Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information to

Visible light photocatalytic acyldifluoroalkylation of unactivated alkenes for the direct synthesis of *gem*-difluorinated ketones

Yulu Zhou, Zhimin Xiong, Jiayan Qiu, Lichun Kong* and Gangguo Zhu*

Key Laboratory of the Ministry of Education for Advanced Catalysis Materials, Department of Chemistry, Zhejiang Normal University, 688 Yingbin Road, Jinhua 321004, China.

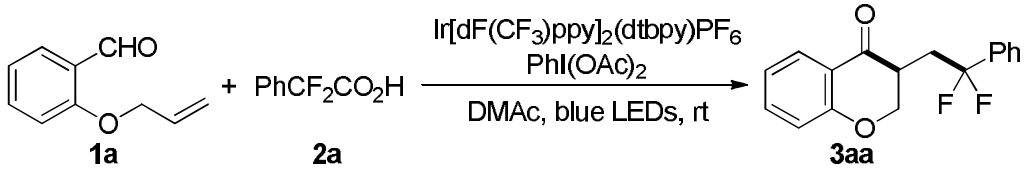
gangguo@zjnu.cn

Table of contents

General information

Unless otherwise noted, the starting materials were used directly from commercial suppliers without further purification. ^{1}H, ^{13}C, and ^{19}F NMR spectra were measured on a 600 or 400 MHz NMR spectrometer using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as the internal standard. Chemical shifts (δ) are given in parts per million relative to TMS, and the coupling constants are given in hertz. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Unless otherwise noted, all melting points are uncorrected. High-resolution mass spectrometry (HRMS) analyses were carried out using a TOF MS instrument with an ESI source. GC-MS measurements were performed on a 7890B GC system with an Agilent 5975 MSD detector. Column chromatography was performed using silica gel (200−300 mesh). Substrates **1** were prepared according to the literature.[1] α,α-Difluoroarylacetic acids **2a-2l** were prepared according to the literature.[2]

General procedure for the photocatalytic acyldifluoroalkylation of unactivated alkenes

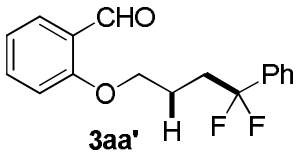


To a mixture of $Ir[dF(CF_3)ppy]_2(dtbpy)PF_6$ (2.8 mg, 0.0025 mmol), **2a** (129.0 mg, 0.75 mmol), and $PhI(OAc)_2$ (161.1 mg, 0.50 mmol) in 2.0 mL of DMAc was added **1a** (40.6 mg, 0.25 mmol) under a nitrogen atmosphere. After 36 h of irradiation with 15 W of blue LEDs at 25 ^{o}C, the reaction mixture was quenched with water, extracted with EtOAc, washed with brine, dried over anhydrous Na_2SO_4, and concentrated. Column chromatography on silica gel (petroleum ethers/EtOAc = 100:1) gave 53 mg (74% yield) of **3aa** as a colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (dd, J = 7.9, 1.7 Hz, 1H), 7.56–7.52 (m, 2H), 7.50–7.46 (m, 1H), 7.46–7.42 (m, 3H), 7.05–6.99 (m, 1H), 6.97 (d, J = 8.4 Hz, 1H), 4.75 (dd, J = 11.4, 5.0 Hz, 1H), 4.26 (t, J = 11.7 Hz, 1H), 3.17–3.05 (m, 2H), 2.15–2.02 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 161.6, 136.5 (t, J = 26.3 Hz), 136.0, 130.1 (t, J = 1.5 Hz), 128.6, 127.5, 124.8 (t, J = 6.0 Hz), 122.7 (t, J = 242.7

[1] (a) K. Hirano, A. T. Biju, I. Piel and F. Glorius, *J. Am. Chem. Soc*., 2009, **131**, 14190; (b) J. Zhao, P. Li, X. Li, C. Xia and F. Li, *Chem. Commun.*, 2016, **52**, 3661; (c) W.-C. Yang, P. Dai, K. Luo, Y.-G. Ji and L. Wu, *Adv. Synth. Catal.*, 2017, **359**, 2390; (d) D. Lu, Y. Wan, L. Kong and G. Zhu, *Org. Lett*., 2017, **19**, 2929.

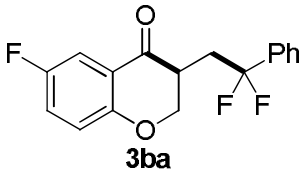
[2] S. Mizuta; I. S. R. Stenhagen; M. O'Duill; J. Wolstenhulme and V. Gouverneur, *Org. Lett.,* 2013, **15**, 2648.

Hz), 121.5, 120.3, 117.8, 70.5 (dd, J = 3.4, 1.8 Hz), 41.2 (t, J = 1.3 Hz), 34.3 (t, J = 28.0 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.1 (d, J = 245.7 Hz), -97.5 (d, J = 245.8 Hz); HRMS (ESI) calcd for $C_{17}H_{14}F_2NaO_2$ $(M+Na)^+$ 311.0854 found 311.0856.



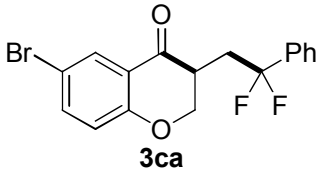
3aa'

*Compound **3aa'***: 5 mg, 7% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.47 (s, 1H), 7.83 (d, J = 7.3 Hz, 1H), 7.53–7.42 (m, 6H), 7.07–6.88 (m, 2H), 4.11 (s, 2H), 2.44–2.28 (m, 2H), 2.05 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.6, 161.0, 136.5 (t, J = 27.5 Hz), 135.9, 129.8, 128.5, 128.4, 125.4, 124.8 (t, J = 6.3 Hz), 122.3 (t, J = 241.3 Hz), 120.8, 112.3, 67.4, 35.8 (t, J = 28.5 Hz), 22.6; ^{19}F NMR (565 MHz, $CDCl_3$) δ -96.1; HRMS (ESI) calcd for $C_{17}H_{16}F_2NaO_2$ $(M+Na)^+$ 313.1011 found 313.1016.



3ba

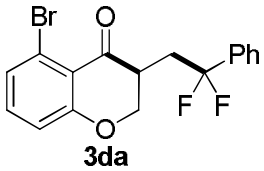
*Compound **3ba***: 65 mg, 85% yield, white solid, mp 95–96 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.55–7.50 (m, 3H), 7.47–7.43 (m, 3H), 7.20 (ddd, J = 8.9, 7.9, 3.2 Hz, 1H), 6.95 (dd, J = 9.1, 4.2 Hz, 1H), 4.75 (dd, J = 11.3, 5.1 Hz, 1H), 4.24 (t, J = 11.7 Hz, 1H), 3.16–3.02 (m, 2H), 2.15–2.01 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.4 (d, J = 1.5 Hz), 157.9 (d, J = 1.5 Hz), 157.2 (d, J = 242.2 Hz), 136.4 (t, J = 26.4 Hz), 130.1, 128.7, 124.8 (t, J = 6.1 Hz), 123.6 (d, J = 24.6 Hz), 122.6 (t, J = 243.1 Hz), 120.7 (d, J = 6.5 Hz), 119.5 (d, J = 34.7 Hz), 112.4 (d, J = 23.4 Hz), 70.6 (dd, J = 3.1, 1.5 Hz), 41.1, 34.3 (t, J = 28.3 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.3 (d, J = 245.6 Hz), -97.5 (d, J = 245.7 Hz), -121.2; HRMS (ESI) calcd for $C_{17}H_{13}F_3NaO_2$ $(M+Na)^+$ 329.0760 found 329.0767.



3ca

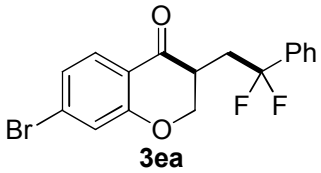
*Compound **3ca***: 68 mg, 74% yield, white solid, mp 102–103 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, J = 2.5 Hz, 1H), 7.57–7.50 (m, 3H), 7.45 (dd, J = 4.9, 1.7 Hz, 3H), 6.88 (d, J = 8.8 Hz,

1H), 4.77 (dd, J = 11.5, 5.3 Hz, 1H), 4.25 (t, J = 11.8 Hz, 1H), 3.15–3.01 (m, 2H), 2.13–2.01 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.9, 160.5, 138.7, 136.4 (t, J = 26.2 Hz), 130.2, 129.9, 128.7, 124.8 (t, J = 6.1 Hz), 122.5 (t, J = 242.6 Hz), 121.6, 119.9, 114.1, 70.5 (dd, J = 3.5, 1.7 Hz), 41.0 (t, J = 1.6 Hz), 34.2 (t, J = 28.2 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.3 (d, J = 245.8 Hz), -97.6 (d, J = 245.7 Hz); HRMS (ESI) calcd for $C_{17}H_{13}BrF_2NaO_2$ $(M+Na)^+$ 388.9959 found 388.9951.



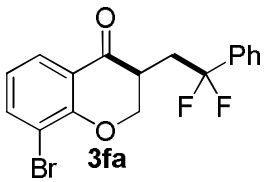
3da

*Compound **3da***: 62 mg, 68% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.54–7.51 (m, 2H), 7.47–7.43 (m, 3H), 7.30–7.26 (m, 1H), 7.23 (t, J = 8.0 Hz, 1H), 6.94 (dd, J = 8.2, 0.9 Hz, 1H), 4.75 (dd, J = 11.5, 5.2 Hz, 1H), 4.26 (t, J = 11.7 Hz, 1H), 3.18–3.04 (m, 2H), 2.15–2.02 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.2, 162.9, 136.4 (t, J = 26.2 Hz), 135.1, 130.1 (t, J = 1.5 Hz), 128.7, 128.4, 124.8 (t, J = 6.1 Hz), 122.6 (t, J = 243.2 Hz), 121.9, 118.6, 117.6, 70.0 (dd, J = 3.5, 1.8 Hz), 41.5, 34.3 (t, J = 28.4 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.3 (d, J = 245.9 Hz), -97.3 (d, J = 245.9 Hz); HRMS (ESI) calcd for $C_{17}H_{13}NaF_2BrO_2$ $(M+Na)^+$ 388.9959 found 388.9962.



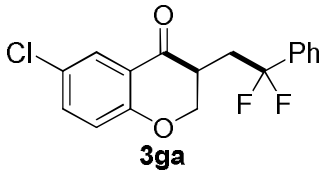
3ea

*Compound **3ea***: 72 mg, 78% yield, white solid, mp 114–116 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (d, J = 8.4 Hz, 1H), 7.53 (dd, J = 6.7, 2.5 Hz, 2H), 7.47–7.43 (m, 3H), 7.19–7.13 (m, 2H), 4.77 (dd, J = 11.4, 5.1 Hz, 1H), 4.26 (t, J = 11.7 Hz, 1H), 3.15–3.02 (m, 2H), 2.13–2.01 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.2, 161.8, 136.4 (t, J = 26.3 Hz), 130.5, 130.5, 128.7, 128.6, 125.2, 124.8 (t, J = 6.0 Hz), 122.6 (t, J = 242.9 Hz), 121.0, 119.2, 70.8 (dd, J = 3.5, 1.7 Hz), 41.1, 34.2 (t, J = 28.2 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.3 (d, J = 245.7 Hz), -97.5 (d, J = 245.9 Hz); HRMS (ESI) calcd for $C_{17}H_{13}BrF_2NaO_2$ $(M+Na)^+$ 388.9959 found 388.9956.



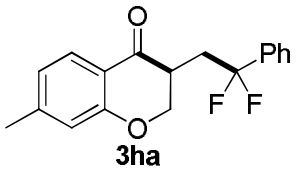
3fa

*Compound **3fa***: 67 mg, 73% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.84 (dd, J = 7.9, 1.5 Hz, 1H), 7.72 (dd, J = 7.7, 1.5 Hz, 1H), 7.53 (dd, J = 6.5, 2.6 Hz, 2H), 7.49–7.41 (m, 3H), 6.91 (t, J = 7.8 Hz, 1H), 4.92 (dd, J = 11.5, 5.3 Hz, 1H), 4.33 (t, J = 11.9 Hz, 1H), 3.21–3.04 (m, 2H), 2.16–2.02 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.2 157.9, 139.2, 136.3 (t, J = 26.2 Hz), 130.2, 128.7, 128.4, 126.9, 124.8 (t, J = 5.9 Hz), 122.5 (t, J = 242.8 Hz), 121.5, 111.4, 71.0 (dd , J = 3.4, 1.9 Hz), 40.8 (t, J = 1.7 Hz), 34.2 (t, J = 28.8 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.3 (d, J = 245.6 Hz), -97.5 (d, J = 245.6 Hz); HRMS (ESI) calcd for $C_{17}H_{13}BrF_2NaO_2$ $(M+Na)^+$ 388.9959 found 388.9952.



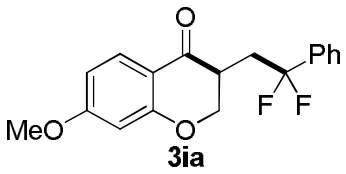
3ga

*Compound **3ga***: 56 mg, 70% yield, white solid, mp 95–97 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, J = 2.6 Hz, 1H), 7.53 (dd, J = 6.7, 2.5 Hz, 2H), 7.47–7.43 (m, 3H), 7.41 (dd, J = 8.9, 2.7 Hz, 1H), 6.93 (d, J = 8.9 Hz, 1H), 4.77 (dd, J = 11.4, 5.2 Hz, 1H), 4.25 (t, J = 11.8 Hz, 1H), 3.16–3.02 (m, 2H), 2.14-2.02 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.0, 160.0, 136.4 (t, J = 26.2 Hz), 135.7, 130.2 (t, J = 1.4 Hz), 128.7, 127.0, 126.8, 124.8 (t, J = 6.1 Hz), 122.5 (t, J = 243.4 Hz), 121.1, 119.5, 70.6 (dd, J = 3.5, 1.7 Hz), 41.0 (t, J = 1.6 Hz), 34.2 (t, J = 27.8 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.3 (d, J = 245.9 Hz), -97.5 (d, J = 245.8 Hz); HRMS (ESI) calcd for $C_{17}H_{13}ClF_2NaO_2$ $(M+Na)^+$ 345.0464 found 345.0460.



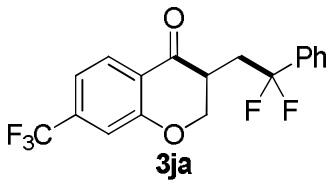
3ha

*Compound **3ha***: 56 mg, 74% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, J = 8.0 Hz, 1H), 7.56–7.50 (m, 2H), 7.47–7.41 (m, 3H), 6.85–6.80 (m, 1H), 6.77 (s, 1H), 4.72 (dd, J = 11.2, 5.1 Hz, 1H), 4.23 (t, J = 11.6 Hz, 1H), 3.15–3.04 (m, 2H), 2.35 (s, 3H), 2.12–2.01 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.8, 161.6, 147.6, 136.6 (t, J = 26.4 Hz), 130.1, 128.6, 127.4, 124.8 (t, J = 5.9 Hz), 122.7 (t, J = 242.9 Hz), 122.9, 118.1, 117.7, 70.5 (dd, J = 3.3, 1.7 Hz), 41.1 (t, J = 1.1 Hz), 34.3 (t, J = 27.9 Hz), 21.9; ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.0 (d, J = 249.7 Hz), -97.6 (d, J = 247.6 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $(M+Na)^+$ 325.1011 found 325.1005.



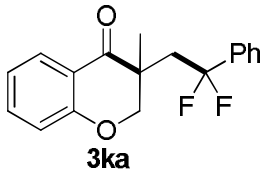
3ia

*Compound **3ia***: 33 mg, 41% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, J = 8.8 Hz, 1H), 7.56–7.51 (m, 2H), 7.46–7.42 (m, 3H), 6.57 (dd, J = 8.8, 2.4 Hz, 1H), 6.40 (d, J = 2.4 Hz, 1H), 4.73 (dd, J = 11.4, 5.1 Hz, 1H), 4.25 (t, J = 11.5 Hz, 1H), 3.83 (s, 3H), 3.16–3.01 (m, 2H), 2.12–2.00 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.7, 166.0, 163.6, 136.6 (t, J = 26.3 Hz), 130.1, 129.2, 128.6, 124.9 (t, J = 5.8 Hz), 122.8 (t, J = 242.3 Hz), 114.2, 110.1, 100.5, 70.8 (dd, J = 3.5, 1.8 Hz), 55.6, 40.8, 40.8, 34.4 (t, J = 28.1 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.0 (d, J = 245.7 Hz), -97.6 (d, J = 245.6 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_3$ $(M+Na)^+$ 341.0960 found 341.0968.



3ja

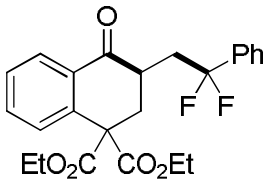
*Compound **3ja***: 61 mg, 68% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (d, J = 8.0 Hz, 1H), 7.75 (s, 1H), 7.63 (d, J = 7.9 Hz, 1H), 7.56–7.51 (m, 2H), 7.49–7.43 (m, 3H), 3.51 (dd, J = 17.6, 8.1 Hz, 1H), 3.10–2.91 (m, 3H), 2.25–2.13 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 205.5, 153.6, 138.5, 136.7 (t, J = 26.1 Hz), 136.3 (q, J = 32.4 Hz), 130.1, 128.6, 124.8 (t, J = 6.2 Hz), 124.7 (q, J = 3.5 Hz), 124.5, 123.6 (q, J = 273.4 Hz), 123.7 (q, J = 3.8 Hz), 122.8, (t, J = 243.0 Hz), 43.1 (t, J = 2.3 Hz), 40.0 (t, J = 27.8 Hz), 33.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -62.9, -92.1 (d, J = 245.4 Hz), -97.4 (d, J = 245.3 Hz); HRMS (ESI) calcd for $C_{18}H_{13}F_5NaO_2$ $(M+Na)^+$ 379.0728 found 379.0730.



3ka

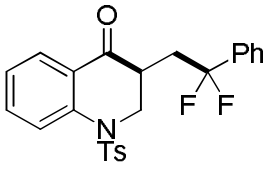
*Compound **3ja***: 65 mg, 86% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (dd, J = 7.9, 1.7 Hz, 1H), 7.51–7.47 (m, 3H), 7.41 (dd, J = 5.1, 2.0 Hz, 3H), 7.06–7.02 (m, 1H), 7.01–6.97 (m, 1H), 4.61 (d, J = 11.6 Hz, 1H), 4.38 (d, J = 11.7 Hz, 1H), 2.82–2.70 (m, 1H), 2.48–2.36 (m, 1H), 1.34 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 195.1, 161.0, 137.7 (t, J = 26.2 Hz), 135.8, 129.8 (t, J

= 1.3 Hz), 128.4, 128.1, 124.7 (t, J = 6.3 Hz), 122.7 (t, J = 244.8 Hz), 121.6, 119.3, 117.7, 74.5 (t, J = 3.4 Hz), 44.2, 41.7, 19.8 (d, J = 2.2 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.9 (d, J = 243.4 Hz), -92.5 (t, J = 243.5 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $(M+Na)^+$ 325.1011 found 325.1011.



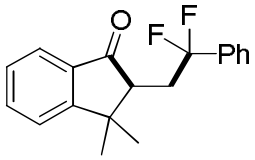
3ma

*Compound **3ma***: 97 mg, 90% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11–8.04 (m, 1H), 7.62–7.53 (m, 3H), 7.49 (d, J = 7.6 Hz, 1H), 7.47–7.39 (m, 4H), 4.35–4.28 (m, 2H), 4.28–4.17 (m, 2H), 3.39–3.24 (m, 2H), 3.01–2.92 (m, 1H), 2.56 (t, J = 13.8 Hz, 1H), 2.16–2.05 (m, 1H), 1.32 (t, J = 7.1 Hz, 3H), 1.26 (t, J = 7.1 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 195.7, 170.3, 169.2, 138.3, 137.0 (t, J = 26.2 Hz), 133.5, 131.5, 129.9 (d, J = 1.0 Hz), 129.4, 128.6, 128.5, 127.8, 124.9 (t, J = 6.1 Hz), 123.0 (t, J = 243.5 Hz), 62.4, 62.3, 58.9, 39.4, 37.8 (t, J = 27.6 Hz), 36.0, 14.0, 13.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -89.4 (d, J = 246.1 Hz), -98.0 (d, J = 246.0 Hz); HRMS (ESI) calcd for $C_{24}H_{24}F_2NaO_5$ $(M+Na)^+$ 453.1484 found 453.1481.



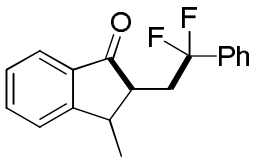
3na

*Compound **3na***: 62 mg, 56% yield, yellow oil ; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96–7.90 (m, 2H), 7.59–7.55 (m, 1H), 7.55–7.51 (m, 2H), 7.51–7.44 (m, 5H), 7.27–7.23 (m, 1H), 7.12 (d, J = 8.1 Hz, 2H), 4.90 (dd, J = 14.4, 5.2 Hz, 1H), 3.70 (t, J = 13.9 Hz, 1H), 3.10–2.97 (m, 1H), 2.59–2.50 (m, 1H), 2.34 (s, 3H), 1.99–1.85 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 193.0, 144.4, 142.2, 136.4, 136.3 (t, J = 26.0 Hz), 134.8, 130.1, 129.9, 128.7, 128.1, 126.8, 125.4, 124.8 (t, J = 6.3 Hz), 124.4, 124.1, 122.6 (t, J = 242.8 Hz), 50.8 (q, J = 2.9, 2.2 Hz), 40.4 (d, J = 1.7 Hz), 35.6 (t, J = 27.5 Hz), 21.5; ^{19}F NMR (565 MHz, $CDCl_3$) δ -88.2 (d, J = 247.4 Hz), -97.3 (d, J = 247.4 Hz); HRMS (ESI) calcd for $C_{24}H_{21}F_2NNaO_3S$ $(M+Na)^+$ 464.1102 found 464.1102.



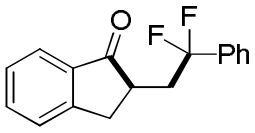
3oa

*Compound **3oa***: 58 mg, 78% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 (d, J = 7.6 Hz, 1H), 7.65–7.57 (m, 3H), 7.55 (d, J = 7.7 Hz, 1H), 7.45 (dd, J = 5.1, 1.9 Hz, 3H), 7.36 (t, J = 7.4 Hz, 1H), 3.10–2.96 (m, 1H), 2.72 (dd, J = 8.9, 2.3 Hz, 1H), 2.38–2.25 (m, 1H), 1.58 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 204.9, 162.8, 137.1 (t, J = 26.6 Hz), 135.0, 133.6, 129.9, 128.5, 127.5, 125.1 (t, J = 5.7 Hz), 123.5, 123.4, 122.8 (t, J = 242.5 Hz), 54.8 (d, J = 1.8 Hz), 42.0, 34.8 (t, J = 28.2 Hz), 27.9, 27.0; ^{19}F NMR (565 MHz, $CDCl_3$) δ -88.7 (d, J = 243.4 Hz), -98.2 (d, J = 243.4 Hz); HRMS (ESI) calcd for $C_{19}H_{19}F_2O$ $(M+H)^+$ 301.1398 found 301.1405.



3pa

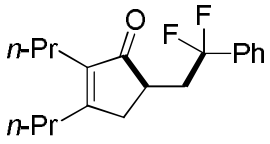
*Compound **3pa***: 49 mg, 68% yield, colorless oil, dr = 3:1; ^{1}H NMR (600 MHz, $CDCl_3$) of major isomer δ 7.74 (d, J = 7.6 Hz, 1H), 7.64 (t, J = 7.8 Hz, 1H), 7.57 (m, 2H), 7.52 (d, J = 7.7 Hz, 1H), 7.45 (m, 3H), 7.39 (t, J = 7.5 Hz, 1H), 3.42–3.27 (m, 1H), 3.07–2.90 (m, 1H), 2.52 (dt, J = 10.6, 3.1 Hz, 1H), 2.25–2.15 (m, 1H), 1.51 (d, J = 7.0 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) of major isomer δ 206.4, 158.7, 137.0 (t, J = 26.4 Hz), 135.2, 135.0, 129.9, 128.5, 127.5, 125.2, 124.9 (t, J = 6.0 Hz), 123.7, 122.8 (t, J = 242.5 Hz), 50.9, 40.5, 40.3 (t, J = 27.6 Hz), 20.1; ^{19}F NMR (565 MHz, $CDCl_3$) of major isomer δ -90.5 (d, J = 244.2 Hz), -98.0 (d, J = 244.2 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO$ $(M+Na)^+$ 309.1061 found 309.1067.



3qa

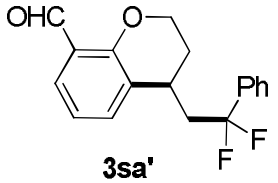
*Compound **3qa***: 38 mg, 56% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (d, J = 7.6 Hz, 1H), 7.63–7.58 (m, 1H), 7.57–7.52 (m, 2H), 7.49–7.42 (m, 4H), 7.37 (t, J = 7.4 Hz, 1H), 3.49-3.39 (m, 1H), 3.08–2.95 (m, 2H), 2.90–2.83 (m, 1H), 2.21–2.09 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 206.5, 153.6, 136.9 (t, J = 26.4 Hz), 135.9, 135.0, 129.9, 128.5, 127.4, 126.4,

124.8 (t, J = 6.2 Hz), 124.0, 122.9 (t, J = 242.9 Hz), 42.7, 40.2 (t, J = 27.6 Hz), 33.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -91.6 (d, J = 245.2 Hz), -97.5 (d, J = 245.3 Hz); HRMS (ESI) calcd for $C_{17}H_{14}F_2NaO$ $(M+Na)^+$ 295.0905 found 295.0912.



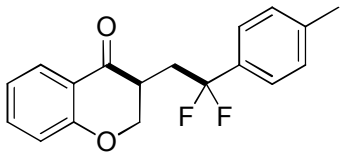
3ra

*Compound **3ra***: 36 mg, 47% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.53–7.47 (m, 2H), 7.47–7.38 (m, 3H), 2.91–2.71 (m, 2H), 2.49 (ddd, J = 9.4, 7.1, 3.5 Hz, 1H), 2.44–2.32 (m, 3H), 2.13 (t, J = 6.7 Hz, 2H), 2.03–1.86 (m, 1H), 1.60–1.51 (m, 2H), 1.43-1.30 (m, 2H), 0.96 (t, J = 7.4 Hz, 3H), 0.86 (t, J = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 209.5, 173.0, 139.2, 137.0 (t, J = 26.1 Hz), 129.8, 128.5, 124.9 (t, J = 6.1 Hz), 123.1 (t, J = 242.9 Hz), 40.5 (t, J = 27.3 Hz), 40.2, 36.8, 32.9, 25.1, 21.8, 20.8, 14.2, 14.1; ^{19}F NMR (565 MHz, $CDCl_3$) δ -91.1 (d, J = 244.7 Hz), -97.8 (d, J = 244.5 Hz); HRMS (ESI) calcd for $C_{19}H_{24}F_2NaO$ $(M+Na)^+$ 329.1687 found 329.1684.



3sa'

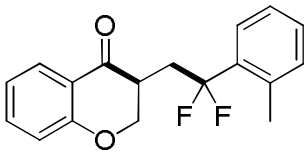
*Compound **3sa'***: 43 mg, 57% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 10.40 (s, 1H), 7.65 (dd, J = 7.7, 1.6 Hz, 1H), 7.53 (td, J = 4.1, 1.9 Hz, 2H), 7.50–7.44 (m, 3H), 7.26 (d, J = 6.2 Hz, 1H), 6.91 (t, J = 7.6 Hz, 1H), 4.35 (dt, J = 10.7, 4.3 Hz, 1H), 4.27 (td, J = 11.2, 10.7, 2.7 Hz, 1H), 3.23 (dq, J = 8.9, 4.5 Hz, 1H), 2.60–2.40 (m, 2H), 2.24–2.16 (m, 1H), 2.10–2.01 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.8, 157.2, 137.0 (t, J = 26.5 Hz), 135.5, 130.0 (t, J = 1.4 Hz), 128.7, 127.1, 126.5, 124.7 (t, J = 6.1 Hz), 124.5, 122.7 (t, J =243.9 Hz), 120.1, 63.4, 45.9 (t, J = 26.7 Hz), 28.5 (t, J = 2.7 Hz), 26.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -91.8 (d, J = 245.8 Hz), -97.5 (d, J = 254.9 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $(M+Na)^+$ 325.1011 found 325.1017.



3ab

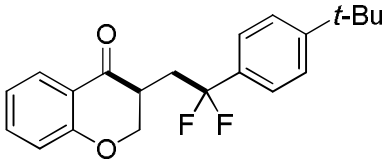
*Compound **3ab***: 60 mg, 80% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (dd, J = 7.9, 1.7 Hz, 1H), 7.47 (ddd, J = 8.6, 7.2, 1.7 Hz, 1H), 7.42 (d, J = 8.1 Hz, 2H), 7.24 (d, J = 8.0 Hz, 2H),

7.03–7.00 (m, 1H), 6.96 (d, J = 8.3 Hz, 1H), 4.74 (dd, J = 11.4, 5.1 Hz, 1H), 4.25 (t, J = 11.7 Hz, 1H), 3.16–3.04 (m, 2H), 2.38 (s, 3H), 2.12–2.01 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 161.6, 140.2, 136.0, 133.6 (t, J = 26.2 Hz), 129.3, 127.5, 124.8 (t, J = 5.6 Hz), 122.9 (t, J = 243.2 Hz), 121.4, 120.3, 117.8, 70.5, 41.3, 34.2 (t, J = 28.4 Hz), 21.2; ^{19}F NMR (565 MHz, $CDCl_3$) δ -89.0 (d, J = 243.2 Hz), -96.9 (d, J = 243.2 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $(M+Na)^+$ 325.1011 found 325.1010.



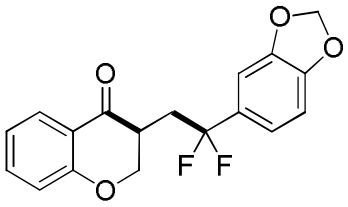
3ac

*Compound **3ac***: 51 mg, 68% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (d, J = 7.9 Hz, 1H), 7.52–7.44 (m, 2H), 7.32 (t, J = 7.4 Hz, 1H), 7.26–7.21 (m, 2H), 7.01 (t, J = 7.5 Hz, 1H), 6.97 (d, J = 8.4 Hz, 1H), 4.77 (dd, J = 11.3, 5.1 Hz, 1H), 4.27 (t, J = 11.7 Hz, 1H), 3.20–3.07(m, 2H), 2.50 (s, 3H), 2.15–2.02 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 161.6, 136.0, 135.4 (t, J = 2.0 Hz), 134.4 (t, J = 24.9 Hz), 132.2, 130.0, 127.5, 125.9, 125.5 (t, J = 8.7 Hz), 123.5 (t, J = 244.0 Hz), 121.5, 120.3, 117.8, 70.5, 41.2, 33.4 (t, J = 27.6 Hz), 20.2 (t, J = 3.1 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.0 (d, J = 248.5 Hz), -94.3 (d, J = 248.6 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO_2$ $(M+Na)^+$ 325.1011 found 325.1013.



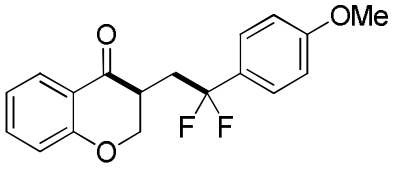
3ad

*Compound **3ad***: 69 mg, 80% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (dd, J = 7.9, 1.7 Hz, 1H), 7.49–7.45 (m, 5H), 7.03–7.00 (m, 1H), 6.97 (d, J = 8.3 Hz, 1H), 4.77 (dd, J = 11.6, 5.1 Hz, 1H), 4.27 (t, J = 11.7 Hz, 1H), 3.17–3.05 (m, 2H), 2.14–2.02 (m, 1H), 1.33 (s, 9H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 161.6, 153.3 (t, J = 1.5 Hz) 136.0, 133.5 (t, J = 26.2 Hz), 131.7, 131.1, 127.5, 125.6, 124.6 (t, J = 6.0 Hz), 122.8 (t, J = 242.6 Hz), 121.5, 120.3, 117.8, 70.5 (dd, J = 3.1, 2.1 Hz), 41.3 (t, J = 1.5 Hz), 34.7, 34.2 (t, J = 28.0 Hz), 31.2; ^{19}F NMR (565 MHz, $CDCl_3$) δ -88.7 (d, J = 246.0 Hz), -97.0 (d, J = 246.3 Hz); HRMS (ESI) calcd for $C_{21}H_{22}F_2NaO_2$ $(M+Na)^+$ 367.1480 found 367.1480.



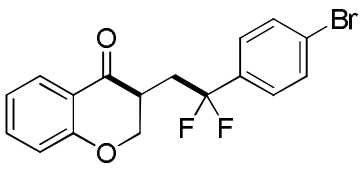
3ae

*Compound **3ae***: 56 mg, 67% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (dd, J = 7.9, 1.5 Hz, 1H), 7.51–7.43 (m, 1H), 7.05–6.94 (m, 4H), 6.83 (d, J = 8.1 Hz, 1H), 6.00 (s, 2H), 4.73 (dd, J = 11.4, 5.1 Hz, 1H), 4.24 (t, J = 11.7 Hz, 1H), 3.15–2.97 (m, 2H), 2.12–1.96 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 161.6, 148.9, 147.9, 136.1, 130.3 (t, J = 26.7 Hz), 127.5, 122.5 (t, J = 243.1 Hz), 121.5, 120.3, 118.9 (t, J = 6.7 Hz), 117.8, 108.2, 105.6 (t, J = 6.1 Hz), 101.5, 70.4, 41.2, 34.2 (t, J = 28.8 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -87.5 (d, J = 244.4 Hz), -95.5 (d, J = 244.4 Hz); HRMS (ESI) calcd for $C_{18}H_{14}F_2NaO_4$ $(M+Na)^+$ 355.0752 found 355.0758.



3af

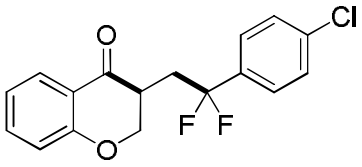
*Compound **3af***: 58 mg, 73% yield, white solid, mp 68–69 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (dd, J = 7.9, 1.6 Hz, 1H), 7.46 (d, J = 8.7 Hz, 3H), 7.01 (t, J = 7.2 Hz, 1H), 6.97–6.91 (m, 3H), 4.73 (dd, J = 11.3, 5.1 Hz, 1H), 4.24 (t, J = 11.7 Hz, 1H), 3.83 (s, 3H), 3.18–3.01 (m, 2H), 2.13–2.00 (m, 1H).; ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 161.6, 160.8, 136.0, 128.7 (t, J = 26.6 Hz), 127.5, 126.4 (t, J = 5.7 Hz), 122.8 (t, J = 242.2 Hz), 121.4, 121.2, 117.8, 113.9, 70.5 (dd, J = 3.0, 2.1 Hz), 55.3, 41.3, 34.2 (t, J = 30.1 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -87.7 (d, J = 245.6 Hz), -95.8 (d, J = 245.6 Hz); HRMS (ESI) calcd for $C_{18}H_{16}F_2NaO3$ $(M+Na)^+$ 341.0960 found 341.0958.



3ag

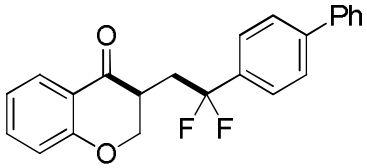
*Compound **3ag***: 78 mg, 85% yield, white solid, mp 81–82 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (dd, J = 7.9, 1.7 Hz, 1H), 7.58 (d, J = 8.5 Hz, 2H), 7.50–7.45 (m, 1H), 7.41 (d, J = 8.5 Hz, 2H), 7.06–6.99 (m, 1H), 6.97 (d, J = 8.4 Hz, 1H), 4.75 (dd, J = 11.4, 5.1 Hz, 1H), 4.26 (t, J = 11.7 Hz, 1H), 3.14–3.01 (m, 2H), 2.12–1.99 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.8, 161.6, 136.1,

135.7 (t, J = 26.8 Hz), 131.9, 127.5, 126.6 (t, J = 6.0 Hz), 124.6 (t, J = 1.8 Hz), 122.3 (t, J = 243.1 Hz), 121.5, 120.2, 117.8, 70.4 (dd, J = 3.2, 1.4 Hz), 41.1, 34.1 (t, J = 27.8 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -90.1 (d, J = 246.8 Hz), -97.7 (d, J = 246.9 Hz); HRMS (ESI) calcd for $C_{17}H_{13}BrF_2NaO_2$ $(M+Na)^+$ 388.9959 found 388.9969.



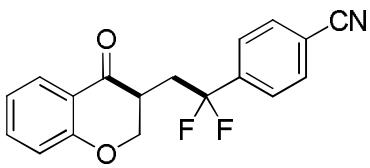
3ah

Compound ***3ah***: 58 mg, 72% yield, yellow solid, mp 64–65 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (dd, J = 7.9, 1.7 Hz, 1H), 7.52–7.45 (m, 3H), 7.42 (d, J = 8.5 Hz, 2H), 7.05–7.00 (m, 1H), 6.97 (d, J = 8.4 Hz, 1H), 4.75 (dd, J = 11.4, 5.1 Hz, 1H), 4.26 (t, J = 11.7 Hz, 1H), 3.15–3.01 (m, 2H), 2.12–2.00 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.9, 161.6, 136.3, 136.1, 135.0 (t, J = 26.9 Hz), 128.9, 127.5, 126.4 (t, J = 6.0 Hz), 122.3 (t, J = 243.6 Hz), 121.5, 120.2, 117.8, 70.4 (dd, J = 3.5, 1.6 Hz), 41.1, 34.2 (t, J = 27.7 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -89.9 (d, J = 247.0 Hz), -97.4 (d, J = 247.1 Hz); HRMS (ESI) calcd for $C_{17}H_{13}ClF_2NaO_2$ $(M+Na)^+$ 345.0464 found 345.0469.



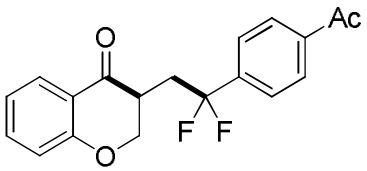
3ai

Compound ***3ai***: 78 mg, 86% yield, white solid, mp 150–151 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, J = 7.3 Hz, 1H), 7.69–7.58 (m, 6H), 7.51–7.38 (m, 4H), 7.09–6.94 (m, 2H), 4.80 (dd, J = 11.4, 5.0 Hz, 1H), 4.29 (t, J = 11.7 Hz, 1H), 3.28–3.07 (m, 2H), 2.22–2.04 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.0, 161.6, 143.0, 140.0, 136.1, 135.3 (t, J = 26.7 Hz), 128.9, 127.8, 127.5, 127.4, 127.2, 125.4 (t, J = 5.8 Hz), 122.7 (t, J = 242.8 Hz), 121.5, 120.3, 117.8, 70.5, 41.2, 34.2 (t, J = 27.9 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -89.4 (d, J = 246.1 Hz), -97.2 (d, J = 246.1 Hz); HRMS (ESI) calcd for $C_{23}H_{18}F_2NaO_2$ $(M+Na)^+$ 387.1167 found 387.1167.



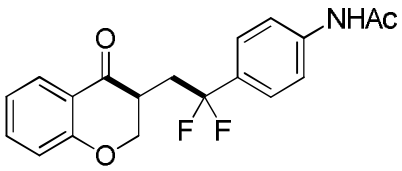
3aj

Compound ***3aj***: 49 mg, 62% yield, yellow solid, mp 100–102 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (dd, J = 7.9, 1.6 Hz, 1H), 7.75 (d, J = 8.3 Hz, 2H), 7.66 (d, J = 8.3 Hz, 2H), 7.52–7.45 (m, 1H), 7.07–6.93 (m, 2H), 4.76 (dd, J = 11.4, 5.2 Hz, 1H), 4.28 (t, J = 11.7 Hz, 1H), 3.22–2.93 (m, 2H), 2.16–1.98 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.6, 161.5, 140.9 (t, J = 26.9 Hz), 136.2, 132.5, 132.2, 127.5, 125.8 (t, J = 6.0 Hz), 121.7 (t, J = 244.4 Hz), 121.6, 120.1, 117.8, 114.3, 70.3 (d, J = 2.8 Hz), 40.9, 34.0 (t, J = 27.0 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -91.9 (d, J = 248.0 Hz), -99.0 (d, J = 248.0 Hz); HRMS (ESI) calcd for $C_{18}H_{13}F_2NNaO_2$ $(M+Na)^+$ 336.0807 found 336.0807.



3ak

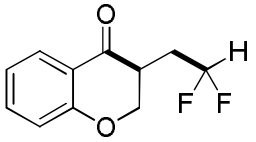
Compound ***3ak***: 58 mg, 70% yield, white solid, mp 123–125 °C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 (d, J = 8.2 Hz, 2H), 7.86 (dd, J = 7.9, 1.5 Hz, 1H), 7.64 (d, J = 8.3 Hz, 2H), 7.53–7.44 (m, 1H), 7.06–6.93 (m, 2H), 4.76 (dd, J = 11.3, 5.2 Hz, 1H), 4.27 (t, J = 11.6 Hz, 1H), 3.18–3.00 (m, 2H), 2.63 (s, 3H), 2.17–1.99 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 197.2, 191.8, 161.5, 140.8 (t, J = 26.8 Hz), 138.3, 136.2, 128.6, 127.5, 125.3 (t, J = 5.8 Hz), 122.2 (t, J = 243.6 Hz), 121.5, 120.2, 117.8, 70.4, 41.0, 34.1 (t, J = 27.6 Hz), 26.7; ^{19}F NMR (565 MHz, $CDCl_3$) δ -91.3 (d, J = 247.0 Hz), -98.4 (d, J = 246.9 Hz); HRMS (ESI) calcd for $C_{19}H_{16}KF_2O_3$ $(M+K)^+$ 369.0699 found 369.0706.



3al

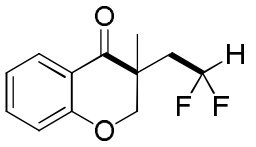
Compound ***3ak***: 62 mg, 72% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (dd, J = 7.9, 1.5 Hz, 1H), 7.59 (d, J = 8.6 Hz, 2H), 7.48–7.44 (m, 3H), 7.01 (t, J = 7.5 Hz, 1H), 6.96 (d, J = 8.4 Hz, 1H), 4.73 (dd, J = 11.4, 5.1 Hz, 1H), 4.25 (t, J = 11.7 Hz, 1H), 3.13–3.00 (m, 2H), 2.18 (s, 3H), 2.11–1.99 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 168.6, 161.6, 139.5, 136.1, 132.0 (t, J = 26.1 Hz), 127.4, 125.7 (t, J = 5.7 Hz), 122.5 (t, J = 242.9 Hz), 121.5, 120.2, 119.5, 117.8, 70.4 (d, J = 1.7 Hz), 41.2, 34.2 (t, J = 28.6 Hz), 24.6; ^{19}F NMR (565 MHz, $CDCl_3$) δ -89.4 (d, J =

245.7 Hz), -96.6 (d, J = 245.8 Hz); HRMS (ESI) calcd for $C_{19}H_{17}F_2NNaO_3$ $(M+Na)^+$ 368.1069 found 368.1070.



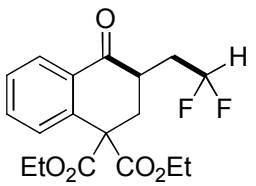
3am

Compound ***3am***: 43 mg, 81% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (dd, J = 7.9, 1.6 Hz, 1H), 7.49 (ddd, J = 8.6, 7.3, 1.7 Hz, 1H), 7.08–6.94 (m, 2H), 6.32–5.59 (m, 1H), 4.62 (dd, J = 11.4, 5.2 Hz, 1H), 4.23 (t, J = 11.7 Hz, 1H), 3.14–3.02 (m, 1H), 2.62–2.40 (m, 1H), 1.98–1.78 (m, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.5, 161.5, 136.2, 127.4, 121.6, 120.2, 117.8, 116.0 (t, J = 239.6 Hz), 70.4, 40.4 (dd, J = 4.9, 3.5 Hz), 30.3 (t, J = 22.4 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -112.5 (d, J = 283.1 Hz), -117.5 (d, J = 283.1 Hz); HRMS (ESI) calcd for $C_{11}H_{10}F_2NaO_2$ $(M+Na)^+$ 235.0541 found 235.0549.



3jm

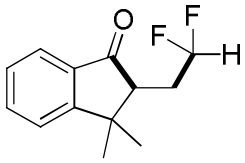
Compound ***3am***: 45 mg, 80% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (dd, J = 7.9, 1.5 Hz, 1H), 7.50 (ddd, J = 8.5, 7.4, 1.7 Hz, 1H), 7.09–7.02 (m, 1H), 6.98 (d, J = 8.4 Hz, 1H), 6.18–5.91 (m, 1H), 4.33 (d, J = 11.7 Hz, 1H), 4.21 (d, J = 11.7 Hz, 1H), 2.25–2.15 (m, 2H), 1.30 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 195.2, 160.9, 136.1, 127.9, 121.8, 119.2, 117.7, 115.7 (t, J = 239.1 Hz), 74.8 (t, J = 1.8 Hz), 42.5 (t, J = 4.2 Hz), 37.5 (t, J = 21.9 Hz), 18.7; ^{19}F NMR (565 MHz, $CDCl_3$) δ -110.9 (d, J = 288.6 Hz), -111.5 (d, J = 288.5 Hz); HRMS (ESI) calcd for $C_{12}H_{13}F_2O_2$ $(M+H)^+$ 227.0878 found 227.0877.



3mm

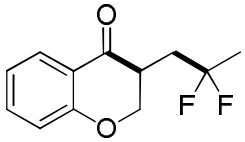
Compound ***3mm***: 74 mg, 84% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11–7.98 (m, 1H), 7.66–7.54 (m, 1H), 7.53–7.38 (m, 2H), 6.34–5.98 (m, 1H), 4.35–4.25 (m, 3H), 4.19 (dq, J =

10.8, 7.1 Hz, 1H), 3.03–2.90 (m, 2H), 2.71–2.58 (m, 1H), 2.56-2.46 (m, 1H), 1.99–1.80 (m, 1H), 1.34–1.20 (m, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 196.3, 170.2, 169.0, 138.1, 133.6, 131.5, 129.7, 128.7, 127.6, 116.4 (t, J = 239.1 Hz), 62.5, 62.4, 58.9, 38.7 (t, J = 5.1 Hz), 35.9, 34.3 (t, J = 22.0 Hz), 13.9, 13.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -112.8 (d, J = 283.9 Hz), -117.5 (d, J = 283.9 Hz); HRMS (ESI) calcd for $C_{18}H_{21}F_2O_5$ $(M+H)^+$ 355.1352 found 355.1347.



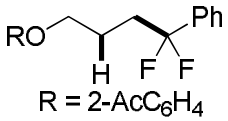
3om

*Compound **3om***: 43 mg, 76% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (d, J = 7.6 Hz, 1H), 7.67–7.61 (m, 1H), 7.52 (d, J = 7.8 Hz, 1H), 7.42–7.35 (m, 1H), 6.60–6.19 (m, 1H), 2.63 (ddd, J = 7.6, 5.8, 1.3 Hz, 1H), 2.45–2.25 (m, 1H), 2.10–1.93 (m, 1H), 1.53 (s, 3H), 1.15 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 205.6, 162.2, 135.2, 133.8, 127.7, 123.6, 123.3, 116.4 (d, J = 239.0 Hz), 53.9 (dd, J = 6.8, 2.6 Hz), 41.7, 30.7 (t, J = 22.2 Hz), 27.8, 26.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -112.7 (d, J = 280.5 Hz), -118.5 (d, J = 280.5 Hz); HRMS (ESI) calcd for $C_{14}H_{17}F_2O$ $(M+H)^+$ 239.1242 found 239.1232.



3an

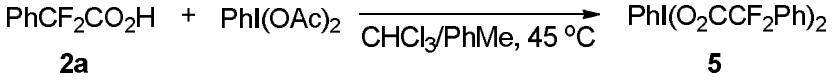
*Compound **3an***: 47 mg, 83% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92–7.86 (m, 1H), 7.52–7.45 (m, 1H), 7.06–6.93 (m, 2H), 4.74 (dd, J = 11.4, 5.2 Hz, 1H), 4.22 (t, J = 11.7 Hz, 1H), 3.19–3.15 (m, 1H), 2.82–2.68 (m, 1H), 1.89–1.77 (m, 1H), 1.74–1.63 (m, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.4, 161.6, 136.0, 127.4, 123.7 (t, J = 238.1 Hz), 121.4, 120.3, 117.8, 70.5 (dd, J = 3.9, 1.0 Hz), 41.0 (t, J = 2.2 Hz), 33.1 (t, J = 25.5 Hz), 24.1 (t, J = 27.6 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -87.7 (d, J = 239.2 Hz), -92.6 (d, J = 240.9 Hz); HRMS (ESI) calcd for $C_{12}H_{12}F_2NaO_2$ $(M+Na)^+$ 249.0698 found 249.0705.



6

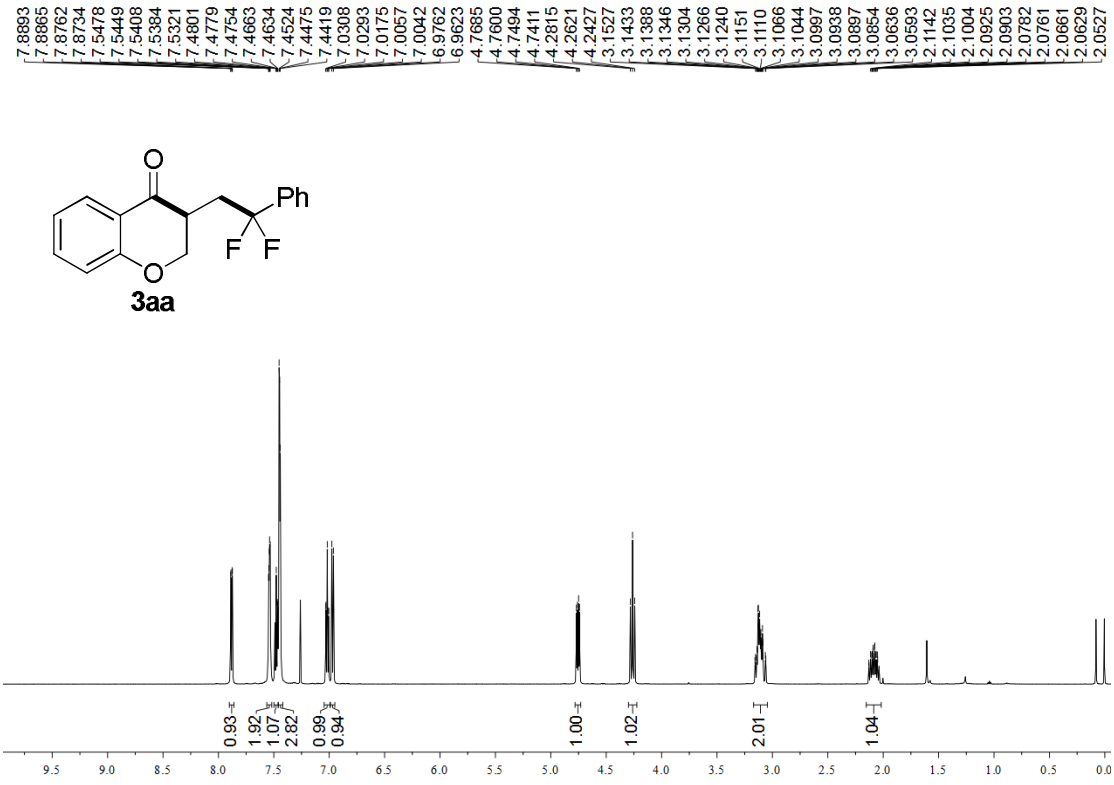
Compound ***6***: 15 mg, 20% yield, colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74–7.71 (m, 1H), 7.51–7.46 (m, 3H), 7.45–7.43 (m, 3H), 7.05–6.97 (m, 1H), 6.90 (d, J = 8.5 Hz, 1H), 4.09 (t, J = 6.1 Hz, 2H), 2.59 (s, 3H), 2.41–2.32 (m, 2H), 2.08–2.01 (m, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 199.7, 158.0, 136.9, 133.7, 130.5, 129.9 (t, J = 1.5 Hz), 128.7, 128.5, 124.8 (t, J = 6.2 Hz), 122.7 (t, J = 242.5 Hz), 120.7, 112.1, 67.5, 36.1 (t, J = 28.3 Hz), 32.0, 22.8; ^{19}F NMR (565 MHz, $CDCl_3$) δ -96.1; HRMS (ESI) calcd for $C_{17}H_{16}F_2NaO_2$ $(M+Na)^+$ 327.1167 found 327.1161.

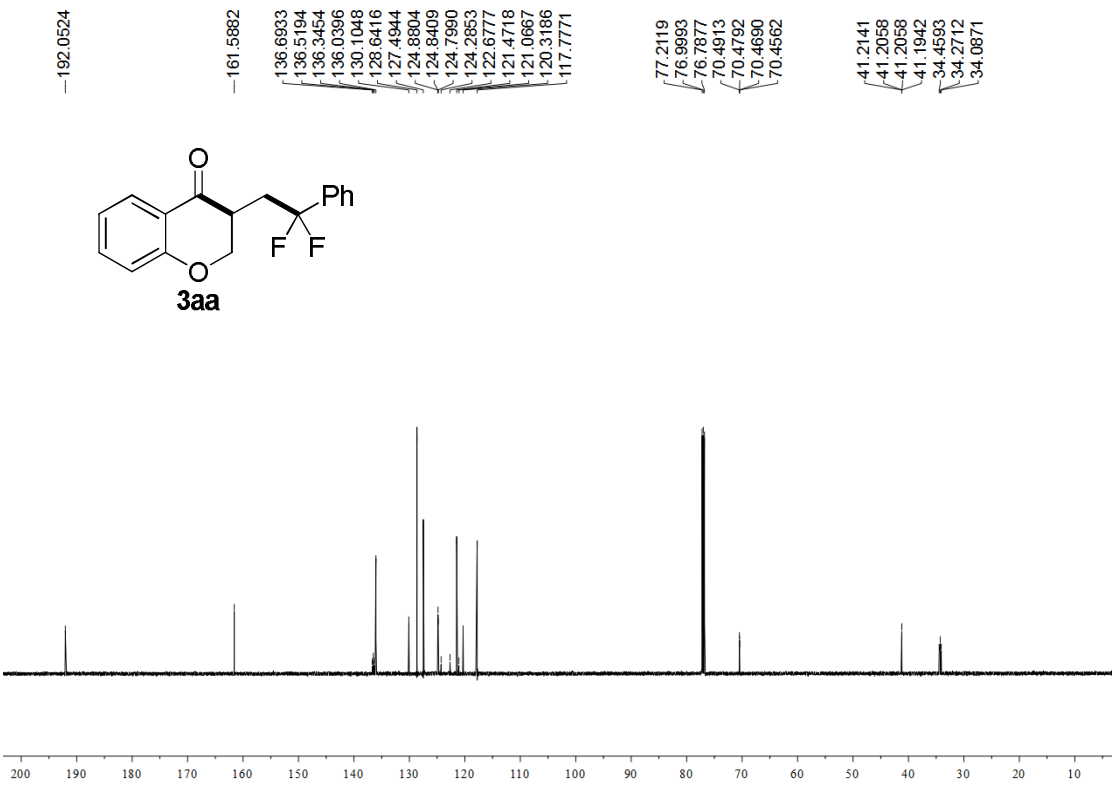
Experiment procedure[3] for the preparation of $PhI(O_2CCF_2Ph)_2$ (5)



Following the literature procedure,[3] a mixture of $PhI(OAc)_2$ (1.5 g, 4.65 mmol) and **2a** (1.6 g, 9.3 mmol) in 40 mL of 1:1 mixture of $CHCl_3$ and PhMe was heated at 45 ^{o}C under a residual pressure until complete dryness, and this operation was repeated about 4 times. The residual solid was purified by recrystallization from dichloromethane/hexane to give 2.2 g (90% yield) of **5** as a white solid, mp 100–101 ^{o}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, J = 7.8 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.48–7.44 (m, 4H), 7.43 (d, J = 7.4 Hz, 2H), 7.41–7.39 (m, 2H), 7.38–7.33 (m, 3H), 7.29 (t, J = 7.6 Hz, 1H); ^{13}C NMR (600 MHz, $CDCl_3$) δ 167.8 (t, J = 34.7 Hz), 134.5, 132.6, 131.4, 130.8, 128.5, 128.3, 125.3 (t, J = 6.0 Hz), 112.5 (t, J = 254.1 Hz); ^{19}F NMR (565 MHz, $CDCl_3$) δ -101.7; HRMS (ESI) calcd for $C_{22}H_{15}F_4INaO_4$ $(M+Na)^+$ 568.9843 found 568.9851.

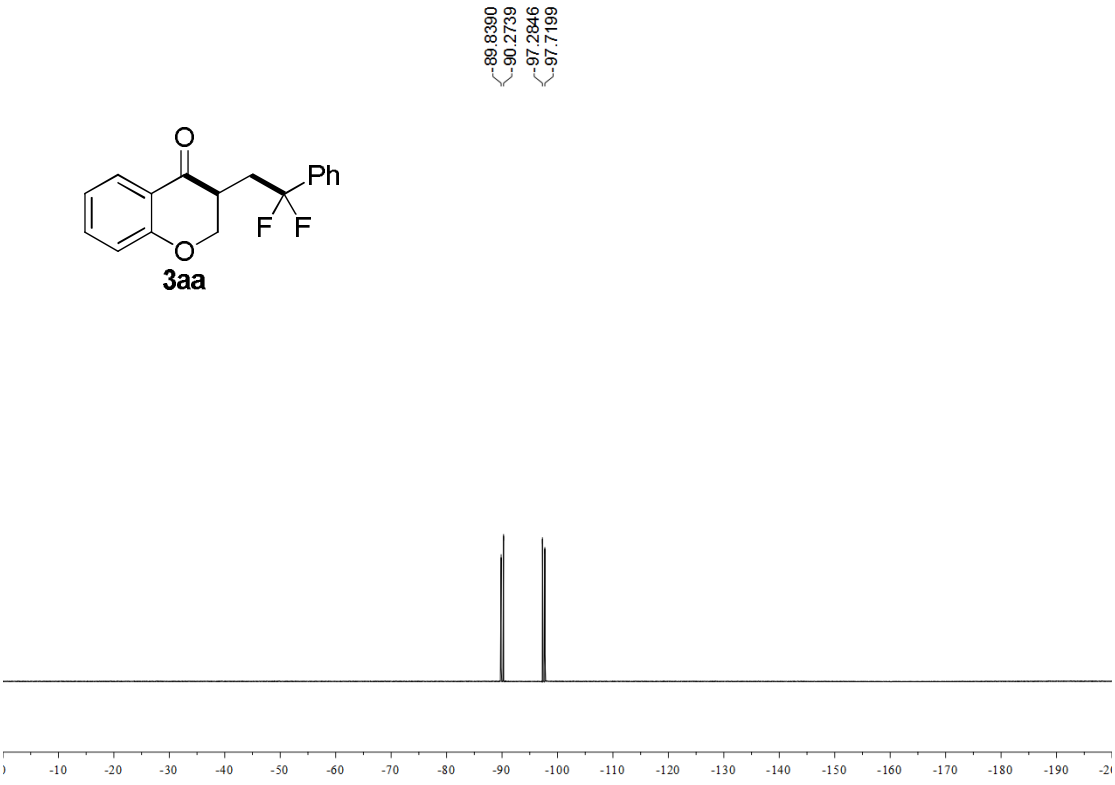
[3] R. Sakamoto, H. Kashiwagi and K. Maruoka, *Org. Lett.*, 2017, **19**, 5126.


7.8893
7.8865
7.8762
7.8734
7.5478
7.5449
7.5408
7.5384
7.5321
7.4801
7.4779
7.4754
7.4663
7.4634
7.4524
7.4475
7.4419
7.0308
7.0293
7.0175
7.0057
7.0042
6.9762
6.9623
4.7685
4.7600
4.7494
4.7411
4.2815
4.2621
4.2427
3.1527
3.1433
3.1388
3.1346
3.1304
3.1266
3.1240
3.1151
3.1110
3.1066
3.1044
3.0997
3.0938
3.0897
3.0854
3.0636
3.0593
2.1142
2.1035
2.1004
2.0925
2.0903
2.0782
2.0761
2.0661
2.0629
2.0527
O
Ph
F F
O
3aa
0.93
1.92
1.07
2.82
0.99
0.94
1.00
1.02
2.01
1.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

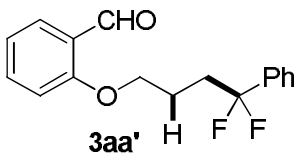

192.0524
161.5882
136.6933
136.5194
136.3454
136.0396
130.1048
128.6416
127.4944
124.8804
124.8409
124.7990
124.2853
122.6777
121.4718
121.0667
120.3186
117.7771
77.2119
76.9993
76.7877
70.4913
70.4792
70.4690
70.4562
41.2141
41.2058
41.2058
41.1942
34.4593
34.2712
34.0871
O
Ph
F F
O
3aa
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

O
Ph
F F
O

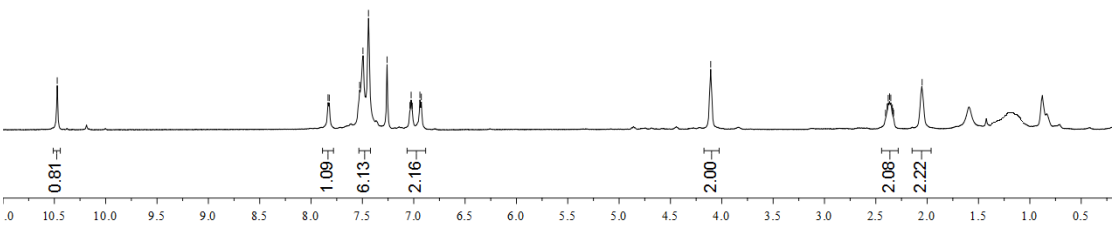
3aa

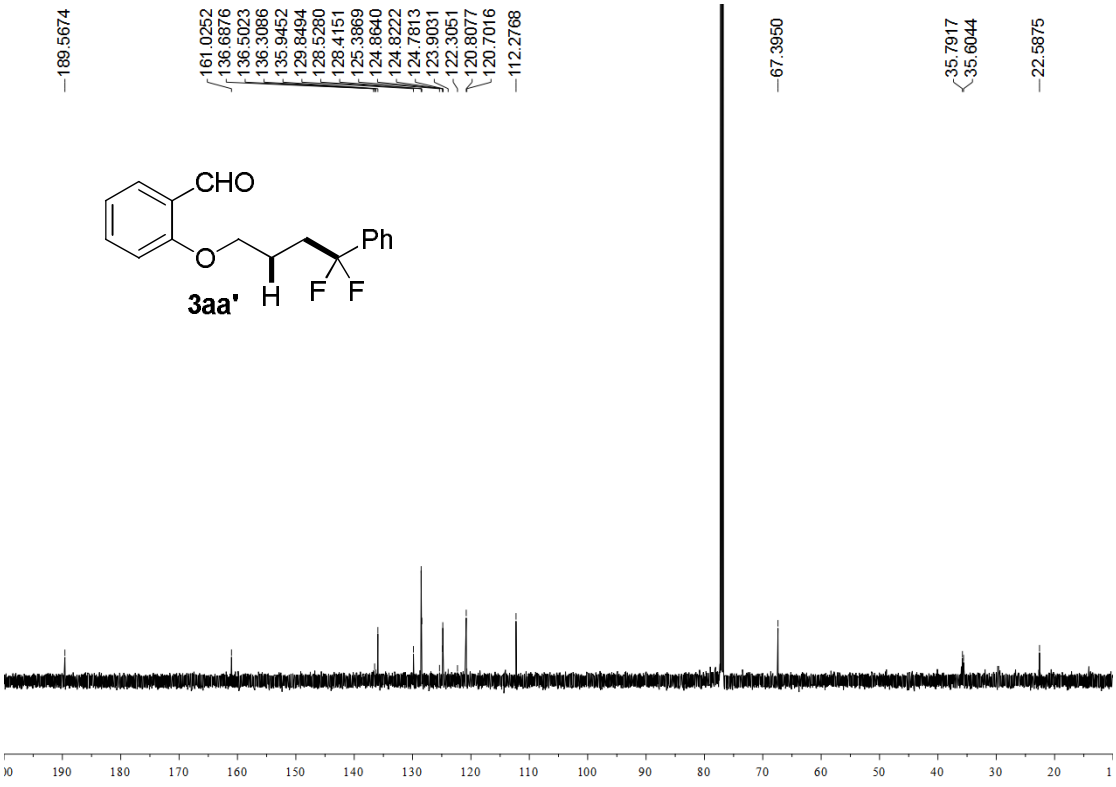

-89.8390
-90.2739
-97.2846
-97.7199
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

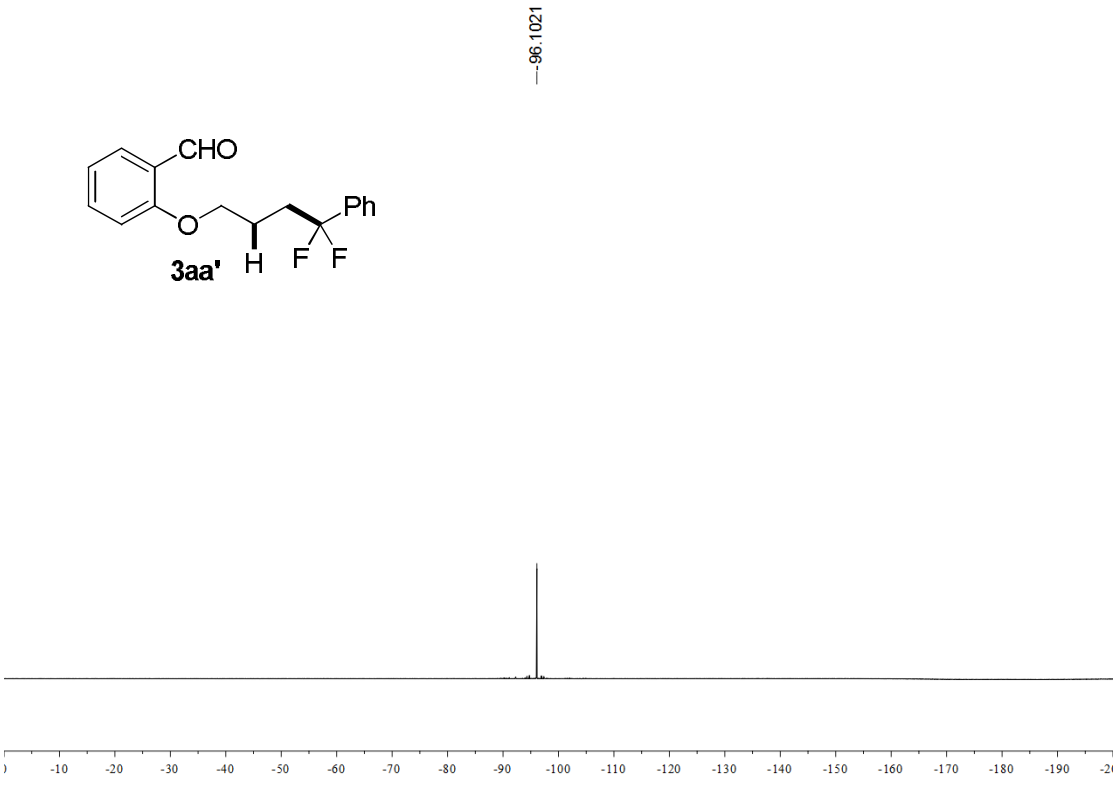

10.4719
7.8347
7.8226
7.5268
7.4946
7.4408
7.2596
7.0370
7.0254
7.0142
6.9399
6.9262
4.1080
2.4054
2.3894
2.3792
2.3658
2.3540
2.3392
2.3335
2.3268
2.0491

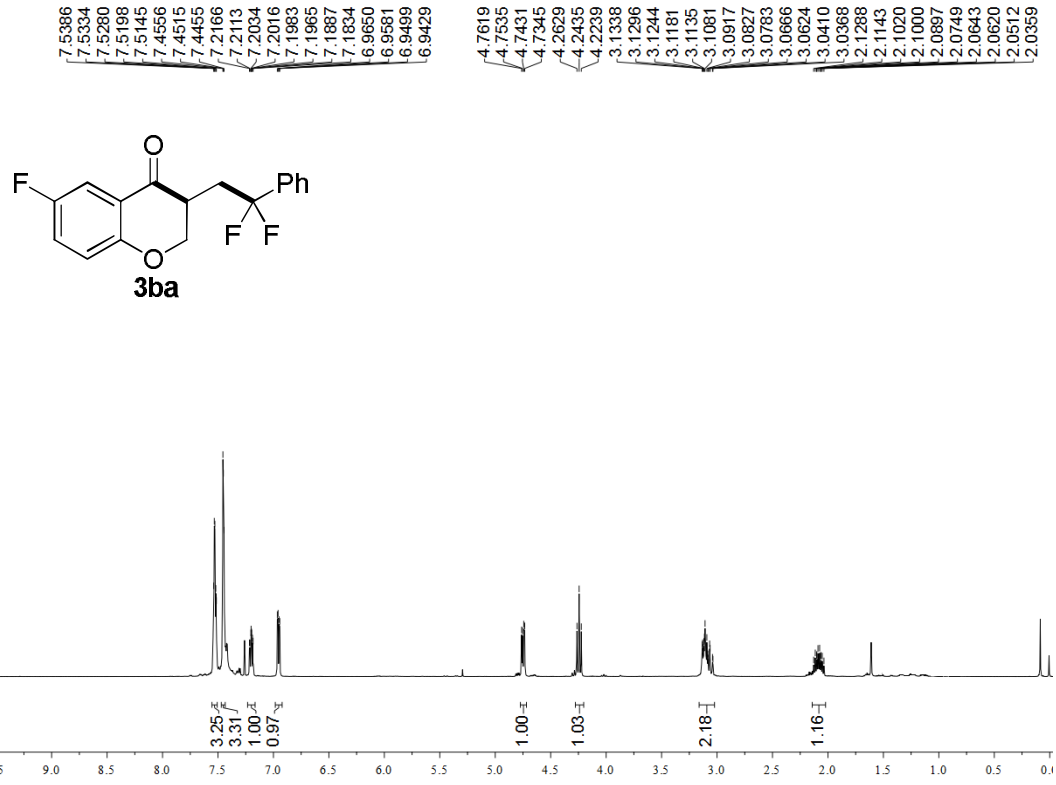

CHO
O
Ph
H
F F

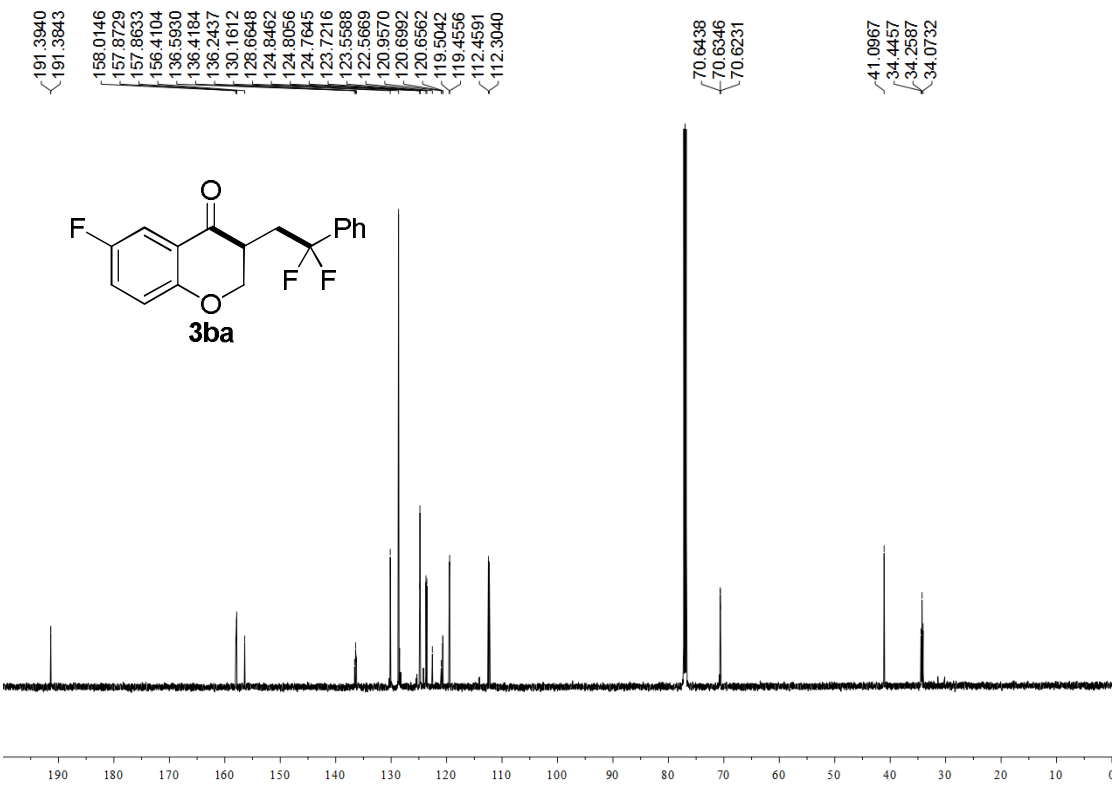
3aa'

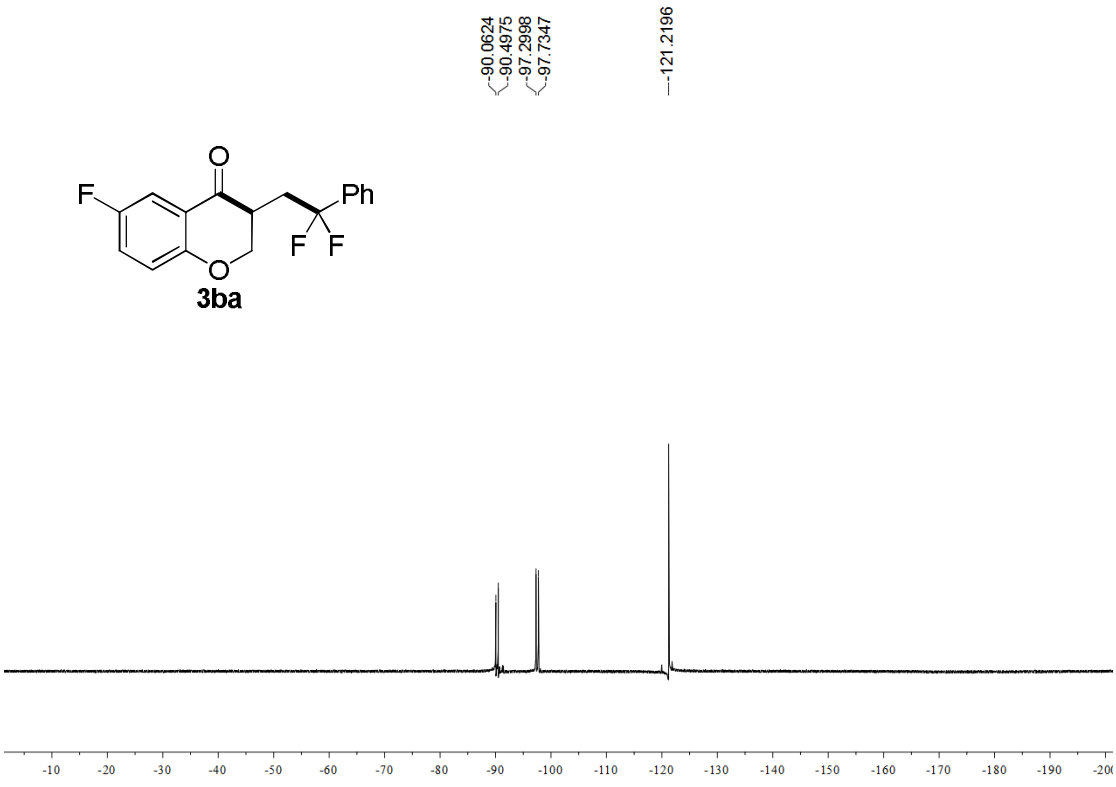

0.81
1.09
6.13
2.16
2.00
2.08
2.22
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

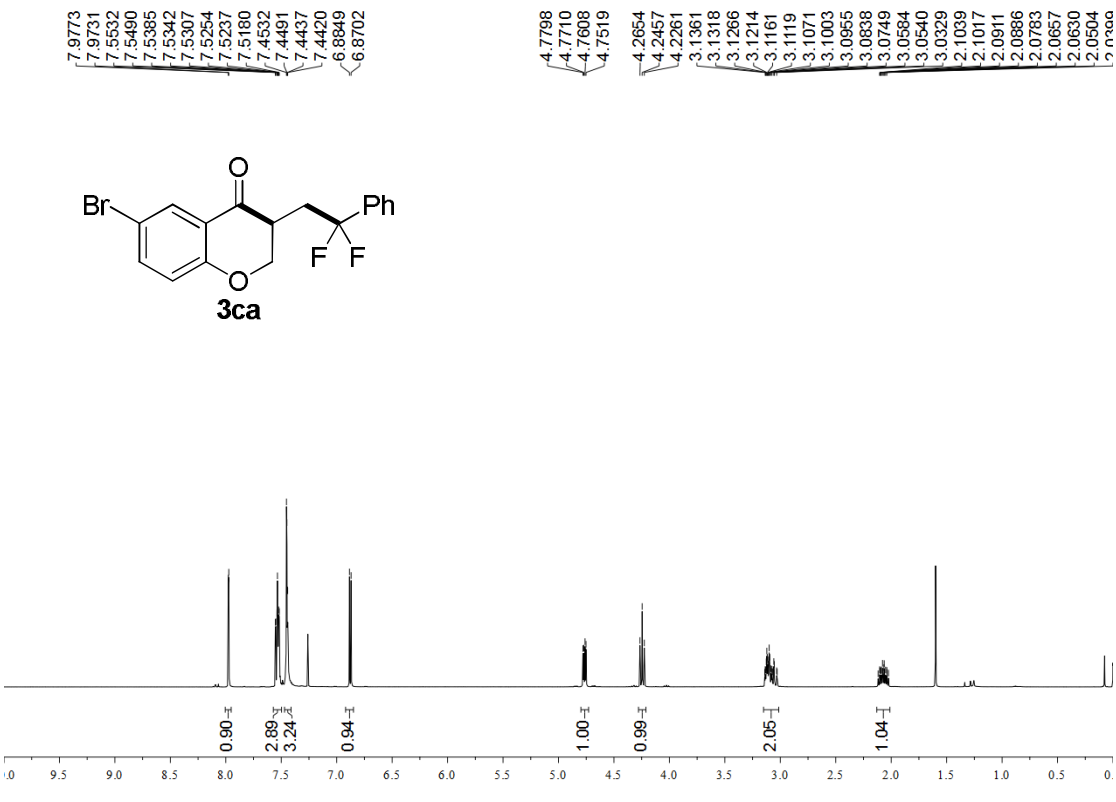

189.5674
161.0252
136.6876
136.5023
136.3086
135.9452
129.8494
128.5280
128.4151
125.3869
124.8640
124.8222
124.7813
123.9031
122.3051
120.8077
120.7016
112.2768
67.3950
35.7917
35.6044
22.5875
CHO
O
Ph
H
F
F
3aa'

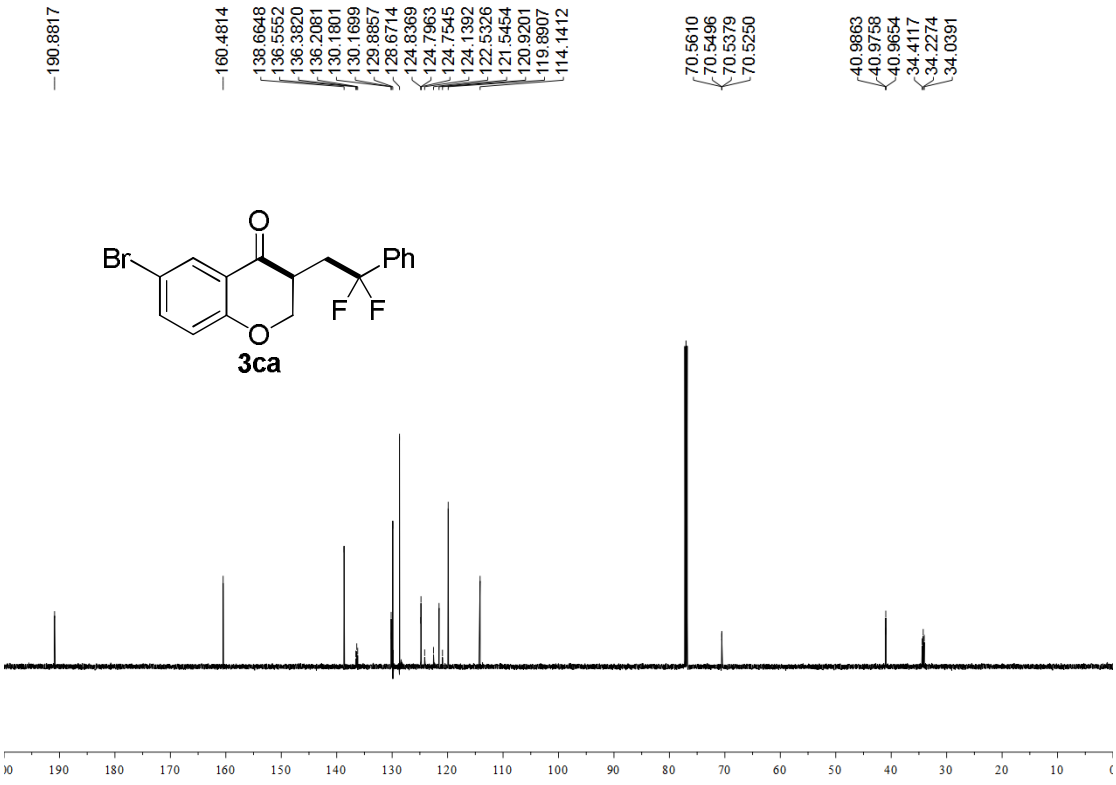

96.1021
CHO
O
Ph
H
F
F
3aa'

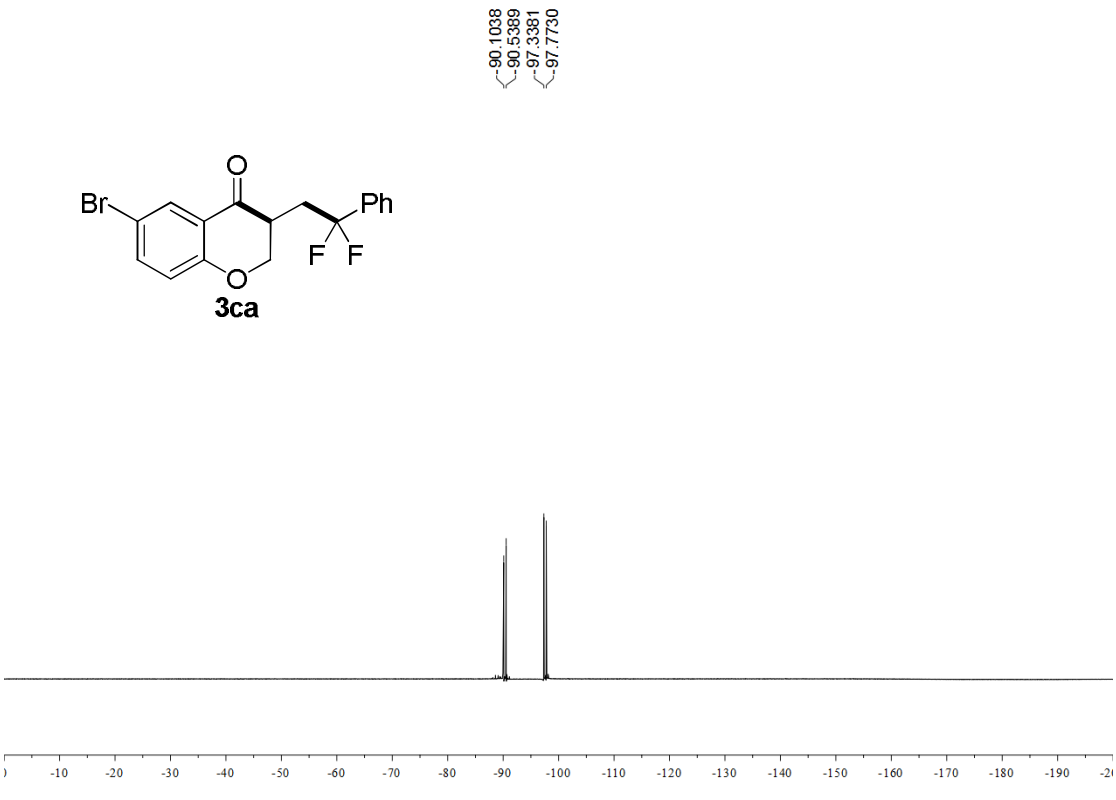

7.5386
7.5334
7.5280
7.5198
7.5145
7.4556
7.4515
7.4455
7.2166
7.2113
7.2034
7.2016
7.1983
7.1965
7.1887
7.1834
6.9650
6.9581
6.9499
6.9429
4.7619
4.7535
4.7431
4.7345
4.2629
4.2435
4.2239
3.1338
3.1296
3.1244
3.1181
3.1135
3.1081
3.0917
3.0827
3.0783
3.0666
3.0624
3.0410
3.0368
2.1288
2.1143
2.1020
2.1000
2.0897
2.0749
2.0643
2.0620
2.0512
2.0359
F
O
Ph
F F
O
3ba
3.25
3.31
1.00
0.97
1.00
1.03
2.18
1.16
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

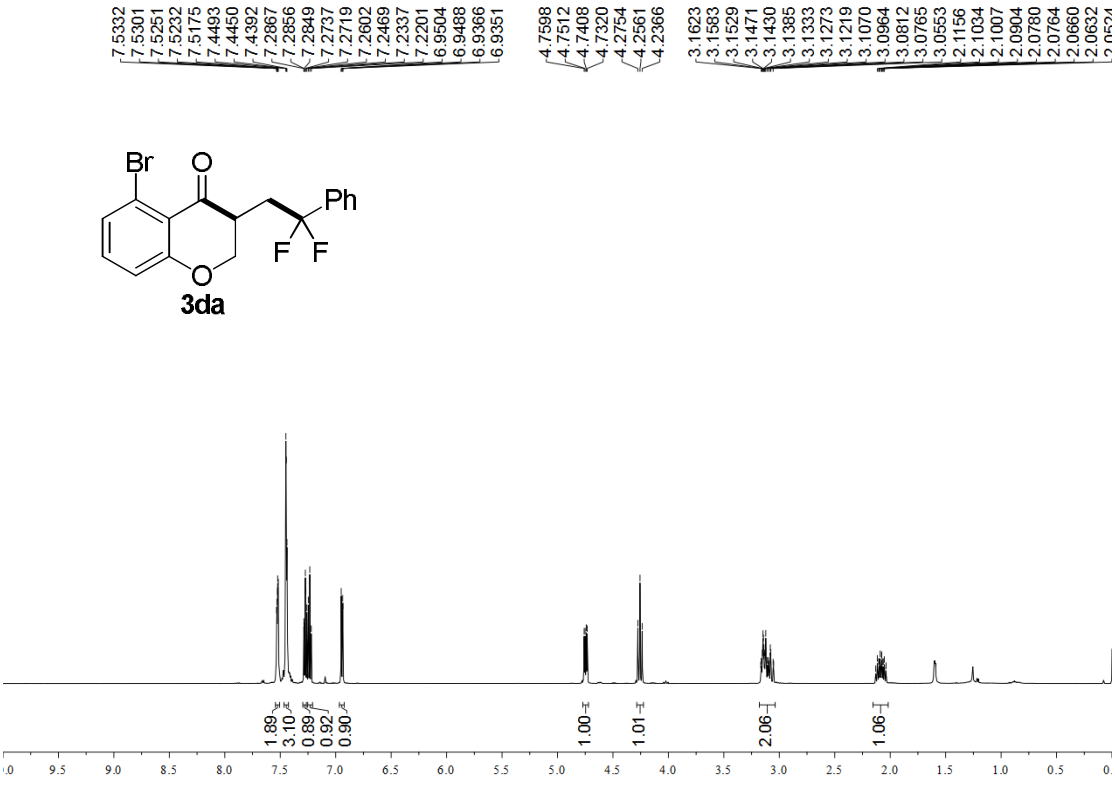

191.3940
191.3843
158.0146
157.8729
157.8633
156.4104
136.5930
136.4184
136.2437
130.1612
128.6648
124.8462
124.8056
124.7645
123.7216
123.5588
122.5669
120.9570
120.6992
120.6562
119.5042
119.4556
112.4591
112.3040
70.6438
70.6346
70.6231
41.0967
34.4457
34.2587
34.0732
F
O
Ph
F F
O
3ba
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

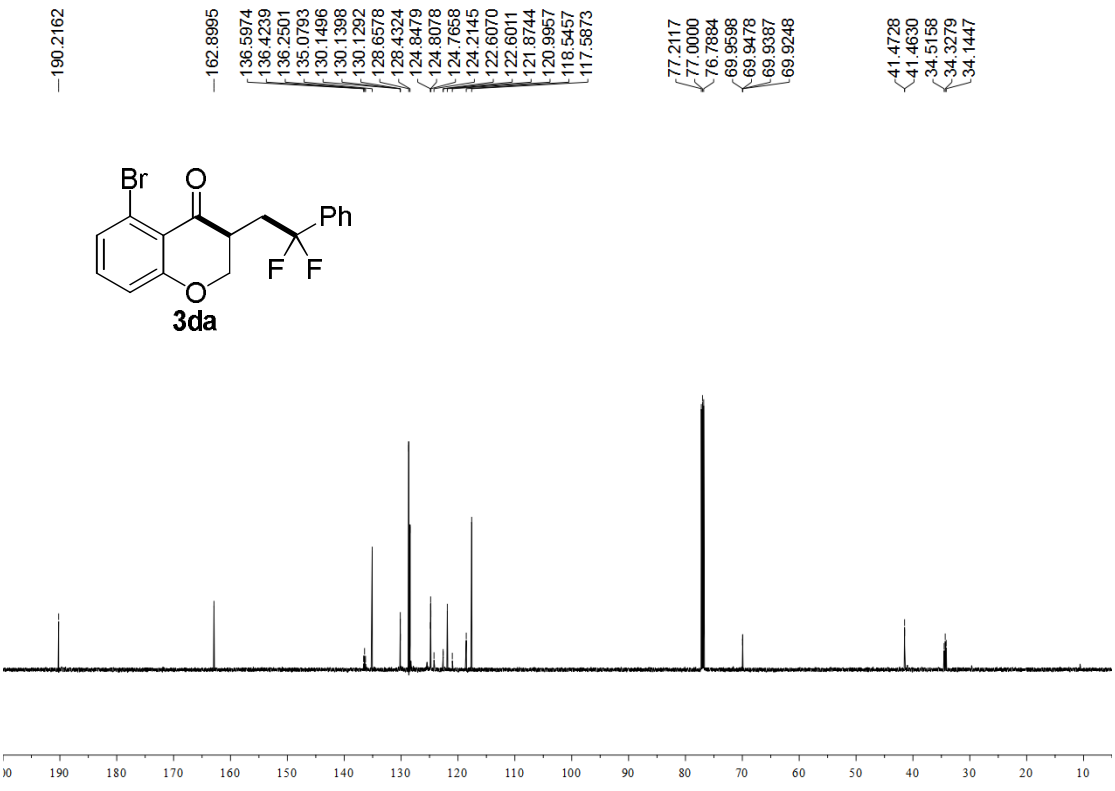

-90.0624
-90.4975
-97.2998
-97.7347
-121.2196
F
O
Ph
F
F
O
3ba
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

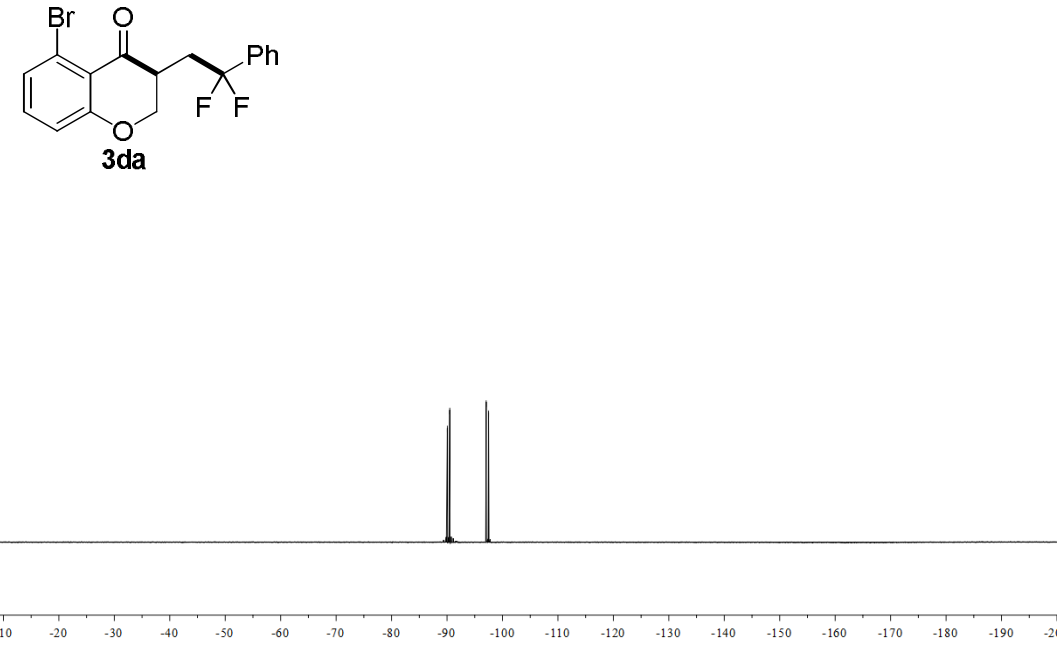

7.9773
7.9731
7.5532
7.5490
7.5385
7.5342
7.5307
7.5254
7.5237
7.5180
7.4532
7.4491
7.4437
7.4420
6.8849
6.8702
4.7798
4.7710
4.7608
4.7519
4.2654
4.2457
4.2261
3.1361
3.1318
3.1266
3.1214
3.1161
3.1119
3.1071
3.1003
3.0955
3.0838
3.0749
3.0584
3.0540
3.0329
2.1039
2.1017
2.0911
2.0886
2.0783
2.0657
2.0630
2.0504
Br
O
Ph
F
F
O
3ca
0.90
2.89
3.24
0.94
1.00
0.99
2.05
1.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

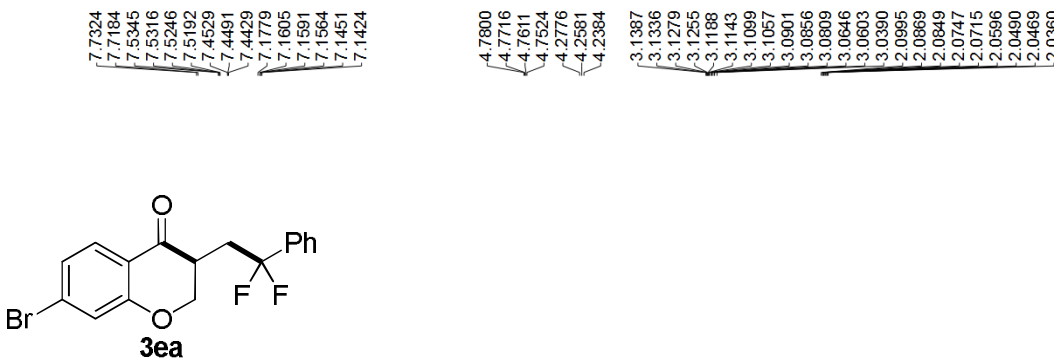

190.8817
160.4814
138.6648
136.5552
136.3820
136.2081
130.1801
130.1699
129.8857
128.6714
124.8369
124.7963
124.7545
124.1392
122.5326
121.5454
120.9201
119.8907
114.1412
70.5610
70.5496
70.5379
70.5250
40.9863
40.9758
40.9654
34.4117
34.2274
34.0391
Br
O
Ph
F F
O
3ca

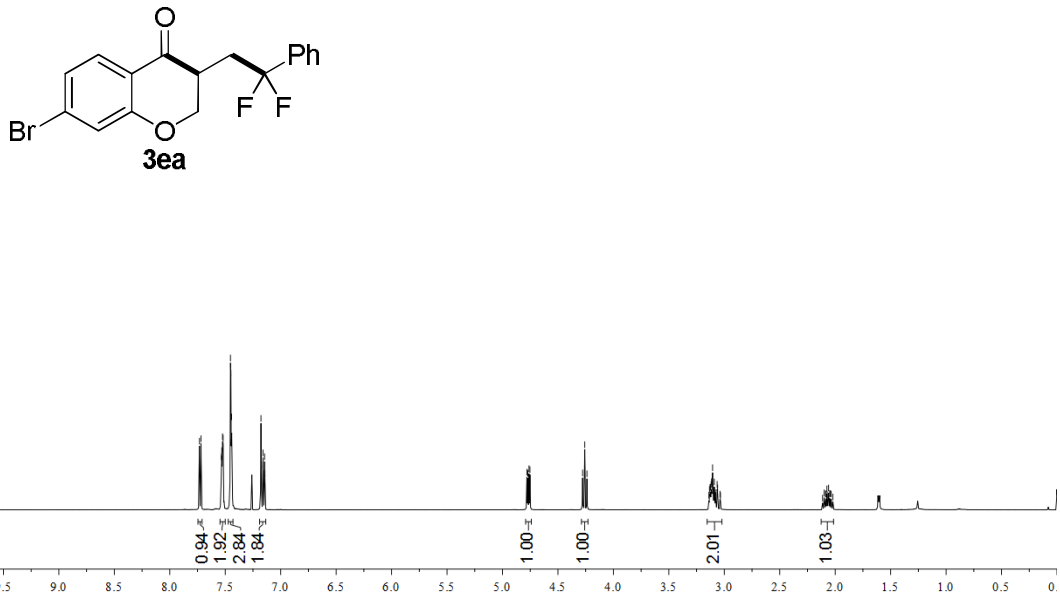

-90.1038
-90.5389
-97.3381
-97.7730
Br
O
Ph
F F
O
3ca

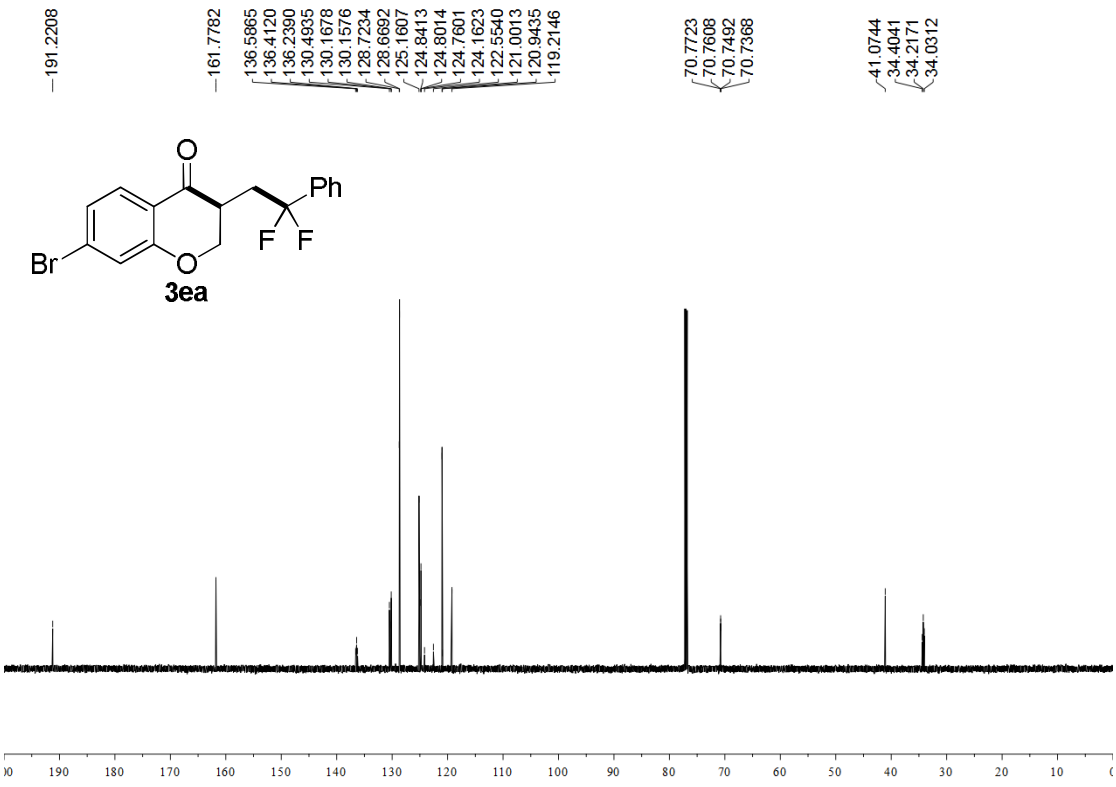

7.5332
7.5301
7.5251
7.5232
7.5175
7.4493
7.4450
7.4392
7.2867
7.2856
7.2849
7.2737
7.2719
7.2602
7.2469
7.2337
7.2201
6.9504
6.9488
6.9366
6.9351
4.7598
4.7512
4.7408
4.7320
4.2754
4.2561
4.2366
3.1623
3.1583
3.1529
3.1471
3.1430
3.1385
3.1333
3.1273
3.1219
3.1070
3.0964
3.0812
3.0765
3.0553
2.1156
2.1034
2.1007
2.0904
2.0780
2.0764
2.0660
2.0632
Br
O
Ph
F
F
O
3da
1.89
3.10
0.89
0.92
0.90
1.00
1.01
2.06
1.06

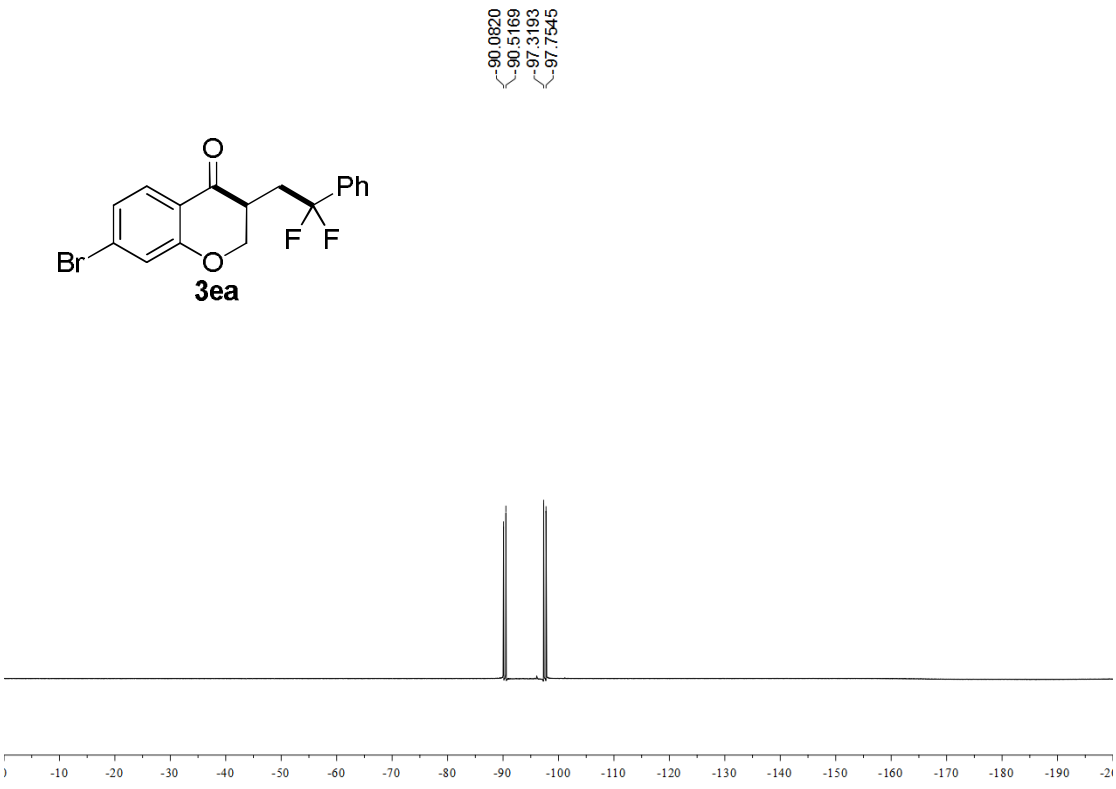

190.2162
162.8995
136.5974
136.4239
136.2501
135.0793
130.1496
130.1398
130.1292
128.6578
128.4324
124.8479
124.8078
124.7658
124.2145
122.6070
122.6011
121.8744
120.9957
118.5457
117.5873
77.2117
77.0000
76.7884
69.9598
69.9478
69.9387
69.9248
41.4728
41.4630
34.5158
34.3279
34.1447
Br
O
Ph
F
F
O
3da

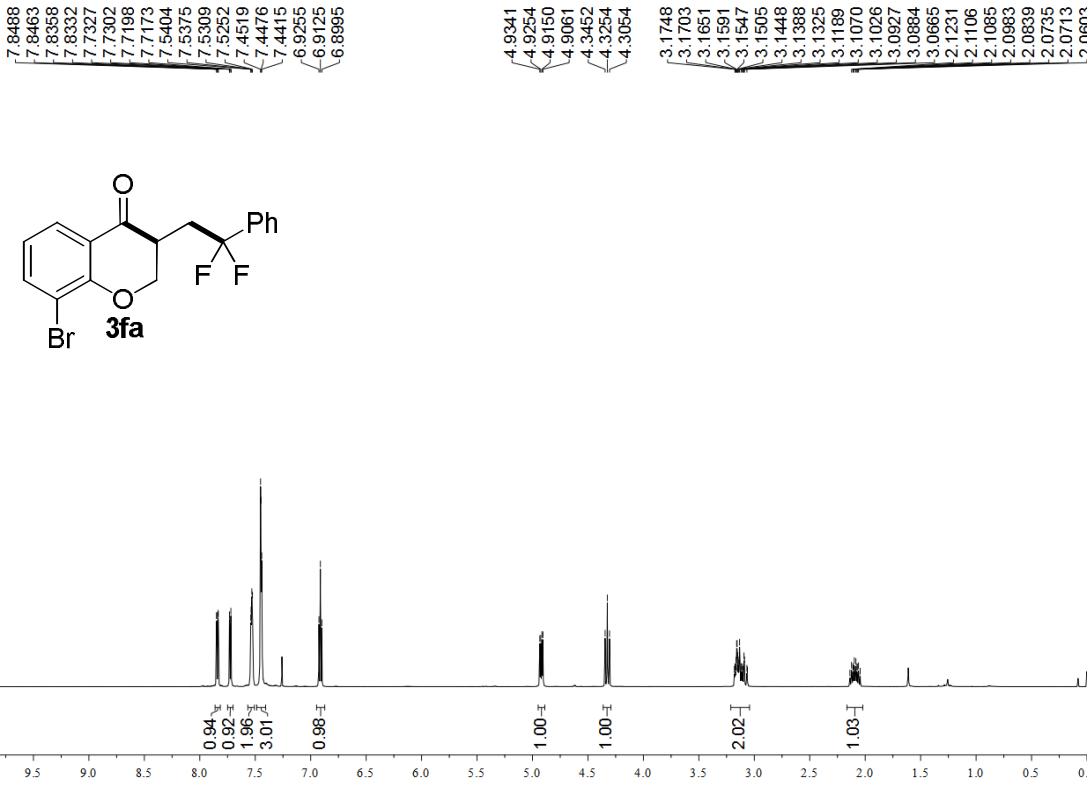

-90.0560
-90.4913
-97.0541
-97.4893
Br
O
Ph
F
F
O
3da
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

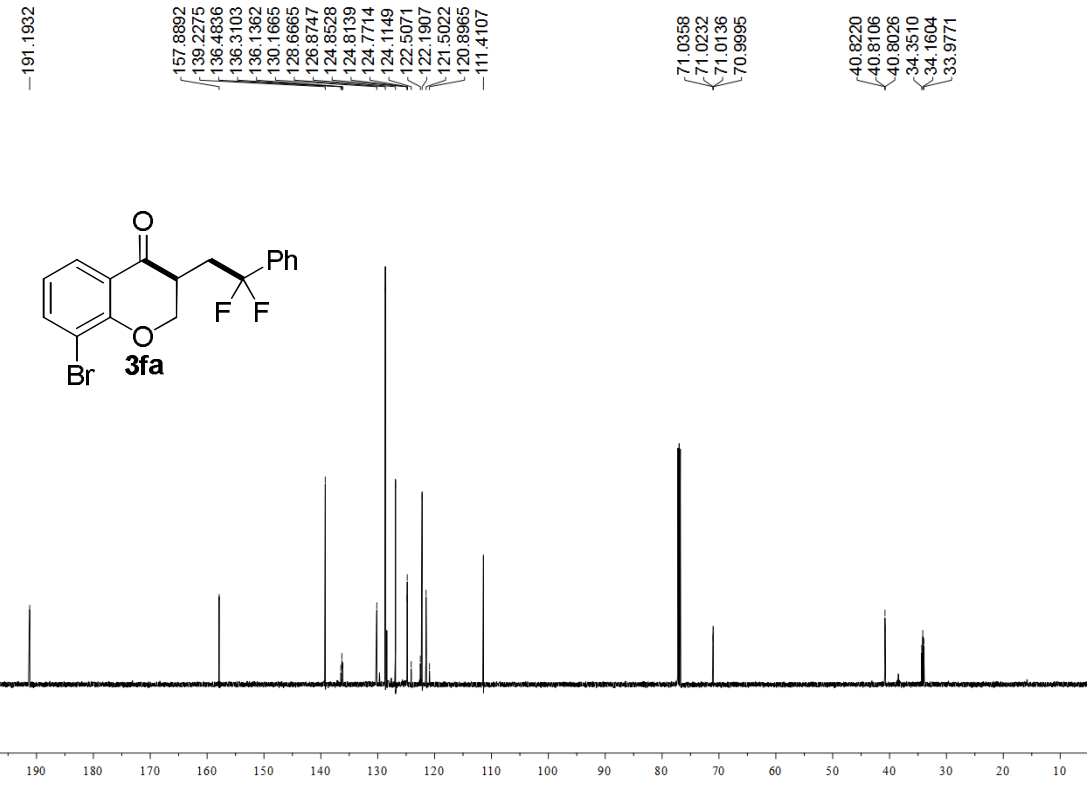

7.7324
7.7184
7.5345
7.5316
7.5246
7.5192
7.4529
7.4491
7.4429
7.1779
7.1605
7.1591
7.1564
7.1451
7.1424
4.7800
4.7716
4.7611
4.7524
4.2776
4.2581
4.2384
3.1387
3.1336
3.1279
3.1255
3.1188
3.1143
3.1099
3.1057
3.0901
3.0856
3.0809
3.0646
3.0603
3.0390
2.0995
2.0869
2.0849
2.0747
2.0715
2.0596
2.0490
2.0469

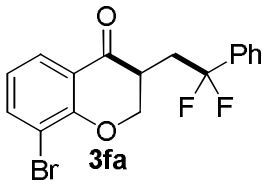

O
Ph
F
F
Br
O
3ea
0.94
1.92
2.84
1.84
1.00
1.00
2.01
1.03
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5


191.2208
161.7782
136.5865
136.4120
136.2390
130.4935
130.1678
130.1576
128.7234
128.6692
125.1607
124.8413
124.8014
124.7601
124.1623
122.5540
121.0013
120.9435
119.2146
70.7723
70.7608
70.7492
70.7368
41.0744
34.4041
34.2171
34.0312
O
Ph
F F
Br
O
3ea
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


90.0820
90.5169
97.3193
97.7545
O
Ph
F F
Br
O
3ea
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

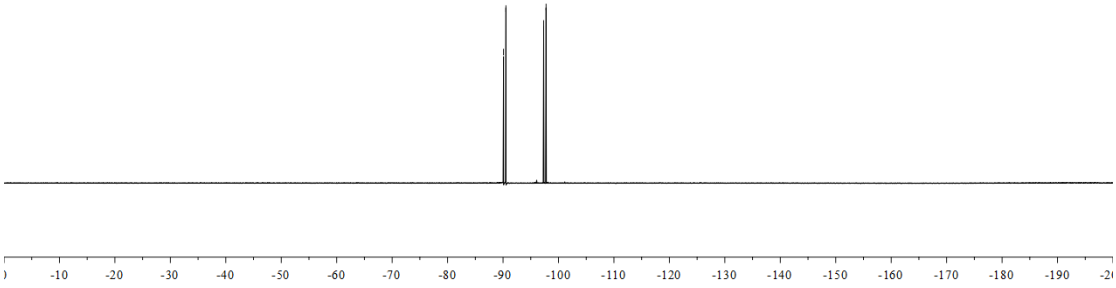

7.8488
7.8463
7.8358
7.8332
7.7327
7.7302
7.7198
7.7173
7.5404
7.5375
7.5309
7.5252
7.4519
7.4476
7.4415
6.9255
6.9125
6.8995
4.9341
4.9254
4.9150
4.9061
4.3452
4.3254
4.3054
3.1748
3.1703
3.1651
3.1591
3.1547
3.1505
3.1448
3.1388
3.1325
3.1189
3.1070
3.1026
3.0927
3.0884
3.0665
2.1231
2.1106
2.1085
2.0983
2.0839
2.0735
2.0713
O
Ph
F
F
O
Br
3fa
0.94
0.92
1.96
3.01
0.98
1.00
1.00
2.02
1.03

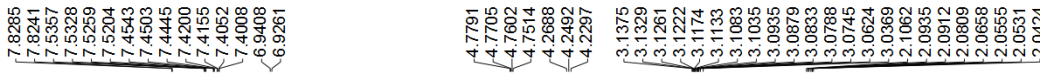

191.1932
157.8892
139.2275
136.4836
136.3103
136.1362
130.1665
128.6665
126.8747
124.8528
124.8139
124.7714
124.1149
122.5071
122.1907
121.5022
120.8965
111.4107
71.0358
71.0232
71.0136
70.9995
40.8220
40.8106
40.8026
34.3510
34.1604
33.9771
O
Ph
F
F
O
Br
3fa

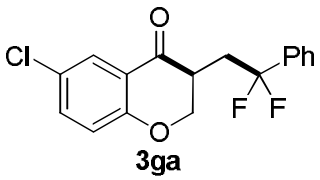

O
Ph
F F
O
Br

3fa

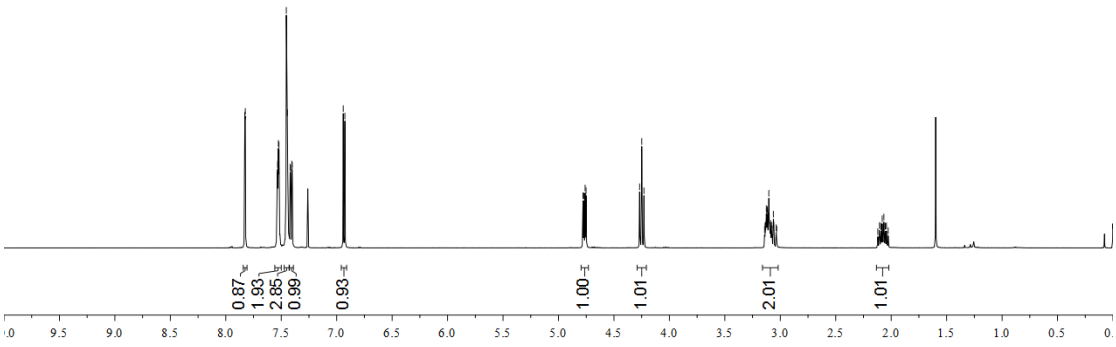

-90.0827
-90.5177
-97.3232
-97.7583

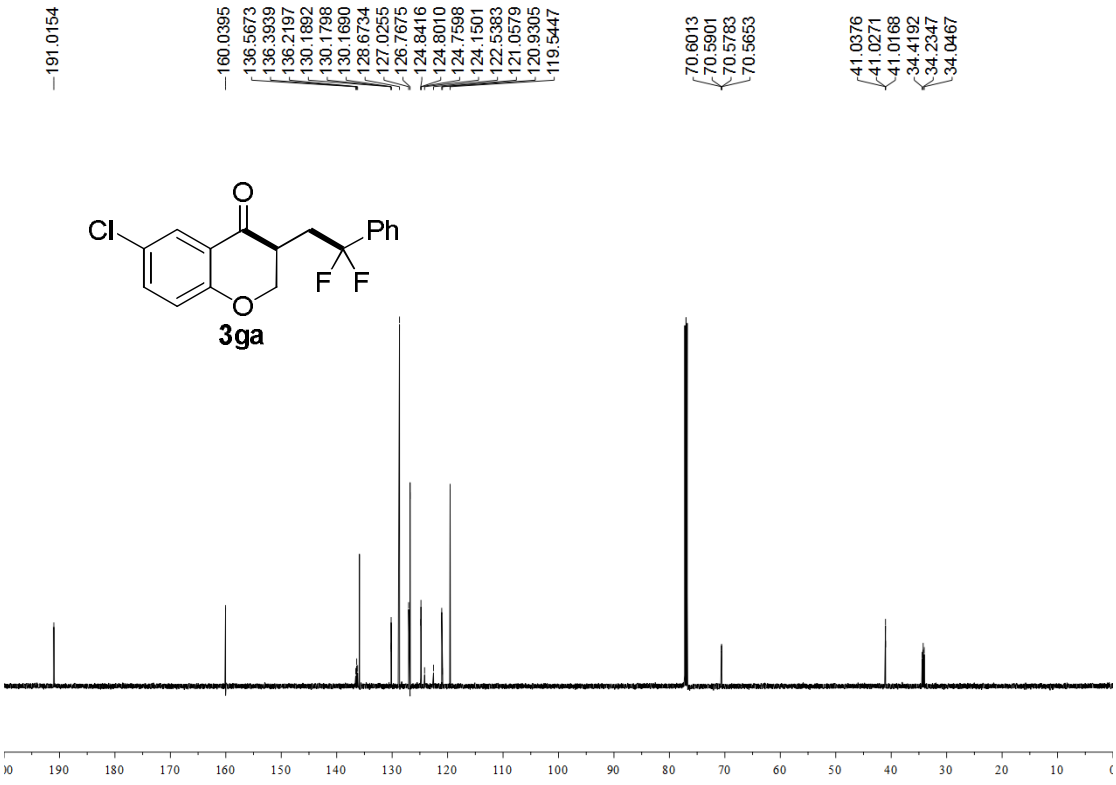

-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

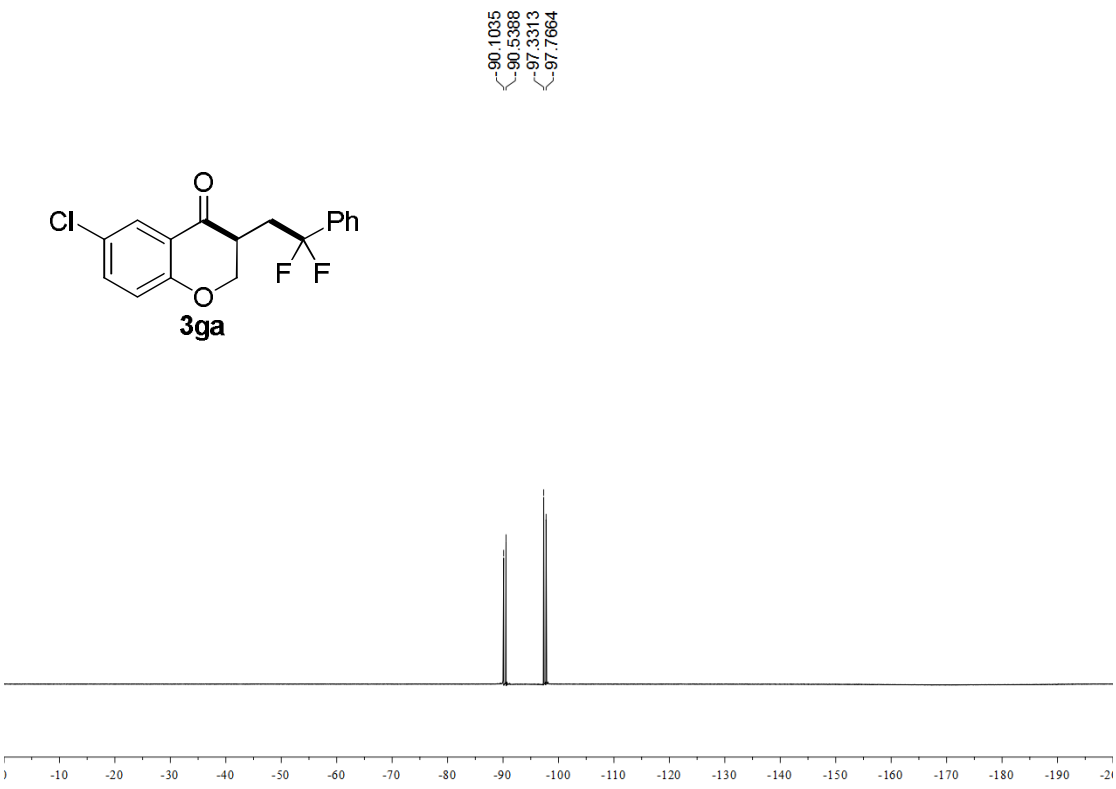

7.8285
7.8241
7.5357
7.5328
7.5259
7.5204
7.4543
7.4503
7.4445
7.4200
7.4155
7.4052
7.4008
6.9408
6.9261
4.7791
4.7705
4.7602
4.7514
4.2688
4.2492
4.2297
3.1375
3.1329
3.1261
3.1222
3.1174
3.1133
3.1083
3.1035
3.0935
3.0879
3.0833
3.0788
3.0745
3.0624
3.0369
2.1062
2.0935
2.0912
2.0809
2.0658
2.0555
2.0531

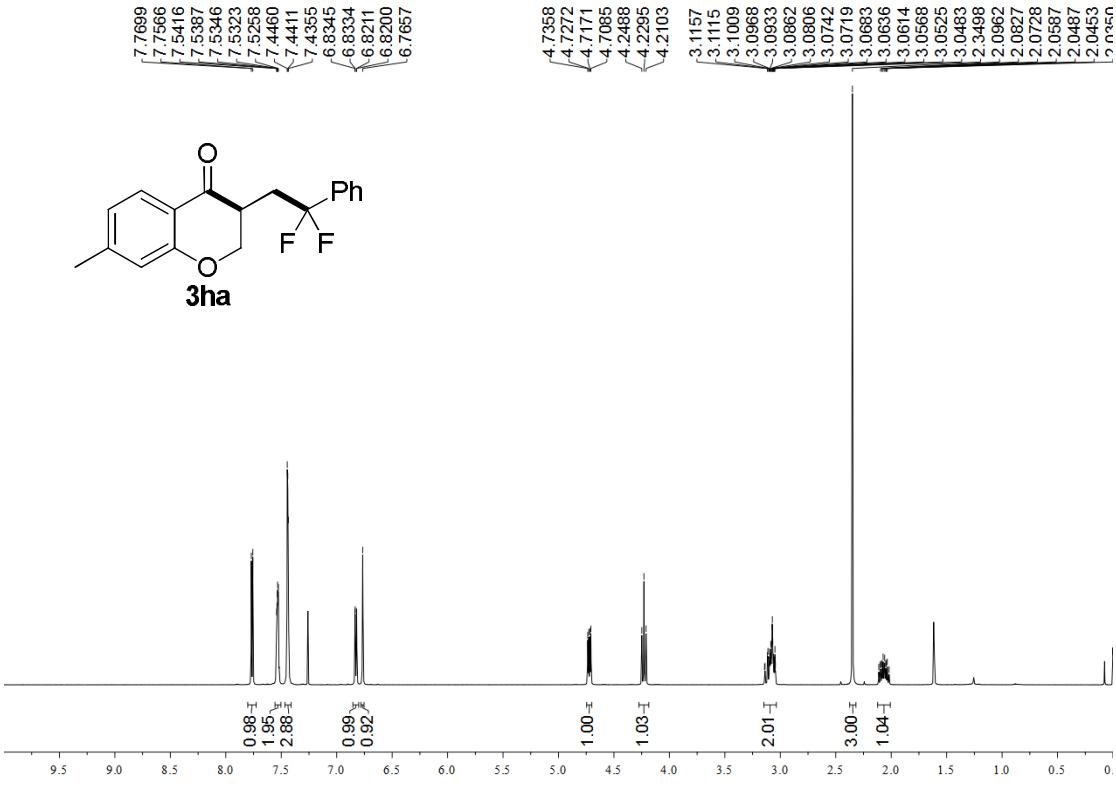

O
Cl
Ph
F F
O

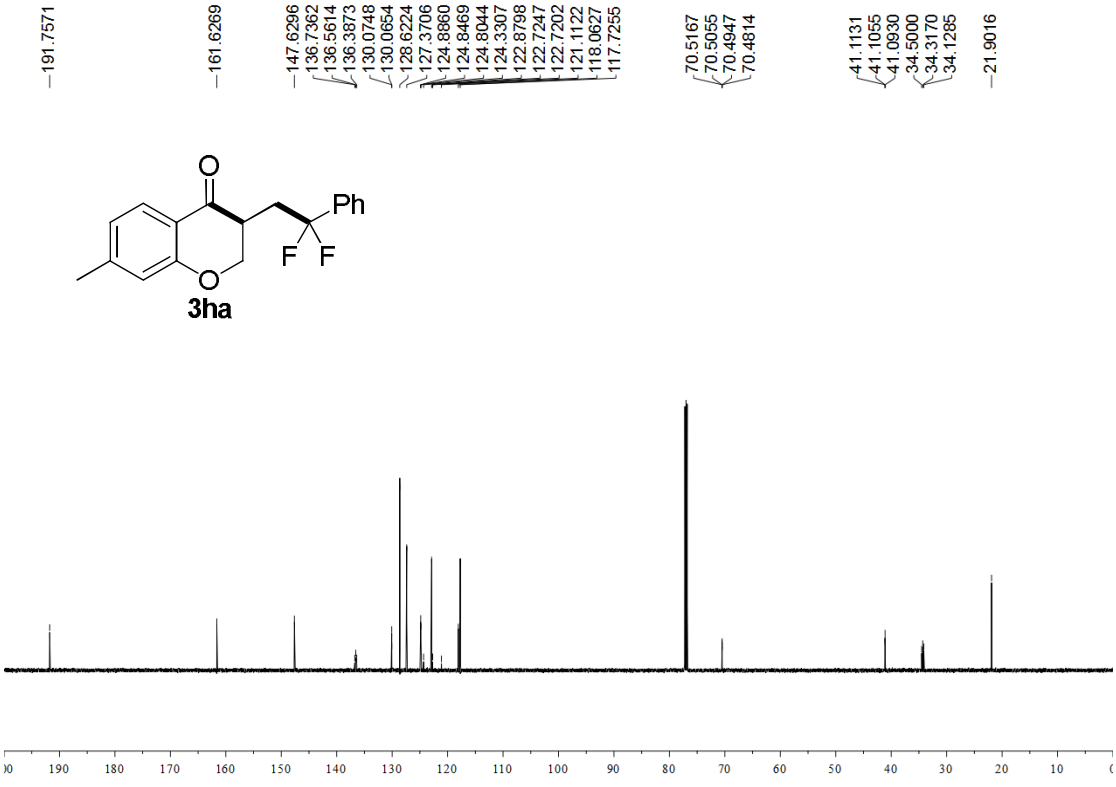
3ga

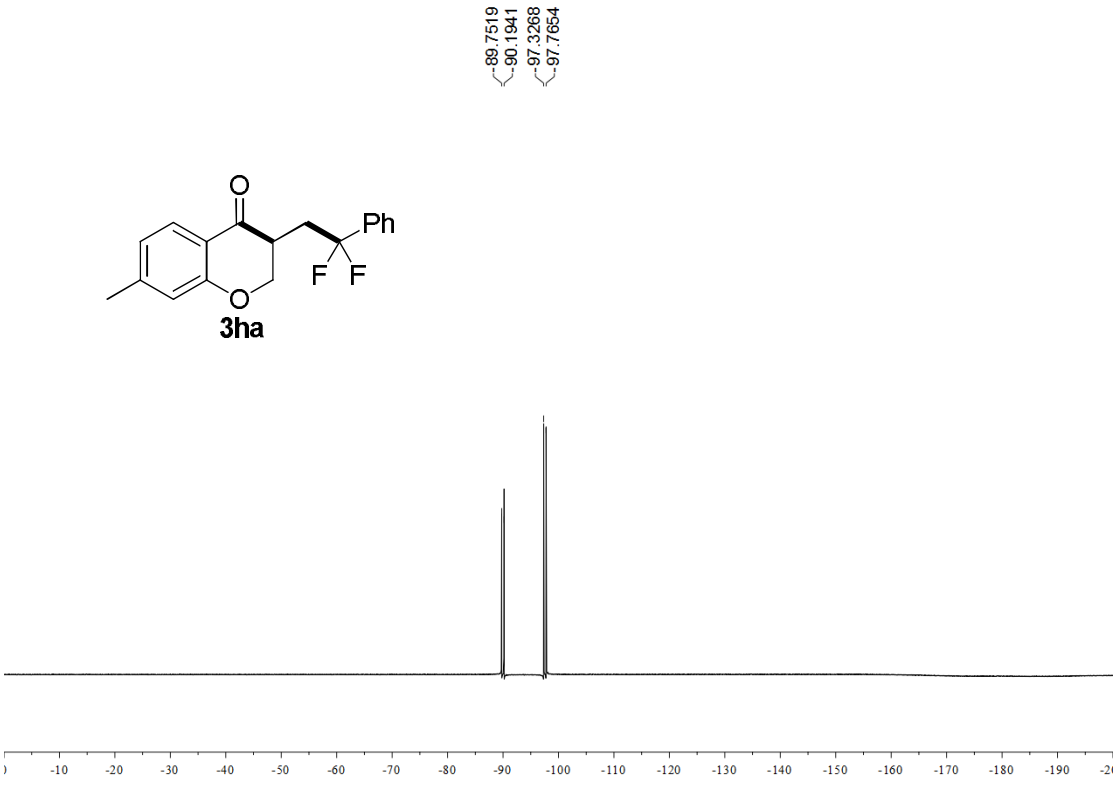

0.87
1.93
2.85
0.99
0.93
1.00
1.01
2.01
1.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

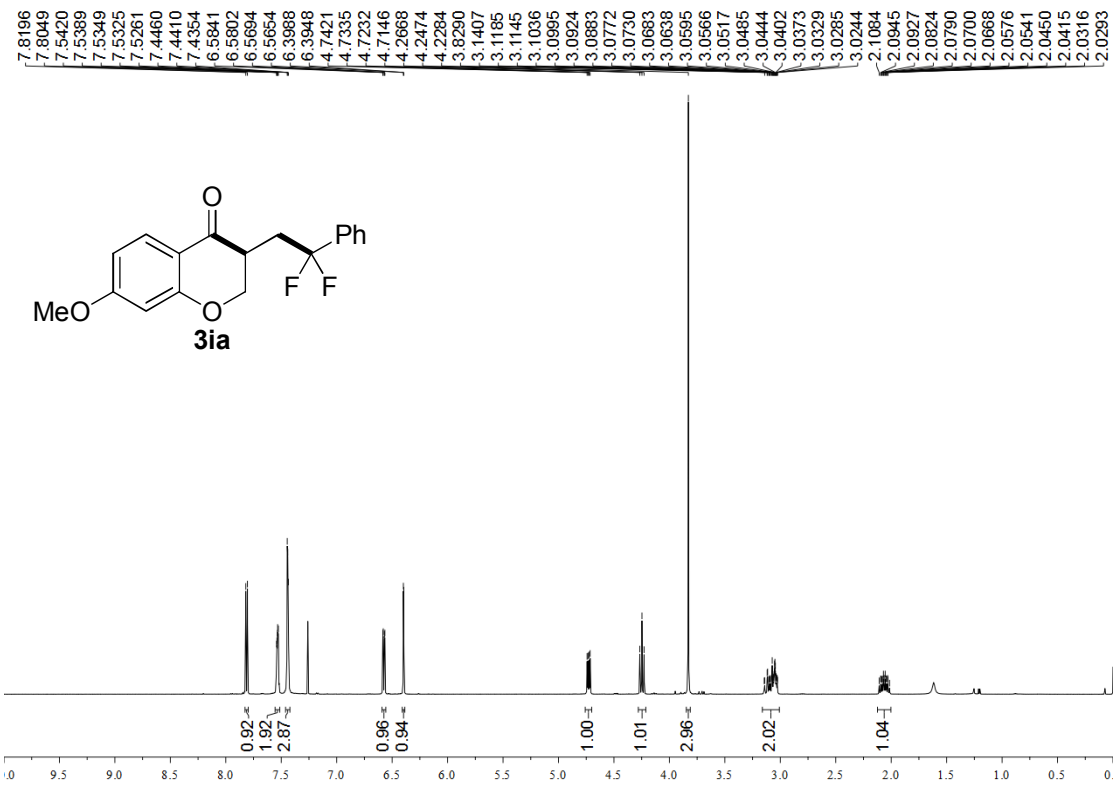

191.0154
160.0395
136.5673
136.3939
136.2197
130.1892
130.1798
130.1690
128.6734
127.0255
126.7675
124.8416
124.8010
124.7598
124.1501
122.5383
121.0579
120.9305
119.5447
70.6013
70.5901
70.5783
70.5653
41.0376
41.0271
41.0168
34.4192
34.2347
34.0467
Cl
O
Ph
F F
O
3ga

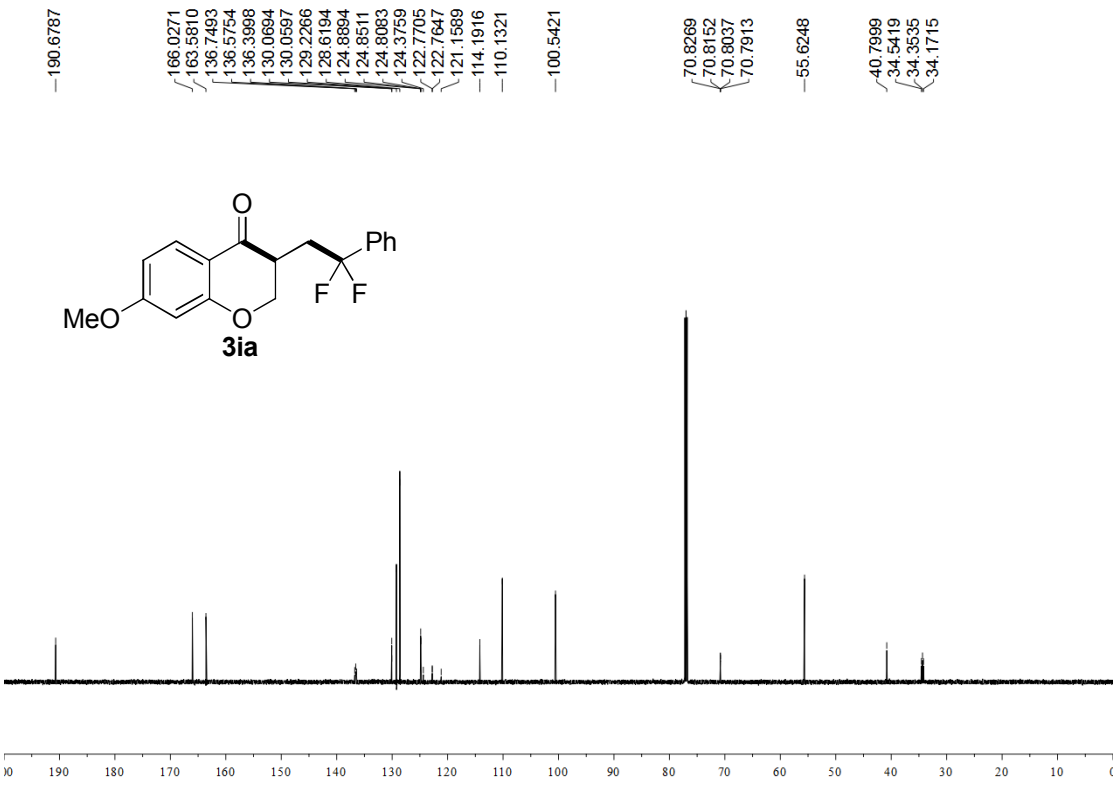

-90.1035
-90.5388
-97.3313
-97.7664
Cl
O
Ph
F F
O
3ga

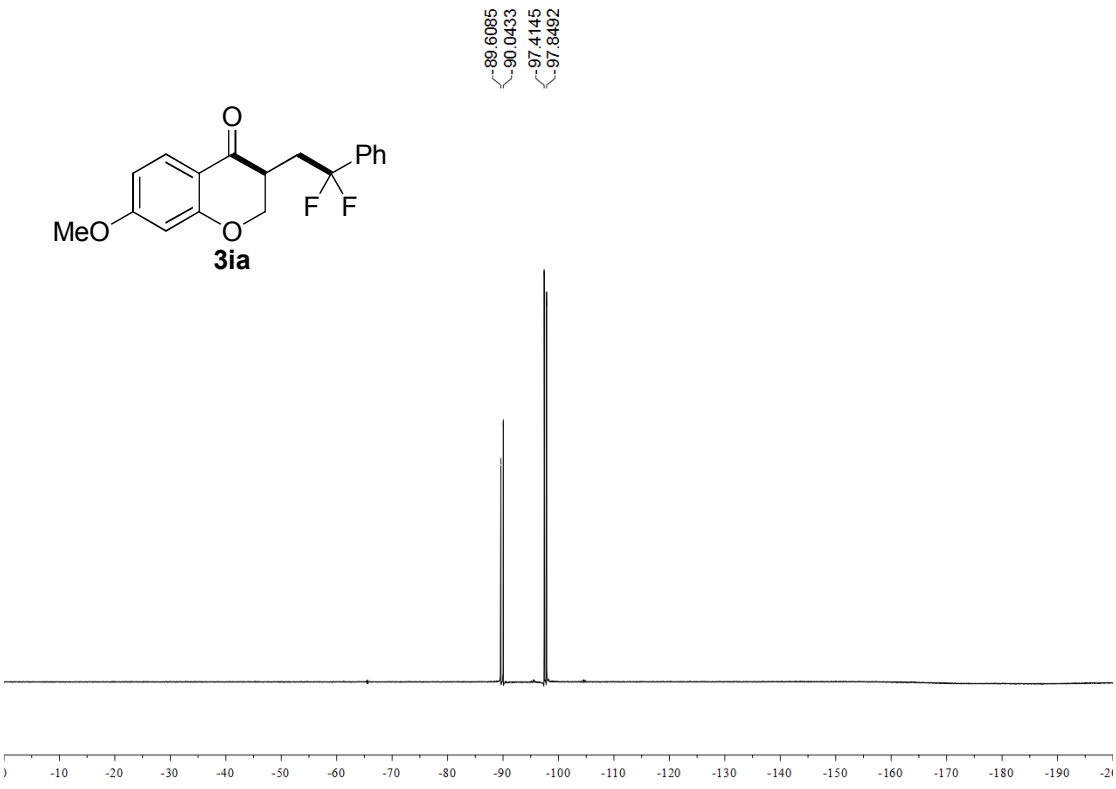

7.7699
7.7566
7.5416
7.5387
7.5346
7.5323
7.5258
7.4460
7.4411
7.4355
6.8345
6.8334
6.8211
6.8200
6.7657
4.7358
4.7272
4.7171
4.7085
4.2488
4.2295
4.2103
3.1157
3.1115
3.1009
3.0968
3.0933
3.0862
3.0806
3.0742
3.0719
3.0683
3.0636
3.0614
3.0568
3.0525
3.0483
2.3498
2.0962
2.0827
2.0728
2.0587
2.0487
2.0453
O
Ph
F
F
O
3ha
0.98
1.95
2.88
0.99
0.92
1.00
1.03
2.01
3.00
1.04

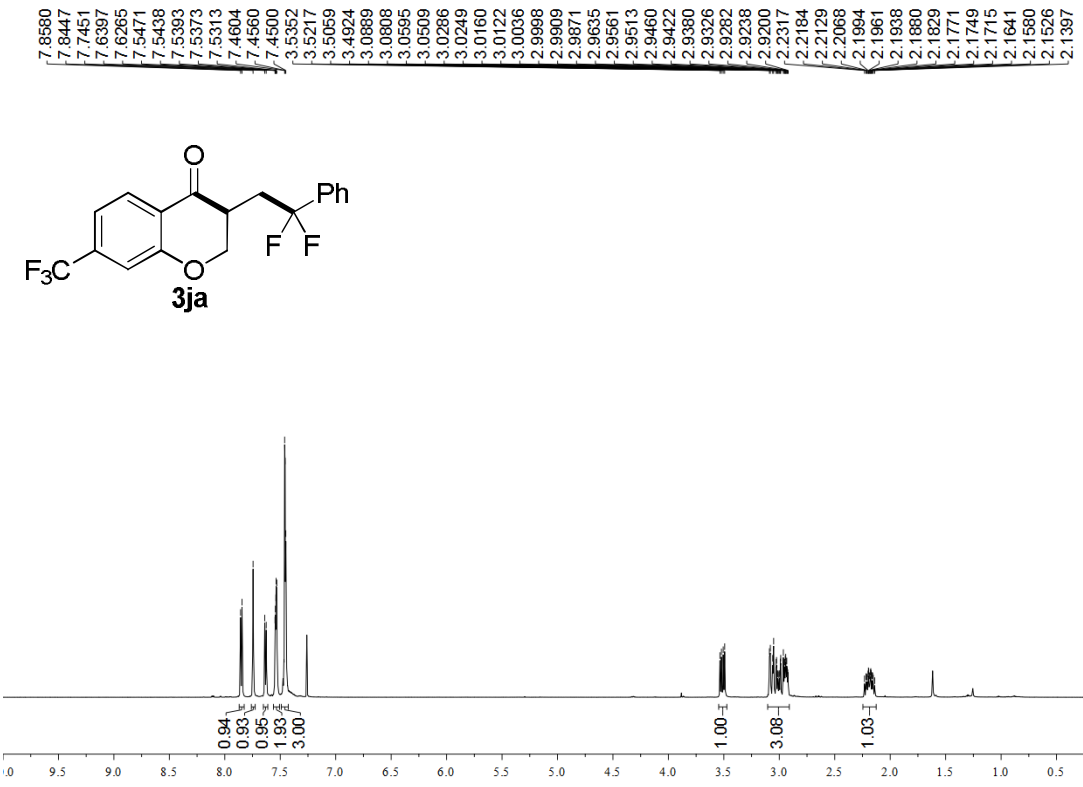

191.7571
161.6269
147.6296
136.7362
136.5614
136.3873
130.0748
130.0654
128.6224
127.3706
124.8860
124.8469
124.8044
124.3307
122.8798
122.7247
122.7202
121.1122
118.0627
117.7255
70.5167
70.5055
70.4947
70.4814
41.1131
41.1055
41.0930
34.5000
34.3170
34.1285
21.9016
O
Ph
F
F
O
3ha

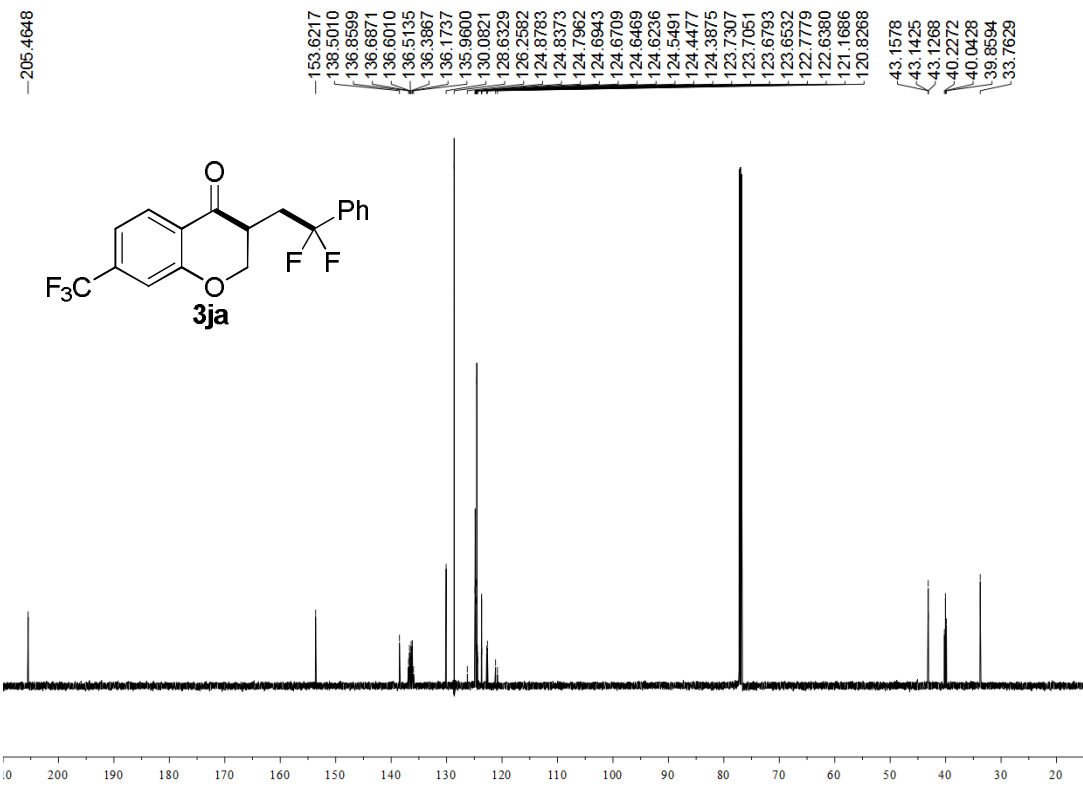

-89.7519
-90.1941
-97.3268
-97.7654
O
Ph
F F
O
3ha

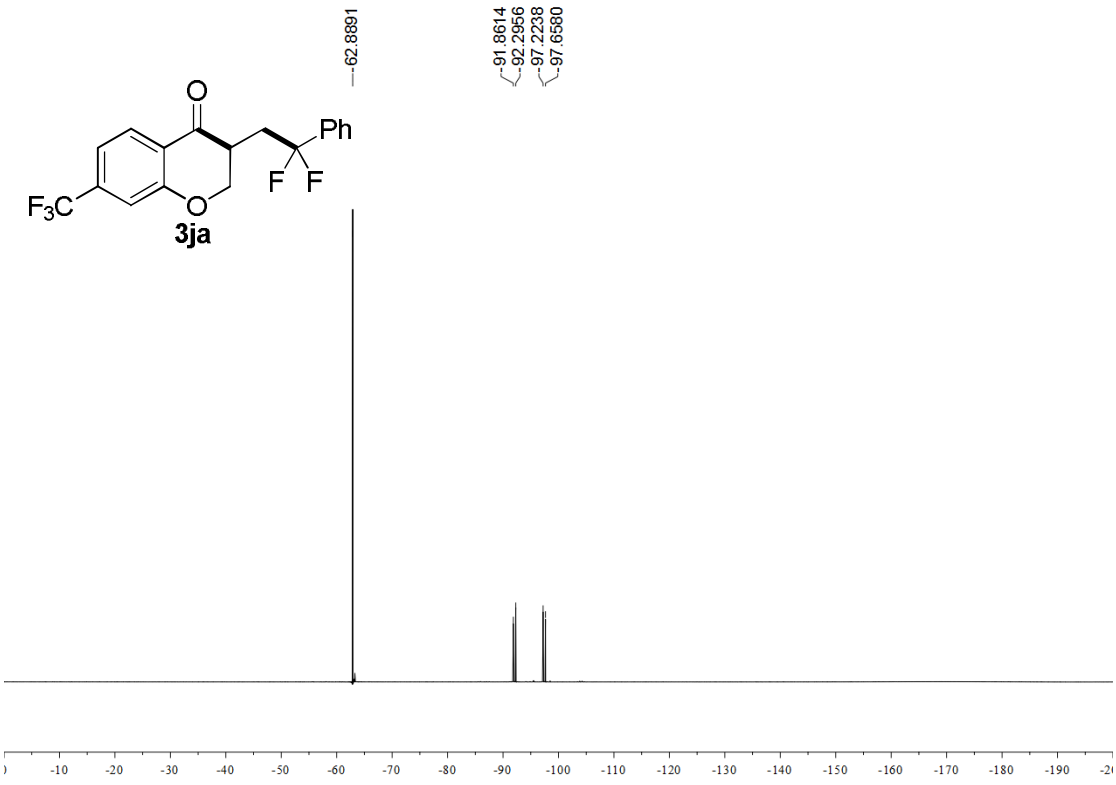

7.8196
7.8049
7.5420
7.5389
7.5349
7.5325
7.5261
7.4460
7.4410
7.4354
6.5841
6.5802
6.5694
6.5654
6.3988
6.3948
4.7421
4.7335
4.7232
4.7146
4.2668
4.2474
4.2284
3.8290
3.1407
3.1185
3.1145
3.1036
3.0995
3.0924
3.0883
3.0772
3.0730
3.0683
3.0638
3.0595
3.0566
3.0517
3.0485
3.0444
3.0402
3.0373
3.0329
3.0285
3.0244
2.1084
2.0945
2.0927
2.0824
2.0790
2.0700
2.0668
2.0576
2.0541
2.0450
2.0415
2.0316
2.0293
O
Ph
F F
MeO
O
3ia
0.92
1.92
2.87
0.96
0.94
1.00
1.01
2.96
2.02
1.04

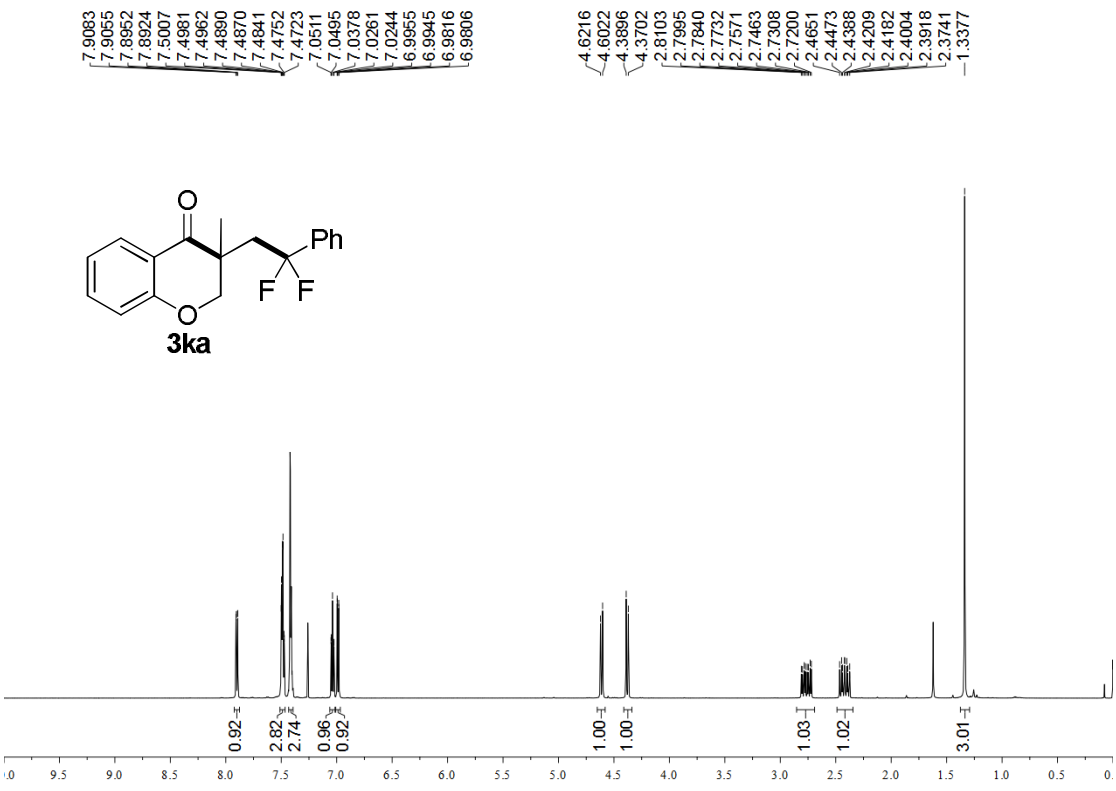

190.6787
166.0271
163.5810
136.7493
136.5754
136.3998
130.0694
130.0597
129.2266
128.6194
124.8894
124.8511
124.8083
124.3759
122.7705
122.7647
121.1589
114.1916
110.1321
100.5421
70.8269
70.8152
70.8037
70.7913
55.6248
40.7999
34.5419
34.3535
34.1715
O
Ph
F F
MeO
O
3ia

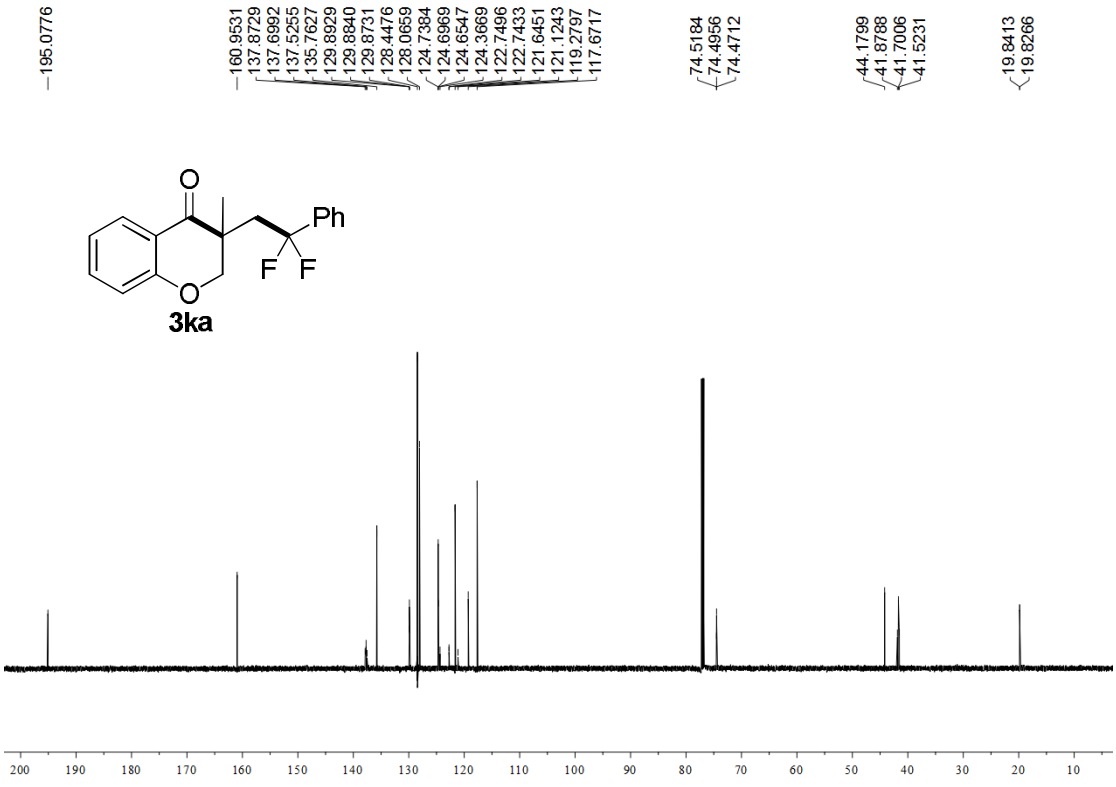

89.6085
90.0433
97.4145
97.8492
O
Ph
F F
MeO
O
3ia

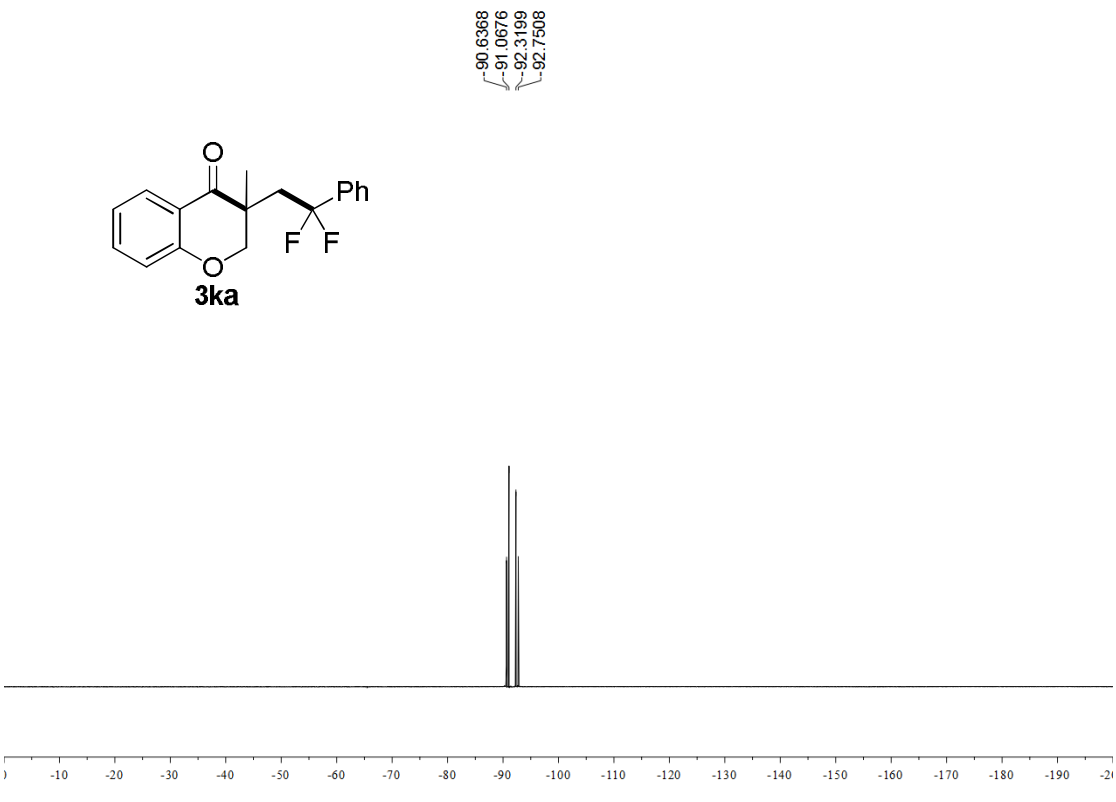

7.8580
7.8447
7.7451
7.6397
7.6265
7.5471
7.5438
7.5393
7.5373
7.5313
7.4604
7.4560
7.4500
3.5352
3.5217
3.5059
3.4924
3.0889
3.0808
3.0595
3.0509
3.0286
3.0249
3.0160
3.0122
3.0036
2.9998
2.9909
2.9871
2.9635
2.9561
2.9513
2.9460
2.9422
2.9380
2.9326
2.9282
2.9238
2.9200
2.2317
2.2184
2.2129
2.2068
2.1994
2.1961
2.1938
2.1880
2.1829
2.1771
2.1749
2.1715
2.1641
2.1580
2.1526
2.1397
O
Ph
F
F
F3C
O
3ja
0.94
0.93
0.95
1.93
3.00
1.00
3.08
1.03

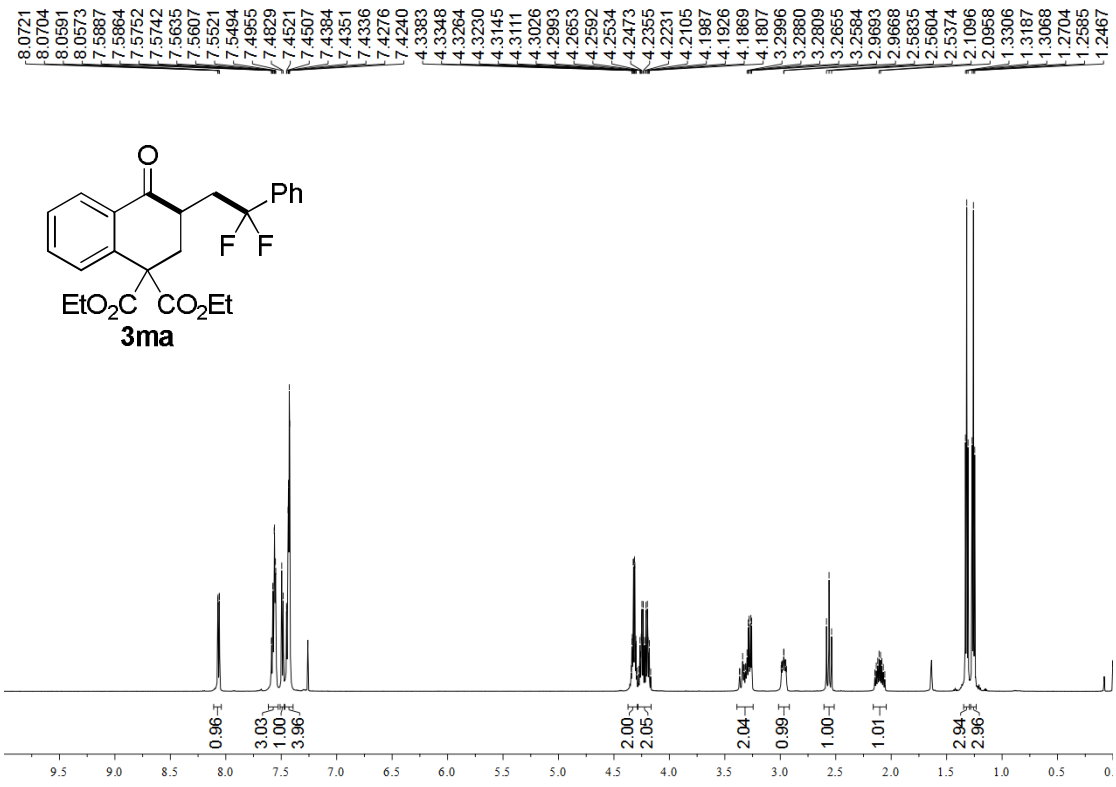

205.4648
153.6217
138.5010
136.8599
136.6871
136.6010
136.5135
136.3867
136.1737
135.9600
130.0821
128.6329
126.2582
124.8783
124.8373
124.7962
124.6943
124.6709
124.6469
124.6236
124.5491
124.4477
124.3875
123.7307
123.7051
123.6793
123.6532
122.7779
122.6380
121.1686
120.8268
43.1578
43.1425
43.1268
40.2272
40.0428
39.8594
33.7629
O
Ph
F
F
F3C
O
3ja

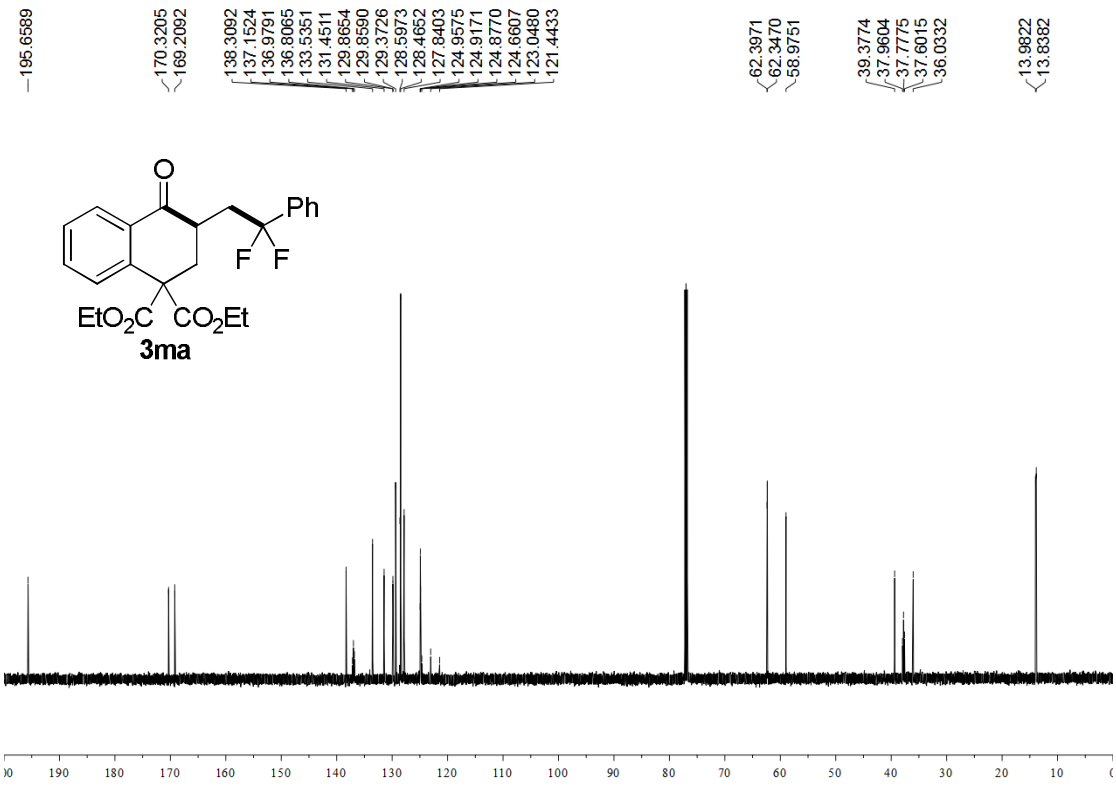

-62.8891
-91.8614
-92.2956
-97.2238
-97.6580
O
Ph
F F
F3C
O
3ja

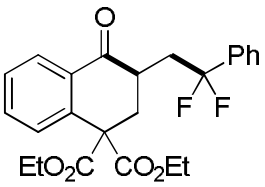

7.9083
7.9055
7.8952
7.8924
7.5007
7.4981
7.4962
7.4890
7.4870
7.4841
7.4752
7.4723
7.0511
7.0495
7.0378
7.0261
7.0244
6.9955
6.9945
6.9816
6.9806
4.6216
4.6022
4.3896
4.3702
2.8103
2.7995
2.7840
2.7732
2.7571
2.7463
2.7308
2.7200
2.4651
2.4473
2.4388
2.4209
2.4182
2.4004
2.3918
2.3741
1.3377
O
Ph
F F
O
3ka
0.92
2.82
2.74
0.96
0.92
1.00
1.00
1.03
1.02
3.01


195.0776
160.9531
137.8729
137.6992
137.5255
135.7627
129.8929
129.8840
129.8731
128.4476
128.0659
124.7384
124.6969
124.6547
124.3669
122.7496
122.7433
121.6451
121.1243
119.2797
117.6717
74.5184
74.4956
74.4712
44.1799
41.8788
41.7006
41.5231
19.8413
19.8266
O
Ph
F F
O
3ka
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10


90.6368
91.0676
92.3199
92.7508
O
Ph
F F
O
3ka
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

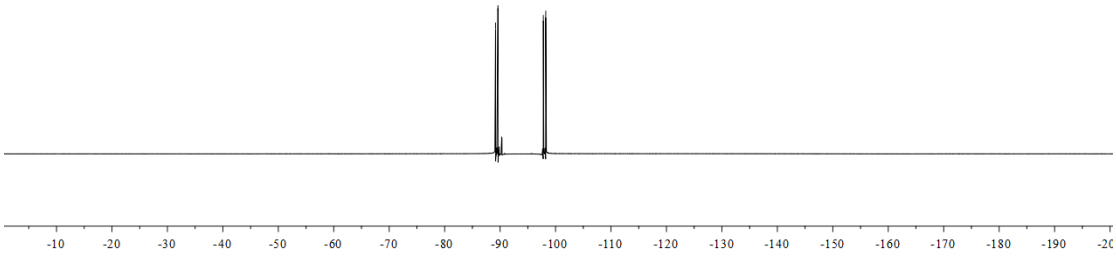

8.0721
8.0704
8.0591
8.0573
7.5887
7.5864
7.5752
7.5742
7.5635
7.5607
7.5521
7.5494
7.4955
7.4829
7.4521
7.4507
7.4384
7.4351
7.4336
7.4276
7.4240
4.3383
4.3348
4.3264
4.3230
4.3145
4.3111
4.3026
4.2993
4.2653
4.2592
4.2534
4.2473
4.2355
4.2231
4.2105
4.1987
4.1926
4.1869
4.1807
3.2996
3.2880
3.2809
3.2655
3.2584
2.9693
2.9668
2.5835
2.5604
2.5374
2.1096
2.0958
1.3306
1.3187
1.3068
1.2704
1.2585
1.2467
O
Ph
F F
EtO2C CO2Et
3ma
0.96
3.03
1.00
3.96
2.00
2.05
2.04
0.99
1.00
1.01
2.94
2.96

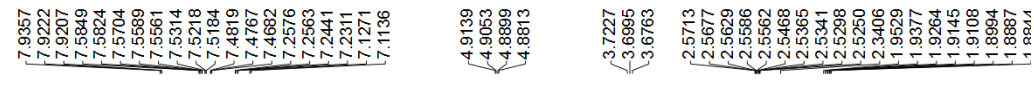

195.6589
170.3205
169.2092
138.3092
137.1524
136.9791
136.8065
133.5351
131.4511
129.8654
129.8590
129.3726
128.5973
128.4652
127.8403
124.9575
124.9171
124.8770
124.6607
123.0480
121.4433
62.3971
62.3470
58.9751
39.3774
37.9604
37.7775
37.6015
36.0332
13.9822
13.8382
O
Ph
F F
EtO2C CO2Et
3ma

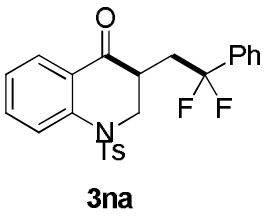

O
Ph
F F
EtO2C CO2Et

3ma

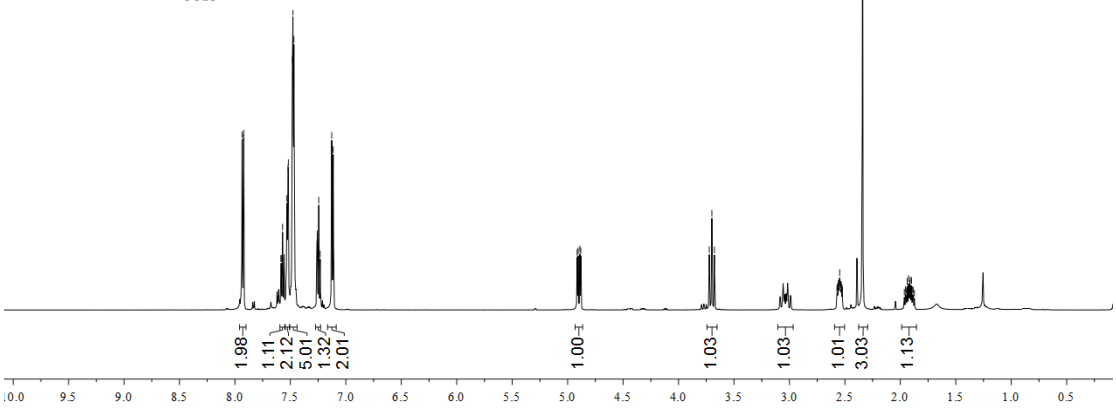

-89.1770
-89.6125
-97.7952
-98.2306

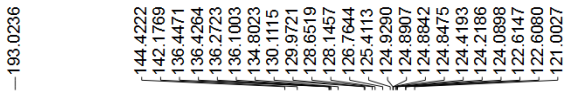

-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

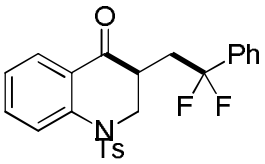

7.9357
7.9222
7.9207
7.5849
7.5824
7.5704
7.5589
7.5561
7.5314
7.5218
7.5184
7.4819
7.4767
7.4682
7.2576
7.2563
7.2441
7.2311
7.1271
7.1136
4.9139
4.9053
4.8899
4.8813
3.7227
3.6995
3.6763
2.5713
2.5677
2.5629
2.5586
2.5562
2.5468
2.5365
2.5341
2.5298
2.5250
2.3406
1.9529
1.9377
1.9264
1.9145
1.9108
1.8994
1.8887


O
Ph
F F
N
Ts

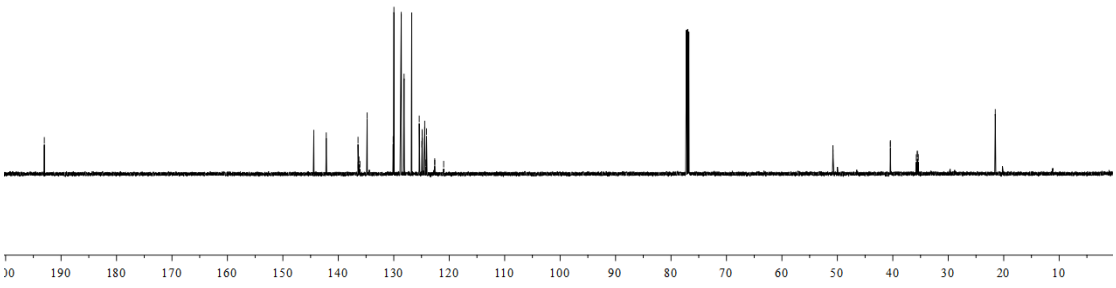
3na


1.98
1.11
2.12
5.01
1.32
2.01
1.00
1.03
1.03
1.01
3.03
1.13
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

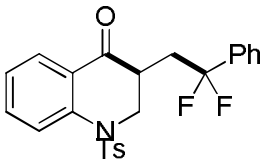

193.0236
144.4222
142.1769
136.4471
136.4264
136.2723
136.1003
134.8023
130.1115
129.9721
128.6519
128.1457
126.7644
125.4113
124.9290
124.8907
124.8842
124.8475
124.4193
124.2186
124.0898
122.6147
122.6080
121.0027
50.8161
50.8013
50.7966
50.7814
40.4486
40.4374
35.7673
35.5880
35.5814
35.4009
21.5252


O
Ph
F
F
N
Ts

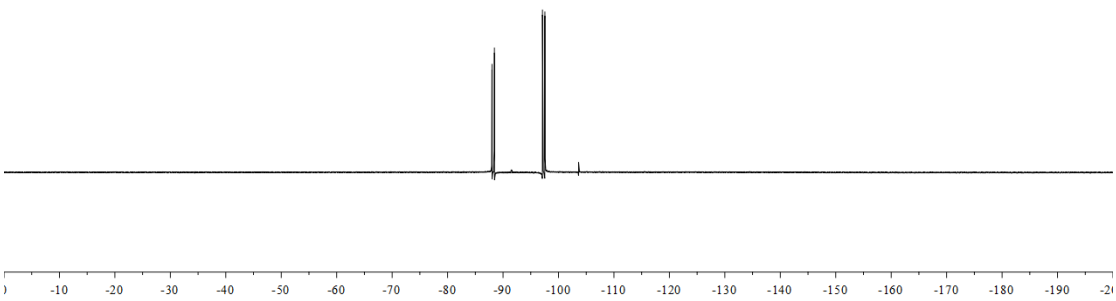
3na

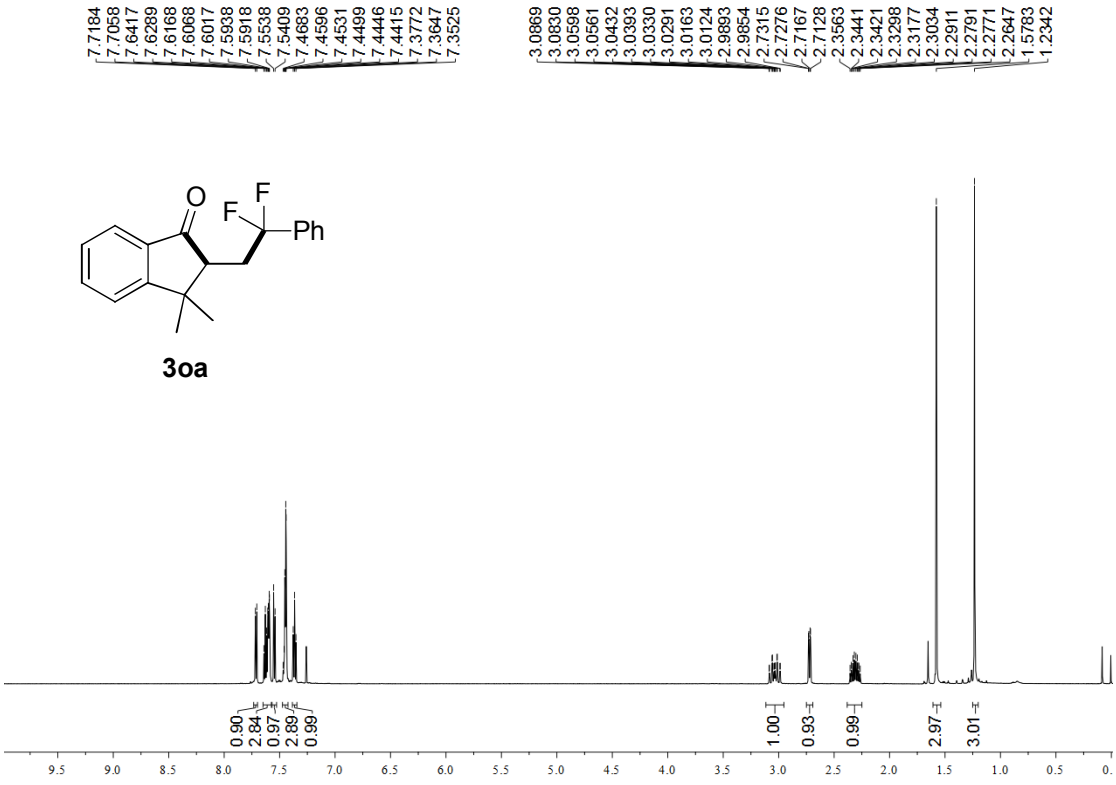

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

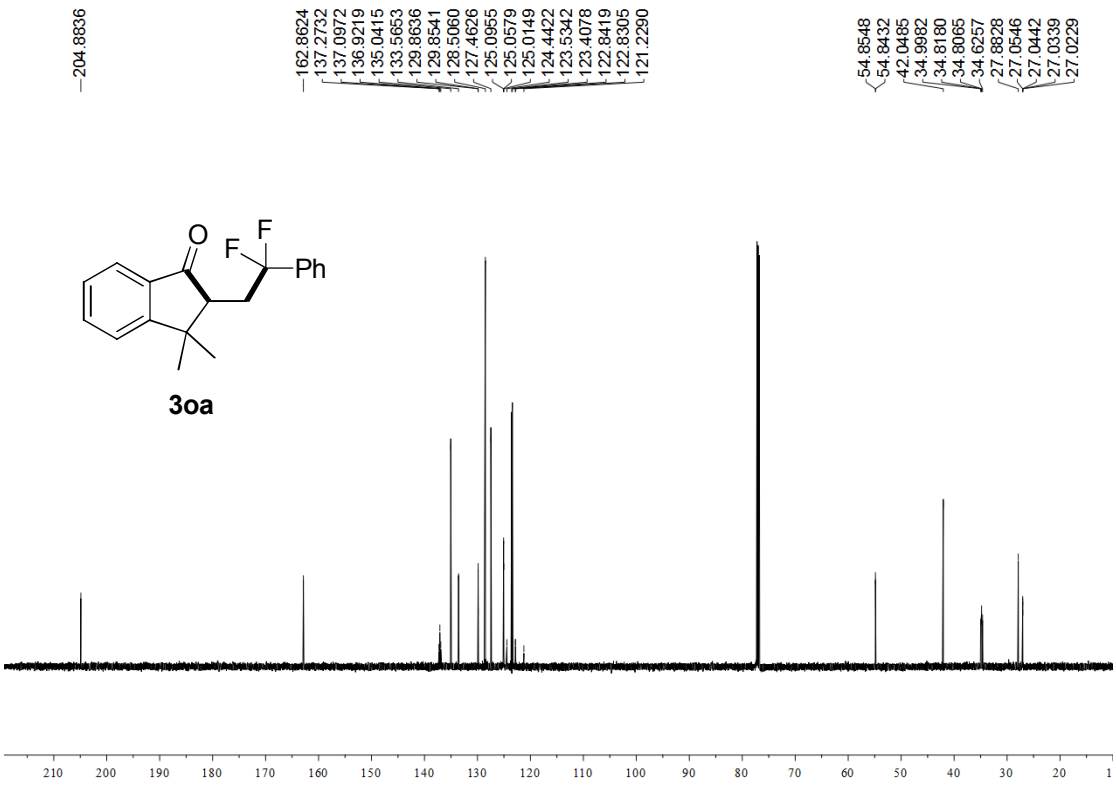

-88.0037
-88.4416
-97.1085
-97.5464

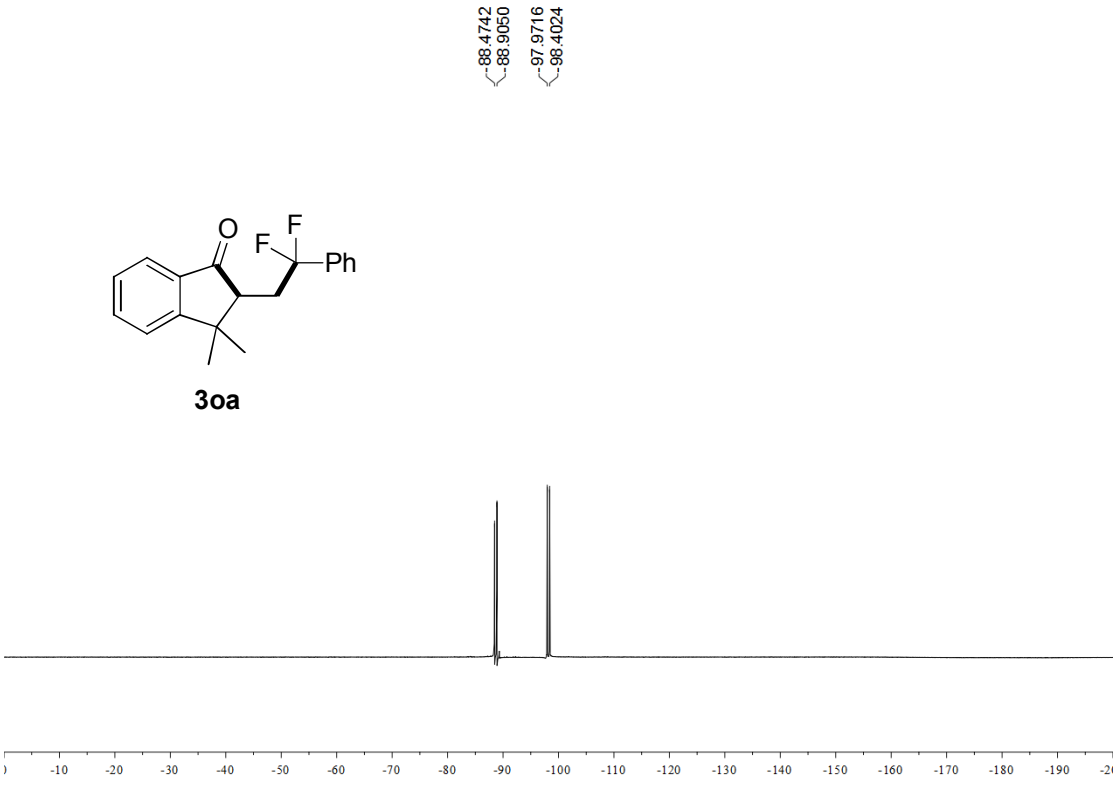

O
Ph
F
F
N
Ts

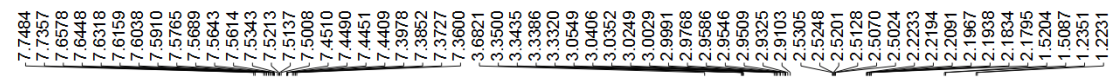
3na

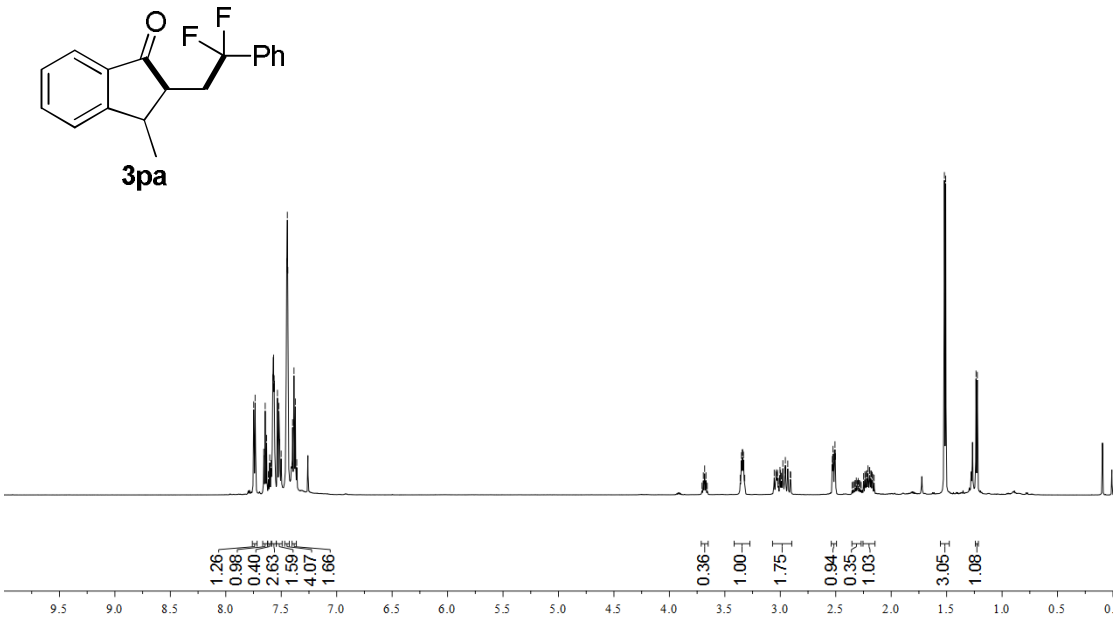

-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

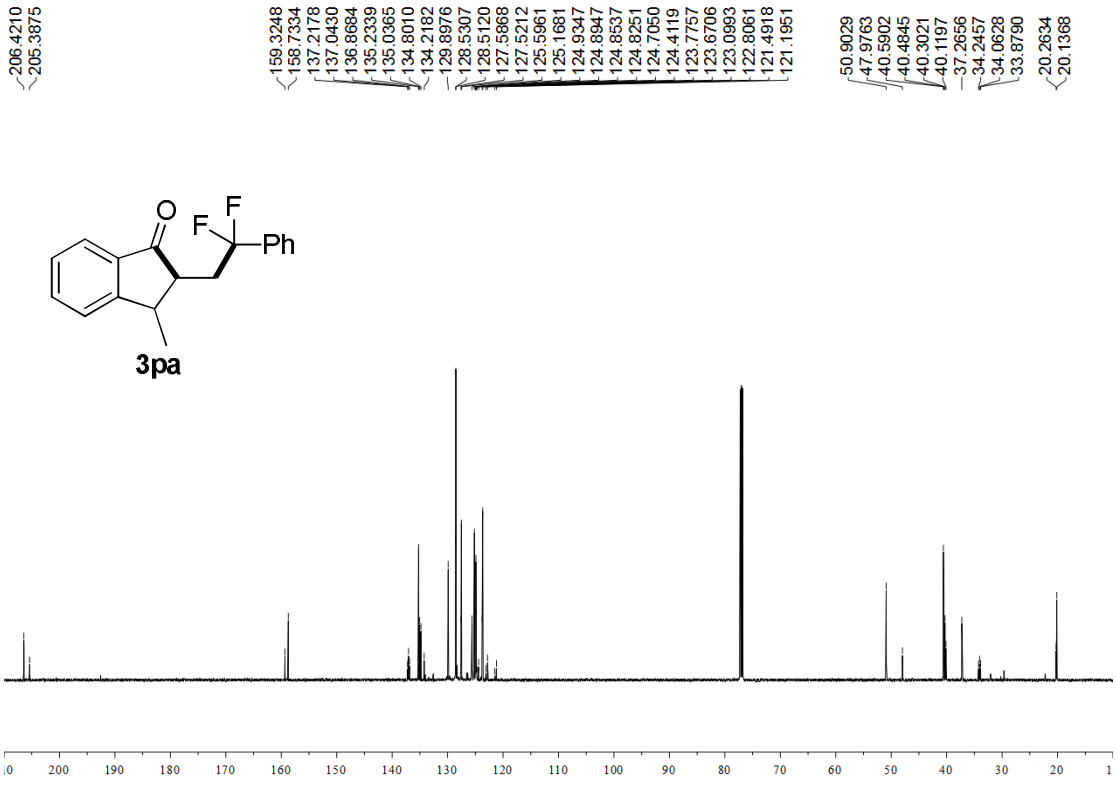

7.7184
7.7058
7.6417
7.6289
7.6168
7.6068
7.6017
7.5938
7.5918
7.5538
7.5409
7.4683
7.4596
7.4531
7.4499
7.4446
7.4415
7.3772
7.3647
7.3525
3.0869
3.0830
3.0598
3.0561
3.0432
3.0393
3.0330
3.0291
3.0163
3.0124
2.9893
2.9854
2.7315
2.7276
2.7167
2.7128
2.3563
2.3441
2.3421
2.3298
2.3177
2.3034
2.2911
2.2791
2.2771
2.2647
1.5783
1.2342
O
F
F
Ph
3oa
0.90
2.84
0.97
2.89
0.99
1.00
0.93
0.99
2.97
3.01

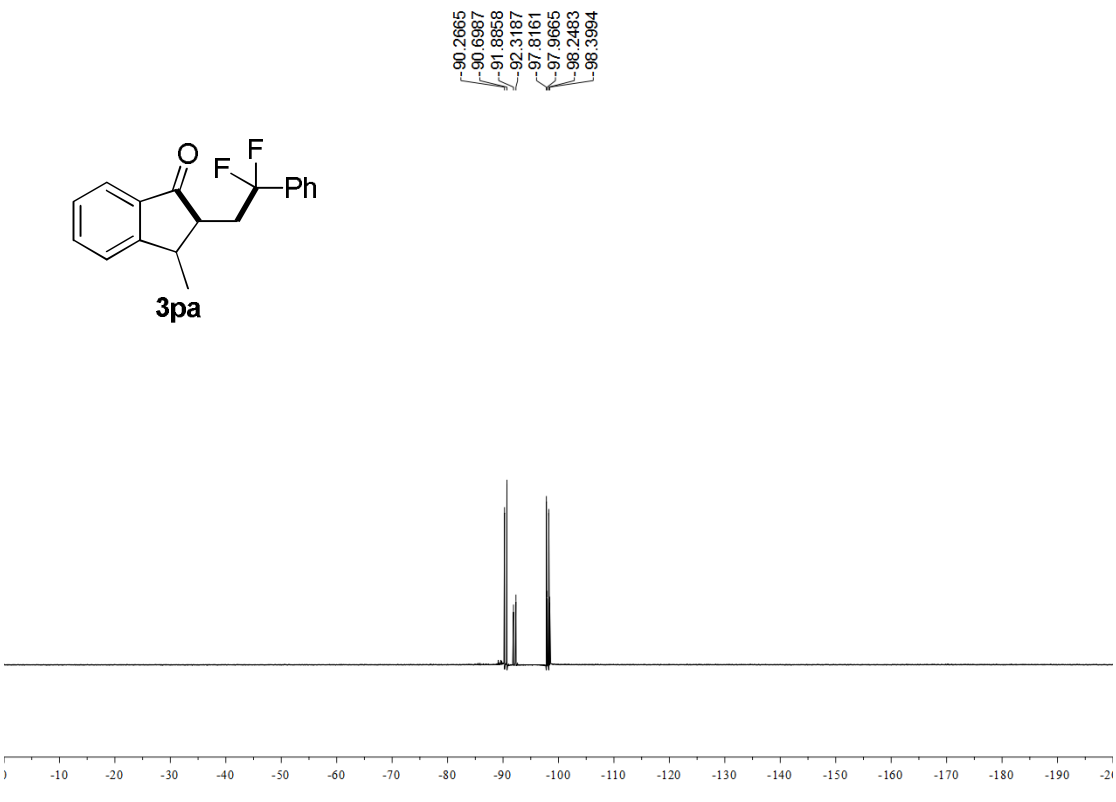

204.8836
162.8624
137.2732
137.0972
136.9219
135.0415
133.5653
129.8636
129.8541
128.5060
127.4626
125.0955
125.0579
125.0149
124.4422
123.5342
123.4078
122.8419
122.8305
121.2290
54.8548
54.8432
42.0485
34.9982
34.8180
34.8065
34.6257
27.8828
27.0546
27.0442
27.0339
27.0229
O
F
F
Ph
3oa

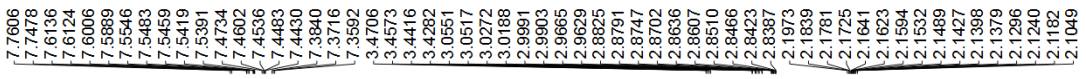

-88.4742
-88.9050
-97.9716
-98.4024
O
F
F
Ph
3oa

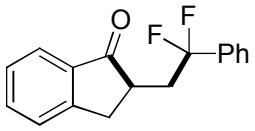

7.7484
7.7357
7.6578
7.6448
7.6318
7.6159
7.6038
7.5910
7.5765
7.5689
7.5643
7.5614
7.5343
7.5213
7.5137
7.5008
7.4510
7.4490
7.4451
7.4409
7.3978
7.3852
7.3727
7.3600
3.6821
3.3500
3.3435
3.3386
3.3320
3.0549
3.0406
3.0352
3.0249
3.0029
2.9991
2.9768
2.9586
2.9546
2.9509
2.9325
2.9103
2.5305
2.5248
2.5201
2.5128
2.5070
2.5024
2.2233
2.2194
2.2091
2.1967
2.1938
2.1834
2.1795
1.5204
1.5087
1.2351
1.2231


O
F
F
Ph
3pa
1.26
0.98
0.40
2.63
1.59
4.07
1.66
0.36
1.00
1.75
0.94
0.35
1.03
3.05
1.08

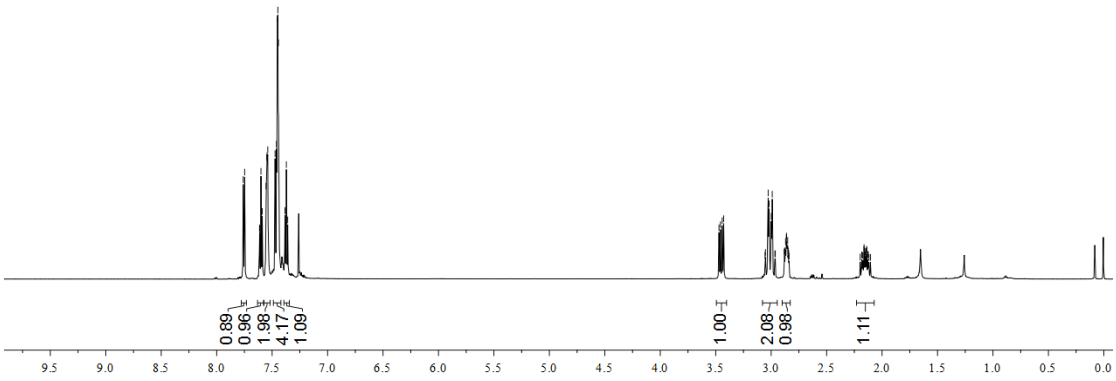

206.4210
205.3875
159.3248
158.7334
137.2178
137.0430
136.8684
135.2339
135.0365
134.8010
134.2182
129.8976
128.5307
128.5120
127.5868
127.5212
125.5961
125.1681
124.9347
124.8947
124.8537
124.8251
124.7050
124.4119
123.7757
123.6706
123.0993
122.8061
121.4918
121.1951
50.9029
47.9763
40.5902
40.4845
40.3021
40.1197
37.2656
34.2457
34.0628
33.8790
20.2634
20.1368
O
F
F
Ph
3pa

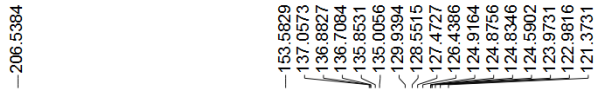

-90.2665
-90.6987
-91.8858
-92.3187
-97.8161
-97.9665
-98.2483
-98.3994
O
F
F
Ph
3pa

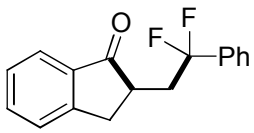

7.7606
7.7478
7.6136
7.6124
7.6006
7.5889
7.5546
7.5483
7.5459
7.5419
7.5391
7.4734
7.4602
7.4536
7.4483
7.4430
7.3840
7.3716
7.3592
3.4706
3.4573
3.4416
3.4282
3.0551
3.0517
3.0272
3.0188
2.9991
2.9903
2.9665
2.9629
2.8825
2.8791
2.8747
2.8702
2.8636
2.8607
2.8510
2.8466
2.8423
2.8387
2.1973
2.1839
2.1781
2.1725
2.1641
2.1623
2.1594
2.1532
2.1489
2.1427
2.1398
2.1379
2.1296
2.1240
2.1182
2.1049


O
F
F
Ph

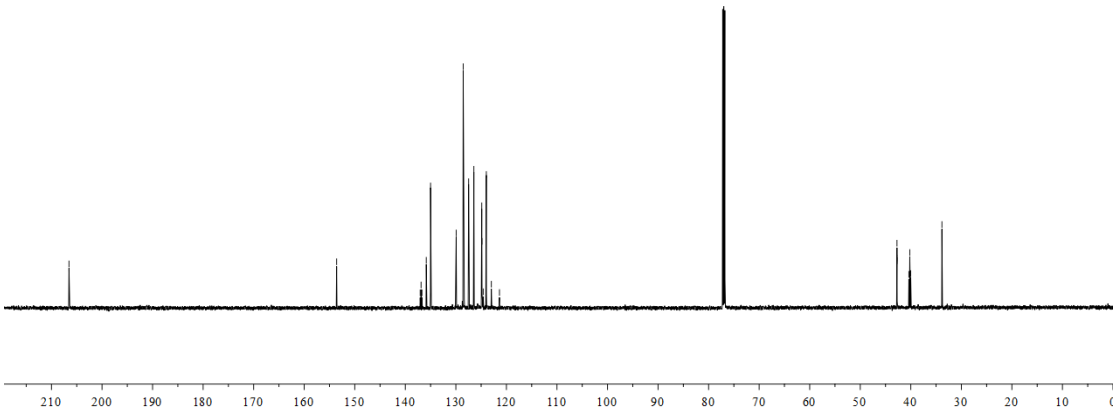
3qa

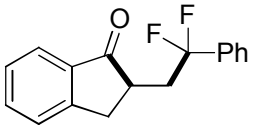

0.89
0.96
1.98
4.17
1.09
1.00
2.08
0.98
1.11
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


206.5384
153.5829
137.0573
136.8827
136.7084
135.8531
135.0056
129.9394
128.5515
127.4727
126.4386
124.9164
124.8756
124.8346
124.5902
123.9731
122.9816
121.3731
42.7473
42.7352
42.7205
40.3828
40.2001
40.0173
33.8434


O
F
F
Ph

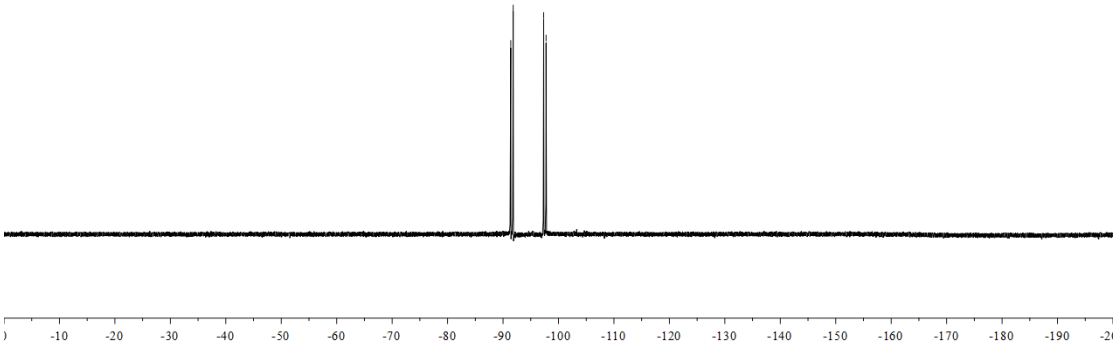
3qa

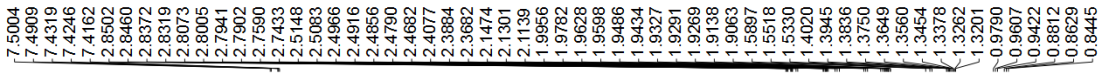

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

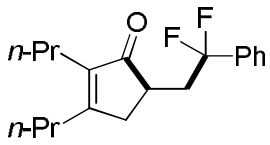

O
F
F
Ph

3qa

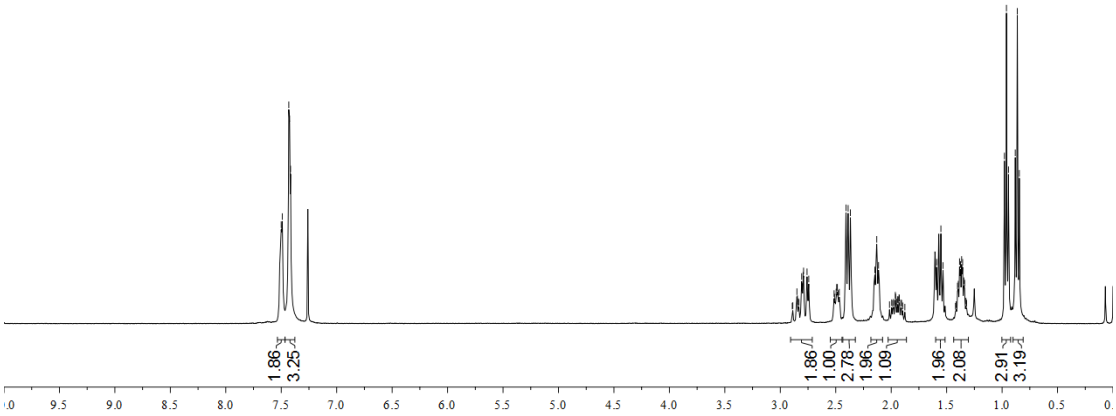

-91.3963
-91.8302
-97.3251
-97.7592

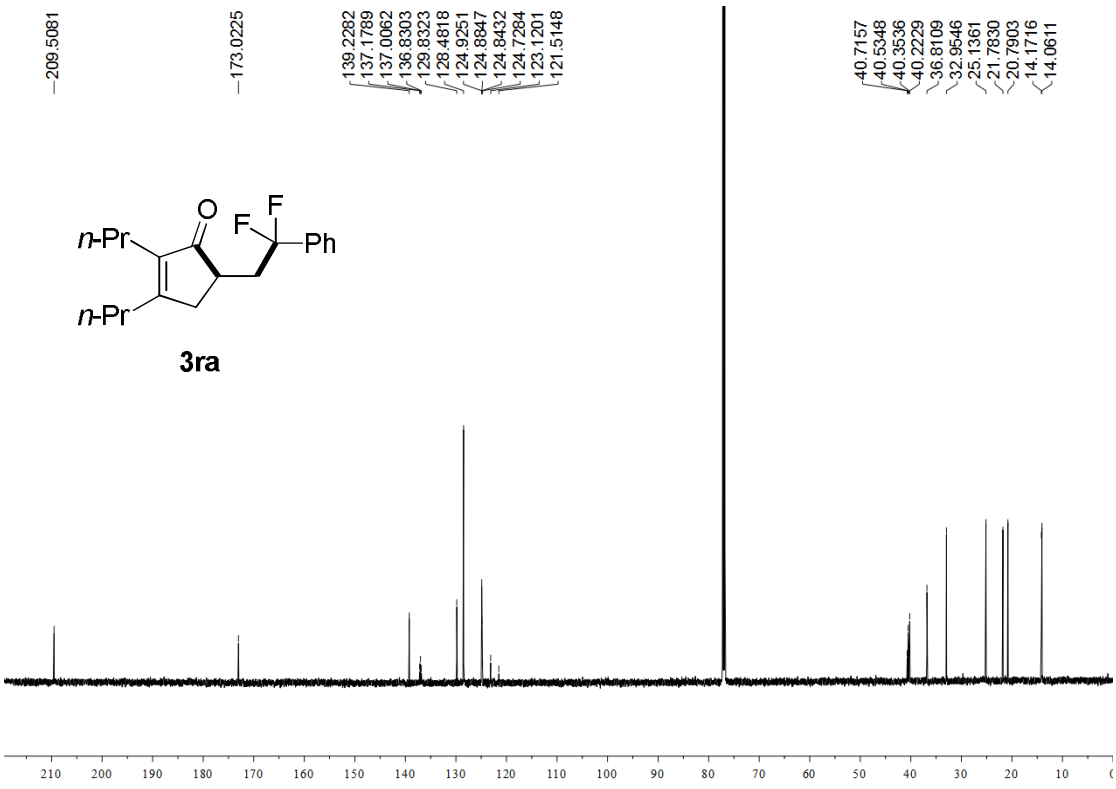

-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

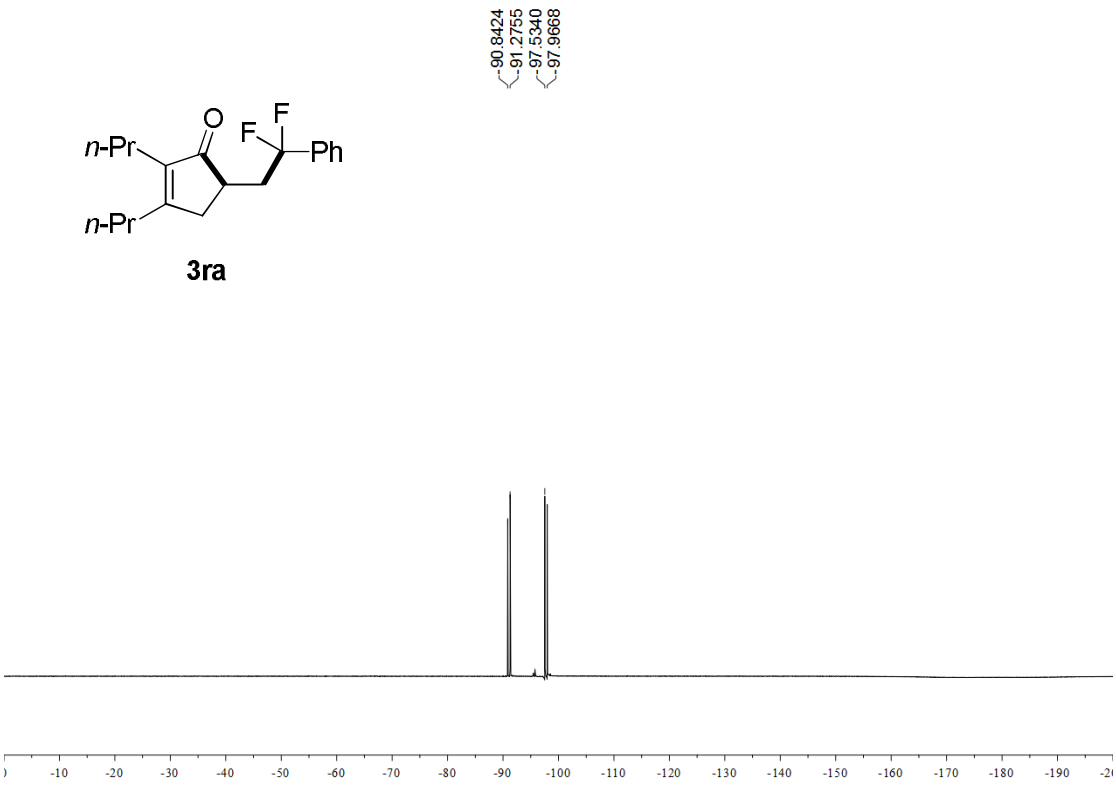

7.5004
7.4909
7.4319
7.4246
7.4162
2.8502
2.8460
2.8372
2.8319
2.8073
2.8005
2.7941
2.7902
2.7590
2.7433
2.5148
2.5083
2.4966
2.4916
2.4856
2.4790
2.4682
2.4077
2.3884
2.3682
2.1474
2.1301
2.1139
1.9956
1.9782
1.9628
1.9598
1.9486
1.9434
1.9327
1.9291
1.9269
1.9138
1.9063
1.5897
1.5518
1.5330
1.4020
1.3945
1.3836
1.3750
1.3649
1.3560
1.3454
1.3378
1.3262
1.3201
0.9790
0.9607
0.9422
0.8812
0.8629
0.8445

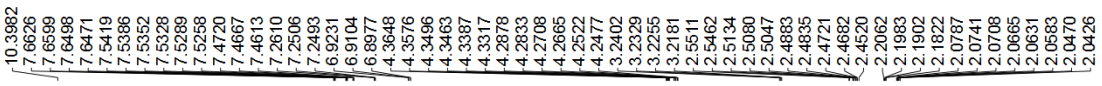

O
F
F
Ph
n-Pr
n-Pr

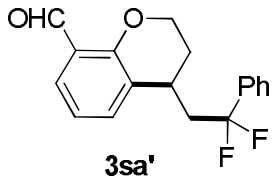
3ra


1.86
3.25
1.86
1.00
2.78
1.96
1.09
1.96
2.08
2.91
3.19
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

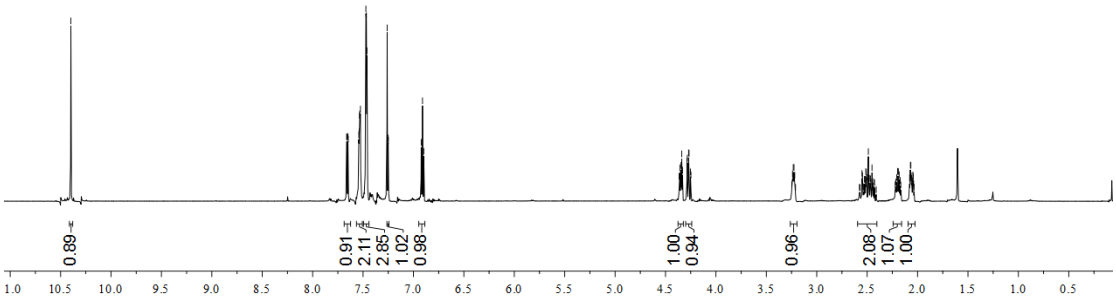

209.5081
173.0225
139.2282
137.1789
137.0062
136.8303
129.8323
128.4818
124.9251
124.8847
124.8432
124.7284
123.1201
121.5148
40.7157
40.5348
40.3536
40.2229
36.8109
32.9546
25.1361
21.7830
20.7903
14.1716
14.0611
O
F
F
Ph
n-Pr
n-Pr
3ra
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

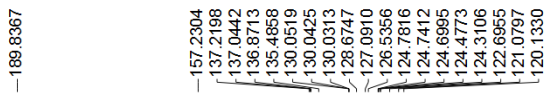

90.8424
91.2755
97.5340
97.9668
O
F
F
Ph
n-Pr
n-Pr
3ra
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

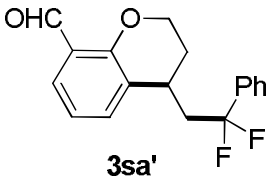

10.3982
7.6626
7.6599
7.6498
7.6471
7.5419
7.5386
7.5352
7.5328
7.5289
7.5258
7.4720
7.4667
7.4613
7.2610
7.2506
7.2493
6.9231
6.9104
6.8977
4.3648
4.3576
4.3496
4.3463
4.3387
4.3317
4.2878
4.2833
4.2708
4.2665
4.2522
4.2477
3.2402
3.2329
3.2255
3.2181
2.5511
2.5462
2.5134
2.5080
2.5047
2.4883
2.4835
2.4721
2.4682
2.4520
2.2062
2.1983
2.1902
2.1822
2.0787
2.0741
2.0708
2.0665
2.0631
2.0583
2.0470
2.0426


OHC
O
Ph
F
F

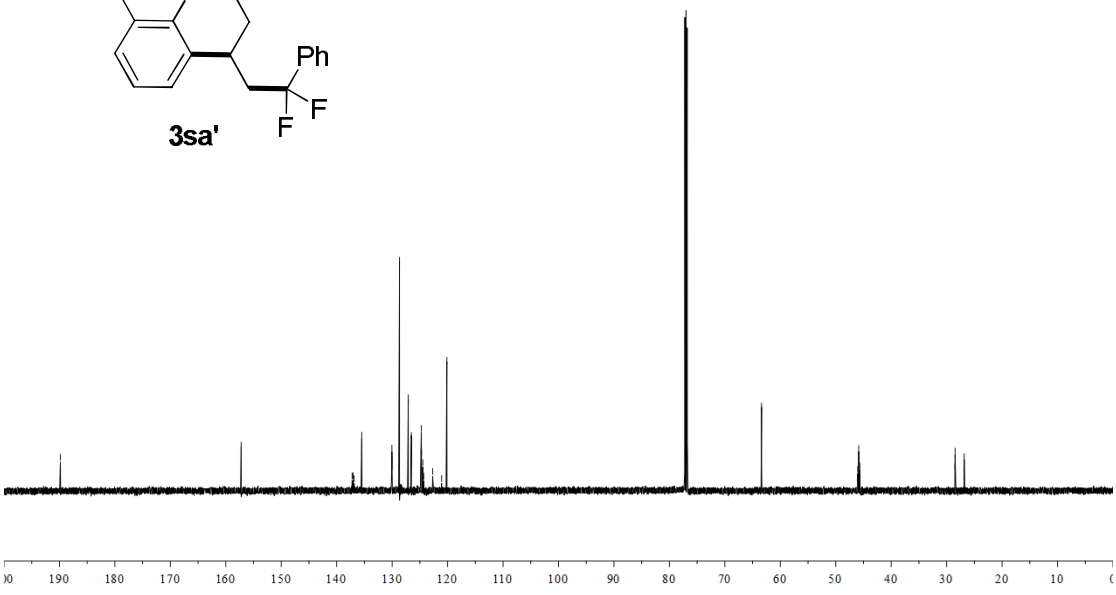
3sa'

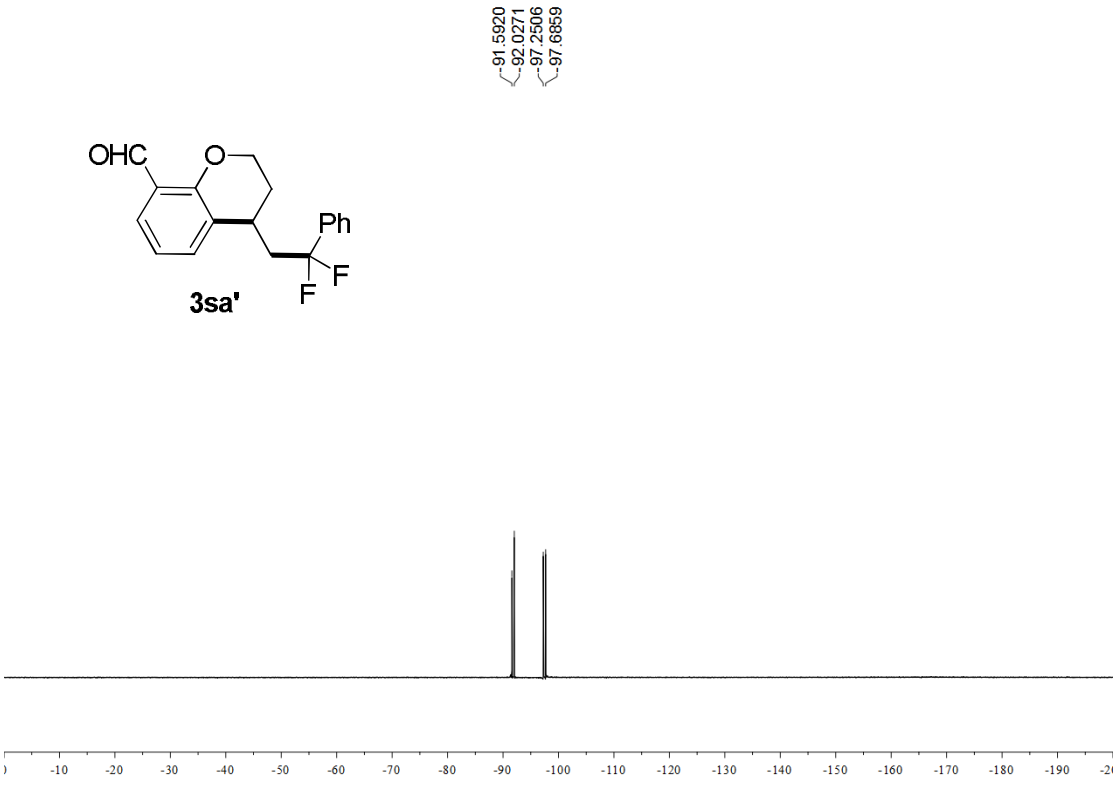

0.89
0.91
2.11
2.85
1.02
0.98
1.00
0.94
0.96
2.08
1.07
1.00
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

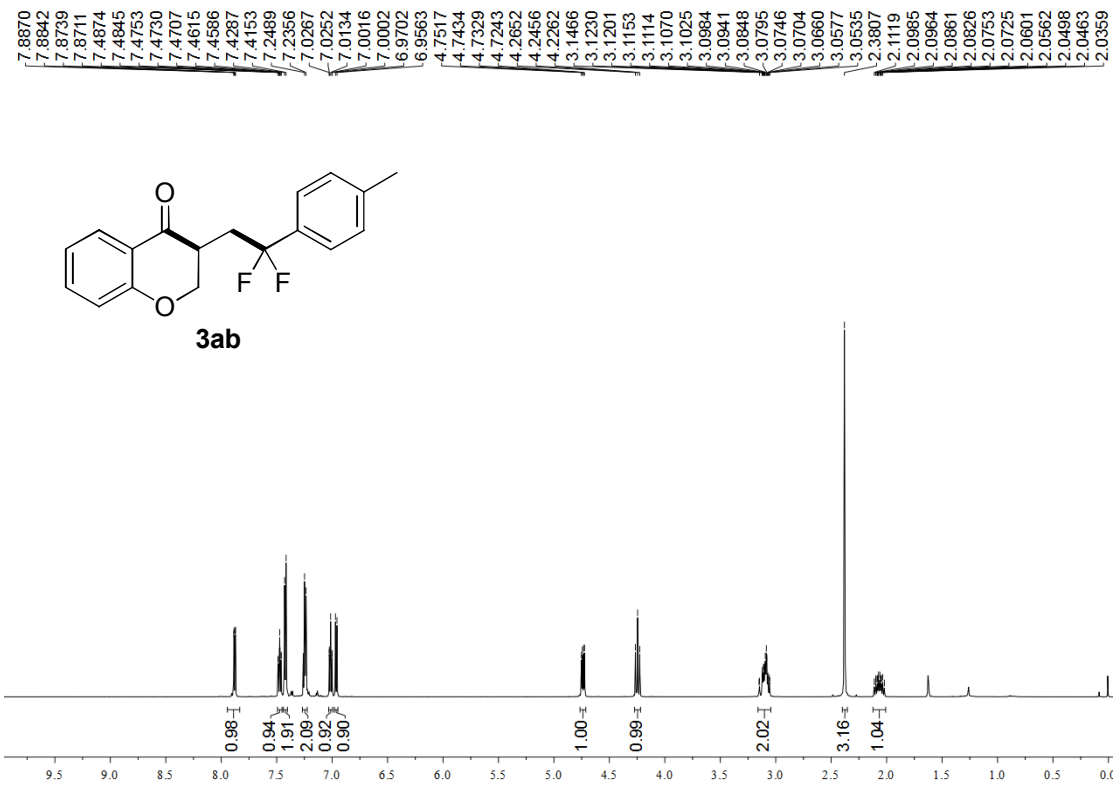

189.8367
157.2304
137.2198
137.0442
136.8713
135.4858
130.0519
130.0425
130.0313
128.6747
127.0910
126.5356
124.7816
124.7412
124.6995
124.4773
124.3106
122.6955
121.0797
120.1330
63.3801
46.0331
45.8566
45.6806
28.4959
28.4779
28.4599
26.8376

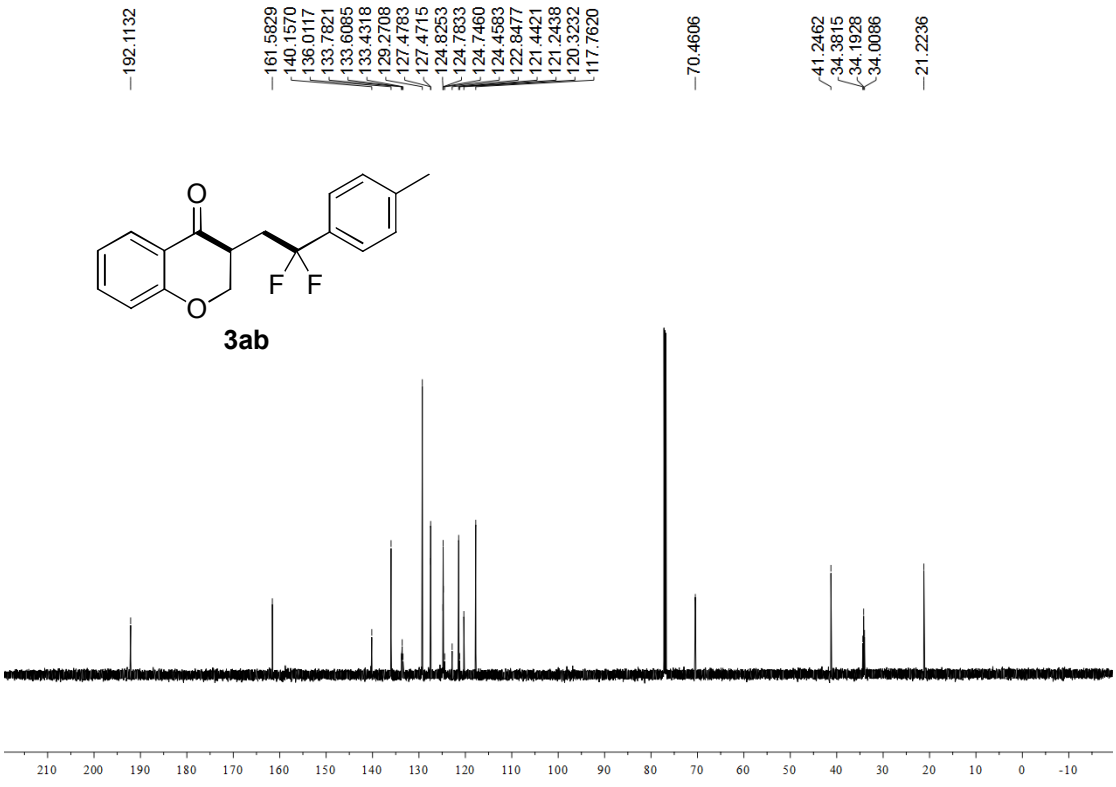

OHC
O
Ph
F
F

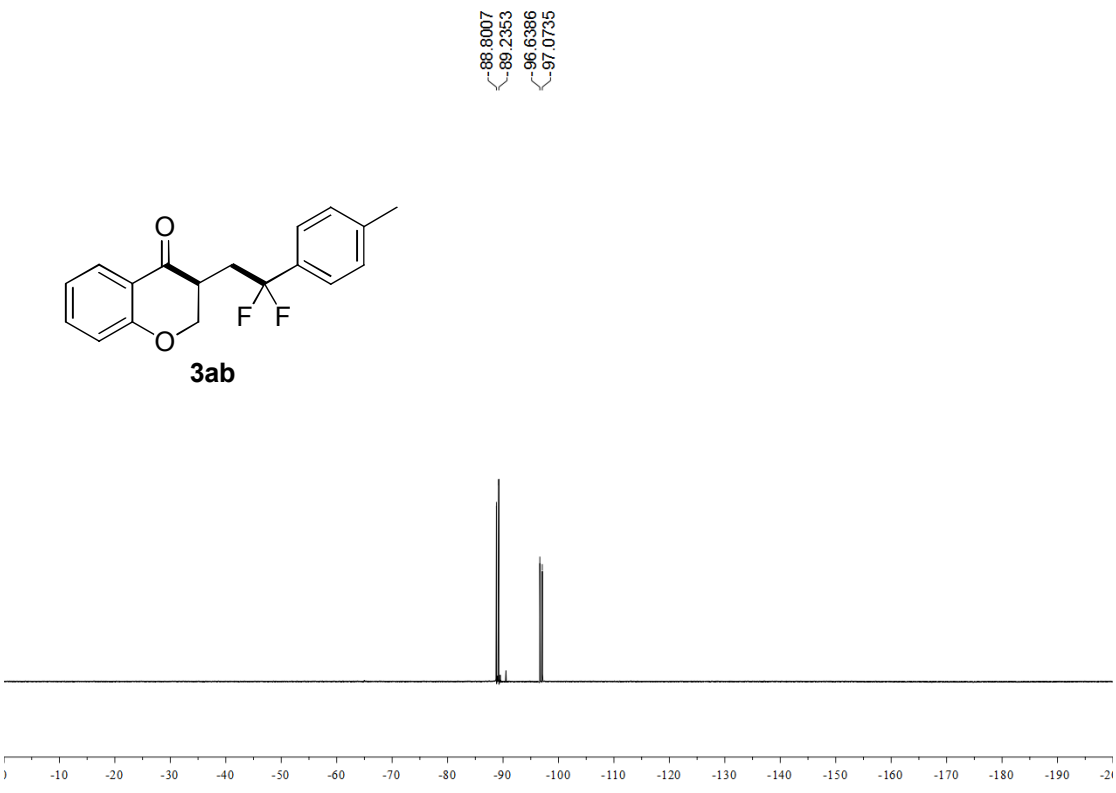
3sa'

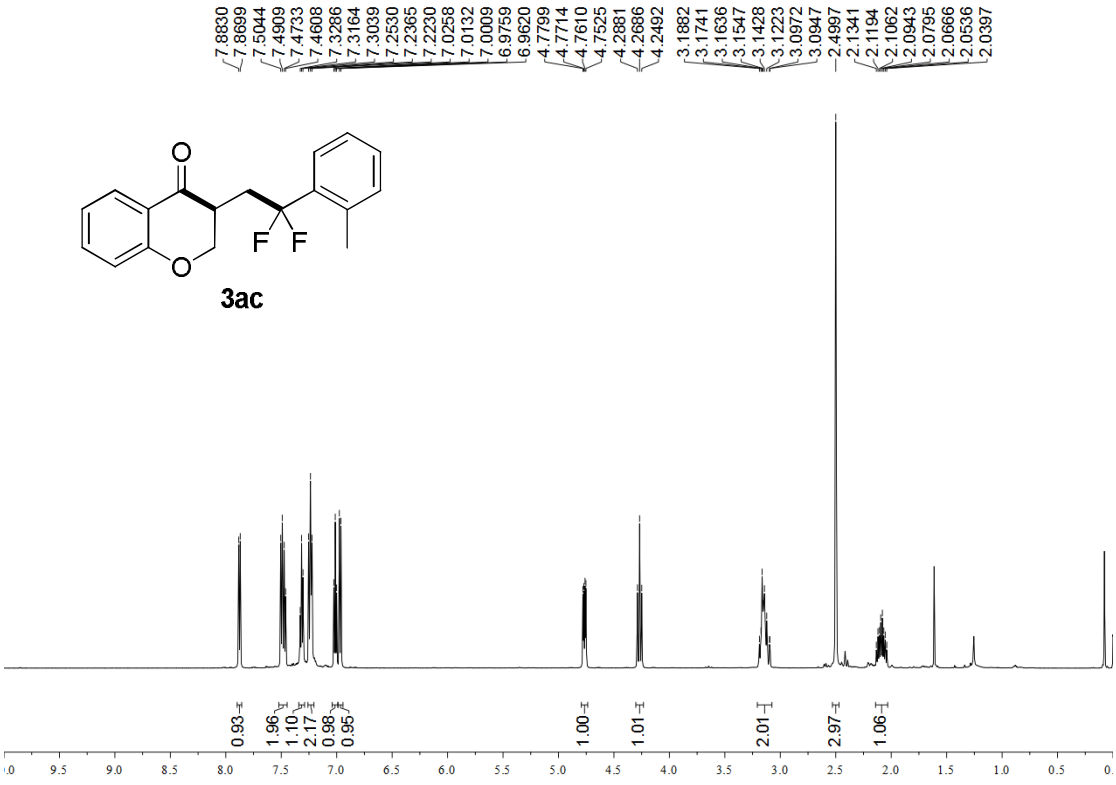

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

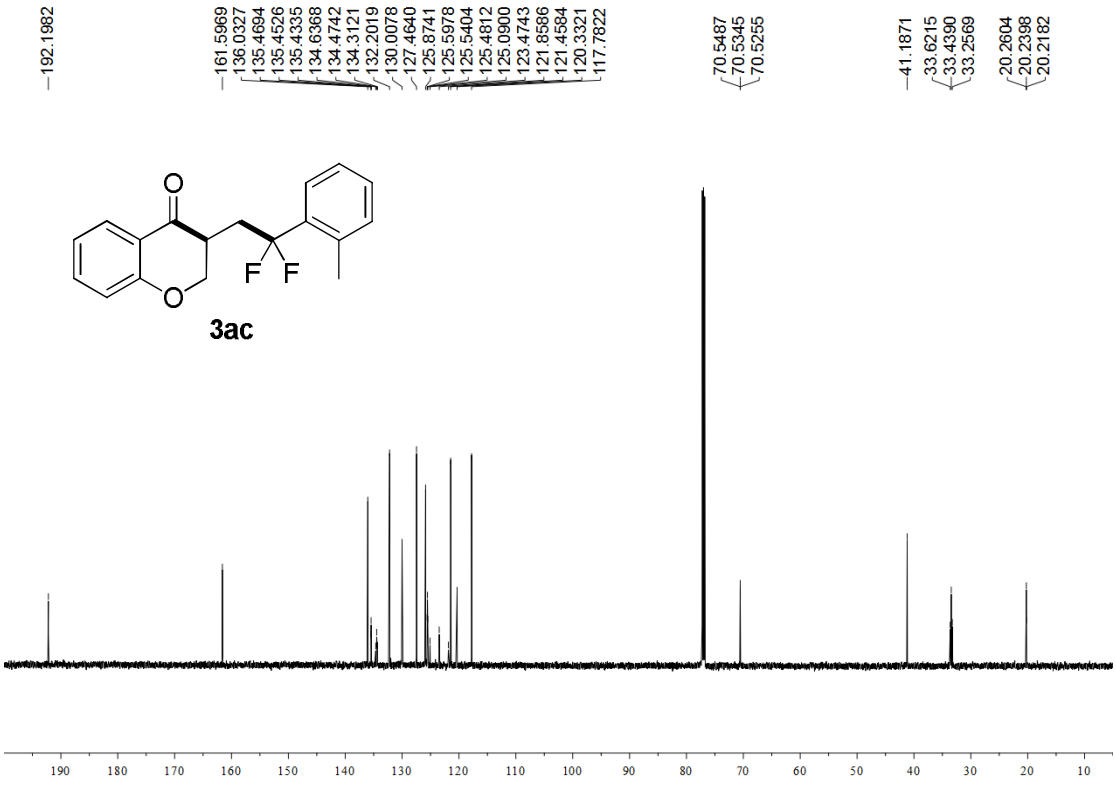

-91.5920
-92.0271
-97.2506
-97.6859
OHC
O
Ph
F
F
3sa'
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190


7.8870
7.8842
7.8739
7.8711
7.4874
7.4845
7.4753
7.4730
7.4707
7.4615
7.4586
7.4287
7.4153
7.2489
7.2356
7.0267
7.0252
7.0134
7.0016
7.0002
6.9702
6.9563
4.7517
4.7434
4.7329
4.7243
4.2652
4.2456
4.2262
3.1466
3.1230
3.1201
3.1153
3.1114
3.1070
3.1025
3.0984
3.0941
3.0848
3.0795
3.0746
3.0704
3.0660
3.0577
3.0535
2.3807
2.1119
2.0985
2.0964
2.0861
2.0826
2.0753
2.0725
2.0601
2.0562
2.0498
2.0463
2.0359
O
O
F
F
3ab
0.98
0.94
1.91
2.09
0.92
0.90
1.00
0.99
2.02
3.16
1.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

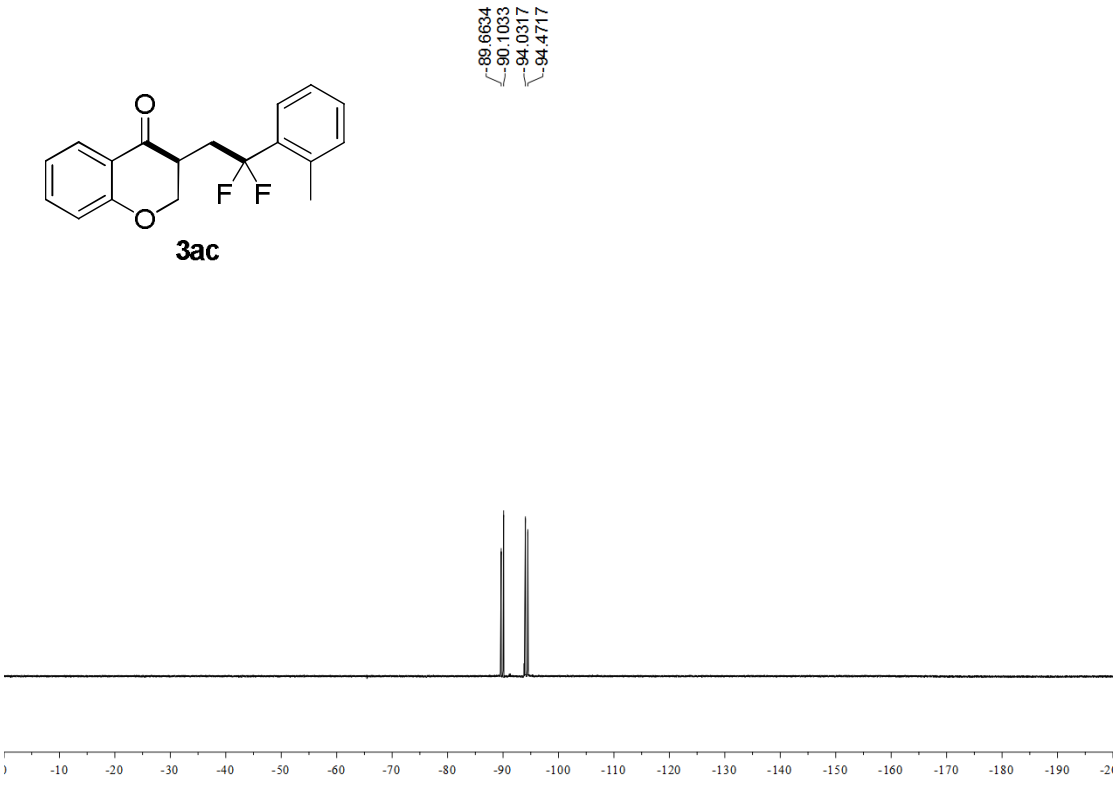

192.1132
161.5829
140.1570
136.0117
133.7821
133.6085
133.4318
129.2708
127.4783
127.4715
124.8253
124.7833
124.7460
124.4583
122.8477
121.4421
121.2438
120.3232
117.7620
70.4606
41.2462
34.3815
34.1928
34.0086
21.2236
O
O
F F
3ab


-88.8007
-89.2353
-96.6386
-97.0735
O
O
F F
3ab

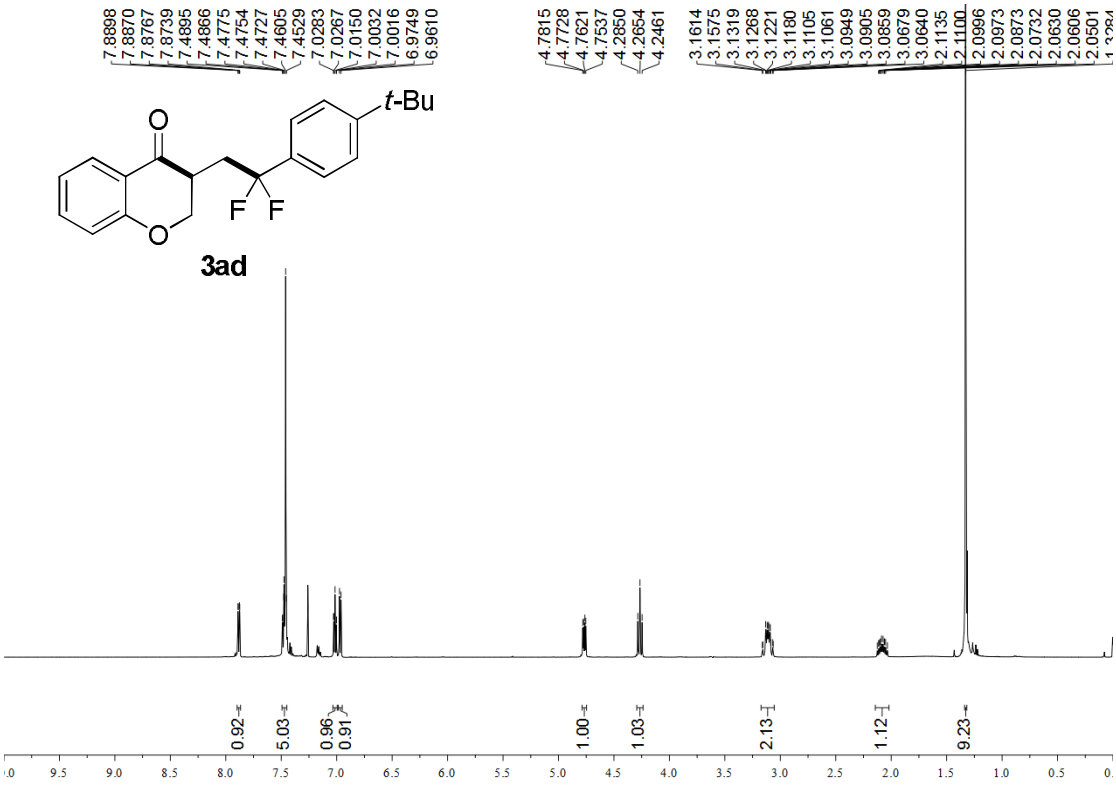

7.8830
7.8699
7.5044
7.4909
7.4733
7.4608
7.3286
7.3164
7.3039
7.2530
7.2365
7.2230
7.0258
7.0132
7.0009
6.9759
6.9620
4.7799
4.7714
4.7610
4.7525
4.2881
4.2686
4.2492
3.1882
3.1741
3.1636
3.1547
3.1428
3.1223
3.0972
3.0947
2.4997
2.1341
2.1194
2.1062
2.0943
2.0795
2.0666
2.0536
2.0397
O
F F
O
3ac
0.93
1.96
1.10
2.17
0.98
0.95
1.00
1.01
2.01
2.97
1.06

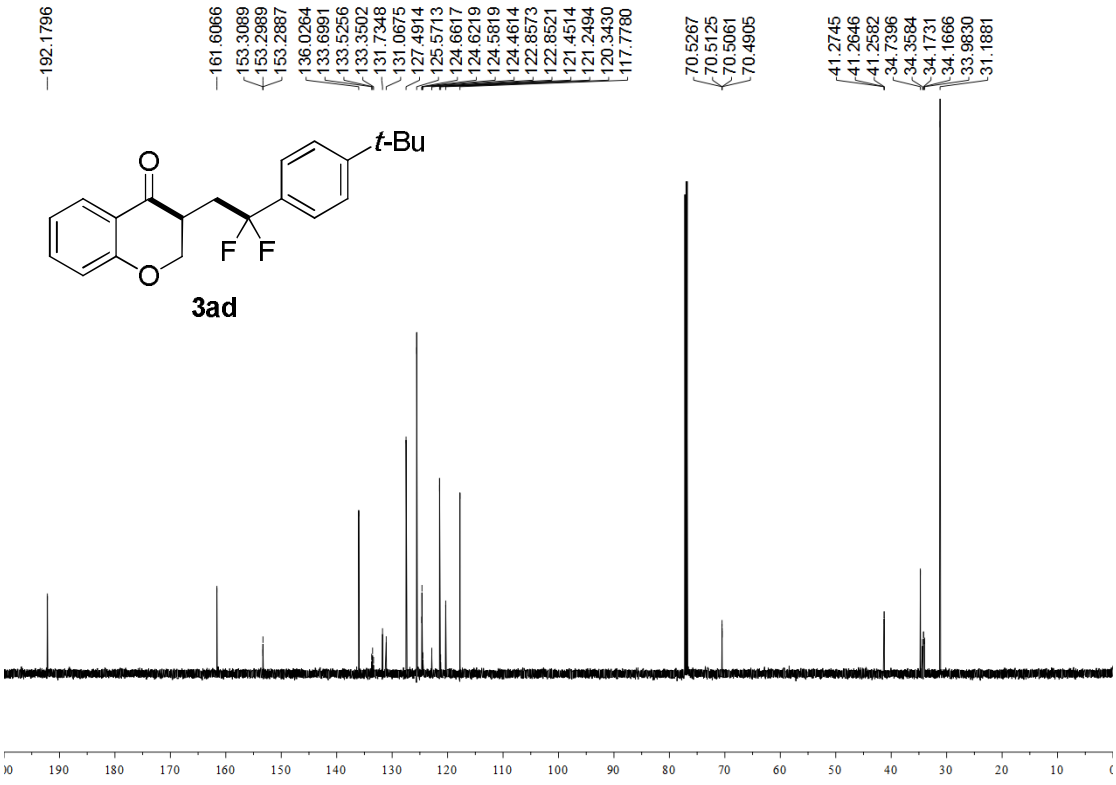

192.1982
161.5969
136.0327
135.4694
135.4526
135.4335
134.6368
134.4742
134.3121
132.2019
130.0078
127.4640
125.8741
125.5978
125.5404
125.4812
125.0900
123.4743
121.8586
121.4584
120.3321
117.7822
70.5487
70.5345
70.5255
41.1871
33.6215
33.4390
33.2569
20.2604
20.2398
20.2182
O
F F
O
3ac

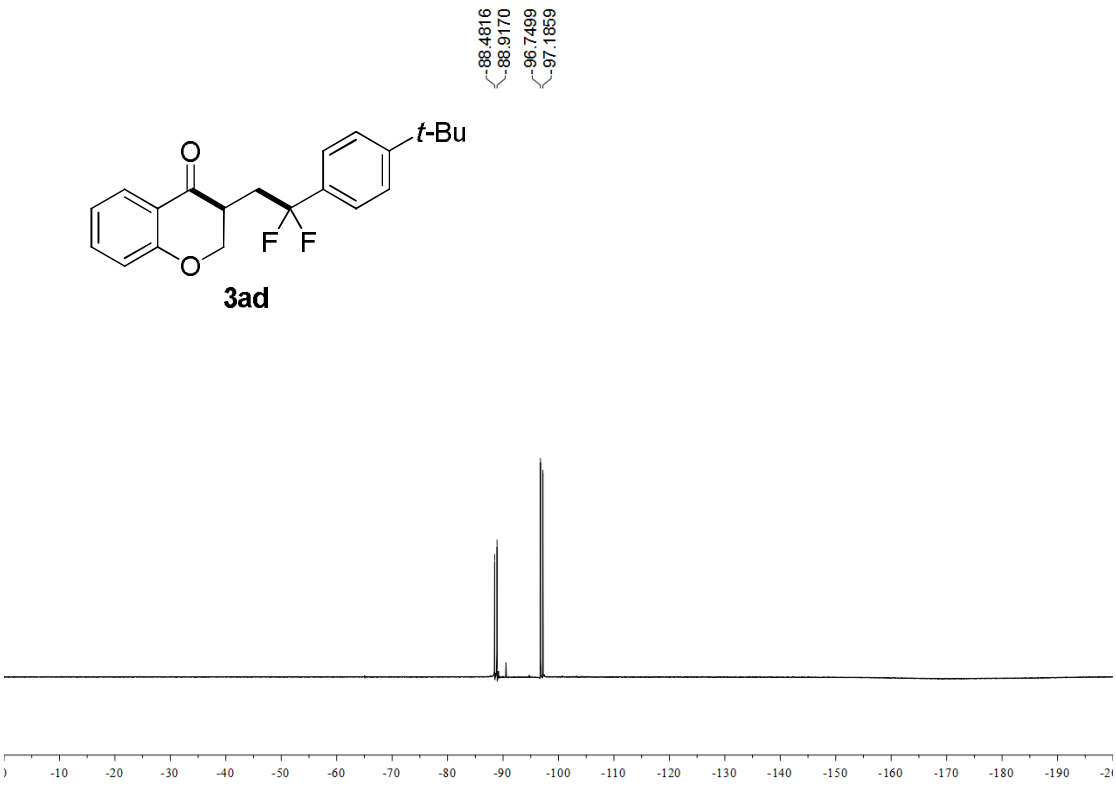
3ac

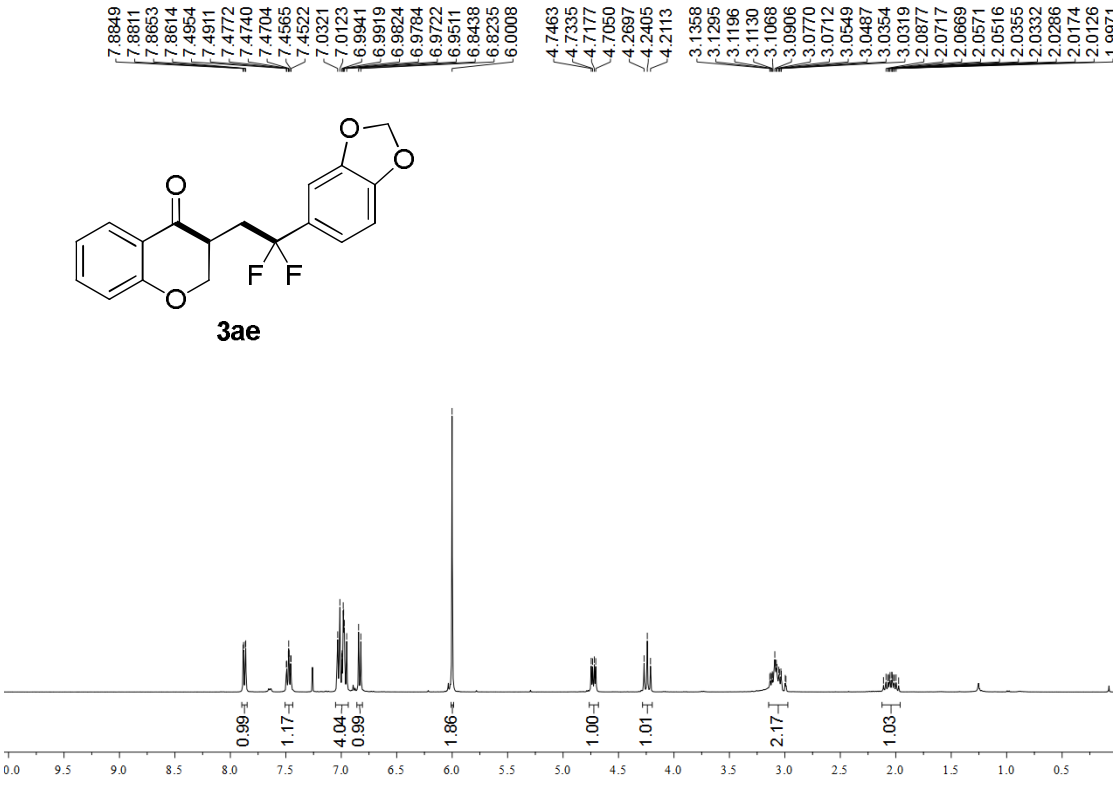

-89.6634
-90.1033
-94.0317
-94.4717

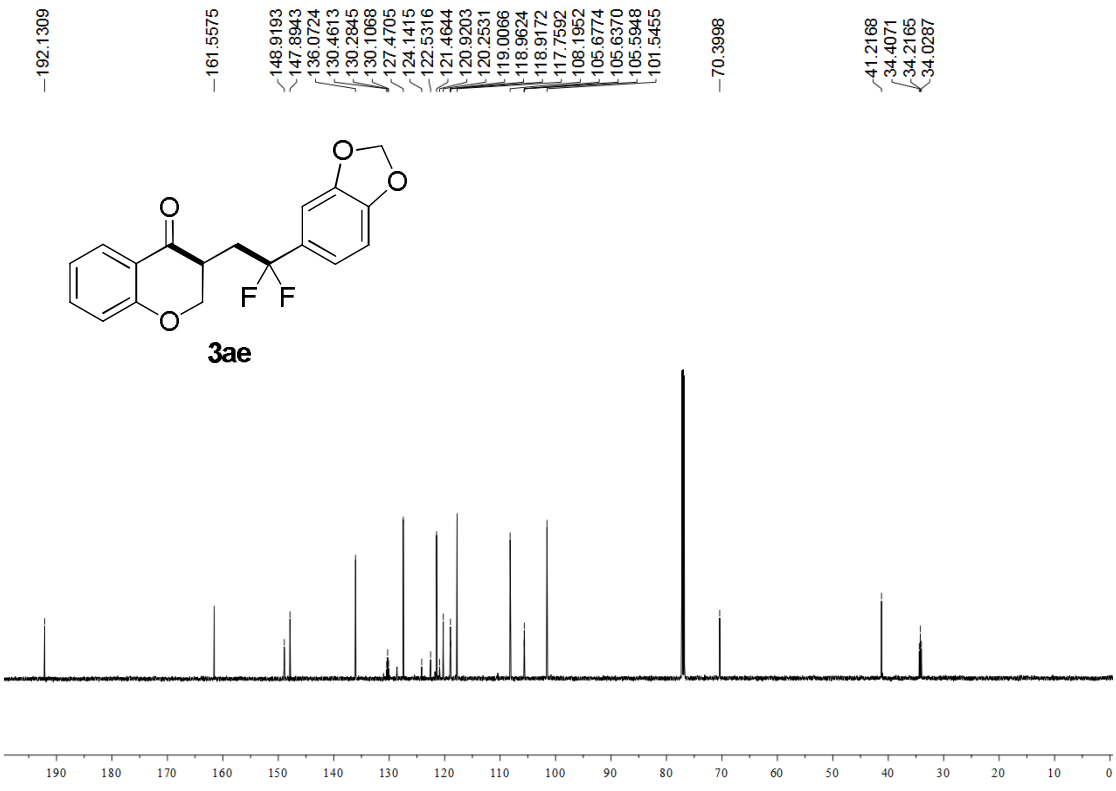
3ad


7.8898
7.8870
7.8767
7.8739
7.4895
7.4866
7.4775
7.4754
7.4727
7.4605
7.4529
7.0283
7.0267
7.0150
7.0032
7.0016
6.9749
6.9610
4.7815
4.7728
4.7621
4.7537
4.2650
4.2654
4.2461
3.1614
3.1575
3.1319
3.1268
3.1221
3.1180
3.1105
3.1061
3.0949
3.0905
3.0859
3.0679
3.0640
2.1135
2.1100
2.0996
2.0973
2.0873
2.0732
2.0630
2.0606
2.0501
0.92
5.03
0.96
0.91
1.00
1.03
2.13
1.12
9.23

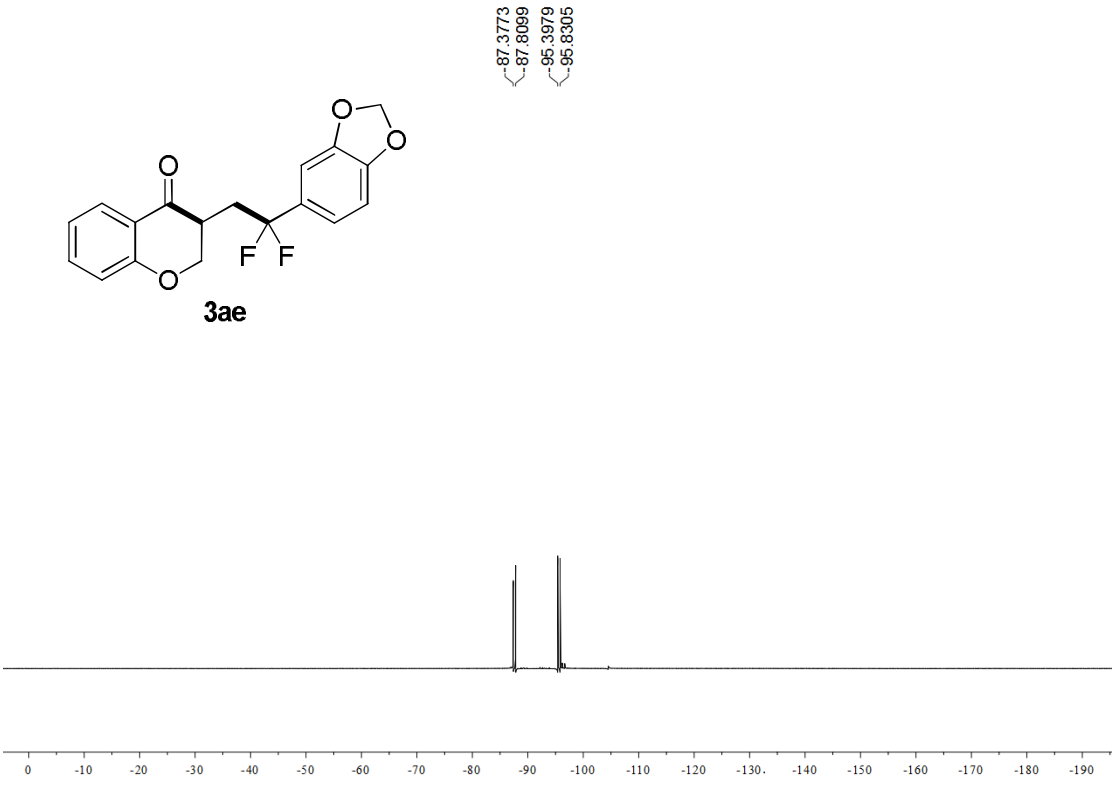

192.1796
161.6066
153.3089
153.2989
153.2887
136.0264
133.6991
133.5256
133.3502
131.7348
131.0675
127.4914
125.5713
124.6617
124.6219
124.5819
124.4614
122.8573
122.8521
121.4514
121.2494
120.3430
117.7780
70.5267
70.5125
70.5061
70.4905
41.2745
41.2646
41.2582
34.7396
34.3584
34.1731
34.1666
33.9830
31.1881
t-Bu
O
O
F F
3ad
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


-88.4816
-88.9170
-96.7499
-97.1659
t-Bu
O
O
F F
3ad
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

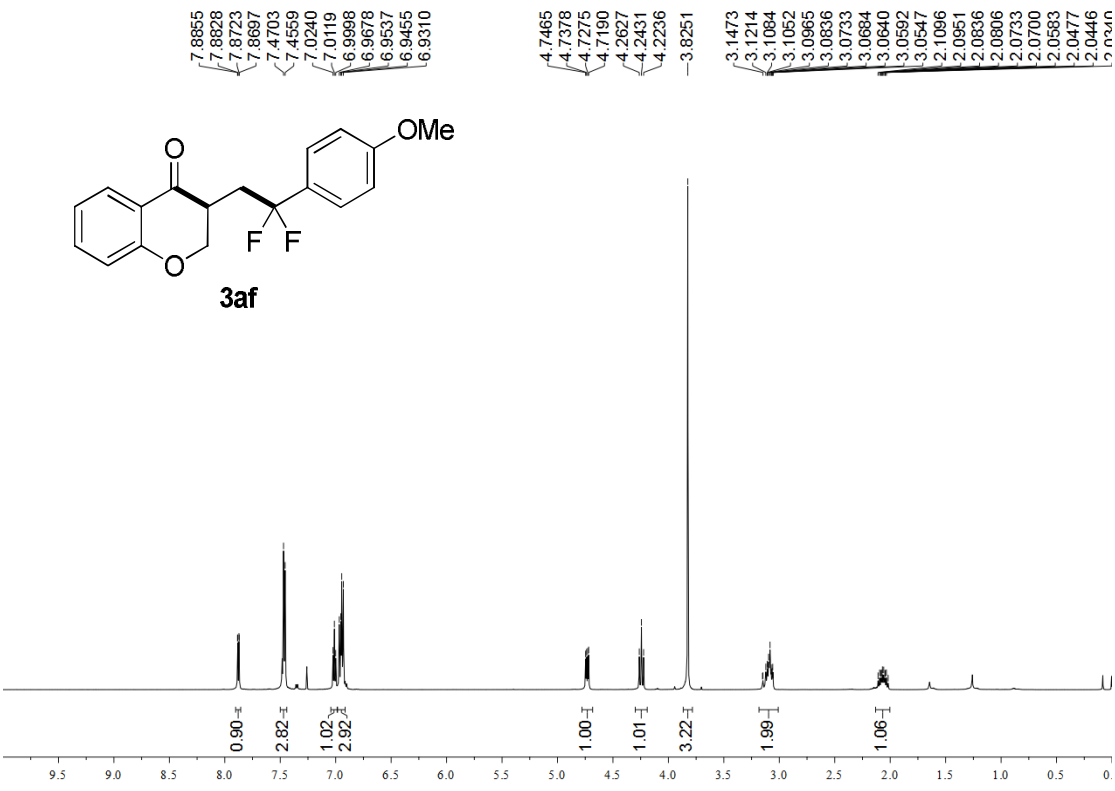

7.8849
7.8811
7.8653
7.8614
7.4954
7.4911
7.4772
7.4740
7.4704
7.4565
7.4522
7.0321
7.0123
6.9941
6.9919
6.9824
6.9784
6.9722
6.9511
6.8438
6.8235
6.0008
4.7463
4.7335
4.7177
4.7050
4.2697
4.2405
4.2113
3.1358
3.1295
3.1196
3.1130
3.1068
3.0906
3.0770
3.0712
3.0549
3.0487
3.0354
3.0319
2.0877
2.0717
2.0669
2.0571
2.0516
2.0355
2.0332
2.0286
2.0174
2.0126
O
O
O
F
F
O
3ae
0.99
1.17
4.04
0.99
1.86
1.00
1.01
2.17
1.03
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

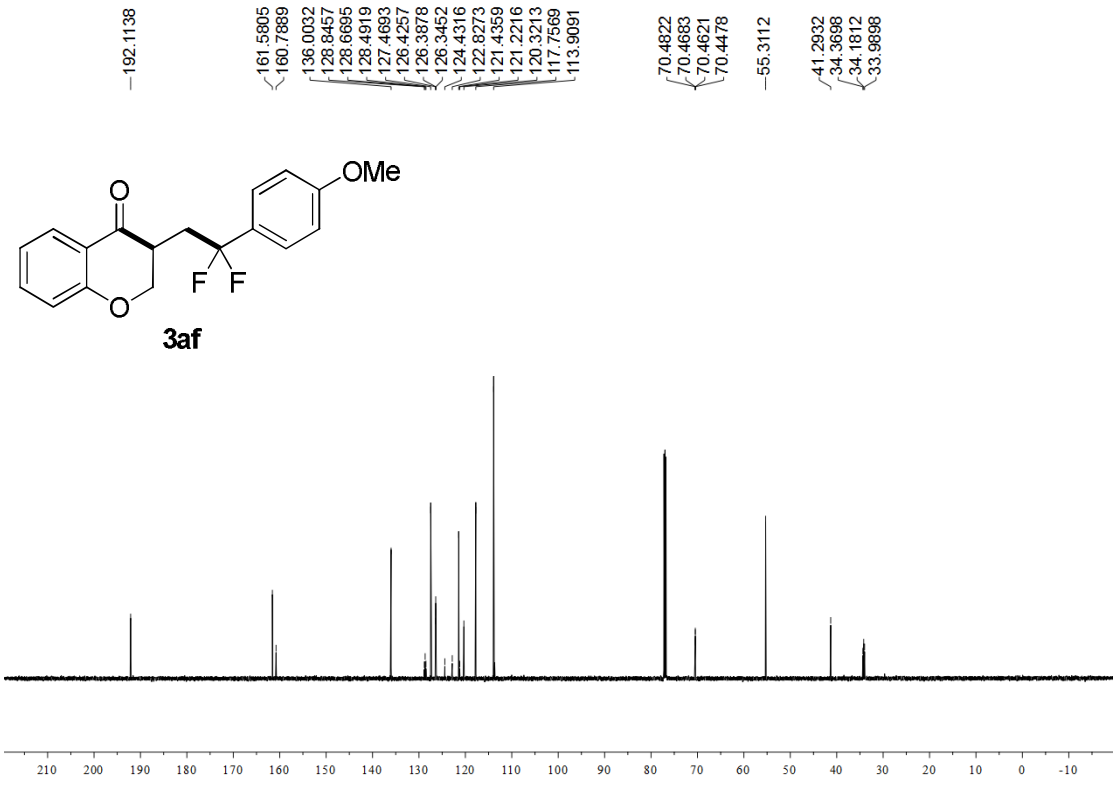

192.1309
161.5575
148.9193
147.8943
136.0724
130.4613
130.2845
130.1068
127.4705
124.1415
122.5316
121.4644
120.9203
120.2531
119.0066
118.9624
118.9172
117.7592
108.1952
105.6774
105.6370
105.5948
101.5455
70.3998
41.2168
34.4071
34.2165
34.0287
O
O
O
F
F
O
3ae
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

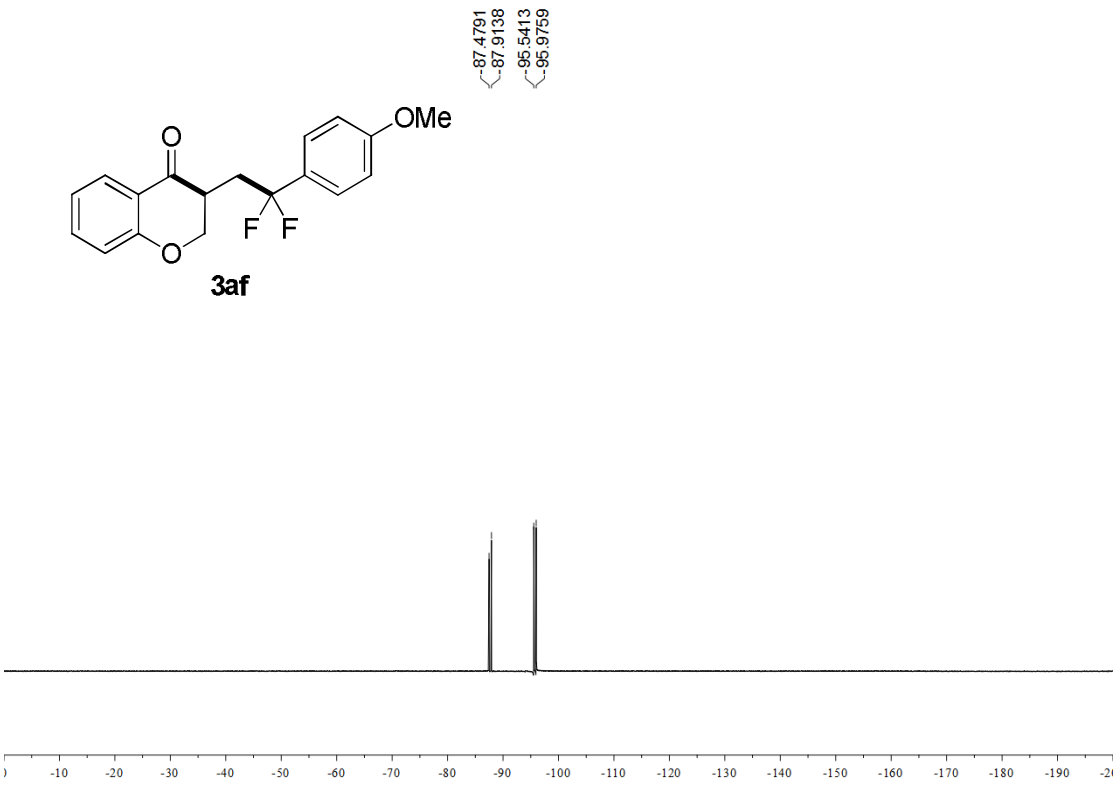
3ae

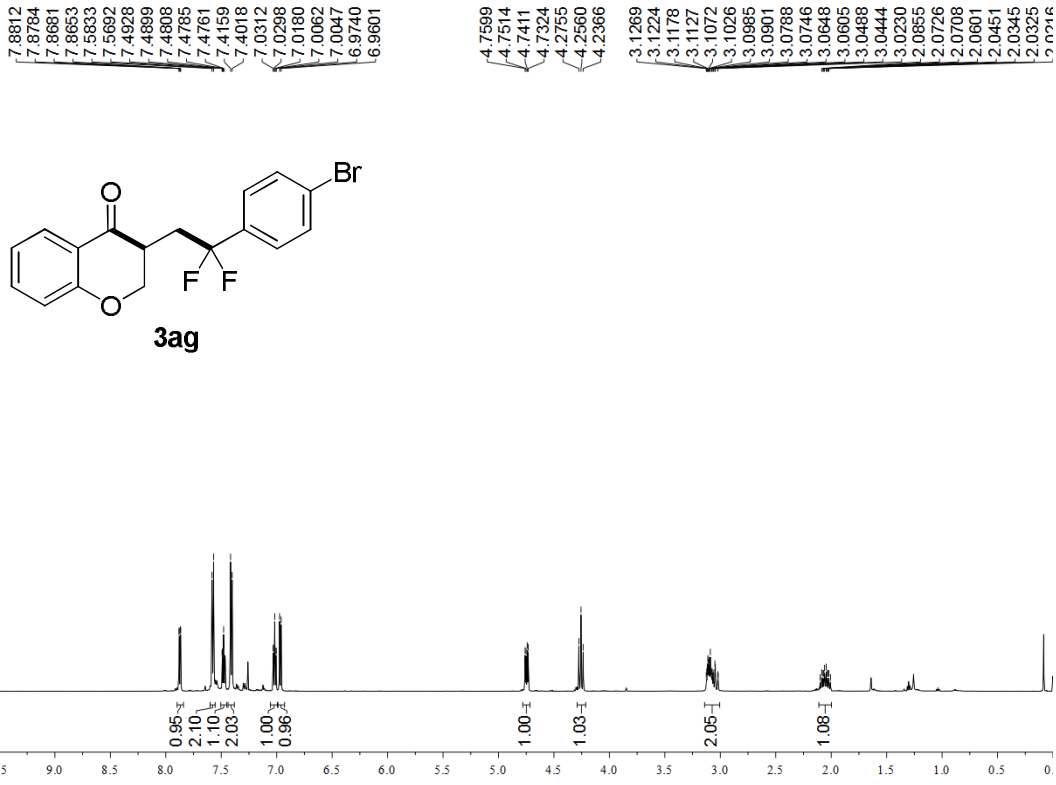

-87.3773
-87.8099
-95.3979
-95.8305

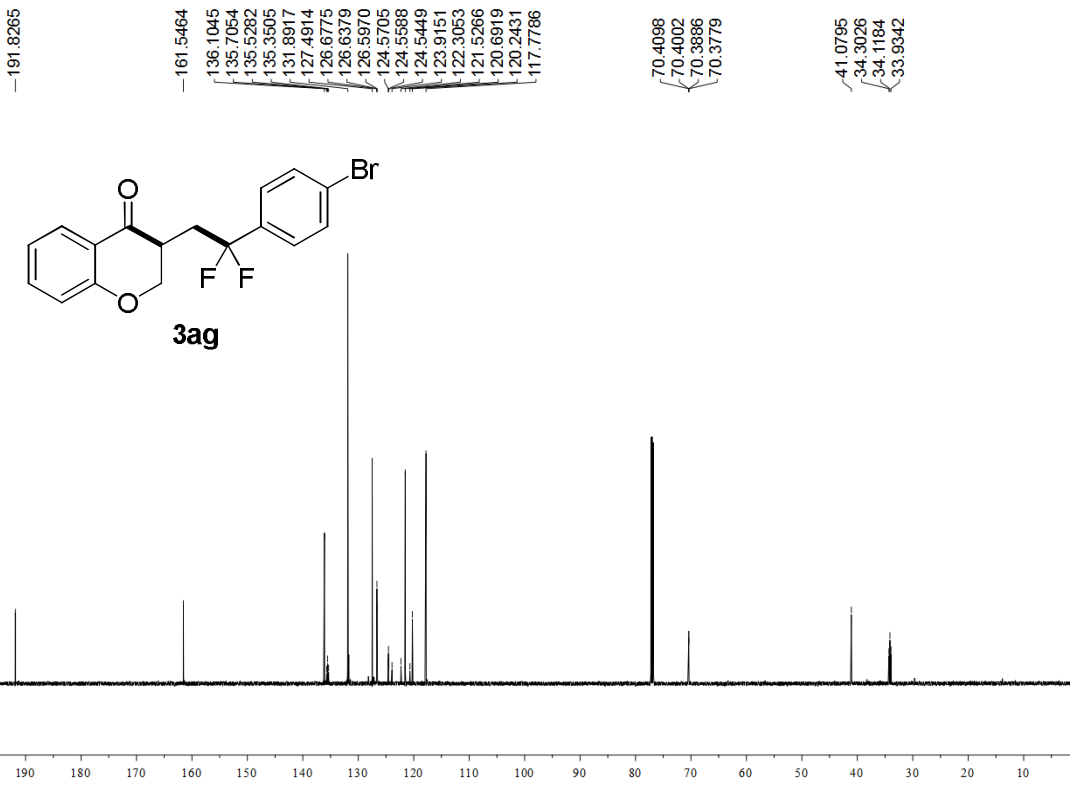
3af

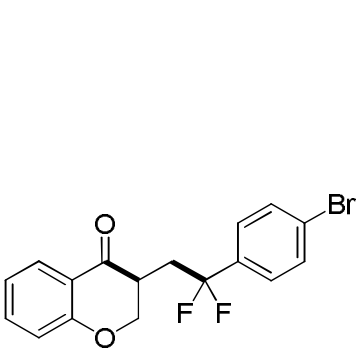

7.8855
7.8828
7.8723
7.8697
7.4703
7.4559
7.0240
7.0119
6.9998
6.9678
6.9537
6.9455
6.9310
4.7465
4.7378
4.7275
4.7190
4.2627
4.2431
4.2236
3.8251
3.1473
3.1214
3.1084
3.1052
3.0965
3.0836
3.0733
3.0684
3.0640
3.0592
3.0547
2.1096
2.0951
2.0836
2.0806
2.0733
2.0700
2.0583
2.0477
2.0446
0.90
2.82
1.02
2.92
1.00
1.01
3.22
1.99
1.06


192.1138
161.5805
160.7889
136.0032
128.8457
128.6695
128.4919
127.4693
126.4257
126.3878
126.3452
124.4316
122.8273
121.4359
121.2216
120.3213
117.7569
113.9091
70.4822
70.4683
70.4621
70.4478
55.3112
41.2932
34.3698
34.1812
33.9898
OMe
O
F F
O
3af
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10


-87.4791
-87.9138
-95.5413
-95.9759
OMe
O
F F
O
3af
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

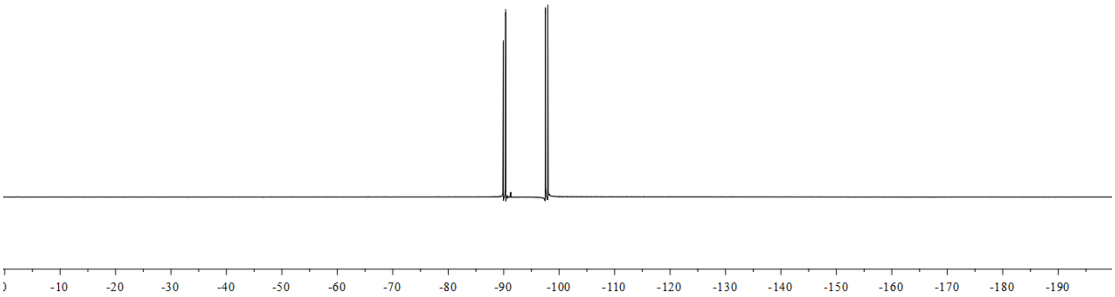

7.8812
7.8784
7.8681
7.8653
7.5833
7.5692
7.4928
7.4899
7.4808
7.4785
7.4761
7.4159
7.4018
7.0312
7.0298
7.0180
7.0062
7.0047
6.9740
6.9601
4.7599
4.7514
4.7411
4.7324
4.2755
4.2560
4.2366
3.1269
3.1224
3.1178
3.1127
3.1072
3.1026
3.0985
3.0901
3.0788
3.0746
3.0648
3.0605
3.0488
3.0444
3.0230
2.0855
2.0726
2.0708
2.0601
2.0451
2.0345
2.0325
Br
O
F F
O
3ag
0.95
2.10
1.10
2.03
1.00
0.96
1.00
1.03
2.05
1.08
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

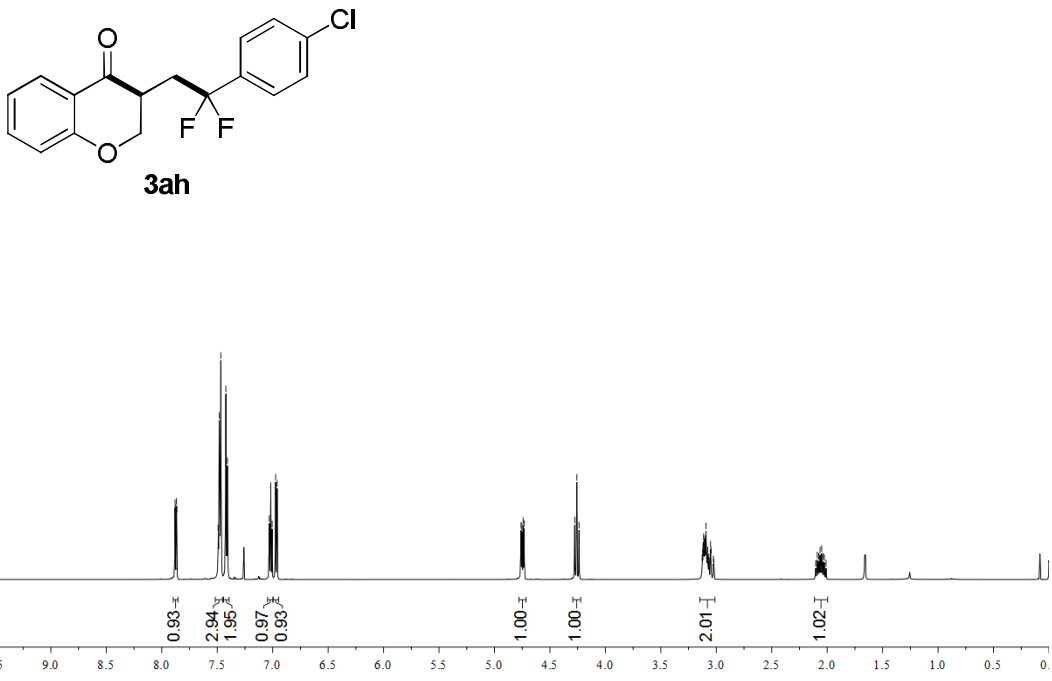

191.8265
161.5464
136.1045
135.7054
135.5282
135.3505
131.8917
127.4914
126.6775
126.6379
126.5970
124.5705
124.5588
124.5449
123.9151
122.3053
121.5266
120.6919
120.2431
117.7786
70.4098
70.4002
70.3886
70.3779
41.0795
34.3026
34.1184
33.9342
Br
O
F F
O
3ag
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10


Br
O
F
F
O

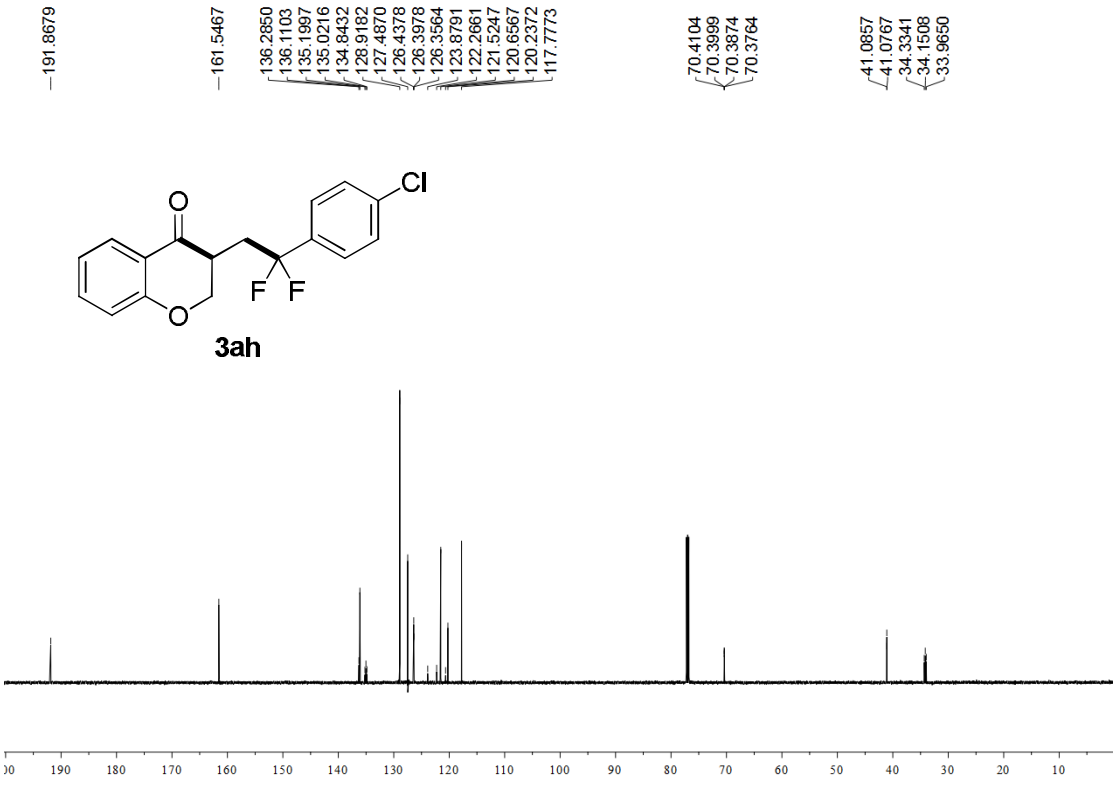
3ag

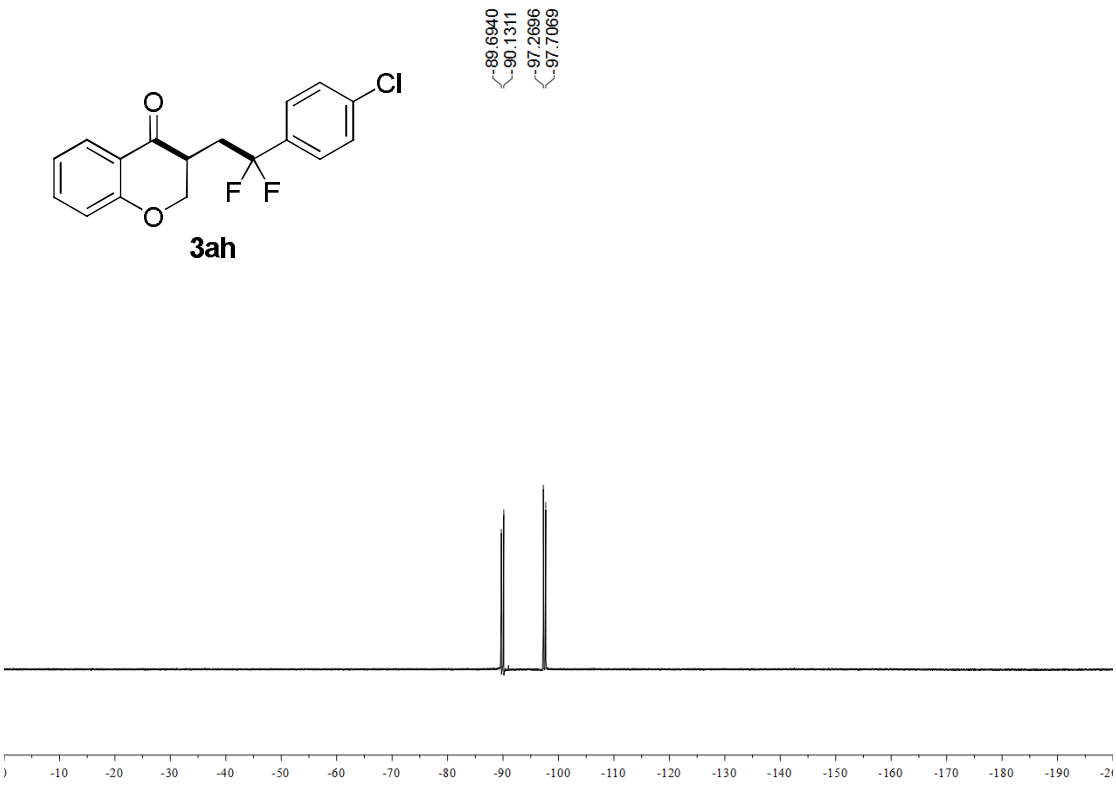
-89.9257
-90.3625
-97.5310
-97.9681

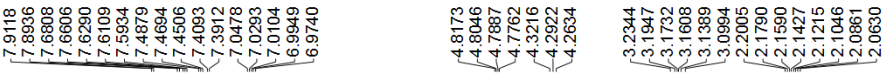

0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

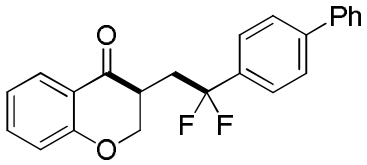

Cl
O
F
F
O
7.8804
7.8776
7.8673
7.8645
7.4922
7.4893
7.4817
7.4677
7.4223
7.4082
7.0303
7.0182
7.0173
7.0053
6.9736
6.9597
4.7607
4.7524
4.7419
4.7332
4.2766
4.2572
4.2377
3.1281
3.1234
3.1195
3.1149
3.1083
3.1038
3.0996
3.0933
3.0830
3.0779
3.0728
3.0677
3.0634
3.0517
3.0473
3.0258
3.0215
2.0894
2.0766
2.0643
2.0494
2.0384
0.93
2.94
1.95
0.97
0.93
1.00
1.00
2.01
1.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

3ah

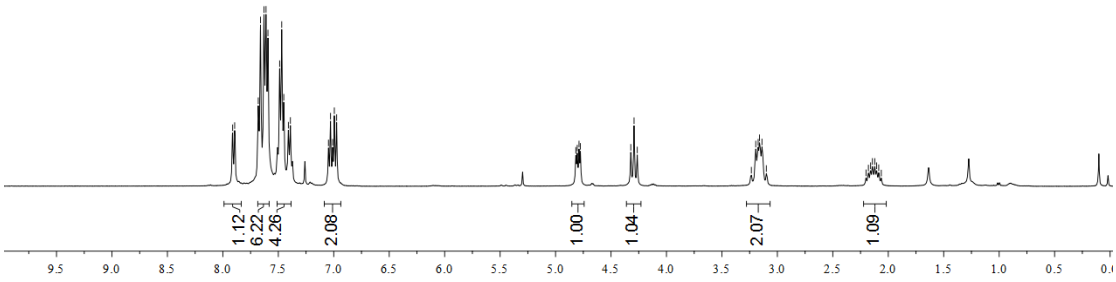

191.8679
161.5467
136.2850
136.1103
135.1997
135.0216
134.8432
128.9182
127.4870
126.4378
126.3978
126.3564
123.8791
122.2661
121.5247
120.6567
120.2372
117.7773
70.4104
70.3999
70.3874
70.3764
41.0857
41.0767
34.3341
34.1508
33.9650
O
Cl
F F
O
3ah

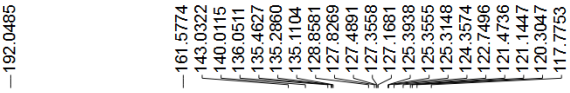

89.6940
90.1311
97.2696
97.7069
O
Cl
F F
O
3ah

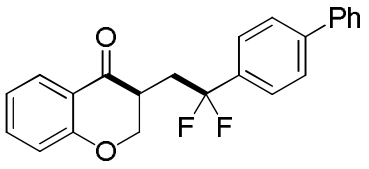

7.9118
7.8936
7.6808
7.6606
7.6290
7.6109
7.5934
7.4879
7.4694
7.4506
7.4093
7.3912
7.0478
7.0293
7.0104
6.9949
6.9740
4.8173
4.8046
4.7887
4.7762
4.3216
4.2922
4.2634
3.2344
3.1947
3.1732
3.1608
3.1389
3.0994
2.2005
2.1790
2.1590
2.1427
2.1215
2.1046
2.0861
2.0630


Ph
O
F
F
O

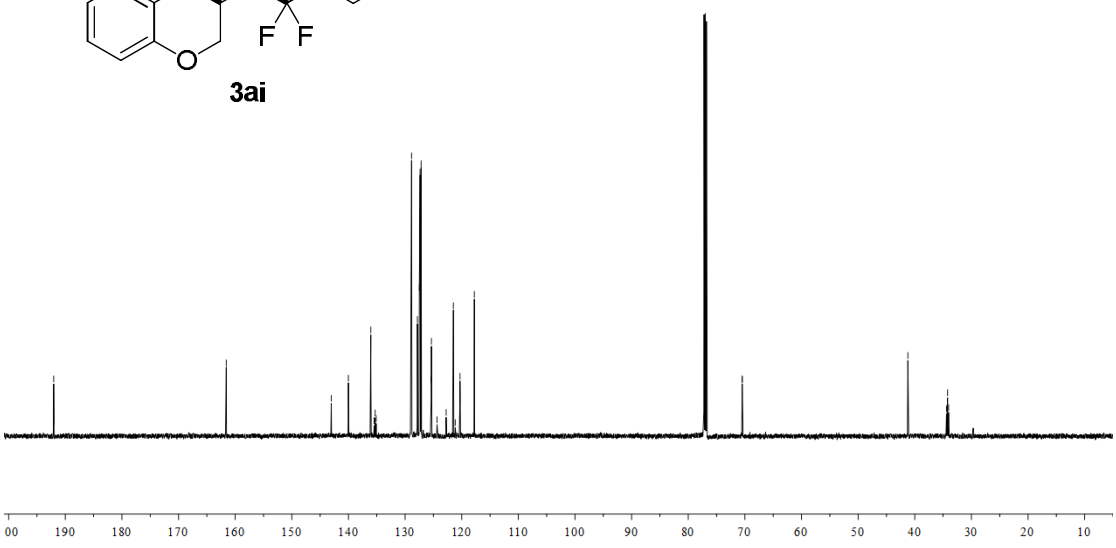
3ai

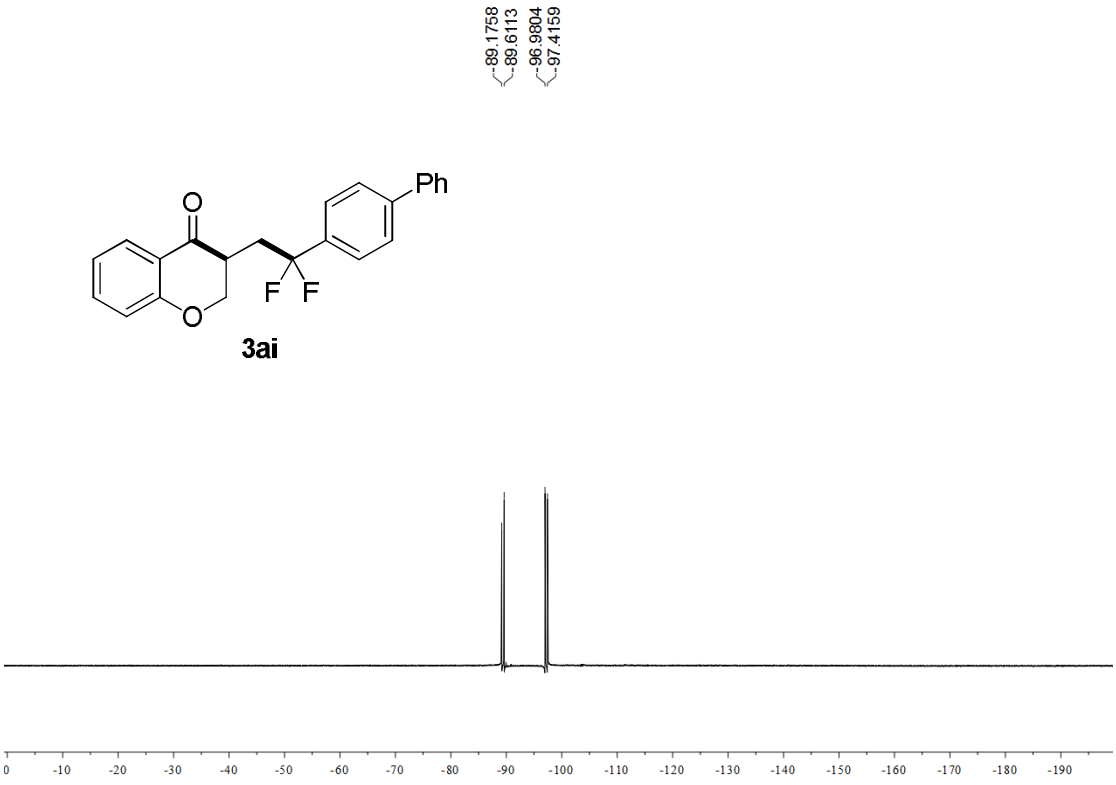

1.12
6.22
4.26
2.08
1.00
1.04
2.07
1.09
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

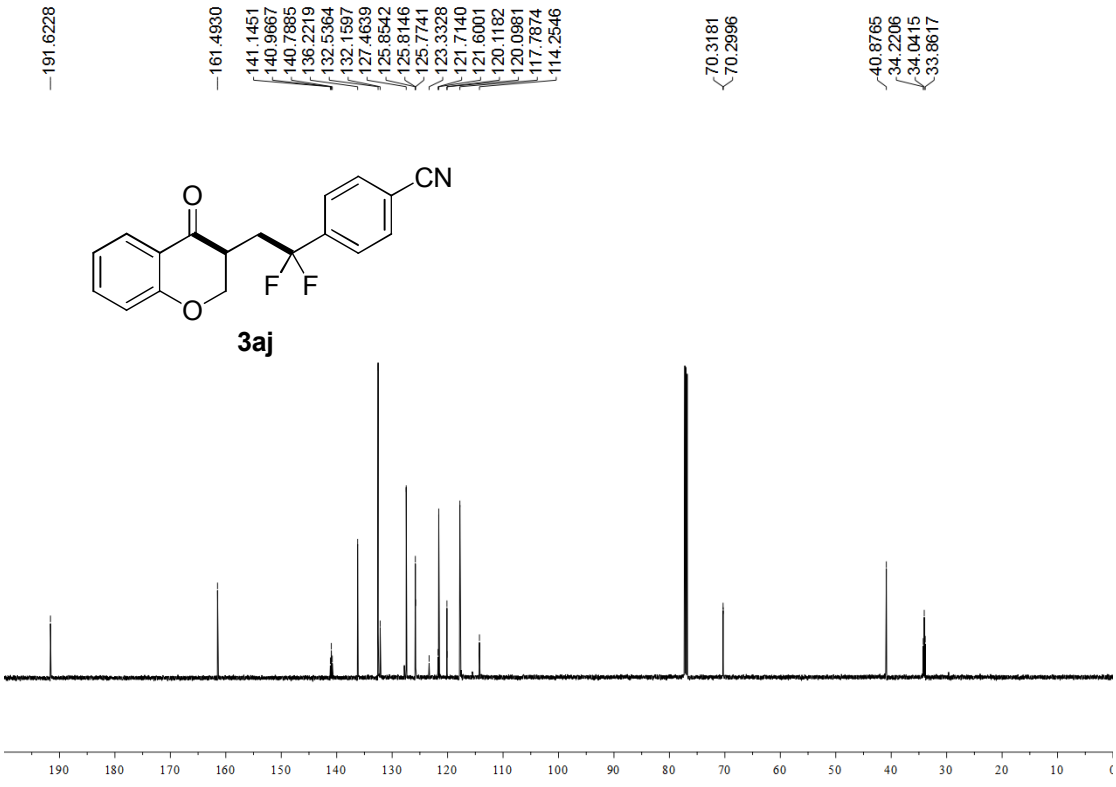

192.0485
161.5774
143.0322
140.0115
136.0511
135.4627
135.2860
135.1104
128.8581
127.8269
127.4891
127.3558
127.1681
125.3938
125.3555
125.3148
124.3574
122.7496
121.4736
121.1447
120.3047
117.7753
70.4720
70.4656
41.2172
34.3985
34.2136
34.0262

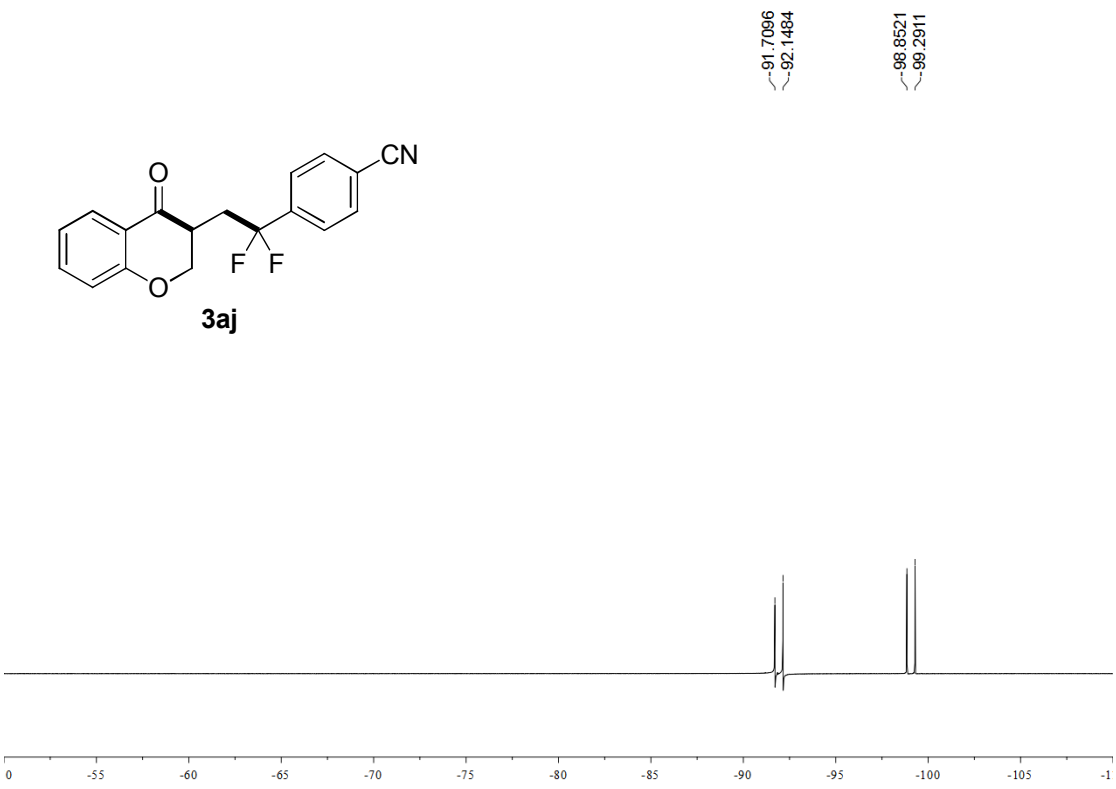

Ph
O
F
F
O

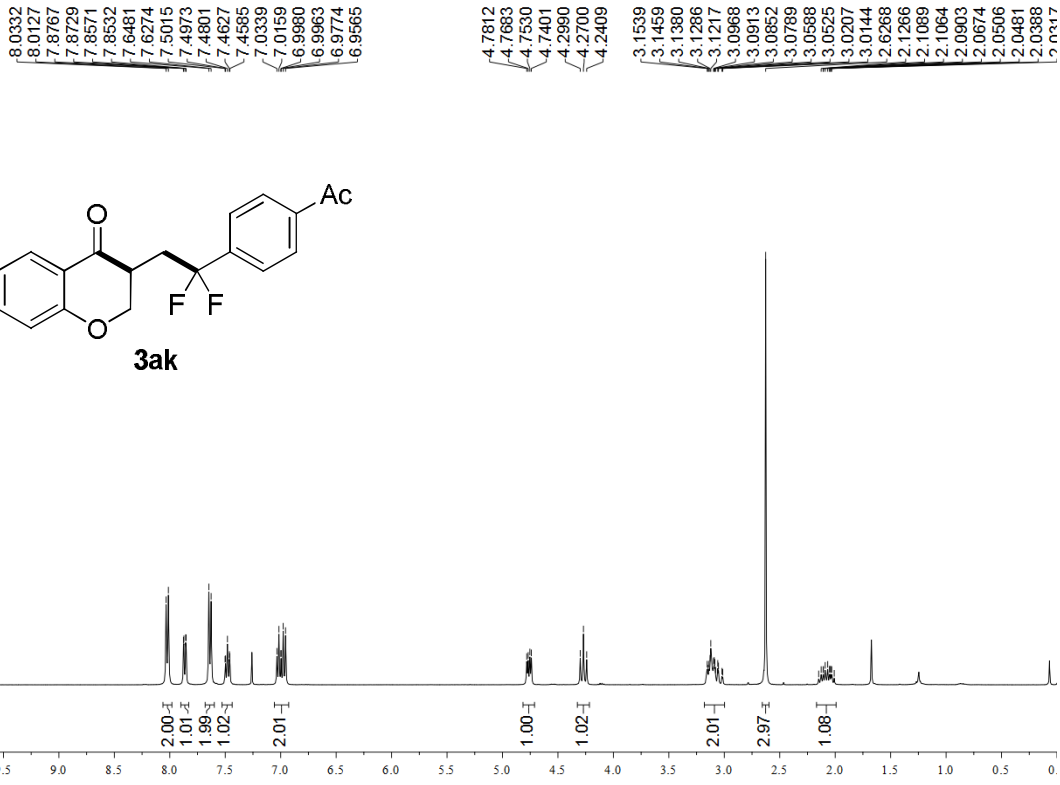
3ai

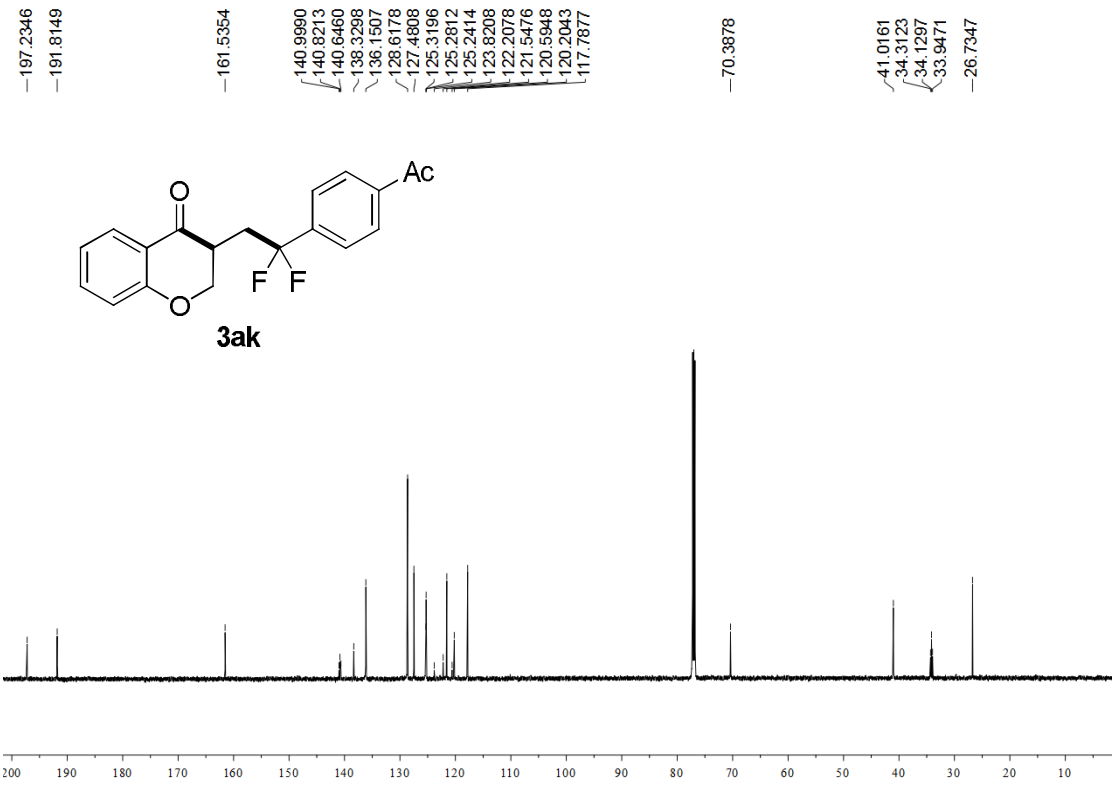

00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

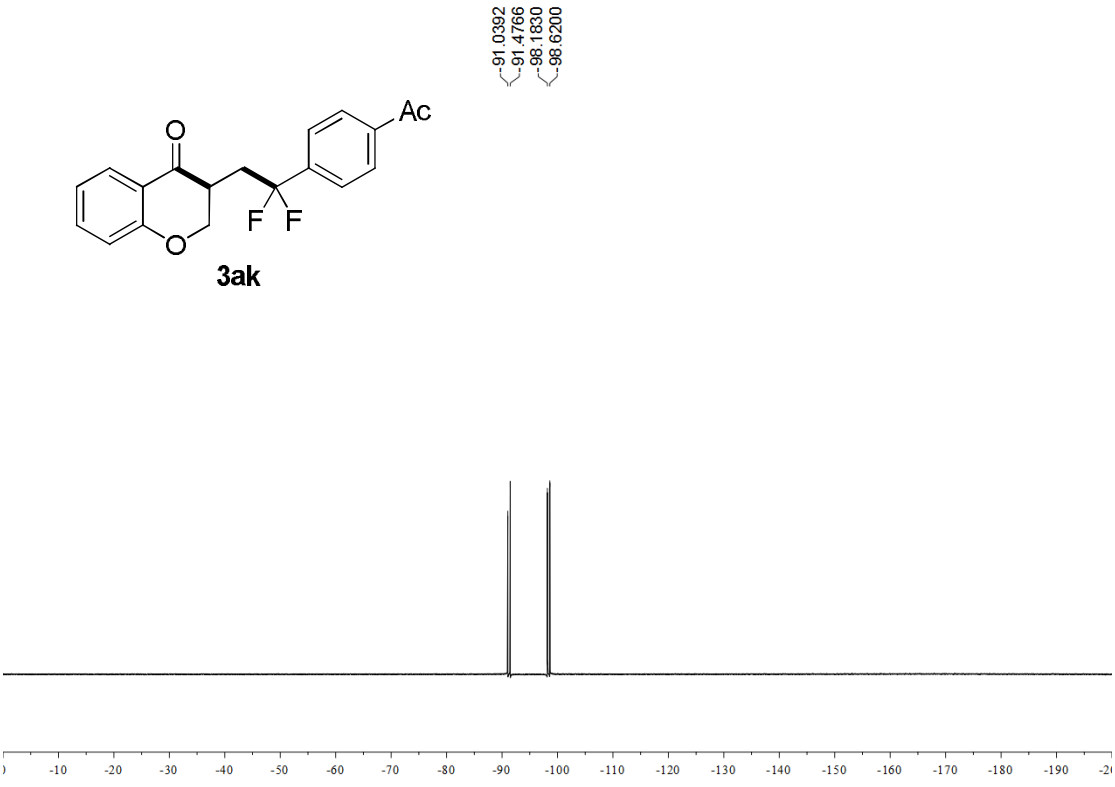

-89.1758
-89.6113
-96.9804
-97.4159
O
Ph
F F
O
3ai
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190
7.8711
7.8672
7.8514
7.8475
7.7643
7.7436
7.6712
7.6505
7.5076
7.5035
7.4860
7.4688
7.4647
7.0391
7.0210
7.0014
6.9807
6.9598
4.7855
4.7726
4.7570
4.7439
4.3060
4.2769
4.2476
3.1696
3.1626
3.1568
3.1485
3.1399
3.1323
3.1277
3.1193
3.1106
3.1045
3.0948
3.0633
3.0562
3.0494
3.0247
3.0219
2.9890
2.1188
2.0977
2.0777
2.0579
2.0370
O
CN
F F
O
3aj
1.00
2.13
2.33
1.01
2.02
1.00
1.00
2.04
1.05
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5

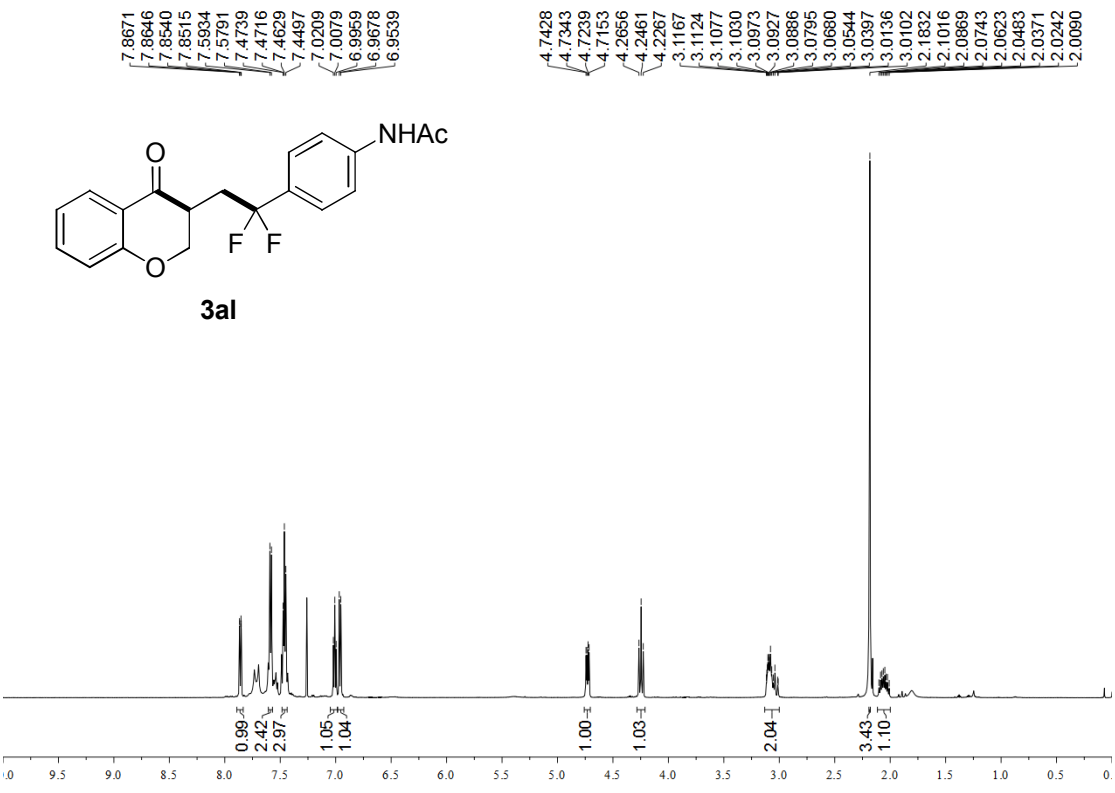

191.6228
161.4930
141.1451
140.9667
140.7885
136.2219
132.5364
132.1597
127.4639
125.8542
125.8146
125.7741
123.3328
121.7140
121.6001
120.1182
120.0981
117.7874
114.2546
70.3181
70.2996
40.8765
34.2206
34.0415
33.8617
O
CN
F F
O
3aj
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

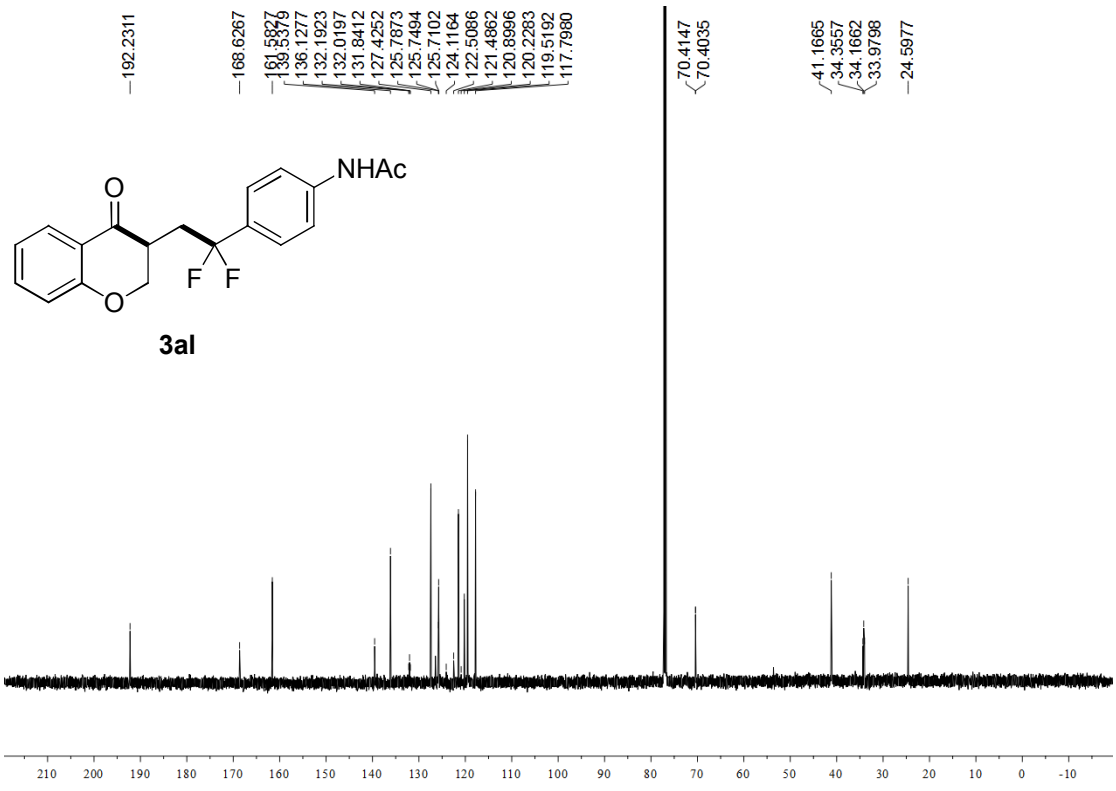

-91.7096
-92.1484
-98.8521
-99.2911
O
CN
F F
O
3aj
0
-55
-60
-65
-70
-75
-80
-85
-90
-95
-100
-105

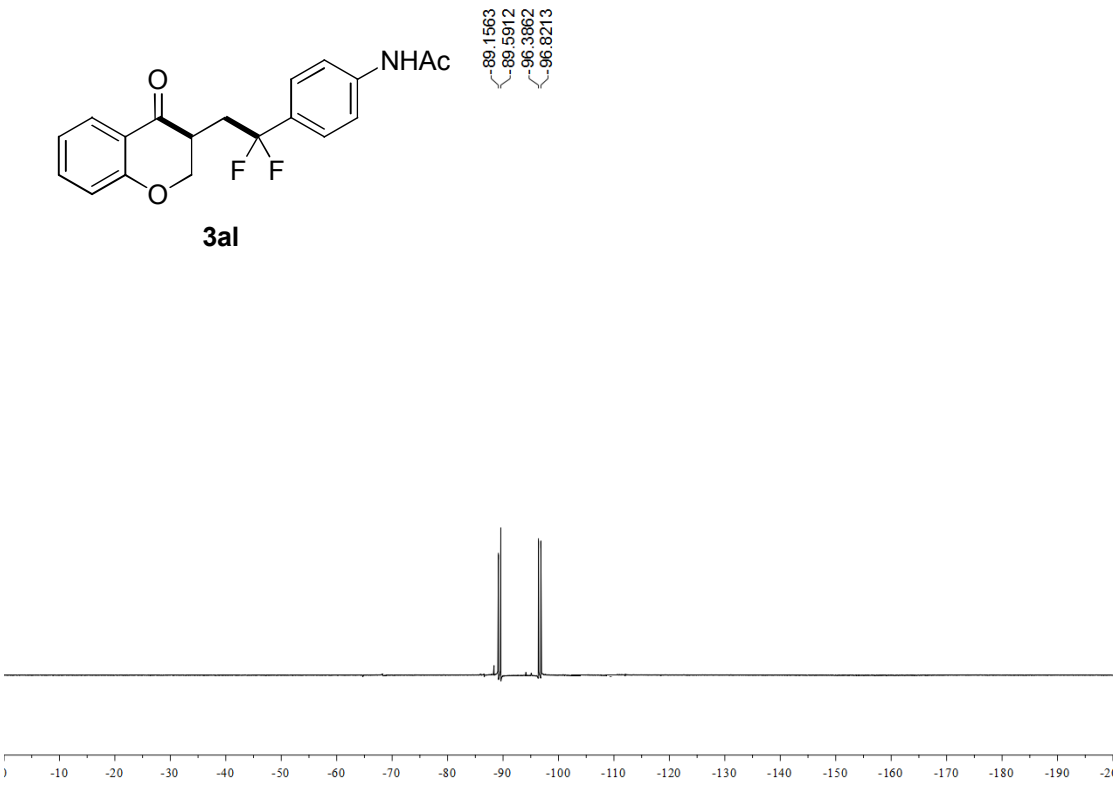

8.0332
8.0127
7.8767
7.8729
7.8571
7.8532
7.6481
7.6274
7.5015
7.4973
7.4801
7.4627
7.4585
7.0339
7.0159
6.9980
6.9963
6.9774
6.9565
4.7812
4.7683
4.7530
4.7401
4.2990
4.2700
4.2409
3.1539
3.1459
3.1380
3.1286
3.1217
3.0968
3.0913
3.0852
3.0789
3.0588
3.0525
3.0207
3.0144
2.6268
2.1266
2.1089
2.1064
2.0903
2.0674
2.0506
2.0481
2.0388
Ac
O
F F
O
3ak
2.00
1.01
1.99
1.02
2.01
1.00
1.02
2.01
2.97
1.08

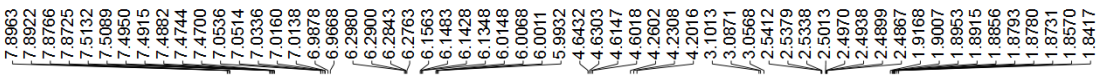

197.2346
191.8149
161.5354
140.9990
140.8213
140.6460
138.3298
136.1507
128.6178
127.4808
125.3196
125.2812
125.2414
123.8208
122.2078
121.5476
120.5948
120.2043
117.7877
70.3878
41.0161
34.3123
34.1297
33.9471
26.7347
Ac
O
F F
O
3ak

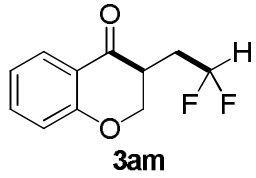

-91.0392
-91.4766
-98.1830
-98.6200
Ac
O
F F
O
3ak


7.8671
7.8646
7.8540
7.8515
7.5934
7.5791
7.4739
7.4716
7.4629
7.4497
7.0209
7.0079
6.9959
6.9678
6.9539
4.7428
4.7343
4.7239
4.7153
4.2656
4.2461
4.2267
3.1167
3.1124
3.1077
3.1030
3.0973
3.0927
3.0886
3.0795
3.0680
3.0544
3.0397
3.0136
3.0102
2.1832
2.1016
2.0869
2.0743
2.0623
2.0483
2.0371
2.0242
2.0090
NHAc
O
F F
O
3al
0.99
2.42
2.97
1.05
1.04
1.00
1.03
2.04
3.43
1.10

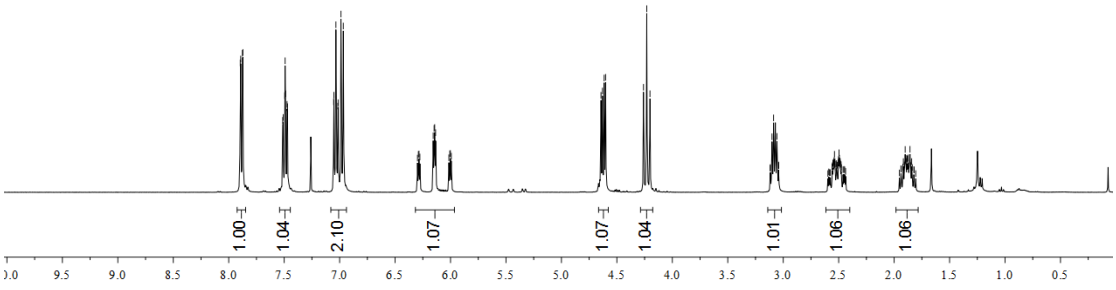

192.2311
168.6267
161.5827
139.5379
136.1277
132.1923
132.0197
131.8412
127.4252
125.7873
125.7494
125.7102
124.1164
122.5086
121.4862
120.8996
120.2283
119.5192
117.7980
70.4147
70.4035
41.1665
34.3557
34.1662
33.9798
24.5977
NHAc
O
F F
O
3aI
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

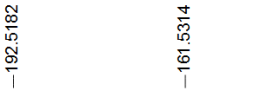

-89.1563
-89.5912
-96.3862
-96.8213
NHAc
O
F F
O
3aI
-10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

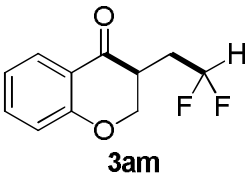

7.8963
7.8922
7.8766
7.8725
7.5132
7.5089
7.4950
7.4915
7.4882
7.4744
7.4700
7.0536
7.0514
7.0336
7.0160
7.0138
6.9878
6.9668
6.2980
6.2900
6.2843
6.2763
6.1563
6.1483
6.1428
6.1348
6.0148
6.0068
6.0011
5.9932
4.6432
4.6303
4.6147
4.6018
4.2602
4.2308
4.2016
3.1013
3.0871
3.0568
2.5412
2.5379
2.5338
2.5013
2.4970
2.4938
2.4899
2.4867
1.9168
1.9007
1.8953
1.8915
1.8856
1.8793
1.8780
1.8731
1.8570
1.8417


O
H
F
F
O

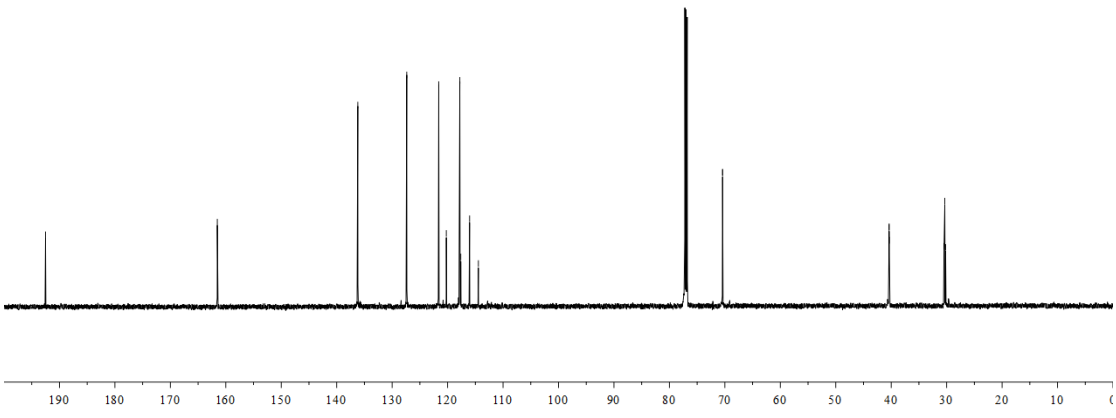
3am


1.00
1.04
2.10
1.07
1.07
1.04
1.01
1.06
1.06
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

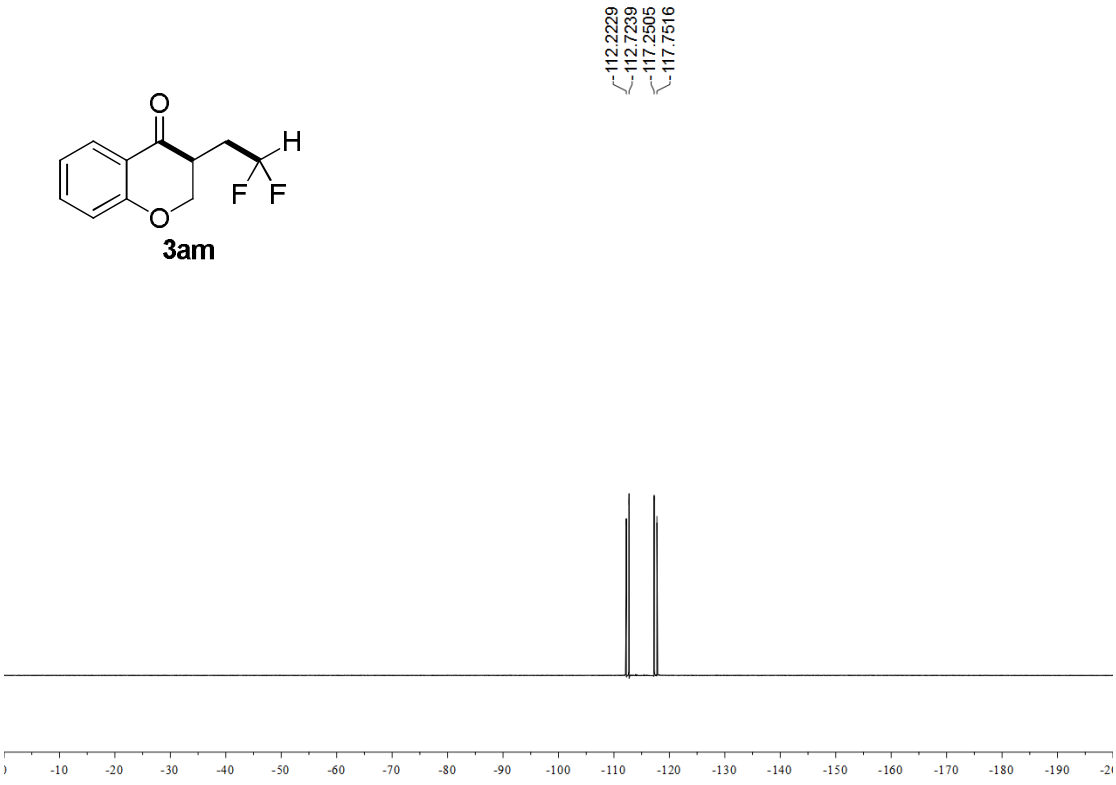

192.5182
161.5314
136.1909
127.3686
121.5987
120.2312
117.7952
117.6210
116.0343
114.4477
70.4117
70.4065
40.4054
40.3822
40.3729
40.3492
30.4815
30.3334
30.1857


O
H
F
F
O

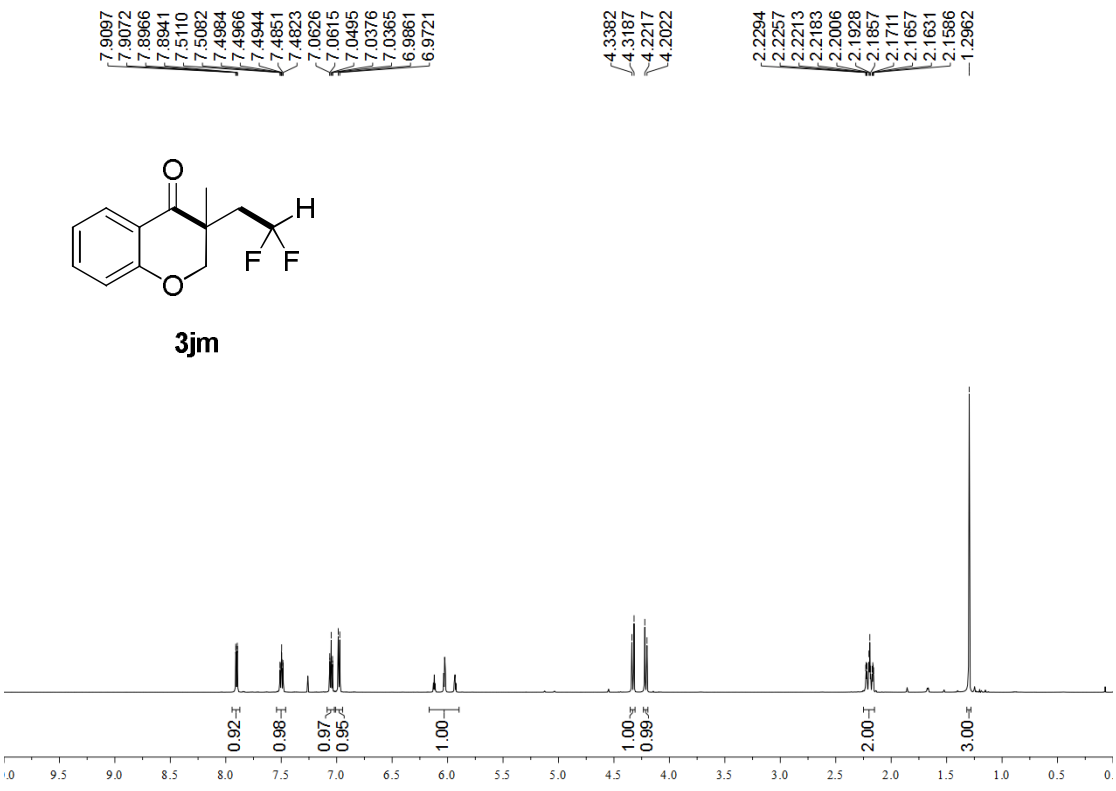
3am

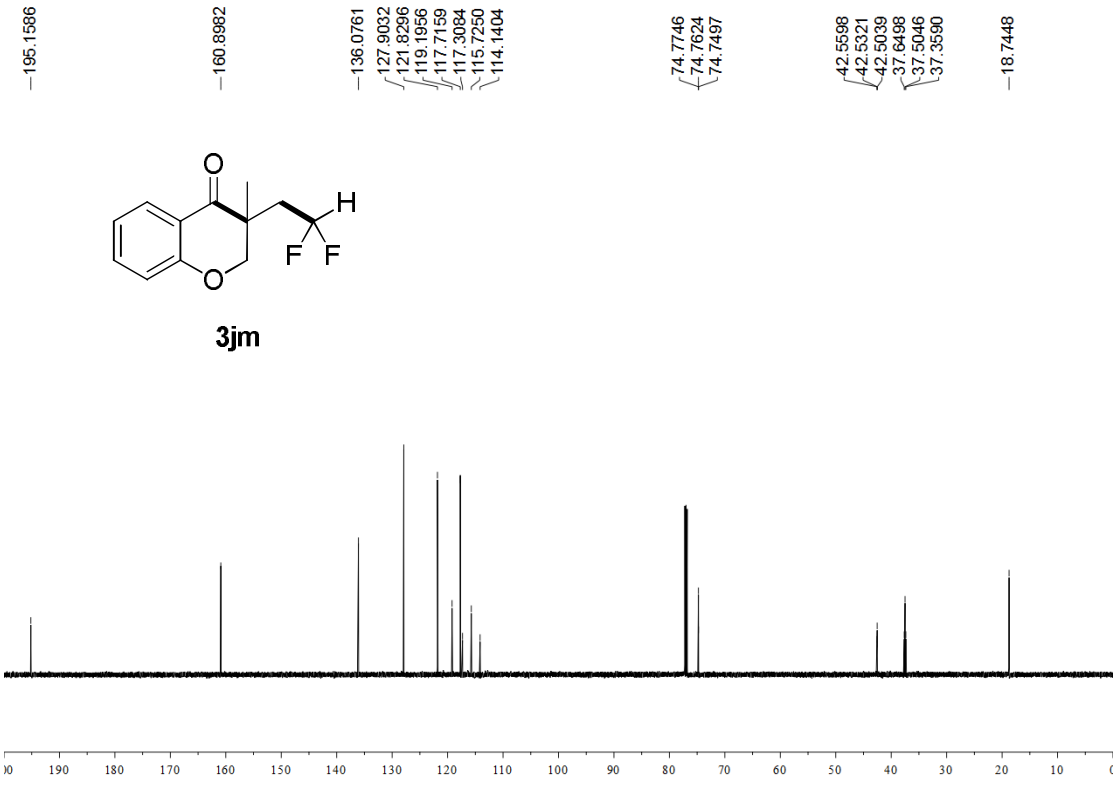

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

3am

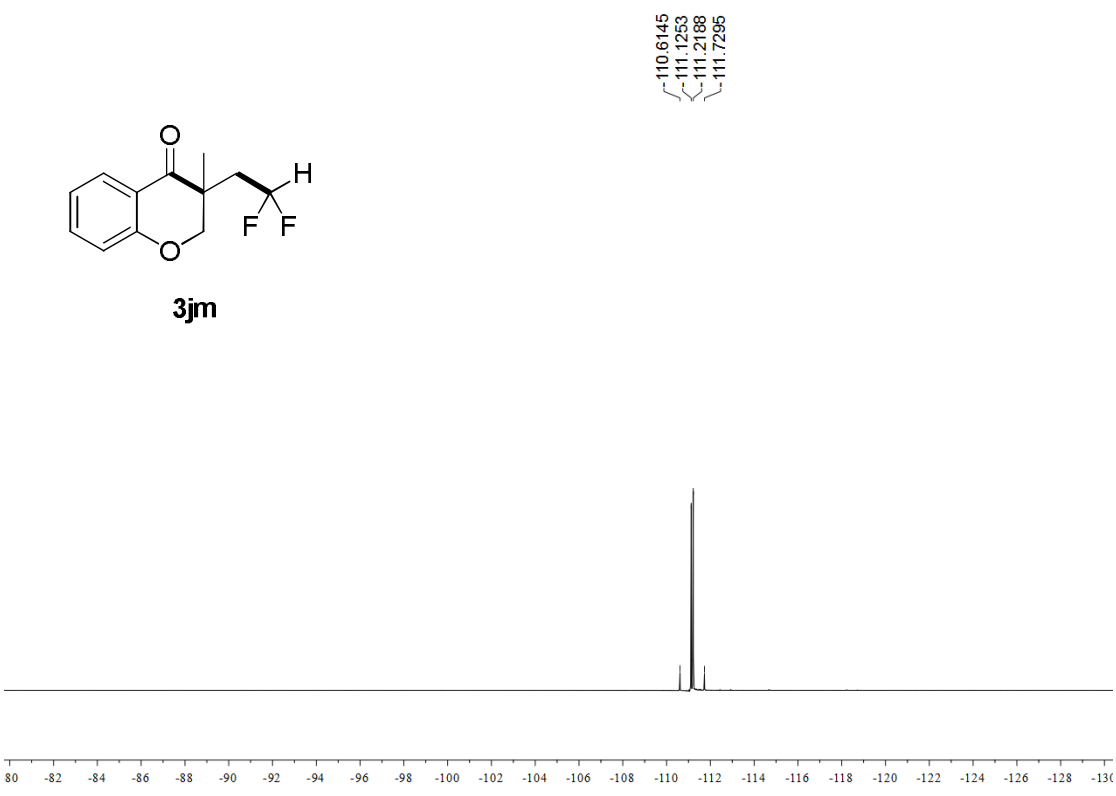

112.2229
112.7239
117.2505
117.7516
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

3jm

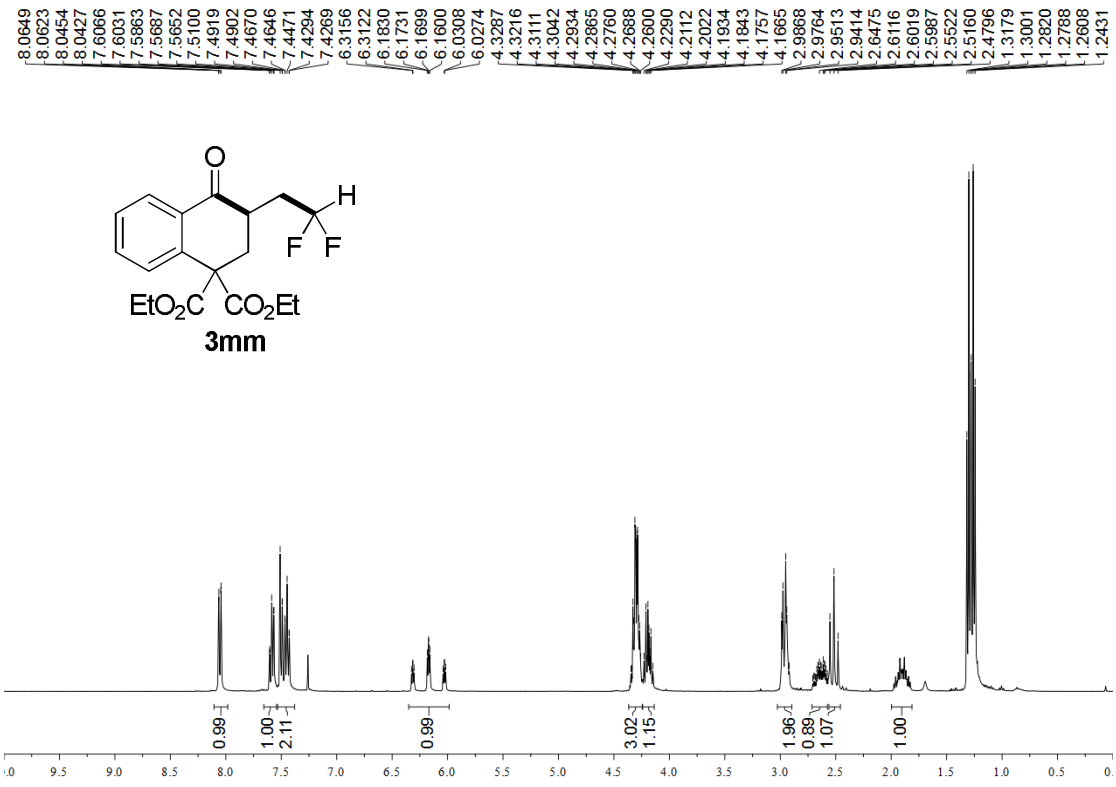

7.9097
7.9072
7.8966
7.8941
7.5110
7.5082
7.4984
7.4966
7.4944
7.4851
7.4823
7.0626
7.0615
7.0495
7.0376
7.0365
6.9861
6.9721
4.3382
4.3187
4.2217
4.2022
2.2294
2.2257
2.2213
2.2183
2.2006
2.1928
2.1857
2.1711
2.1657
2.1631
2.1586
1.2962
0.92
0.98
0.97
0.95
1.00
1.00
0.99
2.00
3.00

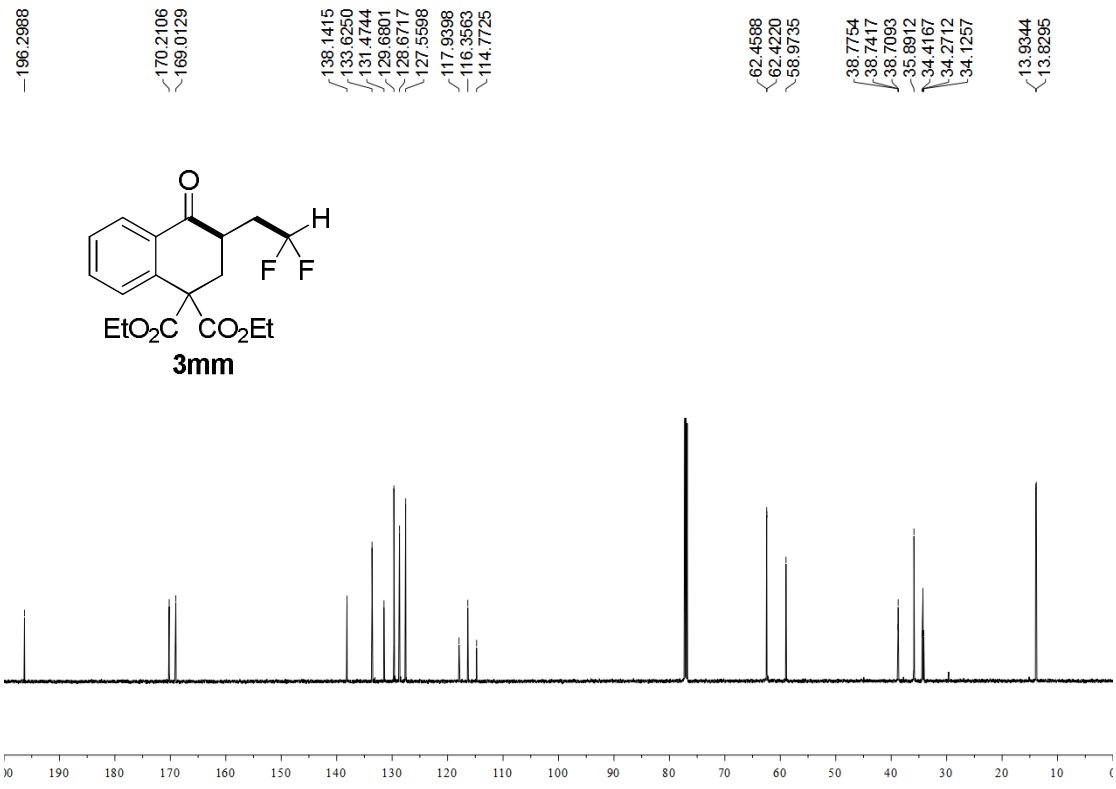

195.1586
160.8982
136.0761
127.9032
121.8296
119.1956
117.7159
117.3084
115.7250
114.1404
74.7746
74.7624
74.7497
42.5598
42.5321
42.5039
37.6498
37.5046
37.3590
18.7448
O
H
F F
O

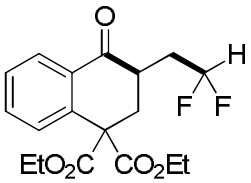
3jm


110.6145
111.1253
111.2188
111.7295
O
H
F F
O

3jm

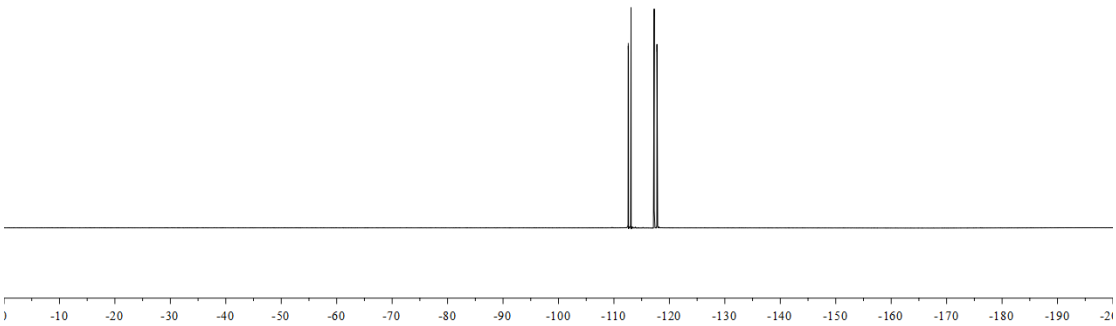

8.0649
8.0623
8.0454
8.0427
7.6066
7.6031
7.5863
7.5687
7.5652
7.5100
7.4919
7.4902
7.4670
7.4646
7.4471
7.4294
7.4269
6.3156
6.3122
6.1830
6.1731
6.1699
6.1600
6.0308
6.0274
4.3287
4.3216
4.3111
4.3042
4.2934
4.2865
4.2760
4.2688
4.2600
4.2290
4.2112
4.2022
4.1934
4.1843
4.1757
4.1665
2.9868
2.9764
2.9513
2.9414
2.6475
2.6116
2.6019
2.5987
2.5522
2.5160
2.4796
1.3179
1.3001
1.2820
1.2788
1.2608
1.2431
O
H
F F
EtO2C CO2Et
3mm
0.99
1.00
2.11
0.99
3.02
1.15
1.96
0.89
1.07
1.00

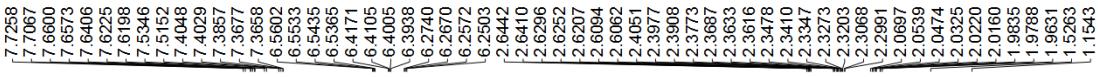

196.2988
170.2106
169.0129
138.1415
133.6250
131.4744
129.6801
128.6717
127.5598
117.9398
116.3563
114.7725
62.4588
62.4220
58.9735
38.7754
38.7417
38.7093
35.8912
34.4167
34.2712
34.1257
13.9344
13.8295
O
H
F F
EtO2C CO2Et
3mm


O
H
F
F
EtO2C
CO2Et

3mm

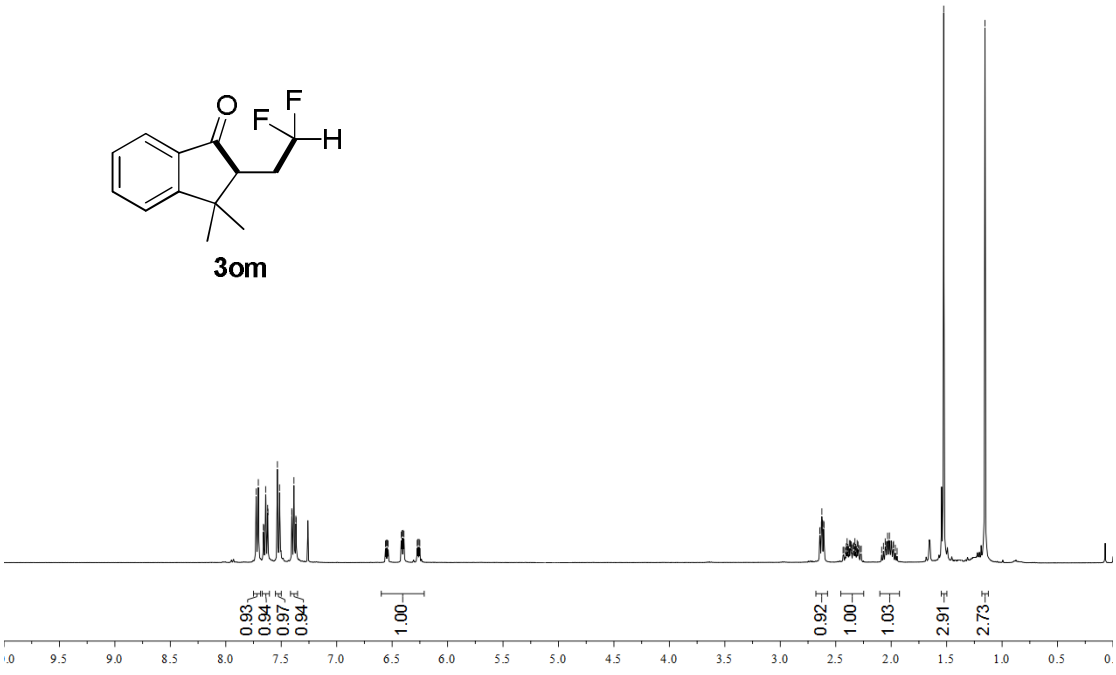

112.5834
113.0858
117.2597
117.7621

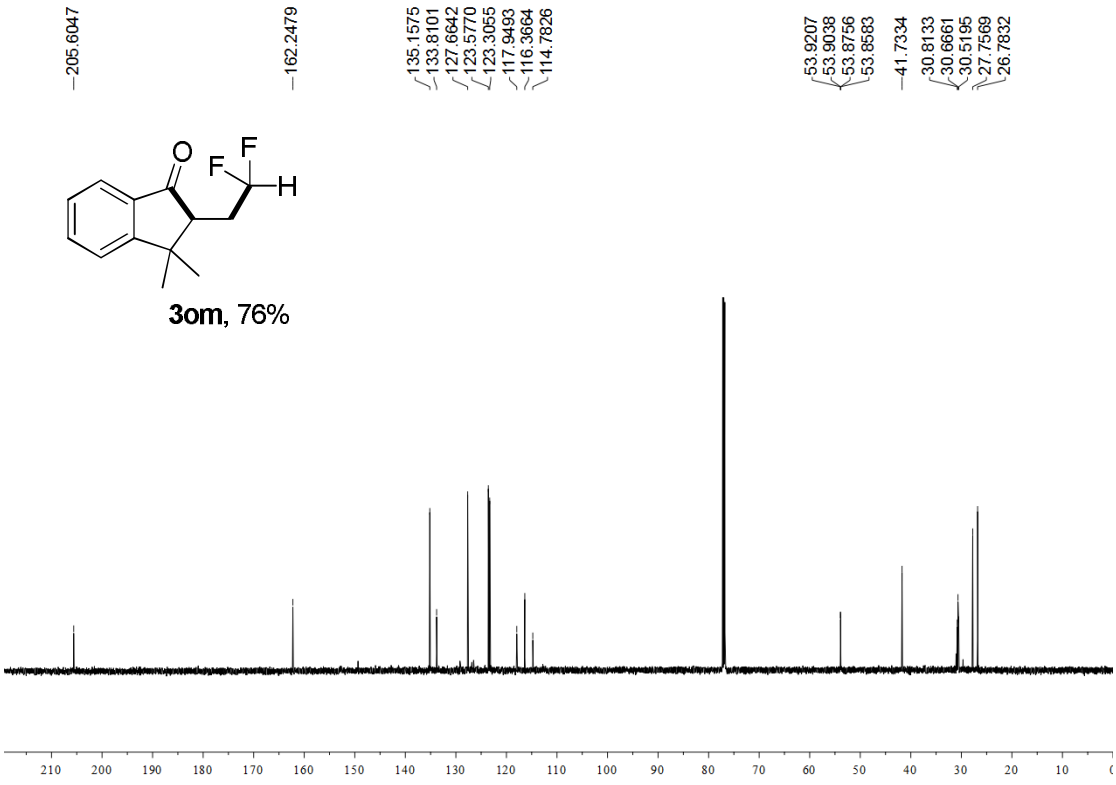

-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

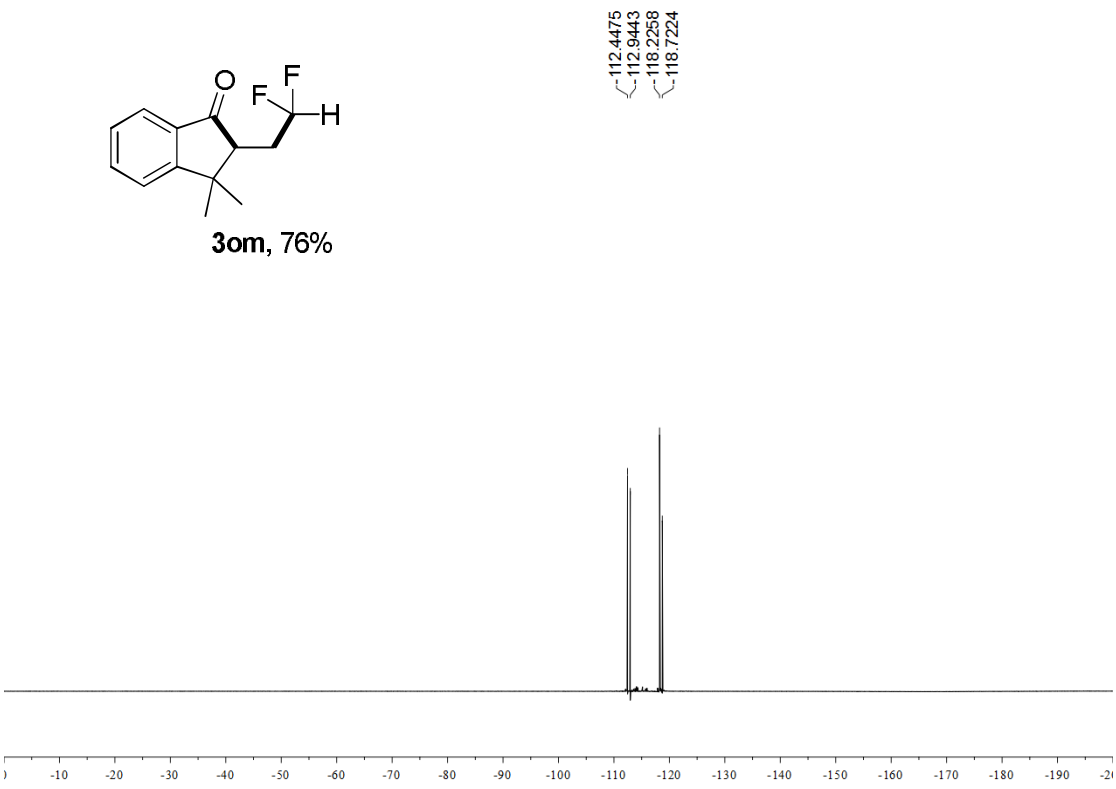

7.7258
7.7067
7.6600
7.6573
7.6406
7.6225
7.6198
7.5346
7.5152
7.4048
7.4029
7.3857
7.3677
7.3658
6.5602
6.5533
6.5435
6.5365
6.4171
6.4105
6.4005
6.3938
6.2740
6.2670
6.2572
6.2503
2.6442
2.6410
2.6296
2.6252
2.6207
2.6094
2.6062
2.4051
2.3977
2.3908
2.3773
2.3687
2.3633
2.3616
2.3478
2.3410
2.3347
2.3273
2.3203
2.3068
2.2991
2.0697
2.0539
2.0474
2.0325
2.0220
2.0160
1.9835
1.9788
1.9631
1.5263
1.1543

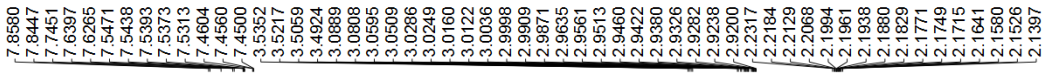
O
F
F
H

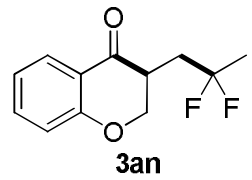
3om


0.93
0.94
0.97
0.94
1.00
0.92
1.00
1.03
2.91
2.73
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

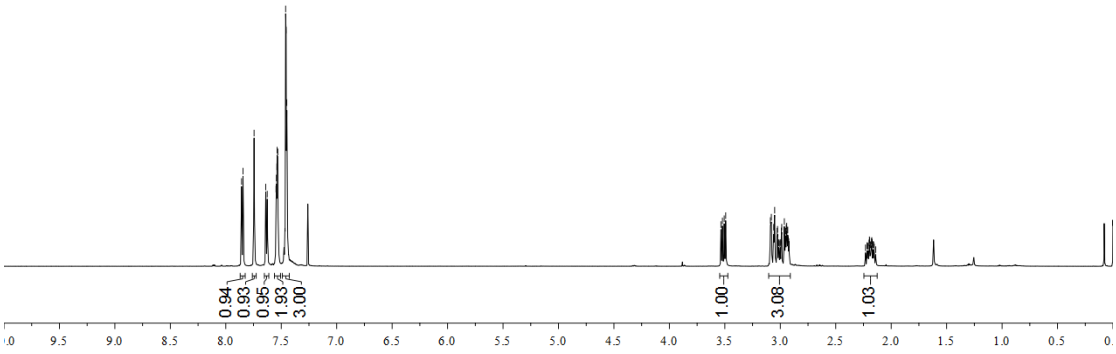

205.6047
162.2479
135.1575
133.8101
127.6642
123.5770
123.3055
117.9493
116.3664
114.7826
53.9207
53.9038
53.8756
53.8583
41.7334
30.8133
30.6661
30.5195
27.7569
26.7832
O
F
F
H
3om, 76%

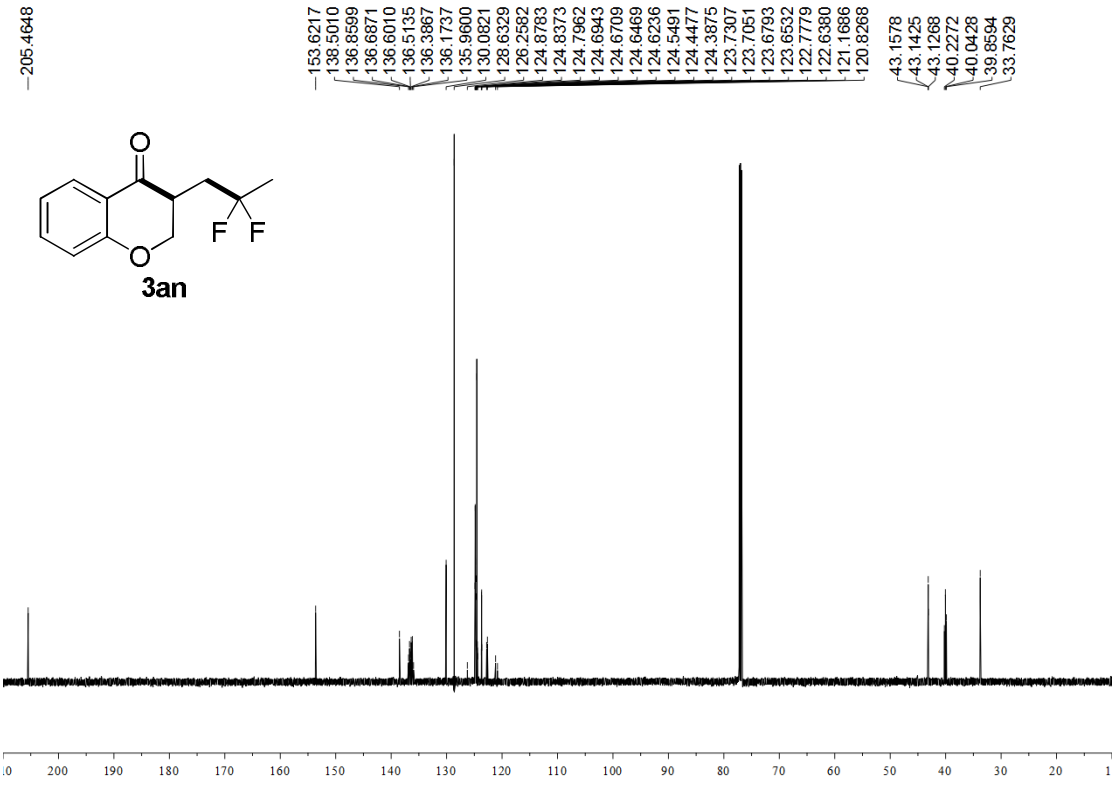

112.4475
112.9443
118.2258
118.7224
O
F
F
H
3om, 76%


7.8580
7.8447
7.7451
7.6397
7.6265
7.5471
7.5438
7.5393
7.5373
7.5313
7.4604
7.4560
7.4500
3.5352
3.5217
3.5059
3.4924
3.0889
3.0808
3.0595
3.0509
3.0286
3.0249
3.0160
3.0122
3.0036
2.9998
2.9909
2.9871
2.9635
2.9561
2.9513
2.9460
2.9422
2.9380
2.9326
2.9282
2.9238
2.9200
2.2317
2.2184
2.2129
2.2068
2.1994
2.1961
2.1938
2.1880
2.1829
2.1771
2.1749
2.1715
2.1641
2.1580
2.1526
2.1397


O
F
F
O

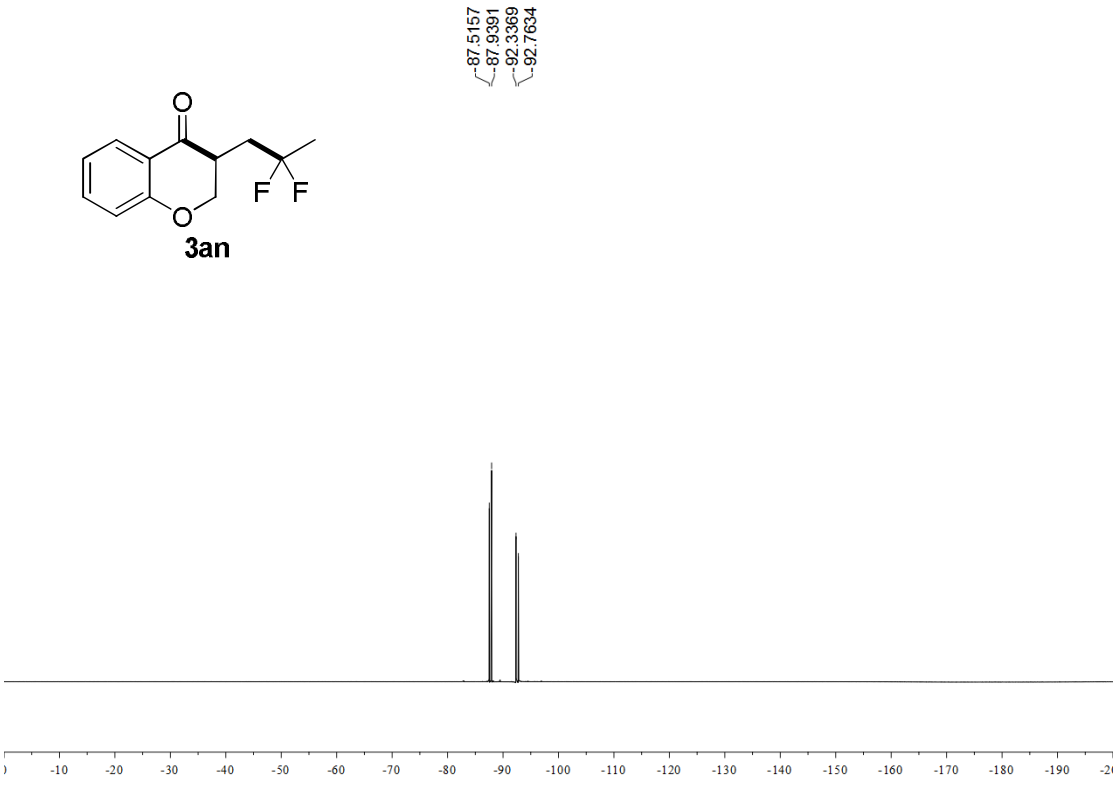
3an


0.94
0.93
0.95
1.93
3.00
1.00
3.08
1.03

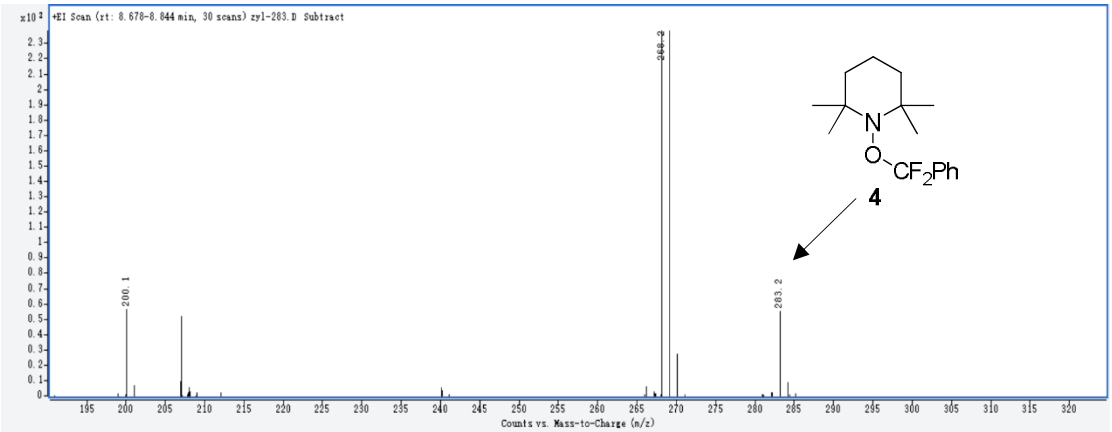

205.4648
153.6217
138.5010
136.8599
136.6871
136.6010
136.5135
136.3867
136.1737
135.9600
130.0821
128.6329
126.2582
124.8783
124.8373
124.7962
124.6943
124.6709
124.6469
124.6236
124.5491
124.4477
124.3875
123.7307
123.7051
123.6793
123.6532
122.7779
122.6380
121.1686
120.8268
43.1578
43.1425
43.1268
40.2272
40.0428
39.8594
33.7629

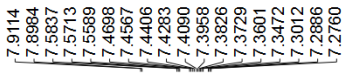
O
F
F
O

3an



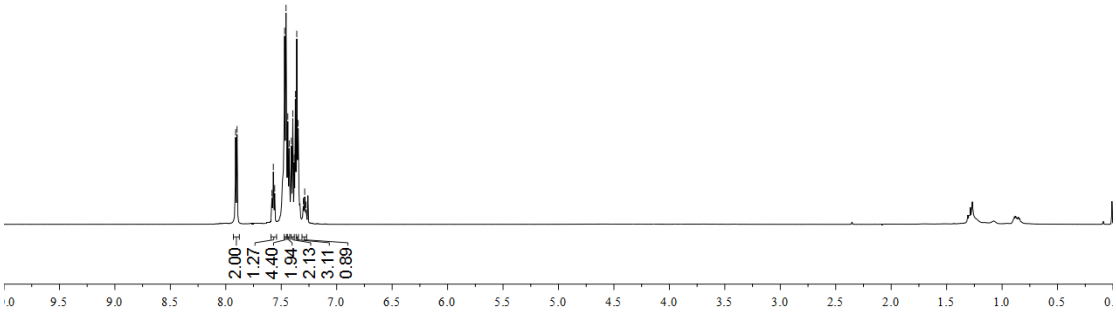
GC-MS analysis of the standard reaction in the presence of TEMPO

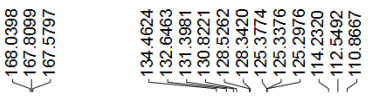




$PhI(O_2CCF_2Ph)_2$

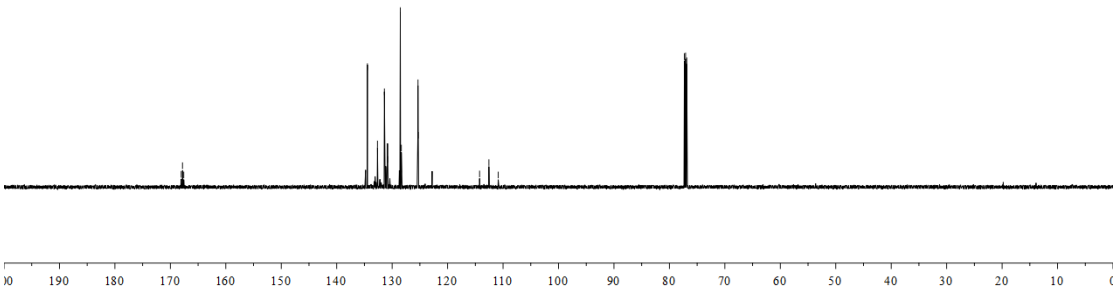
5

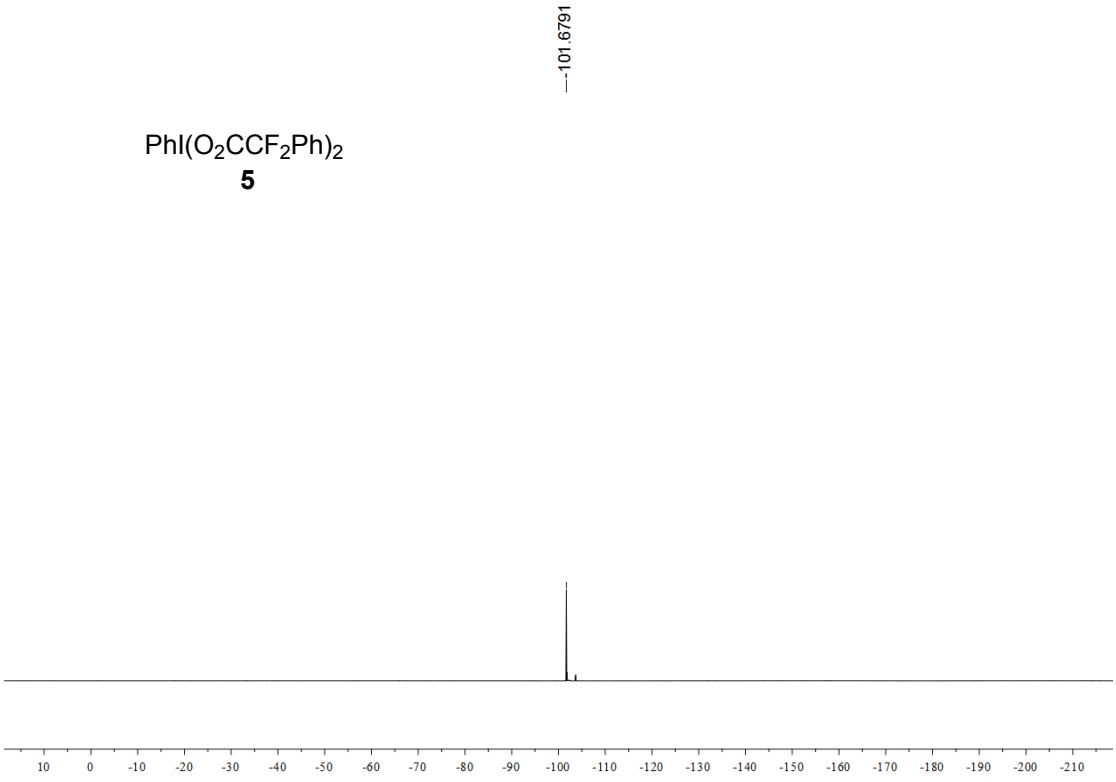




$PhI(O_2CCF_2Ph)_2$

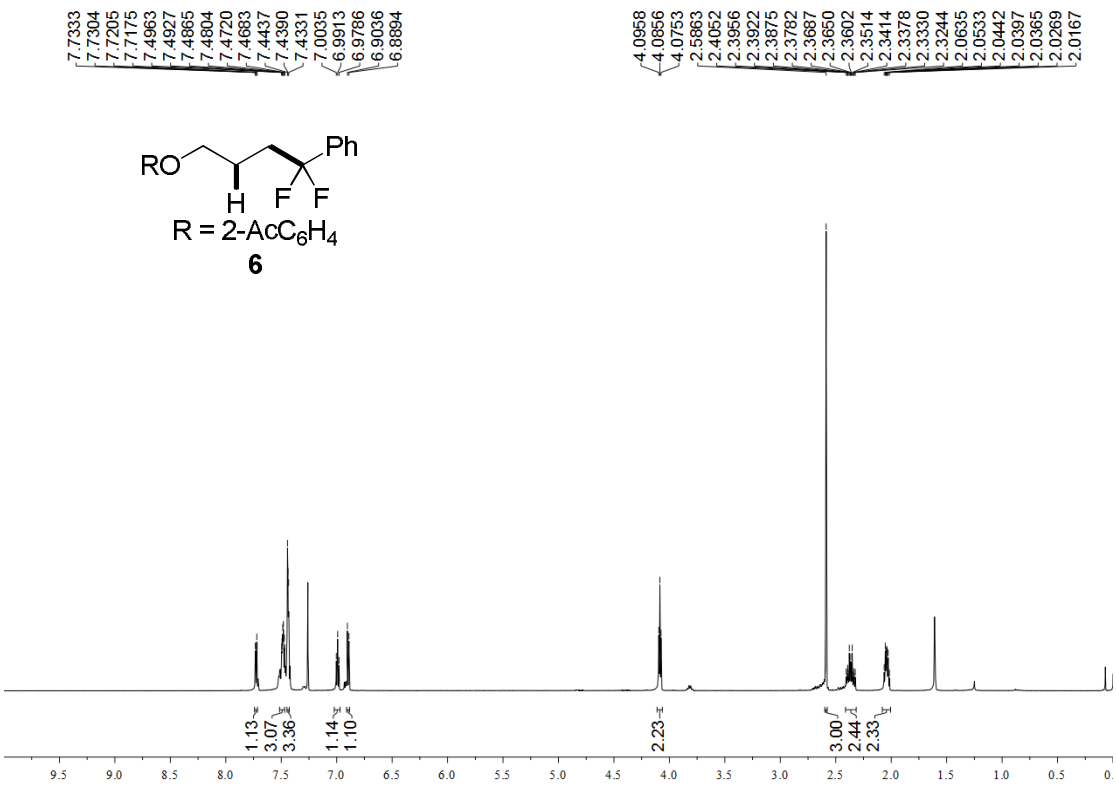
5





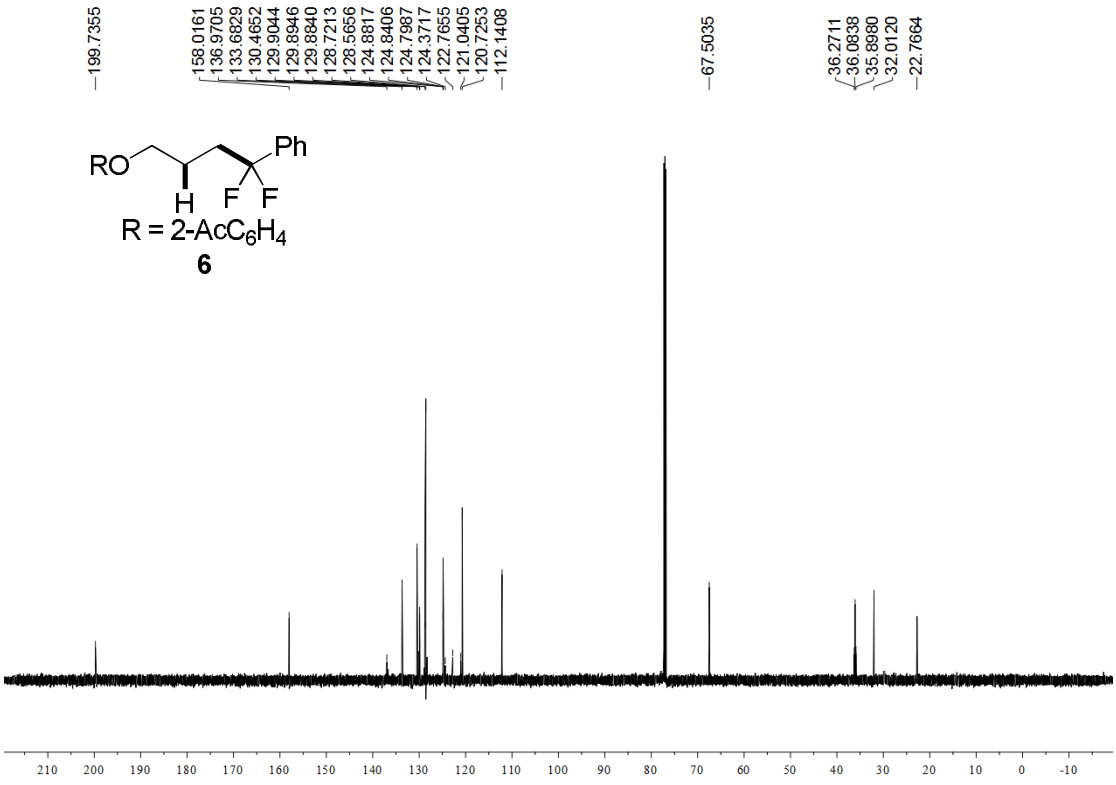
$PhI(O_2CCF_2Ph)_2$

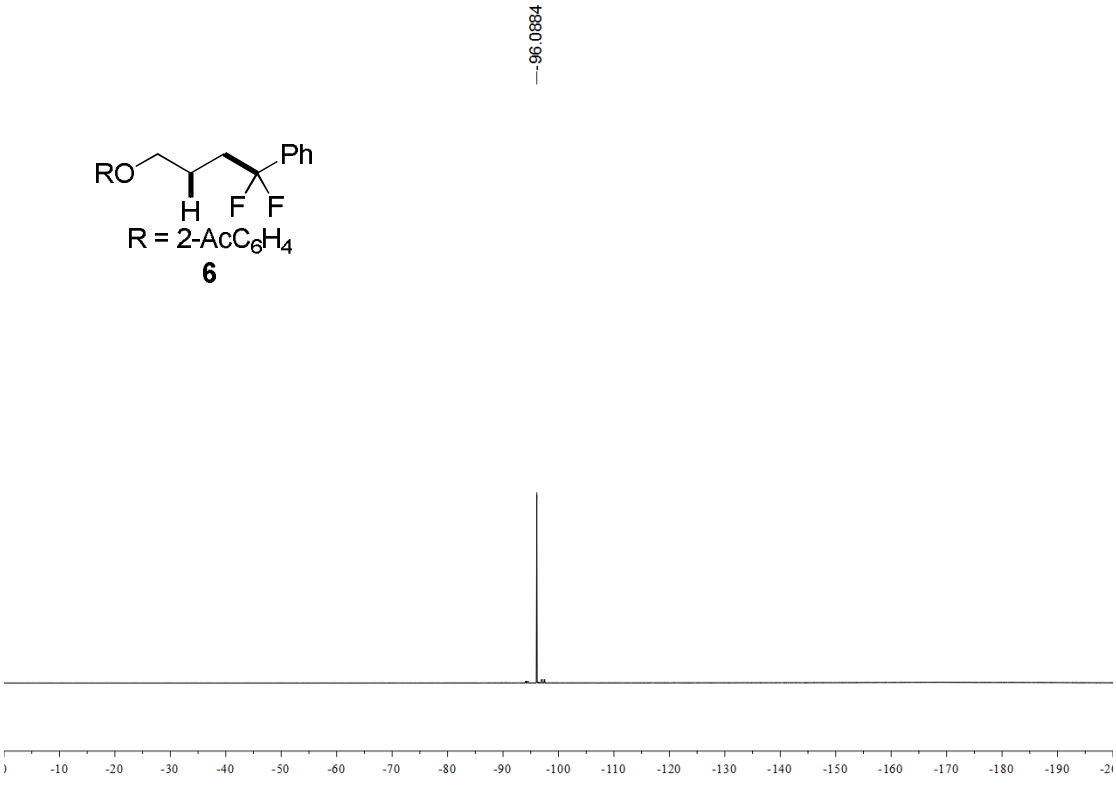
5



$R = 2\text{-}AcC_6H_4$

6


199.7355
158.0161
136.9705
133.6829
130.4652
129.9044
129.8946
129.8840
128.7213
128.5656
124.8817
124.8406
124.7987
124.3717
122.7655
121.0405
120.7253
112.1408
67.5035
36.2711
36.0838
35.8980
32.0120
22.7664
RO
Ph
H
F
F
R = 2-AcC6H4
6
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


-96.0884
RO
Ph
H
F
F
R = 2-AcC6H4
6
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190