Supporting Information

Catalytic Stereoselective Cascade Reactions of Quinols with Trifluoromethyl Ketones: Direct Access to CF_3-containing 1,3-dioxolanes

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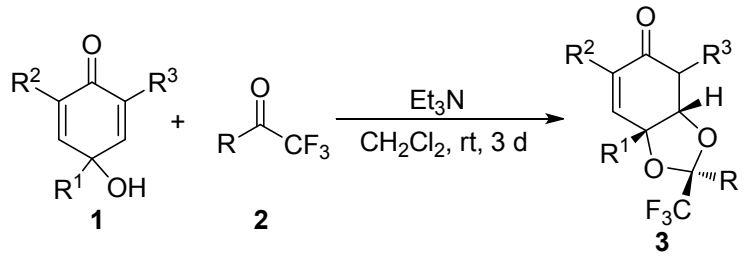
General information:

^{1}H, ^{13}C, and ^{19}F were recorded at Bruker 400 MHz (^{1}H NMR), 100 MHz (^{13}C NMR), as well as 376 MHz (^{19}F NMR). Chemical shifts were reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm, 77.0 ppm). For ^{19}F NMR, the (trifluoromethyl)benzene (δ = –63.72) was used as an external standard. Multiplicity was indicated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet), br (broad). Coupling constants were reported in Hertz (Hz). IR spectra were recorded on an AVATAR 360 FT-IR spectrometer. HRMS were recorded on an APEXIII 7.0 TESLA FTMS (ESI resource). X-ray structural analysis was conducted on the XtaLAB mini (600 W, CCD, 75mn, 0.1 electorns / pixel / sec). Chiral HPLC analysis were carried out on a Agilent 1260 infinity instrument with commercial ChiralPak columns and hexane and isopropyl alcohol as eluents.

Materials: All commercially available reagents and solvent were used without further purification. Analytical thin layer chromatography was performed on 0.25 mm silica gel plates. Silica gel (200-300 mesh) was used for flash chromatography. Trifluoromethyl ketones [1] and *p*-Quinols [2] were prepared according to literatures.

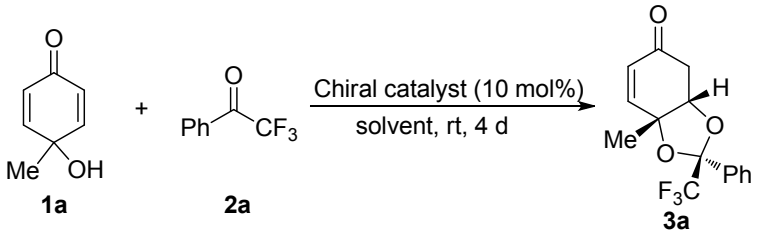
General procedure for the ketalization/oxa-Michael cascade of *p*-Quinols with Trifluoromethyl ketones:



To a solution of the *p*-Quinol **1** (0.3 mmol) and trifluoromethyl ketone **2** (0.36 mmol) in CH_2Cl_2 (1.0 mL) was added Et_3N (0.06 mmol, 20 mol%) via a syringe in one portion. Then the mixture was vigorously stirred at room temperature for 3 days. After the reaction was complete, the mixture was directly purified by column

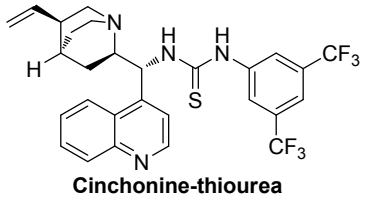
chromatography on silica gel (petroleum ether/EtOAc as the eluent) to furnish the corresponding products **3**.

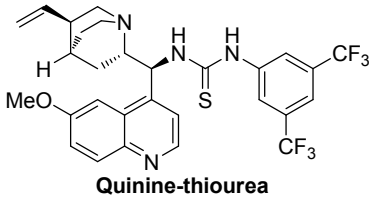
Screening of the catalyst for the enantioselective ketalization/oxa-Michael cascade[a]



Entry	Chiral catalyst	Solvent	Yield[b]	ee[c]	dr[d]
1	Cinchonine-thiourea	CH_2Cl_2	89	-25	94:6
2	Quinine-thiourea	CH_2Cl_2	84	-3	92:8
3	$(DHQD)_2AQN$	CH_2Cl_2	91	-74	90:10
4	$(DHQ)_2AQN$	CH_2Cl_2	90	60	95:5
5	$(DHQ)_2PHAL$	CH_2Cl_2	92	80	97:3

[a] Reaction conditions: **1a** (0.10 mmol), **2a** (0.15 mmol), chiral catalyst (10 mol %), solvent (1.0 mL), rt, 4 d; [b] Isolated yields; [c] Values are for the major diastereomers of **3a**; [d] Diastereomeric ratios were determined by ^{19}F NMR.



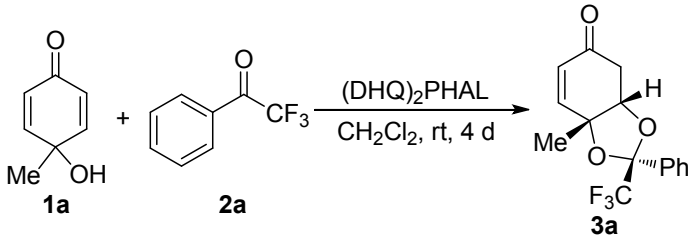


$(DHQD)_2AQN$: CAS 176298-44-5

$(DHQ)_2AQN$: CAS 176097-24-8

$(DHQ)_2PHAL$: CAS 140924-50-1

General procedure for the enantioselective ketalization/oxa-Michael cascade of *p*-Quinol 1a with Trifluoromethyl ketone 2a:

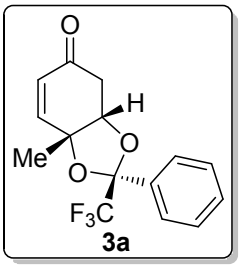


To a solution of the **1a** (0.1 mmol) and **2a** (0.15 mmol) in CH_2Cl_2 (1.0 mL) was added $(DHQ)_2PHAL$ (7.8 mg, 0.01 mmol, 10 mol%). Then the mixture was vigorously

stirred at room temperature for 4 days. After the reaction was complete, the mixture was directly purified by column chromatography on silica gel (petroleum ether/EtOAc as the eluent) to give the desired product **3a** as white solid.

27.4 mg, 92% yield; 80% ee; 97:3 dr; [Determined by HPLC analysis Chiralcel IB column, Hexane/*i*-PrOH = 90/10, Flow rate: 0.8 mL/min, UV detection at 254 nm, Retention time: t_{major}= 9.370 min, t_{minor}= 8.254 min].

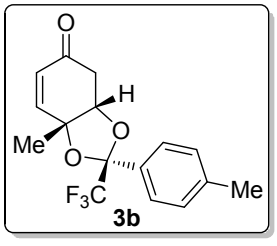
7a-methyl-2-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3a

White solid; 80.6 mg, 90% yield; 82:18 dr; mp 62−65 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.60 (s, 3H), 2.61 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.05 (d, *J* = 20.0 Hz, 1H), 4.67 (d, *J* = 3.2 Hz, 1H), 5.58 (d, *J* = 10.4 Hz, 1H), 6.25 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 7.28–7.34 (m, 3H), 7.48 (d, *J* = 7.2 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.6, 147.3, 134.4, 129.4, 127.8, 127.6, 126.5, 122.4 (q, $J_{C\text{-}F}$ = 286.4 Hz), 103.6 (q, $J_{C\text{-}F}$ = 32.1 Hz), 81.5, 78.9, 37.7, 20.9; **IR** (KBr) ν 2981, 2933, 1694, 1451, 1389, 1262, 1241, 1176, 1096, 1053, 957, 719 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.36 (s, 2.46F), –81.30 (s, 0.54F); **HRMS** (ESI) found: *m/z* 321.0717 $[M+Na]^+$; calcd. for $C_{15}H_{13}F_3O_3$+Na 321.0714.

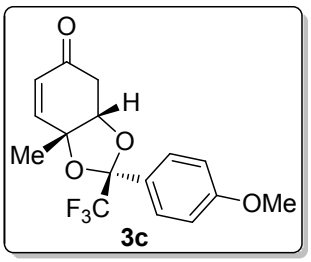
7a-methyl-2-(p-tolyl)-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3b

White solid; 81.5 mg, 87% yield; 78:22 dr; mp 45−47 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.60 (s, 3H), 2.32 (s, 3H), 2.62 (dd, *J* = 18.4 Hz, 1H), 3.06 (d, *J* = 18.4 Hz, 1H), 4.66 (s, 1H), 5.61 (d, *J* = 10.4 Hz, 1H), 6.27 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 7.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.8, 147.5, 139.4, 131.5, 128.5, 127.6, 126.4, 122.4 (q, $J_{C\text{-}F}$ = 286.7 Hz), 103.6 (q, $J_{C\text{-}F}$ = 32.2 Hz), 81.5, 78.9, 37.7, 21.1, 21.0; **IR** (KBr) ν 2979, 2928, 1694, 1616, 1515, 1456, 1387, 1310, 1241, 1178, 1094, 1049, 961, 941, 824, 734 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.44 (s, 2.34F), –81.42 (s, 0.66F); **HRMS** (ESI) found: *m/z* 335.0870 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_3$+Na 335.0871.

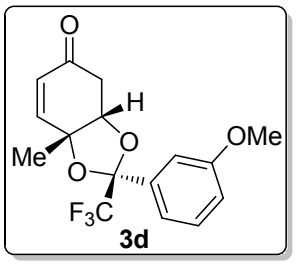
2-(4-methoxyphenyl)-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3c

Colorless oily liquid; 79.6 mg, 81% yield; 82:18 dr; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.59 (s, 3H), 2.61 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.05 (d, *J* = 18.0 Hz, 1H), 3.77 (s, 3H), 4.65 (s, 1H), 5.62 (d, *J* = 10.0 Hz, 1H), 6.26 (dd, *J* = 10.4 Hz, 1.6 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 2H), 7.38 (d, *J* = 8.4 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.9, 160.3, 147.6, 127.9, 127.5, 126.5, 122.4 (q, $J_{C\text{-}F}$ = 286.6 Hz), 113.1, 103.6 (q, $J_{C\text{-}F}$ = 32.3 Hz), 81.5, 78.9, 55.0, 37.7, 21.0; **IR** (KBr) ν 2982, 2922, 1694, 1611, 1513, 1463, 1388, 1304, 1251, 1173, 1094, 1048, 959, 834, 790 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.58 (s, 2.46F), –81.58 (s, 0.54F); **HRMS** (ESI) found: *m/z* 351.0822 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_4$+Na 351.0820.

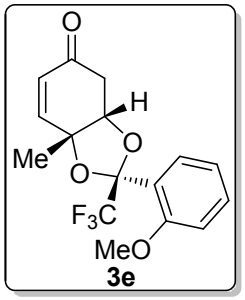
2-(3-methoxyphenyl)-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3d

White solid; 84.6 mg, 86% yield; 80:20 dr; mp 50−52 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.59 (s, 3H), 2.60 (dd, *J* = 18.0 Hz, 3.6 Hz, 1H), 3.05 (d, *J* = 17.6 Hz, 1H), 3.75 (s, 3H), 4.65 (d, *J* = 2.4 Hz, 1H), 5.61 (d, *J* = 10.4 Hz, 1H), 6.27 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 6.86 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H), 6.99 (s, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.8, 159.0, 147.4, 135.8, 128.9, 127.7, 122.4 (q, $J_{C\text{-}F}$ = 287.1 Hz), 118.9, 115.1, 112.1, 103.5 (q, $J_{C\text{-}F}$ = 32.4 Hz), 81.6, 79.0, 55.2, 37.7, 21.0; **IR** (KBr) ν 2935, 2838, 1697, 1603, 1587, 1489, 1456, 1388, 1316, 1270, 1241, 1127, 1046, 977, 898, 795, 730 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.31 (s, 2.40F), –81.23 (s, 0.60F); **HRMS** (ESI) found: *m/z* 351.0821 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_4$+Na 351.0820.

2-(2-methoxyphenyl)-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:

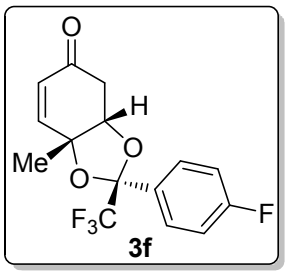


3e

White solid; 76.8 mg, 78% yield; 90:10 dr; mp 114−116 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.61 (s, 3H), 2.60 (dd, *J* = 17.6 Hz, 3.2 Hz, 1H), 3.08 (d, *J* = 17.6 Hz, 1H), 3.78 (s, 3H), 4.63 (s, 1H), 5.62 (d, *J* = 10.4 Hz, 1H), 6.32 (d, *J* = 10.0 Hz, 1H), 6.89 (t, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 7.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.0, 158.0, 147.5, 131.2, 129.1, 127.6, 122.7 (q, $J_{C\text{-}F}$ = 287.6 Hz), 122.5, 119.9, 112.8, 104.3 (q, $J_{C\text{-}F}$ = 34.1 Hz), 81.0, 78.5, 56.2, 37.7, 21.1; **IR** (KBr) ν 2922, 2841, 1693, 1601, 1585, 1489, 1456, 1436, 1388, 1285, 1253, 1173, 1119, 1081,

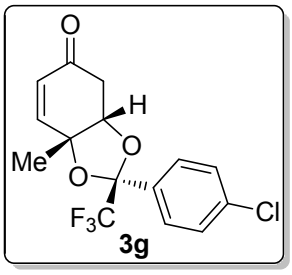
956, 937, 789, 759, 713 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.69 (s, 2.70F), –80.51 (s, 0.30F); **HRMS** (ESI) found: *m/z* 351.0818 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_4$+Na 351.0820.

2-(4-fluorophenyl)-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



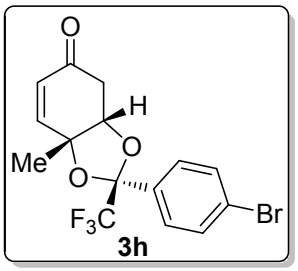
White solid; 90.1 mg, 95% yield; 82:18 dr; mp 63−65 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.65 (s, 3H), 2.66 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.03–3.13 (m, 1H), 4.72 (d, *J* = 2.4 Hz, 1H), 5.65 (dd, *J* = 10.4 Hz, 0.8 Hz, 1H), 6.30 (dd, *J* = 10.4 Hz, 2.4 Hz, 1H), 7.10–7.17 (m, 2H), 7.49–7.53 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.7, 162.6 (d, $J_{C\text{-}F}$ = 245.5 Hz), 147.3, 139.9, 128.8 (d, $J_{C\text{-}F}$ = 8.0 Hz), 128.3 (d, $J_{C\text{-}F}$ = 27.8 Hz), 127.8, 125.3 (d, $J_{C\text{-}F}$ = 37.3 Hz), 122.4 (q, $J_{C\text{-}F}$ = 286.8 Hz), 115.6 (d, $J_{C\text{-}F}$ = 21.4 Hz), 103.6 (q, $J_{C\text{-}F}$ = 32.1 Hz), 81.7, 79.1, 37.8, 21.1; **IR** (KBr) ν 3043, 2964, 2931, 1697, 1607, 1514, 1496, 1481, 1456, 1313, 1239, 1261, 1126, 1048, 968, 839, 793, 726 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.25 (s, 2.46F), –81.19 (s, 0.54F), –115.26 (s, 0.82F), –115.00 (s, 0.18F); **HRMS** (ESI) found: *m/z* 339.0622 $[M+Na]^+$; calcd. for $C_{15}H_{12}F_4O_3$+Na 339.0620.

2-(4-chlorophenyl)-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



Colorless oily liquid; 92.8 mg, 93% yield; 82:18 dr; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.59 (s, 3H), 2.63 (dd, *J* = 17.6 Hz, 2.0 Hz, 1H), 3.03 (d, *J* = 17.6 Hz, 1H), 4.66 (s, 1H), 5.61 (d, *J* = 10.4 Hz, 1H), 6.24 (dd, *J* = 10.0 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.4, 147.0, 135.6, 133.0, 128.1, 128.0, 127.7, 122.1 (q, $J_{C\text{-}F}$ = 286.7 Hz), 113.1, 103.2 (q, $J_{C\text{-}F}$ = 32.7 Hz), 81.7, 79.2, 37.6, 20.9; **IR** (KBr) ν 2988, 2937, 1694, 1599, 1491, 1402, 1307, 1247, 1179, 1099, 1045, 1018, 959, 831, 736 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.51 (s, 2.46F), –81.39 (s, 0.54F); **HRMS** (ESI) found: *m/z* 355.0327 $[M+Na]^+$; calcd. for $C_{15}H_{12}ClF_3O_3$+Na 355.0325.

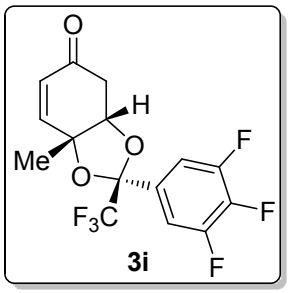
2-(4-bromophenyl)-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3h

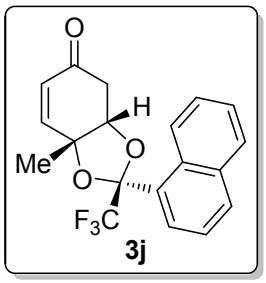
Colorless oily liquid; 102.9 mg, 91% yield; 80:20 dr; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.60 (s, 3H), 2.62 (dd, *J* = 18.0 Hz, 3.6 Hz, 1H), 3.04 (dd, *J* = 18.0 Hz, 1.6 Hz, 1H), 4.66 (d, *J* = 2.4 Hz, 1H), 5.62 (d, *J* = 10.4 Hz, 1H), 6.24 (dd, *J* = 10.4 Hz, 1.6 Hz, 1H), 7.34 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.8 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.5, 147.1, 133.5, 131.1, 128.3, 127.8, 124.1, 122.1 (q, $J_{C\text{-}F}$ = 286.6 Hz), 103.2 (q, $J_{C\text{-}F}$ = 32.6 Hz), 81.7, 79.2, 37.7, 21.0; **IR** (KBr) ν 2964, 2930, 1703, 1697, 1592, 1488, 1456, 1397, 1313, 1260, 1176, 1093, 1013, 961, 941, 826, 790, 736 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.47 (s, 2.40F), –81.35 (s, 0.60F); **HRMS** (ESI) found: *m/z* 398.9820 $[M+Na]^+$; calcd. for $C_{15}H_{12}BrF_3O_3$+Na 398.9820.

7a-methyl-2-(trifluoromethyl)-2-(3,4,5-trifluorophenyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



White solid; 96.1 mg, 91% yield; 84:16 dr; mp 88−90 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.67 (s, 3H), 2.69 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.10 (dd, *J* = 18.0 Hz, 1.6 Hz, 1H), 4.73 (d, *J* = 2.0 Hz, 1H), 5.74 (d, *J* = 10.4 Hz, 1H), 6.33 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 7.17 (t, *J* = 7.2 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.1, 150.7 (dddd, $J_{C\text{-}F}$ = 250.0 Hz, 3.5 Hz), 146.6, 140.6 (dt, $J_{C\text{-}F}$ = 253.4 Hz, 15.1 Hz), 130.9 (q, $J_{C\text{-}F}$ = 4.5 Hz), 128.1, 121.9 (m), 111.5 (dd, $J_{C\text{-}F}$ = 23.1 Hz, 6.5 Hz), 102.4 (q, $J_{C\text{-}F}$ = 33.1 Hz), 82.1, 79.7, 37.6, 21.0; **IR** (KBr) ν 3088, 2983, 2935, 1697, 1626, 1601, 1532, 1443, 1358, 1316, 1241, 1177, 1128, 1088, 1041, 1012, 851, 805, 742 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.50 (s, 2.52F), –81.29 (s, 0.48F), –133.00 (d, *J* = 20.3 Hz, 1.68F), –132.70 (d, *J* = 20.3 Hz, 0.32F), –157.93 (t, *J* = 20.3 Hz, 0.84F), –157.48 (t, *J* = 20.3 Hz, 0.16F); **HRMS** (ESI) found: *m/z* 375.0431 $[M+Na]^+$; calcd. for $C_{15}H_{10}F_6O_3$+Na 375.0432.

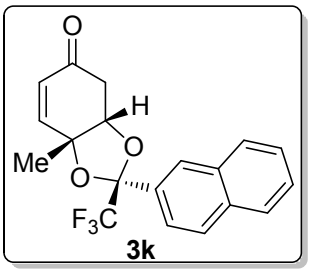
7a-methyl-2-(naphthalen-1-yl)-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



White solid; 83.6 mg, 80% yield; 78:22 dr; mp 45−47 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.62 (s, 3H), 2.62 (d, *J* = 18.0 Hz, 1H), 3.11 (d, *J* = 18.0 Hz, 1H), 4.75 (s, 1H), 5.47 (d, *J* = 10.4 Hz, 1H), 6.21 (d, *J* = 9.6 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 7.46–7.52 (m, 2H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 8.0 Hz, 2H), 8.62 (d, *J* = 5.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.4, 147.0, 133.8, 130.8, 130.4, 130.2,

128.3, 127.5, 126.9, 126.5, 126.0, 125.4, 124.1, 122.8 (q, $J_{C\text{-}F}$ = 287.9 Hz), 105.3 (q, $J_{C\text{-}F}$ = 33.1 Hz), 81.2, 78.9, 37.5, 20.7; **IR** (KBr) ν 3052, 2980, 2932, 1694, 1510, 1388, 1307, 1240, 1176, 1121, 1082, 1050, 1016, 941, 803, 776 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.01 (s, 2.34F), –79.93 (s, 0.66F); **HRMS** (ESI) found: *m/z* 371.0868 $[M+Na]^+$; calcd. for $C_{19}H_{15}F_3O_3$+Na 371.0871.

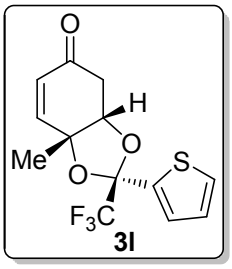
7a-methyl-2-(naphthalen-2-yl)-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3k

White solid; 86.7 mg, 83% yield; 76:24 dr; mp 105−107 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.64 (s, 3H), 2.64 (dd, *J* = 18.0 Hz, 2.8 Hz, 1H), 3.13 (d, *J* = 18.8 Hz, 1H), 4.73 (s, 1H), 5.55 (d, *J* = 10.4 Hz, 1H), 6.28 (d, *J* = 10.0 Hz, 1H), 7.52 (t, *J* = 4.0 Hz, 2H), 7.62 (d, *J* = 8.4 Hz, 1H), 7.81–7.89 (m, 3H), 8.04 (s, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.6, 147.3, 133.5, 132.2, 131.8, 128.5, 127.7, 127.6, 127.5, 126.9, 126.5, 126.3, 123.6, 122.5 (q, $J_{C\text{-}F}$ = 286.7 Hz), 103.7 (q, $J_{C\text{-}F}$ = 32.6 Hz), 81.6, 79.1, 37.6, 20.9; **IR** (KBr) ν 3060, 2963, 2928, 1694, 1508, 1455, 1388, 1313, 1275, 1240, 1179, 1127, 1089, 1048, 935, 819, 750 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.01 (s, 2.28F), –80.92 (s, 0.72F); **HRMS** (ESI) found: *m/z* 371.0873 $[M+Na]^+$; calcd. for $C_{19}H_{15}F_3O_3$+Na 371.0871.

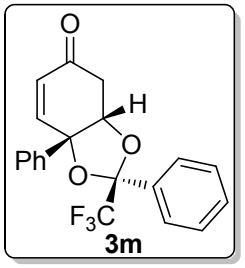
7a-methyl-2-(thiophen-2-yl)-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3l

White solid; 65.7 mg, 72% yield; 75:25 dr; mp 71−73 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.62 (s, 3H), 2.66 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.07 (d, *J* = 18.0 Hz, 1H), 4.66 (s, 1H), 5.76 (d, *J* = 10.4 Hz, 1H), 6.41 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 6.97 (t, *J* = 4.0 Hz, 1H), 7.19 (d, *J* = 2.8 Hz, 1H), 7.32 (d, *J* = 5.2 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.5, 147.1, 136.8, 127.8, 127.5, 127.2, 126.9, 122.0 (q, $J_{C\text{-}F}$ = 286.4 Hz), 102.7 (q, $J_{C\text{-}F}$ = 33.5 Hz), 81.9, 79.6, 37.6, 21.1; **IR** (KBr) ν 2985, 2931, 1694, 1589, 1490, 1328, 1241, 1179, 1088, 1075, 923, 798, 721 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.63 (s, 2.25F), –81.59 (s, 0.75F); **HRMS** (ESI) found: *m/z* 327.0279 $[M+Na]^+$; calcd. for $C_{13}H_{11}F_3O_3S$+Na 327.0279.

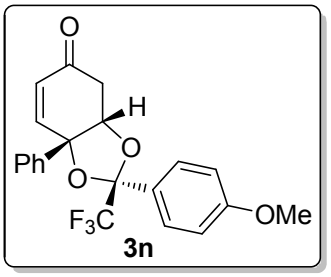
2,7a-diphenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3m

White solid; 91.9 mg, 85% yield; 94:6 dr; mp 133−135 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.67 (dd, *J* = 18.0 Hz, 2.8 Hz, 1H), 3.06 (d, *J* = 18.0 Hz, 1H), 4.80 (s, 1H), 5.89 (d, *J* = 10.4 Hz, 1H), 6.41 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 7.36–7.40 (m, 3H), 7.44–7.50 (m, 3H), 7.57–7.60 (m, 4H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.0, 145.1, 135.1, 134.3, 129.6, 129.3, 129.2, 129.1, 127.9, 126.6, 125.4, 122.6 (q, $J_{C\text{-}F}$ = 287.1 Hz), 104.2 (q, $J_{C\text{-}F}$ = 32.3 Hz), 83.3, 82.6, 37.0; **IR** (KBr) ν 3062, 2931, 1694, 1494, 1450, 1385, 1313, 1273, 1176, 1095, 1063, 1031, 955, 762, 725, 697 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.40 (s, 2.82F), –81.08 (s, 0.18F); **HRMS** (ESI) found: *m/z* 383.0874 $[M+Na]^+$; calcd. for $C_{20}H_{15}F_3O_3$+Na 383.0871.

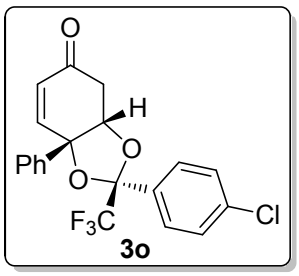
2-(4-methoxyphenyl)-7a-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3n

White solid; 92.5 mg, 79% yield; 86:14 dr; mp 131−133 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.65 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.05 (dd, *J* = 18.0 Hz, 2.0 Hz, 1H), 3.81 (s, 3H), 4.77 (d, *J* = 2.4 Hz, 1H), 5.91 (d, *J* = 10.4 Hz, 1H), 6.40 (dd, *J* = 10.4 Hz, 2.4 Hz, 1H), 6.87 (d, *J* = 8.8 Hz, 2H), 7.41–7.48 (m, 5H), 7.53–7.56 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.1, 160.5, 145.3, 135.2, 129.3, 129.2, 129.1, 128.0, 126.5, 125.4, 122.7 (q, $J_{C\text{-}F}$ = 286.7 Hz), 113.3, 104.3 (q, $J_{C\text{-}F}$ = 32.7 Hz), 83.3, 82.6, 55.1, 37.1; **IR** (KBr) ν 3062, 2963, 2936, 2840, 1697, 1611, 1514, 1449, 1385, 1304, 1252, 1173, 1098, 1014, 956, 834, 760, 699 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.60 (s, 2.58F), –81.35 (s, 0.42F); **HRMS** (ESI) found: *m/z* 413.0975 $[M+Na]^+$; calcd. for $C_{21}H_{17}F_3O_4$+Na 413.0977.

2-(4-chlorophenyl)-7a-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:

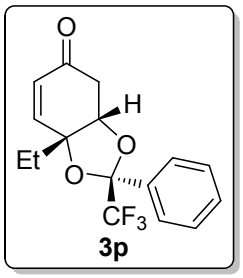


3o

White solid; 105.3 mg, 89% yield; 97:3 dr; mp 140−142 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 2.66 (dd, *J* = 18.0 Hz, 3.2 Hz, 1H), 3.05 (dd, *J* = 18.0 Hz, 2.0 Hz, 1H), 4.79 (d, *J* = 2.4 Hz, 1H), 5.92 (d, *J* = 10.4 Hz, 1H), 6.39 (dd, *J* = 10.4 Hz, 2.4 Hz, 1H), 7.34 (d, *J* = 8.8 Hz, 2H), 7.43–7.55 (m, 7H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.7, 144.8, 135.9, 134.9, 133.0, 129.5, 129.4, 129.1, 128.3, 128.1, 125.4, 122.4 (q, $J_{C\text{-}F}$ = 286.8

Hz), 103.9 (q, $J_{C\text{-}F}$ = 32.8 Hz), 83.5, 82.8, 37.0; **IR** (KBr) ν 3063, 2933, 1694, 1600, 1493, 1449, 1403, 1385, 1313, 1272, 1219, 1177, 1016, 1001, 958, 941, 830, 792, 739, 698 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.51 (s, 2.91F), –81.12 (s, 0.09F); **HRMS** (ESI) found: *m/z* 417.0484 $[M+Na]^+$; calcd. for $C_{20}H_{14}ClF_3O_3$+Na 417.0481.

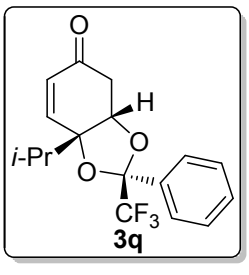
7a-ethyl-2-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3p

White solid; 82.5 mg, 88% yield; 80:20 dr; mp 44−46 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.08 (t, *J* = 7.6 Hz, 3H), 1.88–2.02 (m, 2H), 2.60 (dd, *J* = 18.0 Hz, 3.6 Hz, 1H), 3.06 (dd, *J* = 18.0 Hz, 2.4 Hz, 1H), 4.72 (d, *J* = 2.4 Hz, 1H), 5.65 (d, *J* = 10.4 Hz, 1H), 6.26 (dd, *J* = 10.4 Hz, 2.4 Hz, 1H), 7.28–7.36 (m, 3H), 7.48 (d, *J* = 6.8 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.0, 146.7, 134.6, 129.4, 128.5, 127.7, 126.5, 122.4 (q, $J_{C\text{-}F}$ = 286.9 Hz), 103.4 (q, $J_{C\text{-}F}$ = 32.5 Hz), 81.3, 79.9, 38.2, 28.0, 7.6; **IR** (KBr) ν 2976, 2943, 1693, 1451, 1463, 1386, 1266, 1176, 1095, 1053, 1031, 954, 919, 763, 722, 697, 664 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.15 (s, 2.40F), –81.40 (s, 0.60F); **HRMS** (ESI) found: *m/z* 335.0869 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_3$+Na 335.0871.

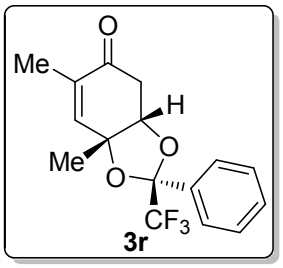
7a-isopropyl-2-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3q

White solid; 85.2 mg, 87% yield; 75:25 dr; mp 43−45 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.05 (d, *J* = 6.8 Hz, 3H), 1.11 (d, *J* = 6.8 Hz, 3H), 2.17–2.28 (m, 1H), 2.61 (dd, *J* = 18.4 Hz, 3.6 Hz, 1H), 3.04 (d, *J* = 17.6 Hz, 1H), 4.75 (dd, *J* = 2.0 Hz, 1H), 5.70 (d, *J* = 10.4 Hz, 1H), 6.27 (dd, *J* = 10.4 Hz, 2.0 Hz, 1H), 7.26–7.34 (m, 3H), 7.47 (d, *J* = 7.6 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.1, 145.4, 134.8, 129.6, 129.3, 127.7, 126.4, 122.3 (q, $J_{C\text{-}F}$ = 286.9 Hz), 103.1 (q, $J_{C\text{-}F}$ = 32.3 Hz), 83.3, 79.0, 39.0, 33.6, 17.4, 16.5; **IR** (KBr) ν 2970, 2876, 1693, 1472, 1451, 1390, 1311, 1272, 1245, 1104, 1063, 1030, 955, 763, 723, 697, 664 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.04 (s, 2.25F), –81.49 (s, 0.75F); **HRMS** (ESI) found: *m/z* 349.1028 $[M+Na]^+$; calcd. for $C_{17}H_{17}F_3O_3$+Na 349.1027.

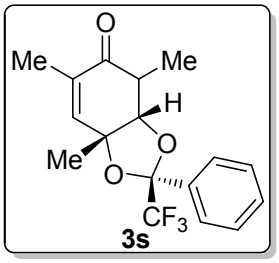
6,7a-dimethyl-2-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3r

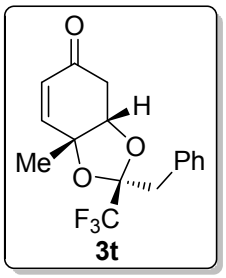
White solid; 83.4 mg, 89% yield; 80:20 dr; mp 76−78 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.33 (s, 3H), 1.58 (s, 3H), 2.59 (dd, *J* = 17.6 Hz, 3.2 Hz, 1H), 3.08 (d, *J* = 17.6 Hz, 1H), 4.61 (s, 1H), 5.96 (s, 1H), 7.30–7.34 (m, 3H), 7.44–7.46 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 194.3, 142.6, 134.9, 134.7, 129.3, 127.7, 126.5, 122.4 (q, $J_{C\text{-}F}$ = 286.0 Hz), 103.5 (q, $J_{C\text{-}F}$ = 32.1 Hz), 81.8, 79.7, 38.0, 21.2, 14.9; **IR** (KBr) ν 2980, 2926, 1688, 1450, 1377, 1363, 1319, 1259, 1177, 1096, 1059, 957, 919, 721, 697, 664 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.46 (s, 2.40F), –81.42 (s, 0.60F); **HRMS** (ESI) found: *m/z* 335.0873 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_3$+Na 335.0871.

4,6,7a-trimethyl-2-phenyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



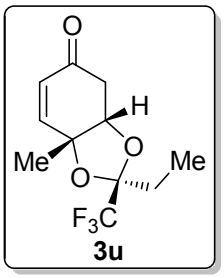
White solid; 79.3 mg, 81% yield; >99:1 dr (recrystallization); mp 81−82 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.31 (s, 3H), 1.43 (d, J = 6.8 Hz, 3H), 1.60 (s, 3H), 2.61 (dq, J = 6.8 Hz, 2.4 Hz, 1H), 4.49 (s, 1H), 5.88 (s, 1H), 7.28–7.31 (m, 3H), 7.41–7.43 (m, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 197.2, 141.2, 135.1, 134.7, 129.2, 127.7, 126.5, 122.4 (q, $J_{C\text{-}F}$ = 286.2 Hz), 103.6 (q, $J_{C\text{-}F}$ = 32.3 Hz), 86.8, 80.0, 41.8, 21.1, 15.0, 11.9; **IR** (KBr) ν 2990, 2906, 1685, 1452, 1385, 1312, 1175, 1161, 1100, 1043, 965, 719, 698, 663 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –82.50 (s, 3F); **HRMS** (ESI) found: *m/z* 349.1029 $[M+Na]^+$; calcd. for $C_{17}H_{17}F_3O_3$+Na 349.1027.

2-benzyl-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



White solid; 74.9 mg, 80% yield; 85:15 dr; mp 58−60 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 1.44 (s, 3H), 2.42 (dd, J = 14.8 Hz, 3.2 Hz, 1H), 2.88 (d, J = 18.0 Hz, 1H), 3.03 (dd, J = 28.0 Hz, 13.6 Hz, 2H), 4.54 (s, 1H), 4.99 (d, J = 10.0 Hz, 1H), 5.78 (d, J = 10.0 Hz, 1H), 7.04–7.17 (m, 2H), 7.18–7.27 (m, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.4, 146.0, 131.9, 131.8, 128.0, 127.3, 124.9, 123.3 (q, $J_{C\text{-}F}$ = 290.0 Hz), 104.5 (q, $J_{C\text{-}F}$ = 30.3 Hz), 81.0, 78.4, 37.5, 37.0, 22.2; **IR** (KBr) ν 3035, 2978, 2935, 1693, 1498, 1456, 1389, 1243, 1158, 1044, 997, 785, 726, 699 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.68 (s, 2.55F), –81.08 (s, 0.45F); **HRMS** (ESI) found: *m/z* 335.0873 $[M+Na]^+$; calcd. for $C_{16}H_{15}F_3O_3$+Na 335.0871.

2-ethyl-7a-methyl-2-(trifluoromethyl)-3a,4-dihydrobenzo[d][1,3]dioxol-5(7aH)-one:



3u

Colorless liquid; 51.1 mg, 68% yield; 80:20 dr; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 0.85 (t, *J* = 6.4 Hz, 3H), 1.55 (s, 3H), 1.78 (d, *J* = 6.8 Hz, 2H), 2.61 (dd, *J* = 16.0 Hz, 1.6 Hz, 1H), 2.97 (d, *J* = 18.0 Hz, 1H), 4.60 (s, 1H), 5.98 (d, *J* = 10.0 Hz, 1H), 6.55 (d, *J* = 10.4 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ 193.9, 148.6, 127.2, 123.3 (q, $J_{C\text{-}F}$ = 289.3 Hz), 105.7 (q, $J_{C\text{-}F}$ = 30.4 Hz), 80.8, 78.3, 37.8, 25.7, 21.9, 6.7; **IR** (KBr) ν 2984, 2944, 1695, 1466, 1389, 1320, 1256, 1169, 1122, 1088, 1057, 1027, 962, 794, 727 cm^{-1}; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –81.19 (s, 2.40F), –80.49 (s, 0.60F); **HRMS** (ESI) found: *m/z* 273.0717 $[M+Na]^+$; calcd. for $C_{11}H_{11}F_3O_3$+Na 273.0714.

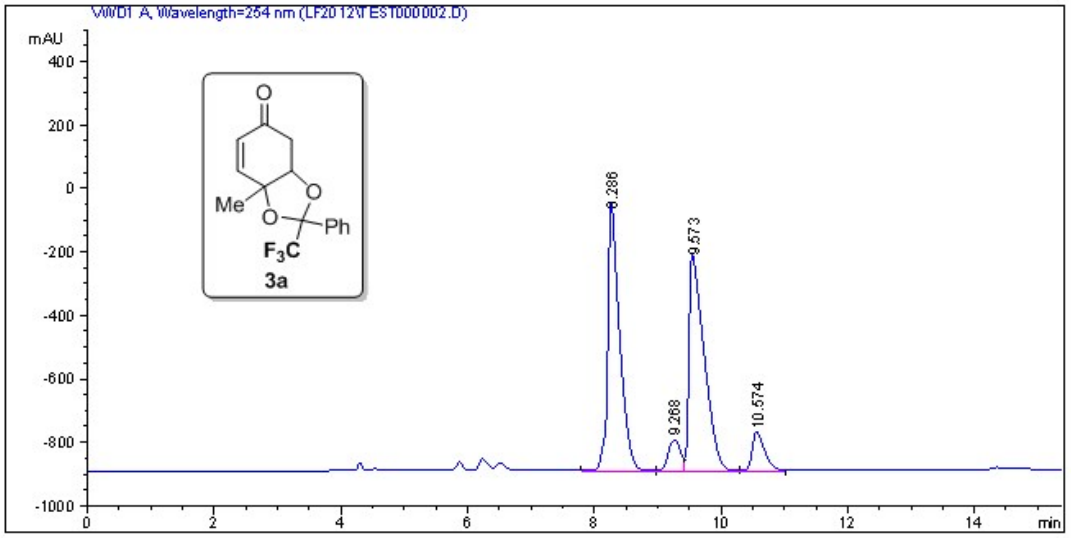
References:

[1] a) Creary, X. *J. Org. Chem.* **1987**, *52*, 5026. b) Chong, J. M.; Mar, E. K. *J. Org. Chem.* **1991**, *56*, 893. c) Prakash, G. K. S.; Panja, C.; Vaghoo, H.; Surampudi, V.; Kultyshev, R.; Mandal, M.; Rasul, G.; Mathew, T.; Olah, G. A. *J. Org. Chem.* **2006**, *71*, 6806. d) Cheng, H.; Pei, Y.; Leng, F.; Li, J.; Liang, A.; Zou, D.; Wu, Y.; Wu, Y. *Tetrahedron Lett.* **2013**, *54*, 4483.

[2] a) Novak, M.; Glover, S. A. *J. Am. Chem. Soc.* **2004**, *126*, 7748. b) Carreño, M. C.; González-López, M.; Urbano, A. *Angew. Chem., Int. Ed.* **2006**, *45*, 2737. c) Tello-Aburto, R.; Kalstabakken, K. A.; Volp, K. A.; Harned, A. M. *Org. Biomol. Chem.* **2011**, *9*, 7849.

Copies of HPLC spectra of racemic /chiral product 3a:

Sample Info : 254nm, IB, i-PrOH : Hexane =10:90, 0.8mL/min



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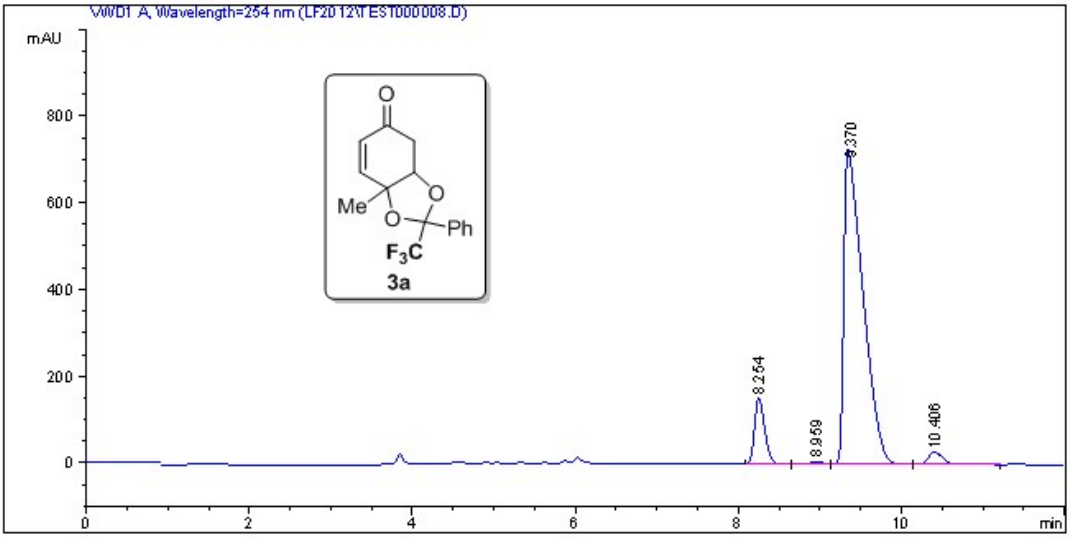
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.286	VV	0.1928	1.09875e4	812.18024	43.2371
2	9.268	VV	0.2093	1256.44019	97.05727	6.1442
3	9.573	VV	0.2685	1.13464e4	666.58215	43.6493
4	10.574	VV	0.2346	1821.90137	120.53816	6.9694

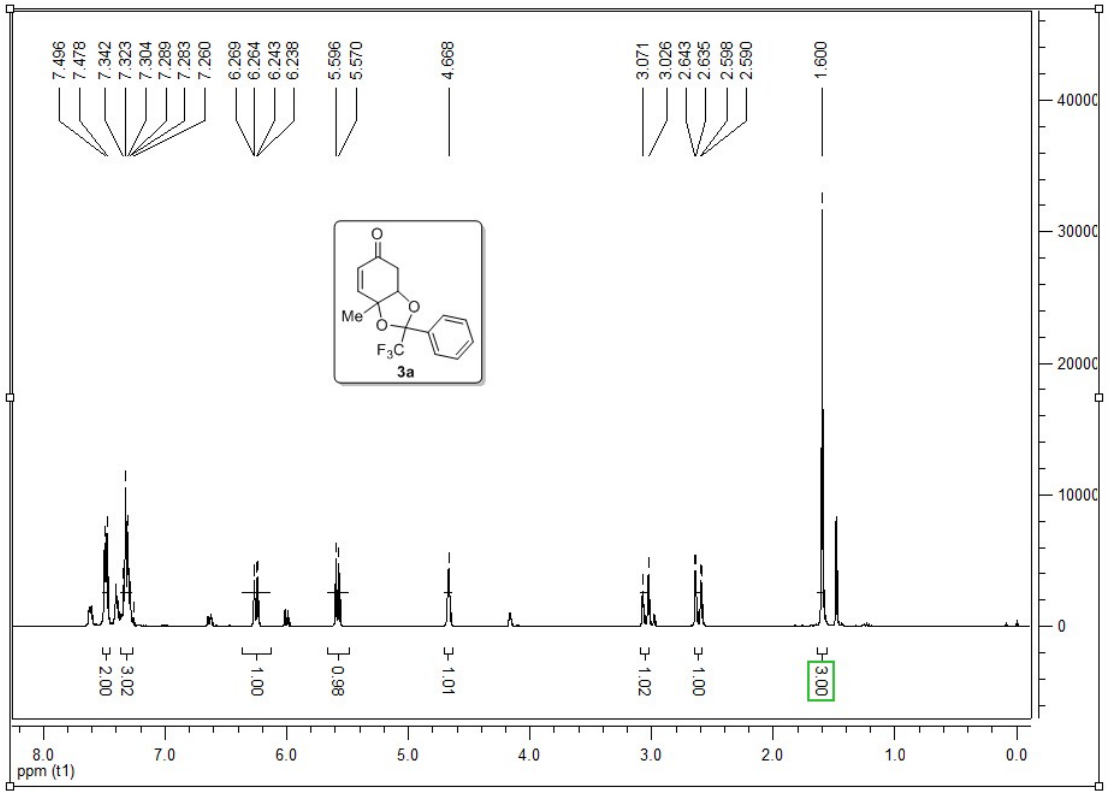
Sample Info : 245nm, IB, i-PrOH : Hexane =10:90, 0.8mL/min

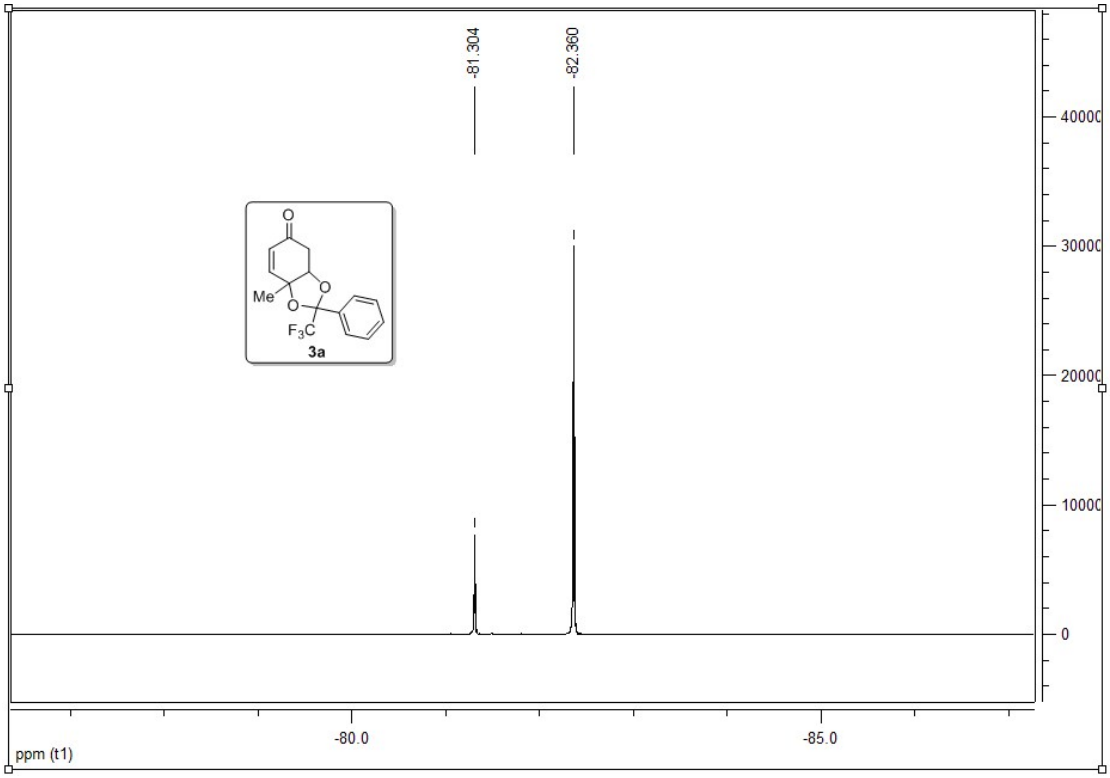


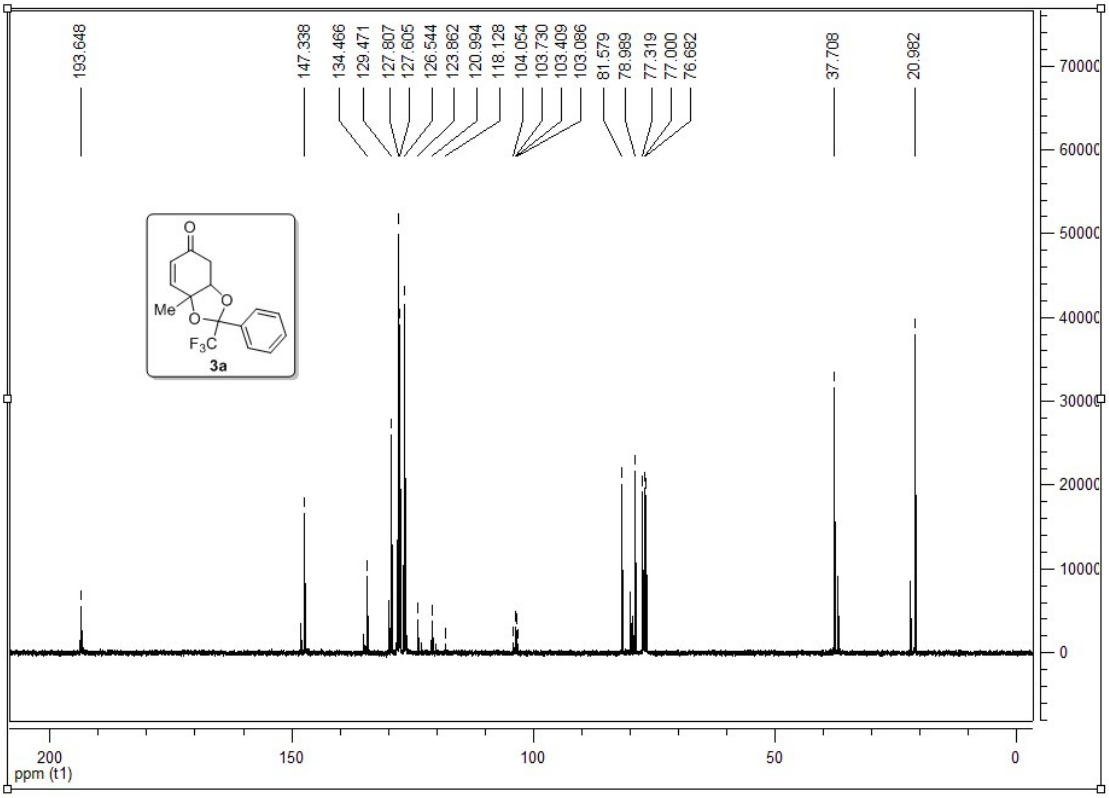
Signal 1: VWD1 A, Wavelength=254 nm

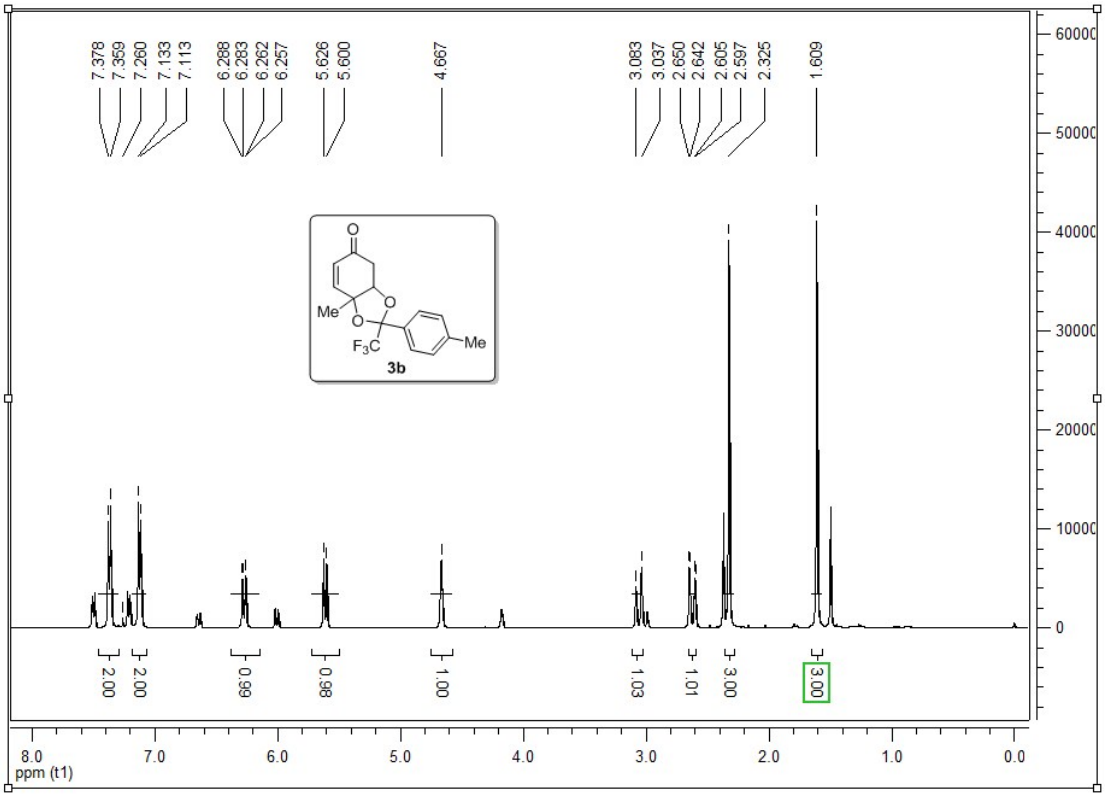
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.254	BV	0.1379	1331.74719	150.90837	9.8191
2	8.959	VV	0.1731	75.75884	6.98477	0.5586
3	9.370	VV	0.2676	1.17889e4	695.67877	86.9210
4	10.406	VV	0.1977	366.36929	28.27770	2.7013

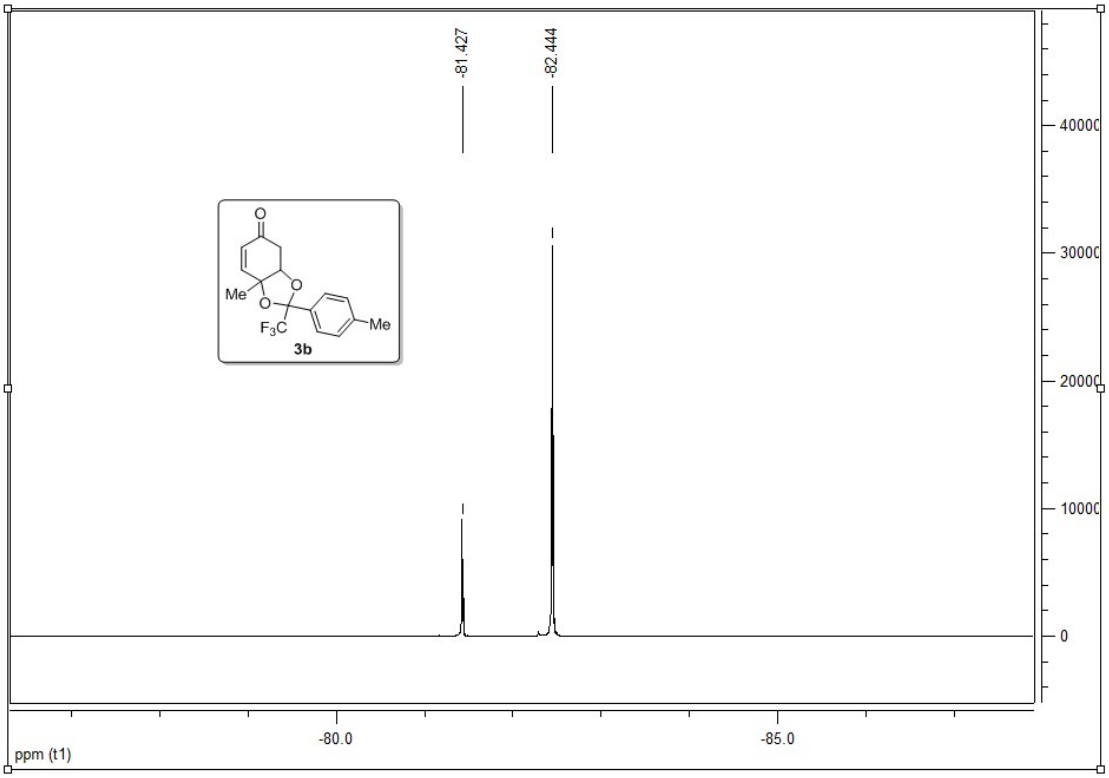
Copies of NMR spectra of the products:

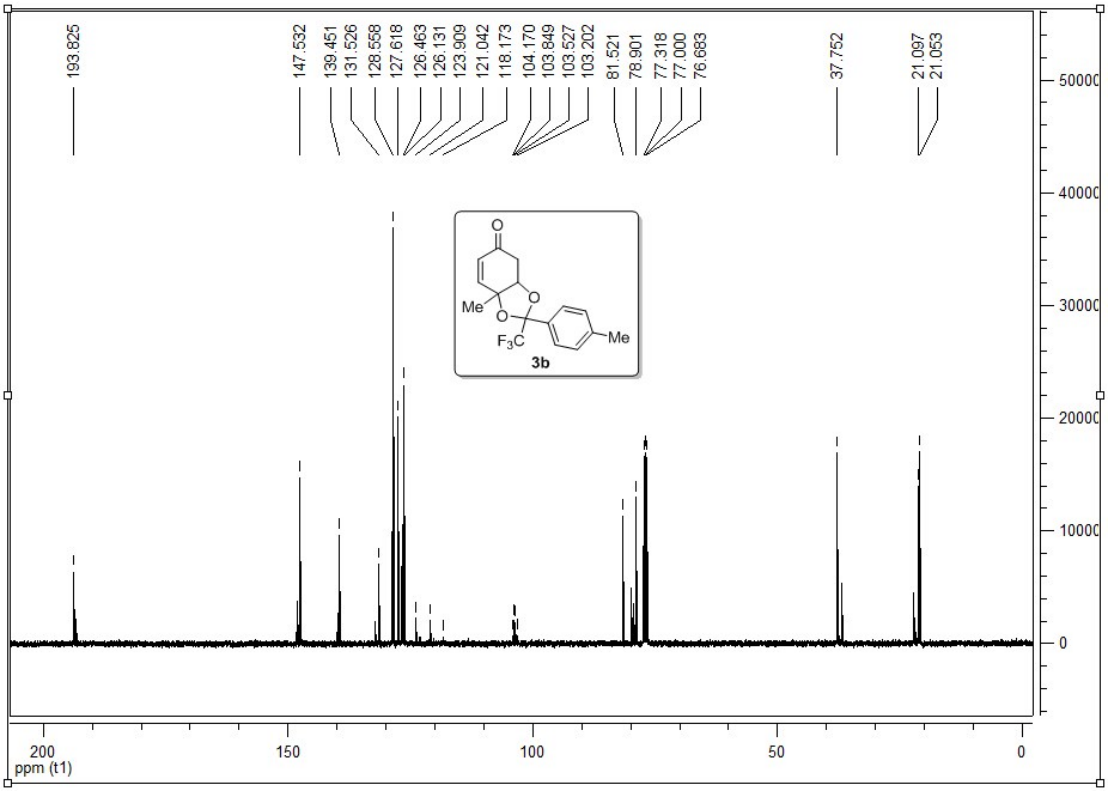


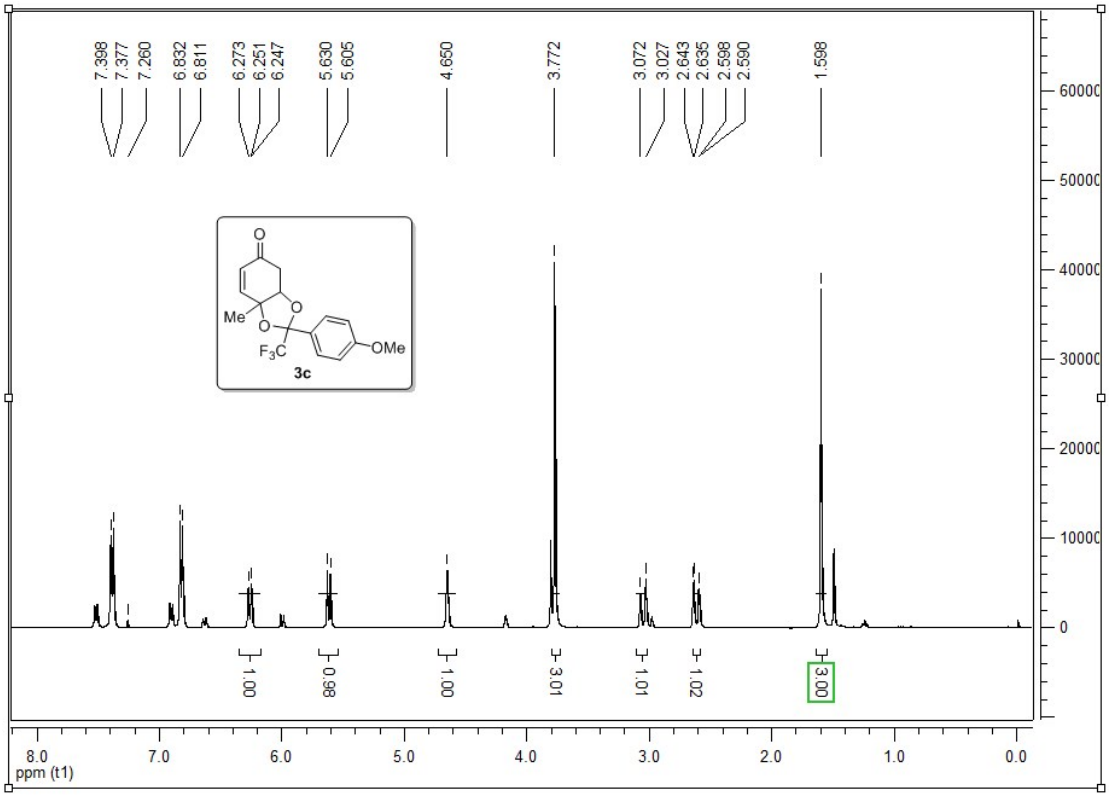


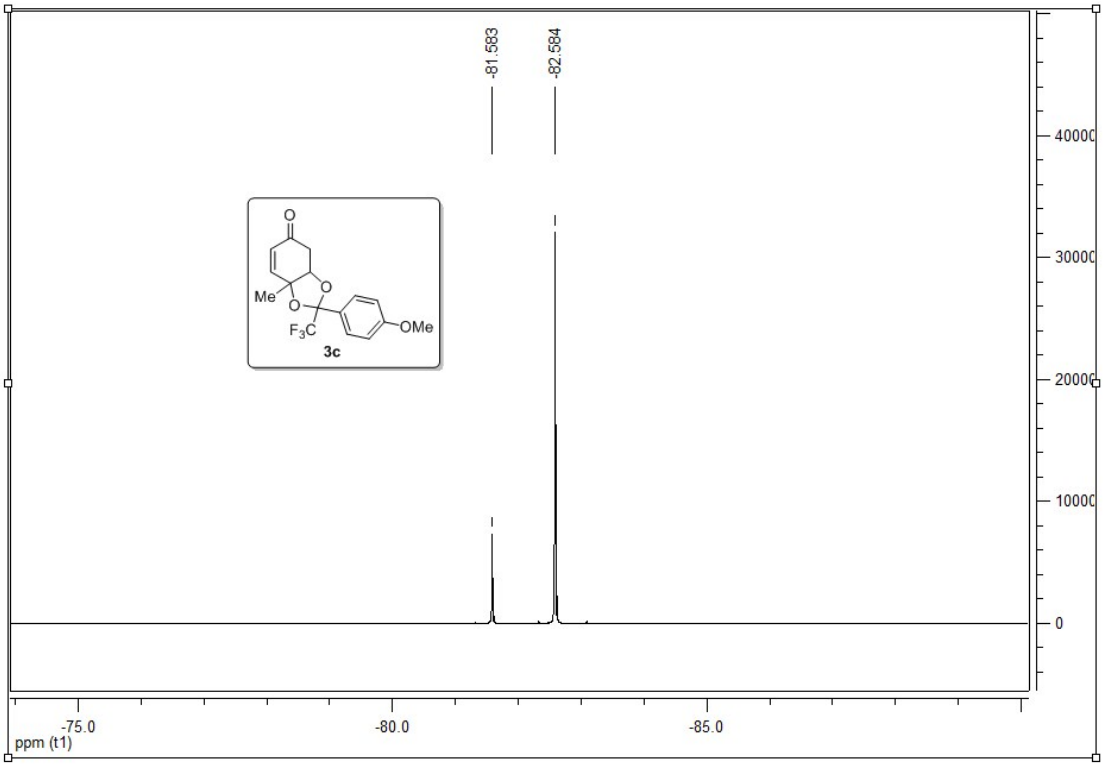

193.648
147.338
134.466
129.471
127.807
127.605
126.544
123.862
120.994
118.128
104.054
103.730
103.409
103.086
81.579
78.989
77.319
77.000
76.682
37.708
20.982
Me
O
F3C
3a
200
150
100
50
0
ppm (t1)


7.378
7.359
7.260
7.133
7.113
6.288
6.283
6.262
6.257
5.626
5.600
4.667
3.083
3.037
2.650
2.642
2.605
2.597
2.325
1.609
Me
O
F3C
Me
3b
2.00
2.00
0.99
0.98
1.00
1.03
1.01
3.00
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


-81.427
-82.444
Me
O
O
F3C
Me
3b
40000
30000
20000
10000
0
-80.0
-85.0
ppm (t1)


193.825
147.532
139.451
131.526
128.558
127.618
126.463
126.131
123.909
121.042
118.173
104.170
103.849
103.527
103.202
81.521
78.901
77.318
77.000
76.683
37.752
21.097
21.053
Me
O
O
F3C
Me
3b
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


7.398
7.377
7.260
6.832
6.811
6.273
6.251
6.247
5.630
5.605
4.650
3.772
3.072
3.027
2.643
2.635
2.598
2.590
1.598
Me
O
F3C
OMe
3c
1.00
0.98
1.00
3.01
1.01
1.02
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)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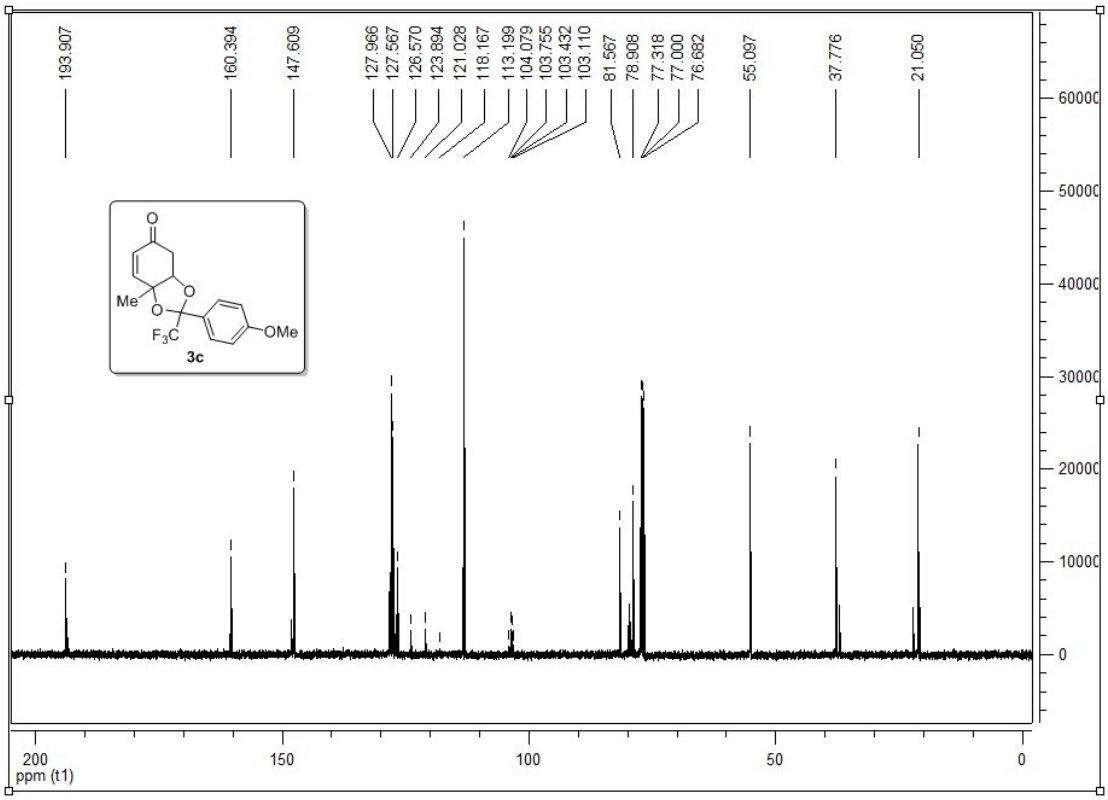
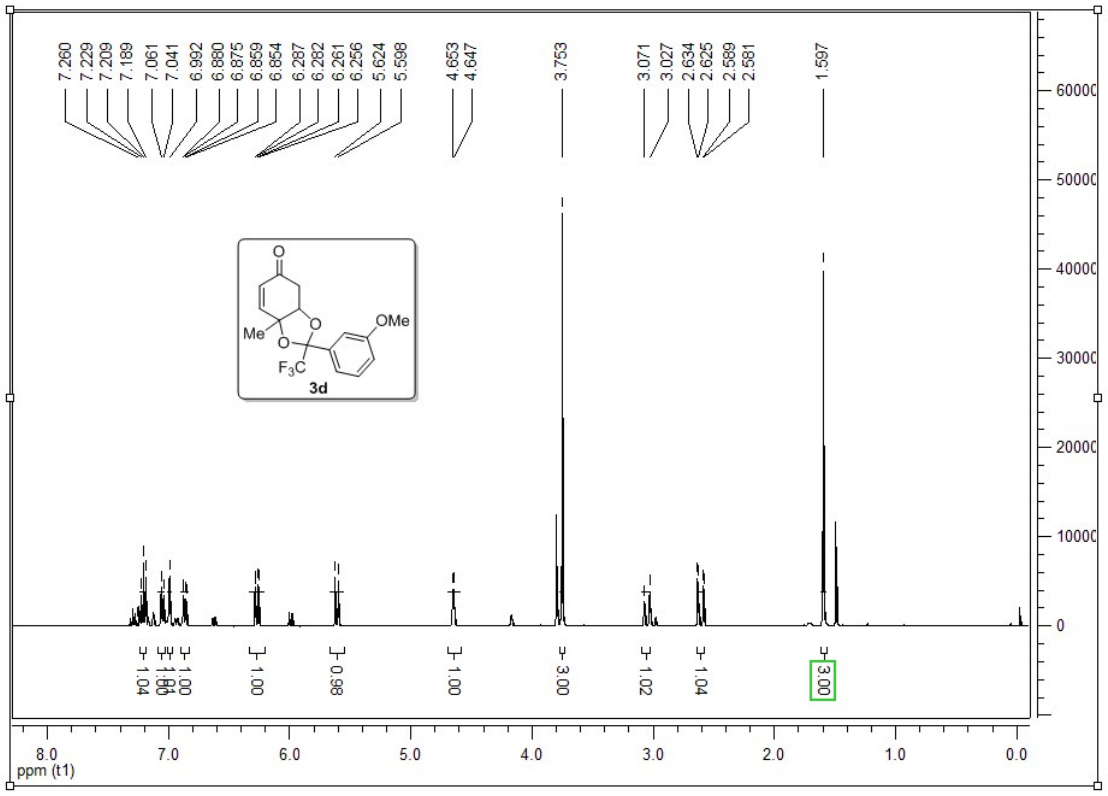
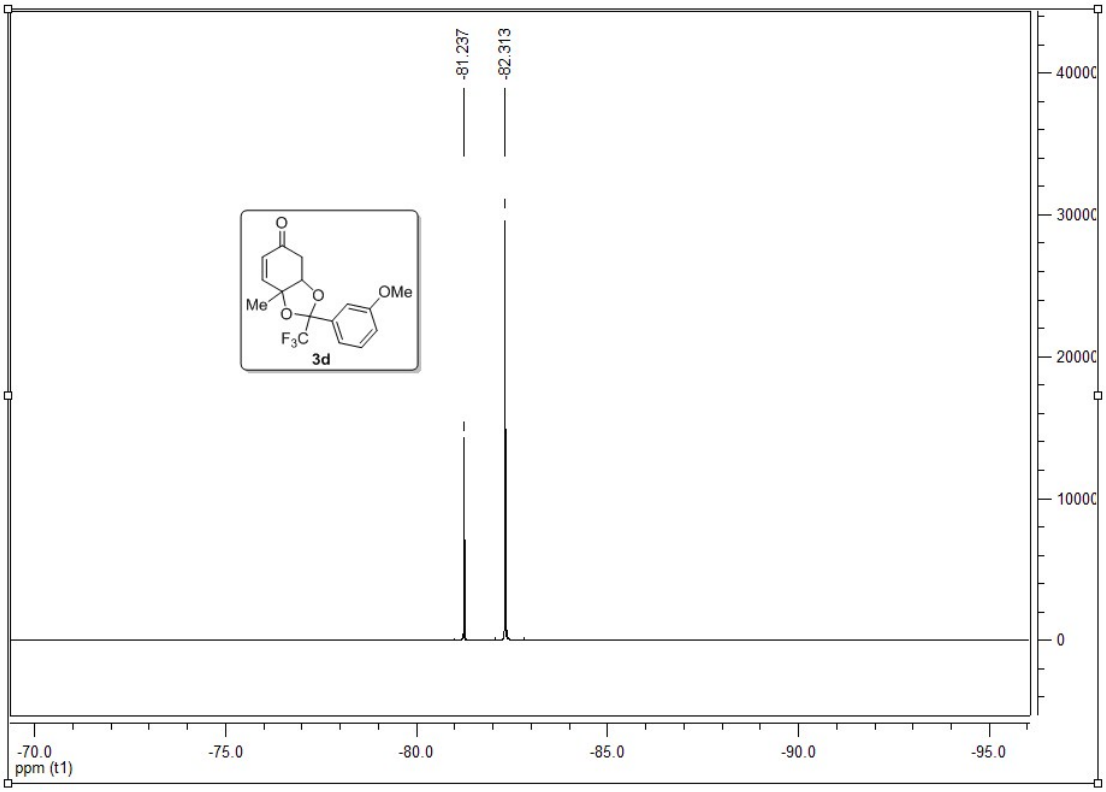
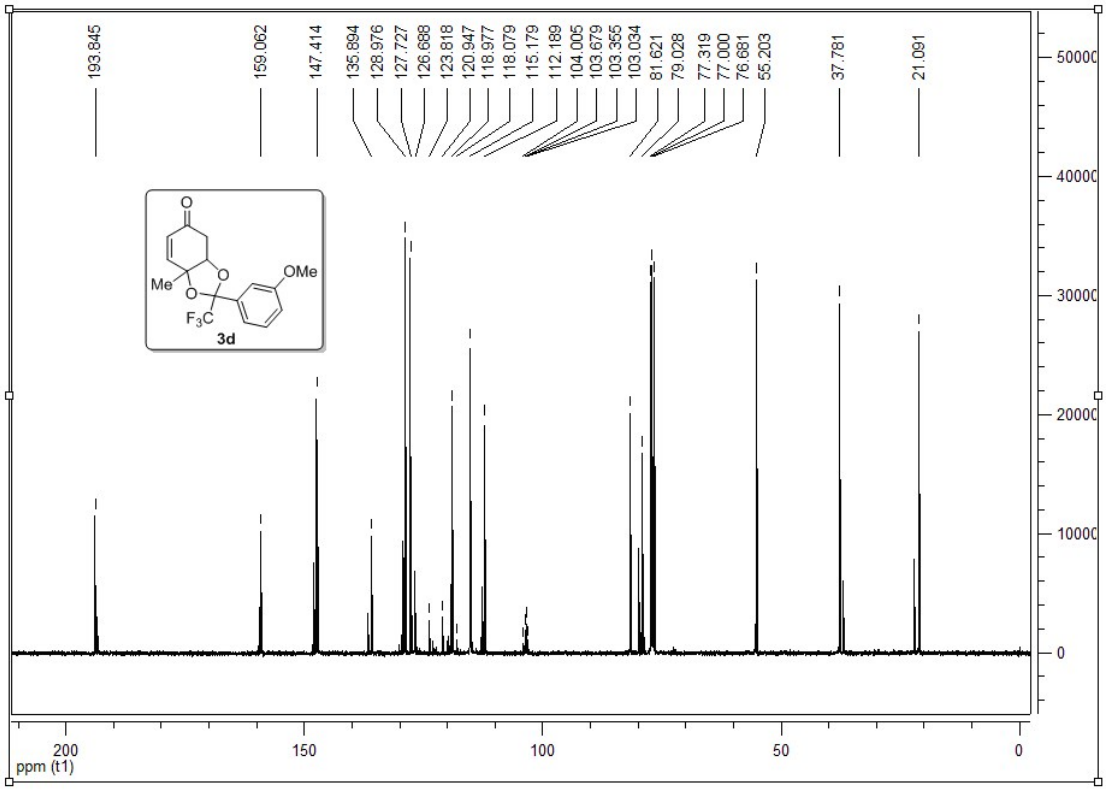
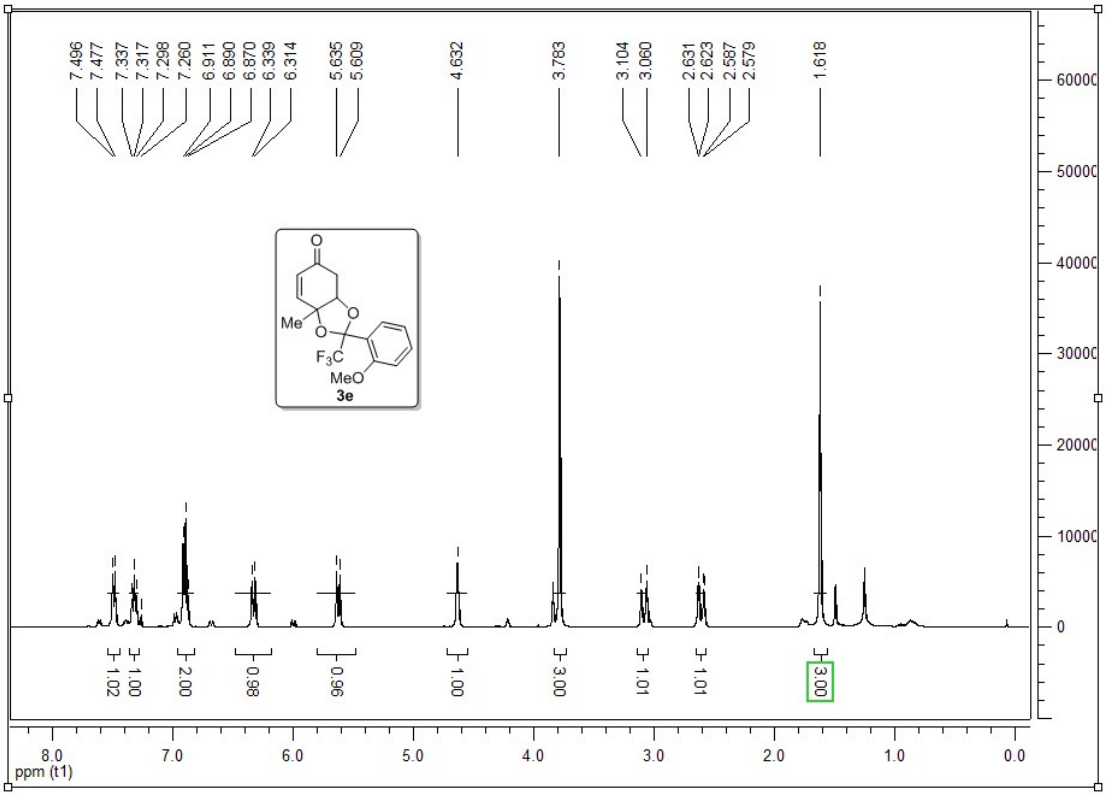
-82.584
Me
O
F3C
OMe
3c
-75.0
-80.0
-85.0
ppm (t1)

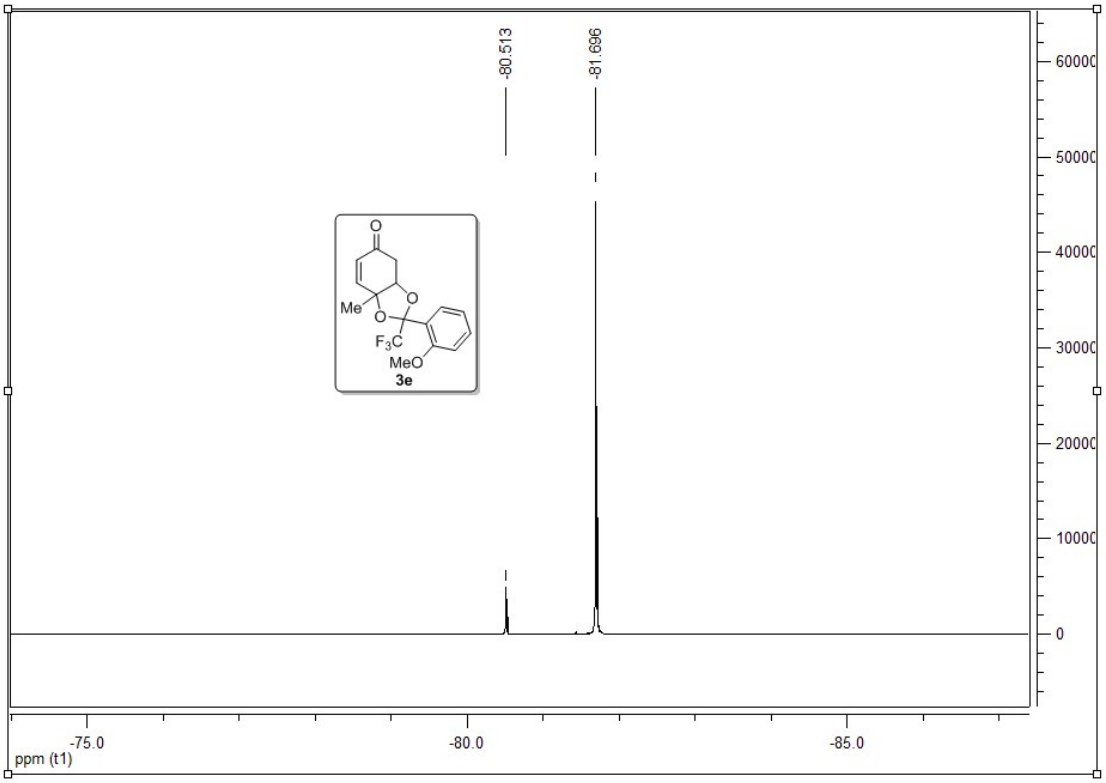

193.907
160.394
147.609
127.966
127.567
126.570
123.894
121.028
118.167
113.199
104.079
103.755
103.432
103.110
81.567
78.908
77.318
77.000
76.682
55.097
37.776
21.050
Me
O
F3C
OMe
3c
200
150
100
50
0
ppm (t1)

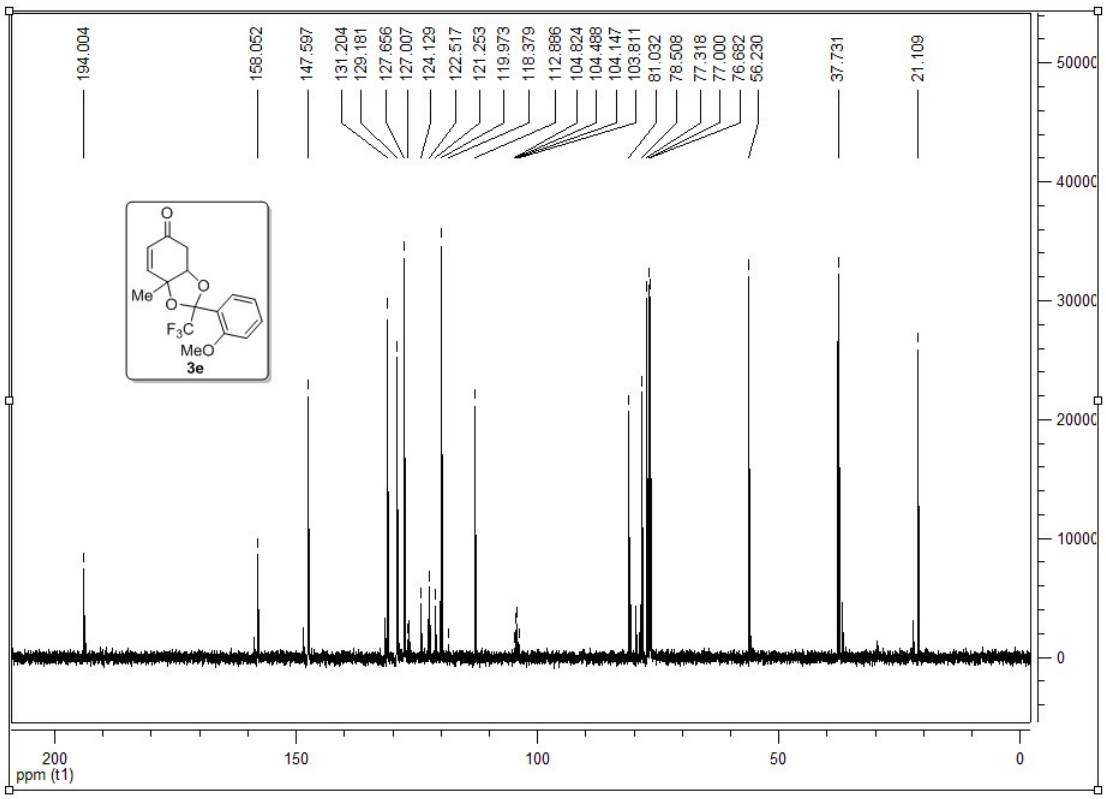

7.260
7.229
7.209
7.189
7.061
7.041
6.992
6.880
6.875
6.859
6.854
6.287
6.282
6.261
6.256
5.624
5.598
4.653
4.647
3.753
3.071
3.027
2.634
2.625
2.589
2.581
1.597
Me
O
F3C
OMe
3d
1.04
3.00
1.00
1.00
0.98
1.00
3.00
1.02
1.04
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

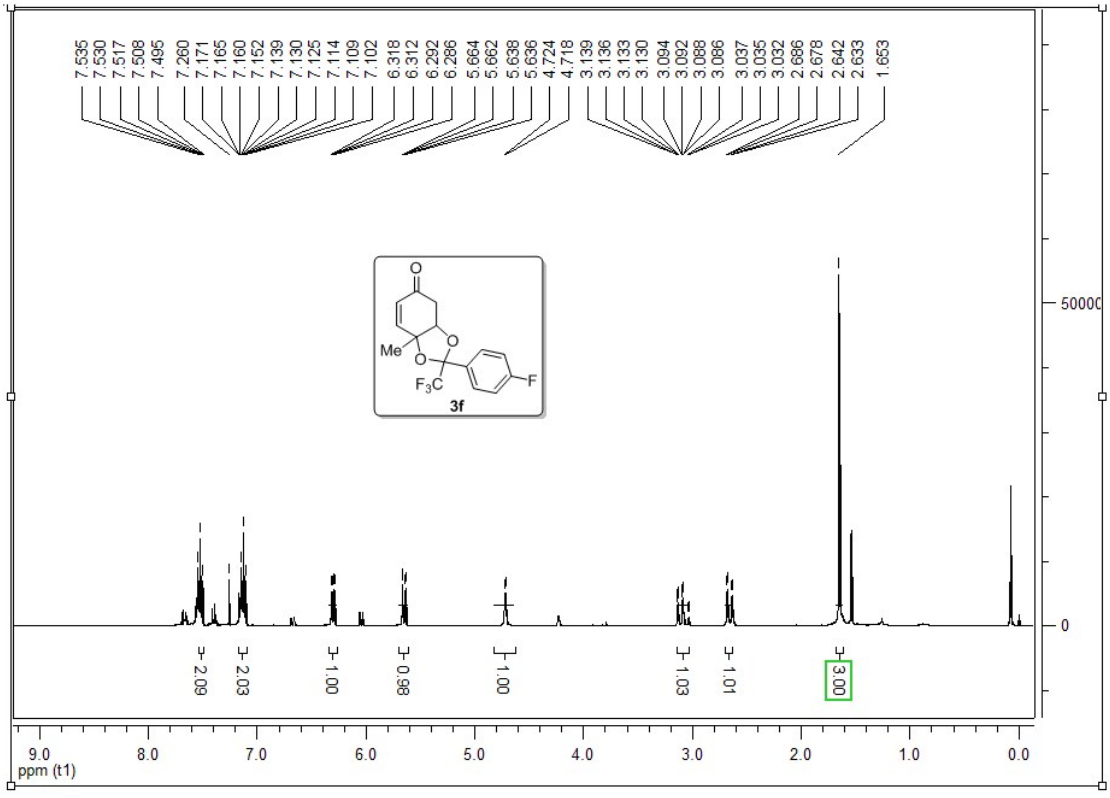

-81.237
-82.313
O
Me
O
OMe
O
F3C
3d
40000
30000
20000
10000
0
-70.0
-75.0
-80.0
-85.0
-90.0
-95.0
ppm (t1)

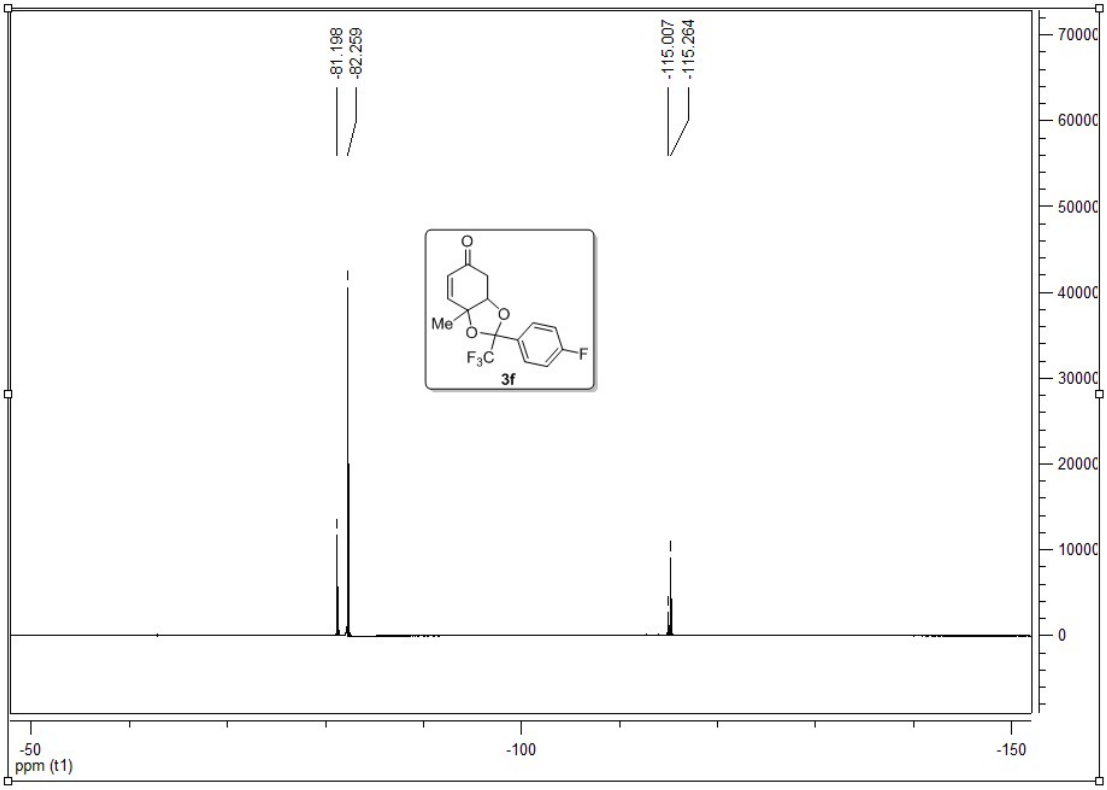

193.845
159.062
147.414
135.894
128.976
127.727
126.688
123.818
120.947
118.977
118.079
115.179
112.189
104.005
103.679
103.355
103.034
81.621
79.028
77.319
77.000
76.681
55.203
37.781
21.091
O
Me
O
OMe
O
F3C
3d
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

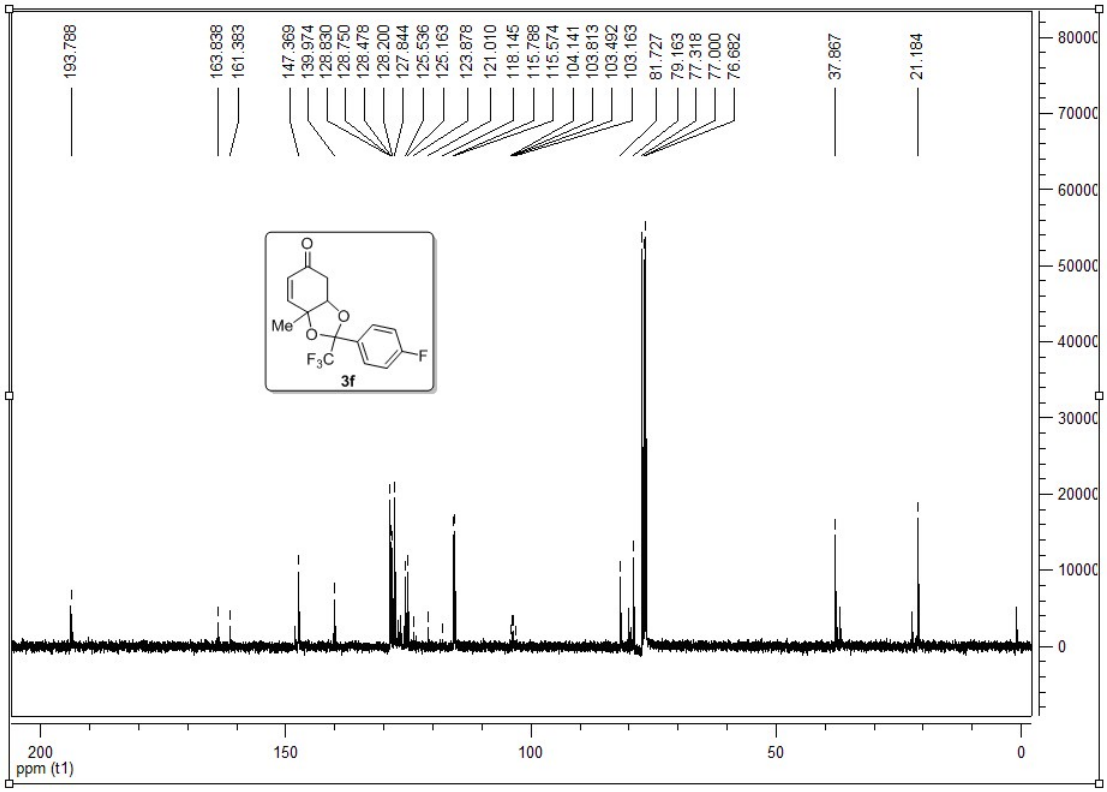

7.496
7.477
7.337
7.317
7.298
7.260
6.911
6.890
6.870
6.339
6.314
5.635
5.609
4.632
3.783
3.104
3.060
2.631
2.623
2.587
2.579
1.618
Me
O
F3C
MeO
3e
1.02
1.00
2.00
0.98
0.96
1.00
3.00
1.01
1.01
3.00
ppm (t1)


-80.513
-81.696
Me
O
F3C
MeO
3e
ppm (t1)


194.004
158.052
147.597
131.204
129.181
127.656
127.007
124.129
122.517
121.253
119.973
118.379
112.886
104.824
104.488
104.147
103.811
81.032
78.508
77.318
77.000
76.682
56.230
37.731
21.109
Me
F3C
MeO
3e
ppm (t1)


7.535
7.530
7.517
7.508
7.495
7.260
7.171
7.165
7.160
7.152
7.139
7.130
7.125
7.114
7.109
7.102
6.318
6.312
6.292
6.286
5.664
5.662
5.638
5.636
4.724
4.718
3.139
3.136
3.133
3.130
3.094
3.092
3.088
3.086
3.037
3.035
3.032
2.686
2.678
2.642
2.633
1.653
Me
F3C
F
3f
2.00
2.03
1.00
0.98
1.00
1.03
1.01
3.00
ppm (t1)


-81.198
-82.259
-115.007
-115.264
Me
F3C
F
3f
-50
ppm (t1)
-100
-150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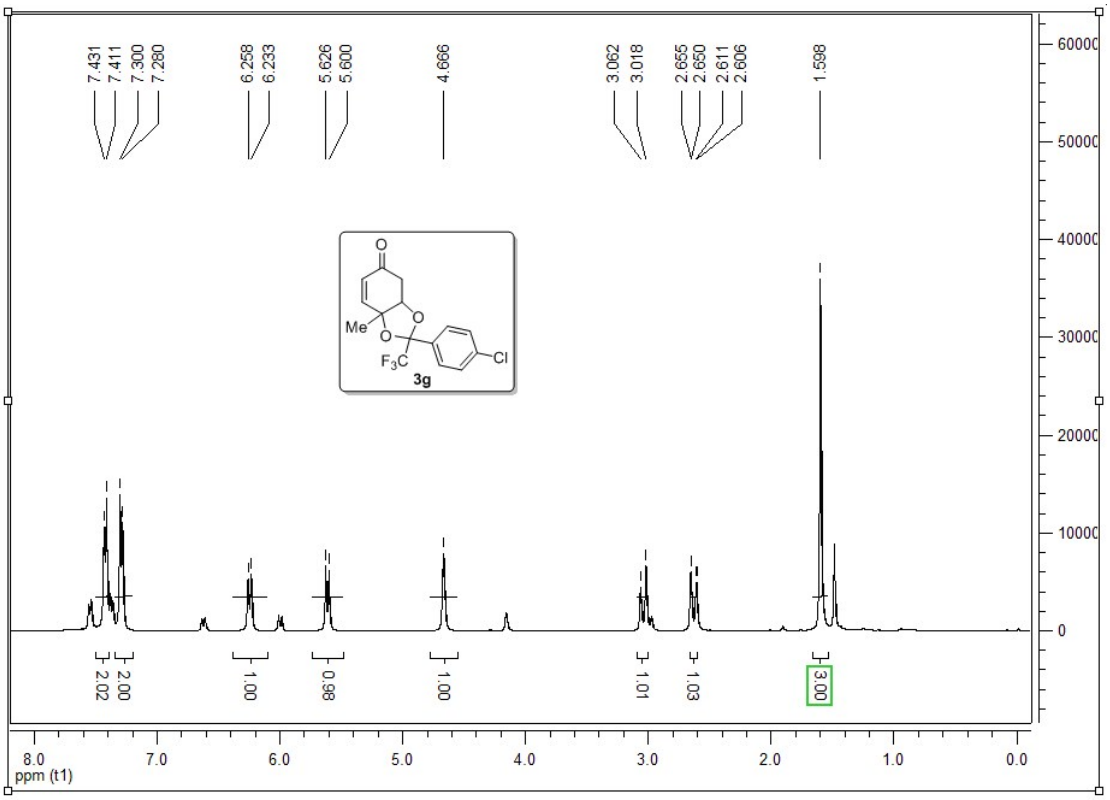
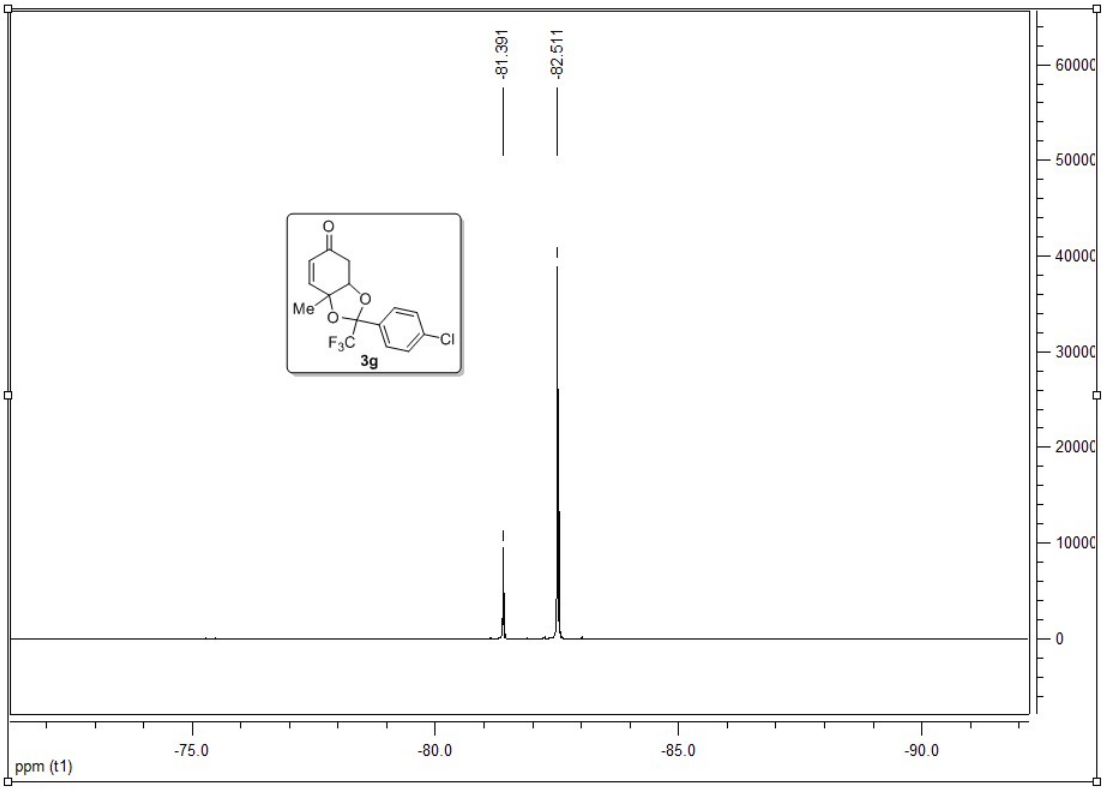
163.838
161.363
147.369
139.974
128.830
128.750
128.478
128.200
127.844
125.536
125.163
123.878
121.010
118.145
115.788
115.574
104.141
103.813
103.492
103.163
81.727
79.163
77.318
77.000
76.682
37.867
21.184
Me
F3C
F
3f
200
ppm (t1)
150
100
50
0


7.431
7.411
7.300
7.280
6.258
6.233
5.626
5.600
4.666
3.062
3.018
2.655
2.650
2.611
2.606
1.598
Me
O
F3C
Cl
3g
2.02
2.00
1.00
0.98
1.00
1.01
1.03
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


-81.391
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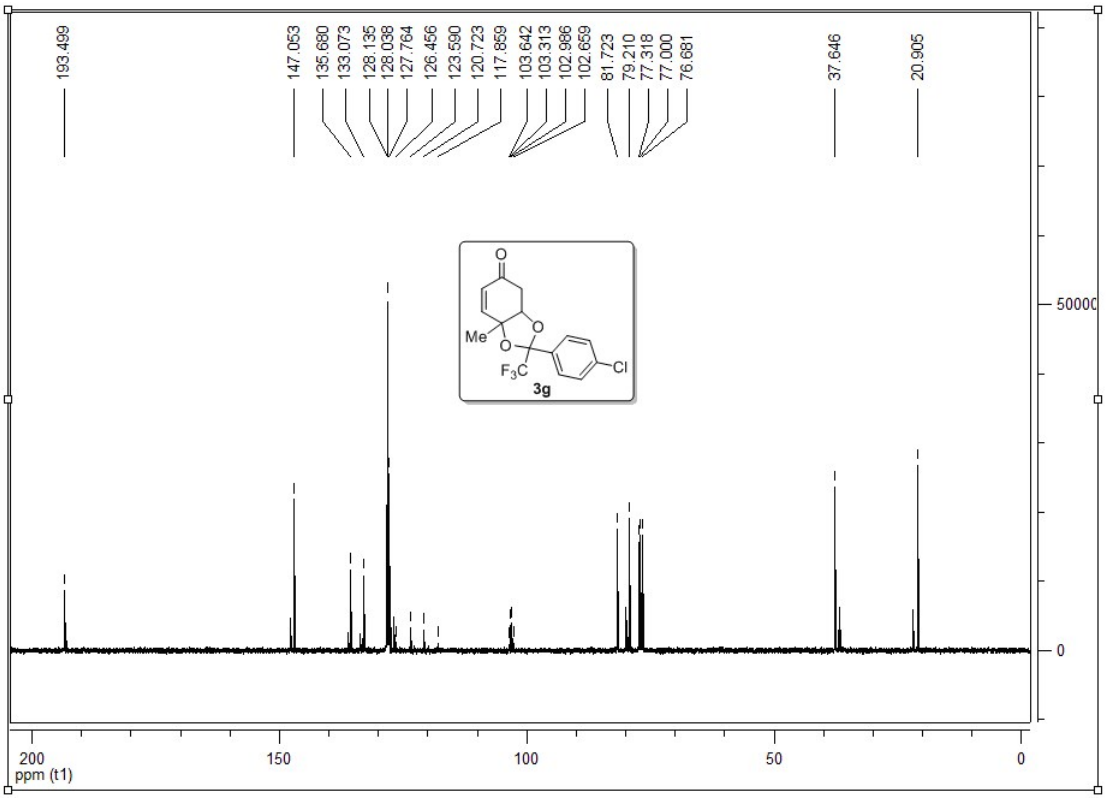
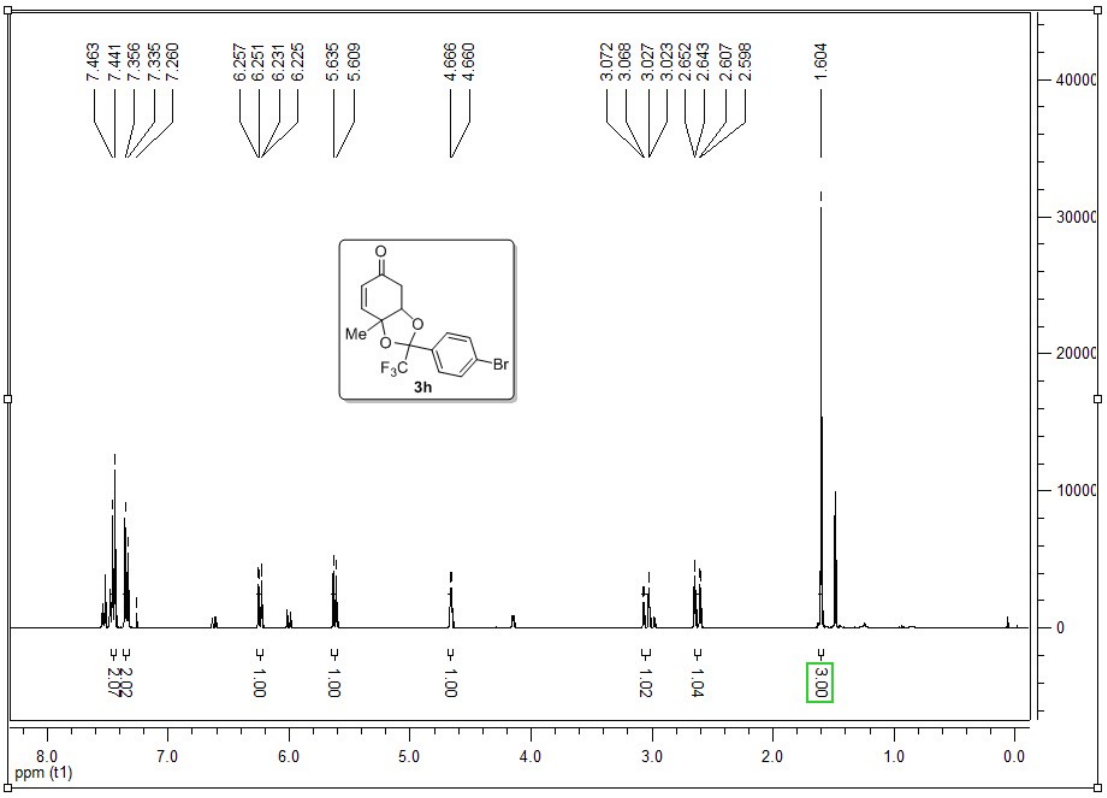
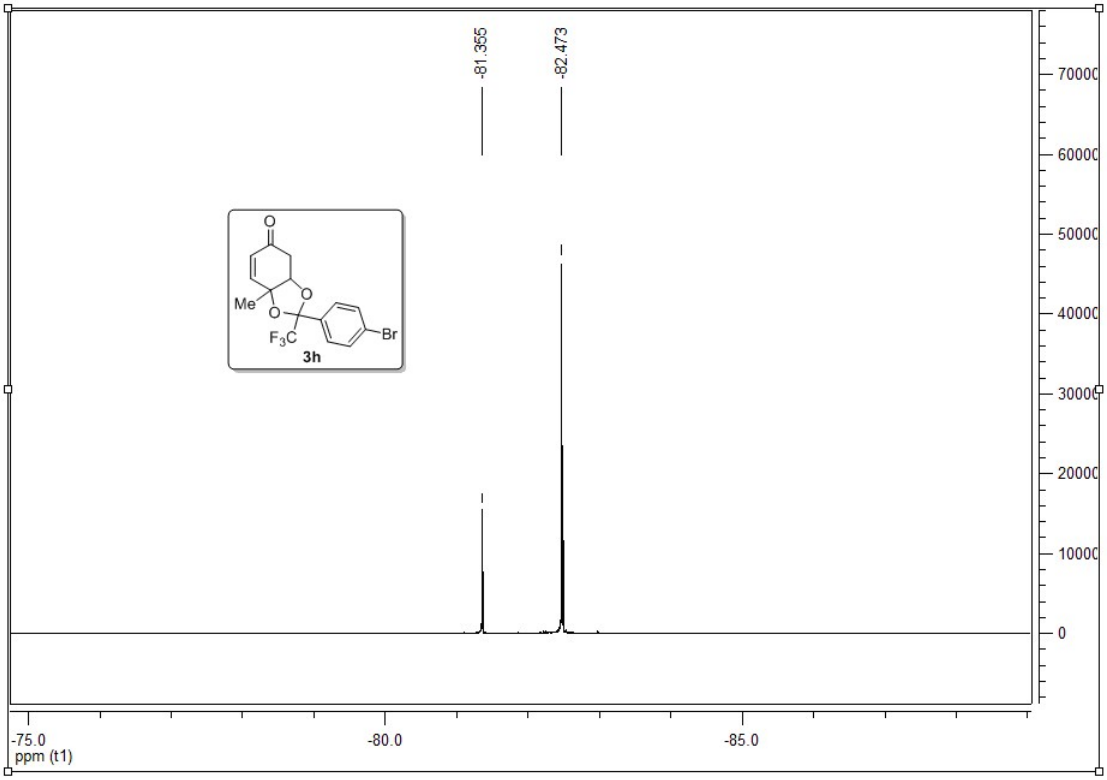
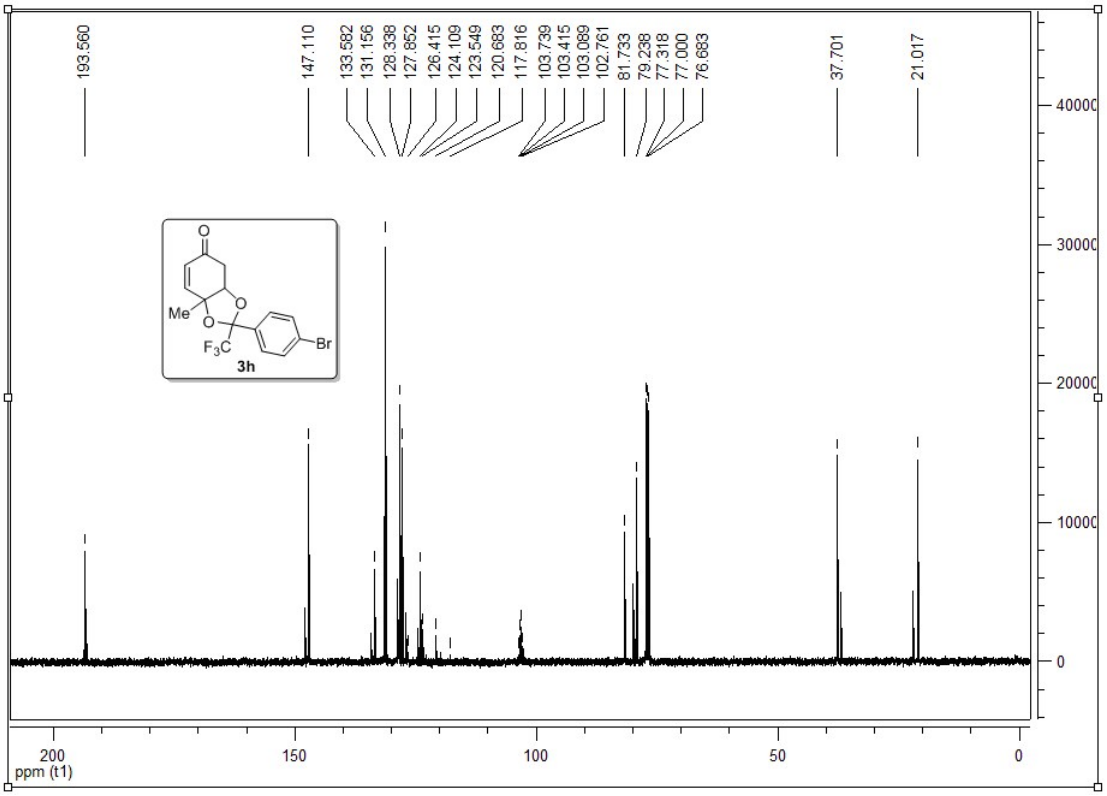
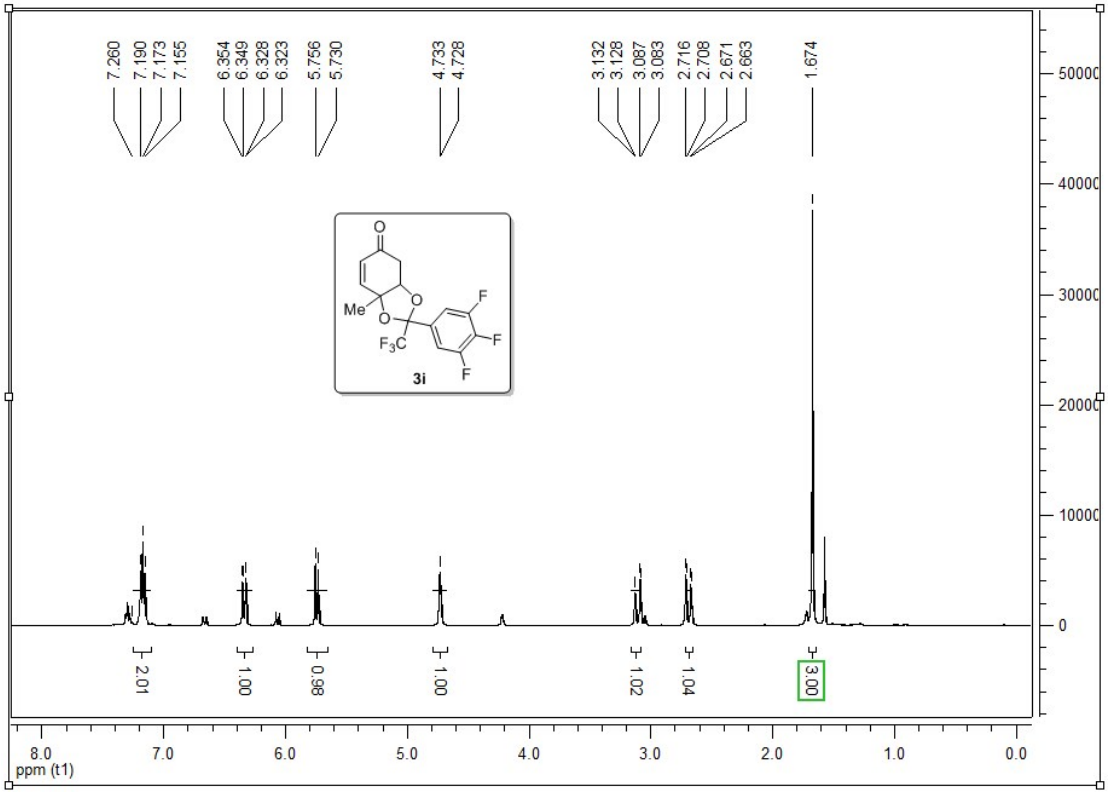
Me
O
F3C
Cl
3g
-75.0
-80.0
-85.0
-90.0
ppm (t1)

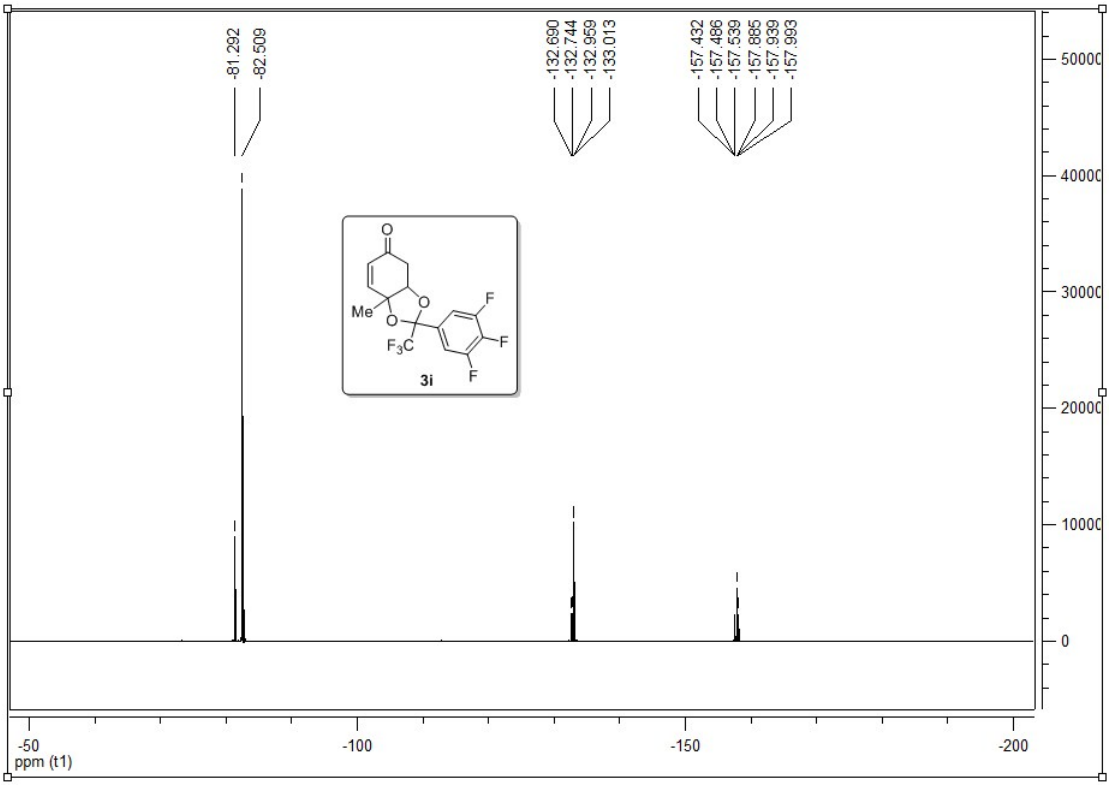

193.499
147.053
135.680
133.073
128.135
128.038
127.764
126.456
123.590
120.723
117.859
103.642
103.313
102.986
102.659
81.723
79.210
77.318
77.000
76.681
37.646
20.905
Me
O
F3C
Cl
3g
50000
0
200
150
100
50
0
ppm (t1)

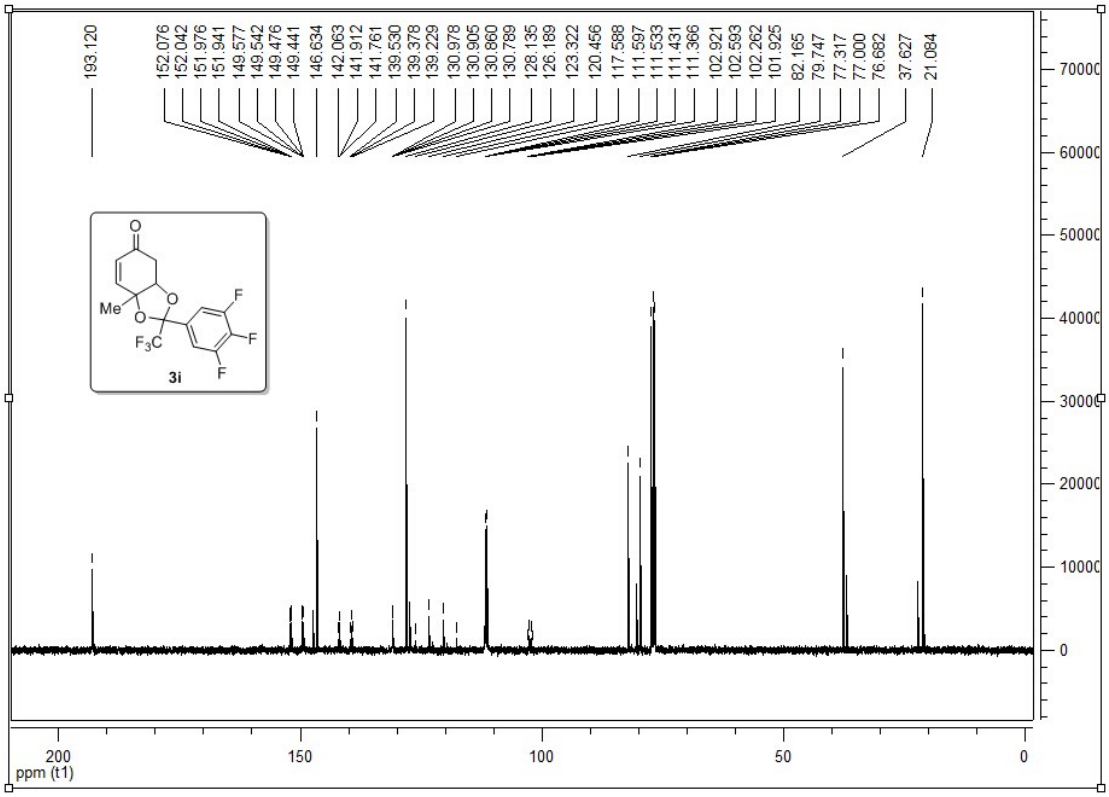

7.463
7.441
7.356
7.335
7.260
6.257
6.251
6.231
6.225
5.635
5.609
4.666
4.660
3.072
3.068
3.027
3.023
2.652
2.643
2.607
2.598
1.604
Me
O
F3C
Br
3h
2.02
1.00
1.00
1.00
2.02
1.04
3.00
40000
30000
20000
10000
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

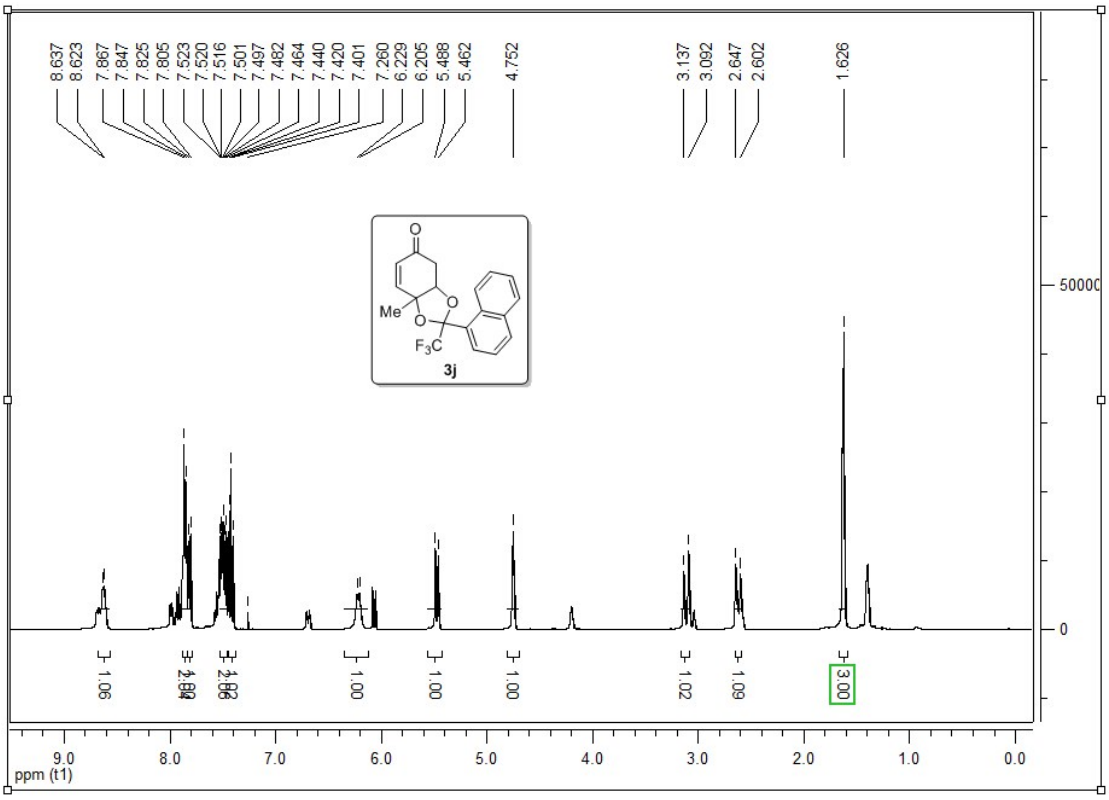

-81.355
-82.473
O
Me
O
O
F3C
Br
3h
70000
60000
50000
40000
30000
20000
10000
0
-75.0
ppm (t1)
-80.0
-85.0


193.560
147.110
133.582
131.156
128.338
127.852
126.415
124.109
123.549
120.683
117.816
103.739
103.415
103.089
102.761
81.733
79.238
77.318
77.000
76.683
37.701
21.017
O
Me
O
O
F3C
Br
3h
40000
30000
20000
10000
0
200
ppm (t1)
150
100
50
0


7.260
7.190
7.173
7.155
6.354
6.349
6.328
6.323
5.756
5.730
4.733
4.728
3.132
3.128
3.087
3.083
2.716
2.708
2.671
2.663
1.674
Me
O
F3C
F
F
F
3i
2.01
1.00
0.98
1.00
1.02
1.04
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


-81.292
-82.509
-132.690
-132.744
-132.959
-133.013
-157.432
-157.486
-157.539
-157.885
-157.939
-157.993
Me
O
F3C
F
F
F
3i
-50
-100
-150
-200
ppm (t1)


193.120
152.076
152.042
151.976
151.941
149.577
149.542
149.476
149.441
146.634
142.063
141.912
141.761
139.530
139.378
139.229
130.978
130.905
130.860
130.789
128.135
126.189
123.322
120.456
117.588
111.597
111.533
111.431
111.366
102.921
102.593
102.262
101.925
82.165
79.747
77.317
77.000
76.682
37.627
21.084
Me
O
F3C
F
F
F
3i
ppm (t1)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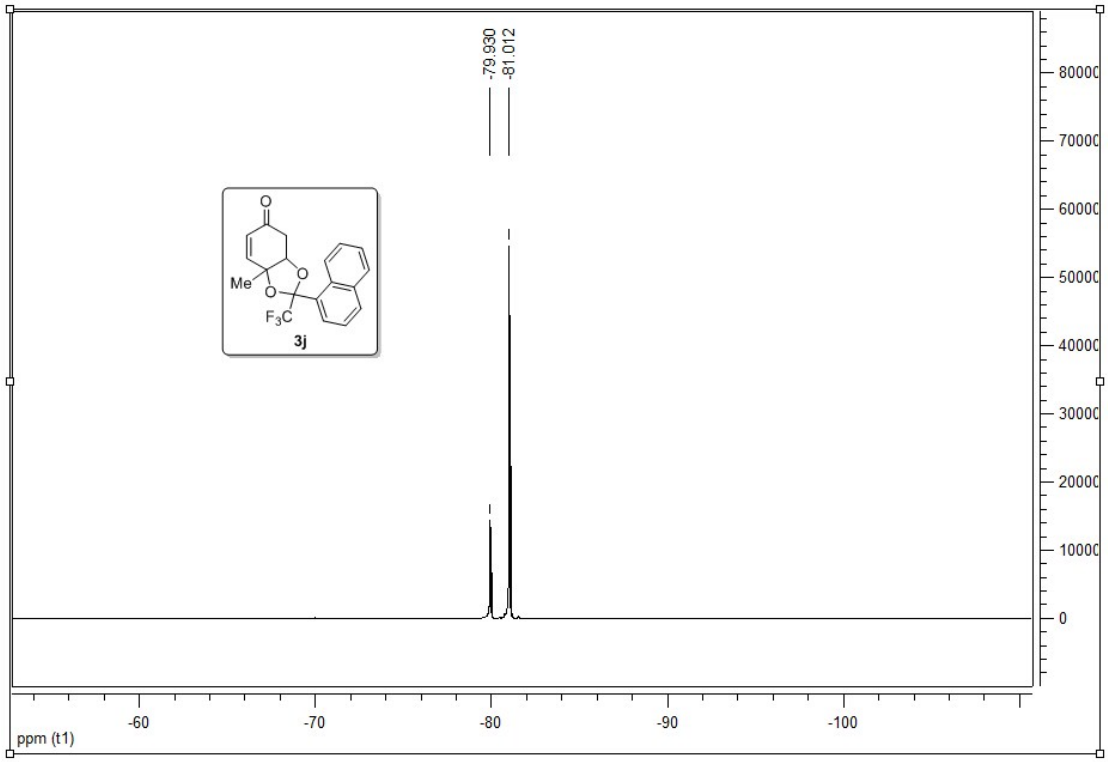
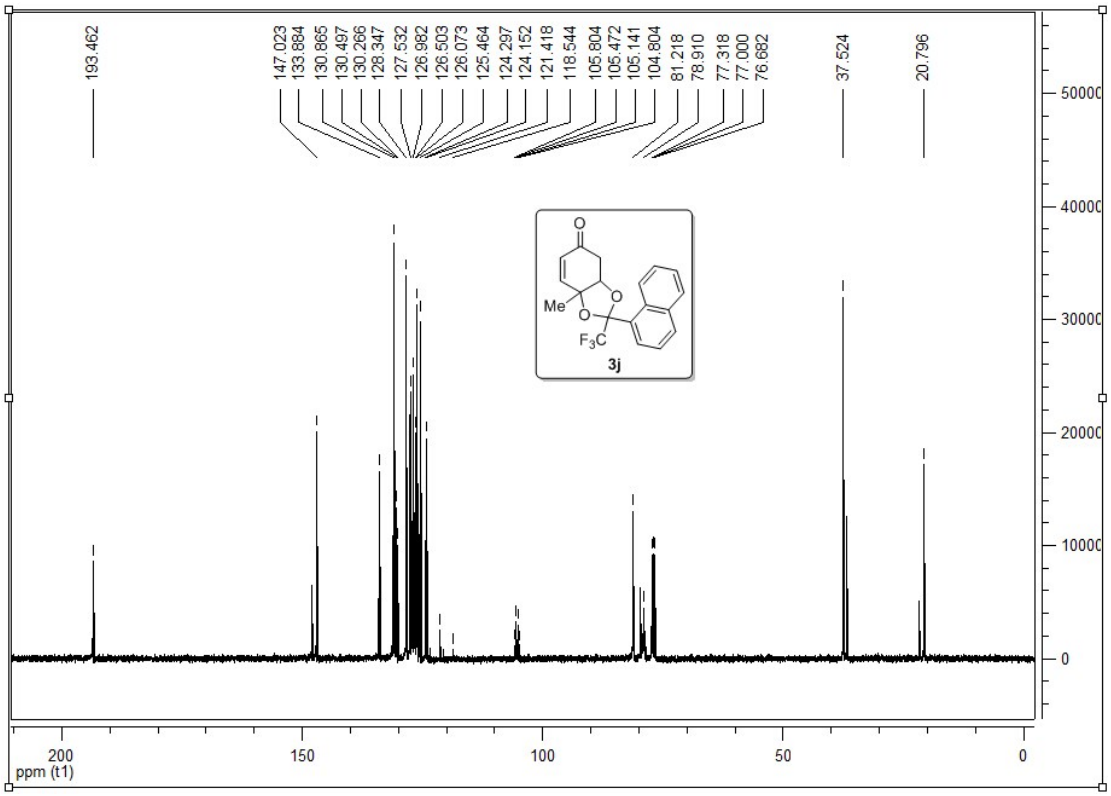
8.623
7.867
7.847
7.825
7.805
7.523
7.520
7.516
7.501
7.497
7.482
7.464
7.440
7.420
7.401
7.260
6.229
6.205
5.488
5.462
4.752
3.137
3.092
2.647
2.602
1.626
Me
O
F3C
3j
1.06
2.04
3.08
1.00
1.00
1.00
1.02
1.09
3.00
ppm (t1)


-79.930
-81.012
Me
O
F3C
3j
80000
70000
60000
50000
40000
30000
20000
10000
0
-60
-70
-80
-90
-100
ppm (t1)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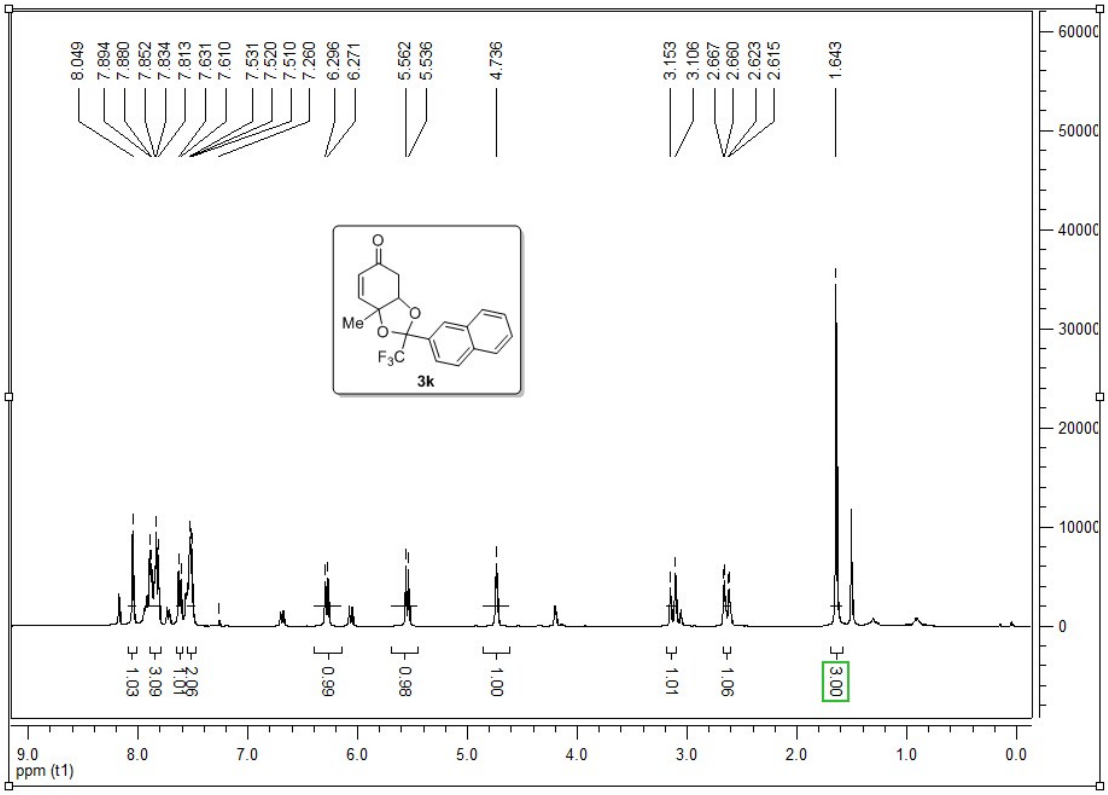
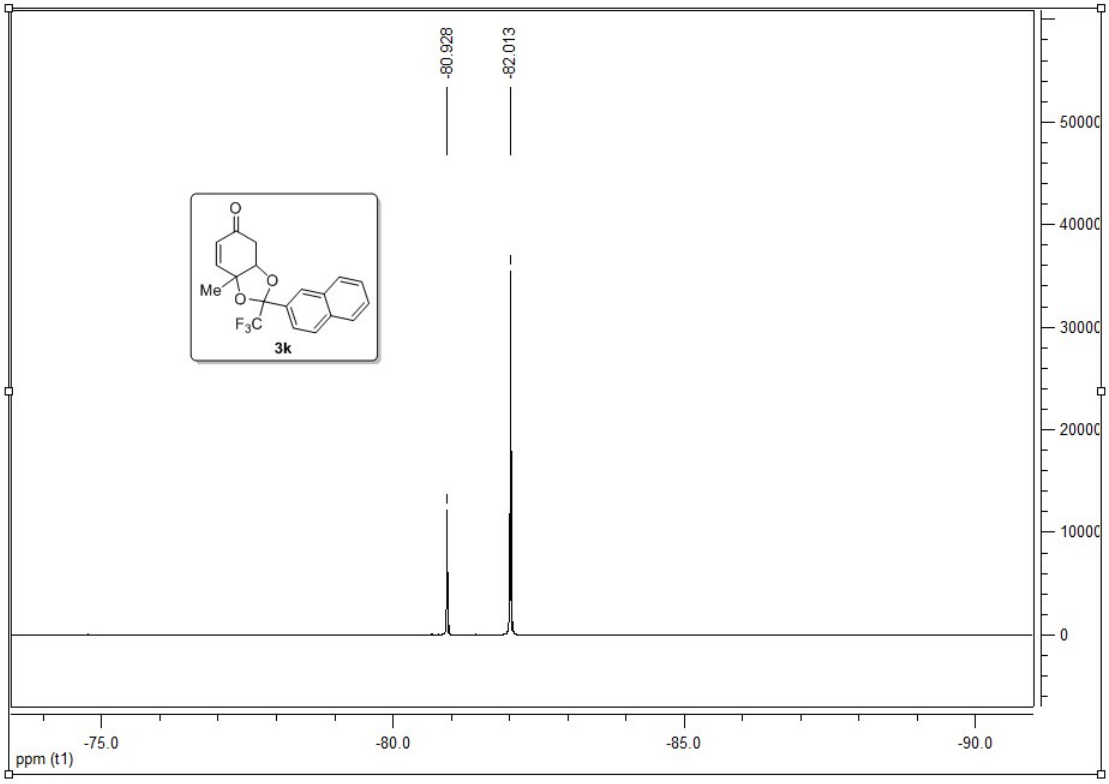
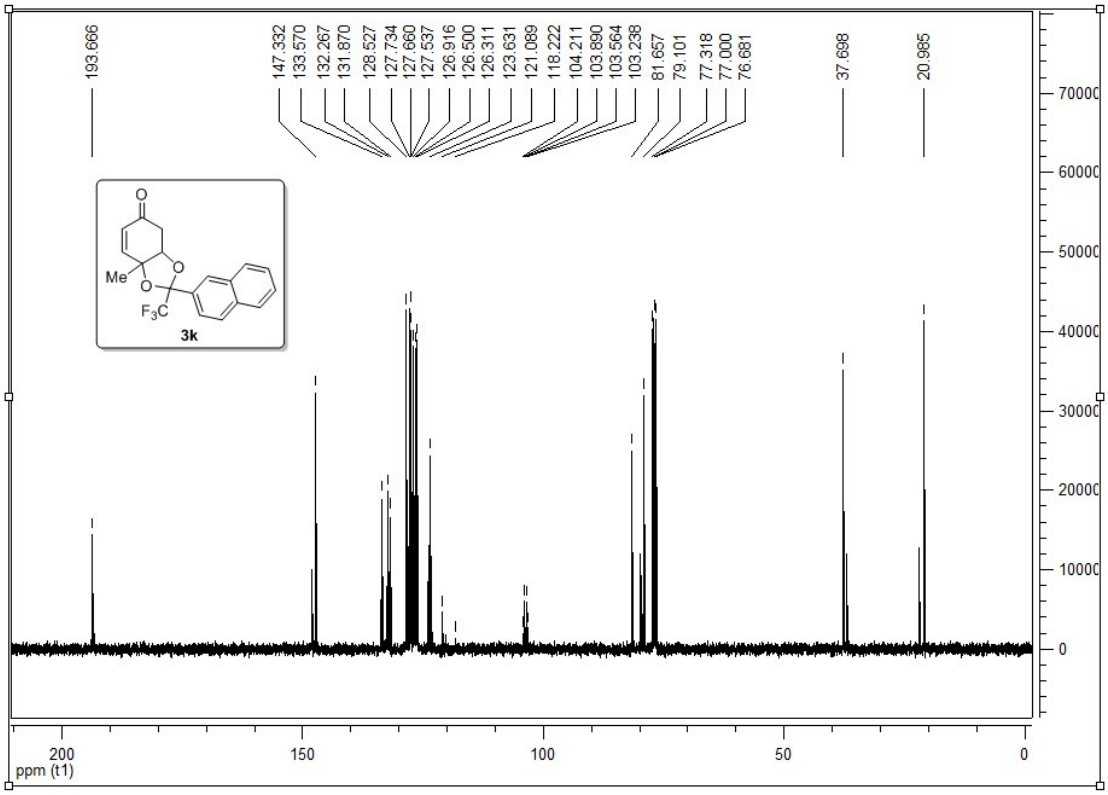
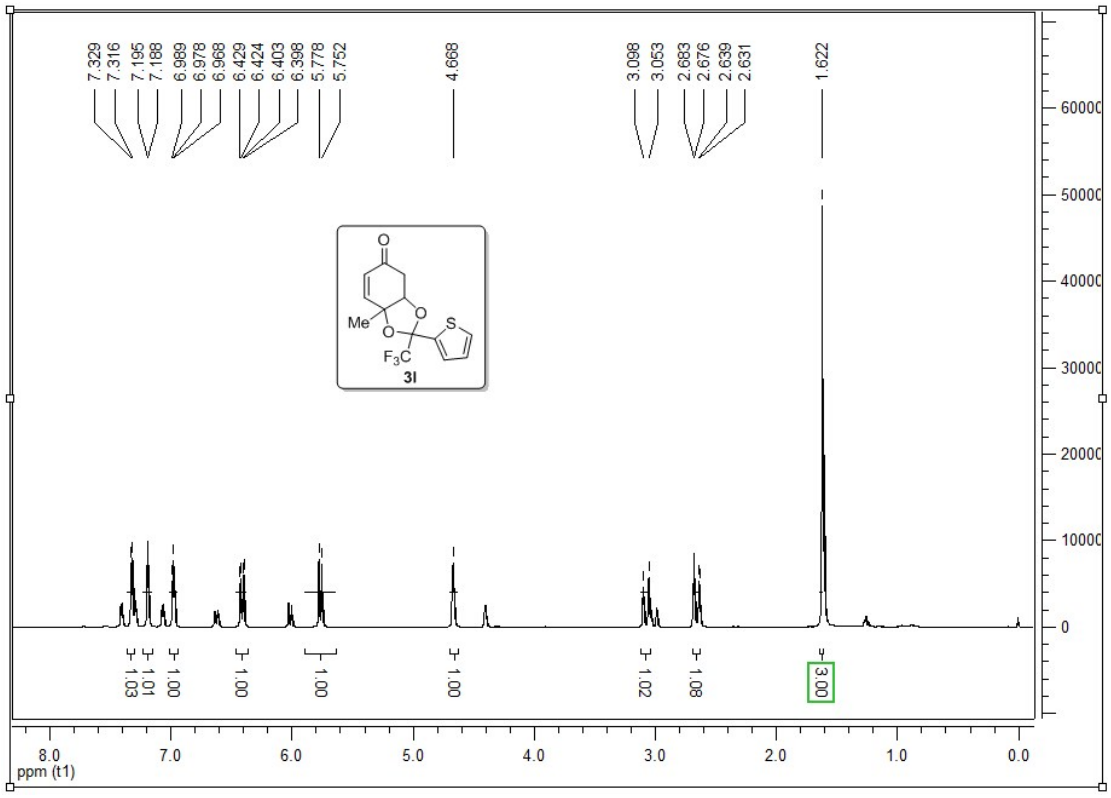
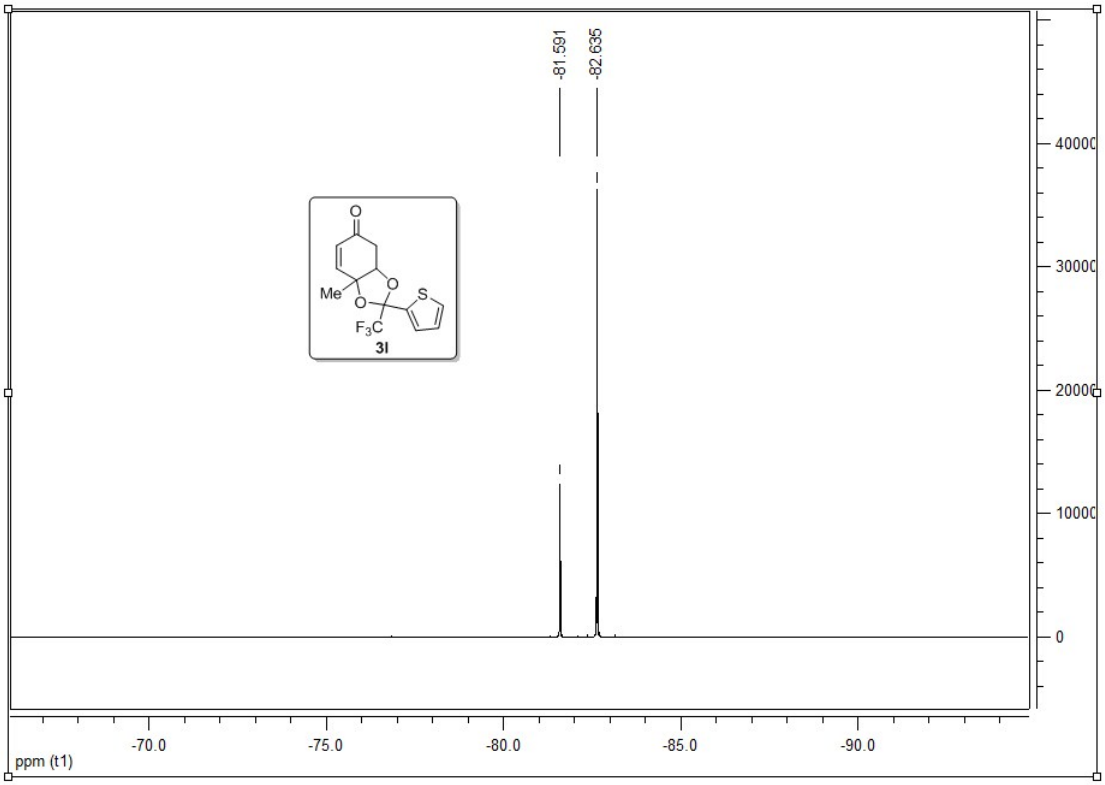
147.023
133.884
130.865
130.497
130.266
128.347
127.532
126.982
126.503
126.073
125.464
124.297
124.152
121.418
118.544
105.804
105.472
105.141
104.804
81.218
78.910
77.318
77.000
76.682
37.524
20.796
Me
O
F3C
3j
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

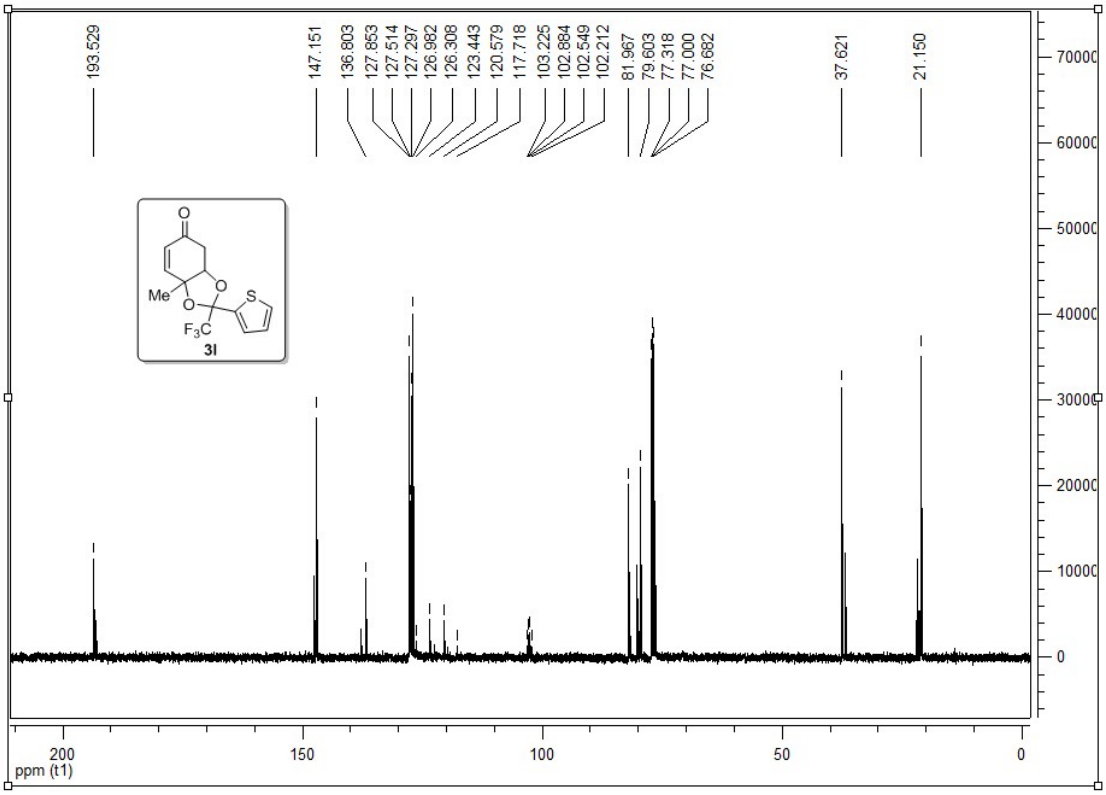

8.049
7.894
7.880
7.852
7.834
7.813
7.631
7.610
7.531
7.520
7.510
7.260
6.296
6.271
5.562
5.536
4.736
3.153
3.106
2.667
2.660
2.623
2.615
1.643
Me
O
F3C
3k
1.03
3.09
2.06
1.01
0.99
0.98
1.00
1.01
1.06
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

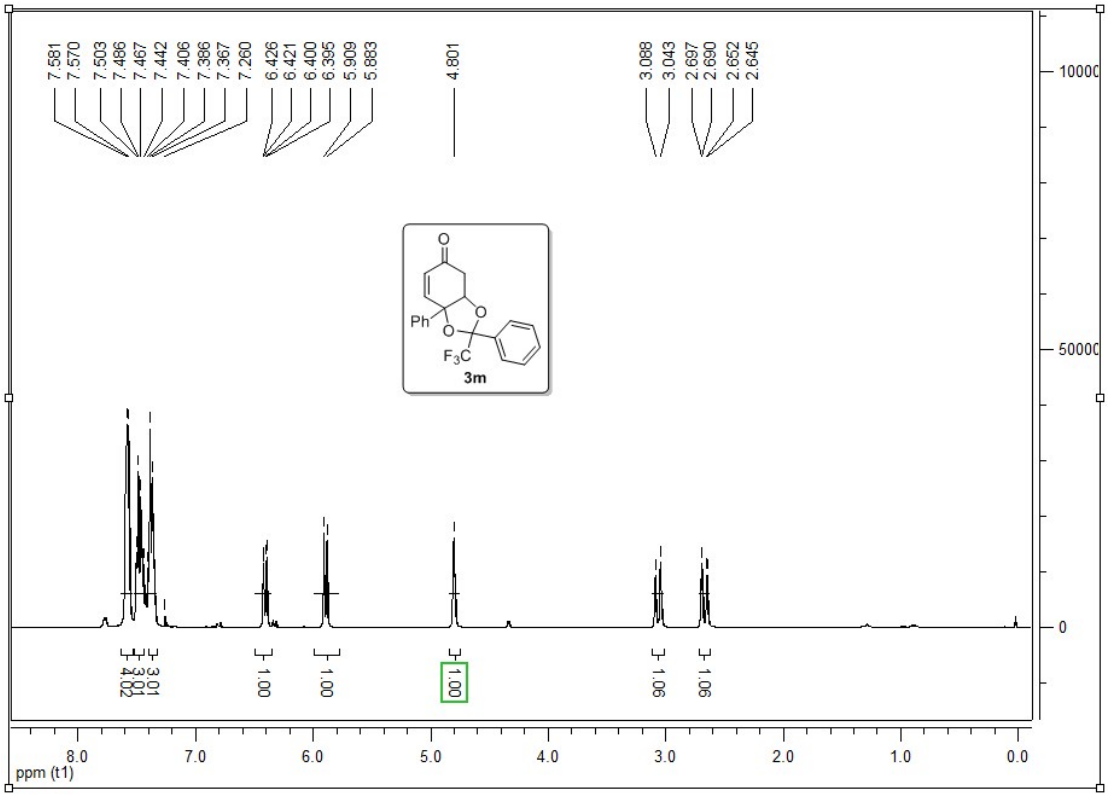

-80.928
-82.013
Me
O
F3C
3k
-75.0
-80.0
-85.0
-90.0
ppm (t1)

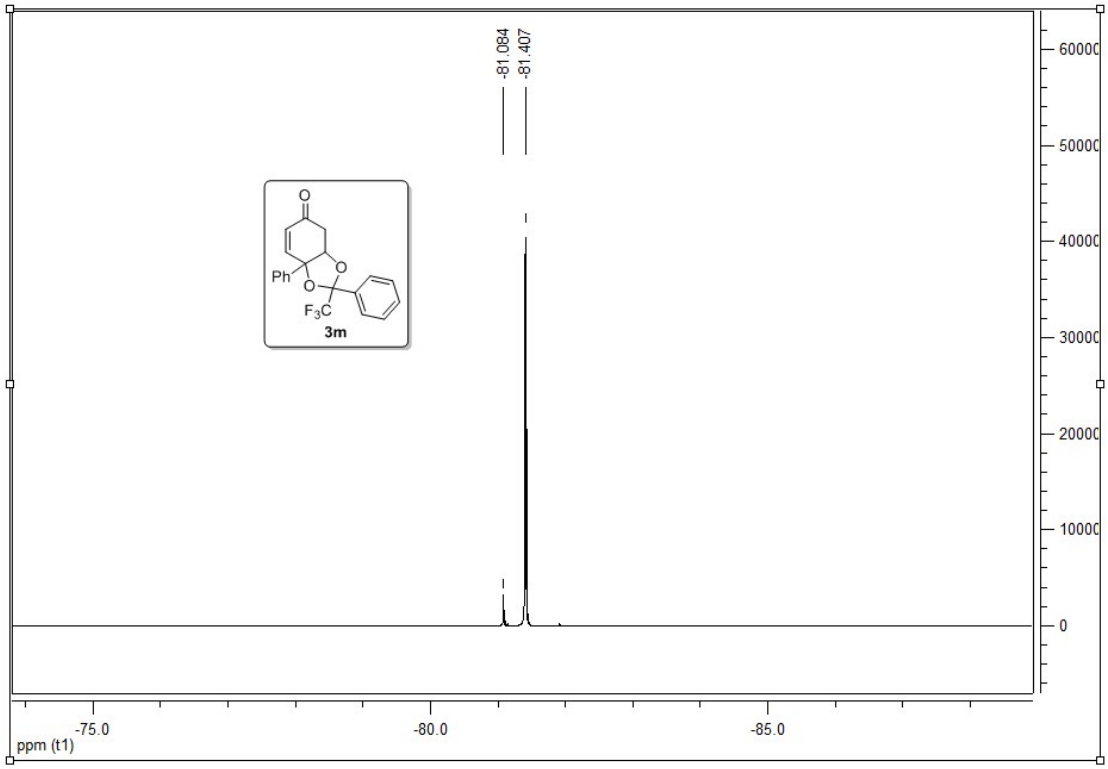

193.666
147.332
133.570
132.267
131.870
128.527
127.734
127.660
127.537
126.916
126.500
126.311
123.631
121.089
118.222
104.211
103.890
103.564
103.238
81.657
79.101
77.318
77.000
76.681
37.698
20.985
Me
O
F3C
3k
200
150
100
50
0
ppm (t1)

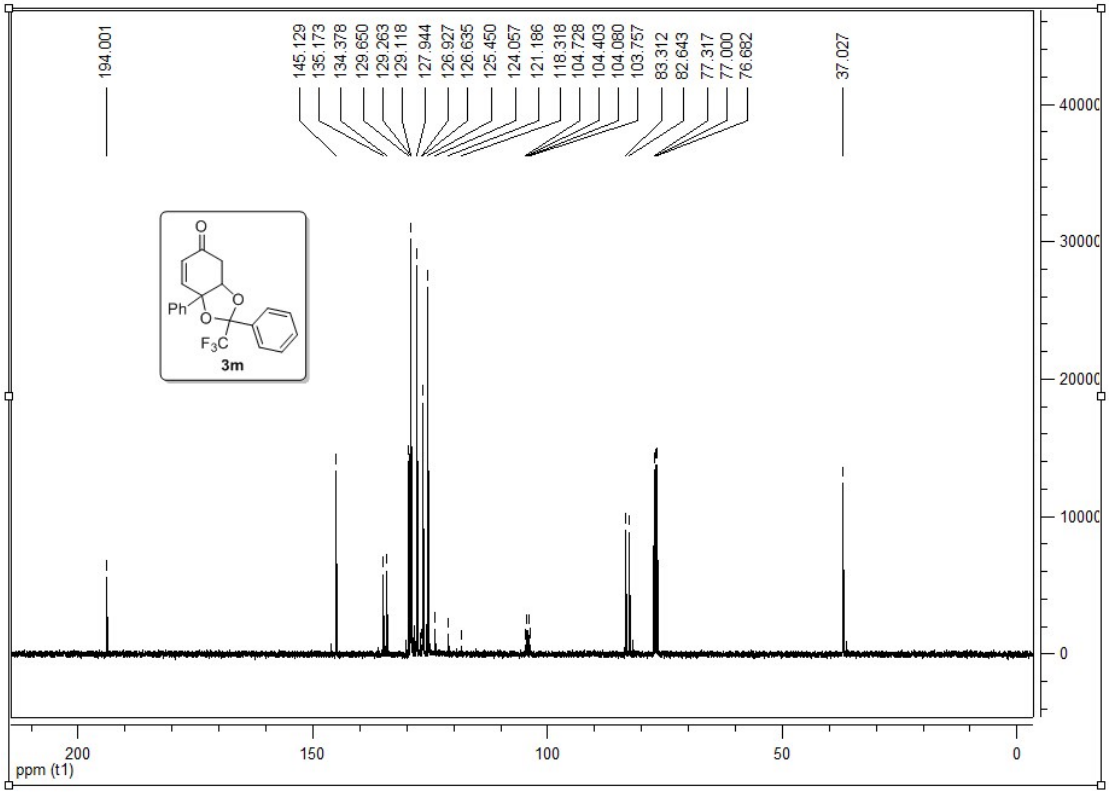

7.329
7.316
7.195
7.188
6.989
6.978
6.968
6.429
6.424
6.403
6.398
5.778
5.752
4.668
3.098
3.053
2.683
2.676
2.639
2.631
1.622
Me
O
S
F3C
3l
1.03
1.01
1.00
1.00
1.00
1.00
1.02
1.08
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

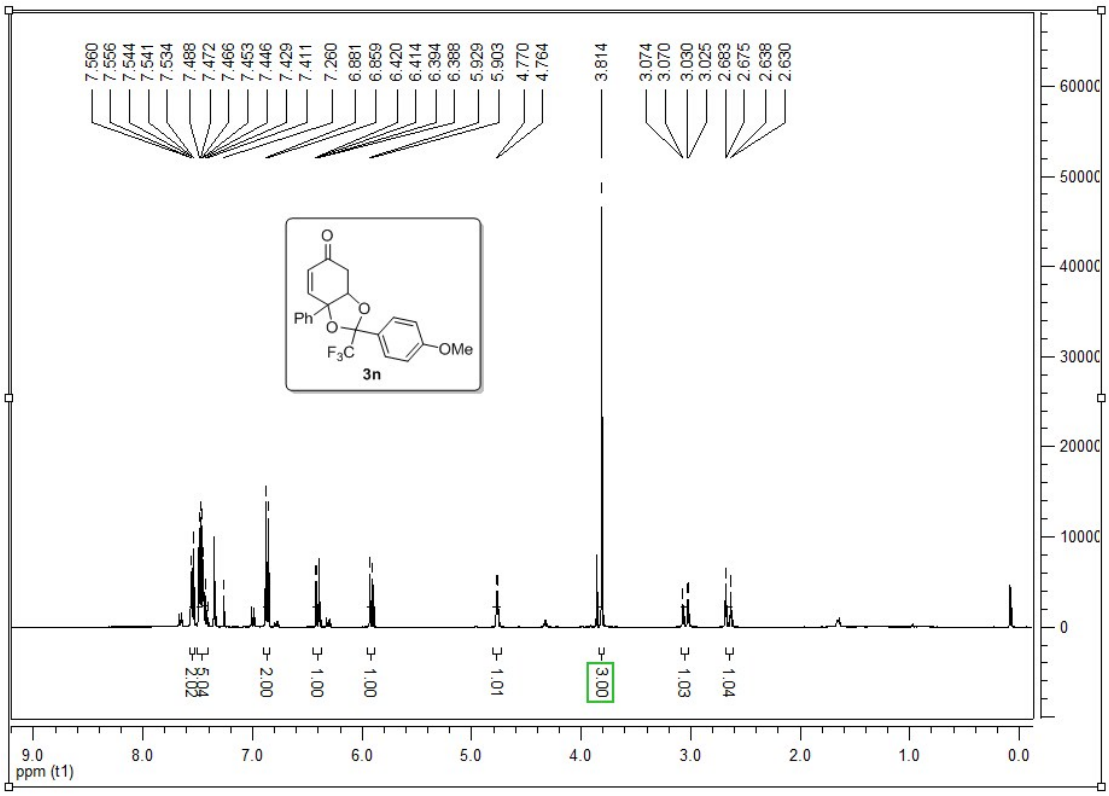

-81.591
-82.635
O
Me
O
O
S
F3C
3l
40000
30000
20000
10000
0
-70.0
-75.0
-80.0
-85.0
-90.0
ppm (t1)

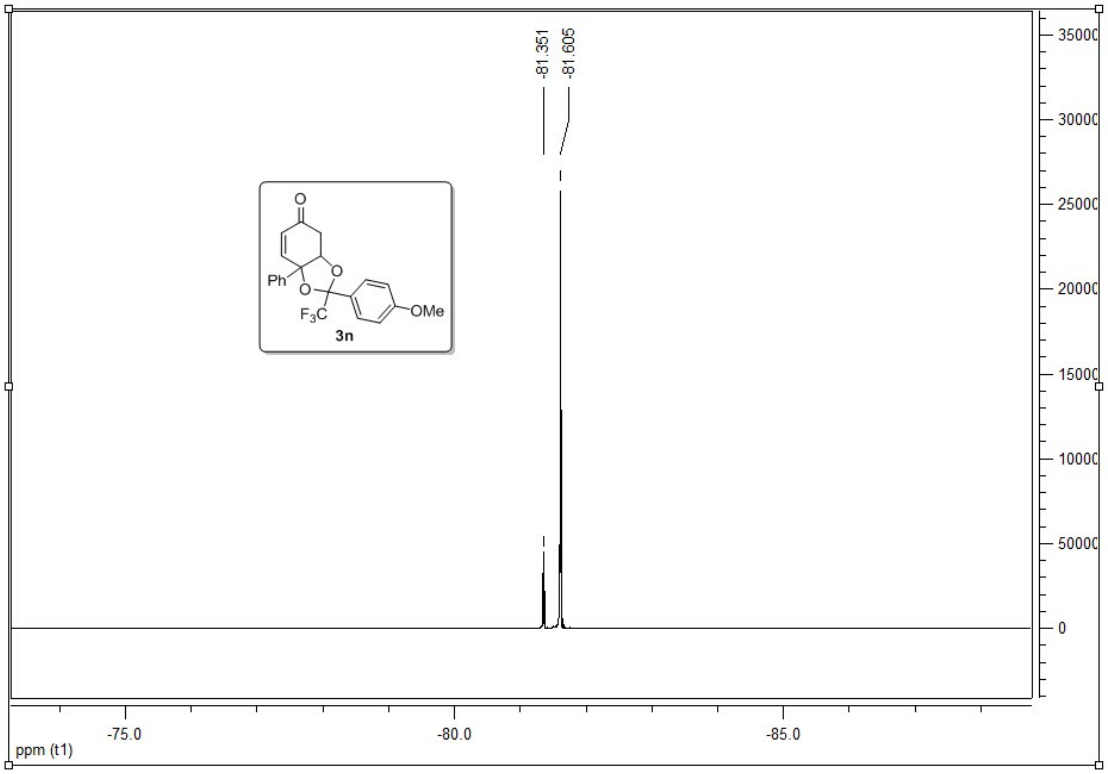

193.529
147.151
136.803
127.853
127.514
127.297
126.982
126.308
123.443
120.579
117.718
103.225
102.884
102.549
102.212
81.967
79.603
77.318
77.000
76.682
37.621
21.150
O
Me
O
O
S
F3C
3l
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

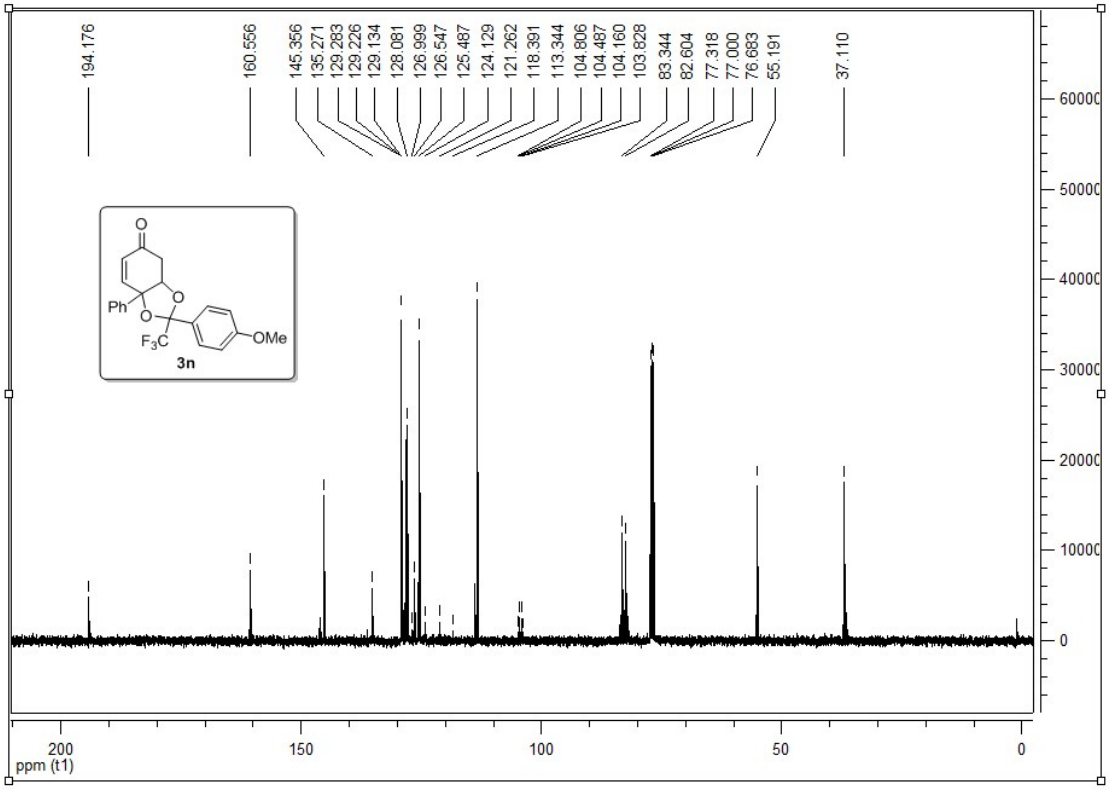

7.581
7.570
7.503
7.486
7.467
7.442
7.406
7.386
7.367
7.260
6.426
6.421
6.400
6.395
5.909
5.883
4.801
3.088
3.043
2.697
2.690
2.652
2.645
Ph
O
F3C
3m
4.02
3.01
3.31
1.00
1.00
1.00
1.06
1.06
ppm (t1)

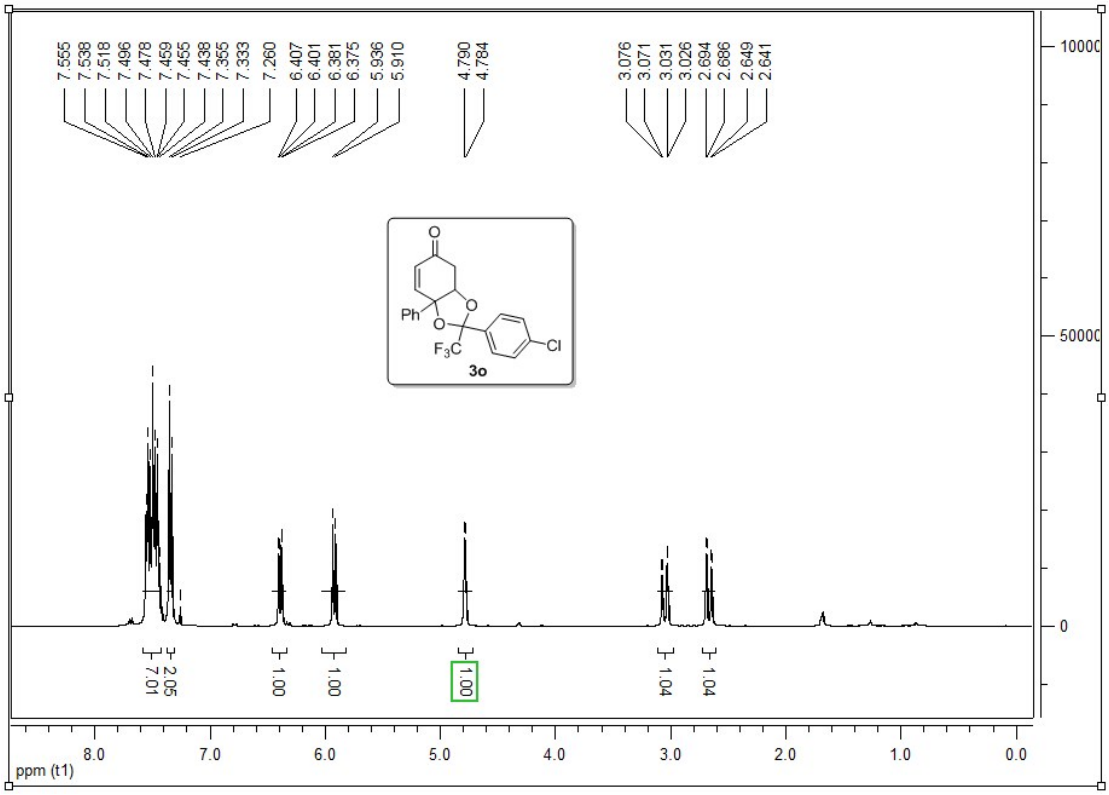

-81.084
-81.407
Ph
O
F3C
3m
ppm (t1)

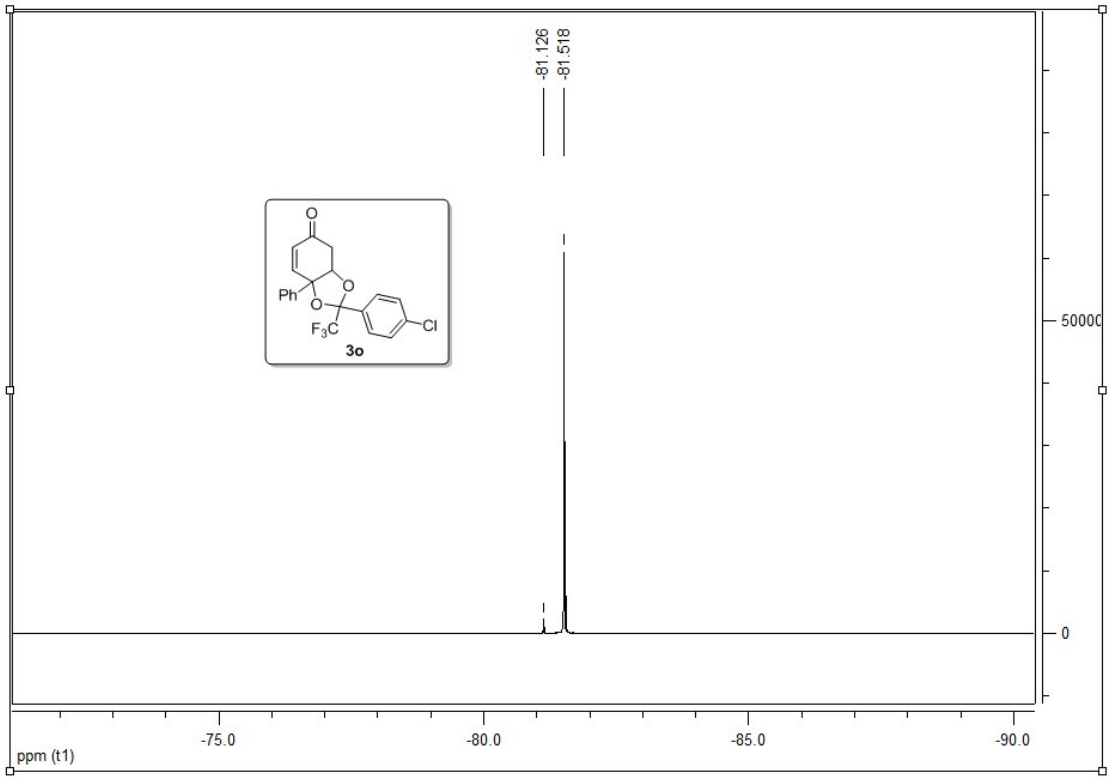

194.001
145.129
135.173
134.378
129.650
129.263
129.118
127.944
126.927
126.635
125.450
124.057
121.186
118.318
104.728
104.403
104.080
103.757
83.312
82.643
77.317
77.000
76.682
37.027
Ph
O
F3C
3m
ppm (t1)

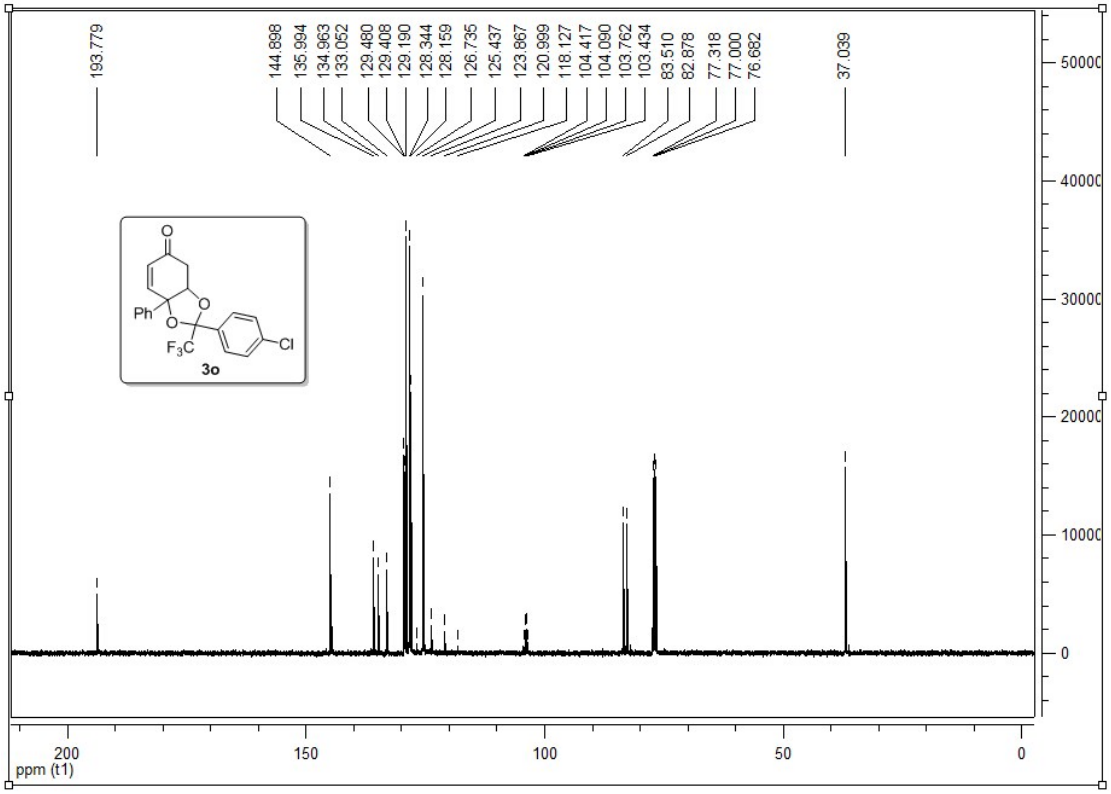

7.560
7.556
7.544
7.541
7.534
7.488
7.472
7.466
7.453
7.446
7.429
7.411
7.260
6.881
6.859
6.420
6.414
6.394
6.388
5.929
5.903
4.770
4.764
3.814
3.074
3.070
3.030
3.025
2.683
2.675
2.638
2.630
Ph
O
F3C
OMe
3n
5.04
2.00
1.00
1.00
1.01
3.00
1.03
1.04
ppm (t1)


-81.351
-81.605
Ph
O
O
F3C
OMe
3n
35000
30000
25000
20000
15000
10000
5000
0
-75.0
-80.0
-85.0
ppm (t1)


194.176
160.556
145.356
135.271
129.283
129.226
129.134
128.081
126.999
126.547
125.487
124.129
121.262
118.391
113.344
104.806
104.487
104.160
103.828
83.344
82.604
77.318
77.000
76.683
55.191
37.110
Ph
O
O
F3C
OMe
3n
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


7.555
7.538
7.518
7.496
7.478
7.459
7.455
7.438
7.365
7.333
7.260
6.407
6.401
6.381
6.375
5.936
5.910
4.790
4.784
3.076
3.071
3.031
3.026
2.694
2.686
2.649
2.641
Ph
O
F3C
Cl
3o
7.01
2.95
1.00
1.00
1.00
1.04
1.04
10000
50000
0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


-81.126
-81.518
Ph
O
F3C
Cl
3o
50000
0
-75.0
-80.0
-85.0
-90.0
ppm (t1)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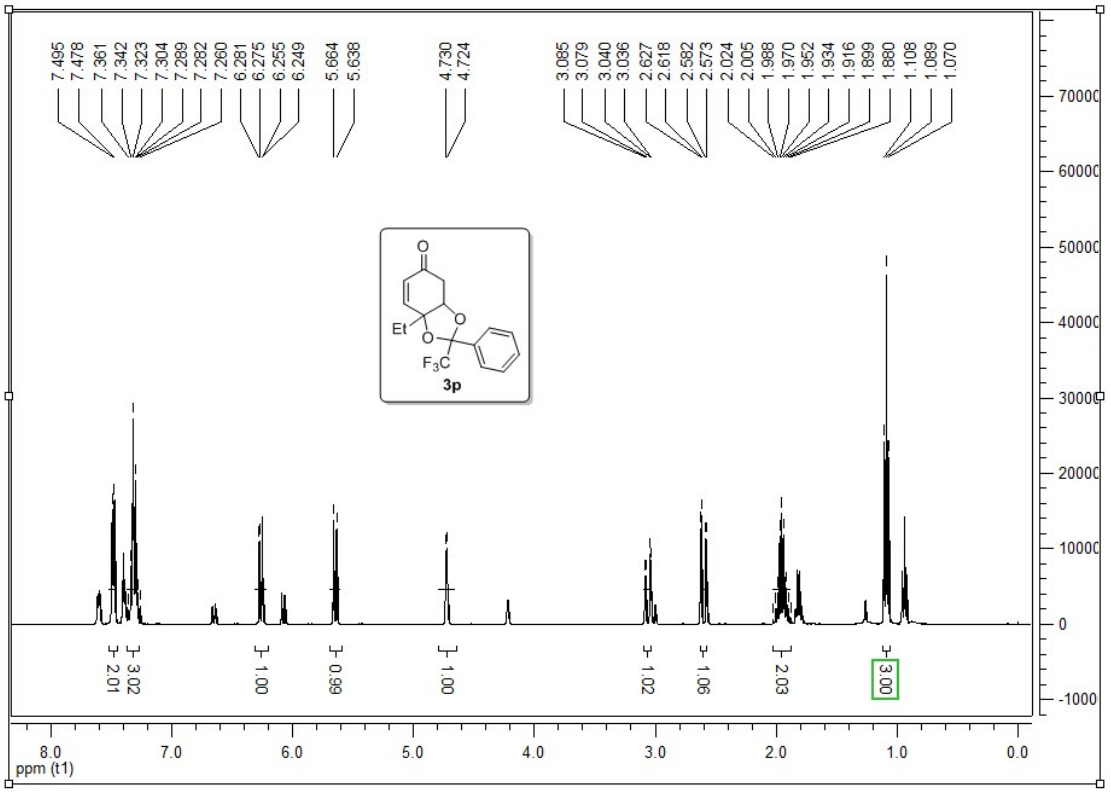
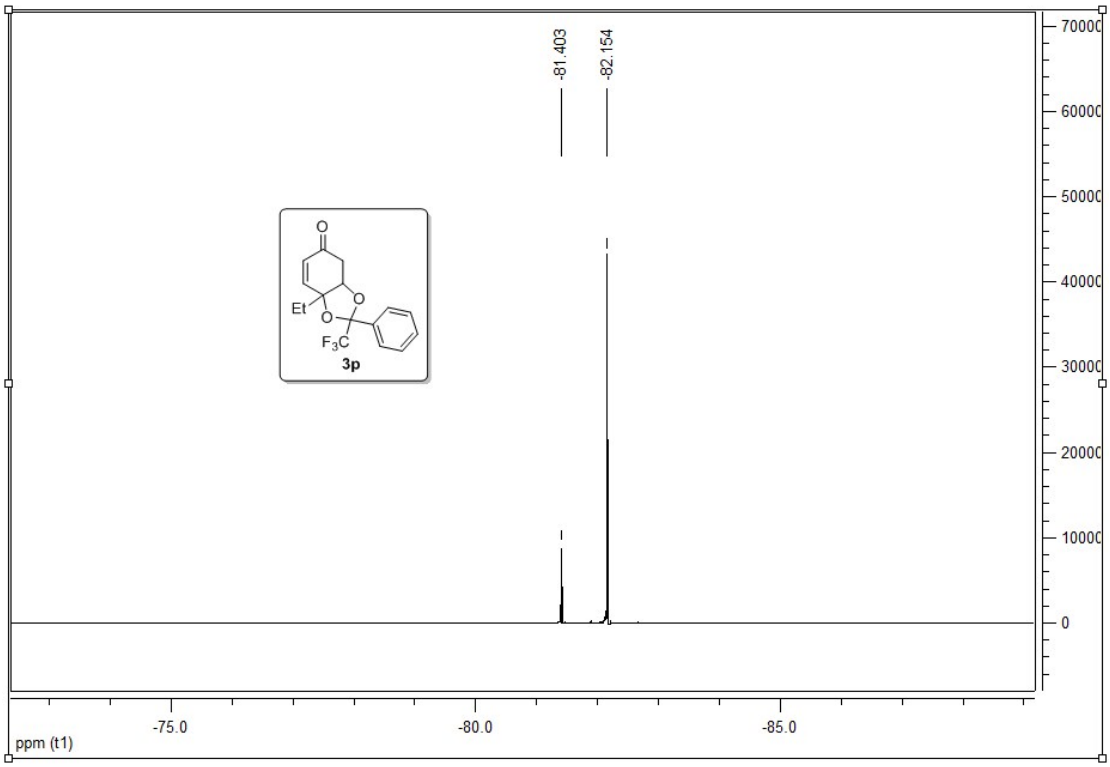
135.994
134.963
133.052
129.480
129.408
129.190
128.344
128.159
126.735
125.437
123.867
120.999
118.127
104.417
104.090
103.762
103.434
83.510
82.878
77.318
77.000
76.682
37.039
Ph
O
F3C
Cl
3o
ppm (t1)


7.495
7.478
7.361
7.342
7.323
7.304
7.289
7.282
7.260
6.281
6.275
6.255
6.249
5.664
5.638
4.730
4.724
3.085
3.079
3.040
3.036
2.627
2.618
2.582
2.573
2.024
2.005
1.988
1.970
1.952
1.934
1.916
1.899
1.880
1.108
1.089
1.070
Et
O
F3C
3p
2.01
3.02
1.00
0.99
1.00
1.02
1.06
2.03
3.00
ppm (t1)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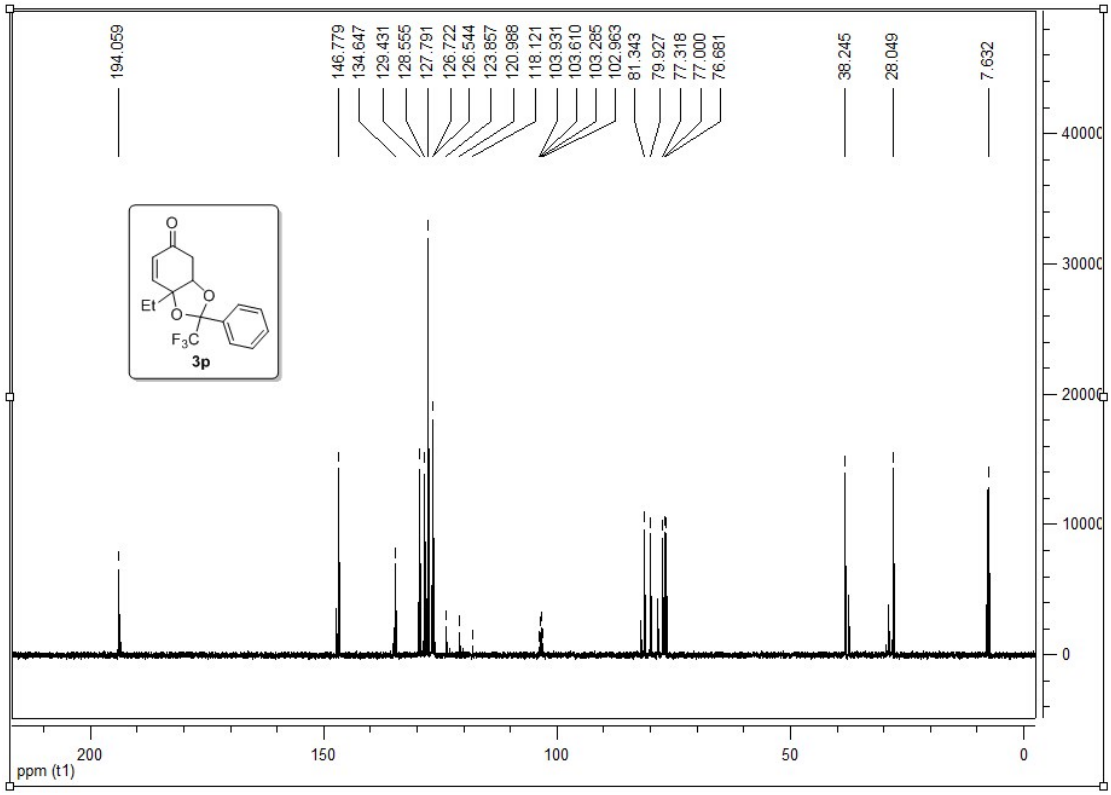
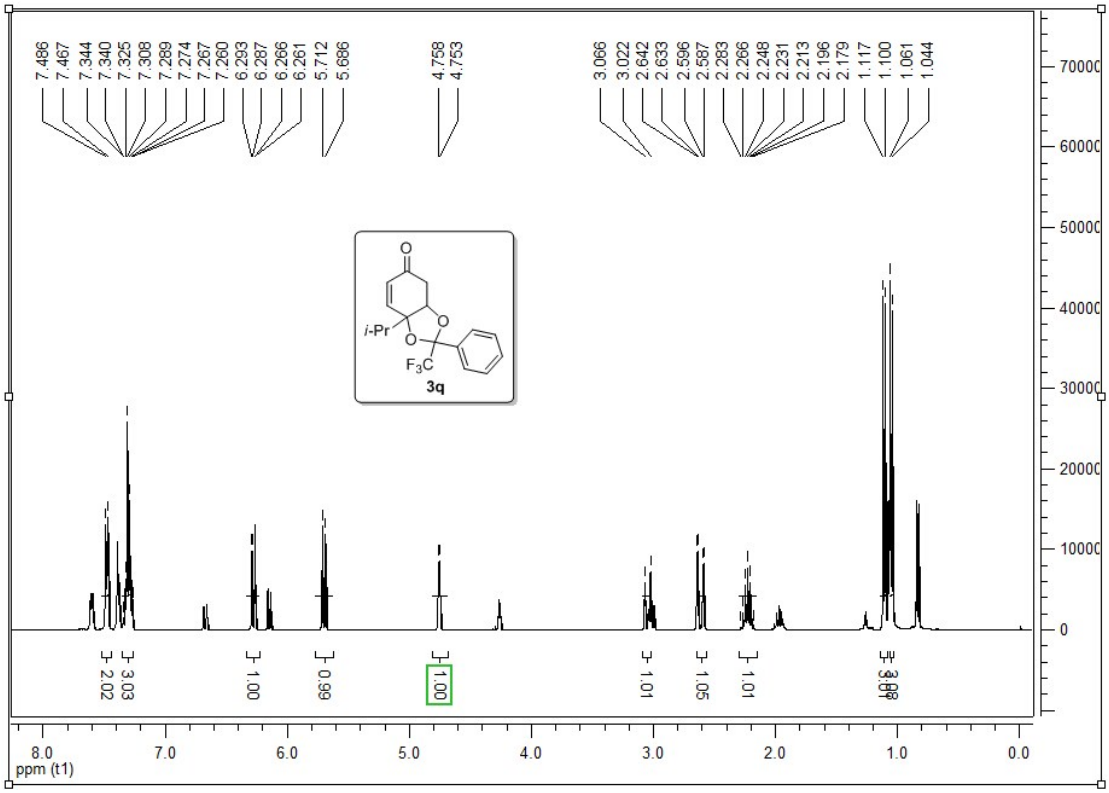
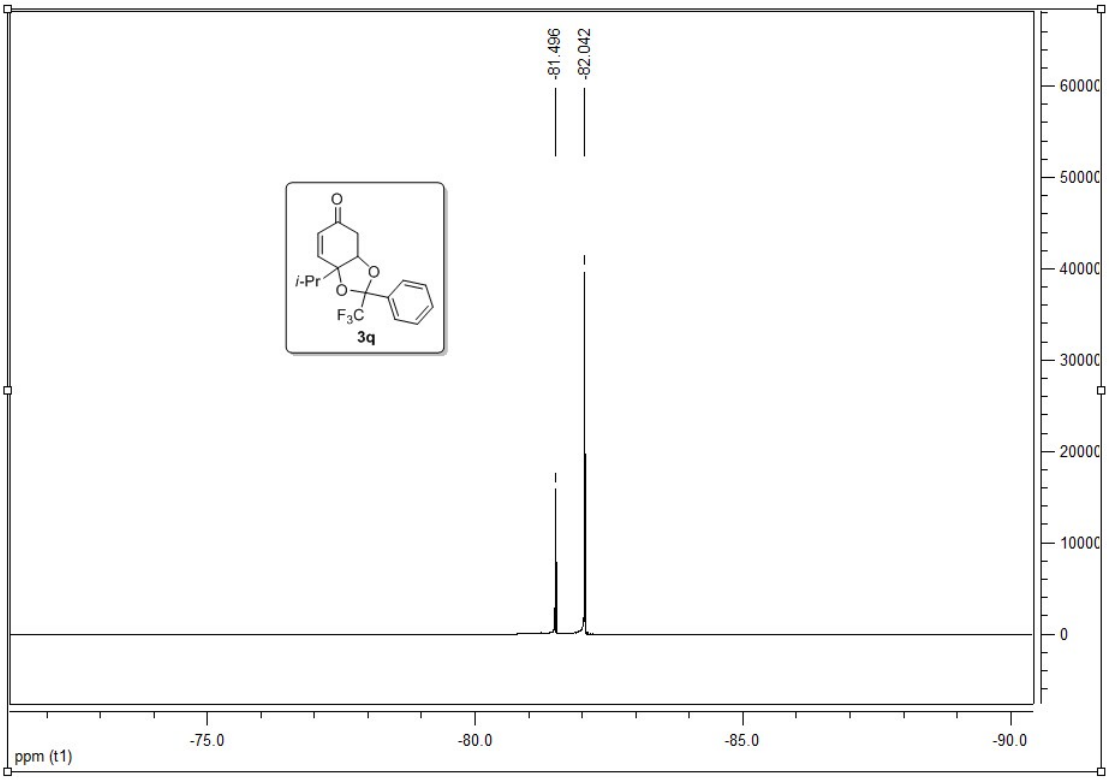
-82.154
Et
F3C
3p
ppm (t1)


194.059
146.779
134.647
129.431
128.555
127.791
126.722
126.544
123.857
120.988
118.121
103.931
103.610
103.285
102.963
81.343
79.927
77.318
77.000
76.681
38.245
28.049
7.632
Et
F3C
3p
ppm (t1)


7.486
7.467
7.344
7.340
7.325
7.308
7.289
7.274
7.267
7.260
6.293
6.287
6.266
6.261
5.712
5.686
4.758
4.753
3.066
3.022
2.642
2.633
2.596
2.587
2.283
2.266
2.248
2.231
2.213
2.196
2.179
1.117
1.100
1.061
1.044
i-Pr
F3C
3q
2.02
3.03
1.00
0.99
1.00
1.01
1.05
1.01
3.08
ppm (t1)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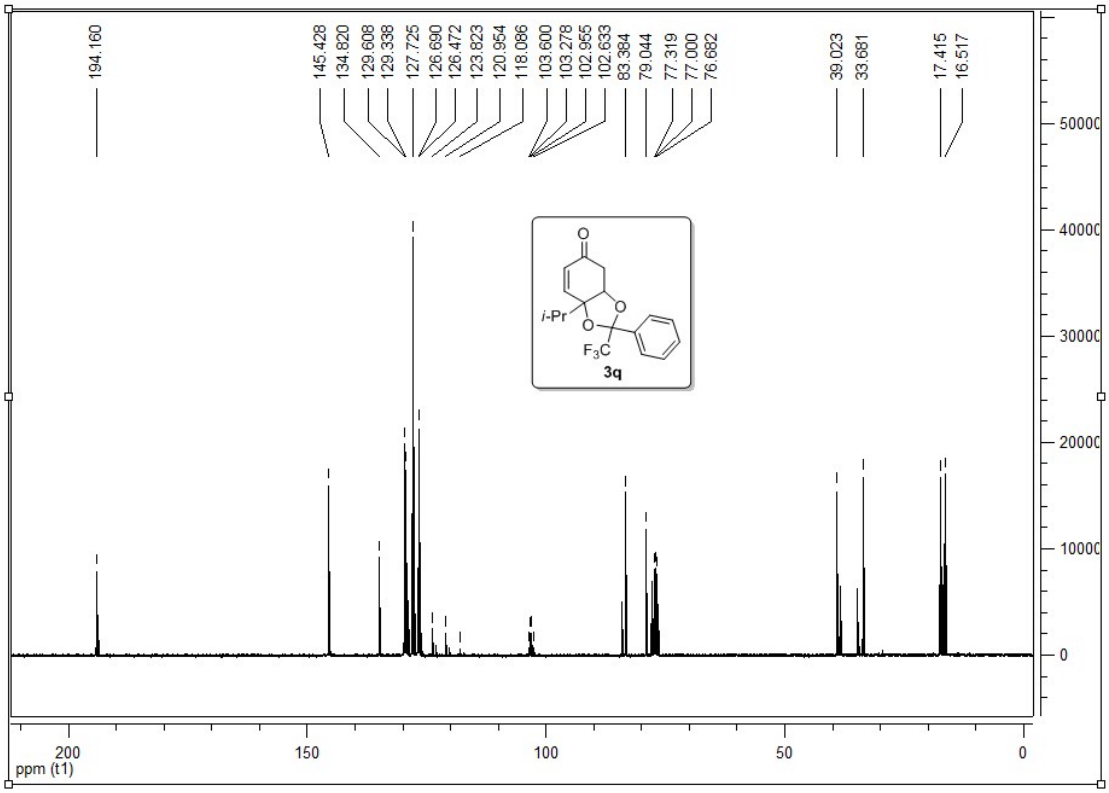
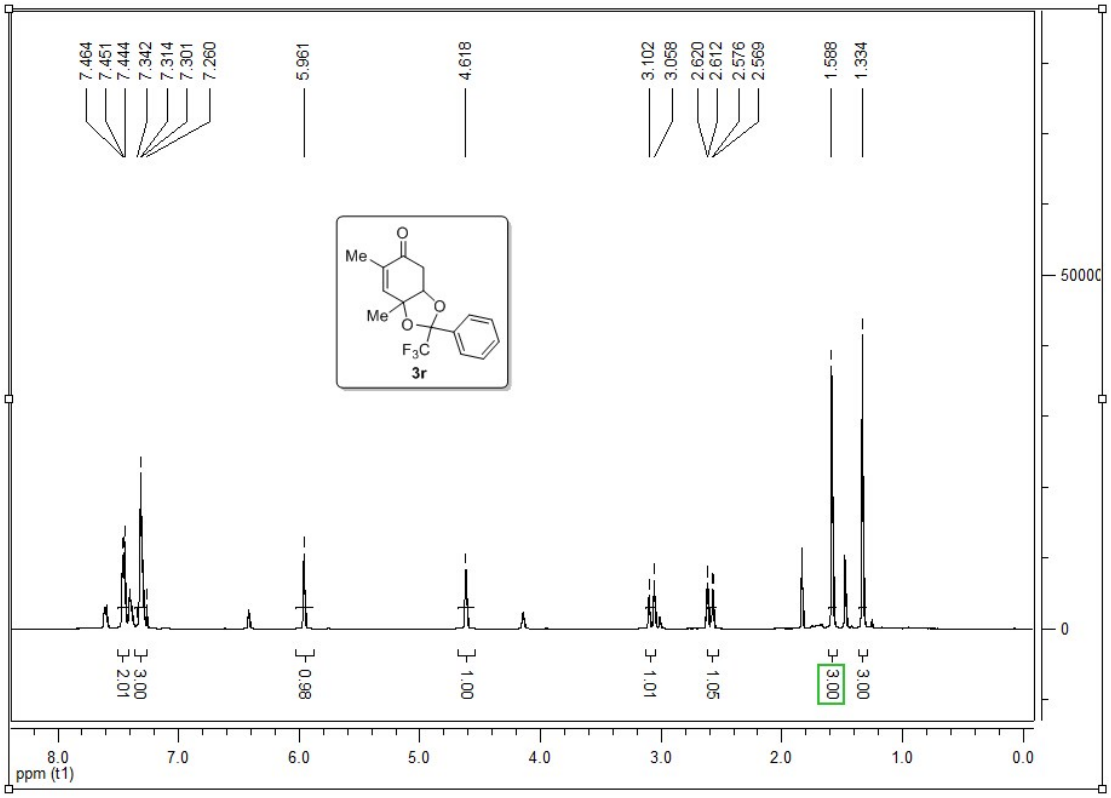
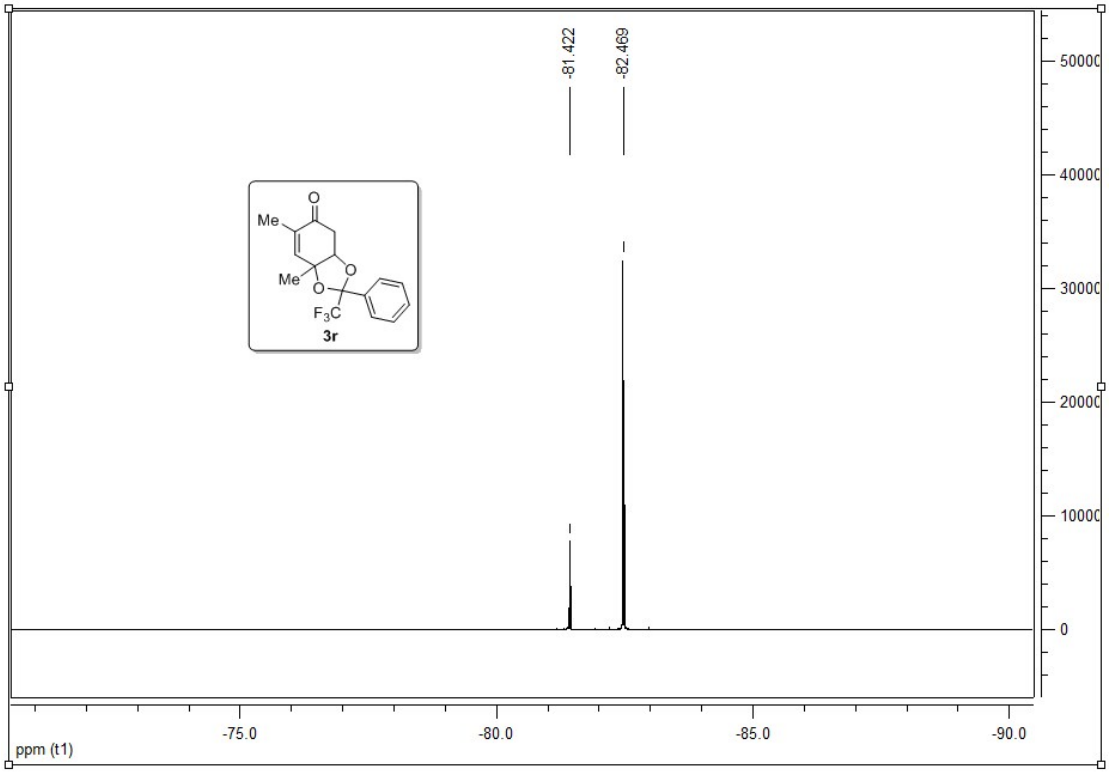
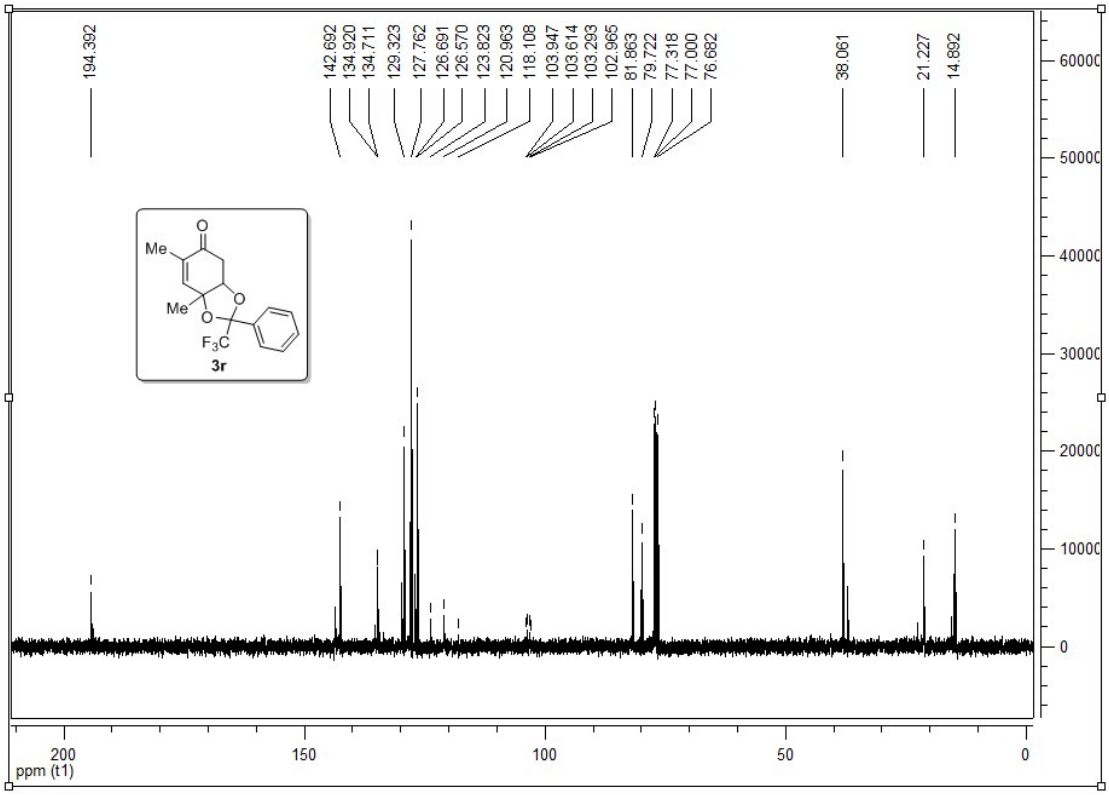
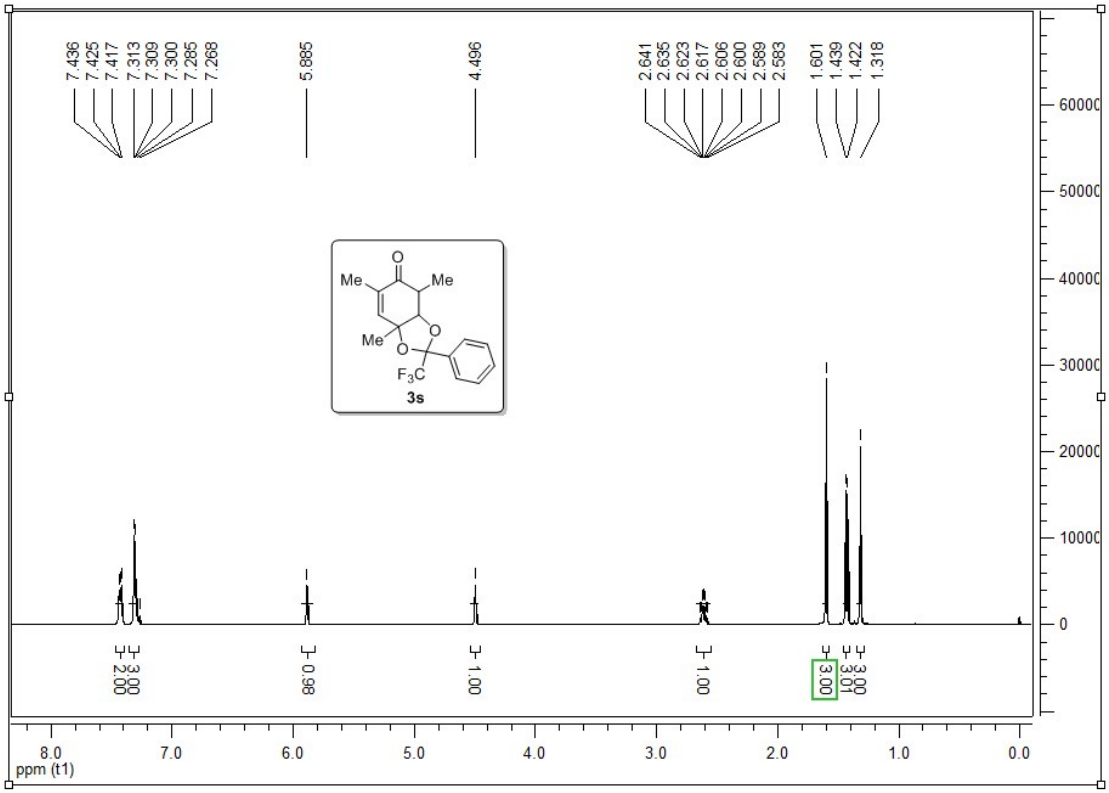
-82.042
i-Pr
F3C
3q
ppm (t1)

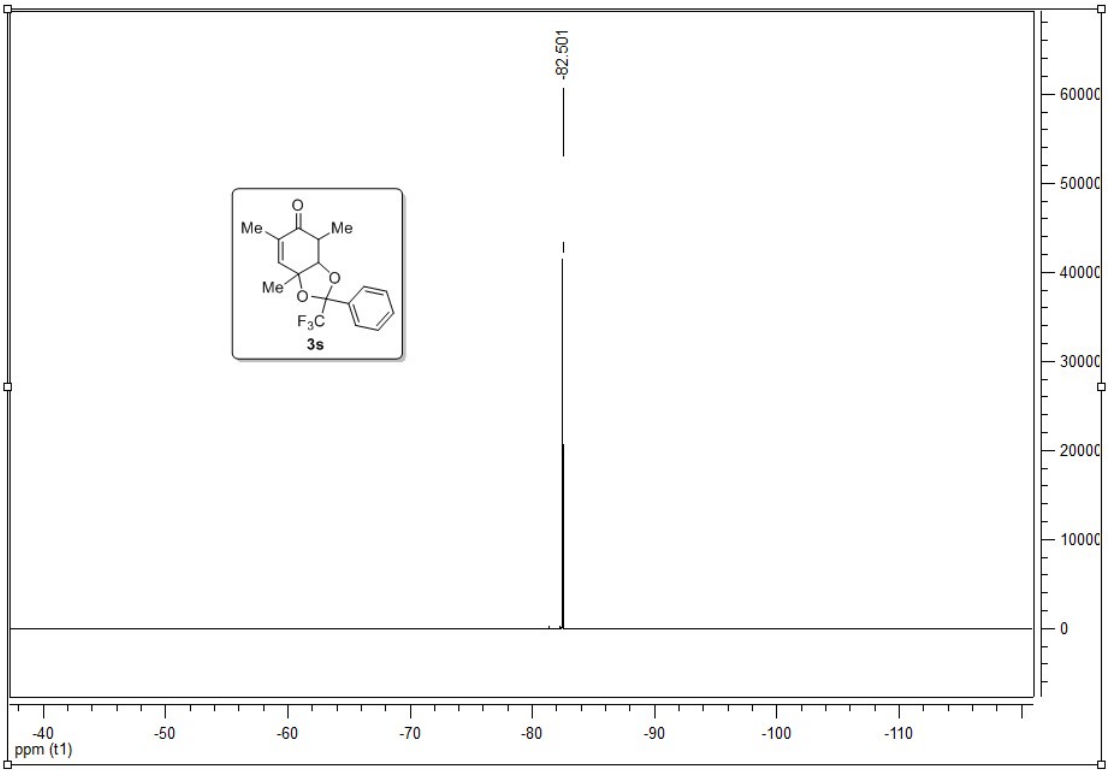

194.160
145.428
134.820
129.608
129.338
127.725
126.690
126.472
123.823
120.954
118.086
103.600
103.278
102.955
102.633
83.384
79.044
77.319
77.000
76.682
39.023
33.681
17.415
16.517
i-Pr
F3C
3q
ppm (t1)


7.464
7.451
7.444
7.342
7.314
7.301
7.260
5.961
4.618
3.102
3.058
2.620
2.612
2.576
2.569
1.588
1.334
Me
Me
F3C
3r
2.01
3.00
0.98
1.00
1.01
1.05
3.00
3.00
ppm (t1)


-81.422
-82.469
Me
Me
O
O
O
F3C
3r
50000
40000
30000
20000
10000
0
-75.0
-80.0
-85.0
-90.0
ppm (t1)


194.392
142.692
134.920
134.711
129.323
127.762
126.691
126.570
123.823
120.963
118.108
103.947
103.614
103.293
102.965
81.863
79.722
77.318
77.000
76.682
38.061
21.227
14.892
Me
Me
O
O
O
F3C
3r
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)


7.436
7.425
7.417
7.313
7.309
7.300
7.285
7.268
5.885
4.496
2.641
2.635
2.623
2.617
2.606
2.600
2.589
2.583
1.601
1.439
1.422
1.318
Me
Me
Me
O
O
F3C
3s
2.00
3.00
0.98
1.00
1.00
3.00
3.01
3.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


-82.501
Me
Me
Me
O
O
F3C
3s
-40
-50
-60
-70
-80
-90
-100
-110
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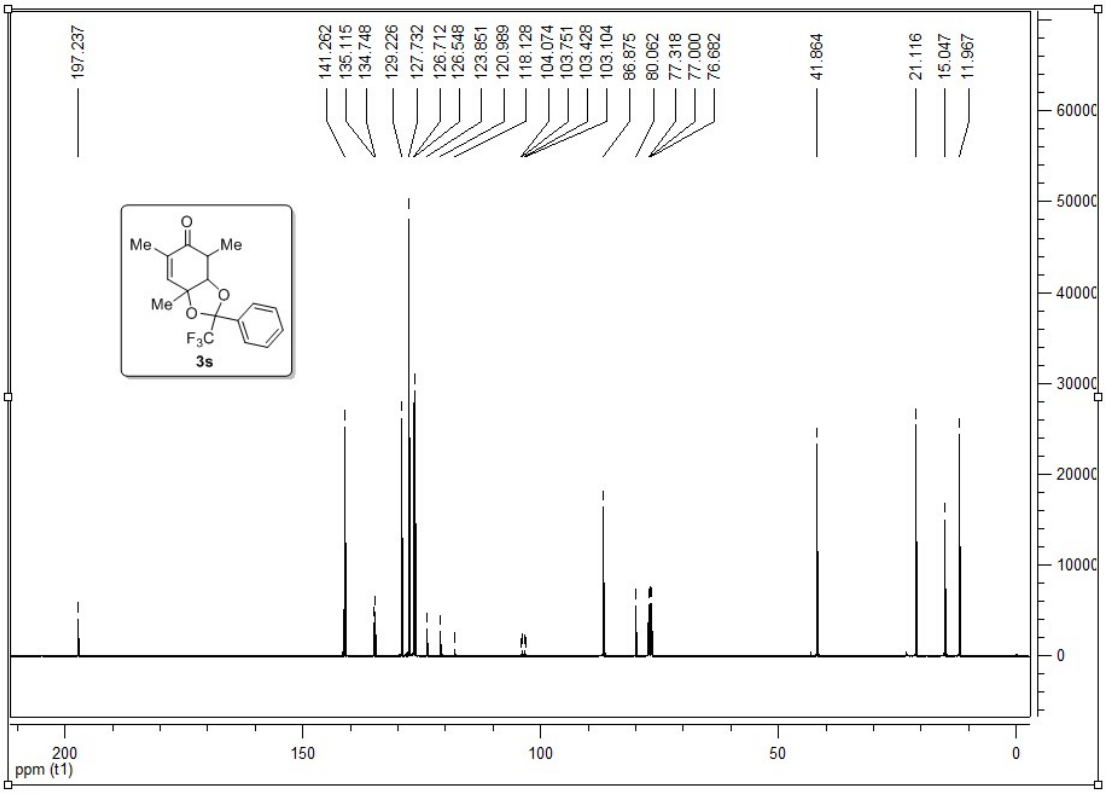
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118.128
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103.428
103.104
86.875
80.062
77.318
77.000
76.682
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21.116
15.047
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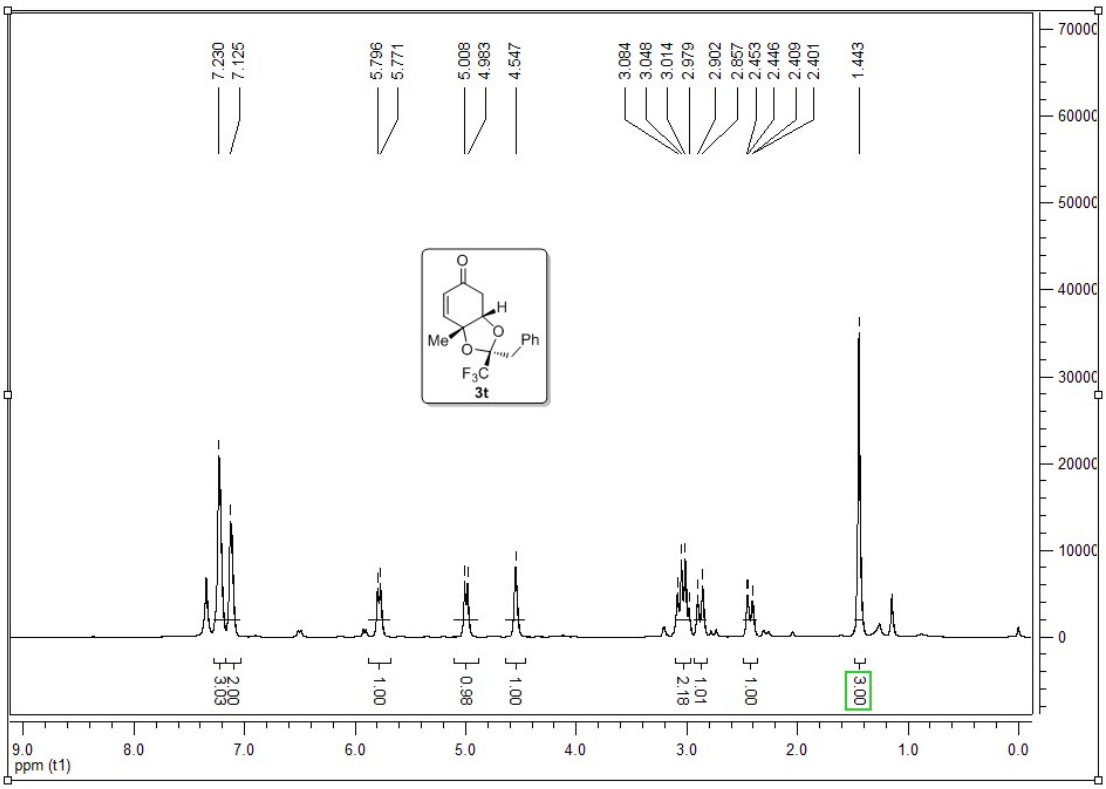
Me
Me
O
O
O
F3C
3s
ppm (t1)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4.547
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3.014
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2.453
2.446
2.409
2.401
1.443
O
H
Me
O
O
Ph
F3C
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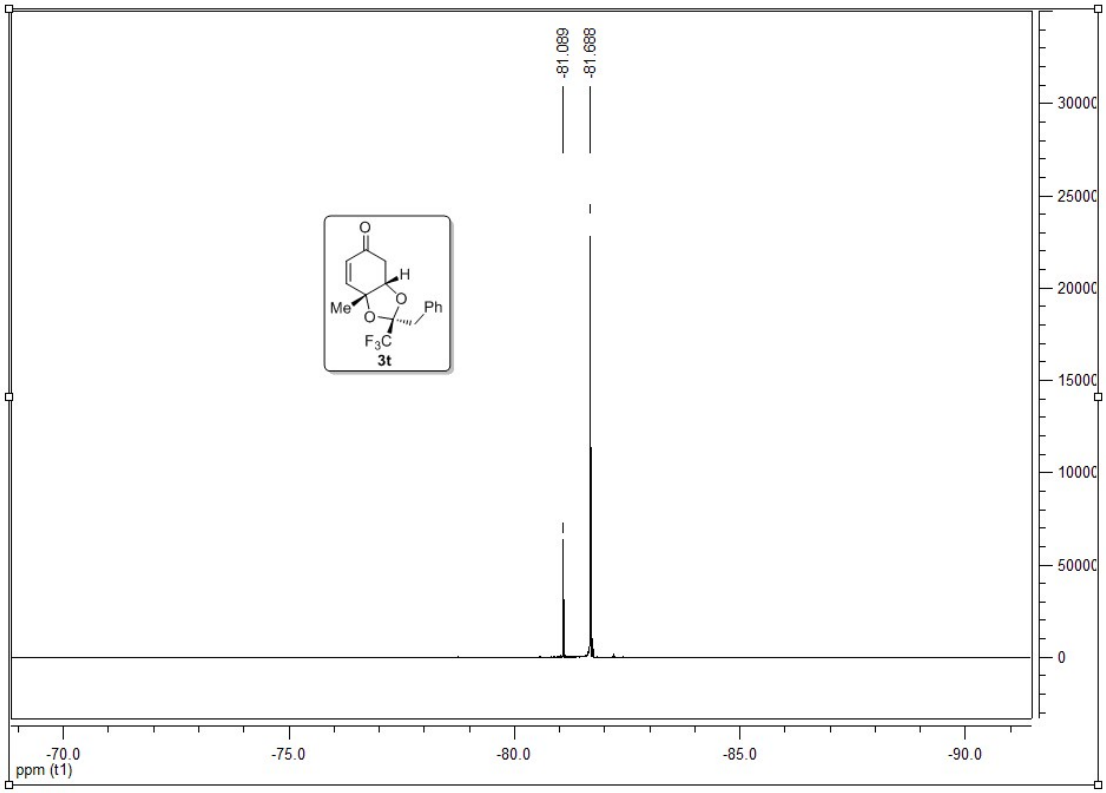
1.00
3.00
ppm (t1)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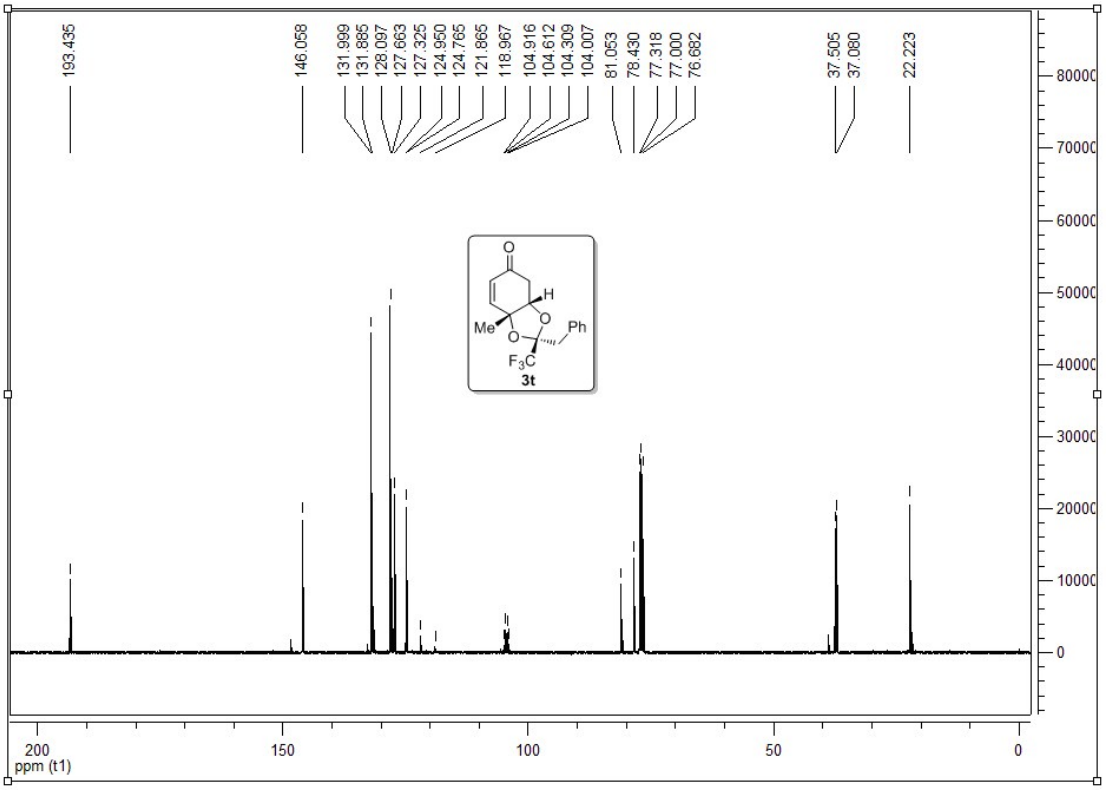
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104.612
104.309
104.007
81.053
78.430
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77.000
76.682
37.505
37.080
22.223
3t
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150
100
50
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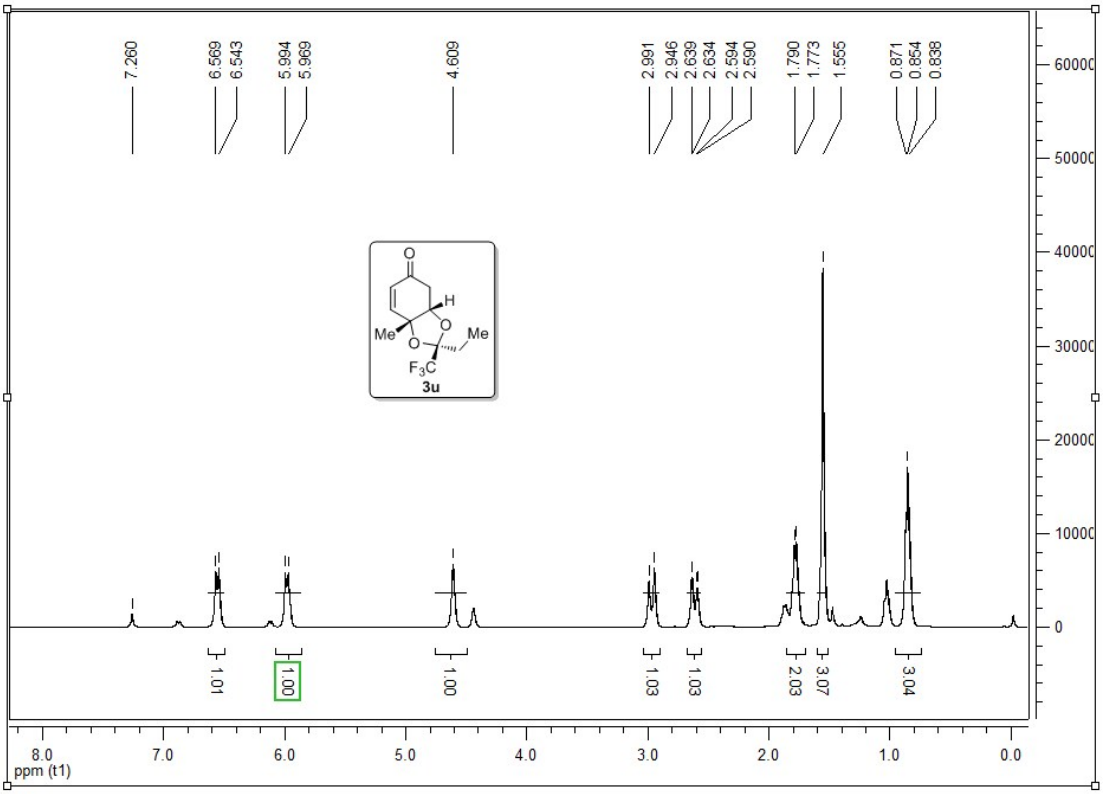
ppm (t1)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2.634
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1.790
1.773
1.555
0.871
0.854
0.838
1.01
1.00
1.00
1.03
1.03
2.03
3.07
3.04
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7.0
6.0
5.0
4.0
3.0
2.0
1.0
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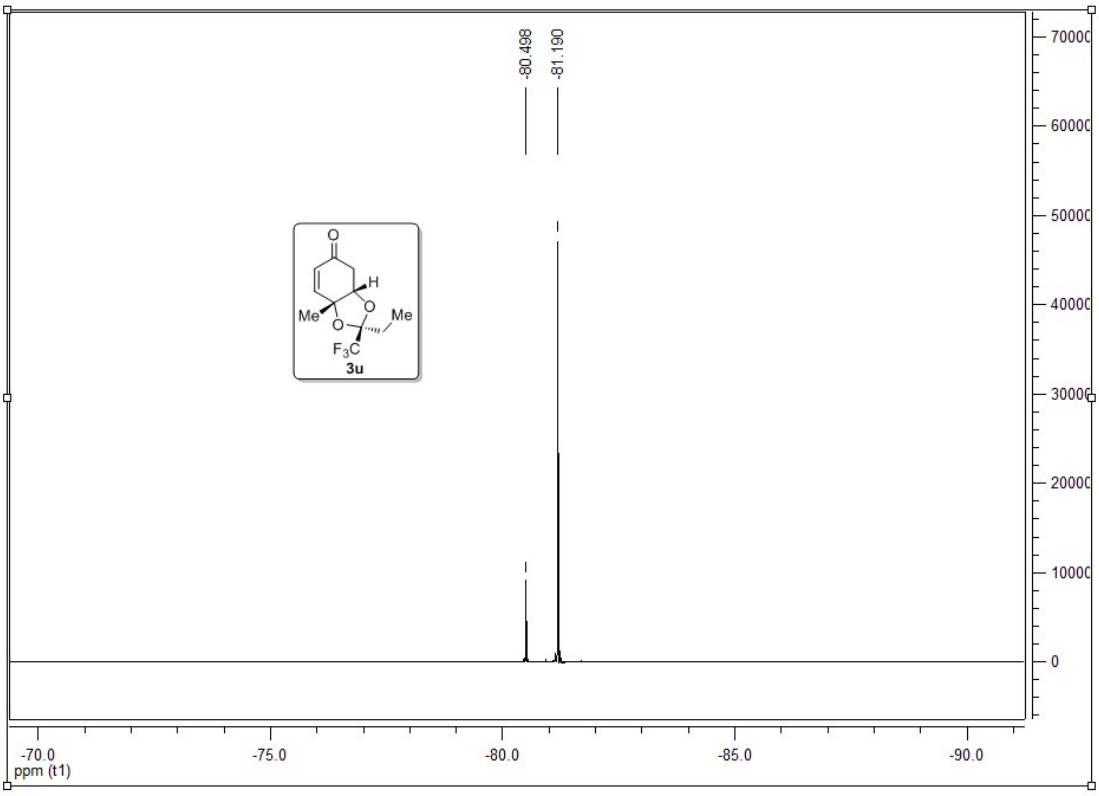
ppm (t1)
3u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-85.0
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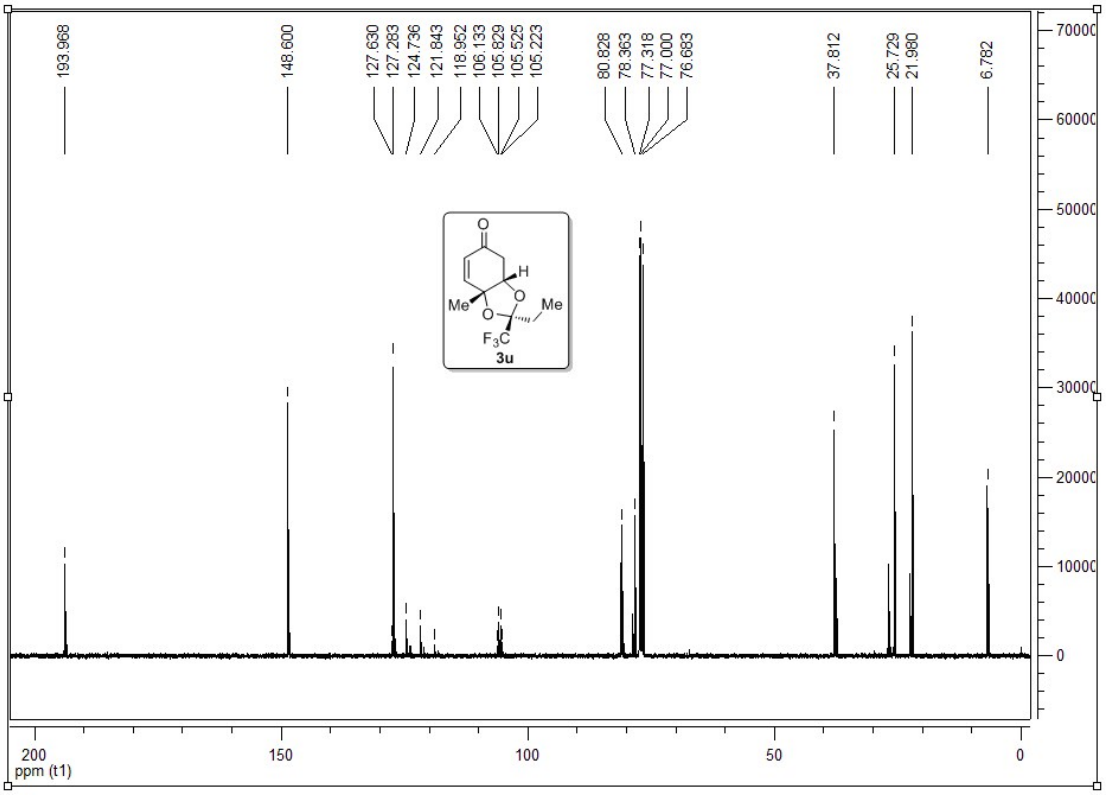
ppm (t1)
3u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78.363
77.318
77.000
76.683
37.812
25.729
21.980
6.782
O
H
Me
O
O
Me
F3C
3u
70000
60000
50000
40000
30000
20000
10000
0
200
150
100
50
0
ppm (t1)

X-Ray Analysis for the product 3a:

CCDC 1061550 contains the supplementary crystallographic data for the product **3a**. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

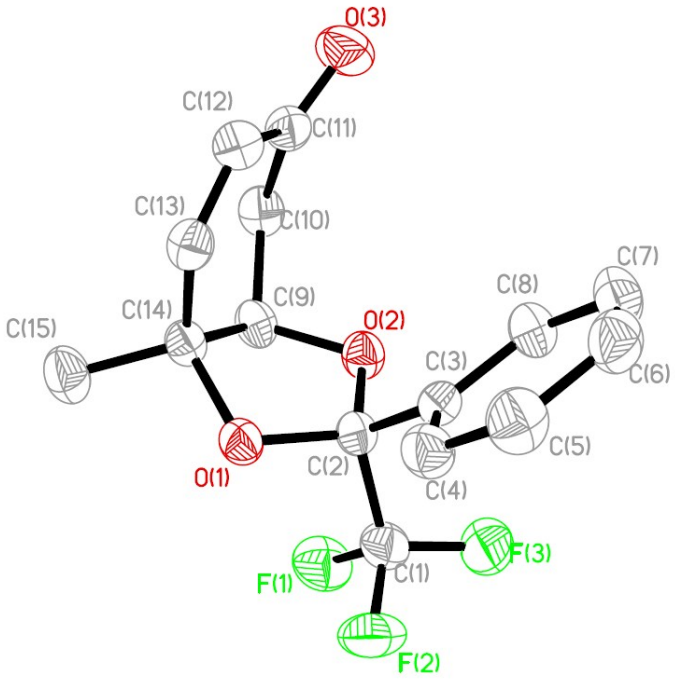

Table 1. Crystal data and structure refinement for 130703A.

Empirical formula	C15 H13 F3 O3
Formula weight	298.25
Temperature	294(2) K
Wavelength	0.71073 Å
Crystal system, space group	MONOCLINIC, C2/c
Unit cell dimensions	a = 28.78(3) A alpha = 90 deg. b = 6.621(7) A beta = 106.775(15) deg. c = 15.245(18) A gamma = 90 deg.
Volume	2782(5) Å ^{3}
Z, Calculated density	8, 1.424 Mg/m^3
Absorption coefficient	0.124 mm^{-1}
F(000)	1232
Crystal size	0.17 x 0.14 x 0.05 mm
Theta range for data collection	3.16 to 25.03 deg.
Limiting indices	-34<=h<=34, -7<=k<=7, -18<=l<=18
Reflections collected / unique	11489 / 2448 [R(int) = 0.0959]
Completeness to theta = 25.03	99.6 %

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9938 and 0.9792
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2448 / 0 / 192
Goodness-of-fit on F^2	1.055
Final R indices [I>2sigma(I)]	R1 = 0.0545, wR2 = 0.1312
R indices (all data)	R1 = 0.0833, wR2 = 0.1499
Extinction coefficient	0.0005(5)
Largest diff. peak and hole	0.161 and -0.166 $e.A^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 130703A. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	2042(1)	2269(5)	8616(2)	65(1)
C(2)	1577(1)	2492(3)	7820(2)	48(1)
C(3)	1137(1)	1870(3)	8099(2)	48(1)
C(4)	1014(1)	-139(4)	8128(2)	63(1)
C(5)	609(1)	-660(5)	8397(2)	74(1)
C(6)	330(1)	777(5)	8632(2)	73(1)
C(7)	449(1)	2758(5)	8601(2)	72(1)
C(8)	851(1)	3316(4)	8345(2)	62(1)
C(9)	1650(1)	4654(4)	6683(2)	53(1)
C(10)	1351(1)	6342(4)	6137(2)	59(1)
C(11)	816(1)	5891(4)	5876(2)	58(1)
C(12)	678(1)	3796(4)	5636(2)	58(1)
C(13)	994(1)	2305(4)	5820(2)	54(1)
C(14)	1525(1)	2555(3)	6283(2)	47(1)
C(15)	1828(1)	1870(4)	5678(2)	72(1)
F(1)	2429(1)	2921(3)	8396(1)	87(1)
F(2)	2132(1)	356(3)	8878(1)	89(1)
F(3)	2013(1)	3313(3)	9338(1)	95(1)
O(1)	1653(1)	1312(2)	7101(1)	53(1)
O(2)	1541(1)	4519(2)	7542(1)	54(1)
O(3)	520(1)	7210(3)	5853(2)	85(1)

Table 3. Bond lengths [A] and angles [deg] for 130703A.

C(1)-F(3)	1.323(3)
C(1)-F(1)	1.325(3)
C(1)-F(2)	1.331(4)
C(1)-C(2)	1.532(4)
C(2)-O(2)	1.402(3)
C(2)-O(1)	1.413(3)

C(2)-C(3)	1.505(4)
C(3)-C(4)	1.381(4)
C(3)-C(8)	1.384(4)
C(4)-C(5)	1.384(4)
C(4)-H(4)	0.9300
C(5)-C(6)	1.360(4)
C(5)-H(5)	0.9300
C(6)-C(7)	1.361(4)
C(6)-H(6)	0.9300
C(7)-C(8)	1.373(4)
C(7)-H(7)	0.9300
C(8)-H(8)	0.9300
C(9)-O(2)	1.435(3)
C(9)-C(10)	1.506(4)
C(9)-C(14)	1.518(3)
C(9)-H(9)	0.9800
C(10)-C(11)	1.507(4)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(11)-O(3)	1.213(3)
C(11)-C(12)	1.460(4)
C(12)-C(13)	1.317(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.495(4)
C(13)-H(13)	0.9300
C(14)-O(1)	1.451(3)
C(14)-C(15)	1.510(4)
C(15)-H(15A)	0.9600
C(15)-H(15B)	0.9600
C(15)-H(15C)	0.9600
F(3)-C(1)-F(1)	107.5(2)
F(3)-C(1)-F(2)	107.6(3)
F(1)-C(1)-F(2)	106.3(2)
F(3)-C(1)-C(2)	111.0(2)
F(1)-C(1)-C(2)	112.0(2)
F(2)-C(1)-C(2)	112.3(2)
O(2)-C(2)-O(1)	107.99(19)
O(2)-C(2)-C(3)	110.61(18)
O(1)-C(2)-C(3)	113.4(2)
O(2)-C(2)-C(1)	107.7(2)
O(1)-C(2)-C(1)	105.4(2)
C(3)-C(2)-C(1)	111.4(2)
C(4)-C(3)-C(8)	118.5(3)
C(4)-C(3)-C(2)	121.3(2)

C(8)-C(3)-C(2)	120.2(2)
C(3)-C(4)-C(5)	119.8(2)
C(3)-C(4)-H(4)	120.1
C(5)-C(4)-H(4)	120.1
C(6)-C(5)-C(4)	121.1(3)
C(6)-C(5)-H(5)	119.5
C(4)-C(5)-H(5)	119.5
C(7)-C(6)-C(5)	119.4(3)
C(7)-C(6)-H(6)	120.3
C(5)-C(6)-H(6)	120.3
C(6)-C(7)-C(8)	120.7(3)
C(6)-C(7)-H(7)	119.6
C(8)-C(7)-H(7)	119.6
C(7)-C(8)-C(3)	120.5(3)
C(7)-C(8)-H(8)	119.7
C(3)-C(8)-H(8)	119.7
O(2)-C(9)-C(10)	108.0(2)
O(2)-C(9)-C(14)	102.73(18)
C(10)-C(9)-C(14)	115.2(2)
O(2)-C(9)-H(9)	110.2
C(10)-C(9)-H(9)	110.2
C(14)-C(9)-H(9)	110.2
C(11)-C(10)-C(9)	112.1(2)
C(11)-C(10)-H(10A)	109.2
C(9)-C(10)-H(10A)	109.2
C(11)-C(10)-H(10B)	109.2
C(9)-C(10)-H(10B)	109.2
H(10A)-C(10)-H(10B)	107.9
O(3)-C(11)-C(12)	122.6(3)
O(3)-C(11)-C(10)	121.4(3)
C(12)-C(11)-C(10)	116.0(2)
C(13)-C(12)-C(11)	122.5(3)
C(13)-C(12)-H(12)	118.7
C(11)-C(12)-H(12)	118.7
C(12)-C(13)-C(14)	124.5(2)
C(12)-C(13)-H(13)	117.7
C(14)-C(13)-H(13)	117.7
O(1)-C(14)-C(13)	108.32(18)
O(1)-C(14)-C(15)	107.9(2)
C(13)-C(14)-C(15)	111.6(2)
O(1)-C(14)-C(9)	101.65(19)
C(13)-C(14)-C(9)	112.5(2)
C(15)-C(14)-C(9)	114.1(2)
C(14)-C(15)-H(15A)	109.5

C(14)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(14)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(2)-O(1)-C(14)	107.19(18)
C(2)-O(2)-C(9)	108.61(16)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 130703A. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	48(2)	80(2)	65(2)	-4(2)	13(1)	7(1)
C(2)	53(1)	46(1)	46(1)	-5(1)	15(1)	4(1)
C(3)	49(1)	54(1)	39(1)	-1(1)	10(1)	2(1)
C(4)	71(2)	56(2)	68(2)	6(1)	28(2)	7(1)
C(5)	82(2)	65(2)	83(2)	7(2)	35(2)	-7(2)
C(6)	63(2)	95(2)	66(2)	1(2)	28(2)	-6(2)
C(7)	66(2)	84(2)	74(2)	-10(2)	34(2)	4(2)
C(8)	65(2)	59(2)	67(2)	-8(1)	26(1)	3(1)
C(9)	50(1)	51(1)	62(2)	-5(1)	26(1)	-5(1)
C(10)	76(2)	41(1)	67(2)	3(1)	31(1)	-4(1)
C(11)	67(2)	56(2)	52(1)	7(1)	18(1)	9(1)
C(12)	58(2)	56(2)	58(1)	2(1)	15(1)	-3(1)
C(13)	62(2)	46(1)	53(1)	-5(1)	18(1)	-7(1)
C(14)	55(1)	41(1)	53(1)	3(1)	25(1)	1(1)
C(15)	88(2)	65(2)	78(2)	3(2)	48(2)	9(2)
F(1)	49(1)	107(1)	100(1)	-4(1)	16(1)	-3(1)
F(2)	73(1)	95(1)	88(1)	27(1)	6(1)	22(1)
F(3)	71(1)	141(2)	62(1)	-27(1)	-1(1)	14(1)
O(1)	63(1)	47(1)	53(1)	1(1)	22(1)	9(1)
O(2)	62(1)	48(1)	55(1)	-5(1)	21(1)	0(1)
O(3)	85(2)	68(1)	95(2)	-2(1)	17(1)	26(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 130703A.

	x	y	z	U(eq)
H(4)	1201	-1138	7968	76
H(5)	527	-2015	8416	89
H(6)	59	409	8813	87

H(7)	257	3746	8756	86
H(8)	930	4675	8336	75
H(9)	1997	4925	6786	63
H(10A)	1415	7575	6496	71
H(10B)	1448	6562	5586	71
H(12)	354	3503	5343	69
H(13)	880	1007	5649	64
H(15A)	2165	1918	6017	108
H(15B)	1770	2744	5155	108
H(15C)	1740	511	5477	108

Table 6. Torsion angles [deg] for 130703A.

F(3)-C(1)-C(2)-O(2)	65.1(3)
F(1)-C(1)-C(2)-O(2)	-54.9(3)
F(2)-C(1)-C(2)-O(2)	-174.47(19)
F(3)-C(1)-C(2)-O(1)	-179.7(2)
F(1)-C(1)-C(2)-O(1)	60.2(3)
F(2)-C(1)-C(2)-O(1)	-59.3(3)
F(3)-C(1)-C(2)-C(3)	-56.3(3)
F(1)-C(1)-C(2)-C(3)	-176.4(2)
F(2)-C(1)-C(2)-C(3)	64.1(3)
O(2)-C(2)-C(3)-C(4)	159.9(2)
O(1)-C(2)-C(3)-C(4)	38.4(3)
C(1)-C(2)-C(3)-C(4)	-80.4(3)
O(2)-C(2)-C(3)-C(8)	-21.0(3)
O(1)-C(2)-C(3)-C(8)	-142.5(2)
C(1)-C(2)-C(3)-C(8)	98.7(3)
C(8)-C(3)-C(4)-C(5)	0.2(4)
C(2)-C(3)-C(4)-C(5)	179.3(2)
C(3)-C(4)-C(5)-C(6)	0.1(5)
C(4)-C(5)-C(6)-C(7)	0.1(5)
C(5)-C(6)-C(7)-C(8)	-0.7(5)
C(6)-C(7)-C(8)-C(3)	1.0(4)
C(4)-C(3)-C(8)-C(7)	-0.7(4)
C(2)-C(3)-C(8)-C(7)	-179.9(2)
O(2)-C(9)-C(10)-C(11)	-65.5(3)
C(14)-C(9)-C(10)-C(11)	48.6(3)
C(9)-C(10)-C(11)-O(3)	143.8(3)
C(9)-C(10)-C(11)-C(12)	-37.5(3)
O(3)-C(11)-C(12)-C(13)	-167.1(3)
C(10)-C(11)-C(12)-C(13)	14.2(4)
C(11)-C(12)-C(13)-C(14)	-0.3(4)
C(12)-C(13)-C(14)-O(1)	122.3(3)

C(12)-C(13)-C(14)-C(15)	-119.1(3)
C(12)-C(13)-C(14)-C(9)	10.7(3)
O(2)-C(9)-C(14)-O(1)	-33.5(2)
C(10)-C(9)-C(14)-O(1)	-150.7(2)
O(2)-C(9)-C(14)-C(13)	82.1(2)
C(10)-C(9)-C(14)-C(13)	-35.0(3)
O(2)-C(9)-C(14)-C(15)	-149.3(2)
C(10)-C(9)-C(14)-C(15)	93.5(3)
O(2)-C(2)-O(1)-C(14)	-16.0(2)
C(3)-C(2)-O(1)-C(14)	106.9(2)
C(1)-C(2)-O(1)-C(14)	-131.0(2)
C(13)-C(14)-O(1)-C(2)	-88.1(2)
C(15)-C(14)-O(1)-C(2)	150.9(2)
C(9)-C(14)-O(1)-C(2)	30.6(2)
O(1)-C(2)-O(2)-C(9)	-6.7(2)
C(3)-C(2)-O(2)-C(9)	-131.35(19)
C(1)-C(2)-O(2)-C(9)	106.7(2)
C(10)-C(9)-O(2)-C(2)	147.5(2)
C(14)-C(9)-O(2)-C(2)	25.3(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for 130703A [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)