 17.3 Hz, 3F), 113.57 (s, 3F). IR (KBr) 3032, 1488, 1409, 1325, 1238, 1179, 1008 cm^{-1}.



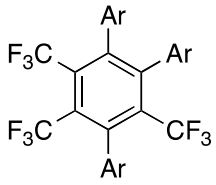
2h: Ar = 4-$MeOC_6H_4$

Benzene derivative 2h[S3]**:** Isolated yield: 73% (Table 2, Entry 7). White solid. Mp 300 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.73 (s, 3H), 3.74 (s, 3H), 3.87 (s, 3H), 6.65 (d, J = 8.7 Hz, 2H), 6.66 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 8.7 Hz, 2H), 6.90 (d, J = 8.7 Hz, 2H), 6.95 (d, J = 8.7 Hz, 2H), 7.27 (d, J = 8.7 Hz, 2H). ^{13}C NMR (150 MHz, acetone-d6): δ 55.3, 55.4, 55.6, 113.10, 113.12, 113.7, 123.9 (q, J_{CF} = 275.5 Hz), 124.0 (q, J_{CF} = 275.5 Hz), 124.2 (q, J_{CF} = 277.7 Hz), 129.0, 129.4, 129.8, 130.1 (q, J_{CF} = 29.7 Hz), 131.9, 132.0, 132.3 (q, J_{CF} = 31.4 Hz), 132.8, 133.5 (q, J_{CF} = 26.4 Hz), 142.6, 145.8, 148.2, 159.8, 159.9, 160.9. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.6 (q, J = 17.3 Hz, 3F), 111.8 (q, J = 17.3 Hz, 3F), 113.6 (s, 3F). IR (KBr) 2937, 2839, 1613, 1465, 1414, 1325, 1249, 1159, 1036 cm^{-1}.



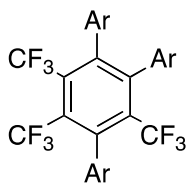
2i: Ar = 4-^{t}BuC$_6$H$_4$

Benzene derivative 2i: Isolated yield: 65% (Table 2, Entry 8). White solid. Mp 236-243 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.18 (s, 9H), 1.19 (s, 9H), 1.37 (s, 9H), 6.81 (d, J = 8.0 Hz, 2H), 6.83 (d, J = 8.0 Hz, 2H), 7.04 (d, J = 3.4 Hz, 2H), 7.06 (d, J = 3.4 Hz, 2H) 7.29 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 31.2, 31.3, 31.4, 34.3, 34.4, 34.7, 122.7 (q, J_{CF} = 277.1 Hz), 122.8 (q, J_{CF} = 277.1 Hz), 123.0 (q, J_{CF} = 278.9 Hz), 123.42, 123.44, 124.1, 129.4, 129.8 (q, J_{CF} = 30.0 Hz), 130.1, 131.5 (q, J_{CF} = 30.0 Hz), 132.2 (q, J_{CF} = 27.2 Hz), 132.9, 133.5, 133.7, 141.9, 144.9, 145.0, 146.9, 149.9, 150.1, 151.4. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.9 (q, J = 17.1 Hz, 3F), 112.1 (q, J = 17.1 Hz, 3F), 113.9 (s, 3F). IR (KBr) 2966, 2906, 2870, 1907, 1411, 1325, 1237, 1164, 1134, 1102 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{39}H_{39}F_9Na^+$ $[M+Na]^+$ 701.2806, found 701.2801.



2j: Ar = 3-MeC$_6$H$_4$

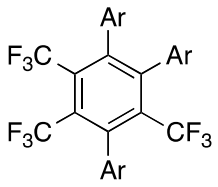
Benzene derivative 2j[S3]**:** Isolated yield: 92% (Table 2, Entry 9). White solid. Mp 55-60 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.17-2.18 (m, 3H), 2.20-2.22 (m, 3H), 2.41 (s, 3H), 6.70-6.83 (m, 4H), 6.89-6.92 (m, 2H), 6.94-7.02 (m, 2H), 7.17-7.20 (m, 2H), 7.24-7.25 (m, 1H), 7.29-7.32 (m, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): 21.11, 21.13, 21.4, 122.7 (q, J_{CF} = 277.2 Hz), 122.8 (q, J_{CF} = 277.2 Hz), 123.0 (q, J_{CF} = 279.7 Hz), 126.4, 126.7, 127.0, 127.1, 127.6, 127.7, 128.0 (d, J_{CF} = 3.8 Hz), 128.2 (d, J_{CF} = 5.8 Hz), 129.2, 129.8 (q, J_{CF} = 30.7 Hz) 130.5, 130.8, 131.3, 131.5 (q, J_{CF} = 31.3 Hz), 131.6, 132.2 (q, J_{CF} = 27.2 Hz), 135.9, 136.1, 136.2 , 136.4, 136.9, 141.9, 144.6, 146.6. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.7 (q, J = 19.2 Hz, 3F), 112.0 (q, J = 15.4 Hz, 3F), 113.7 (q, J = 13.4 Hz, 3F). IR (KBr) 2923, 1607, 1403, 1326, 1232, 1201, 1050 cm^{-1}.



2k: Ar = 3,5-MeC$_6$H$_3$

Benzene derivative 2k: Isolated yield: 83% (Table 2, Entry 10). White solid. Mp 150-154 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.15 (s, 6H), 2.16 (s, 6H), 2.36 (s, 6H), 6.55-6.56 (m, 2H), 6.58-6.59 (m, 2H), 6.69 (s, 1H), 6.71 (s, 1H), 6.98-6.99 (m, 2H), 7.06

(s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 20.92, 20.94, 21.2, 122.8 (q, J_{CF} = 276.7 Hz), 122.9 (q, J_{CF} = 275.9 Hz), 123.1 (q, J_{CF} = 279.1 Hz), 127.7, 127.87, 127.88, 128.6, 128.7, 128.8, 129.5 (q, J_{CF} = 31.2 Hz), 130.0, 131.4 (q, J_{CF} = 31.2 Hz), 132.0 (q, J_{CF} = 27.2 Hz), 135.81, 135.88, 135.9, 136.2, 136.6, 141.9, 144.7, 146.8. ^{19}F NMR (470 MHz, $CDCl_3$): δ 110.8 (q, J = 16.5 Hz, 3F), 112.1 (q, J = 16.5 Hz, 3F), 113.8 (s, 3F). IR (KBr) 2921, 1605, 1336, 1168, 1078 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{33}H_{27}F_9Na^+$ $[M+Na]^+$ 617.1861, found 617.1844.

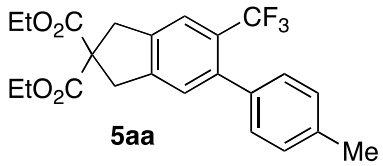


2l: Ar = 3-MeOC$_6$H$_4$

Benzene derivative 2l: Isolated yield: 84% (Table 2, Entry 11). White solid. Mp 57-61 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.63-3.65 (m, 3H), 3.66-3.68 (m, 3H), 3.81 (s, 3H), 6.48-6.70 (m, 6H), 6.93-7.07 (m, 5H), 7.32 (t, J = 8.0 Hz, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 55.1, 55.22, 55.23, 113.2, 113.6, 114.1, 114.2, 115.8, 116.0, 116.5, 116.6, 122.6, 122.6 (q, J_{CF} = 277.1 Hz), 122.7 (q, J_{CF} = 277.1 Hz), 122.9 (q, J_{CF} = 279.5 Hz), 123.4, 127.8, 127.9, 128.3, 129.9 (q, J_{CF} = 30.8 Hz), 131.7 (q, J_{CF} = 30.0 Hz), 132.2 (q, J_{CF} = 27.6 Hz), 137.2, 137.4, 141.7, 144.2, 146.1, 158.0, 158.1, 158.6. ^{19}F NMR (470 MHz, $CDCl_3$): δ 111.0 (q, J = 16.5 Hz, 3F), 112.2 (q, J = 17.1 Hz, 3F), 113.9 (d, J = 9.2 Hz, 3F). IR (KBr) 3066, 3005, 2943, 2837, 1602, 1581, 1488, 1431, 1327, 1236, 1165, 1052 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{30}H_{22}F_9O_3^+$ $[M+H]^+$ 601.1425, found 601.1443.

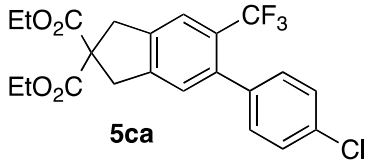
General Procedure for the iron catalyzed [2+2+2] cycloaddition of trifluoromethylated alkynes 1 with diynes 4a-g: A typical procedure is given for the reaction of **1c** with **4a** (Table 3, entry 3). A solution of FeI_2 (6.2 mg, 0.02 mmol), Zn dust (13.1 mg, 0.2 mmol), 1,6-diyne **4a** (23.6 mg, 0.1 mmol), and alkyne **1c** (61.4 mg,

0.3 mmol) in CH_3CN (0.1 mL) was stirred at 80 ºC for 12 h. The reaction mixture was filtered through Celite and Florisil. The combined organic layers were evaporated. The residue was chromatographed on silica gel (hexane/ethyl acetate = 9/1) to give 40 mg (90%) of **5ca**.



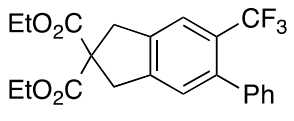
5aa

Diethyl 5-(*p*-tolyl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5aa): Isolated yield: 92% (Table 4, Entry 1). Pale yellow oil. ^{1}H NMR (270 MHz, $CDCl_3$): δ 1.27 (t, J = 7.0 Hz, 6H), 2.38 (s, 3H), 3.64 (s, 2H), 3.67 (s, 2H), 4.23 (q, J = 7.0 Hz, 4H), 7.13 (s, 1H), 7.18 (s, 4H), 7.55 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 21.1, 40.1, 40.3, 60.3, 61.9, 121.8 (q, J_{CF} = 6.0 Hz), 124.3 (q, J_{CF} = 273.5 Hz), 127.4 (q, J_{CF} = 30.0 Hz), 127.9, 128.3, 128.8, 137.0, 137.1, 139.2, 140.6, 143.7, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.3 (s, 3F). IR (neat) 2983, 1732, 1621, 1573, 1490, 1236, 1189, 911 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{23}H_{23}F_3NaO_4^+$ $[M+Na]^+$ 443.1446, found 443.1427.



5ca

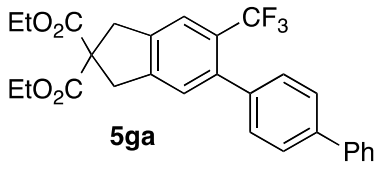
Diethyl 5-(4-chlorophenyl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5ca)[S5]**:** Isolated yield: 90% (Table 4, Entry 2). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, J = 7.0 Hz, 6H), 3.64 (s, 2H), 3.67 (s, 2H), 4.24 (q, J = 7.0 Hz, 4H), 7.11 (s, 1H), 7.22 (d, J = 8.0 Hz, 2H), 7.35 (d, J = 8.0 Hz, 2H), 7.56 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 40.1, 40.3, 60.3, 62.0, 121.9 (q, J_{CF} = 5.8 Hz), 124.1 (q, J_{CF} = 273.5 Hz), 127.4 (q, J_{CF} = 28.7 Hz), 127.7, 127.9, 130.3, 133.6, 138.3, 139.2, 139.8, 143.9, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.3 (s, 3F). IR (neat) 2983, 1732, 1622, 1576, 1485, 1236, 1157, 912 cm^{-1}.



5fa

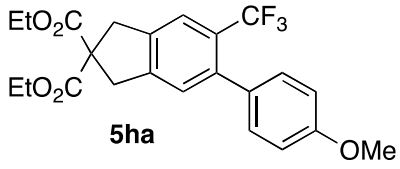
Diethyl 5-phenyl-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5fa): Isolated yield: 92% (Table 4, Entry 3). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, J = 7.0 Hz, 6H), 3.64 (s, 2H), 3.68 (s, 2H), 4.23 (q, J = 7.0 Hz, 4H), 7.15 (s, 1H), 7.29-7.38

(m, 5H), 7.56 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 40.1, 40.3, 60.3, 61.9, 121.8 (q, J_{CF} = 5.8 Hz), 124.3 (q, J_{CF} = 274.8 Hz), 127.4, 127.5 (q, J_{CF} = 28.8 Hz), 127.6, 127.8, 128.9, 133.6, 139.9, 140.5, 143.7, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.4 (s, 3F). IR (neat) 2983, 1732, 1623, 1572, 1486, 1416, 1237, 1156, 911 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{22}H_{22}F_3O_4^+$ $[M+H]^+$ 407.1470, found 407.1463.



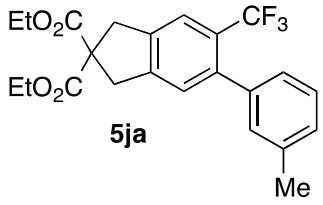
5ga

Diethyl 5-([1,1'-biphenyl]-4-yl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5ga): Isolated yield: 90% (Table 4, Entry 4). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.29 (t, *J* = 7.3 Hz, 6H), 3.66 (s, 2H), 3.69 (s, 2H), 4.24 (q, *J* = 7.3 Hz, 4H), 7.19 (s, 1H), 7.36-7.38 (m, 3H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.59 (s, 1H), 7.61 (d, *J* = 8.5 Hz, 2H), 7.65 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 40.1, 40.3, 60.3, 62.0, 121.9 (q, J_{CF} = 6.0 Hz), 124.3 (q, J_{CF} = 273.5 Hz), 126.4, 127.1, 127.3, 127.4 (q, J_{CF} = 28.7 Hz), 127.9, 128.8, 129.4, 138.9, 139.5, 140.1, 140.2, 140.6, 143.8, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.5 (s, 3F). IR (neat) 2982, 1734, 1623, 1574, 1484, 1235, 1119, 911 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{28}H_{26}F_3O_4^+$ $[M+H]^+$ 483.1783, found 483.1755.



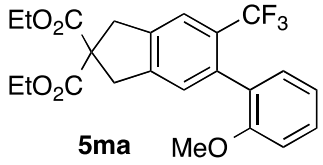
5ha

Diethyl 5-(4-methoxyphenyl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5ha): Isolated yield: 94% (Table 4, Entry 5). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, *J* = 7.0 Hz, 6H), 3.64 (s, 2H), 3.67 (s, 2H), 3.84 (s, 3H), 4.23 (q, *J* = 7.0 Hz, 4H), 6.96 (d, *J* = 8.0 Hz, 2H), 7.13 (s, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.55 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 40.1, 40.3, 55.2, 60.4, 62.0, 113.1, 121.9 (q, J_{CF} = 4.8 Hz), 124.3 (q, J_{CF} = 273.4 Hz), 127.5 (q, J_{CF} = 28.8 Hz), 128.1, 130.1, 132.3, 139.2, 140.3, 143.7, 159.0, 171.3. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.3 (s, 3F). IR (neat) 2939, 1732, 1611, 1063, 837, 759 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{23}H_{24}F_3O_5^+$ $[M+H]^+$ 437.1576, found 437.1566.



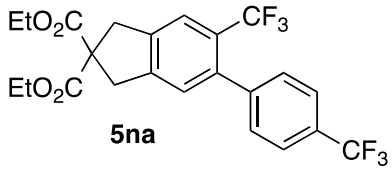
5ja

Diethyl 5-(*m*-tolyl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5ja): Isolated yield: 88% (Table 4, Entry 6). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, J = 7.0 Hz, 6H), 2.38 (s, 3H), 3.64 (s, 2H), 3.68 (s, 2H), 4.23 (q, J = 7.0 Hz, 4H), 7.09-7.19 (m, 4H), 7.26 (t, J = 8.0 Hz, 1H), 7.56 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 21.4, 40.1, 40.3, 60.4, 62.0, 121.8 (q, J_{CF} = 6.0 Hz), 124.3 (q, J_{CF} = 273.5 Hz), 126.1, 127.2 (q, J_{CF} = 30.1 Hz), 127.5, 127.8, 128.2, 129.7, 137.2, 139.3, 139.8, 140.7, 143.6, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.4 (s, 3F). IR (neat) 2983, 1733, 1607, 1574, 1482, 1238, 1190, 910 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{23}H_{24}F_3O_4^+$ $[M+H]^+$ 421.1627, found 421.1649.



5ma

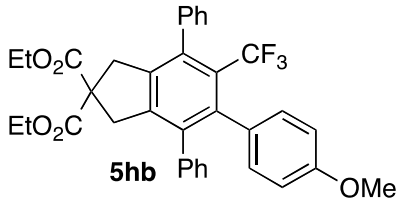
Diethyl 5-(2-methoxyphenyl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5ca): Isolated yield: 82% (Table 4, Entry 7). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.27 (t, J = 7.0 Hz, 6H), 3.65 (s, 2H), 3.68 (s, 2H), 3.80 (s, 3H), 4.23 (q, J = 7.0 Hz, 4H), 6.93-6.98 (m, 2H), 7.09-7.12 (m, 2H), 7.35 (q, J = 7.8 Hz, 1H), 7.55 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 40.2, 40.4, 55.5, 60.3, 62.0, 110.5, 119.8, 121.9 (q, J_{CF} = 4.8 Hz), 124.3 (q, J_{CF} = 273.5 Hz), 127.9, 128.1 (q, J_{CF} = 29.9 Hz), 128.6, 129.2, 130.8, 136.6, 139.4, 143.6, 156.8, 171.3, 171.5. ^{19}F NMR (470 MHz, $CDCl_3$): δ 102.9 (s, 3F). IR (neat) 2982, 1732, 1611, 1574, 1490, 1298, 1121, 912 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{23}H_{23}F_3NaO_5^+$ $[M+Na]^+$ 459.1395, found 459.1377..



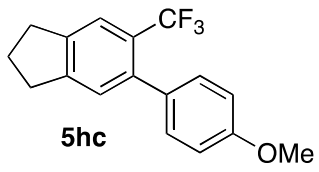
5na

Diethyl 5-(4-trifluoromethylphenyl)-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5na): Isolated yield: 94% (Table 4, Entry 8). Pale yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, J = 7.0 Hz, 6H), 3.65 (s, 2H), 3.68 (s, 2H), 4.24 (q, J = 7.0 Hz, 4H), 7.12 (s, 1H), 7.41 (d, J = 8.0 Hz, 2H), 7.57 (s, 1H), 7.64 (d, J = 8.0 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 14.0, 40.1, 40.3, 60.3, 62.0, 122.0 (q, J_{CF} = 4.9 Hz), 124.2 (q, J_{CF} = 273.5 Hz), 124.3 (q, J_{CF} = 273.5 Hz), 124.6 (q, J_{CF} = 3.6 Hz), 127.3 (q, J_{CF} = 28.8 Hz), 127.5, 129.4, 129.7 (q, J_{CF}

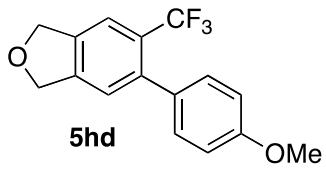
= 32.4 Hz), 138.9, 140.2, 143.5, 144.0, 171.1. ^{19}F NMR (470 MHz, $CDCl_3$): δ 99.2 (s, 3F), 105.4 (s, 3F). IR (neat) 2985, 1733, 1619, 1574, 1447, 1325, 1159, 912 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{23}H_{20}F_6NaO_4^+$ $[M+Na]^+$ 497.1164, found 497.1163.



Diethyl 5-(4-methoxyphenyl)-4,7-diphenyl-6-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5hb): Isolated yield: 97% (Table 4, Entry 9). White solid. Mp 167-173 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.18 (t, J = 7.3 Hz, 6H), 3.30 (s, 2H), 3.39 (s, 2H), 3.72 (s, 3H), 4.23 (q, J = 7.3 Hz, 4H), 6.65 (d, J = 7.0 Hz, 2H), 6.94 (d, J = 6.5 Hz, 2H), 6.95 (d, J = 6.5 Hz, 2H), 7.13-7.19 (m, 3H) 7.31 (d, J = 7.0 Hz, 2H), 7.38-7.46 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 13.9, 40.8, 41.1, 55.0, 59.5, 61.8, 112.4, 124.5 (q, J_{CF} = 279.1 Hz), 126.5, 126.6 (q, J_{CF} = 27.5 Hz), 127.2, 127.8, 128.1, 128.2, 129.4, 131.0, 131.1, 137.1, 138.6, 139.40, 139.41, 139.9, 140.0, 142.1, 158.1, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 113.9 (s, 3F). IR (KBr) 2985, 1734, 1613, 1578, 1440, 1245, 1186, 1038 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{35}H_{32}F_3O_5^+$ $[M+H]^+$ 589.21964 found 589.2181.

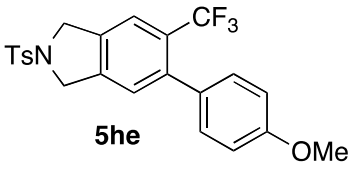


5-(4-Methoxyphenyl)-6-trifluoromethyl-2,3-dihydro-1*H*-indene (5hc): Isolated yield: 79% (Table 4, Entry 10). Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.12-2.18 (m, 2H), 2.94-3.00 (m, 4H), 3.85 (s, 3H), 6.91 (d, J = 8.5 Hz, 2H), 7.15 (s, 1H), 7.23 (d, J = 8.5 Hz, 2H), 7.57 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 25.4, 32.5, 32.8, 55.2, 113.0, 121.8 (q, J_{CF} = 4.8 Hz), 124.6 (q, J_{CF} = 273.5 Hz), 126.5 (q, J_{CF} = 28.8 Hz), 128.2, 130.2, 132.8, 139.3, 143.3, 147.9, 158.8. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.5 (s, 3F). IR (neat) 2955, 2838, 1612, 1579, 1487, 1249, 1152, 927 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{17}H_{16}F_3O^+$ $[M+H]^+$ 293.1153 , found 293.1164.



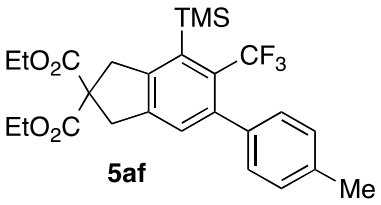
5-(4-Methoxyphenyl)-6-trifluoromethyl-1,3-dihydroisobenzofuran (5hd): Isolated yield: 75% (Table 4,

Entry 11). White solid. Mp 98-101 ºC. ^{1}H NMR (270 MHz, $CDCl_3$): δ 3.85 (s, 3H), 5.16 (s, 2H), 5.18 (s, 2H), 6.92 (d, J = 8.6 Hz, 2H), 7.18 (s, 1H), 7.20 (d, J = 8.6 Hz, 2H), 7.60 (s, 1H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 55.2, 73.2, 73.3, 113.2, 118.9 (q, J_{CF} = 6.0 Hz), 124.2 (q, J_{CF} = 273.4 Hz), 124.9, 128.0 (q, J_{CF} = 30.1 Hz), 130.1, 132.0, 138.3, 140.9, 142.7, 159.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 105.2 (s, 3F). IR (KBr) 2941, 1614, 1577, 1489, 1292, 1159, 932 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{16}H_{14}F_3O_2^+$ $[M+H]^+$ 295.0946, found 295.0958.



5he

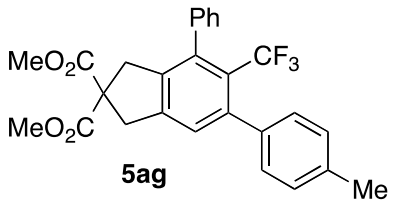
5-(4-Methoxyphenyl)-2-tosyl-6-trifluoromethylisoindoline (5he): Isolated yield: 76% (Table 4, Entry 12). White solid. Mp 127-133 ºC. ^{1}H NMR (270 MHz, $CDCl_3$): δ 2.42 (s, 3H), 3.84 (s, 3H), 4.67 (s, 2H), 4.68 (s, 2H), 6.90 (d, J = 8.6 Hz, 2H), 7.12 (s, 1H), 7.16 (d, J = 8.6 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 7.53 (s, 1H), 7.79 (d, J = 8.2 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.5, 53.4, 53.5, 55.2, 113.2, 120.5 (q, J_{CF} = 4.8 Hz), 123.9 (q, J_{CF} = 274.8 Hz), 126.6, 127.6, 128.4 (q, J_{CF} = 28.8 Hz), 129.2, 130.0, 131.4, 133.4, 135.3, 139.7, 141.3, 144.0, 159.3. ^{19}F NMR (470 MHz, $CDCl_3$): δ 104.9 (s, 3F). IR (KBr) 2966, 1733, 1613, 1520, 1492, 1257, 1182, 935 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{23}H_{20}F_3NNaO_3S^+$ $[M+Na]^+$ 470.1014, found 443.1022.



5af

Diethyl 6-(*p*-tolyl)-5-trifluoromethyl-4-trimethylsilyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5af): Isolated yield: 82%. The ratio of two regioisomers was determined by ^{19}F NMR of the crude materials to be **5af**/**5'af** = 72/28. The major isomer was purified by silica gel chromatography (hexane). White solid. Mp 97-101 ºC. ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.44 (s, 9H), 1.27 (t, J = 6.8 Hz, 6H), 2.40 (s, 3H), 3.56 (s, 2H), 3.71 (s, 2H), 4.23 (q, J = 6.8 Hz, 4H), 7.13 (s, 1H), 7.18-7.20 (m, 4H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 2.2 (d, J_{CF} = 2.6 Hz), 14.0, 21.8, 39.0, 43.1, 60.5, 61.9, 125.2 (q, J_{CF} = 275.9 Hz), 128.4, 128.5, 131.8 (q, J_{CF} = 27.7 Hz), 136.8, 139.2, 141.6, 142.4, 145.7, 171.2. ^{19}F NMR (470 MHz, $CDCl_3$): δ 114.5 (s, 3F). IR (KBr) 2986, 1730, 1598, 1541, 1426,

1297, 1173, 919 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{26}H_{32}F_3O_4Si^+$ $[M+H]^+$ 493.2022, found 493.2038.



5ag

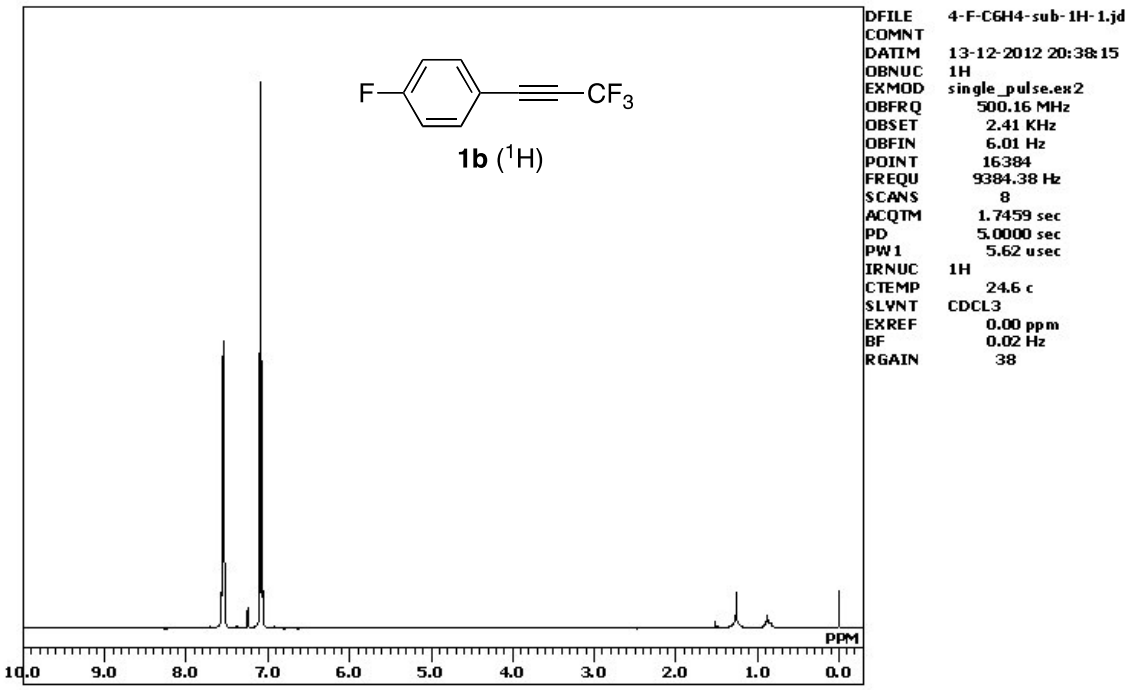
Dimethyl 4-phenyl-6-(*p*-tolyl)-5-trifluoromethyl-1*H*-indene-2,2(3*H*)-dicarboxylate (5ag): Isolated yield: 85%. The ratio of two regioisomers was determined by ^{19}F NMR of the crude materials to be **5ag**/**5'ag** = 85/15. The major isomer was purified by silica gel chromatography (hexane). White solid. Mp 163-166 ºC. ^1H NMR (500 MHz, $CDCl_3$): δ 2.40 (s, 3H), 3.26 (s, 2H), 3.69 (s, 2H), 3.73 (s, 6H), 7.12 (s, 1H), 7.19-7.28 (m, 6H), 7.36-7.44 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.2, 40.3, 40.8, 53.1, 59.7, 124.5 (q, J_{CF} = 276.0 Hz), 125.4 (q, J_{CF} = 27.7 Hz), 127.2, 127.3, 128.1, 128.2, 128.39, 128.40, 136.9, 138.2, 138.7, 139.0, 139.9, 141.8, 142.4, 171.7. ^{19}F NMR (470 MHz, $CDCl_3$): δ 113.8 (s, 3F). IR (KBr) 2957, 1736, 1617, 1577, 1490, 1240, 1181, 915 cm^{-1}. HRMS (ESI): *m/z*: calcd for $C_{27}H_{24}F_3O_4^+$ $[M+H]^+$ 469.1627, found 469.1612.

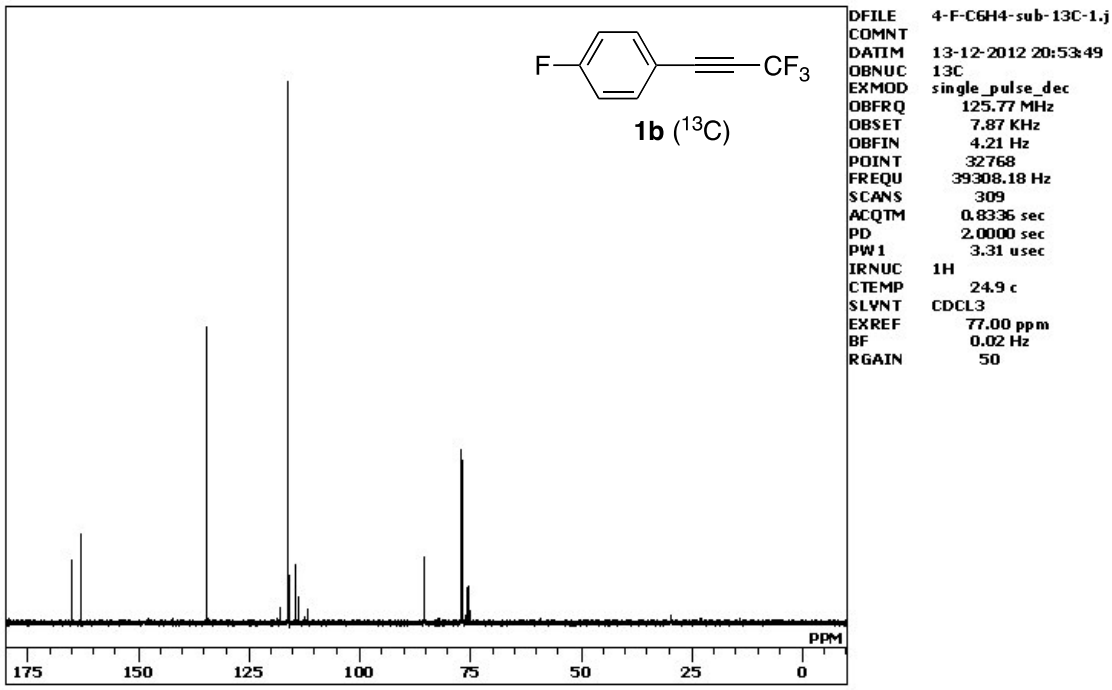
References

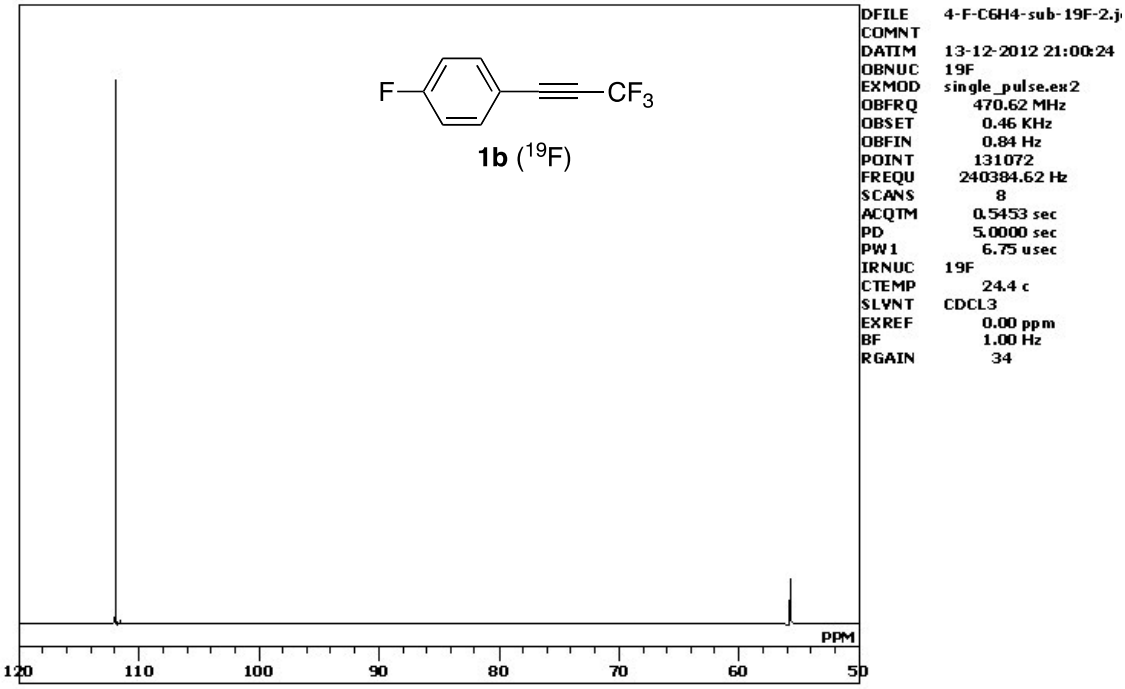
1. (a) Konno, T.; Chae, J.; Kanda, M.; Nagai, G.; Tamura, K.; Ishihara, T.; Yamanaka, H. *Tetrahedron* **2003**, *59,* 7571-7580. (b) Chu, L.; Qing, F. L. *J. Am. Chem. Soc*. **2010**, *132*, 7262-7263.
2. (a) Saino, N.; Amemiya, F.; Tanabe, E.; Kase, K.; Okamoto, S. *Org. Lett*. **2006**, *8*, 1439-1442. (b) Zhang, D.-H.; Yao, L.-F.; Wei, Y.; Shi, M. *Angew. Chem. Int. Ed.* **2011**, *50*, 2583–2587. (c) García-Rubín, S.; Varela, J. A.; Castedo, L.; Saá, C.

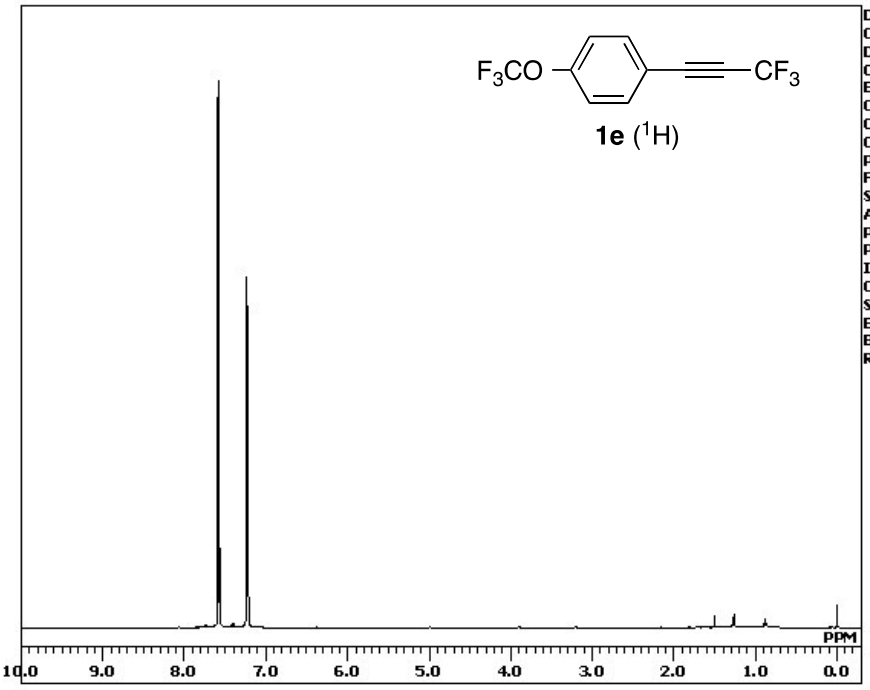
Chem. Eur. J. **2008**, *14*, 9772–9778. (d) Yamamoto, Y.; Arakawa, T.; Ogawa, R.; Itoh, K. *J. Am. Chem. Soc*. **2003**, *125*, 12143-12160.

3. Kawatsura, M.; Yamamoto, M.; Namioka, J.; Kajita, K.; Hirakawa, T.; Itoh, T. *Org. Lett*. **2011**, *13*, 1001–1003.
4. Müller, C.; Lachicotte, R. J.; Jones, W. D. *Organometallics* **2002**, *21*, 1975–1981.
5. Konno, T.; Moriyasu, K.; Kinugawa, R.; Ishihara, T. *Org. Biomol. Chem.* **2010**, *8*, 1718–1724.

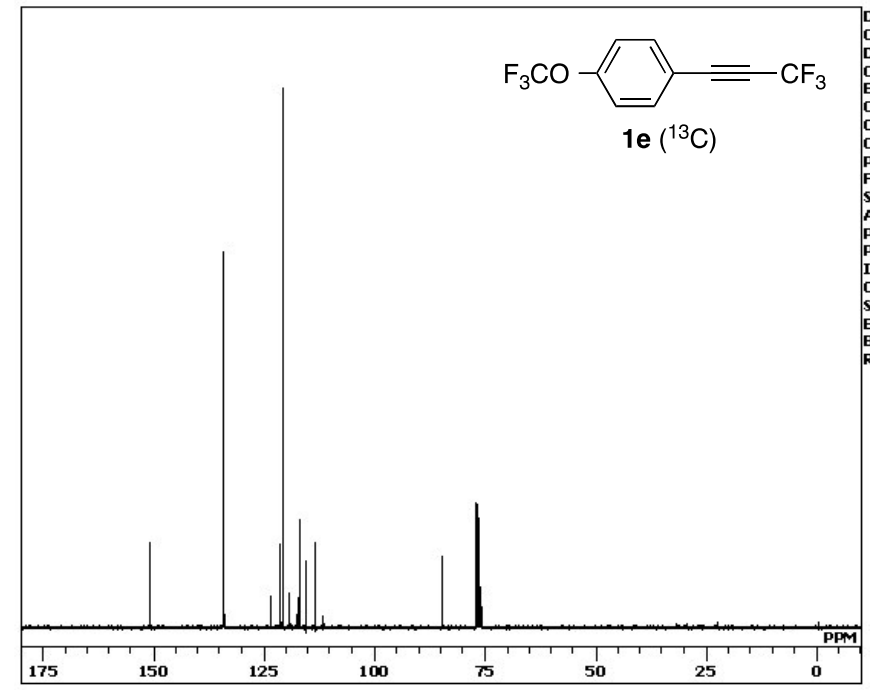



F
CF3
1b (13C)
DFILE 4-F-C6H4-sub-13C-1.j
COMNT
DATIM 13-12-2012 20:53:49
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 309
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.31 usec
IRNUC 1H
CTEMP 24.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.02 Hz
RGAIN 50
PPM
175 150 125 100 75 50 25 0

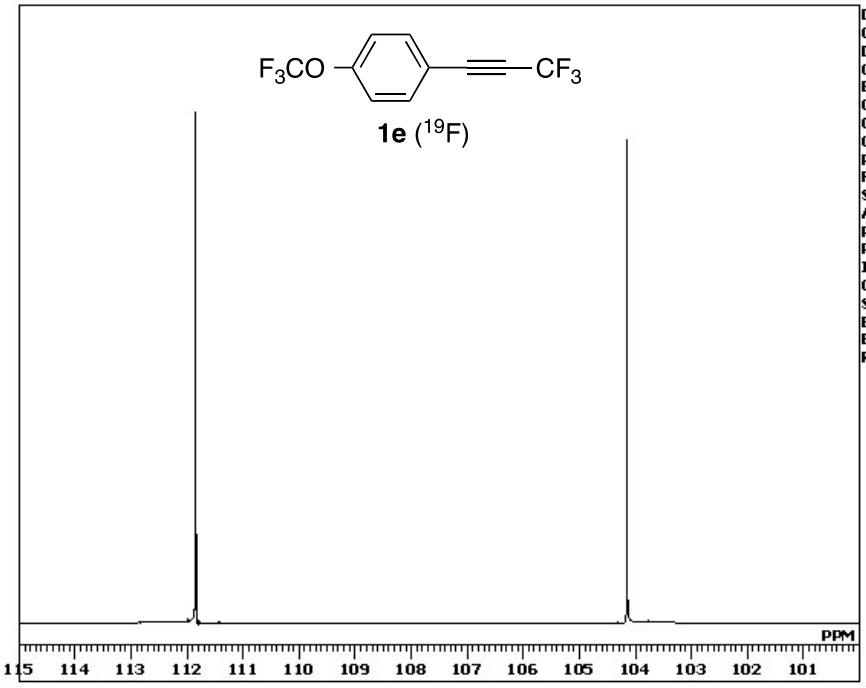

F
CF3
1b (19F)
DFILE 4-F-C6H4-sub-19F-2.j
COMNT
DATIM 13-12-2012 21:00:24
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 34
PPM
120 110 100 90 80 70 60 50



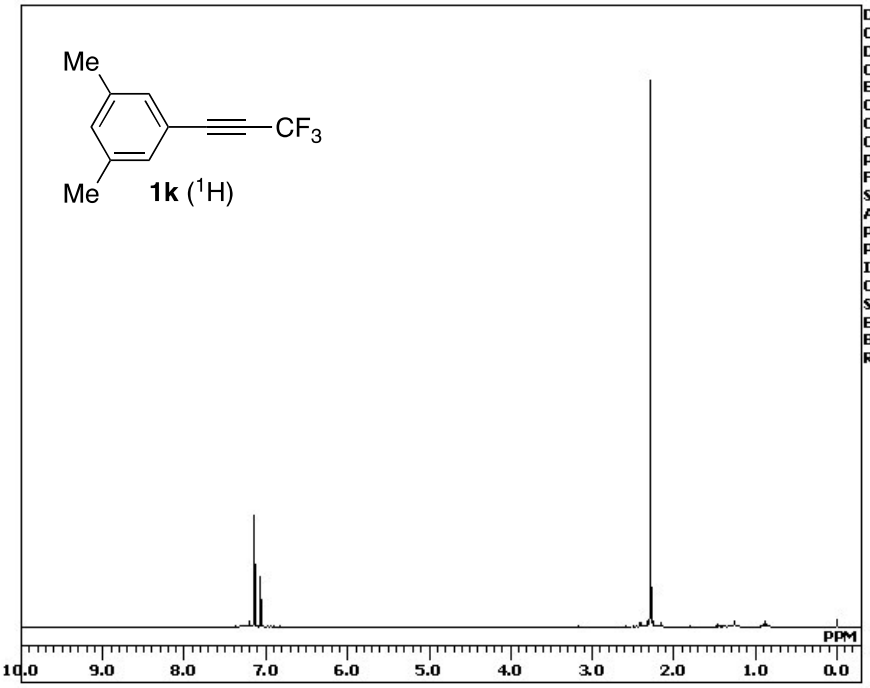
DFILE	4-CF3O-C6H4-sub-1H-
COMNT	
DATIM	10-01-2013 14:55:25
OBNUC	1H
EXMOD	single_pulse.ex2
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	9384.38 Hz
SCANS	8
ACQTM	1.7459 sec
PD	5.0000 sec
PW1	5.61 usec
IRNUC	1H
CTEMP	25.4 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.02 Hz
RGAIN	34



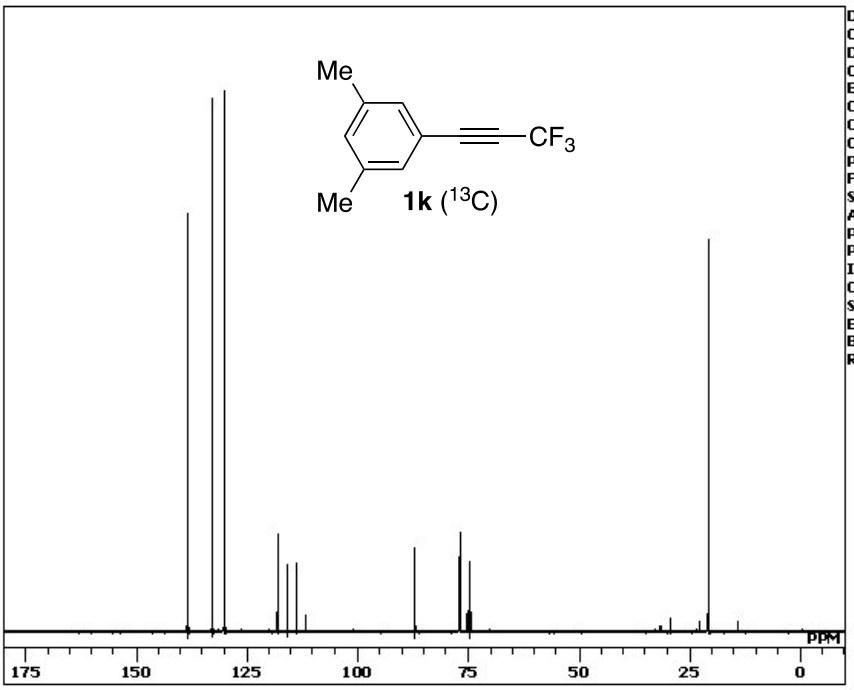
DFILE	4-CF3O-C6H4-sub-13C
COMNT	
DATIM	10-01-2013 15:16:10
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	392
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.34 usec
IRNUC	1H
CTEMP	25.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	0.02 Hz
RGAIN	56



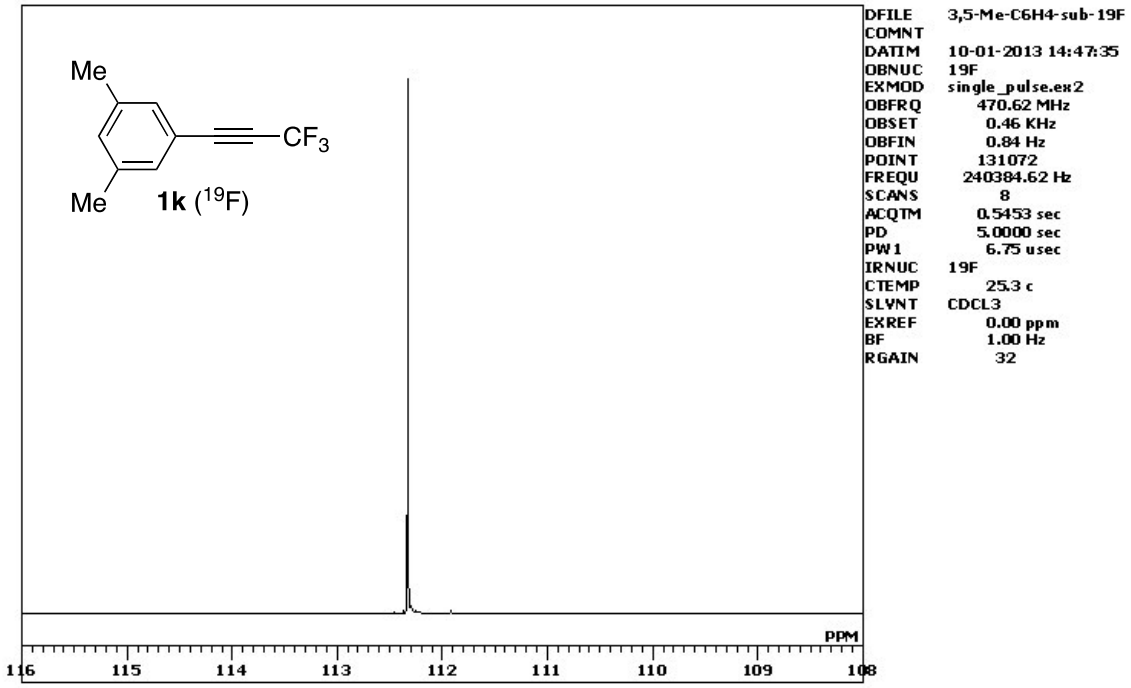
DFILE	4-CF3O-C6H4-sub-19F
COMNT	
DATIM	10-01-2013 15:21:30
OBNUC	19F
EXMOD	single_pulse.ex2
OBFRQ	470.62 MHz
OBSET	0.46 KHz
OBFIN	0.84 Hz
POINT	131072
FREQU	240384.62 Hz
SCANS	8
ACQTM	0.5453 sec
PD	5.0000 sec
PW1	6.75 usec
IRNUC	19F
CTEMP	25.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	28

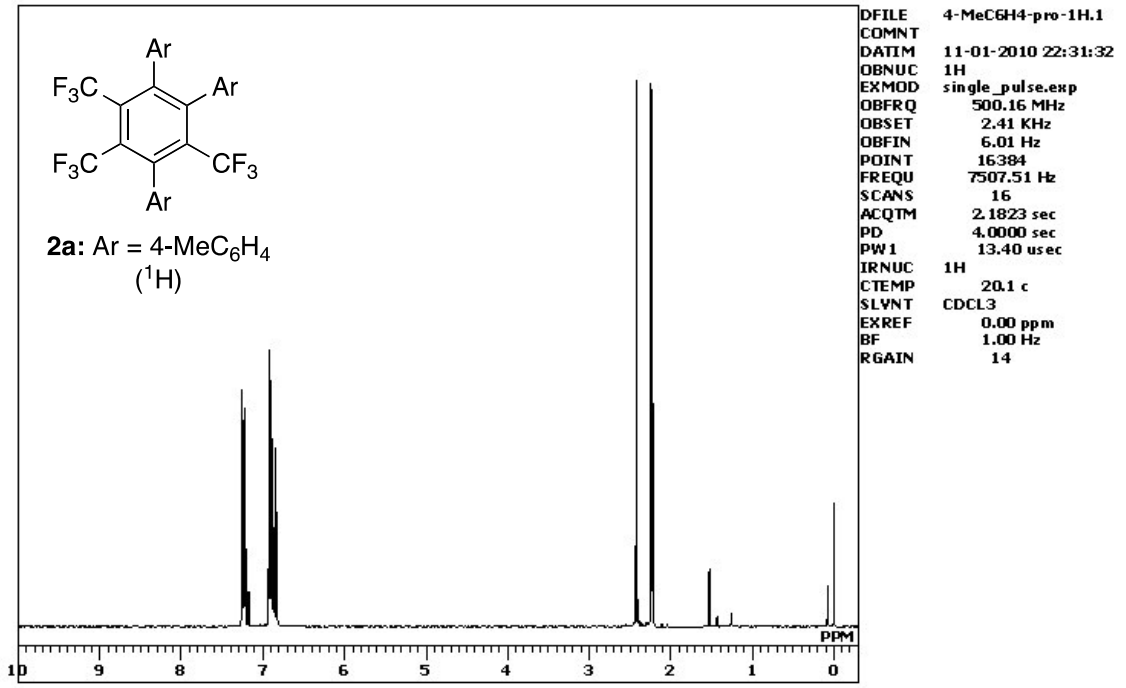


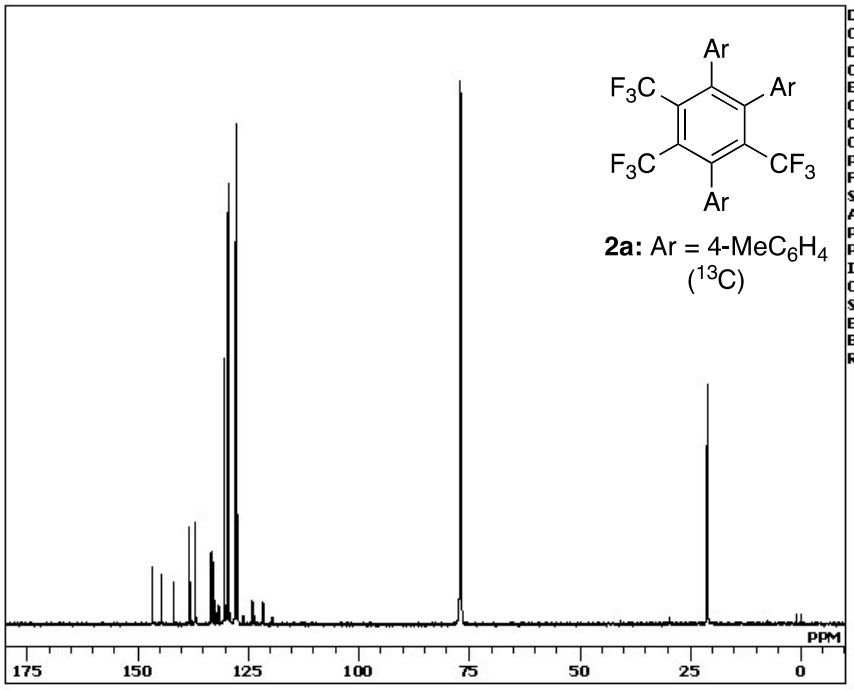
DFILE 3,5-Me-C6H4-sub-1H-
COMNT
DATIM 10-01-2013 14:24:20
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.61 usec
IRNUC 1H
CTEMP 25.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.02 Hz
RGAIN 26



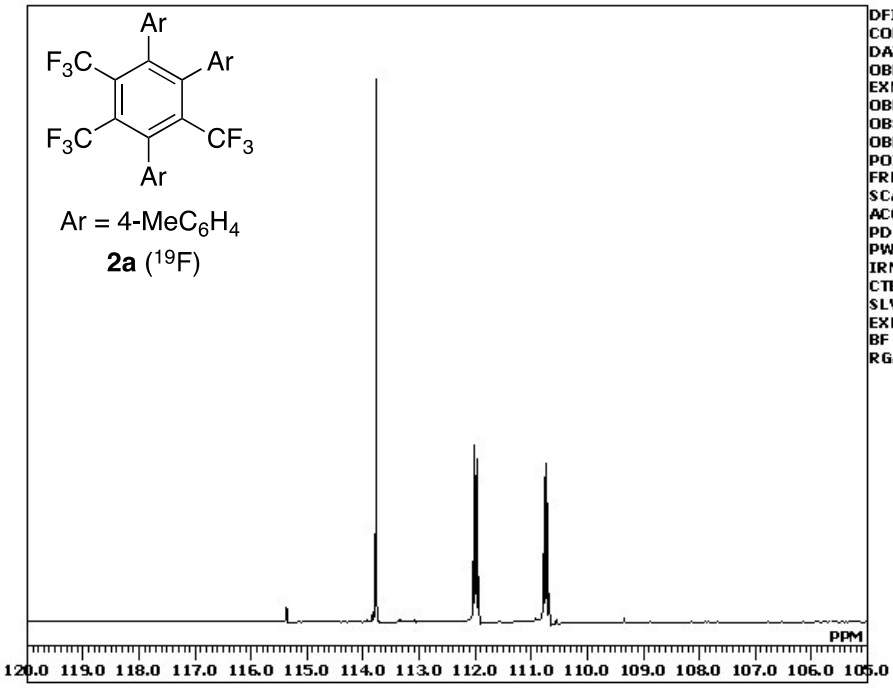
DFILE 3,5-Me-C6H4-sub-13C
COMNT
DATIM 10-01-2013 14:41:22
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 312
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 25.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.02 Hz
RGAIN 56


Me
CF3
Me
1k (19F)
PPM
116
115
114
113
112
111
110
109
108
DFILE 3,5-Me-C6H4-sub-19F
COMNT
DATIM 10-01-2013 14:47:35
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 25.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 32

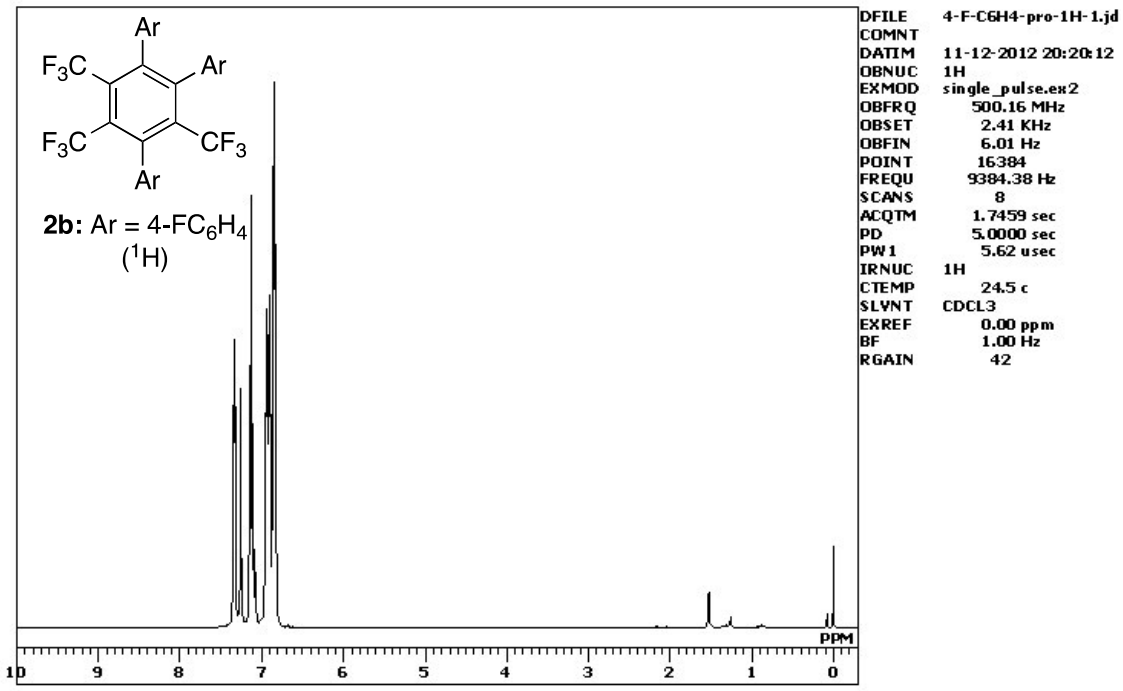

Ar
F3C
Ar
F3C
CF3
Ar
2a: Ar = 4-MeC6H4
(1H)
PPM
10
9
8
7
6
5
4
3
2
1
0
DFILE 4-MeC6H4-pro-1H.1
COMNT
DATIM 11-01-2010 22:31:32
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 16
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 13.40 usec
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 14

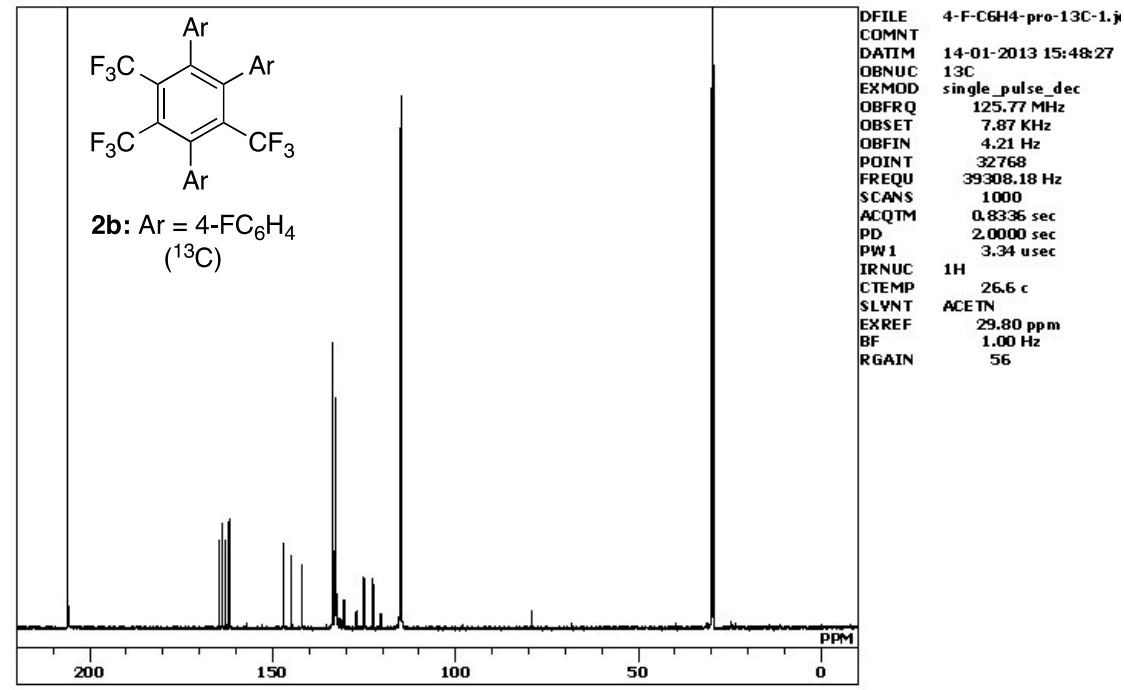


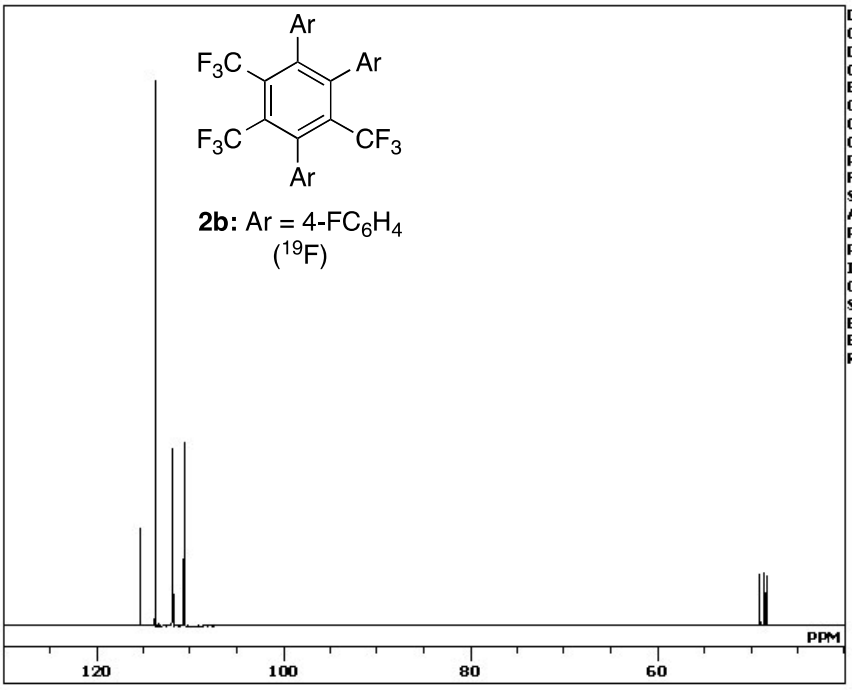
DFILE	4-MeC6H4-pro-13C.1
COMNT	
DATIM	25-12-2009 08:30:09
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	31446.54 Hz
SCANS	6500
ACQTM	1.0420 sec
PD	1.0000 sec
PW1	4.33 usec
IRNUC	1H
CTEMP	24.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	29



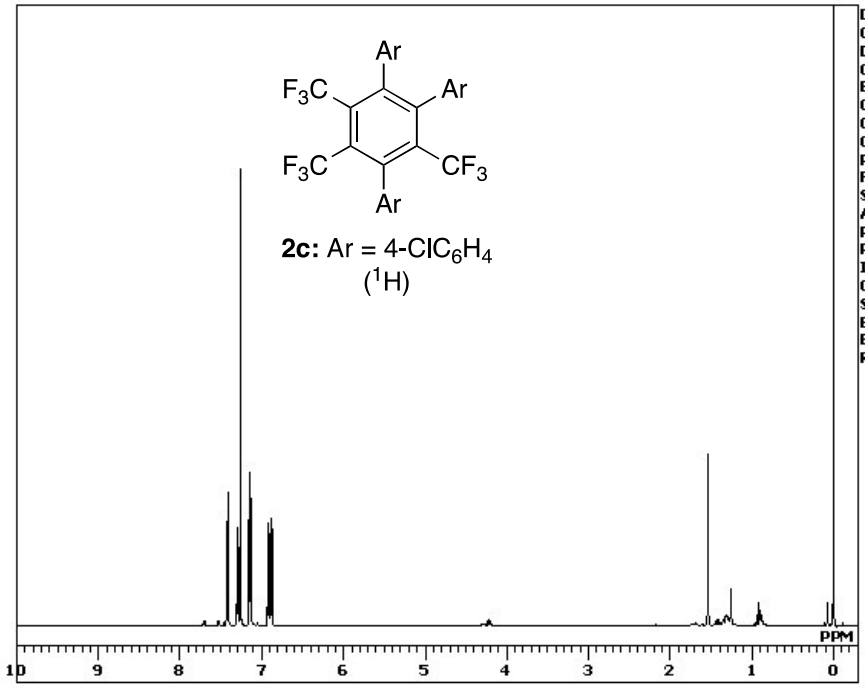
DFILE	4-MeC6H4-pro-19F.1
COMNT	
DATIM	02-11-2009 10:40:02
OBNUC	19F
EXMOD	single_pulse.exp
OBFRQ	470.62 MHz
OBSET	0.46 KHz
OBFIN	0.84 Hz
POINT	32768
FREQU	188679.25 Hz
SCANS	16
ACQTM	0.1737 sec
PD	4.0000 sec
PW1	14.00 usec
IRNUC	19F
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	362.10 ppm
BF	1.00 Hz
RGAIN	13


Ar
F3C
Ar
F3C
CF3
Ar
2b: Ar = 4-FC6H4
(1H)
PPM
10
9
8
7
6
5
4
3
2
1
0
DFILE 4-F-C6H4-pro-1H-1.jd
COMNT
DATIM 11-12-2012 20:20:12
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.62 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 42

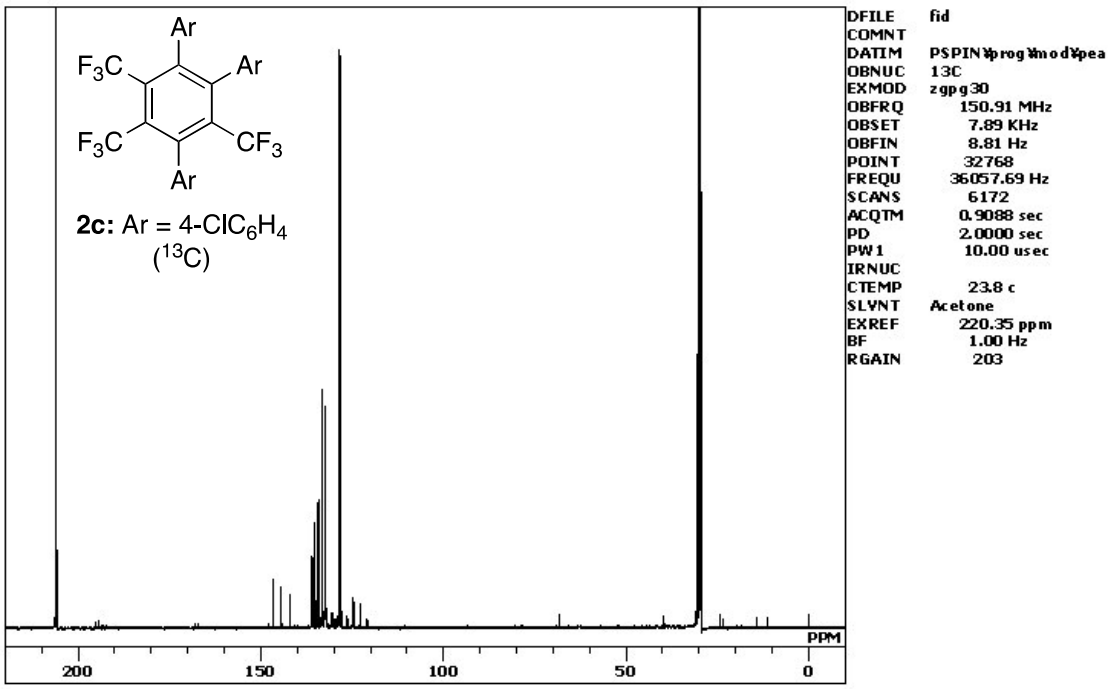

Ar
F3C
Ar
F3C
CF3
Ar
2b: Ar = 4-FC6H4
(13C)
PPM
200
150
100
50
0
DFILE 4-F-C6H4-pro-13C-1.j
COMNT
DATIM 14-01-2013 15:48:27
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 1000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT ACETN
EXREF 29.80 ppm
BF 1.00 Hz
RGAIN 56

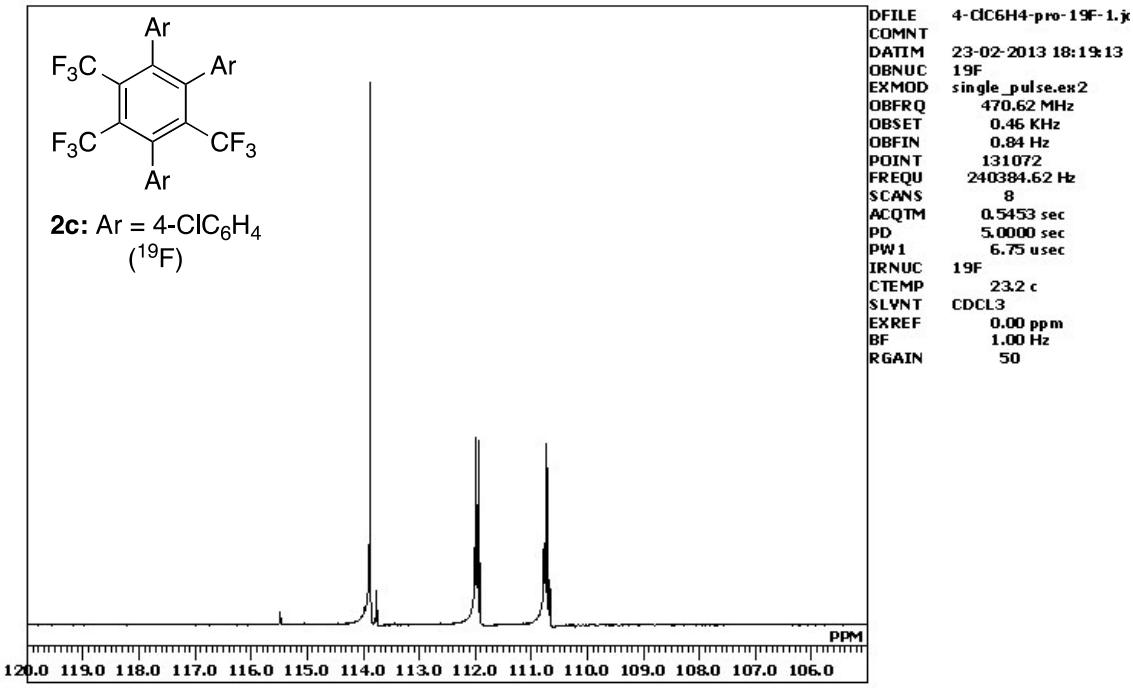


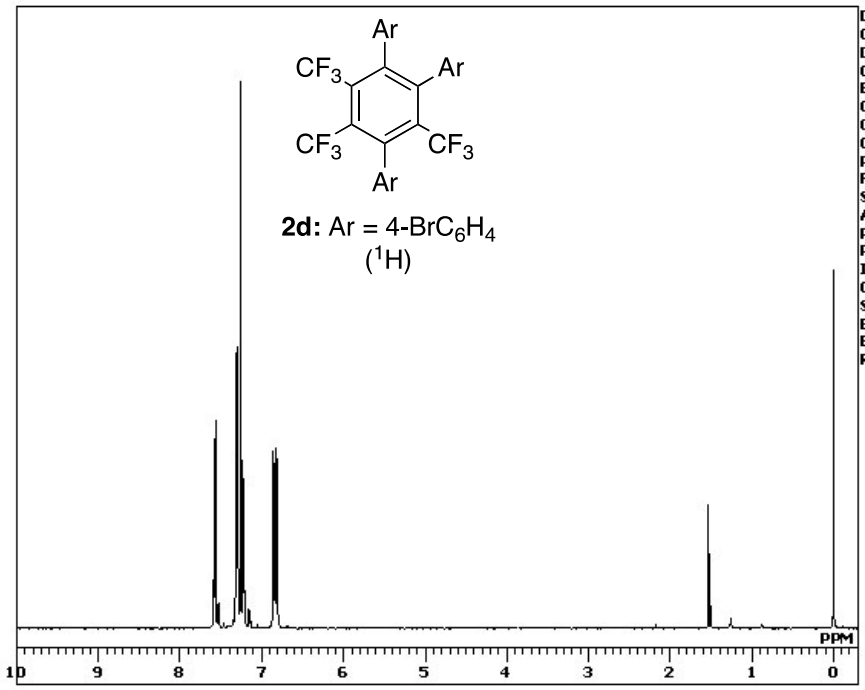
DFILE	4-F-C6H4-pro-19F-1.j
COMNT	
DATIM	11-12-2012 20:24:02
OBNUC	19F
EXMOD	single_pulse.ex2
OBFRQ	470.62 MHz
OBSET	0.46 KHz
OBFIN	0.84 Hz
POINT	131072
FREQU	240384.62 Hz
SCANS	8
ACQTM	0.5453 sec
PD	5.0000 sec
PW1	6.75 usec
IRNUC	19F
CTEMP	24.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	50



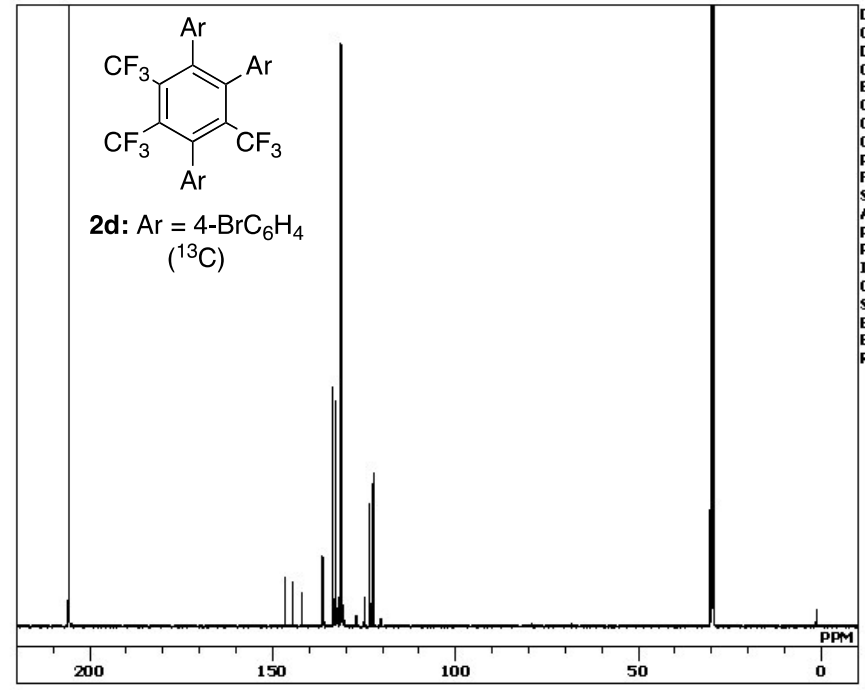
DFILE	4-ClC6H4-pro-1H.1
COMNT	
DATIM	16-11-2009 16:04:53
OBNUC	1H
EXMOD	single_pulse.exp
OBFRQ	500.16 MHz
OBSET	2.41 KHz
OBFIN	6.01 Hz
POINT	16384
FREQU	7507.51 Hz
SCANS	16
ACQTM	2.1823 sec
PD	4.0000 sec
PW1	13.40 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	22


Ar
F3C
Ar
F3C
CF3
Ar
2c: Ar = 4-ClC6H4
(13C)
DFILE fid
COMNT
DATIM PSPIN¥prog¥mod¥pea
OBNUC 13C
EXMOD zgpg30
OBFRQ 150.91 MHz
OBSET 7.89 KHz
OBFIN 8.81 Hz
POINT 32768
FREQU 36057.69 Hz
SCANS 6172
ACQTM 0.9088 sec
PD 2.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 23.8 c
SLVNT Acetone
EXREF 220.35 ppm
BF 1.00 Hz
RGAIN 203
PPM
200
150
100
50
0

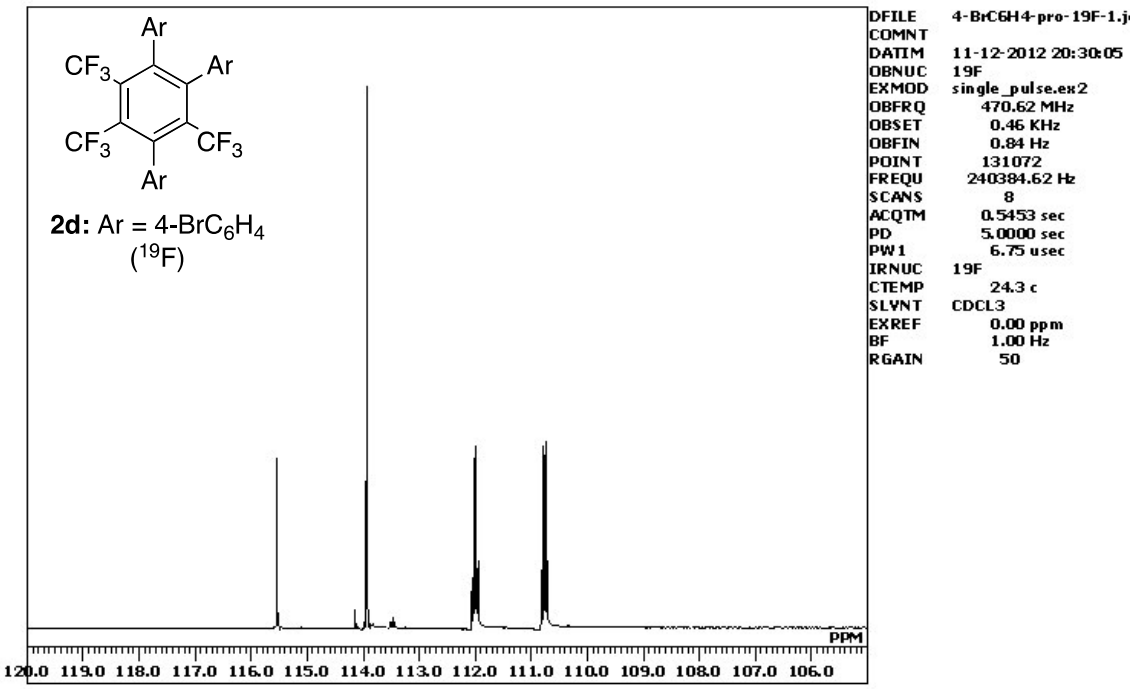

Ar
F3C
Ar
F3C
CF3
Ar
2c: Ar = 4-ClC6H4
(19F)
DFILE 4-ClC6H4-pro-19F-1.jx
COMNT
DATIM 23-02-2013 18:19:13
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 50
PPM
120.0 119.0 118.0 117.0 116.0 115.0 114.0 113.0 112.0 111.0 110.0 109.0 108.0 107.0 106.0

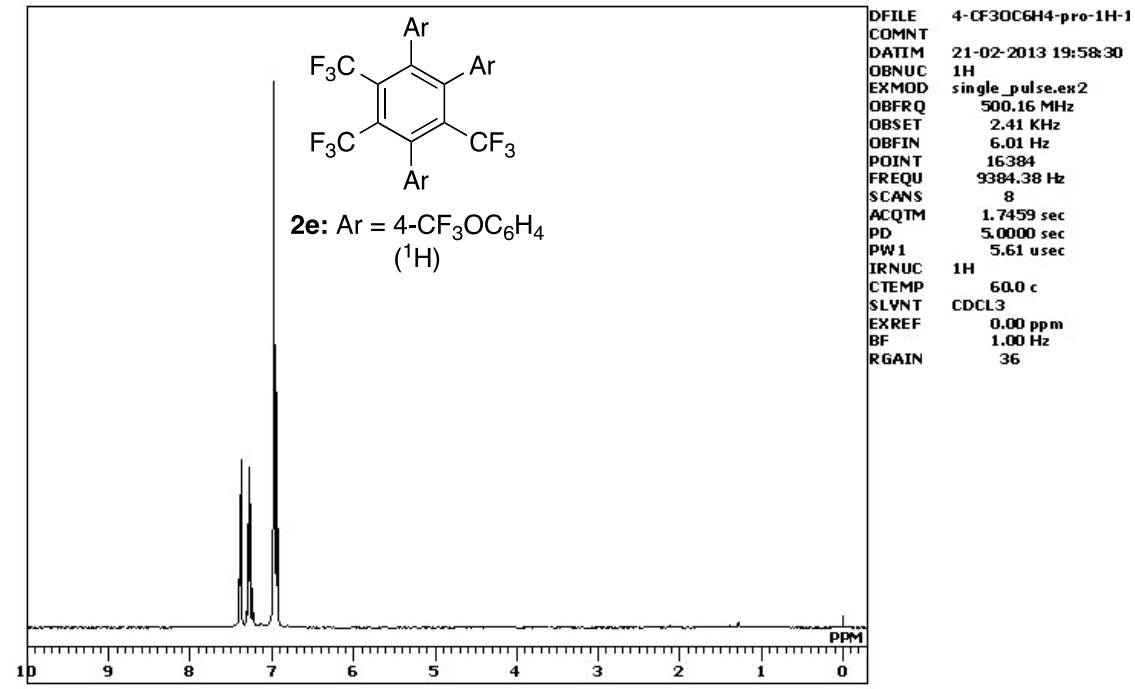


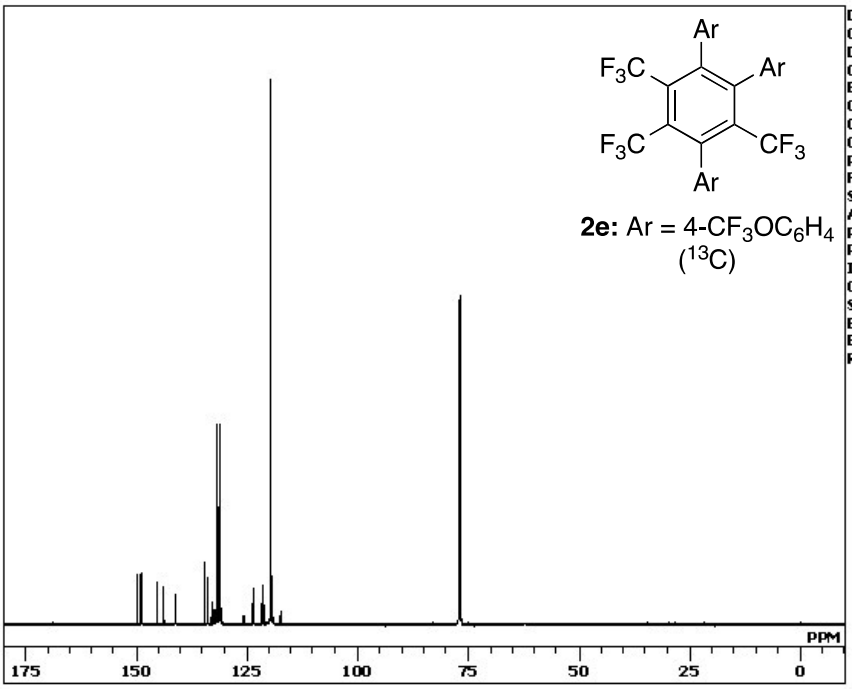
DFILE 4-Br-C6H4-product-1H
COMNT
DATIM 12-12-2012 17:39:45
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.62 usec
IRNUC 1H
CTEMP 24.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 52



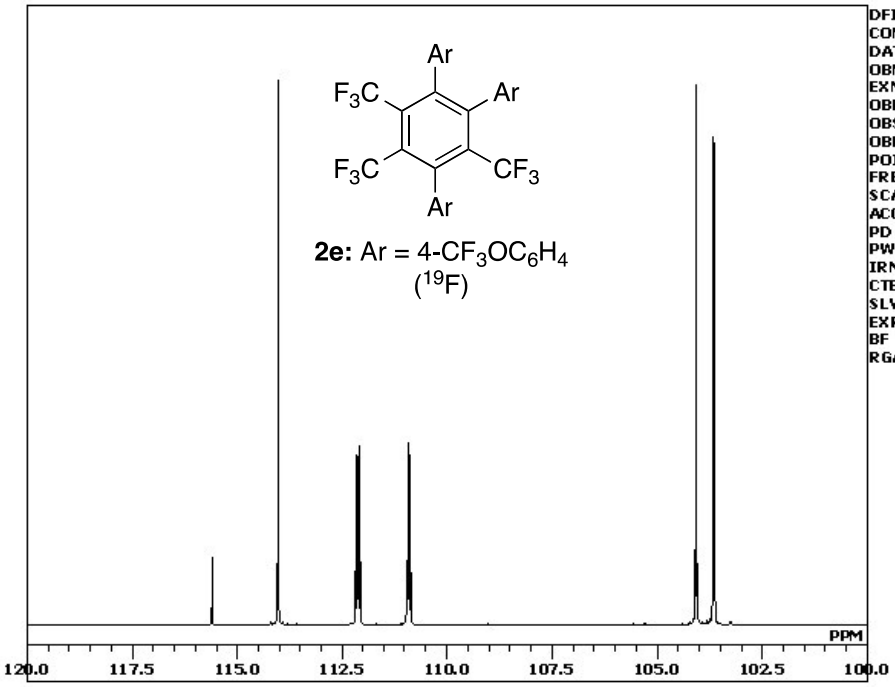
DFILE 4-BrC6H4-pro-13C-4.j
COMNT
DATIM 23-02-2013 21:37:28
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 4061
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 50.0 c
SLVNT ACETN
EXREF 29.80 ppm
BF 1.00 Hz
RGAIN 56


Ar
CF3
Ar
CF3
CF3
Ar
2d: Ar = 4-BrC6H4
(19F)
DFILE 4-BrC6H4-pro-19F-1.j
COMNT
DATIM 11-12-2012 20:30:05
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 24.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 50
PPM
120.0 119.0 118.0 117.0 116.0 115.0 114.0 113.0 112.0 111.0 110.0 109.0 108.0 107.0 106.0

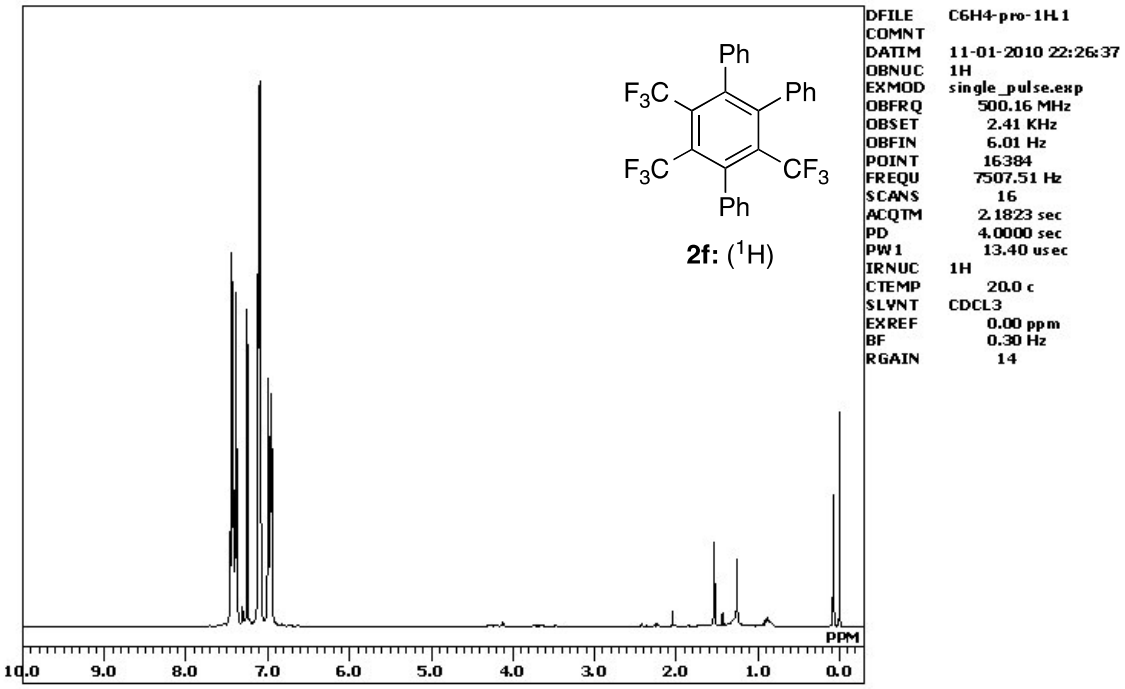

Ar
F3C
Ar
F3C
CF3
Ar
2e: Ar = 4-CF3OC6H4
(1H)
DFILE 4-CF3OC6H4-pro-1H-1
COMNT
DATIM 21-02-2013 19:58:30
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.61 usec
IRNUC 1H
CTEMP 60.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 36
PPM
10 9 8 7 6 5 4 3 2 1 0

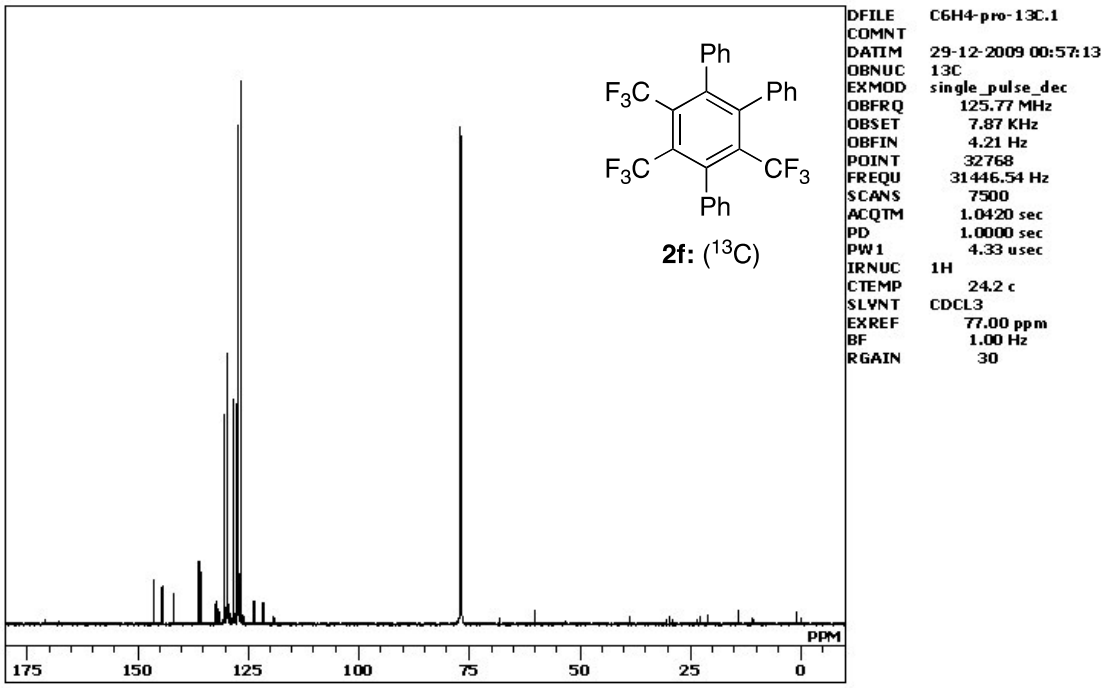


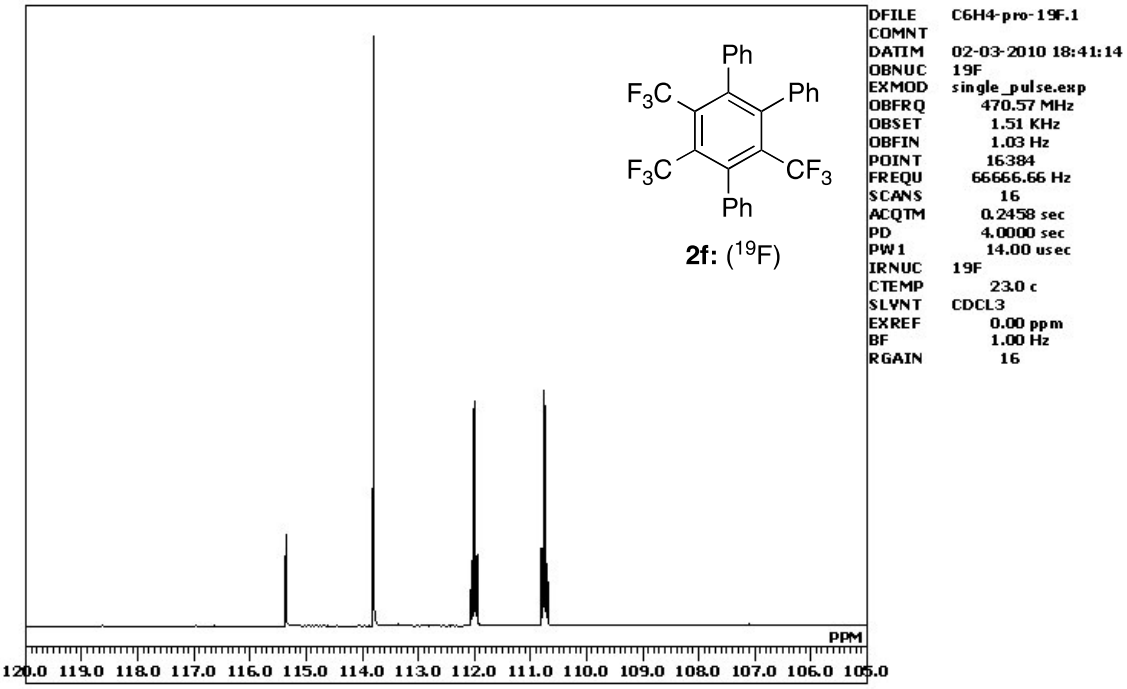

```
DFILE    4-CF3OC6H4-pro-13C-
COMNT
DATIM    21-02-2013 21:14:34
OBNUC    13C
EXMOD    single_pulse_dec
OBFRQ      125.77 MHz
OBSET        7.87 KHz
OBFIN        4.21 Hz
POINT       32768
FREQU     39308.18 Hz
SCANS        1578
ACQTM      0.8336 sec
PD         2.0000 sec
PW1          3.34 usec
IRNUC    1H
CTEMP       60.0 c
SLVNT    CDCL3
EXREF       77.00 ppm
BF           1.00 Hz
RGAIN          58
```

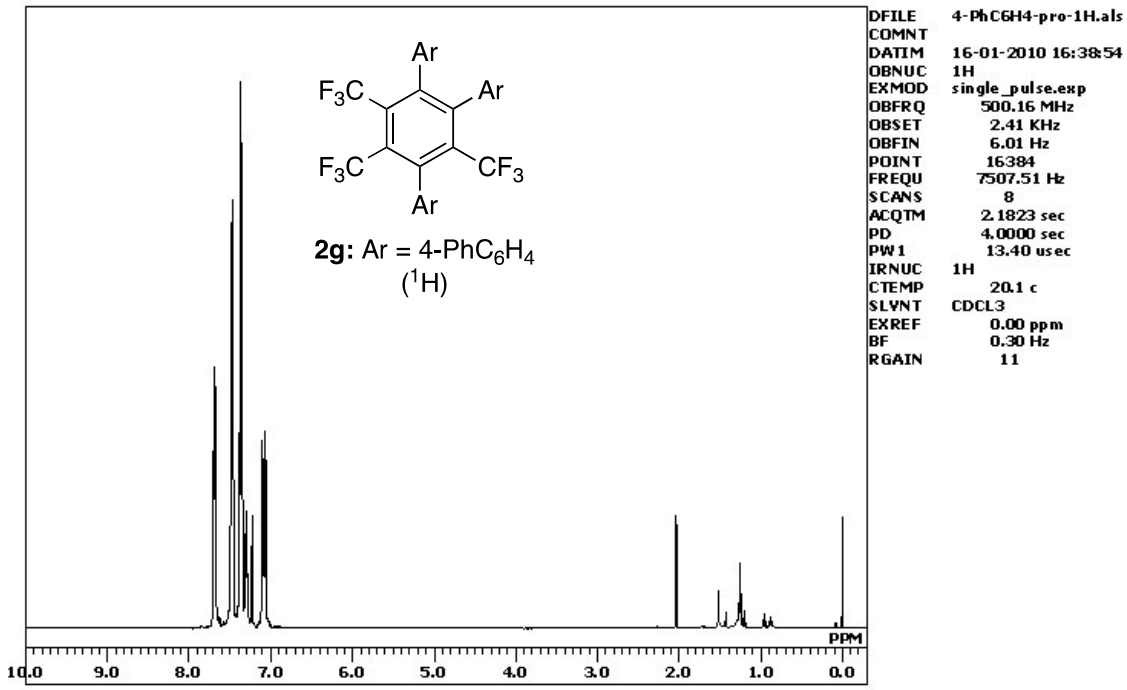


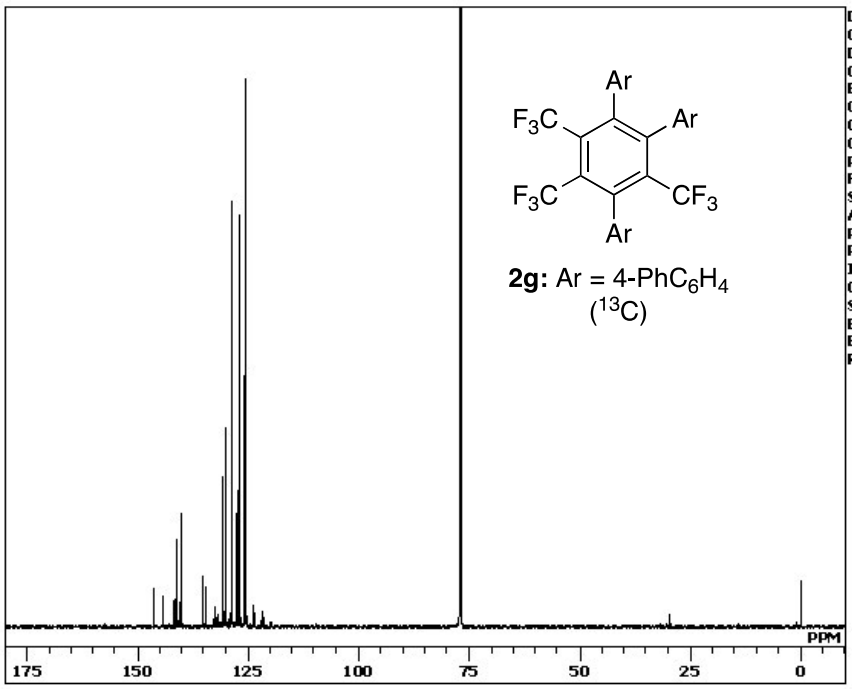

```
DFILE    4-CF3OC6H4-pro-19F-
COMNT
DATIM    21-02-2013 21:20:22
OBNUC    19F
EXMOD    single_pulse.ex2
OBFRQ      470.62 MHz
OBSET        0.46 KHz
OBFIN        0.84 Hz
POINT      131072
FREQU    240384.62 Hz
SCANS           8
ACQTM      0.5453 sec
PD         5.0000 sec
PW1          6.75 usec
IRNUC    19F
CTEMP       25.8 c
SLVNT    CDCL3
EXREF        0.00 ppm
BF           1.00 Hz
RGAIN          30
```


Ph
F3C
Ph
F3C
CF3
Ph
2f: (1H)
DFILE C6H4-pro-1H.1
COMNT
DATIM 11-01-2010 22:26:37
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 16
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 13.40 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.30 Hz
RGAIN 14
PPM
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

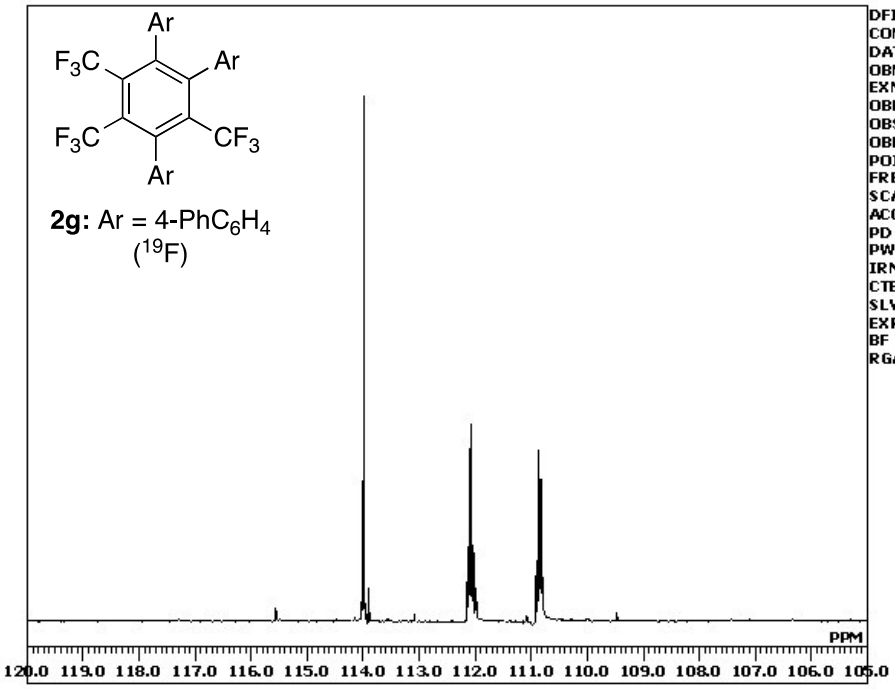

Ph
F3C
Ph
F3C
CF3
Ph
2f: (13C)
DFILE C6H4-pro-13C.1
COMNT
DATIM 29-12-2009 00:57:13
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 31446.54 Hz
SCANS 7500
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.33 usec
IRNUC 1H
CTEMP 24.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 30
PPM
175 150 125 100 75 50 25 0


Ph
F3C
Ph
F3C
CF3
Ph
2f: (19F)
DFILE C6H4-pro-19F.1
COMNT
DATIM 02-03-2010 18:41:14
OBNUC 19F
EXMOD single_pulse.exp
OBFRQ 470.57 MHz
OBSET 1.51 KHz
OBFIN 1.03 Hz
POINT 16384
FREQU 66666.66 Hz
SCANS 16
ACQTM 0.2458 sec
PD 4.0000 sec
PW1 14.00 usec
IRNUC 19F
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 16
PPM
120.0 119.0 118.0 117.0 116.0 115.0 114.0 113.0 112.0 111.0 110.0 109.0 108.0 107.0 106.0 105.0

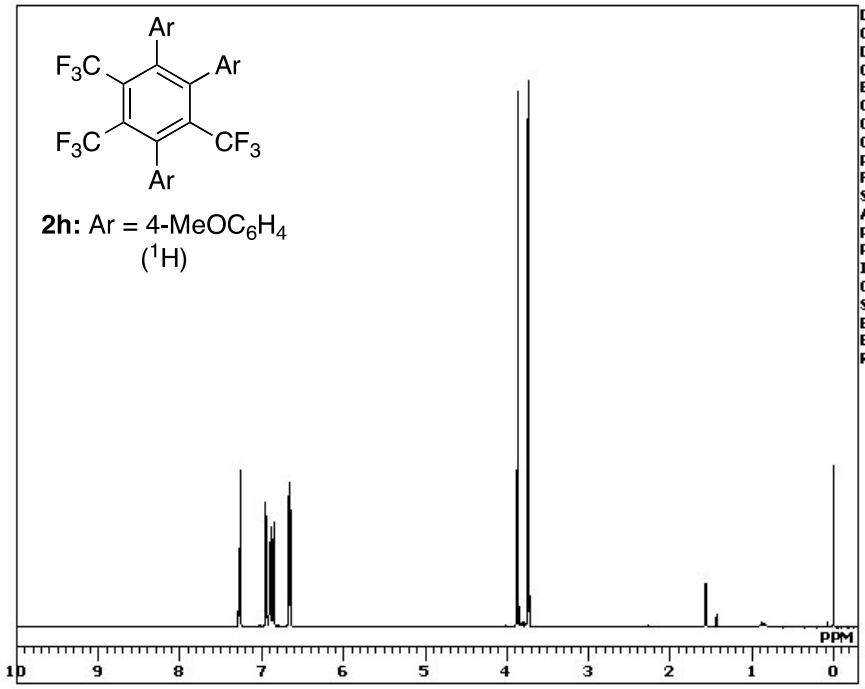

Ar
F3C
Ar
F3C
CF3
Ar
2g: Ar = 4-PhC6H4
(1H)
DFILE 4-PhC6H4-pro-1H.als
COMNT
DATIM 16-01-2010 16:38:54
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 13.40 usec
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.30 Hz
RGAIN 11
PPM
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0

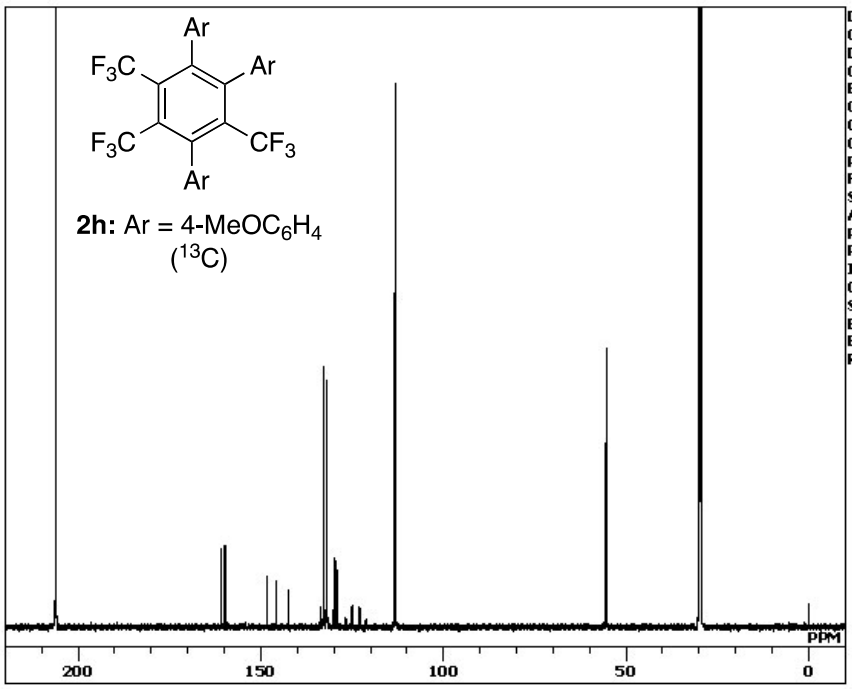


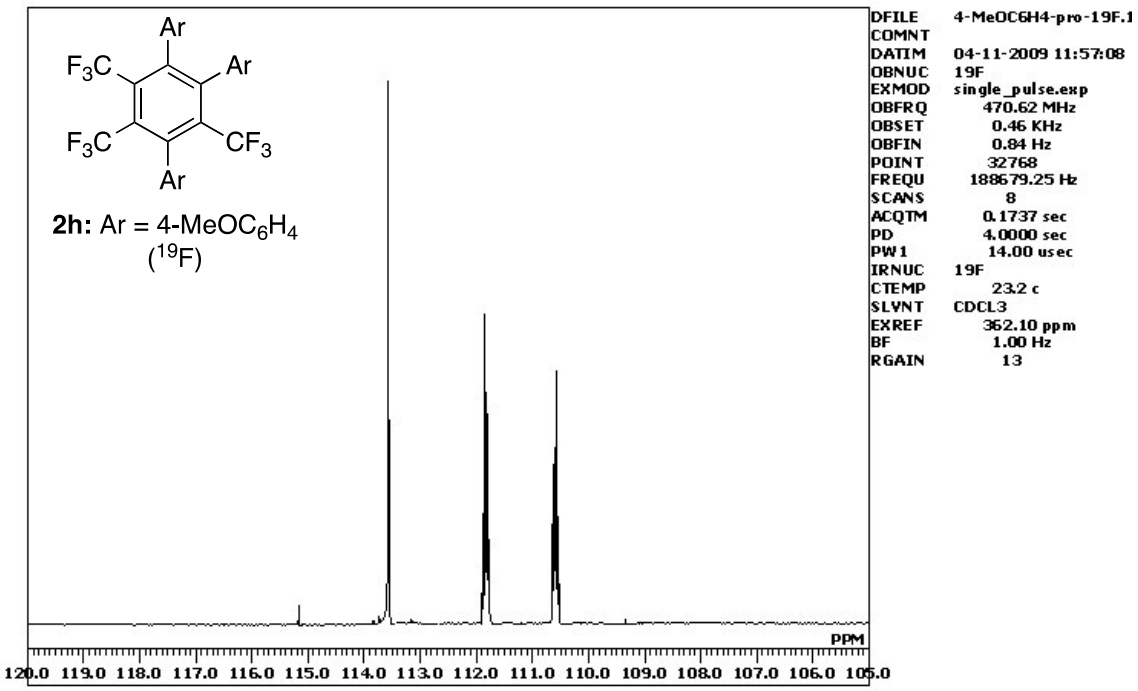
DFILE fid
COMNT
DATIM PSPIN¥prog¥mod¥dat
OBNUC 13C
EXMOD zgpg30
OBFRQ 150.91 MHz
OBSET 7.89 KHz
OBFIN 8.81 Hz
POINT 32768
FREQU 36057.69 Hz
SCANS 14500
ACQTM 0.9088 sec
PD 2.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 22.7 c
SLVNT CDCl3
EXREF 219.45 ppm
BF 1.00 Hz
RGAIN 203

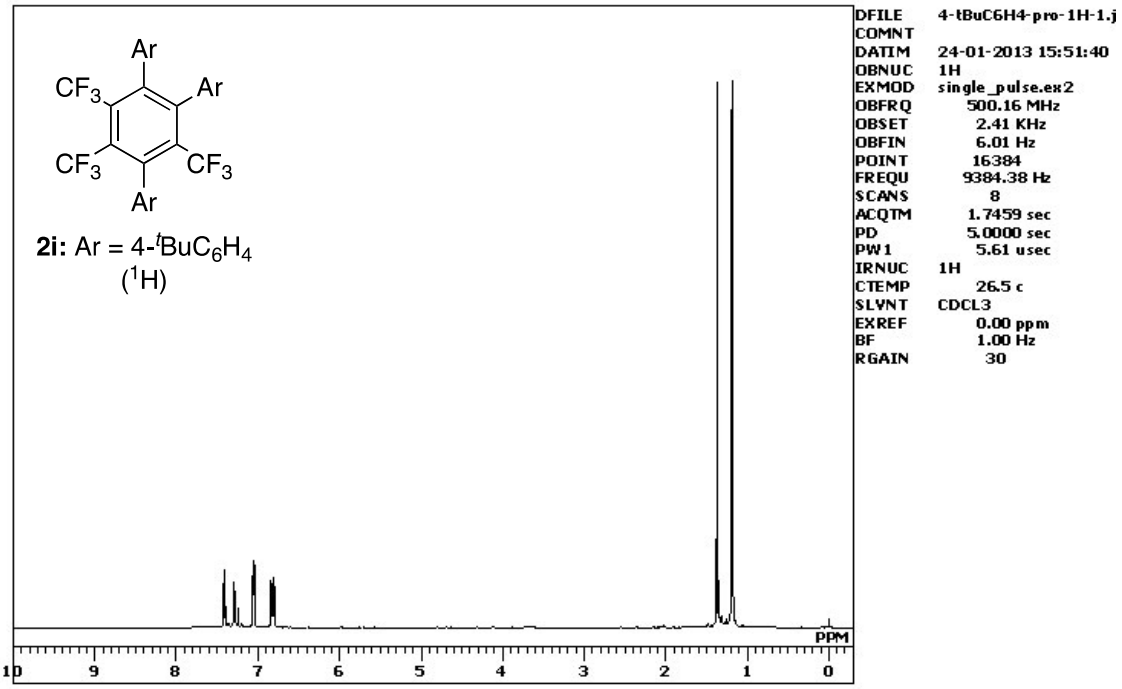


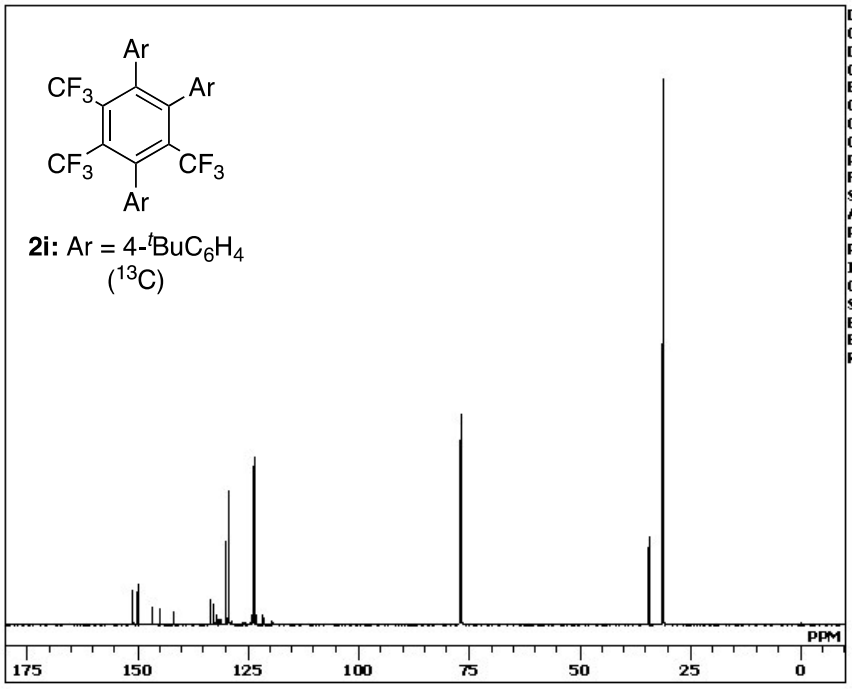
DFILE 4-PhC6H4-pro-19F.1
COMNT
DATIM 20-01-2010 12:08:39
OBNUC 19F
EXMOD single_pulse.exp
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 32768
FREQU 188679.25 Hz
SCANS 16
ACQTM 0.1737 sec
PD 4.0000 sec
PW1 14.00 usec
IRNUC 19F
CTEMP 22.4 c
SLVNT CDCL3
EXREF 362.10 ppm
BF 1.00 Hz
RGAIN 13


Ar
F3C
Ar
F3C
CF3
Ar
2h: Ar = 4-MeOC6H4
(1H)
DFILE 4-MeOC6H4-pro-1H.al
COMNT
DATIM 16-01-2010 16:29:16
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 13.40 usec
IRNUC
CTEMP 20.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 16
PPM
10 9 8 7 6 5 4 3 2 1 0

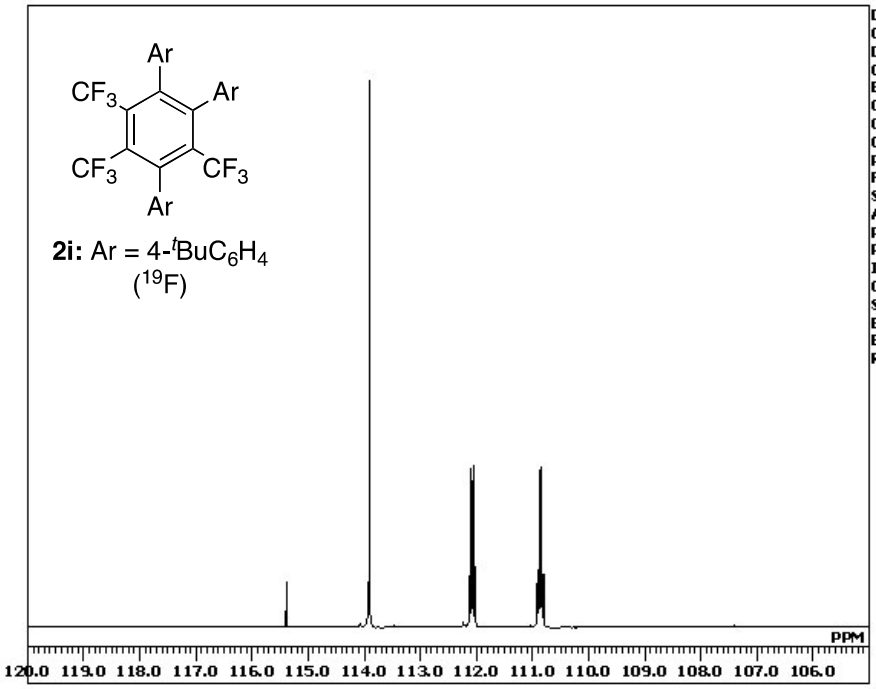

Ar
F3C
Ar
F3C
CF3
Ar
2h: Ar = 4-MeOC6H4
(13C)
DFILE fid
COMNT
DATIM P$PIN¥prog¥mod¥pea
OBNUC 13C
EXMOD zgpg30
OBFRQ 150.91 MHz
OBSET 7.89 KHz
OBFIN 8.81 Hz
POINT 32768
FREQU 36057.69 Hz
SCANS 8500
ACQTM 0.9088 sec
PD 2.0000 sec
PW1 10.00 usec
IRNUC
CTEMP 23.3 c
SLVNT Acetone
EXREF 220.37 ppm
BF 1.00 Hz
RGAIN 203
PPM
200 150 100 50 0


Ar
F3C
Ar
F3C
CF3
Ar
2h: Ar = 4-MeOC6H4
(19F)
DFILE 4-MeOC6H4-pro-19F.1
COMNT
DATIM 04-11-2009 11:57:08
OBNUC 19F
EXMOD single_pulse.exp
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 32768
FREQU 188679.25 Hz
SCANS 8
ACQTM 0.1737 sec
PD 4.0000 sec
PW1 14.00 usec
IRNUC 19F
CTEMP 23.2 c
SLVNT CDCL3
EXREF 362.10 ppm
BF 1.00 Hz
RGAIN 13
PPM
120.0 119.0 118.0 117.0 116.0 115.0 114.0 113.0 112.0 111.0 110.0 109.0 108.0 107.0 106.0 105.0

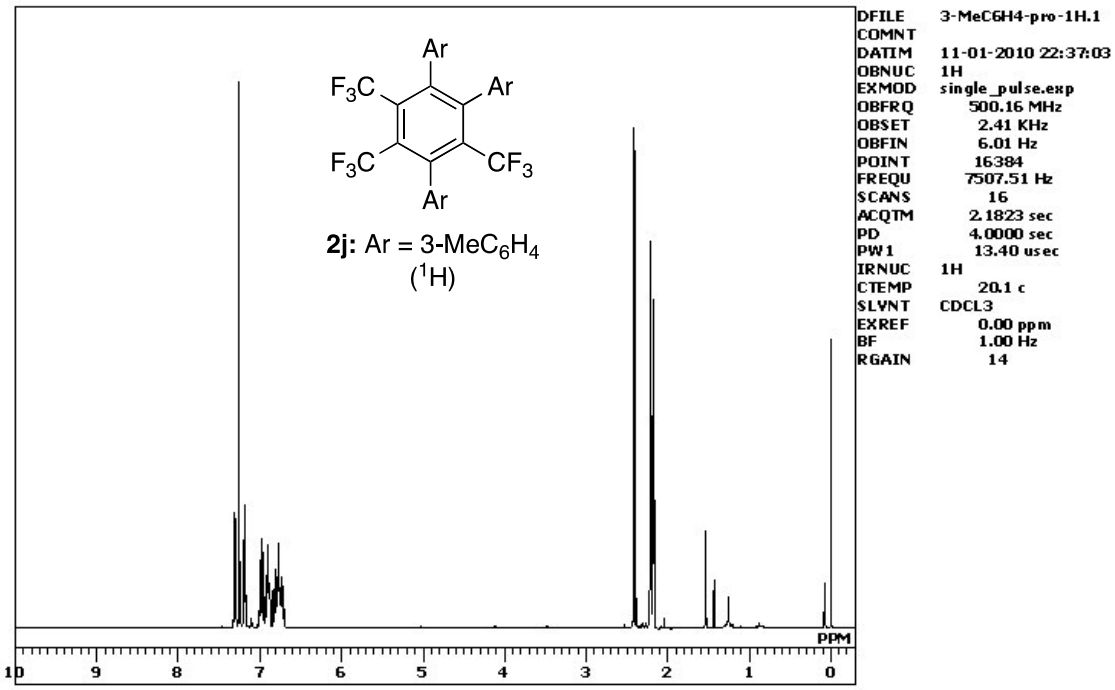

Ar
CF3
Ar
CF3
CF3
Ar
2i: Ar = 4-tBuC6H4
(1H)
DFILE 4-tBuC6H4-pro-1H-1.j
COMNT
DATIM 24-01-2013 15:51:40
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.61 usec
IRNUC 1H
CTEMP 26.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 30
PPM
10 9 8 7 6 5 4 3 2 1 0



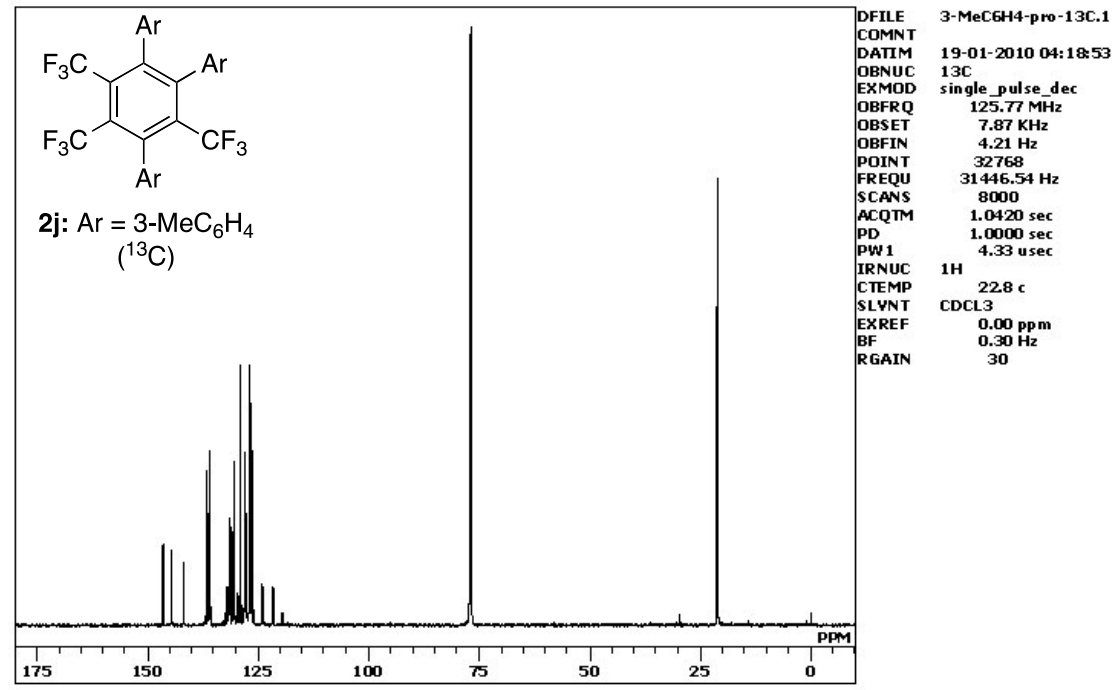
DFILE 4-tBuC6H4-pro-13C-1.
COMNT
DATIM 24-01-2013 16:31:28
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 800
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 26.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 58



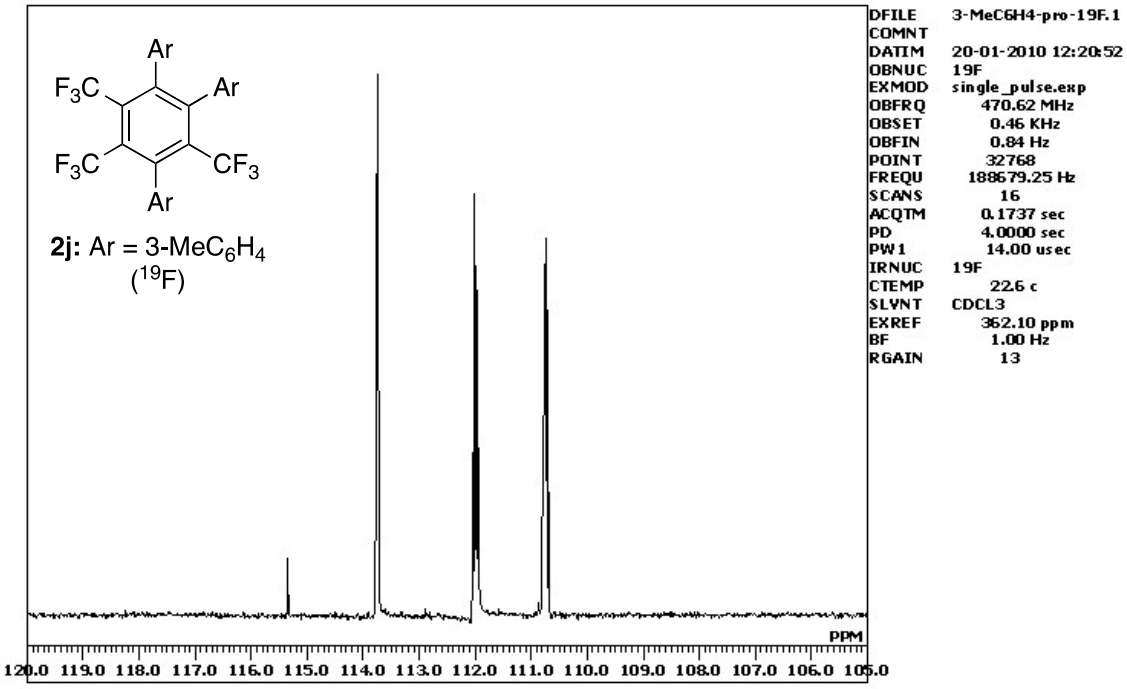
DFILE 4-tBuC6H4-pro-19F-1.
COMNT
DATIM 24-01-2013 16:37:16
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 26.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 36

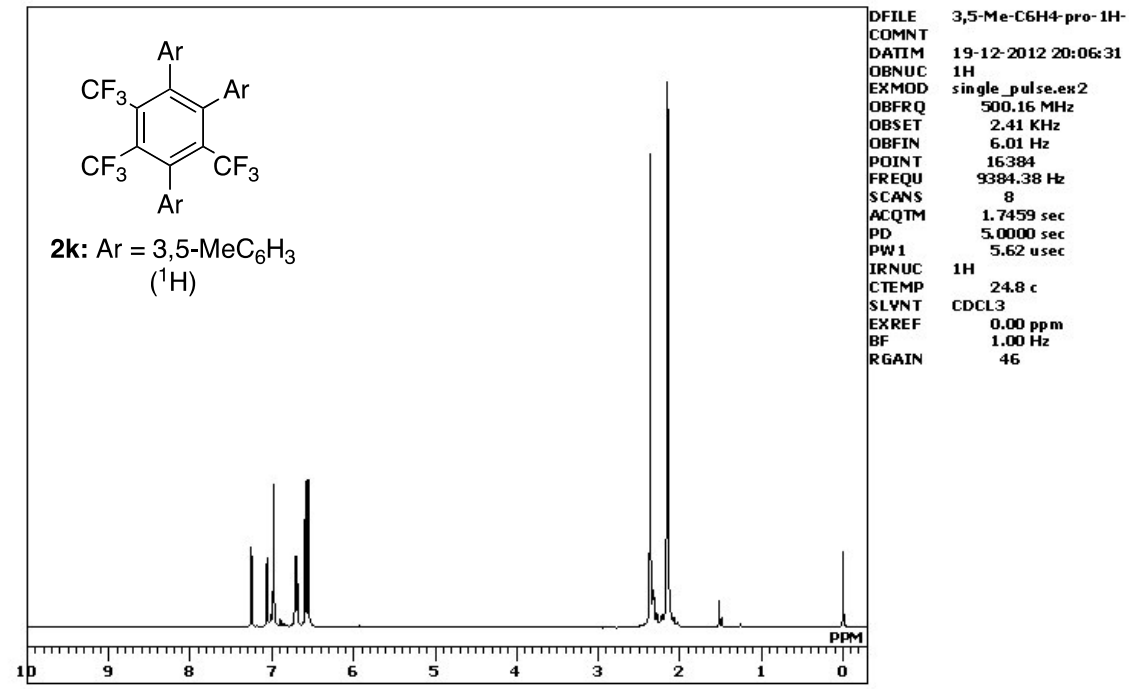


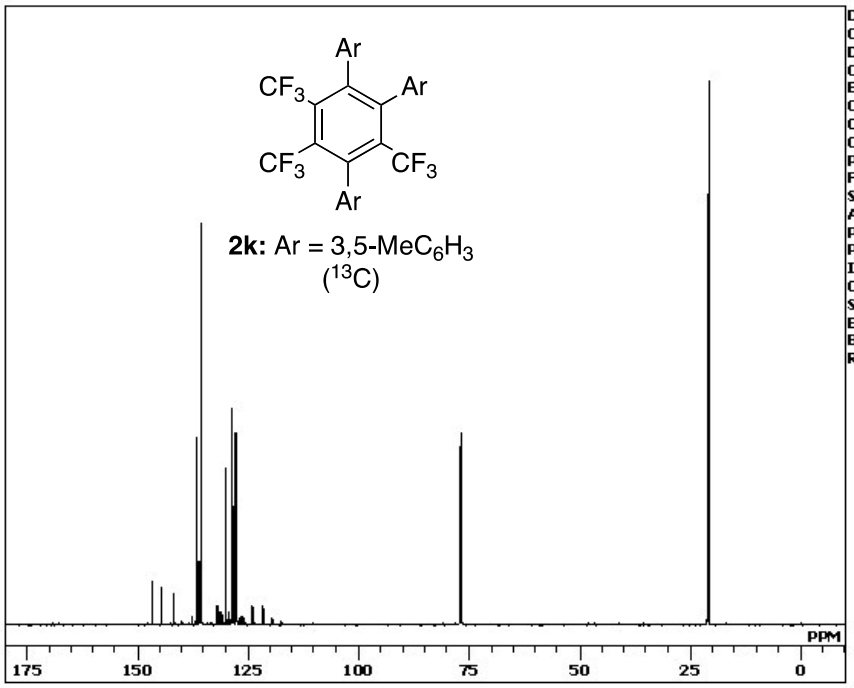
DFILE 3-MeC6H4-pro-1H.1
COMNT
DATIM 11-01-2010 22:37:03
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 16
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 13.40 usec
IRNUC 1H
CTEMP 20.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 14



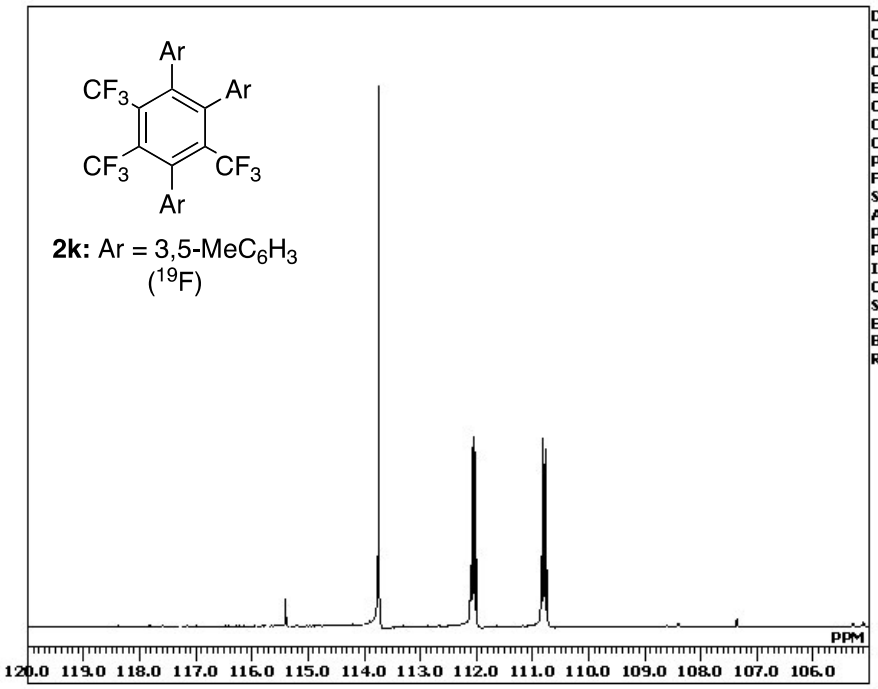
DFILE 3-MeC6H4-pro-13C.1
COMNT
DATIM 19-01-2010 04:18:53
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 31446.54 Hz
SCANS 8000
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.33 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.30 Hz
RGAIN 30


Ar
F3C
Ar
F3C
CF3
Ar
2j: Ar = 3-MeC6H4
(19F)
DFILE 3-MeC6H4-pro-19F.1
COMNT
DATIM 20-01-2010 12:20:52
OBNUC 19F
EXMOD single_pulse.exp
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 32768
FREQU 188679.25 Hz
SCANS 16
ACQTM 0.1737 sec
PD 4.0000 sec
PW1 14.00 usec
IRNUC 19F
CTEMP 22.6 c
SLVNT CDCL3
EXREF 362.10 ppm
BF 1.00 Hz
RGAIN 13
PPM
120.0 119.0 118.0 117.0 116.0 115.0 114.0 113.0 112.0 111.0 110.0 109.0 108.0 107.0 106.0 105.0

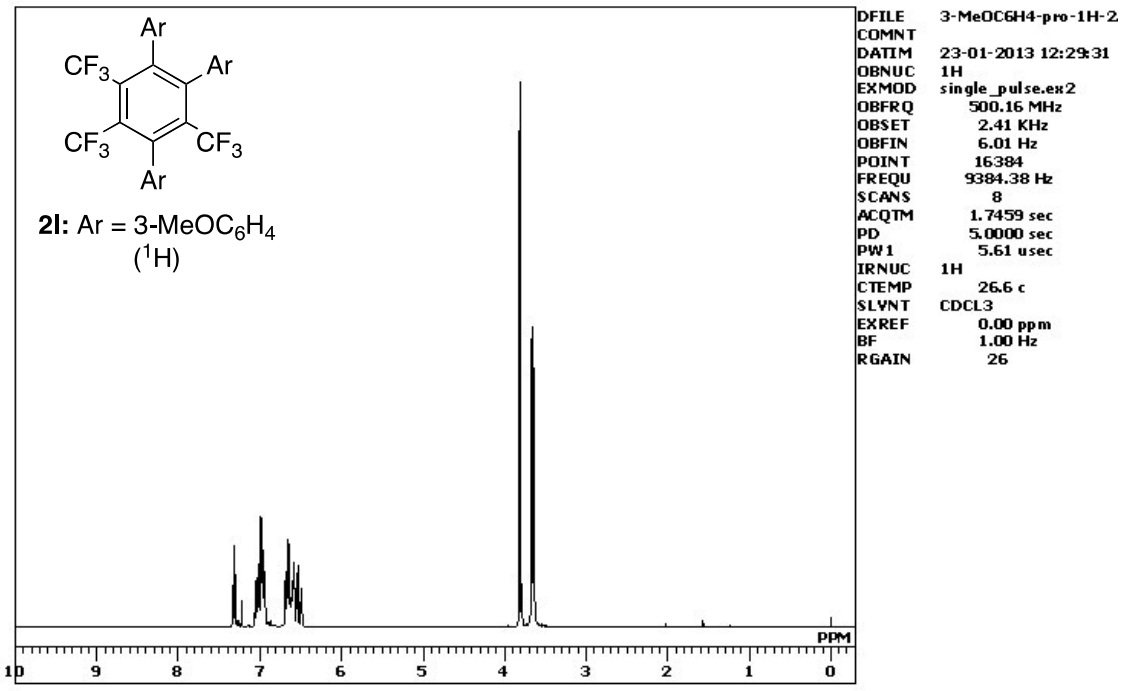

Ar
CF3
Ar
CF3
CF3
Ar
2k: Ar = 3,5-MeC6H3
(1H)
DFILE 3,5-Me-C6H4-pro-1H-
COMNT
DATIM 19-12-2012 20:06:31
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.62 usec
IRNUC 1H
CTEMP 24.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 46
PPM
10 9 8 7 6 5 4 3 2 1 0

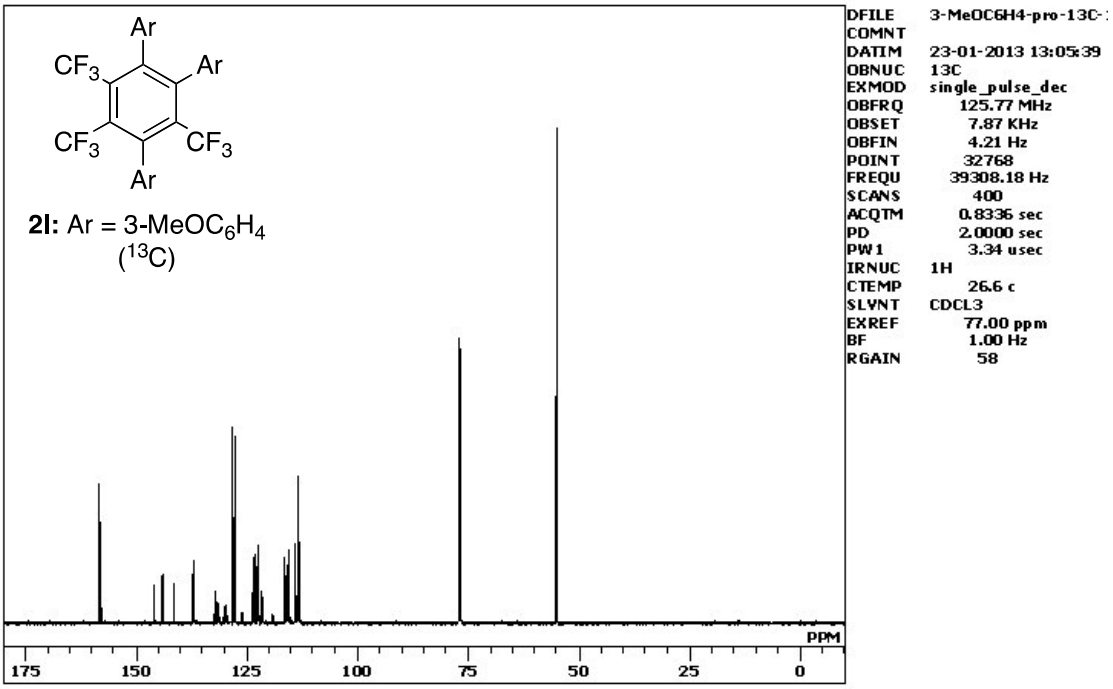


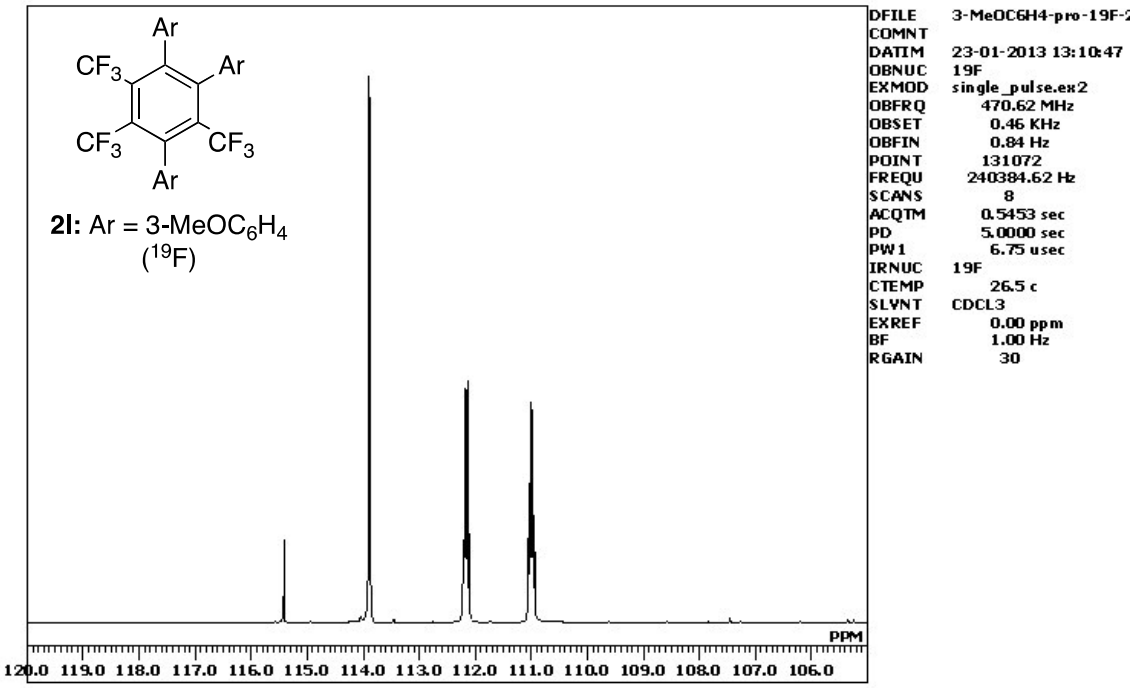
DFILE	3,5-Me-C6H4-pro-13C
COMNT	
DATIM	19-12-2012 20:47:53
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	125.77 MHz
OBSET	7.87 KHz
OBFIN	4.21 Hz
POINT	32768
FREQU	39308.18 Hz
SCANS	694
ACQTM	0.8336 sec
PD	2.0000 sec
PW1	3.31 usec
IRNUC	1H
CTEMP	24.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.00 Hz
RGAIN	50

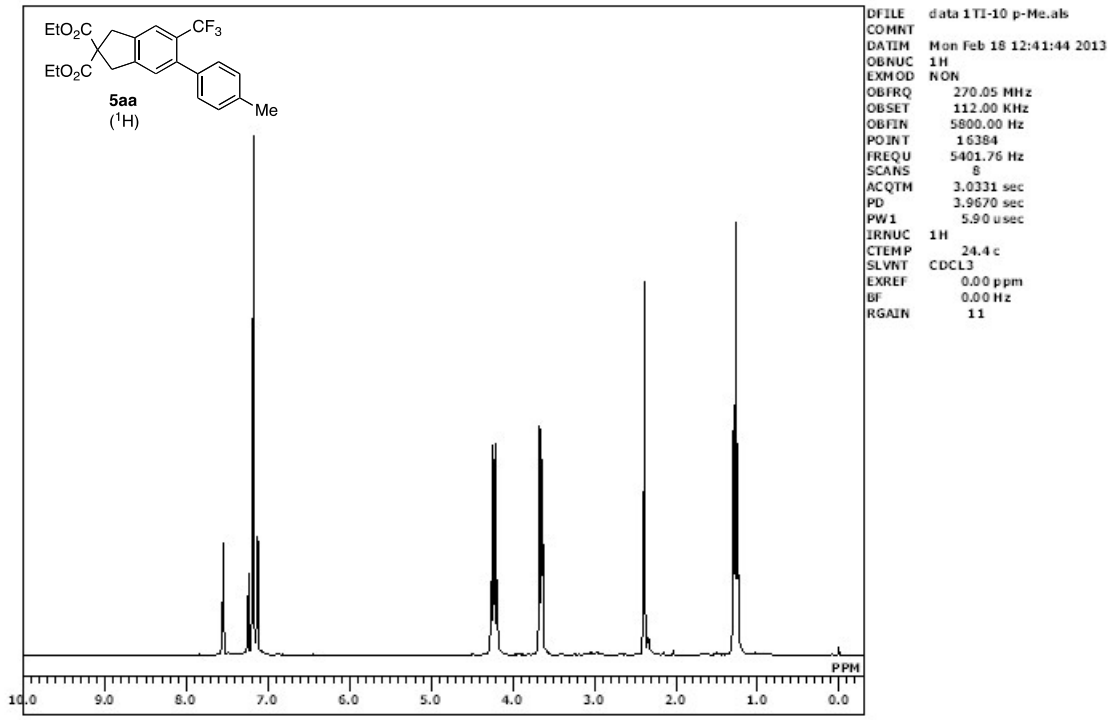


DFILE	3,5-Me-C6H4-pro-19f-
COMNT	
DATIM	19-12-2012 20:09:25
OBNUC	19F
EXMOD	single_pulse.ex2
OBFRQ	470.62 MHz
OBSET	0.46 KHz
OBFIN	0.84 Hz
POINT	131072
FREQU	240384.62 Hz
SCANS	8
ACQTM	0.5453 sec
PD	5.0000 sec
PW1	6.75 usec
IRNUC	19F
CTEMP	24.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	1.00 Hz
RGAIN	40

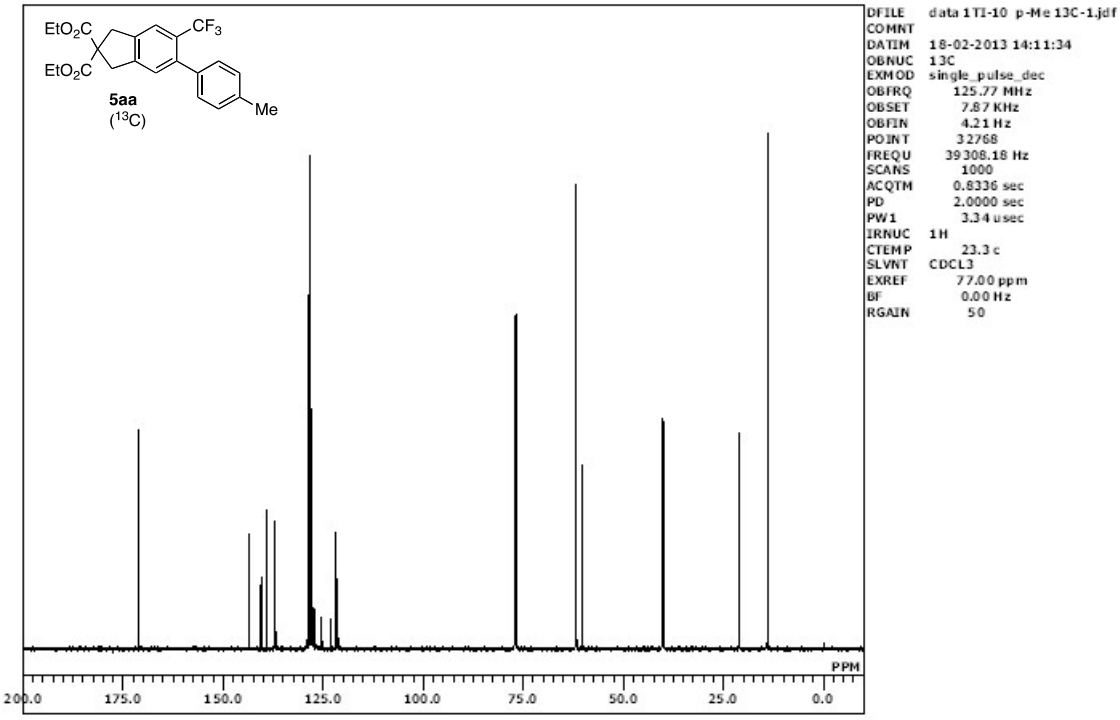

Ar
CF3
Ar
CF3
CF3
Ar
2l: Ar = 3-MeOC6H4
(1H)
DFILE 3-MeOC6H4-pro-1H-2
COMNT
DATIM 23-01-2013 12:29:31
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.61 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 26
PPM
10 9 8 7 6 5 4 3 2 1 0


Ar
CF3
Ar
CF3
CF3
Ar
2l: Ar = 3-MeOC6H4
(13C)
DFILE 3-MeOC6H4-pro-13C-
COMNT
DATIM 23-01-2013 13:05:39
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 400
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 26.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.00 Hz
RGAIN 58
PPM
175 150 125 100 75 50 25 0

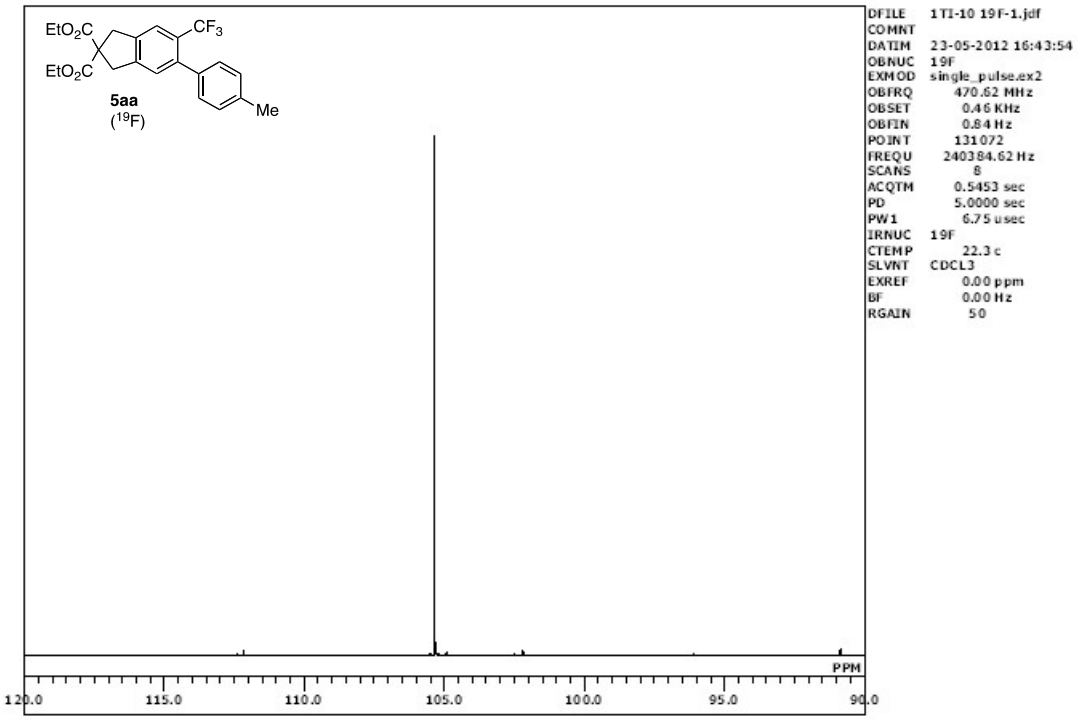

Ar
CF3
Ar
CF3
CF3
Ar
2l: Ar = 3-MeOC6H4
(19F)
DFILE 3-MeOC6H4-pro-19F-
COMNT
DATIM 23-01-2013 13:10:47
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 26.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.00 Hz
RGAIN 30
PPM
120.0 119.0 118.0 117.0 116.0 115.0 114.0 113.0 112.0 111.0 110.0 109.0 108.0 107.0 106.0



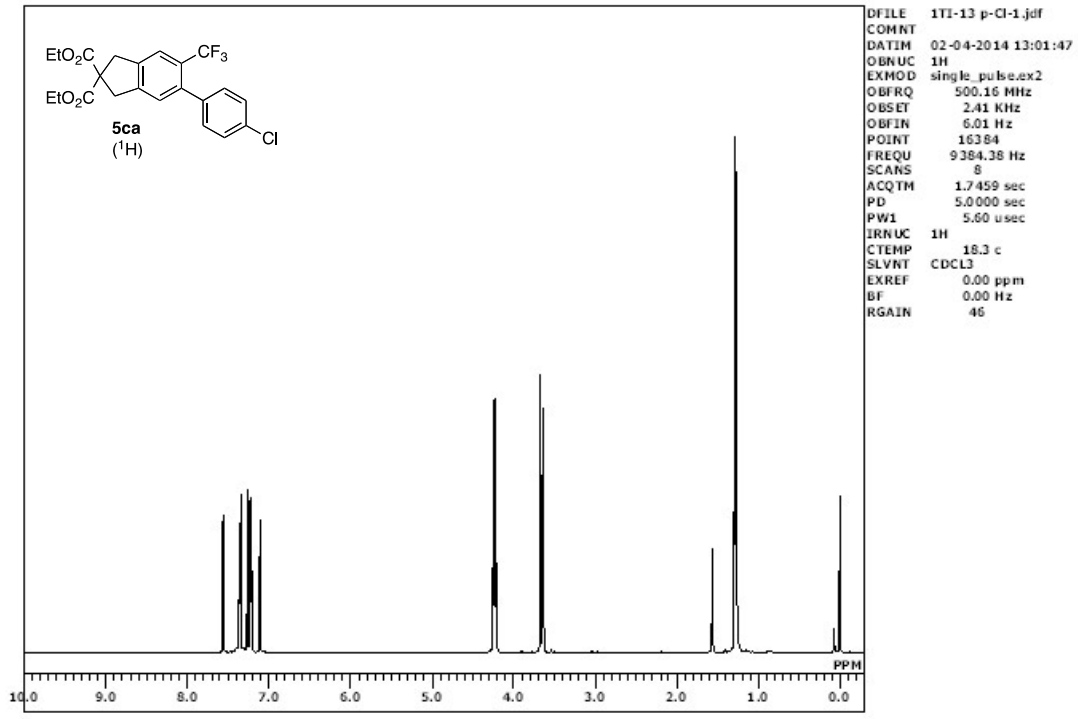

```
DFILE   data 1TI-10 p-Me.als
COMNT
DATIM   Mon Feb 18 12:41:44 2013
OBNUC   1H
EXMOD   NON
OBFRQ     270.05 MHz
OBSET     112.00 KHz
OBFIN    5800.00 Hz
POINT      16384
FREQU    5401.76 Hz
SCANS          8
ACQTM     3.0331 sec
PD        3.9670 sec
PW1        5.90 usec
IRNUC   1H
CTEMP      24.4 c
SLVNT   CDCL3
EXREF      0.00 ppm
BF         0.00 Hz
RGAIN        11
```



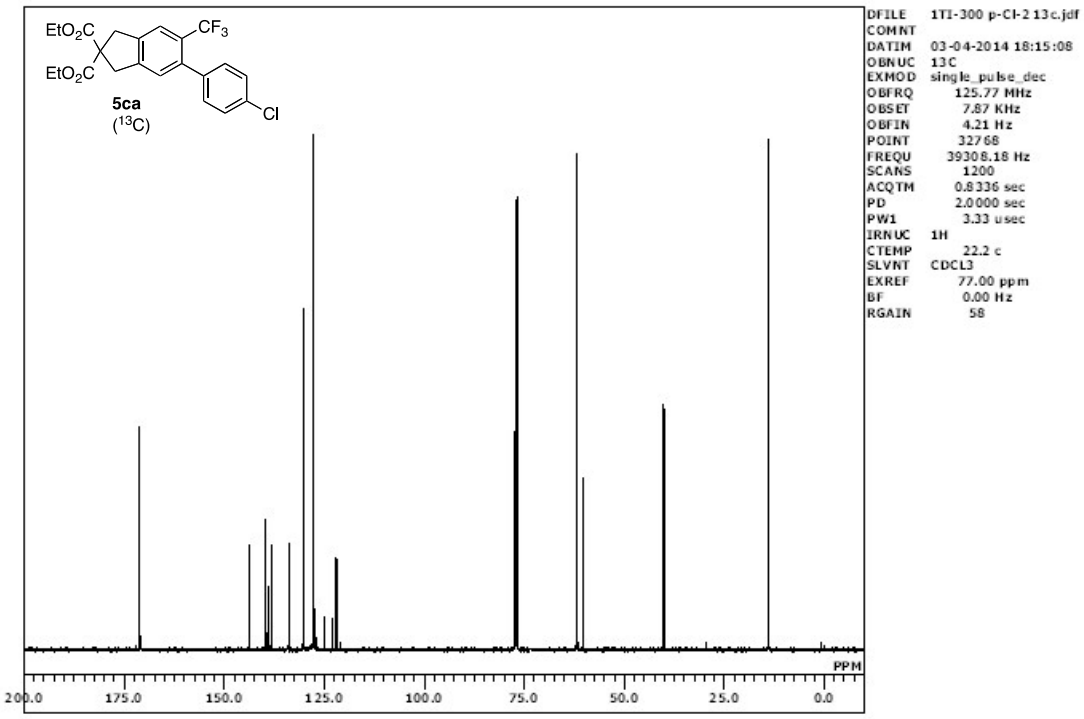

```
DFILE   data 1TI-10  p-Me 13C-1.jdf
COMNT
DATIM   18-02-2013 14:11:34
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ     125.77 MHz
OBSET       7.87 KHz
OBFIN       4.21 Hz
POINT      32768
FREQU   39308.18 Hz
SCANS        1000
ACQTM     0.8338 sec
PD        2.0000 sec
PW1        3.34 usec
IRNUC   1H
CTEMP      23.3 c
SLVNT   CDCL3
EXREF     77.00 ppm
BF         0.00 Hz
RGAIN        50
```

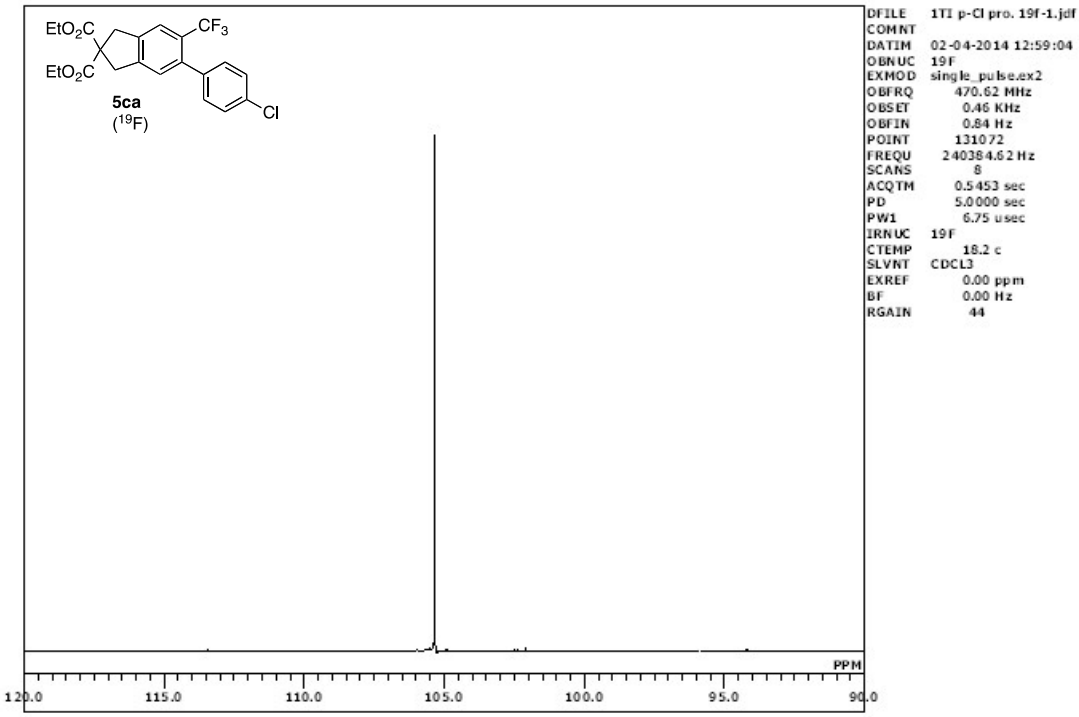


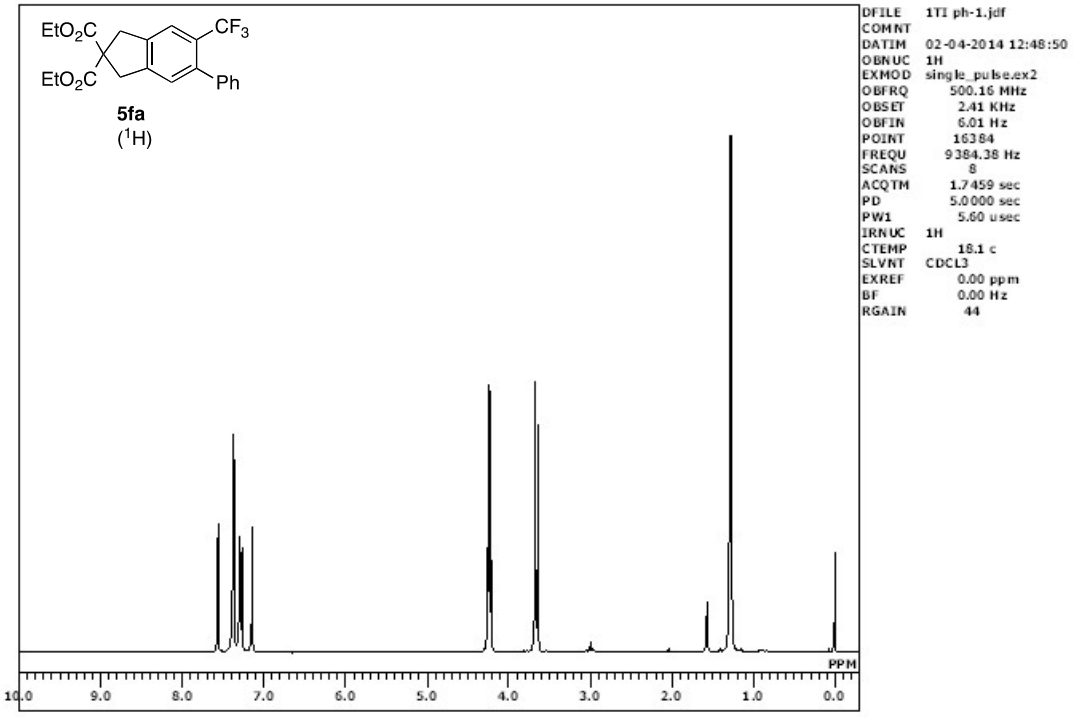

```
DFILE   1TI-10 19F-1.jdf
COMNT
DATIM   23-05-2012 16:43:54
OBNUC   19F
EXMOD   single_pulse.ex2
OBFRQ     470.62 MHz
OBSET       0.46 KHz
OBFIN       0.84 Hz
POINT     131072
FREQU   240384.62 Hz
SCANS          8
ACQTM       0.5453 sec
PD          5.0000 sec
PW1         6.75 usec
IRNUC   19F
CTEMP      22.3 c
SLVNT   CDCL3
EXREF       0.00 ppm
BF          0.00 Hz
RGAIN         50
```

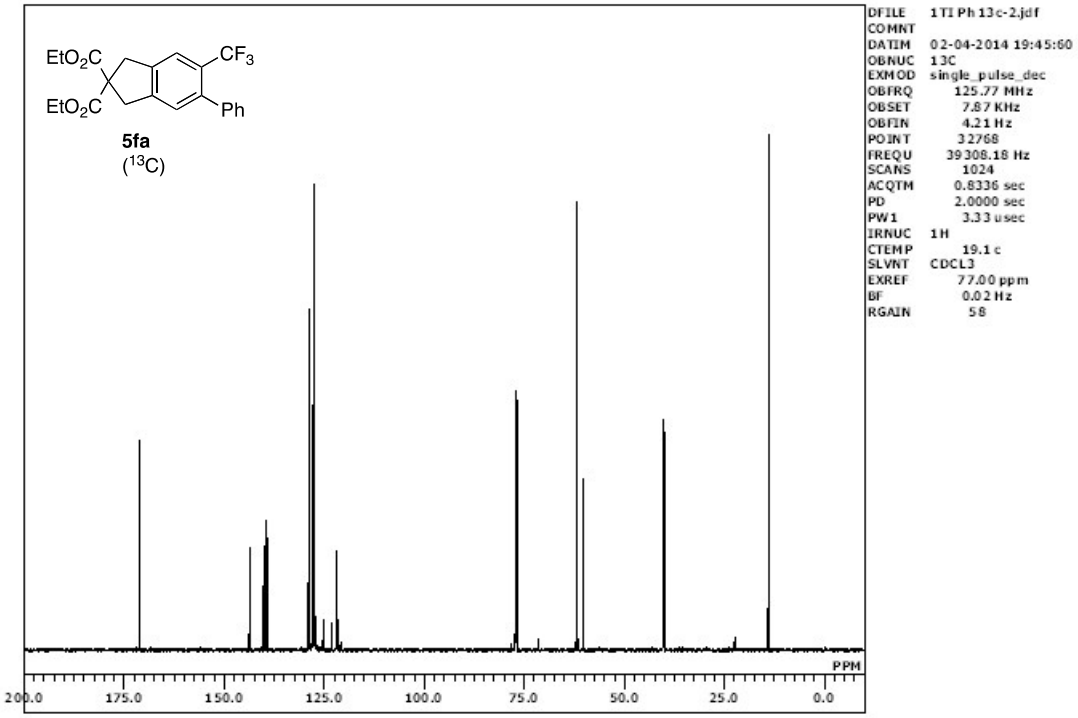


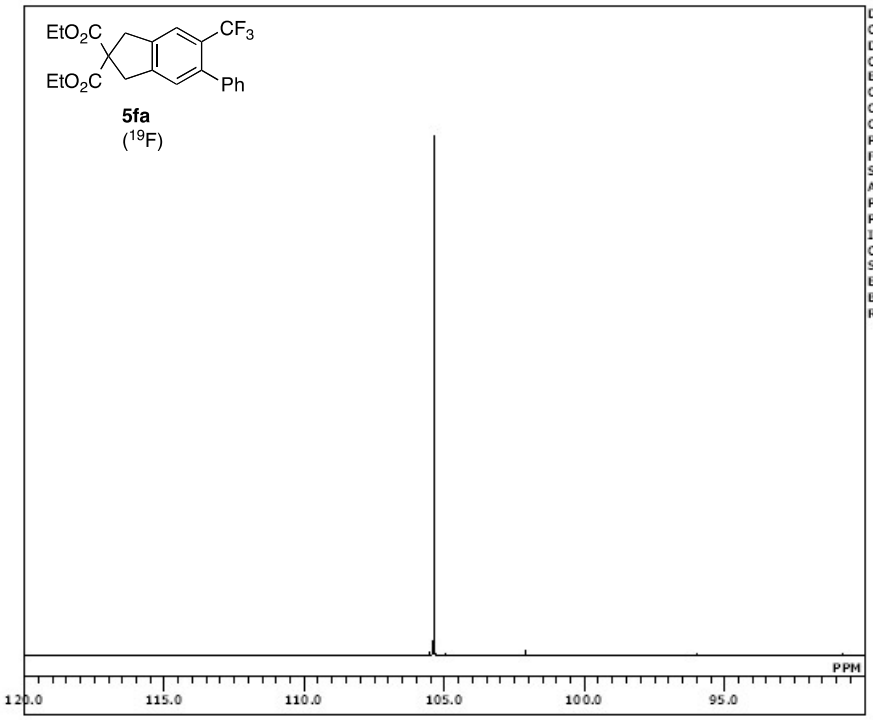

```
DFILE   1TI-13 p-Cl-1.jdf
COMNT
DATIM   02-04-2014 13:01:47
OBNUC   1H
EXMOD   single_pulse.ex2
OBFRQ     500.16 MHz
OBSET       2.41 KHz
OBFIN       6.01 Hz
POINT       16384
FREQU     9384.38 Hz
SCANS          8
ACQTM       1.7459 sec
PD          5.0000 sec
PW1         5.60 usec
IRNUC   1H
CTEMP      18.3 c
SLVNT   CDCL3
EXREF       0.00 ppm
BF          0.00 Hz
RGAIN         46
```


EtO2C
EtO2C
CF3
Cl
5ca
(13C)
DFILE 1TI-300 p-Cl-2 13c.jdf
COMNT
DATIM 03-04-2014 18:15:08
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 1200
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 22.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 58
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0

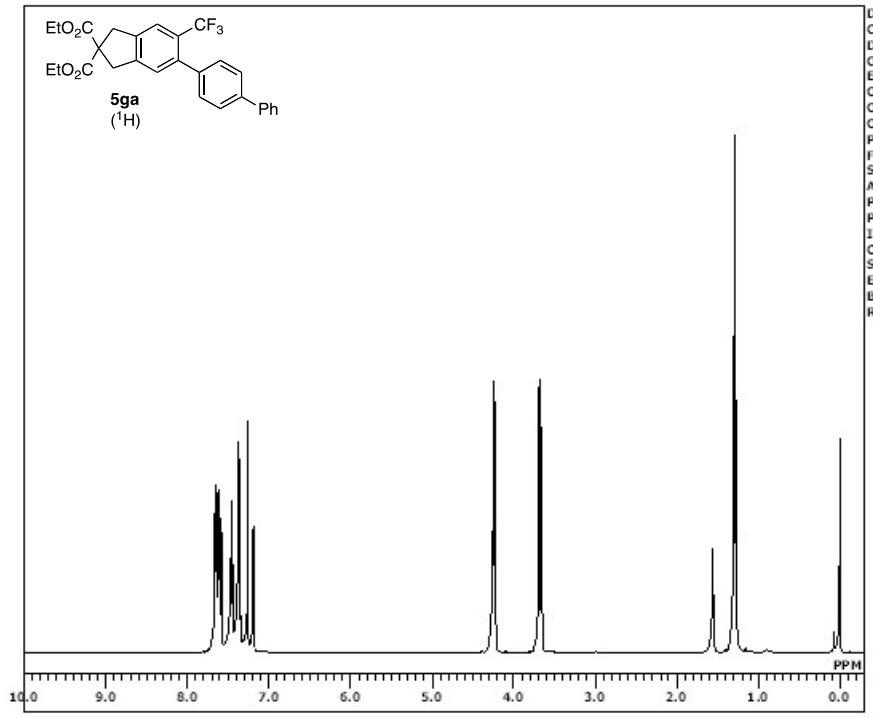

EtO2C
EtO2C
CF3
Cl
5ca
(19F)
DFILE 1TI p-Cl pro. 19f-1.jdf
COMNT
DATIM 02-04-2014 12:59:04
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 18.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 44
PPM
120.0
115.0
110.0
105.0
100.0
95.0
90.0


EtO2C
EtO2C
CF3
Ph
5fa
(1H)
DFILE 1TI ph-1.jdf
COMNT
DATIM 02-04-2014 12:48:50
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 18.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 44
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

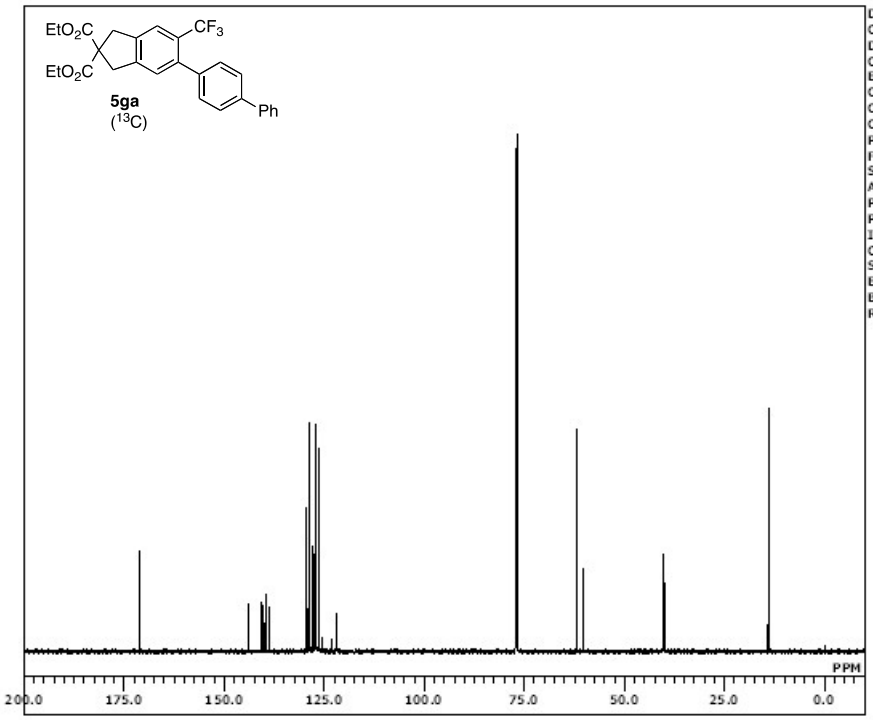

EtO2C
EtO2C
CF3
Ph
5fa
(13C)
DFILE 1TI Ph 13c-2.jdf
COMNT
DATIM 02-04-2014 19:45:60
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.02 Hz
RGAIN 58
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0



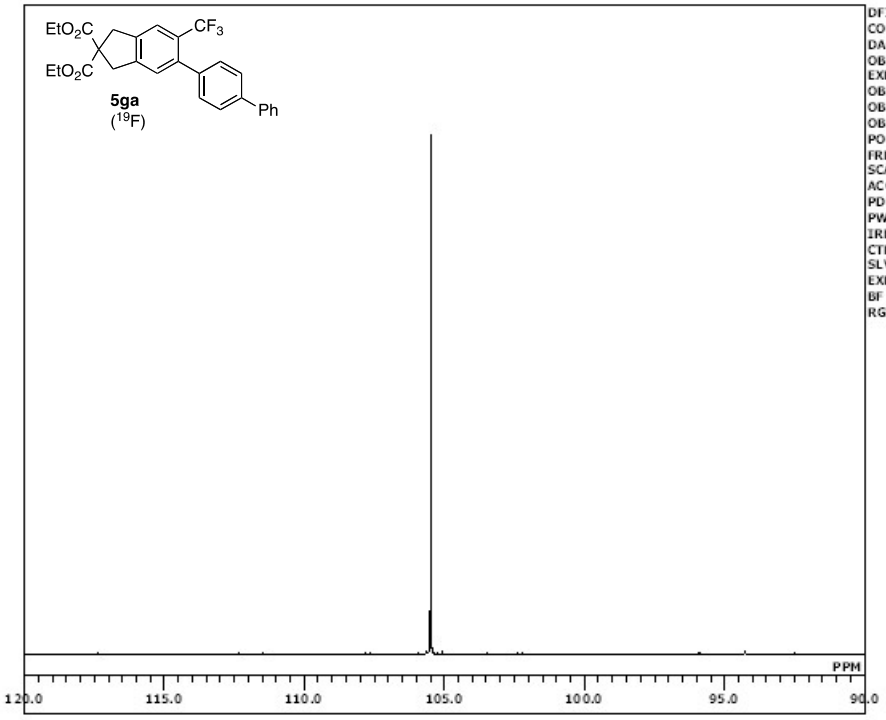
DFILE 1TI ph 19f-1.jdf
COMNT
DATIM 02-04-2014 12:52:55
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 18.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 44



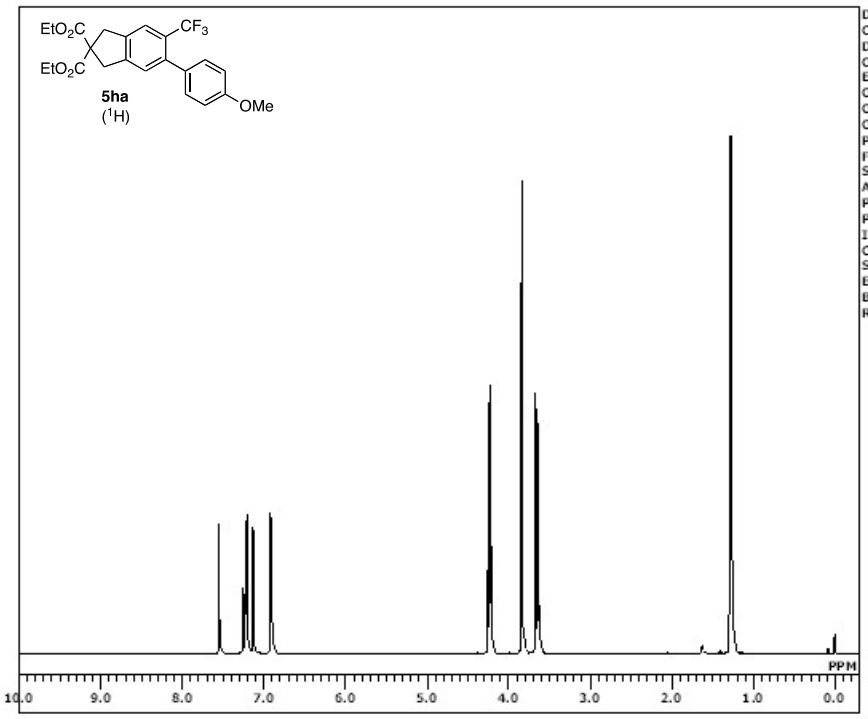
DFILE 1TI p-Ph pro. -1.jdf
COMNT
DATIM 02-04-2014 14:18:20
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 48



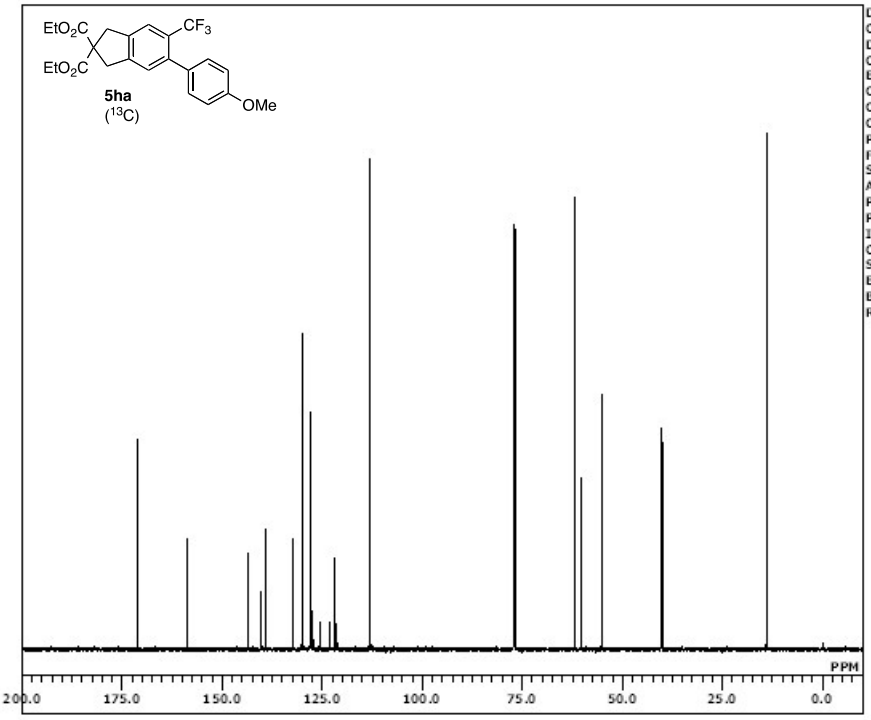
DFILE 1TI p-Ph 13c-2.jdf
COMNT
DATIM 02-04-2014 20:42:46
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 1024
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.02 Hz
RGAIN 56



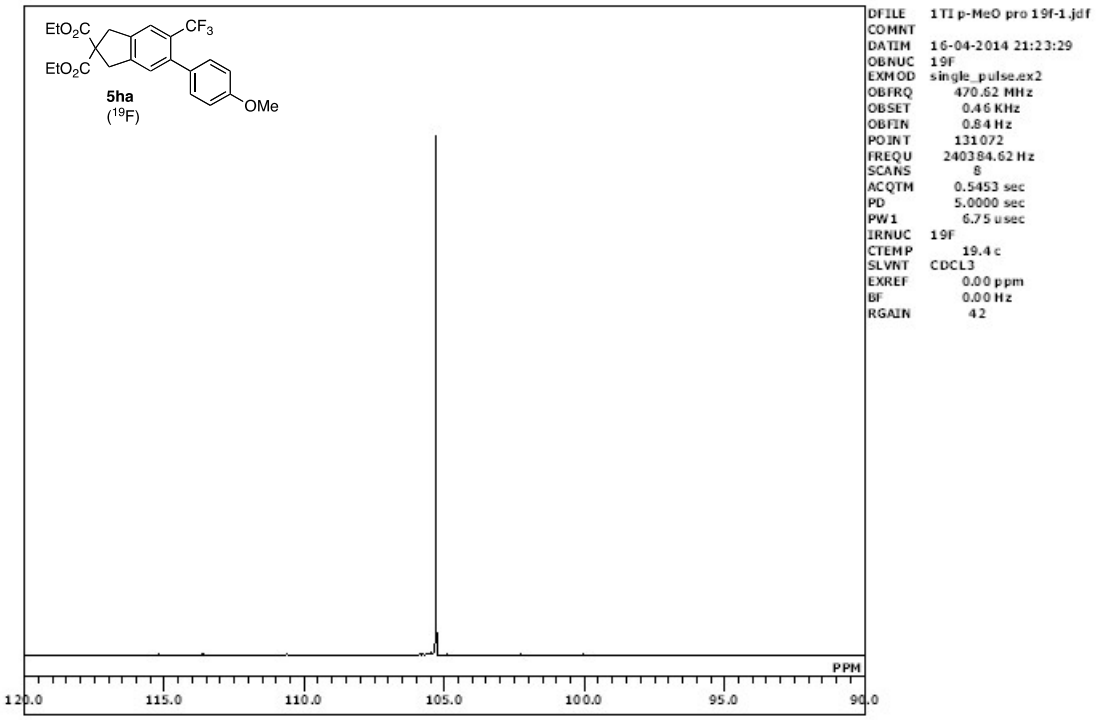
DFILE 1TI p-Ph pro. 19f-1.jdf
COMNT
DATIM 02-04-2014 14:14:48
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 18.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 44



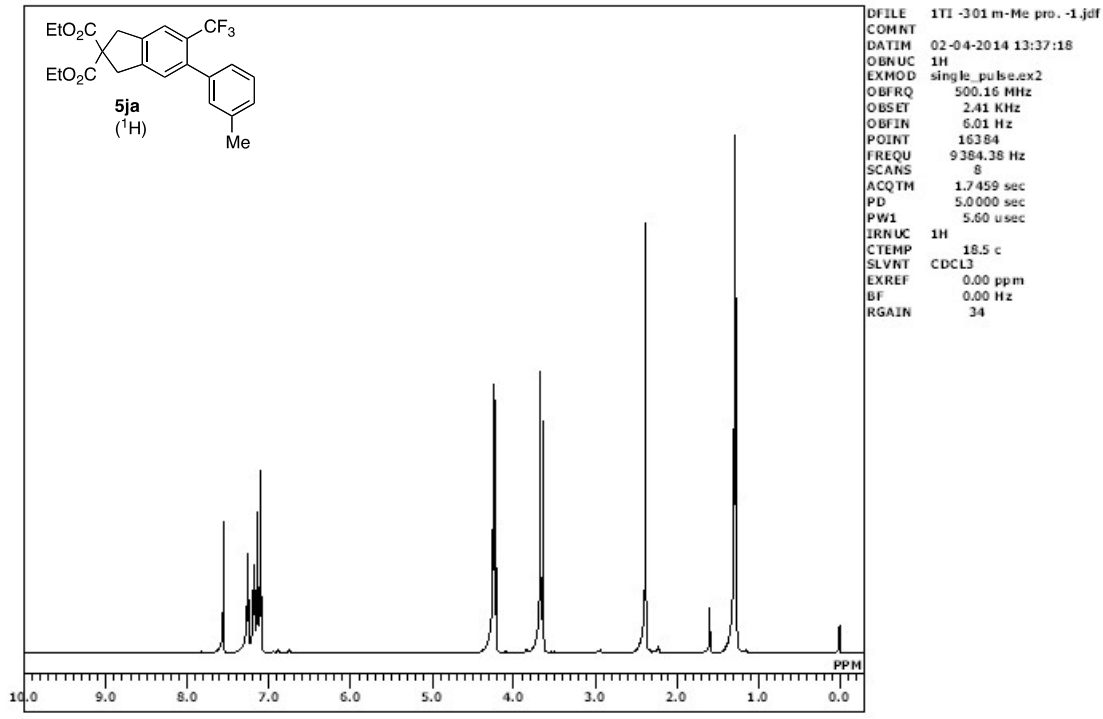
DFILE 1TI p-MeO pro-2.jdf
COMNT
DATIM 16-04-2014 21:19:15
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 19.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 30



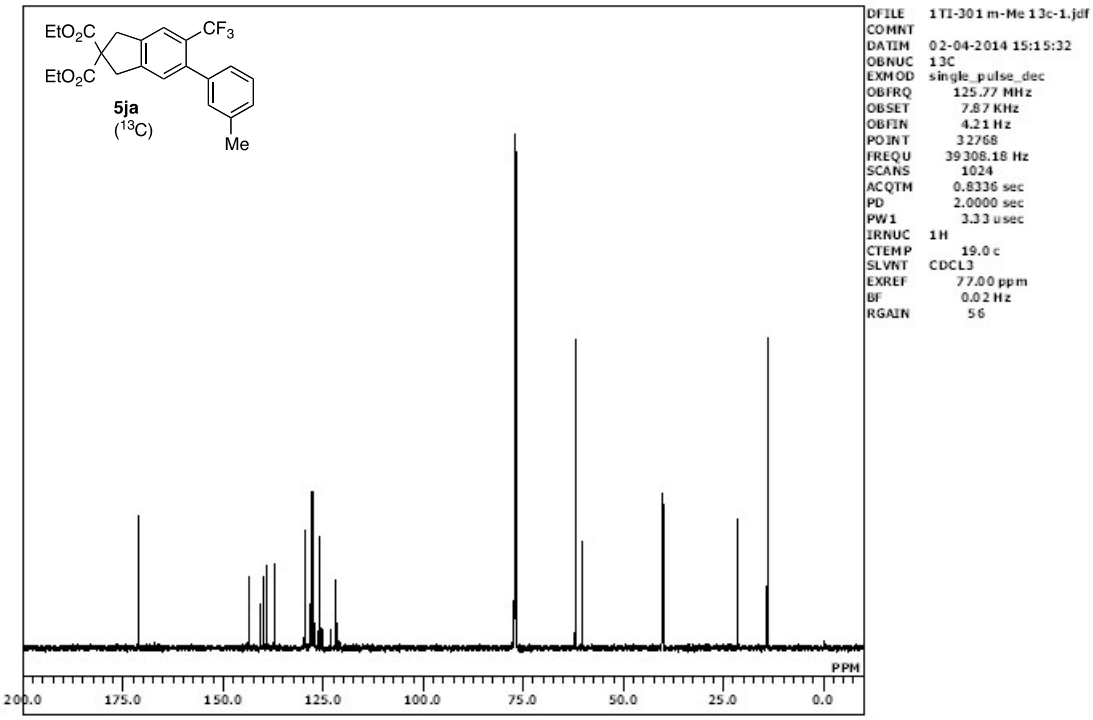
DFILE 1TI p-MeO pro 13c-1.jdf
COMNT
DATIM 16-04-2014 22:15:22
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 1024
ACQTM 0.8338 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 20.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.02 Hz
RGAIN 58



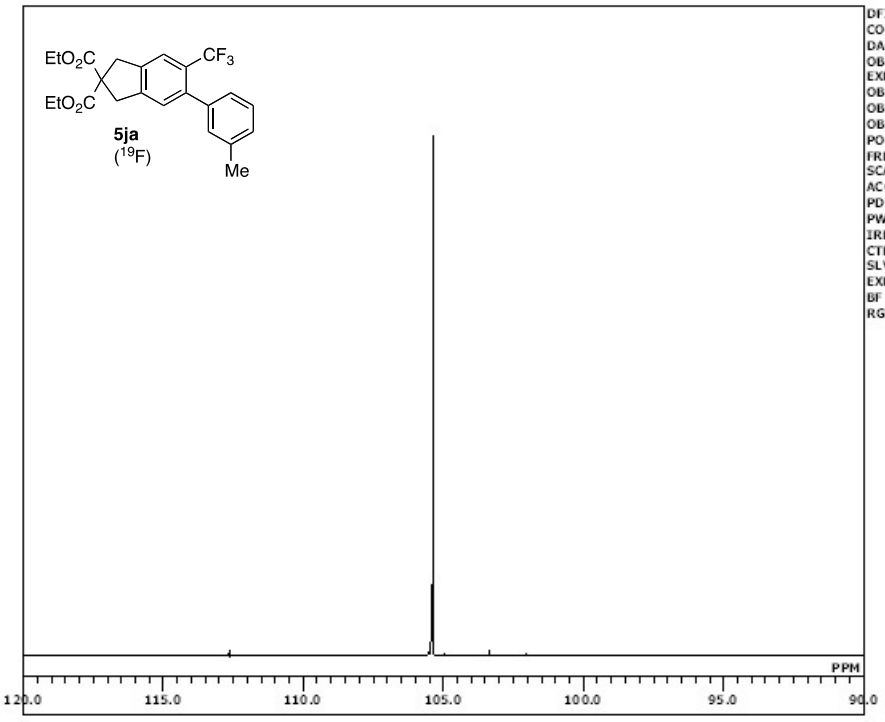

```
DFILE   1TI p-MeO pro 19f-1.jdf
COMNT
DATIM   16-04-2014 21:23:29
OBNUC   19F
EXMOD   single_pulse.ex2
OBFRQ     470.62 MHz
OBSET       0.46 KHz
OBFIN       0.84 Hz
POINT     131072
FREQU   240384.62 Hz
SCANS          8
ACQTM       0.5453 sec
PD          5.0000 sec
PW1         6.75 usec
IRNUC   19F
CTEMP      19.4 c
SLVNT   CDCL3
EXREF       0.00 ppm
BF          0.00 Hz
RGAIN         42
```



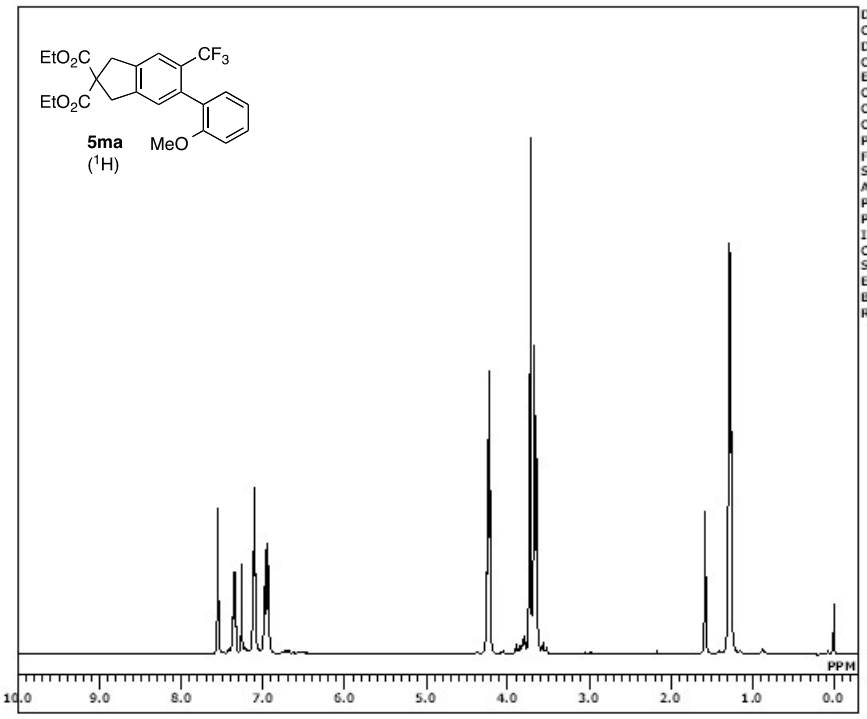

```
DFILE   1TI -301 m-Me pro. -1.jdf
COMNT
DATIM   02-04-2014 13:37:18
OBNUC   1H
EXMOD   single_pulse.ex2
OBFRQ     500.16 MHz
OBSET       2.41 KHz
OBFIN       6.01 Hz
POINT       16384
FREQU     9384.38 Hz
SCANS          8
ACQTM       1.7459 sec
PD          5.0000 sec
PW1         5.60 usec
IRNUC   1H
CTEMP      18.5 c
SLVNT   CDCL3
EXREF       0.00 ppm
BF          0.00 Hz
RGAIN         34
```

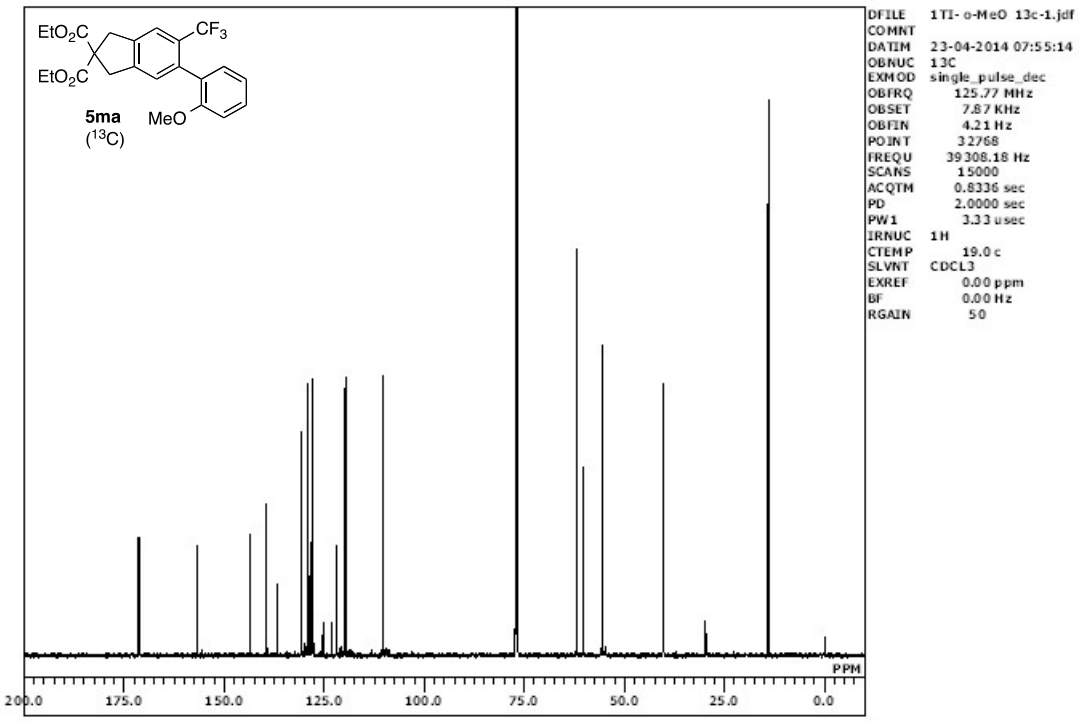


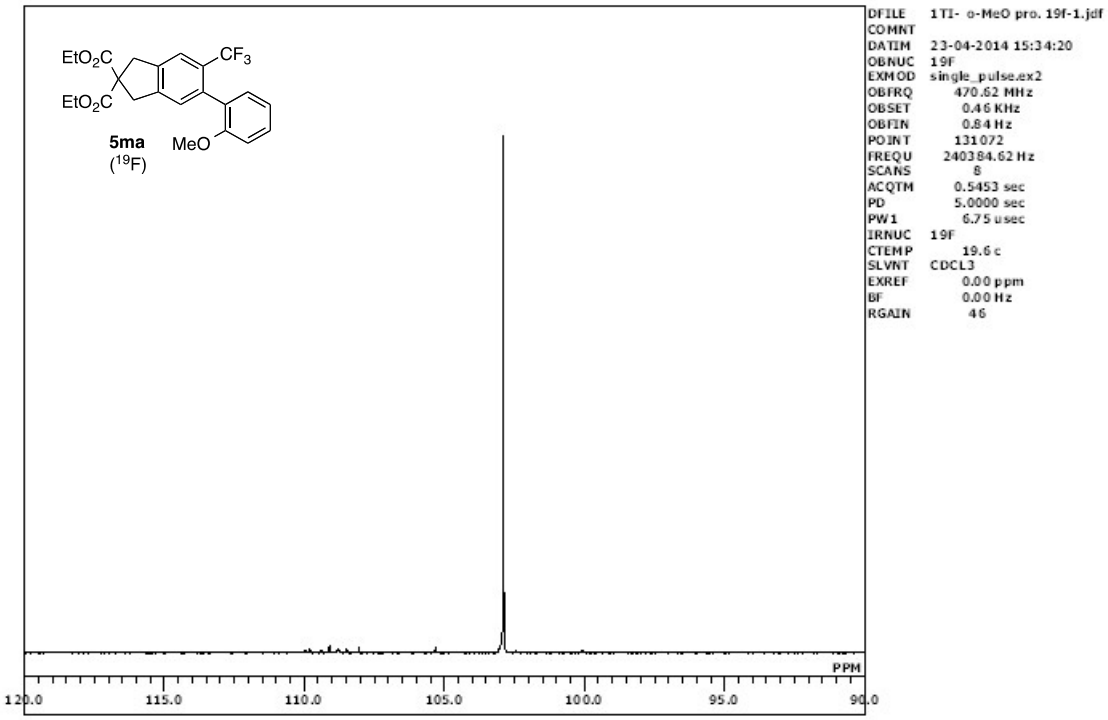

```
DFILE   1TI-301 m-Me 13c-1.jdf
COMNT
DATIM   02-04-2014 15:15:32
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ     125.77 MHz
OBSET       7.87 KHz
OBFIN       4.21 Hz
POINT      32768
FREQU   39308.18 Hz
SCANS       1024
ACQTM     0.8336 sec
PD        2.0000 sec
PW1        3.33 usec
IRNUC   1H
CTEMP      19.0 c
SLVNT   CDCL3
EXREF     77.00 ppm
BF         0.02 Hz
RGAIN        56
```

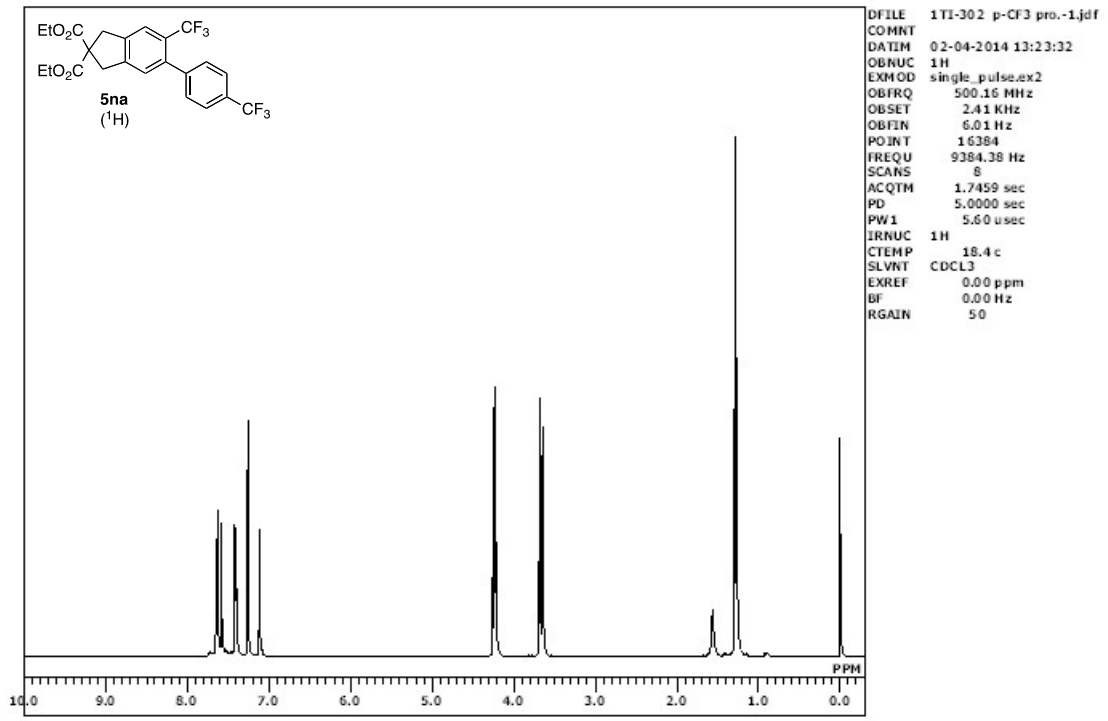


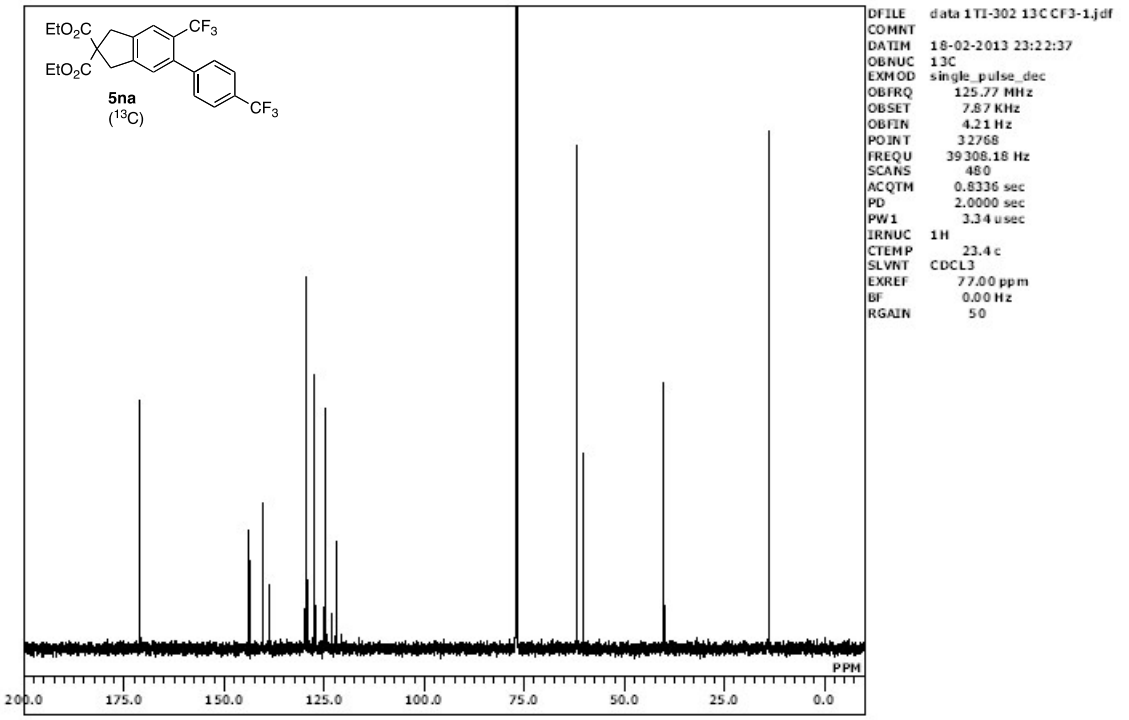

```
DFILE   1TI-301 m-Me pro. 19f-1.jdf
COMNT
DATIM   02-04-2014 13:34:07
OBNUC   19F
EXMOD   single_pulse.ex2
OBFRQ     470.62 MHz
OBSET       0.46 KHz
OBFIN       0.84 Hz
POINT     131072
FREQU   240384.62 Hz
SCANS          8
ACQTM     0.5453 sec
PD        5.0000 sec
PW1        6.75 usec
IRNUC   19F
CTEMP      18.4 c
SLVNT   CDCL3
EXREF      0.00 ppm
BF         0.00 Hz
RGAIN        42
```

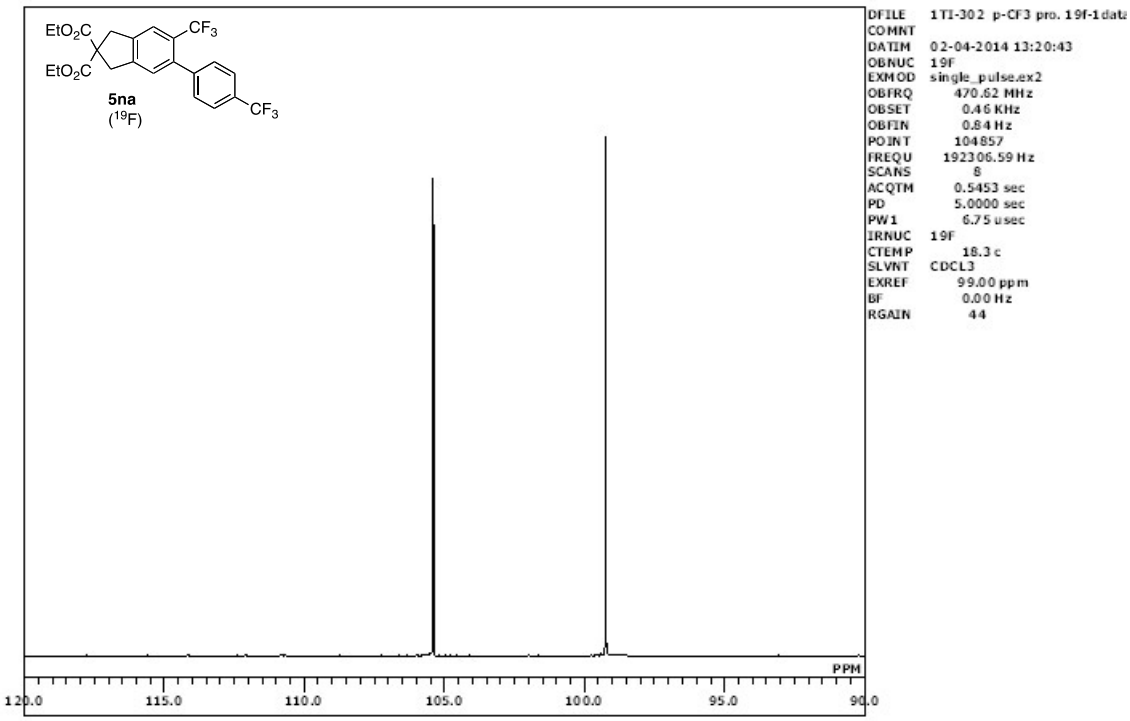

EtO2C
EtO2C
CF3
MeO
5ma
(1H)
DFILE 1TI- o-MeO -2 .jdf
COMNT
DATIM 22-04-2014 20:04:59
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 16
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 40
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

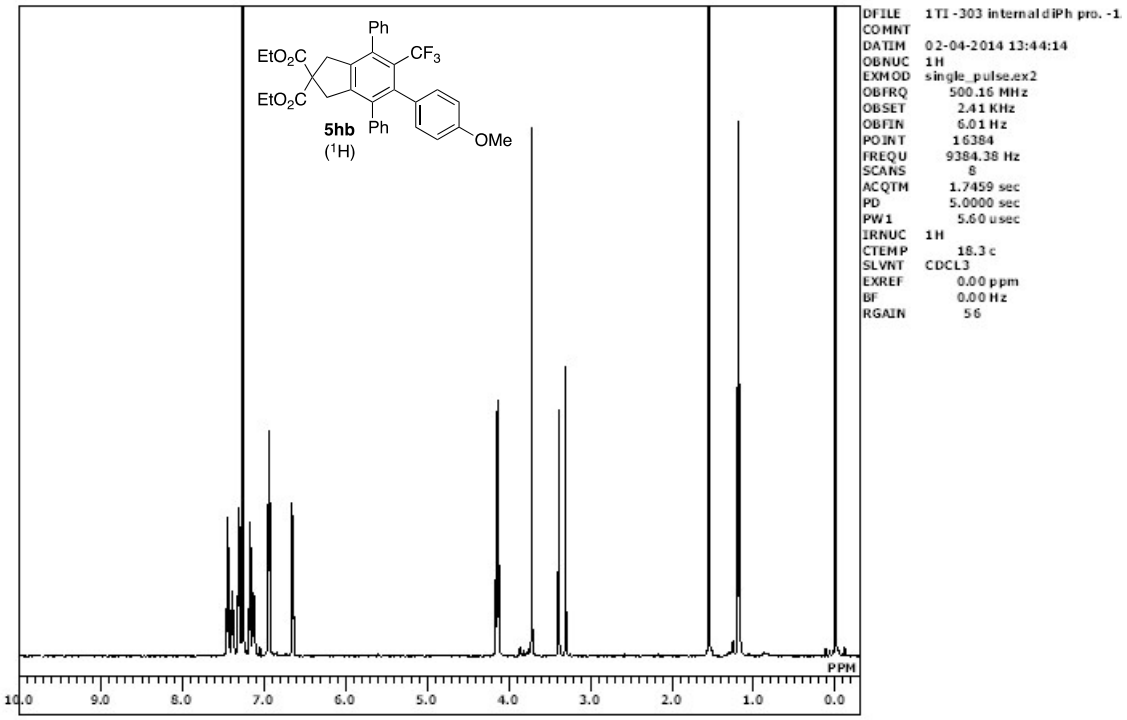

EtO2C
EtO2C
CF3
MeO
5ma
(13C)
DFILE 1TI- o-MeO 13c-1.jdf
COMNT
DATIM 23-04-2014 07:55:14
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 15000
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 50
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0

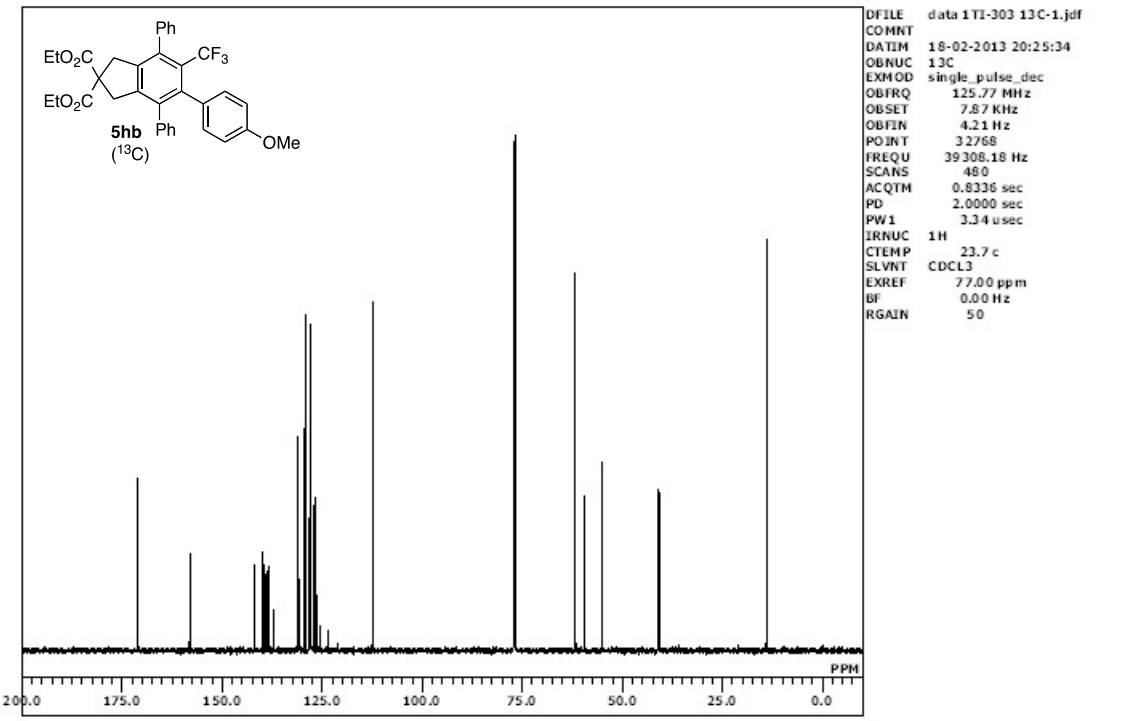

EtO2C
EtO2C
CF3
MeO
5ma
(19F)
DFILE 1TI- o-MeO pro. 19f-1.jdf
COMNT
DATIM 23-04-2014 15:34:20
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 19.6 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 46
PPM
120.0
115.0
110.0
105.0
100.0
95.0
90.0

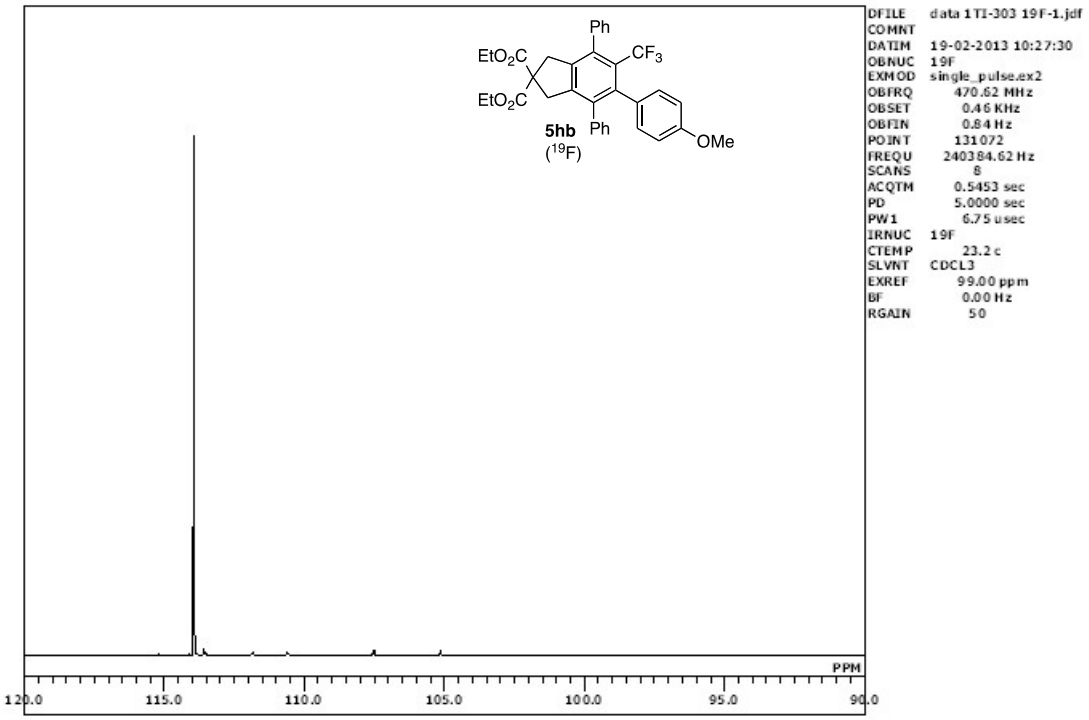

EtO2C
EtO2C
CF3
CF3
5na
(1H)
DFILE 1TI-302 p-CF3 pro.-1.jdf
COMNT
DATIM 02-04-2014 13:23:32
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 18.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 50
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


EtO2C
EtO2C
CF3
CF3
5na
(13C)
DFILE data 1TI-302 13C CF3-1.jdf
COMNT
DATIM 18-02-2013 23:22:37
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 480
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 23.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 50
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0

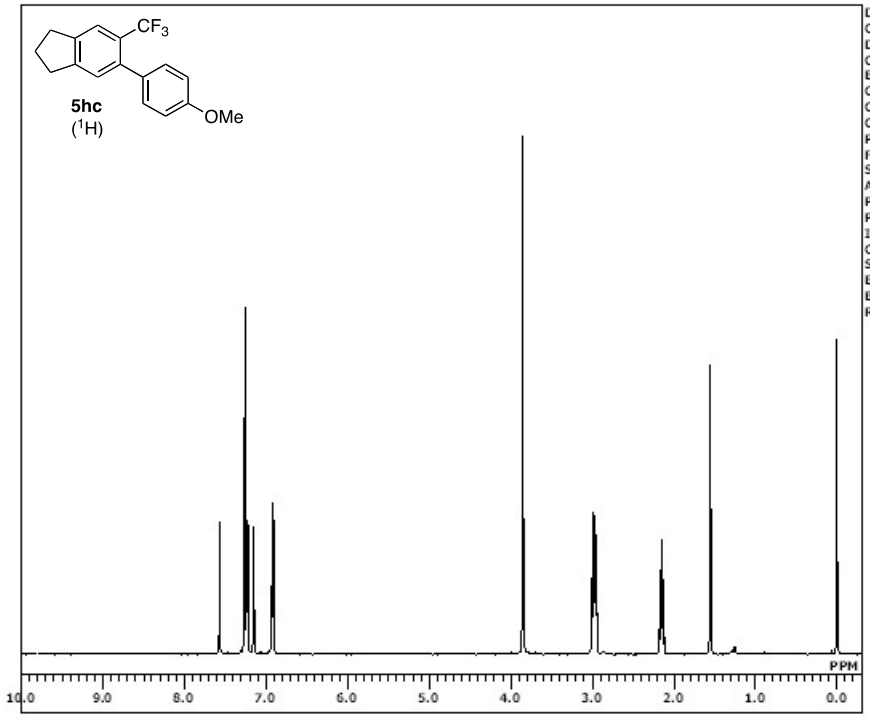

EtO2C
EtO2C
CF3
CF3
5na
(19F)
DFILE 1TI-302 p-CF3 pro. 19f-1 data
COMNT
DATIM 02-04-2014 13:20:43
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 104857
FREQU 192306.59 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 18.3 c
SLVNT CDCL3
EXREF 99.00 ppm
BF 0.00 Hz
RGAIN 44
PPM
120.0
115.0
110.0
105.0
100.0
95.0
90.0


Ph
EtO2C
CF3
EtO2C
5hb
(1H)
Ph
OMe
DFILE 1TI-303 internal diPh pro. -1
COMNT
DATIM 02-04-2014 13:44:14
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 18.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 56
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

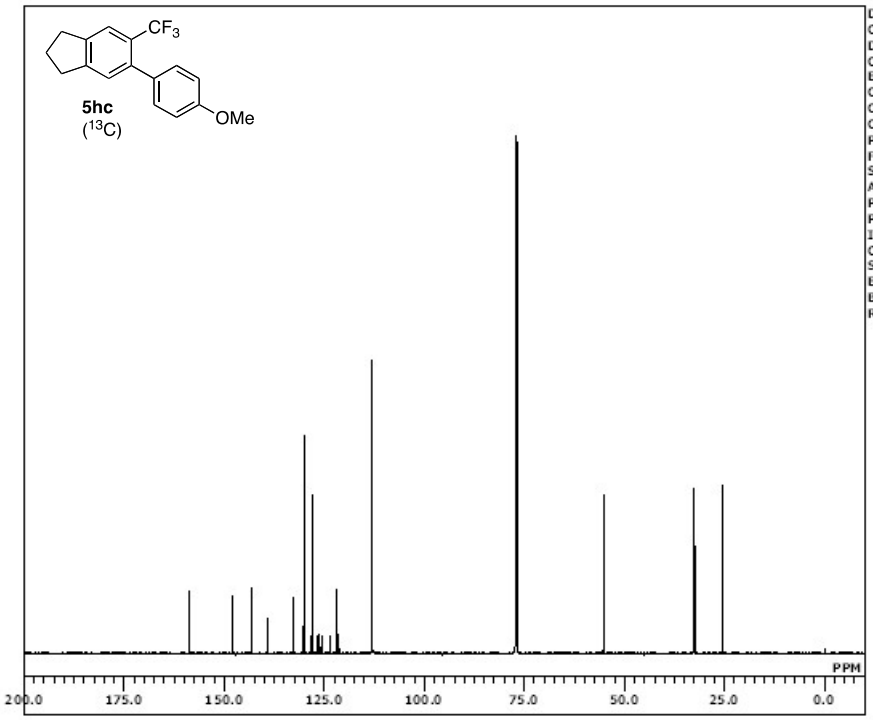

Ph
EtO2C
CF3
EtO2C
5hb
(13C)
Ph
OMe
DFILE data 1TI-303 13C-1.jdf
COMNT
DATIM 18-02-2013 20:25:34
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 480
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 50
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0



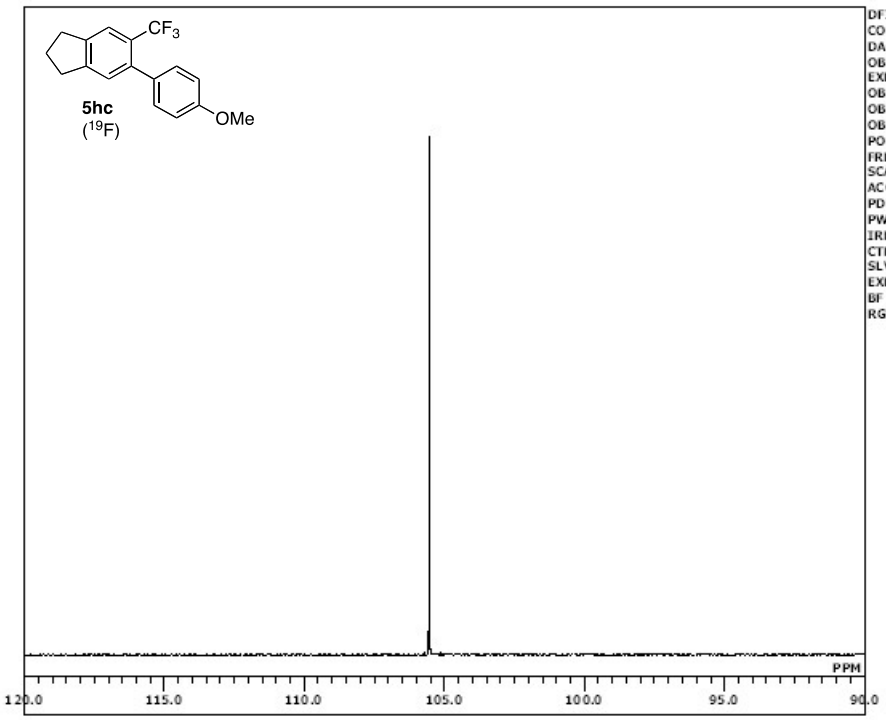
DFILE data 1TI-303 19F-1.jdf
COMNT
DATIM 19-02-2013 10:27:30
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 23.2 c
SLVNT CDCL3
EXREF 99.00 ppm
BF 0.00 Hz
RGAIN 50



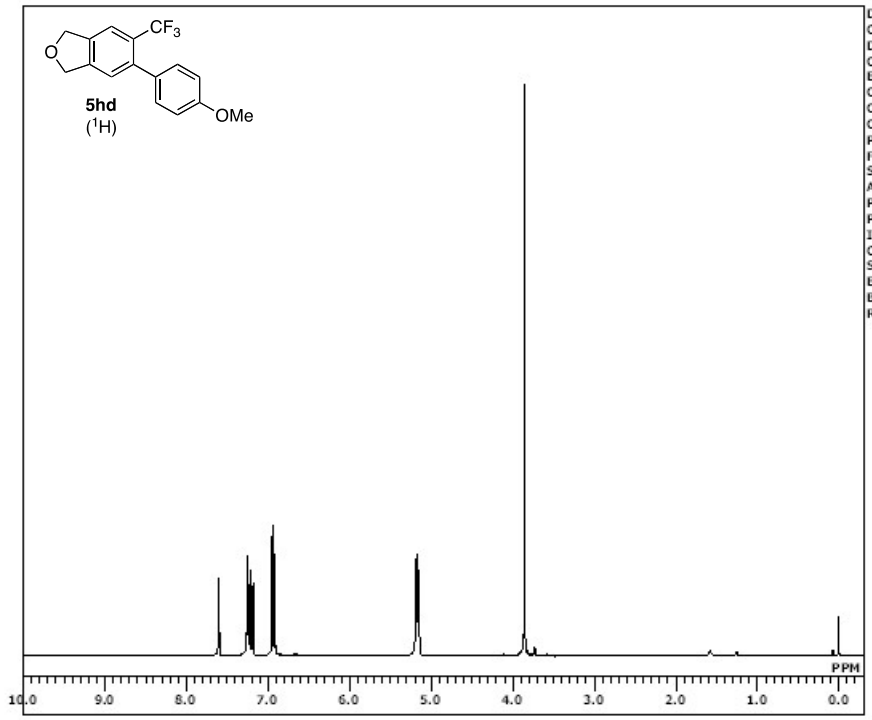
DFILE 1TI heptadiyne pro. -1.jdf
COMNT
DATIM 02-04-2014 13:50:22
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 18.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.00 Hz
RGAIN 50



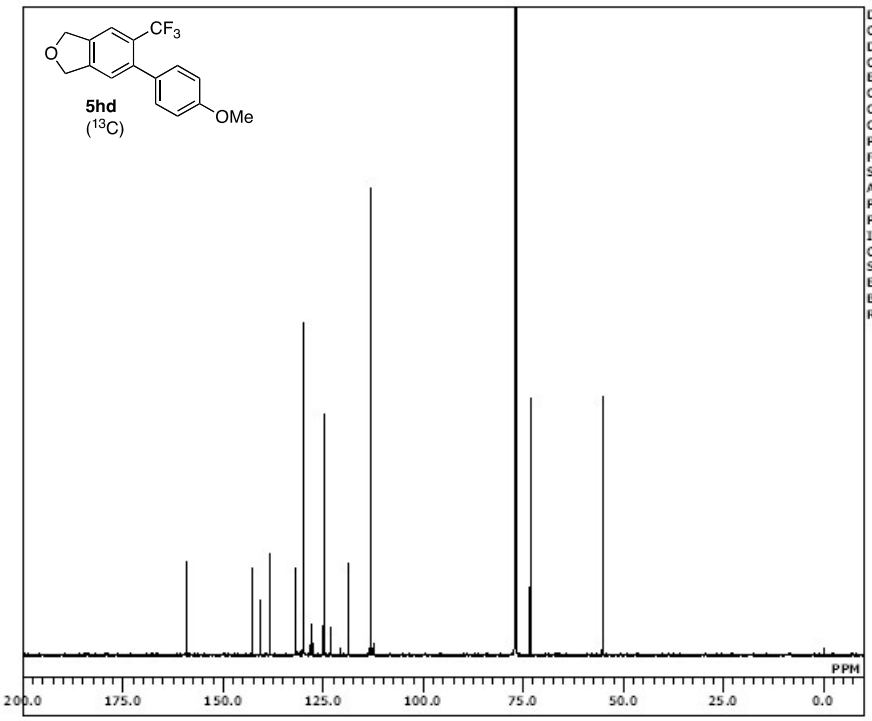
DFILE 1TI Heptadiyne pro. 13c-1.jdf
COMNT
DATIM 03-04-2014 06:11:36
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 9600
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 18.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 56



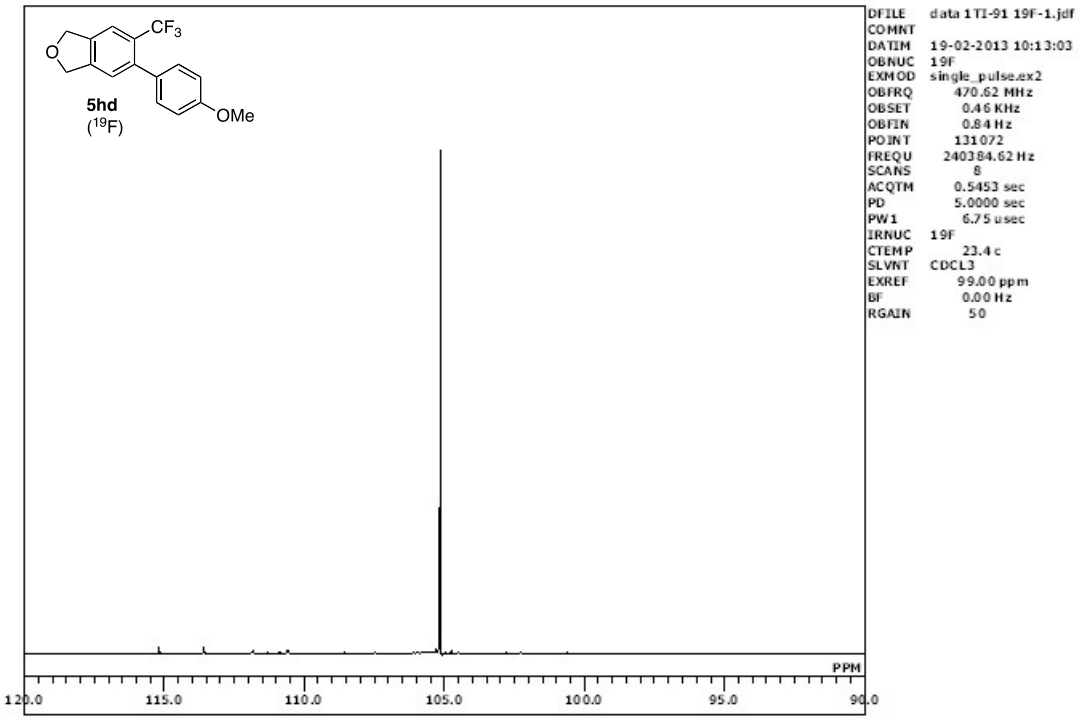
DFILE 1TI heptadiyne pro. 19f-1.jd
COMNT
DATIM 02-04-2014 13:53:05
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 18.6 c
SLVNT CDCL3
EXREF 99.00 ppm
BF 0.00 Hz
RGAIN 44



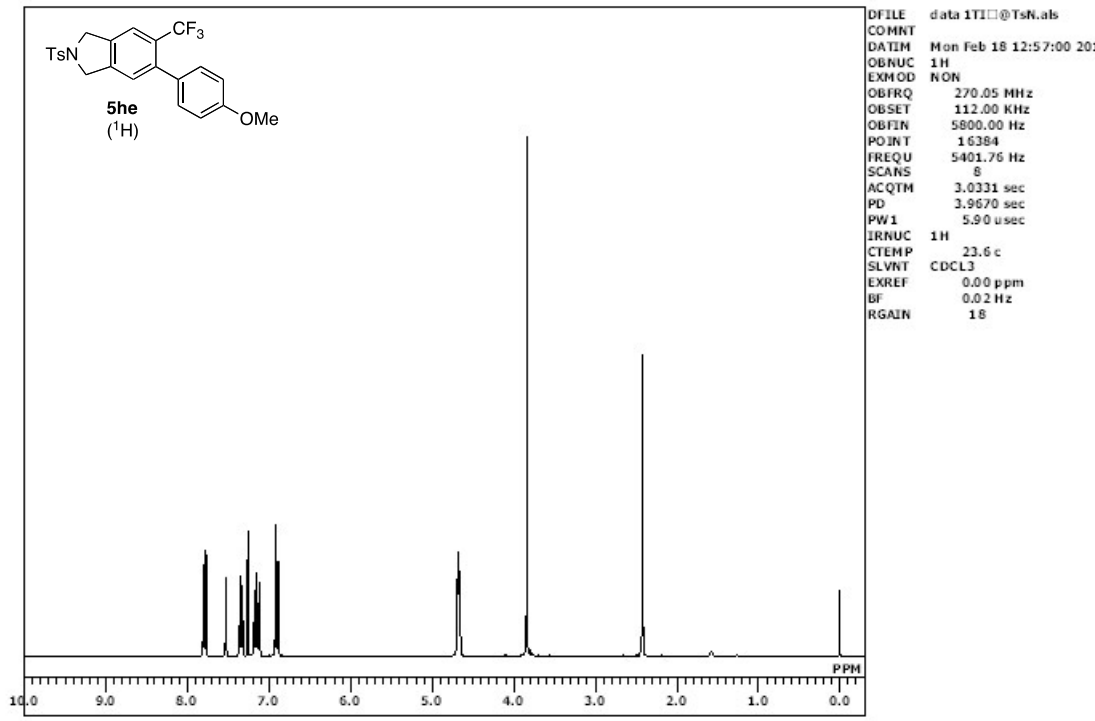
DFILE data 1TI-91.als
COMNT
DATIM Mon Feb 18 12:49:32 2013
OBNUC 1H
EXMOD NON
OBFRQ 270.05 MHz
OBSET 112.00 KHz
OBFIN 5800.00 Hz
POINT 16384
FREQU 5401.76 Hz
SCANS 8
ACQTM 3.0331 sec
PD 3.9670 sec
PW1 5.90 usec
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.02 Hz
RGAIN 18



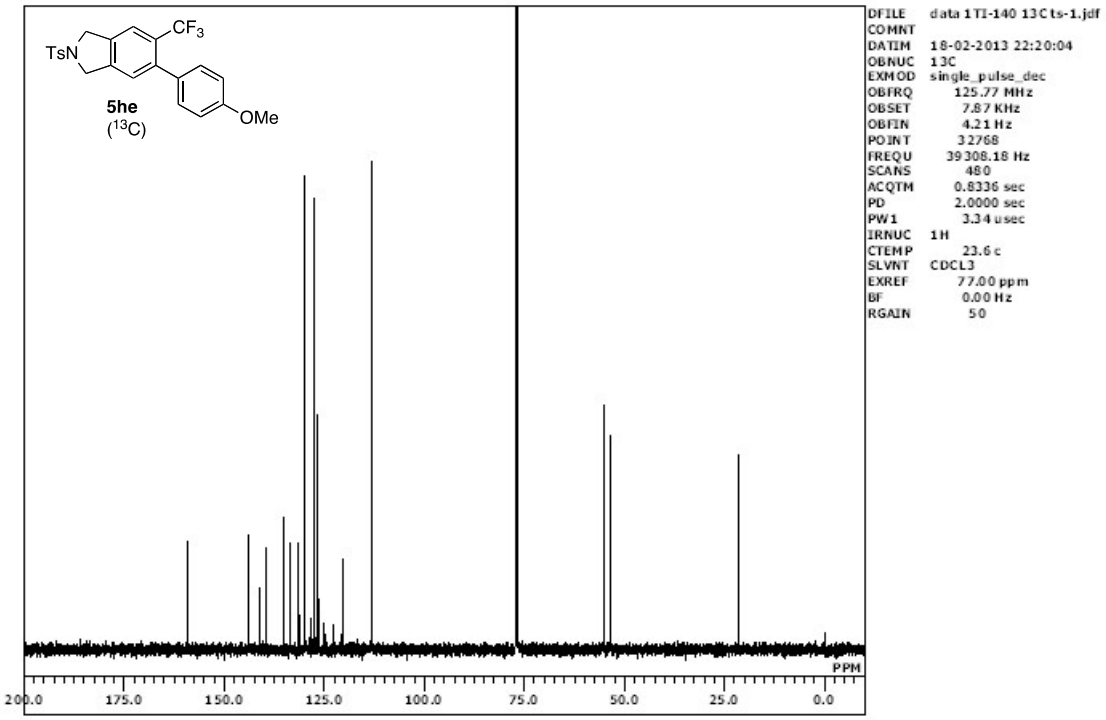
DFILE 1TI propargyl ether pro 13c-1
COMNT
DATIM 17-04-2014 07:19:20
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 11264
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 58

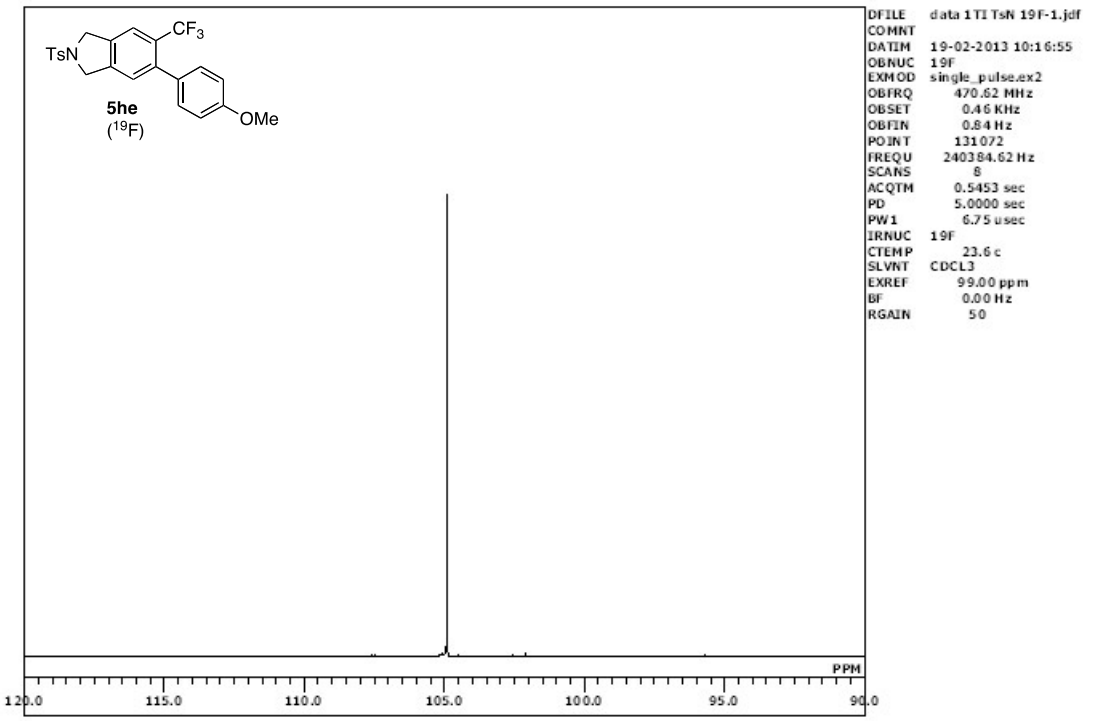


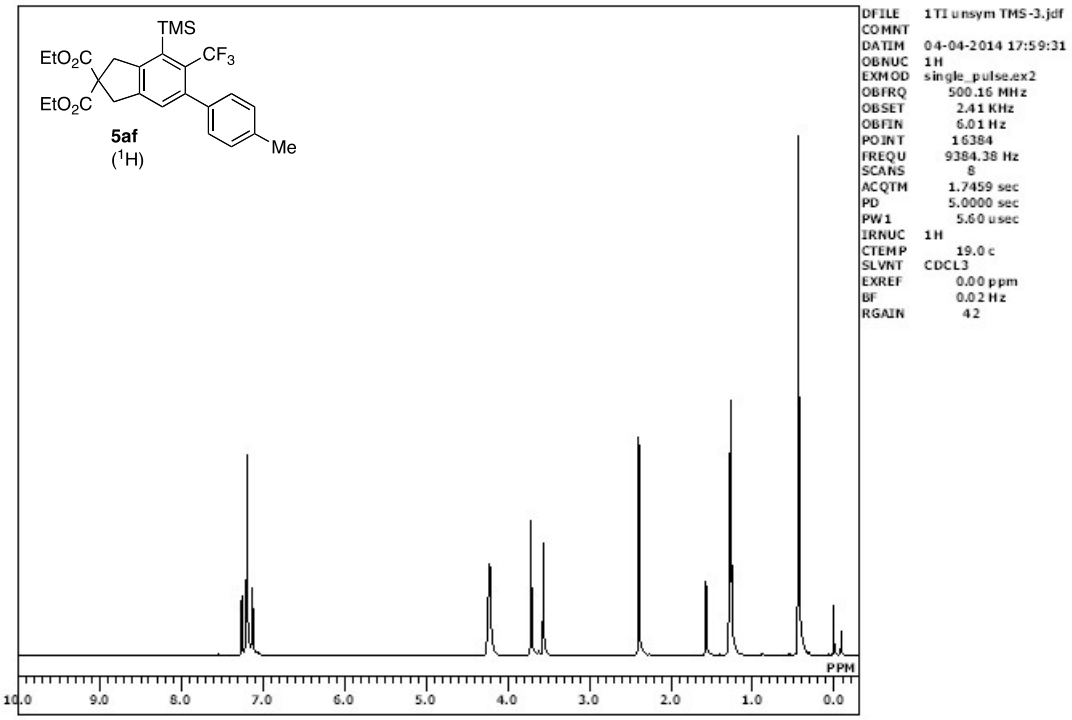

```
DFILE   data 1TI-91 19F-1.jdf
COMNT
DATIM   19-02-2013 10:13:03
OBNUC   19F
EXMOD   single_pulse.ex2
OBFRQ     470.62 MHz
OBSET       0.46 KHz
OBFIN       0.84 Hz
POINT     131072
FREQU   240384.62 Hz
SCANS          8
ACQTM       0.5453 sec
PD          5.0000 sec
PW1         6.75 usec
IRNUC   19F
CTEMP      23.4 c
SLVNT   CDCL3
EXREF      99.00 ppm
BF          0.00 Hz
RGAIN         50
```

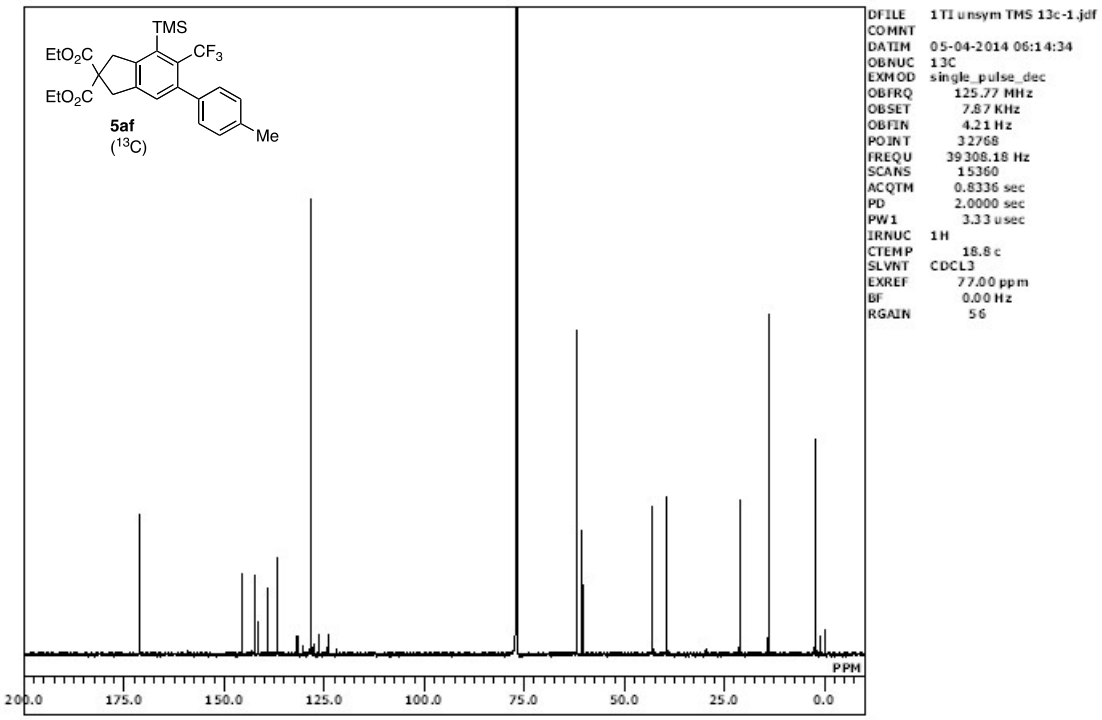


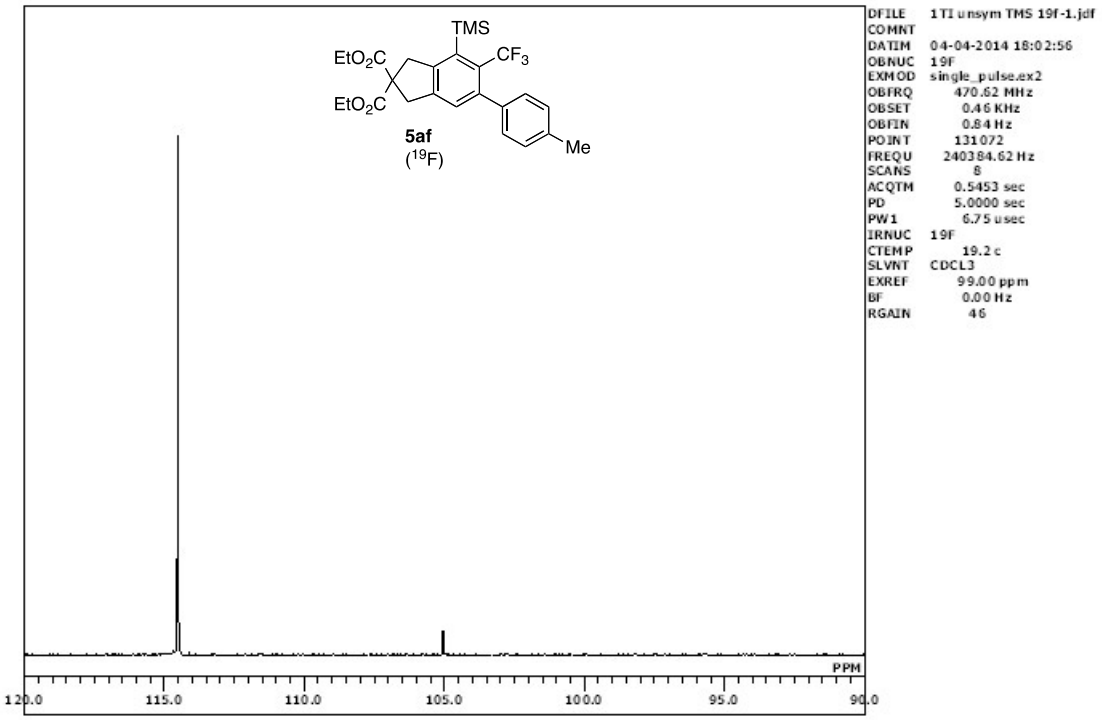

```
DFILE   data 1TI□@TsN.als
COMNT
DATIM   Mon Feb 18 12:57:00 2013
OBNUC   1H
EXMOD   NON
OBFRQ     270.05 MHz
OBSET     112.00 KHz
OBFIN    5800.00 Hz
POINT      16384
FREQU    5401.76 Hz
SCANS          8
ACQTM       3.0331 sec
PD          3.9670 sec
PW1         5.90 usec
IRNUC   1H
CTEMP      23.6 c
SLVNT   CDCL3
EXREF       0.00 ppm
BF          0.02 Hz
RGAIN         18
```

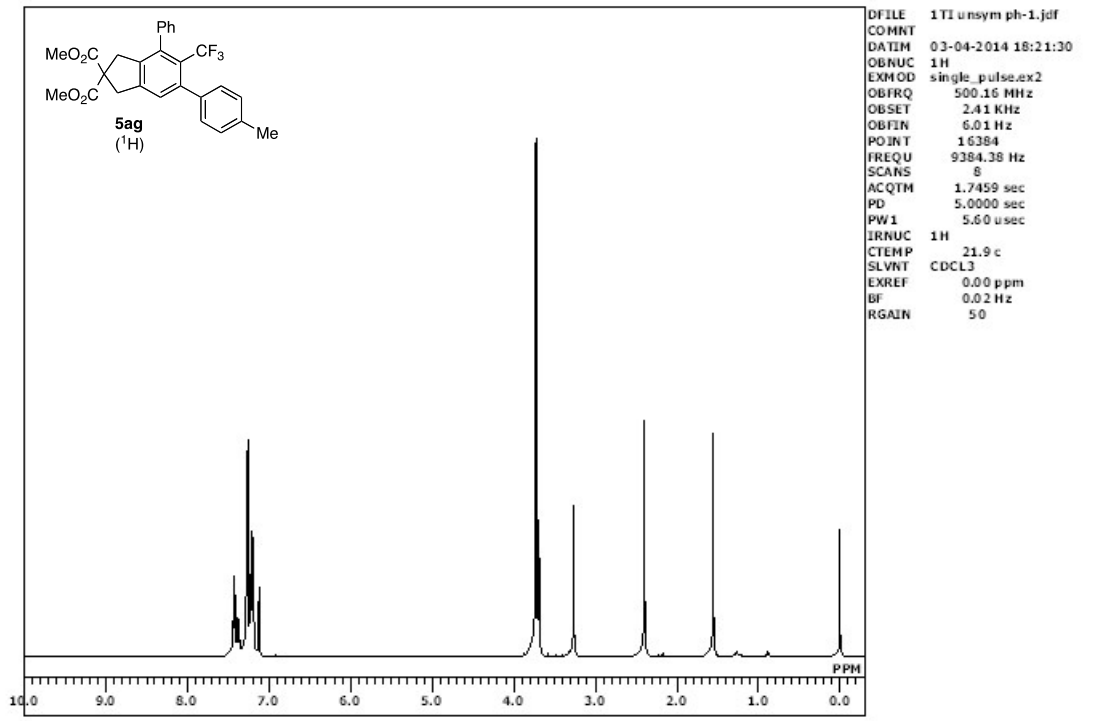

TsN
CF3
OMe
5he
(13C)
DFILE data 1TI-140 13C ts-1.jdf
COMNT
DATIM 18-02-2013 22:20:04
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 480
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.34 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 50
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0

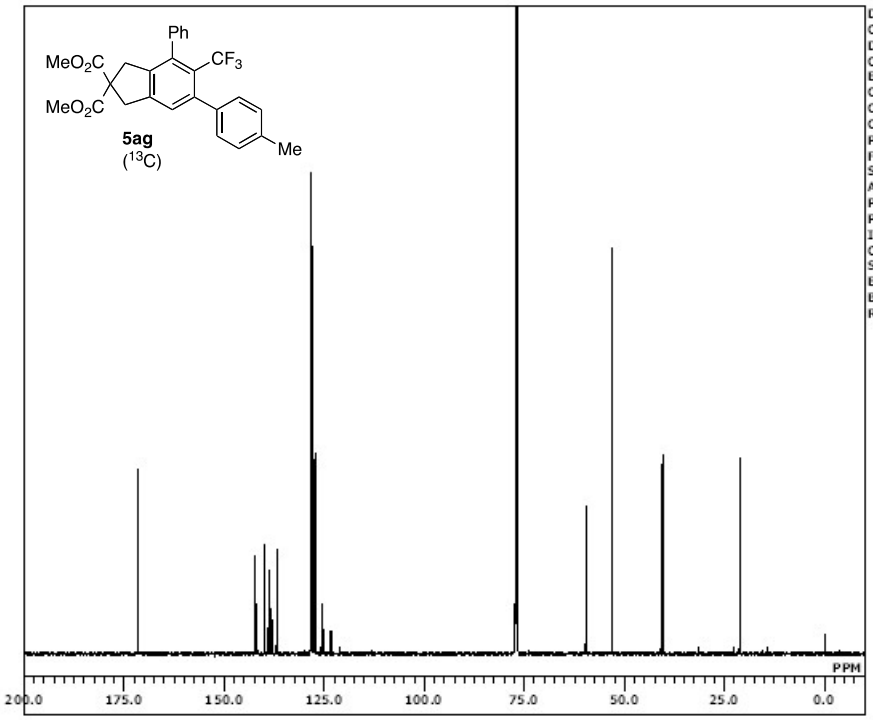

TsN
CF3
OMe
5he
(19F)
DFILE data 1TI TsN 19F-1.jdf
COMNT
DATIM 19-02-2013 10:16:55
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 23.6 c
SLVNT CDCL3
EXREF 99.00 ppm
BF 0.00 Hz
RGAIN 50
PPM
120.0
115.0
110.0
105.0
100.0
95.0
90.0


TMS
EtO2C
EtO2C
CF3
Me
5af
(1H)
DFILE 1TI unsym TMS-3.jdf
COMNT
DATIM 04-04-2014 17:59:31
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.02 Hz
RGAIN 42
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

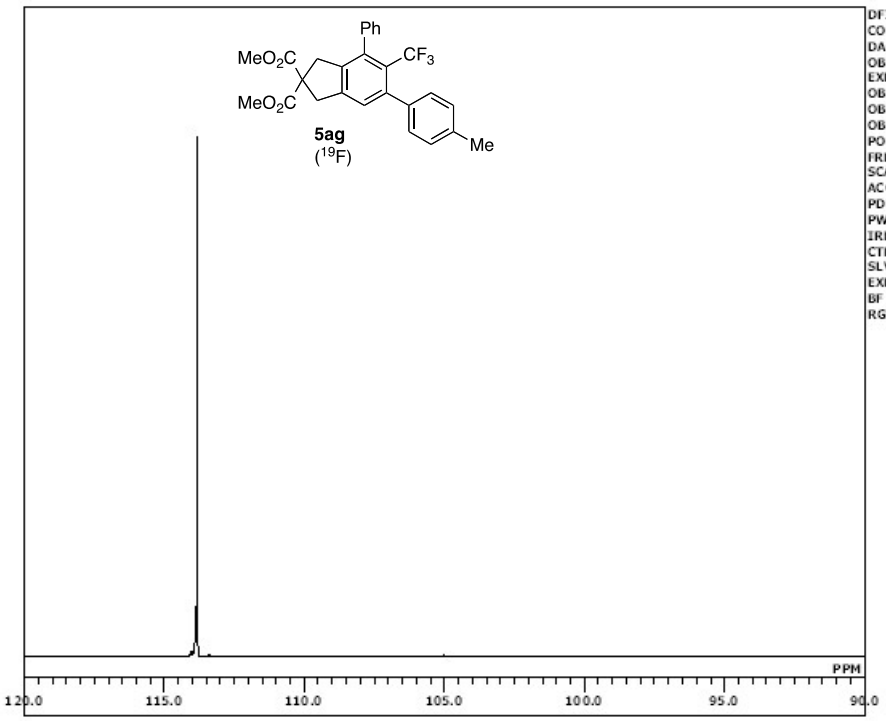

TMS
EtO2C
EtO2C
CF3
Me
5af
(13C)
DFILE 1TI unsym TMS 13c-1.jdf
COMNT
DATIM 05-04-2014 06:14:34
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 15360
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 18.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 56
PPM
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0


TMS
EtO2C
EtO2C
CF3
Me
5af
(19F)
DFILE 1TI unsym TMS 19f-1.jdf
COMNT
DATIM 04-04-2014 18:02:56
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 19.2 c
SLVNT CDCL3
EXREF 99.00 ppm
BF 0.00 Hz
RGAIN 46
PPM
120.0
115.0
110.0
105.0
100.0
95.0
90.0


Ph
MeO2C
MeO2C
CF3
Me
5ag
(1H)
DFILE 1TI unsym ph-1.jdf
COMNT
DATIM 03-04-2014 18:21:30
OBNUC 1H
EXMOD single_pulse.ex2
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 9384.38 Hz
SCANS 8
ACQTM 1.7459 sec
PD 5.0000 sec
PW1 5.60 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.02 Hz
RGAIN 50
PPM
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0



DFILE 1TI unsym Ph 13c-2.jdf
COMNT
DATIM 04-04-2014 07:48:29
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 39308.18 Hz
SCANS 12288
ACQTM 0.8336 sec
PD 2.0000 sec
PW1 3.33 usec
IRNUC 1H
CTEMP 19.0 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.00 Hz
RGAIN 54



DFILE 1TI unsym Ph -1 @,P,X,1.jdf
COMNT
DATIM 04-04-2014 08:56:60
OBNUC 19F
EXMOD single_pulse.ex2
OBFRQ 470.62 MHz
OBSET 0.46 KHz
OBFIN 0.84 Hz
POINT 131072
FREQU 240384.62 Hz
SCANS 8
ACQTM 0.5453 sec
PD 5.0000 sec
PW1 6.75 usec
IRNUC 19F
CTEMP 18.8 c
SLVNT CDCL3
EXREF 99.00 ppm
BF 0.00 Hz
RGAIN 44