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Supporting Information for

Electrooxidative Dearomatization of Biaryls: Synthesis of Tri- and Difluoromethylated Spiro[5.5]trienones

Yan Zhang,[a] Chanchan Ma,[a] Julia Struwe,[b] Jian Feng,[a] Wangyu Zeng,[a] Gangguo Zhu[a] and Lutz Ackermann*[b]*

[a] Key Laboratory of the Ministry of Education for Advanced Catalysis Materials, Zhejiang Normal University, 688 Yingbin Road, 321004 Jinhua P. R. China
*zhangyan001@zjnu.edu.cn
[b] Institut für Organische und Biomolekulare Chemie, Georg-August-Universität Göttingen
Tammannstraße 2, 37077 Göttingen, Germany
*Lutz.Ackermann@chemie.uni-goettingen.de

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1. General Remarks

Catalytic reactions were carried out in undivided electrochemical cells using pre-dried glassware under air, if not noted otherwise. Hydrogen gas is generated during the reaction and can lead to an explosive gaseous mixture with atmospheric oxygen. Therefore, the reactions should be performed in a well-ventilated fume-hood and specialized reaction equipment should be used. NMR spectra were recorded on BRUKER AVANCE III 400 or BRUKER AVANCE III 600. $CDCl_3$ and DMSO-d_6 were used as the solvent. Chemical shifts were referenced relative to residual solvent signal ($CDCl_3$: ^{1}H NMR: δ 7.26 ppm, ^{13}C NMR: δ 77.16 ppm; DMSO-d_6, ^{1}H NMR: δ 2.50 ppm, ^{13}C NMR: δ 39.52 ppm). The following abbreviations are used to describe peak patterns where appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants (*J*) are reported in Hertz (Hz). All IR spectra were recorded on a Bruker FT-IR Alpha-P device. Electrospray-ionization (ESI) mass spectra were obtained on AB Sciex LC 30A-Triple TOF 4600 apparatus. All systems are equipped with time-of-flight (TOF) analyzers. Melting points were measured with micro melting point apparatus. Electrocatalysis was conducted using an AXIOMET AX-3003P potentiostat in constant current mode. Platinum electrodes (10 mm × 15 mm × 0.25 mm, 99.9%; obtained from ChemPur® Karlsruhe, Germany) and graphite felt (GF) electrodes (10 mm × 15 mm × 6 mm, SIGRACELL® GFA 6 EA, obtained from SGL Carbon, Wiesbaden, Germany) were connected using stainless steel adapters. Unless otherwise noted, some materials obtained from commercial suppliers were used directly without further purification. Biarylynone **1** were prepared according to the literature.[1, 2]

Table S-1. Scopes of Biarylynone **1**. (PMP = *para*-methoxyphenyl)

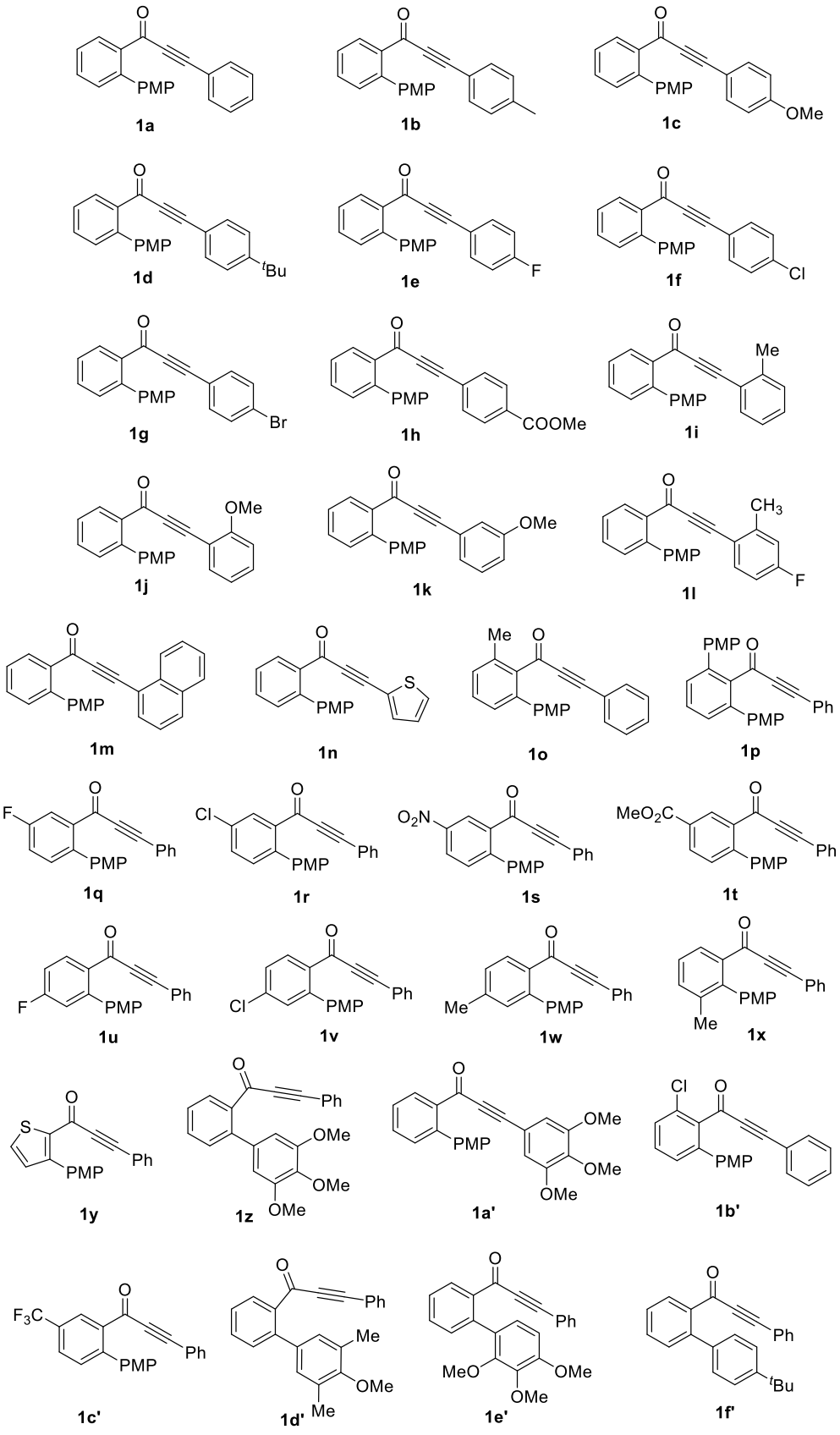
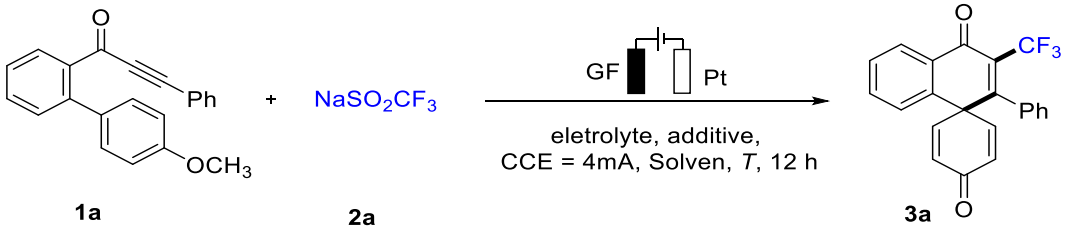


Table S-2. Electrooxidative dearomatization of biaryls.[a]

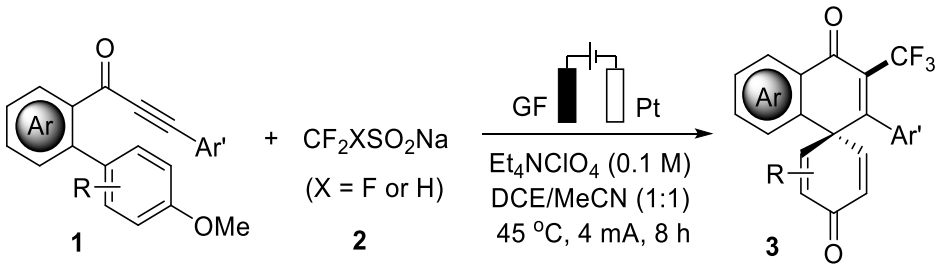


1a **2a** **3a**

Entry	Solvent	Additive	Eletrolyte	*T* / °C	Yield / %
1	MeCN	Na_2CO_3 (2.0 eq)	–	50	40
2	MeCN/THF (1:1)	Na_2CO_3 (2.0 eq)	–	50	10
3	MeCN/H_2O (2:1)	Na_2CO_3 (2.0 eq)	–	50	15
4	DCE/MeCN/MeOH (2:2:1)	Na_2CO_3 (2.0 eq)	–	50	25
5	DCE/MeCN/*i*-PrOH (2:2:1)	Na_2CO_3 (2.0 eq)	–	50	16
6	DCE/EA/H_2O (2:2:1)	Na_2CO_3 (2.0 eq)	–	50	20
7	DCE/MeCN/DMF (2:2:1)	Na_2CO_3 (2.0 eq)	–	50	28
8	DCE/DMF/H_2O (2:2:1)	Na_2CO_3 (2.0 eq)	–	50	34
9	DCE/MeCN/H_2O (2:2:1)	Na_2CO_3 (2.0 eq)	–	50	40
10	DCE/MeCN (1:1)	Na_2CO_3 (2.0 eq)	–	50	46
11	DCE/MeCN (1:1)	–	–	50	46
12	DCE/MeCN (1:1)	–	*n*-Bu_4NBF_4	50	20
13	DCE/MeCN (1:1)	–	LiOBu	50	30
14	DCE/MeCN (1:1)	–	*n*-Bu_4NI	50	0
15	DCE/MeCN (1:1)	–	Et_4NClO_4	50	60
16	DCE/MeCN (1:1)	–	Et_4NPF_6	50	18
17	DCE/MeCN (1:1)	–	*n*-	50	40
18[b]	DCE/MeCN (1:1)	Cp_2Fe (10% mol)	Et_4NClO_4	50	45
19[c]	DCE/MeCN (1:1)	[Mes-Acr]ClO_4 (10% mol)	Et_4NClO_4	50	49
20[d]	DCE/MeCN (1:1)	BQ (10% mol)	Et_4NClO_4	50	55
21[e]	DCE/MeCN (1:1)	–	Et_4NClO_4	50	37
22[f]	DCE/MeCN (1:1)	–	Et_4NClO_4	50	30
23	DCE/MeCN (1:1)	–	Et_4NClO_4	30	38
24	DCE/MeCN (1:1)	–	Et_4NClO_4	60	36
25[g]	DCE/MeCN (1:1)	–	Et_4NClO_4	50	58
27[h]	DCE/MeCN (1:1)	–	Et_4NClO_4	50	60
27[i]	DCE/MeCN (1:1)	–	Et_4NClO_4	50	0

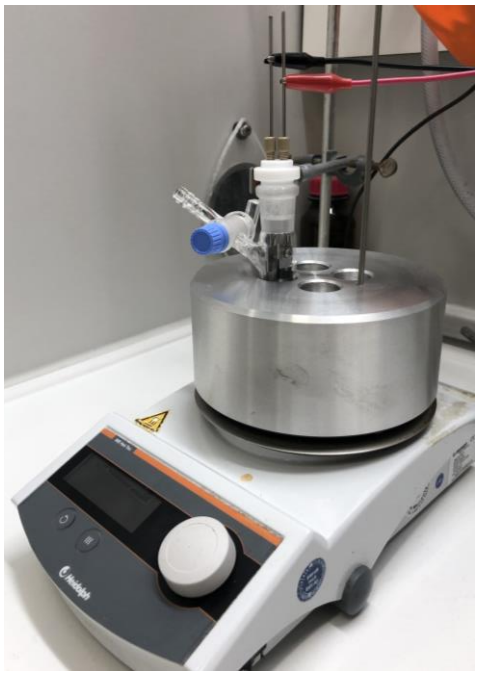
[a] Undivided cell, GF anode, Pt cathode, constant current = 4 mA, **1** (0.30 mmol), **2a** (0.90 mmol, 3.0 equiv), additive (0.6 mmol, 2.0 equiv), eletrolyte (0.1 M), solvent (4 mL), under air, 8 h. Yield of isolated products. [b] Cp_2Fe = ferrocene. [c] [Mes-Acr]ClO_4, blue LEDs, 30 W. [d] BQ = benzoquinone. [e] Constant potential electrolysis (CPE) at 2.5 V. [f] Constant potential electrolysis (CPE) at 2.0 V. [g] Pt(+)|Ni(−) instead of GF(+)|Pt(−). [h] Experiment under N_2. [i] No electricity.

2. General Procedure for Electrooxidative dearomatization of biaryls.

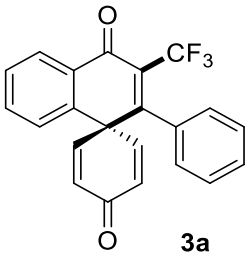


The electrocatalysis was carried out in an undivided cell under air with a graphite felt (GF) anode (10 mm × 15 mm × 6 mm) and a platinum cathode (10 mm × 15 mm × 0.25 mm). Biarylynone **1** (0.3 mmol, 1.0 equiv), [CF_3SO_2Na (**2a**, 140 mg, 0.9 mmol, 3.0 equiv) or CF_2HSO_2Na (**2b**, 110 mg, 0.9 mmol, 3.0 equiv)] and Et_4NClO_4 (92 mg, 0.1 M) were dissolved in a mixture of DCE/MeCN (1:1, 4 mL). Electrocatalysis was performed at 45 °C with a constant current of 4.0 mA maintained for 8 h. The GF anode was washed with ethyl acetate (3 × 5 mL) in an ultrasonic bath and transfered to the round bottom flask with the crude reaction solution. Silica was added to the flask and all volatiles were evaporated under vacuum. Purification was performed by flash column chromatography on silica gel using *n*-hexane/EtOAc as the eluent to give the corresponding products **3**.


lsr tools



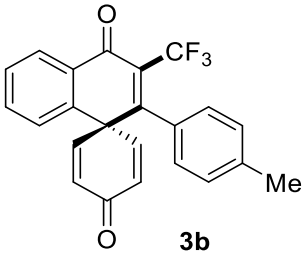
3. Characterization Data of Products 3 and 4



3a

2'-Phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3a)

The general procedure was followed using **1a** (93.6 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3a** (65.9 mg, 60%) as a white solid. **M.p.**: 185–187 °C. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.22 (dd, J = 7.8, 1.6 Hz, 1H), 7.57 (ddd, J = 7.5, 7.5, 1.6 Hz, 1H), 7.50 (ddd, J = 7.6, 7.6, 1.3 Hz, 1H), 7.34–7.14 (m, 4H), 7.04–6.93 (m, 2H), 6.69 (d, J = 10.0 Hz, 1H), 6.27 (d, J = 10.0 Hz, 1H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 184.1, 179.4, 159.0 (q, J = 2.3 Hz), 147.0, 137.1, 134.2, 134.0, 130.7, 129.9, 129.2, 129.0, 128.9 (q, J = 26.9 Hz), 128.0, 127.9, 127.3, 126.7 (q, J = 1.9 Hz), 121.8 (q, J = 278.0 Hz), 51.3. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.46. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}F_3O_2$ $[M+H]^+$: 367.0940, found: 367.0942.

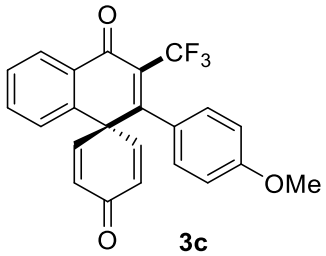


3b

2'-(*p*-Tolyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3b)

The general procedure was followed using **1b** (97.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3b** (70.8 mg, 62%) as a white solid. **M.p.**: 109–111 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.31 (dd, J = 7.8, 1.6 Hz, 1H), 7.62 (ddd, J = 7.6, 7.6, 1.3 Hz, 1H), 7.57 (ddd, J = 7.6, 7.6, 1.3 Hz, 1H), 7.27–7.26 (m, 1H), 7.10 (d, J = 7.8 Hz, 2H), 6.94 (d, J = 8.1 Hz, 2H), 6.72 (d, J = 10.1 Hz, 2H), 6.35 (d, J = 10.1 Hz, 2H), 2.35 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.4, 179.5, 159.4 (q, J = 2.0 Hz), 147.3, 139.2, 137.1,

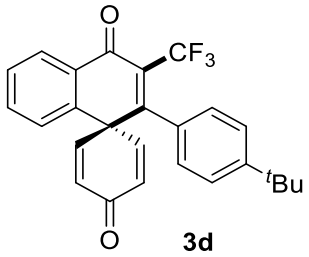
134.0, 131.5, 130.8, 130.1, 129.3, 129.1 (q, J = 26.9 Hz), 128.2, 128.1, 128.0, 126.7 (q, J = 1.4 Hz), 121.9 (q, J = 277.8 Hz), 51.5, 21.3. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.48. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_2$ $[M+H]^+$: 381.1097, found: 381.1098.



3c

2'-(4-Methoxyphenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3c)

The general procedure was followed using **1c** (102.6 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3c** (64.2 mg, 54%) as a white solid. **M.p.**: 125–127 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.27 (dd, J = 7.8, 1.1 Hz, 1H), 7.60 (ddd, J = 7.7, 7.7, 1.3 Hz, 1H), 7.55 (ddd, J = 7.7, 7.7, 1.3 Hz, 1H), 7.26–7.25 (m, 1H), 6.98 (d, J = 8.7 Hz, 2H), 6.80 (d, J = 8.7 Hz, 2H), 6.71 (d, J = 10.0 Hz, 2H), 6.34 (d, J = 10.0 Hz, 2H), 3.79 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.3, 179.5, 160.1, 159.2 (q, J = 2.8 Hz), 147.4, 137.1, 134.0, 130.7, 130.0, 129.3 (q, J = 26.7 Hz), 129.2, 128.9, 128.2, 128.0 (q, J = 2.2 Hz), 126.6, 121.9 (q, J = 277.8 Hz), 113.0, 55.2, 51.6. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.46. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_3$ $[M+H]^+$: 397.1046, found: 397.1050.

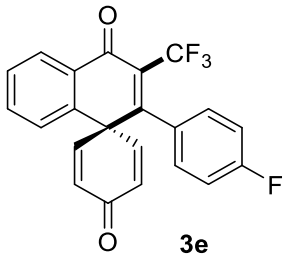


3d

2'-(4-(*tert*-Butyl)phenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3d)

The general procedure was followed using **1d** (110.4 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3d** (71 mg, 56%) as a white solid. **M.p.**: 158–160 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.27 (s, 1H), 7.58 (ddd, J = 7.6, 7.6, 1.6 Hz, 1H), 7.52 (ddd, J = 7.6, 7.6, 1.5

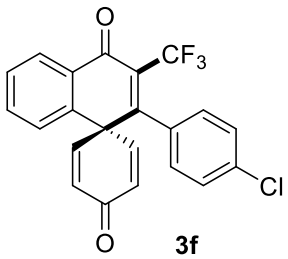
Hz, 1H), 7.27 (d, *J* = 8.3 Hz, 2H), 7.25–7.22 (m, 1H), 6.95 (d, *J* = 8.3 Hz, 2H), 6.70 (d, *J* = 10.0 Hz, 2H), 6.31 (d, *J* = 10.0 Hz, 2H), 1.26 (s, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) *δ* 184.4, 179.6, 159.6, 152.4, 147.4, 137.2, 134.0, 131.5, 130.7, 130.1, 129.3, 129.1 (q, *J* = 26.6 Hz), 128.0 (q, *J* = 6.8 Hz), 126.5, 124.3, 121.9 (q, *J* = 278.0 Hz), 51.5, 34.7, 31.2, 26.9. **^{19}F NMR** (565 MHz, $CDCl_3$) *δ* -56.50. **HR-MS** (ESI) *m/z* calc. for $C_{26}H_{22}F_3O_2$ $[M+H]^+$: 423.1566, found: 423.1566.



3e

2'-(4-Fluorophenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3e)

The general procedure was followed using **1e** (99.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3e** (57.6 mg, 50%) as a white solid. **M.p.**: 163–165 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) *δ* 8.26 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.62 (ddd, *J* = 7.8, 7.8, 1.4 Hz, 1H), 7.56 (dd, *J* = 7.6, 7.6 Hz, 1H), 7.30–7.22 (m, 1H), 7.06–7.02 (m, 2H), 7.00 (dd, *J* = 11.7, 5.4 Hz, 2H), 6.71 (dd, *J* = 6.4, 6.4 Hz, 2H), 6.40–6.27 (m, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) *δ* 184.0, 179.3, 162.9 (d, *J* = 249.9 Hz), 158.0 (q, *J* = 2.0 Hz), 147.0, 137.0, 134.2, 131.0, 130.2 (q, *J* = 3.7 Hz), 129.9, 129.6 (q, *J* = 27.0 Hz), 129.4, 128.9 (q, *J* = 1.6 Hz), 128.9 (q, *J* = 1.5 Hz), 128.1, 121.8 (q, *J* = 278.1 Hz), 114.9 (q, *J* = 22.0 Hz), 51.4. **^{19}F NMR** (565 MHz, $CDCl_3$) *δ* -56.43, -111.24. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}F_4O_2$ $[M+H]^+$: 385.0846, found: 385.0846.

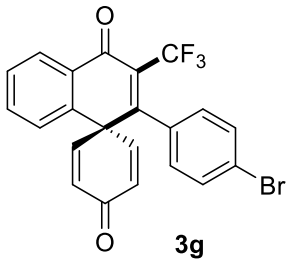


3f

2'-(4-Chlorophenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3f)

The general procedure was followed using **1f** (103.8 mg, 0.30 mmol) and **2a** (140 mg,

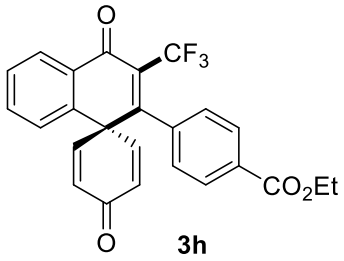
0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3f** (62.4 mg, 52%) as a white solid. **M.p.**: 178–180 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.26 (d, J = 7.8 Hz, 1H), 7.62 (dd, J = 11.0, 4.2 Hz, 1H), 7.56 (dd, J = 7.5, 7.5 Hz, 1H), 7.27 (dd, J = 17.1, 7.7 Hz, 3H), 7.00 (d, J = 8.4 Hz, 2H), 6.71 (d, J = 10.0 Hz, 2H), 6.35 (d, J = 9.9 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.0, 179.2, 157.7, 146.9, 136.9, 135.4, 134.2, 132.6, 131.1, 130.7 (q, J = 13.4 Hz), 129.9, 129.5 (q, J = 26.9 Hz), 128.3 (q, J = 1.6 Hz), 128.1, 128.0, 127.9, 121.7 (q, J = 278.1 Hz), 51.2. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.39. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}{}^{35}ClF_3O_2$ $[M+H]^+$: 401.0551, found: 401.0549.



3g

2'-(4-Bromophenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3g)

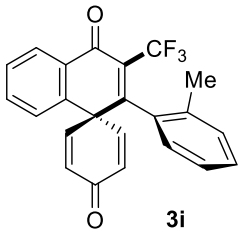
The general procedure was followed using **1g** (117.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3g** (60.1 mg, 45%) as a white solid. **M.p.**: 177–179 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.32–8.24 (m, 1H), 7.68–7.60 (m, 1H), 7.57 (ddd, J = 8.2, 4.2, 1.0 Hz, 1H), 7.48–7.42 (m, 2H), 7.26 (d, J = 6.5 Hz, 1H), 7.11–6.92 (m, 2H), 6.77–6.66 (m, 2H), 6.41–6.30 (m, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 179.1, 157.6 (q, J = 2.1 Hz), 146.8, 136.9, 134.2, 131.1, 130.9, 129.9, 129.5 (q, J = 27.1 Hz), 129.5, 128.5 (q, J = 1.5 Hz), 128.1, 128.0, 127.5, 123.7, 121.7 (q, J = 278.0 Hz), 51.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.38. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}{}^{79}BrF_3O_2$ $[M+H]^+$: 445.0046, found: 445.0041.



3h

Methyl-4-(4,4'-dioxo-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-dien-2'-yl)benzoate (3h)

The general procedure was followed using **1h** (111.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3h** (60.1 mg, 45%) as a white solid. **M.p.**: 160–162 °C. **^{1}H NMR** (600 MHz, DMSO-d_6) δ 8.21 (d, J = 7.8 Hz, 1H), 7.92 (d, J = 8.2 Hz, 2H), 7.77–7.71 (m, 1H), 7.68–7.65 (m, 1H), 7.39 (d, J = 8.2 Hz, 2H), 7.30 (d, J = 7.9 Hz, 1H), 7.24 (d, J = 10.0 Hz, 2H), 6.29 (d, J = 10.0 Hz, 2H), 4.31 (q, J = 7.1 Hz, 1H), 1.32 (t, J = 7.1 Hz, 1H). **^{13}C NMR** (151 MHz, DMSO-d_6) δ 184.4, 179.6, 165.7, 158.5 (q, J = 1.8 Hz), 148.4, 139.7, 137.8, 134.8, 130.6, 130.4, 130.0, 129.6, 128.8 (q, J = 37.7 Hz), 128.2, 128.1, 127.4, 122.5 (q, J = 277.5 Hz), 61.4, 51.1, 14.6. **^{19}F NMR** (565 MHz, DMSO-d_6) δ -55.43. **HR-MS** (ESI) *m/z* calc. for $C_{25}H_{18}F_3O_4$ $[M+H]^+$: 439.1152, found: 439.1156.

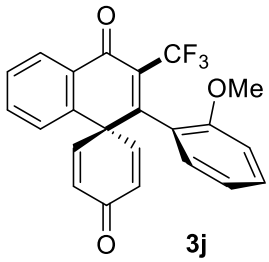


3i

2'-(*o*-Tolyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3i)

The general procedure was followed using **1i** (97.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3i** (68.4 mg, 60%) as a white solid. **M.p.**: 145–147 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.31 (dd, J = 7.8, 1.1 Hz, 1H), 7.61 (tdd, J = 15.0, 10.8, 4.3 Hz, 2H), 7.27 (d, J = 7.9 Hz, 1H), 7.24 (dd, J = 7.5, 7.5 Hz, 1H), 7.17 (d, J = 7.7 Hz, 1H), 7.09 (dd, J = 7.5, 7.5 Hz, 1H), 6.90 (d, J = 7.6 Hz, 1H), 6.81 (dd, J = 10.0, 3.1 Hz, 1H), 6.67 (dd, J = 9.9, 3.1 Hz, 1H), 6.40 (dd, J = 9.9, 1.5 Hz, 1H), 6.24 (dd, J = 10.1, 1.5 Hz, 1H), 2.22 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.1, 179.3, 158.9 (q, J = 2.2 Hz), 147.9, 145.9, 137.8, 134.1, 133.8 (q, J = 1.9 Hz), 133.4, 131.0, 130.7, 130.2, 130.1, 129.8 (q, J = 27.2 Hz), 129.4, 129.2, 128.2, 127.7, 126.5, 124.5, 121.9 (q, J = 277.9 Hz), 52.2, 20.4. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -58.65. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_2$ $[M+H]^+$:

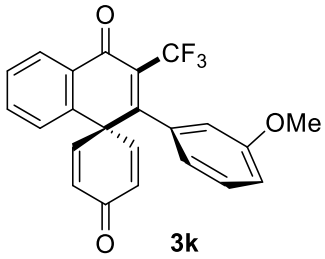
381.1097, found: 381.1098.



3j

2'-(2-Methoxyphenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3j)

The general procedure was followed using **1j** (102.6 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3j** (67.7 mg, 57%) as a white solid. **M.p.**: 170–172 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.30 (d, J = 7.8 Hz, 1H), 7.59 (ddd, J = 7.8, 3.8, 3.8 Hz, 1H), 7.55 (dd, J = 7.5, 7.5 Hz, 1H), 7.31–7.28 (m, 1H), 7.26–7.24 (m, 1H), 6.91 (dd, J = 10.0, 3.1 Hz, 1H), 6.87–6.83 (m, 3H), 6.64 (dd, J = 9.9, 3.0 Hz, 1H), 6.36 (dd, J = 9.9, 1.5 Hz, 1H), 6.21 (dd, J = 10.0, 1.5 Hz, 1H), 3.75 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.4, 179.5, 156.1, 155.2, 147.5 (q, J = 17.9 Hz), 137.7, 133.9, 130.7 (q, J = 4.7 Hz), 130.2, 130.1 (q, J = 26.9 Hz), 129.8, 129.2, 128.0, 128.0, 127.9, 123.0, 121.9 (q, J = 277.7 Hz), 120.0, 119.3, 111.3, 110.8, 55.4, 51.9. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -59.05. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_3$ $[M+H]^+$: 397.1046, found: 397.1048.

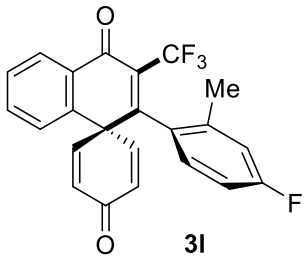


3k

2'-(3-Methoxyphenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3k)

The general procedure was followed using **1k** (102.6 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3k** (71.3 mg, 60%) as a white solid. **M.p.**: 88–90 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.29 (dd, J = 7.9, 1.3 Hz, 1H), 7.62 (ddd, J = 7.7, 7.7, 1.5 Hz, 1H), 7.56 (ddd, J = 7.7, 7.7, 1.1 Hz, 1H), 7.26 (dd, J = 6.2, 1.6 Hz, 1H), 7.22 (dd, J = 8.0, 8.0 Hz, 1H), 6.90–6.85 (m, 1H), 6.75–6.69 (m, 2H), 6.63 (d, J = 7.6 Hz, 1H), 6.60–6.56 (m, 1H),

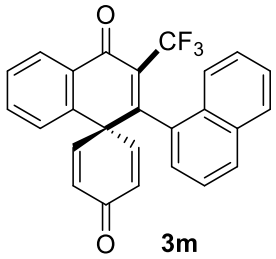
6.35 (ddd, *J* = 23.1, 10.0, 1.6 Hz, 2H), 3.77 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.3, 179.4, 158.8 (q, *J* = 2.1 Hz), 158.5, 147.2 (q, *J* = 4.7 Hz), 137.1, 135.6, 134.1, 130.8, 130.6, 130.0, 129.3, 129.0 (q, *J* = 26.5 Hz), 128.8, 128.1, 128.0, 121.8 (q, *J* = 278.0 Hz), 119.4 (q, *J* = 1.4 Hz), 114.0, 113.8, 113.4 (q, *J* = 1.5 Hz), 55.3, 51.3. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.66. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_3$ $[M+H]^+$: 397.1046, found: 397.1048.



3l

2'-(4-Fluoro-2-methylphenyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3l)

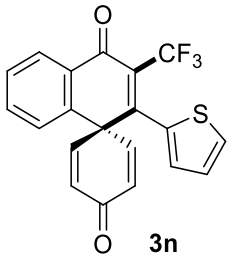
The general procedure was followed using **1l** (99.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3l** (56.1 mg, 47%) as a white solid. **M.p.**: 88–90 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.63 (ddd, *J* = 7.6, 7.6, 1.3 Hz, 1H), 7.59 (dd, *J* = 11.1, 4.0 Hz, 1H), 7.29–7.26 (m, 1H), 6.93–6.86 (m, 2H), 6.80 (ddd, *J* = 13.2, 9.1, 2.7 Hz, 2H), 6.67 (dd, *J* = 9.9, 3.1 Hz, 1H), 6.42 (dd, *J* = 9.9, 1.4 Hz, 1H), 6.28 (dd, *J* = 10.1, 1.4 Hz, 1H), 2.22 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 179.1, 162.6 (d, *J* = 249.1 Hz), 157.8, 147.7, 145.7, 137.6, 136.9 (dq, *J* = 8.0, 1.9 Hz), 134.2, 131.2, 130.8, 130.4 (q, *J* = 26.9 Hz), 129.9, 129.5, 129.3 (d, *J* = 3.4 Hz), 128.3 (d, *J* = 8.6 Hz), 128.2, 127.8, 121.8 (q, *J* = 278.0 Hz), 117.1 (d, *J* = 21.6 Hz), 111.9 (d, *J* = 21.7 Hz), 52.2, 20.5. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -58.59, -112.31. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{15}F_4O_2$ $[M+H]^+$: 399.1003, found: 399.1002.



3m

2'-(Naphthalen-1-yl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3m)

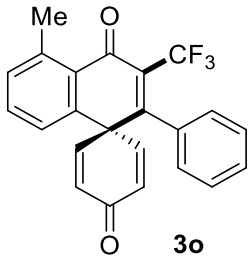
The general procedure was followed using **1m** (108.6 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3m** (31.2 mg, 22%) with a purity of > 90% as a yellow oil. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.37 (dd, J = 7.6, 1.6 Hz, 1H), 7.84 (dd, J = 17.9, 8.3 Hz, 2H), 7.68–7.58 (m, 3H), 7.48 (ddd, J = 15.1, 13.7, 6.4 Hz, 2H), 7.38–7.33 (m, 1H), 7.29–7.23 (m, 1H), 7.12 (d, J = 7.0 Hz, 1H), 6.81–6.58 (m, 2H), 6.41 (dd, J = 9.8, 1.5 Hz, 1H), 5.87 (dd, J = 10.1, 1.5 Hz, 1H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.0, 179.3, 158.0, 148.1, 146.5, 137.9, 134.2, 132.9, 131.3, 130.6, 130.3, 130.2, 129.7, 129.5, 129.0, 128.3, 127.8, 126.9, 126.2, 125.5 (q, J = 6.9 Hz), 124.9 (q, J = 35.0 Hz), 124.3, 124.1, 123.9, 121.8 (q, J = 278.1 Hz), 52.3. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -58.29. **HR-MS** (ESI) *m/z* calc. for $C_{26}H_{16}F_3O_2$ $[M+H]^+$: 417.1097, found: 417.1092.



3n

2'-(Thiophen-2-yl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3n)

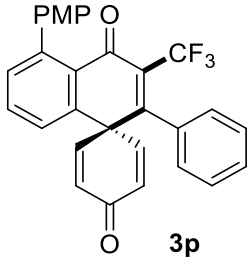
The general procedure was followed using **1n** (95.4 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3n** (33.5 mg, 30%) as a yellow oil. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.29 (dd, J = 7.9, 1.3 Hz, 1H), 7.62 (ddd, J = 7.6, 7.6, 1.4 Hz, 1H), 7.56 (dd, J = 11.1, 4.0 Hz, 1H), 7.41 (dd, J = 5.0, 0.9 Hz, 1H), 7.27 (s, 1H), 7.01 (dd, J = 4.9, 3.7 Hz, 1H), 6.96 (dd, J = 3.5, 0.9 Hz, 1H), 6.69 (d, J = 10.0 Hz, 2H), 6.44 (d, J = 9.7 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.4, 179.2, 152.9 (q, J = 1.9 Hz), 146.8, 136.8, 134.2, 133.8, 131.3, 130.6 (q, J = 26.8 Hz), 129.9, 129.4, 129.2 (q, J = 2.0 Hz), 128.4, 128.2, 128.1, 126.8, 121.9 (q, J = 277.8 Hz), 51.3. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.90. **HR-MS** (ESI) *m/z* calc. for $C_{20}H_{12}F_3O_2S$ $[M+H]^+$: 373.0505, found: 373.0505.



3o

5'-Methyl-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3o)

The general procedure was followed using **1o** (97.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3o** (58.1 mg, 51%) as a white solid. **M.p.**: 130–132 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.45 (dd, J = 7.7, 7.7 Hz, 1H), 7.34 (dd, J = 7.4, 7.4 Hz, 2H), 7.31–7.26 (m, 2H), 7.15 (d, J = 7.8 Hz, 1H), 7.07–7.00 (m, 2H), 6.73 (dd, J = 6.4, 6.4 Hz, 2H), 6.29 (d, J = 10.0 Hz, 2H), 2.79 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.3, 181.7, 156.0 (q, J = 2.2 Hz), 147.8, 142.3, 138.6, 133.9, 133.0, 132.9, 130.5 (q, J = 24.0 Hz), 130.8, 129.0, 128.8, 127.3, 127.0, 125.9, 121.9 (q, J = 277.7 Hz), 51.7, 23.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.45. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_2$ $[M+H]^+$: 381.1097, found: 381.1095.

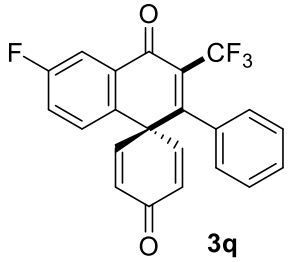


3p

2'-Phenyl-5'-(*p*-tolyl)-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3p)

The general procedure was followed using **1p** (125.4 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3p** (51 mg, 36%) as a white solid. **M.p.**: 73–75 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.55 (dd, J = 7.8, 7.8 Hz, 1H), 7.40 (d, J = 6.9 Hz, 1H), 7.34 (d, J = 7.4 Hz, 1H), 7.31 (d, J = 7.8 Hz, 1H), 7.26 (dd, J = 3.6, 3.6 Hz, 2H), 7.23 (d, J = 8.6 Hz, 2H), 7.05 (d, J = 7.3 Hz, 2H), 6.96 (d, J = 8.6 Hz, 2H), 6.76 (d, J = 10.0 Hz, 2H), 6.32 (d, J = 10.0 Hz, 2H), 3.86 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.2, 181.2, 159.1, 156.4 (q, J = 2.1 Hz), 148.2, 147.6, 144.1, 138.4, 133.6, 133.0, 132.8, 132.6, 130.7, 129.6,

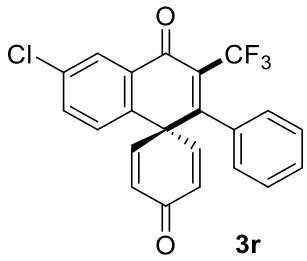
129.6 (q, *J* = 45.8 Hz), 129.1 (q, *J* = 3.8 Hz), 127.5, 127.1, 126.6, 121.6 (q, *J* = 277.6 Hz), 113.8, 55.3, 51.6. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.57; **HR-MS** (ESI) *m/z* calc. for $C_{29}H_{20}F_3O_3$ $[M+H]^+$: 473.1359, found: 473.1356.



3q

6'-Fluoro-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3q)

The general procedure was followed using **1q** (99.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3q** (54.1 mg, 47%) as a white solid. **M.p.**: 115–117 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 8.5, 2.8 Hz, 1H), 7.39–7.24 (m, 5H), 7.07–7.01 (m, 2H), 6.70 (d, *J* = 10.1 Hz, 2H), 6.33 (d, *J* = 10.1 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 178.5 (d, *J* = 2.0 Hz), 162.9 (d, *J* = 251.9 Hz), 159.5, 146.7, 134.1, 133.0 (d, *J* = 3.0 Hz), 132.1 (d, *J* = 7.3 Hz), 131.0, 130.6 (d, *J* = 7.8 Hz), 129.3, 128.9 (q, *J* = 27.1 Hz), 127.5, 126.8 (q, *J* = 1.5 Hz), 121.9 (d, *J* = 22.9 Hz), 121.7 (q, *J* = 278.1 Hz), 114.0 (d, *J* = 23.1 Hz), 51.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.60, -109.82. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}F_4O_2$ $[M+H]^+$: 385.0846, found: 385.0844.

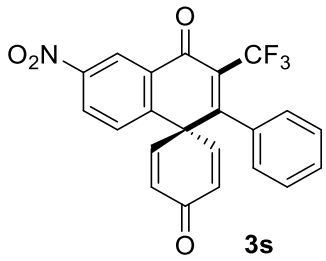


3r

6'-Chloro-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3r)

The general procedure was followed using **1r** (103.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3r** (60 mg, 50%) as a white solid. **M.p.**: 100–102 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.24 (d, *J* = 2.3 Hz, 1H), 7.57 (ddd, *J* = 8.5, 7.6, 2.3 Hz, 1H), 7.38–7.33 (m, 1H), 7.30 (dd, *J* = 7.6, 7.6 Hz, 2H), 7.21 (dd, *J* = 8.5, 4.2 Hz, 1H), 7.10–6.91 (m, 2H),

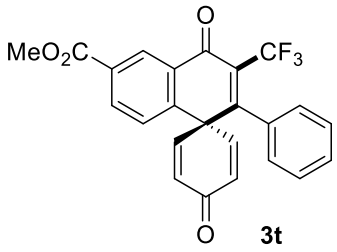
6.81–6.57 (m, 2H), 6.44–6.19 (m, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 178.4, 159.4, 146.5, 136.1, 135.5, 134.2, 134.0, 131.3, 131.1, 129.8, 129.4, 129.2 (q, J = 31.7 Hz), 127.7, 127.5, 126.7 (q, J = 1.7 Hz), 121.7 (q, J = 278.1 Hz), 51.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.59. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}{}^{35}ClF_3O_2$ $[M+H]^+$: 401.0551, found: 401.0547.



3s

6'-Nitro-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3s)

The general procedure was followed using **1s** (107.1 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3s** (40.7 mg, 33%) as a white solid. **M.p.**: 160–162 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 9.04 (d, J = 2.4 Hz, 1H), 8.42 (dd, J = 8.7, 2.5 Hz, 1H), 7.52 (d, J = 8.7 Hz, 1H), 7.41 (dd, J = 7.5, 7.5 Hz, 1H), 7.35 (dd, J = 7.6, 7.6 Hz, 2H), 7.10–7.06 (m, 2H), 6.77 (d, J = 10.0 Hz, 2H), 6.42 (d, J = 10.0 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.4, 177.7, 159.8 (q, J = 2.0 Hz), 148.4, 145.4, 143.3, 133.6, 131.8, 131.2, 130.2, 129.6, 129.0 (q, J = 27.6 Hz), 127.8, 127.6, 126.7 (q, J = 1.0 Hz), 123.2, 121.5 (q, J = 278.2 Hz), 51.3. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.65. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}F_3NO_4$ $[M+H]^+$: 482.0791, found: 482.0804.

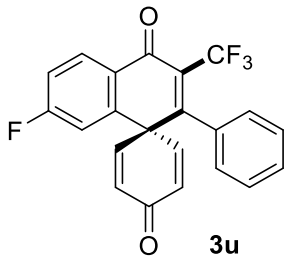


3t

Methyl-4,4'-dioxo-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-6'-carboxylate (3t)

The general procedure was followed using **1t** (111.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc =

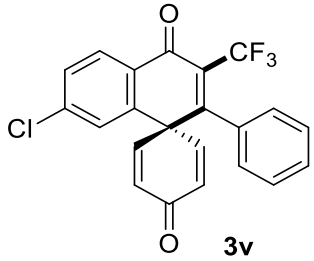
3:1) yielded **3t** (80.1 mg, 63%) as a white solid. **M.p.**: 215–217 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.89 (d, J = 1.8 Hz, 1H), 8.23 (dd, J = 8.3, 1.9 Hz, 1H), 7.36 (dd, J = 7.9, 4.1 Hz, 2H), 7.31 (dd, J = 7.6, 7.6 Hz, 2H), 7.08–7.03 (m, 2H), 6.76–6.72 (m, 2H), 6.40 – 6.35 (m, 2H), 3.98 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.8, 178.7, 165.4, 159.2 (q, J = 2.0 Hz), 146.3, 141.4, 134.3, 134.0, 131.4, 131.3, 130.2, 129.4, 129.1 (q, J = 27.4 Hz), 128.6, 127.5, 126.8, 121.7 (q, J = 278.2 Hz), 52.7, 51.4. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.55. **HR-MS** (ESI) *m/z* calc. for $C_{24}H_{16}F_3O_4$ $[M+H]^+$: 425.0995, found: 425.0992.



3u

7'-Fluoro-2'-phenyl-3'-(trifluoromethyl)-4′*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3u)

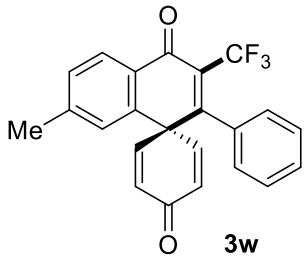
The general procedure was followed using **1u** (99.0 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3u** (58.8 mg, 51%) as a white solid. **M.p.**: 153–155 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.34 (dd, J = 8.8, 5.8 Hz, 1H), 7.36 (dd, J = 7.5, 7.5 Hz, 1H), 7.30 (dd, J = 7.5, 7.5 Hz, 2H), 7.27–7.24 (m, 1H), 7.05–7.02 (m, 2H), 6.93 (dd, J = 9.0, 2.4 Hz, 1H), 6.74–6.69 (m, 2H), 6.40–6.32 (m, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.8, 178.3, 166.0 (d, J = 258.1 Hz), 158.9, 146.4, 140.3 (d, J = 8.6 Hz), 134.0, 131.4 (d, J = 9.7 Hz), 131.2, 129.3, 129.0 (q, J = 27.0 Hz), 127.5, 126.8 (q, J = 1.5 Hz), 126.6, 121.7 (q, J = 278.0 Hz), 117.5 (d, J = 22.2 Hz), 114.7 (d, J = 23.5 Hz), 51.2. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.51, -101.60. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}F_4O_2$ $[M+H]^+$: 385.0846, found: 385.0841.



3v

7'-Chloro-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3v)

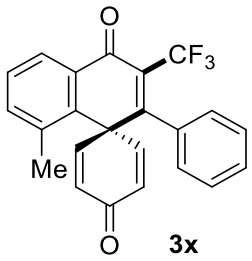
The general procedure was followed using **1v** (103.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3v** (69.6 mg, 58%) as a white solid. **M.p.**: 158–160 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.21 (d, J = 8.5 Hz, 1H), 7.52 (dd, J = 8.5, 2.0 Hz, 1H), 7.36 (dd, J = 8.4, 6.5 Hz, 1H), 7.30 (dd, J = 16.6, 9.4 Hz, 2H), 7.23 (d, J = 1.9 Hz, 1H), 7.07–7.02 (m, 2H), 6.73 (d, J = 10.1 Hz, 2H), 6.36 (d, J = 10.1 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.8, 178.5, 159.0 (q, J = 2.0 Hz), 146.3, 140.9, 139.0, 134.0, 131.3, 130.0, 129.7, 129.3, 129.0 (q, J = 27.1 Hz), 128.4, 128.0, 127.5, 126.8 (q, J = 1.4 Hz), 121.7 (q, J = 278.0 Hz), 51.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.51. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{13}{}^{35}ClF_3O_2$ $[M+H]^+$: 401.0551, found: 401.0540.



3w

7'-Methyl-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3w)

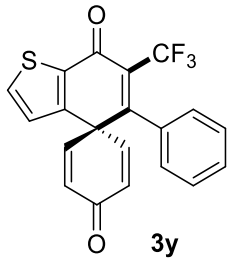
The general procedure was followed using **1w** (97.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3w** (45.6 mg, 40%) as a white solid. **M.p.**: 158–160 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.18 (d, J = 8.1 Hz, 1H), 7.35 (ddd, J = 10.8, 6.2, 3.2 Hz, 2H), 7.31–7.25 (m, 2H), 7.03 (dd, J = 9.8, 8.5 Hz, 3H), 6.80–6.59 (m, 2H), 6.39–6.27 (m, 2H), 2.40 (d, J = 6.1 Hz, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.3, 179.2, 158.6, 147.3, 145.3, 137.1, 134.4, 131.1 130.8, 130.5, 129.1, 129.0 (q, J = 26.8 Hz), 128.1, 127.7, 127.4, 126.9 (q, J = 1.5 Hz), 121.9 (q, J = 278.3 Hz), 51.3, 21.9. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.43. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_2$ $[M+H]^+$: 381.1097, found: 381.1089.



3x

8'-Methyl-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3x)

The general procedure was followed using **1x** (97.8 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3x** (51.3 mg, 45%) as a white solid. **M.p.**: 143–145 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.30 (dd, J = 7.8, 1.1 Hz, 1H), 7.53 (dd, J = 7.7, 7.7 Hz, 1H), 7.47–7.45 (m, 1H), 7.33 (ddd, J = 6.9, 4.0, 1.1 Hz, 1H), 7.26 (ddd, J = 7.4, 3.6, 3.6 Hz, 3H), 6.96 (dd, J = 5.1, 3.4 Hz, 2H), 6.68–6.63 (m, 2H), 6.45–6.40 (m, 2H), 2.27 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.7, 180.0, 158.3 (q, J = 1.9 Hz), 144.9, 138.3, 137.8, 135.4, 133.6, 132.7, 130.8, 129.5 (q, J = 27.0 Hz), 129.5, 128.9, 128.9 (q, J = 1.6 Hz), 126.9, 126.7, 121.7 (q, J = 278.2 Hz), 51.6, 21.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.67. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O_2$ $[M+H]^+$: 381.1097, found: 381.1092.

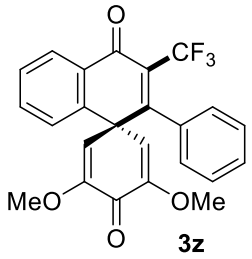


3y

5-Phenyl-6-(trifluoromethyl)-7*H*-spiro[benzo[b]thiophene-4,1'-cyclohexane]-2',5'-diene-4',7-dione (3y)

The general procedure was followed using **1y** (95.4 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3y** (31.2 mg, 28%) as a white solid. **M.p.**: 170–172 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.76 (d, J = 5.1 Hz, 1H), 7.36 (dd, J = 7.5, 7.5 Hz, 1H), 7.30 (t, J = 7.6, 7.6 Hz, 2H), 7.05 (d, J = 7.4 Hz, 2H), 6.86 (d, J = 5.1 Hz, 1H), 6.68 (d, J = 10.0 Hz, 2H), 6.35 (d, J = 10.0 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 174.2, 159.3 (q, J = 2.2 Hz), 145.3, 144.5, 136.6, 135.4, 134.1, 131.2, 129.3, 129.0 (q, J = 26.8 Hz), 127.6, 127.3, 126.7 (q, J = 1.6 Hz), 121.8 (q, J = 278.2 Hz), 51.3. **^{19}F NMR** (565 MHz,

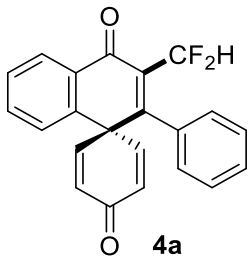
$CDCl_3$) δ -55.99. **HR-MS** (ESI) *m/z* calc. for $C_{20}H_{12}F_3O_2S$ $[M+H]^+$: 373.0505, found: 373.0498.



3z

3,5-Dimethoxy-2'-phenyl-3'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3z)

The general procedure was followed using **1z** (111.6 mg, 0.30 mmol) and **2a** (140 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **3z** (63.9 mg, 50%) as a white solid. **M.p.**: 170–172 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.61–7.57 (m, 1H), 7.54–7.50 (m, 1H), 7.34–7.31 (m, 1H), 7.27 (dd, *J* = 8.0, 7.1 Hz, 3H), 7.00 (dd, *J* = 5.2, 3.4 Hz, 2H), 5.69 (s, 2H), 3.63 (s, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 179.6, 174.9, 161.3 (q, *J* = 2.0 Hz), 152.6, 140.4, 134.1, 134.0, 129.6, 129.0, 128.8, 128.1 (q, *J* = 24.1 Hz), 127.8, 127.8, 127.2, 127.1 (q, *J* = 1.0 Hz), 122.0 (q, *J* = 277.9 Hz), 115.4, 55.6, 48.7. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.34. **HR-MS** (ESI) *m/z* calc. for $C_{24}H_{18}F_3O_4$ $[M+H]^+$: 427.1152, found: 427.1146.

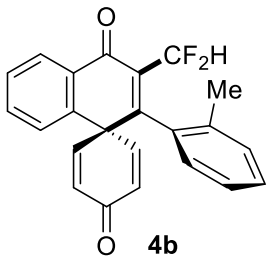


4a

3'-(Difluoromethyl)-2'-phenyl-4′*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4a)

The general procedure was followed using **1a** (93.6 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4a** (57.4 mg, 55%) as a white solid. **M.p.**: 175–177 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.61 (d, *J* = 1.4 Hz, 1H), 7.58–7.54 (m, 1H), 7.41–7.31 (m, 1H), 7.27 (dd, *J* = 3.6, 3.6 Hz, 1H), 7.15–7.05 (m, 1H), 6.76–6.71 (m, 1H), 6.42 (t, *J* = 53.4 Hz, 1H), 6.37–6.33 (m, 1H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ

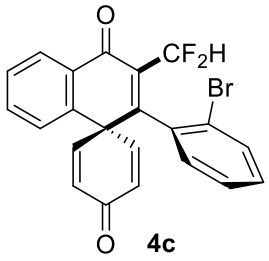
184.4, 180.6, 158.5 (t, *J* = 4.8 Hz), 149.4, 147.5, 137.6, 133.9, 133.2, 131.3 (t, *J* = 20.7 Hz), 130.7, 130.1, 129.4, 129.2, 128.2, 128.0, 127.8, 111.8 (t, *J* = 240.3 Hz), 50.8. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.92. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{15}F_2O_2$ $[M+H]^+$: 349.1035, found: 349.1026.



4b

3'-(Difluoromethyl)-2'-(*o*-tolyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4b)

The general procedure was followed using **1l** (97.8 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4b** (60.8 mg, 56%) as a white solid. **M.p.**: 175–178 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.34 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.60 (m, 2H), 7.28–7.25 (m, 2H), 7.21 (d, *J* = 7.6 Hz, 1H), 7.12 (dd, *J* = 7.5, 7.5 Hz, 1H), 6.94 (d, *J* = 7.6 Hz, 1H), 6.84 (dd, *J* = 10.0, 3.1 Hz, 1H), 6.71 (dd, *J* = 9.9, 3.1 Hz, 1H), 6.40 (dd, *J* = 9.9, 1.5 Hz, 1H), 6.28–6.25 (m, 1H), 6.26 (t, *J* = 53.4 Hz, 1H), 2.23 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.2, 180.3, 158.4 (t, *J* = 5.4 Hz), 148.2, 146.3, 138.1, 134.8, 133.9, 132.1, 131.9 (t, *J* = 20.9 Hz), 130.9, 130.6, 130.5, 130.2, 129.5, 129.3, 128.0, 124.8, 112.1 (t, *J* = 240.5 Hz), 51.6, 20.5. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -117.58 (d, *J* = 11.1 Hz, 2F). **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{17}F_2O_2$ $[M+H]^+$: 363.1191, found: 363.1184.

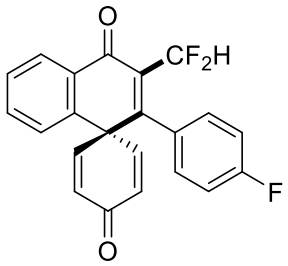


4c

2'-(2-Bromophenyl)-3'-(difluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4c)

The general procedure was followed using **1g** (117.0 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc =

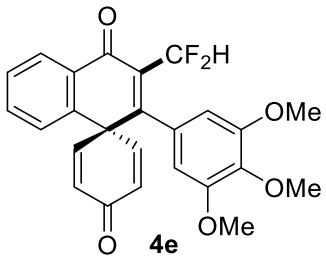
3:1) yielded **4c** (76.7 mg, 60%) as a white solid. **M.p.**: 164–168 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.31 (dd, J = 7.8, 1.5 Hz, 1H), 7.66–7.42 (m, 3H), 7.34–7.19 (m, 4H), 7.06 (dd, J = 7.4, 2.0 Hz, 1H), 6.62 (dd, J = 9.8, 3.1 Hz, 1H), 6.48–6.12 (m, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.3, 180.5, 156.3, 148.0, 146.5, 138.0, 134.1, 133.7, 133.1, 132.4 (t, J = 20.8 Hz), 131.0, 131.0, 130.5, 130.0, 129.6, 129.3, 128.0, 128.0, 126.5, 122.43, 111.7 (t, J = 241.1 Hz), 51.4. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -116.73 (d, J = 320.8 Hz, 1F), -118.00 (d, J = 320.9 Hz, F). **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}{}^{79}BrF_2O_2$ $[M+H]^+$: 427.014, found: 427.0127.



4d

3'-(Difluoromethyl)-2'-(4-fluorophenyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4d)

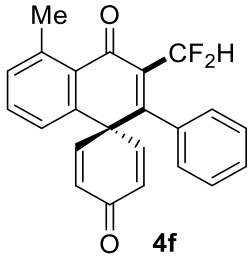
The general procedure was followed using **1e** (99.0 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4d** (67 mg, 61%) as a white solid. **M.p.**: 175–179 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.29 (dd, J = 7.8, 1.0 Hz, 1H), 7.63 (dd, J = 7.8, 1.3 Hz, 1H), 7.55 (dd, J = 11.1, 4.0 Hz, 1H), 7.29–7.26 (m, 1H), 7.14–7.08 (m, 2H), 7.03 (dd, J = 8.6, 8.6 Hz, 2H), 6.74 (d, J = 10.0 Hz, 2H), 6.53 (t, J = 53.5 Hz, 1H), 6.37 (t, J = 6.3 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.2, 180.7, 163.8, 162.2, 157.4 (t, J = 3.8 Hz), 147.4, 137.6, 134.0, 131.9 (t, J = 20.2 Hz), 130.9, 129.9 (d, J = 8.4 Hz), 129.8, 129.3, 129.3, 128.1 (d, J = 49.6 Hz), 115.0 (d, J = 21.9 Hz), 111.4 (t, J = 240.5 Hz), 50.9. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -111.01, -115.48. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}F_3O_2$ $[M+H]^+$: 367.094, found: 367.0933.



4e

3'-(Difluoromethyl)-2'-(3,4,5-trimethoxyphenyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4e)

The general procedure was followed using **1a'** (111.6 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4e** (68.3 mg, 52%) as a white solid. **M.p.**: 163–165 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.28 (dd, J = 7.9, 1.5 Hz, 1H), 7.59 (ddd, J = 7.5, 7.5, 1.5 Hz, 1H), 7.53 (ddd, J = 7.5, 7.5, 1.1 Hz, 1H), 7.28–7.20 (m, 1H), 6.79–6.65 (m, 2H), 6.52–6.24 (m, 5H), 3.85 (s, 3H), 3.77 (s, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.5, 180.5, 158.3 (t, J = 5.3 Hz), 152.8, 148.0, 139.0, 137.5, 133.9, 131.1 (t, J = 20.8 Hz), 130.4, 130.1, 129.3, 128.9, 128.0, 128.0, 112.2 (t, J = 240.3 Hz), 105.4, 61.0, 56.3, 50.8. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.85. **HR-MS** (ESI) *m/z* calc. for $C_{25}H_{21}F_2O_5$ $[M+H]^+$: 439.1352, found: 439.1343.

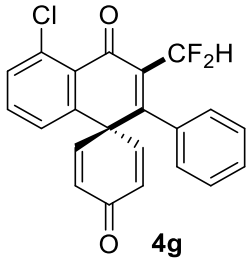


4f

3'-(Difluoromethyl)-5'-methyl-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4f)

The general procedure was followed using **1o** (97.8 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4f** (58.6 mg, 54%) as a white solid. **M.p.**: 178–182 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.44 (dd, J = 7.7, 7.7 Hz, 1H), 7.38–7.31 (m, 4H), 7.15 (d, J = 7.8 Hz, 1H), 7.08 (dd, J = 9.2, 7.8 Hz, 2H), 6.78–6.72 (m, 2H), 6.39 (t, J = 46.5 Hz, 1H), 6.30 (d, J = 10.1 Hz, 2H), 2.82 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.5, 182.8, 156.2 (t, J = 4.8 Hz), 148.3, 142.7, 139.0, 133.1, 132.9, 132.9, 132.4 (t, J = 20.3 Hz), 130.3, 129.3, 128.4, 127.8, 127.8, 126.3, 112.0 (t, J = 240.3 Hz), 51.3, 23.5. **^{19}F NMR** (565 MHz,

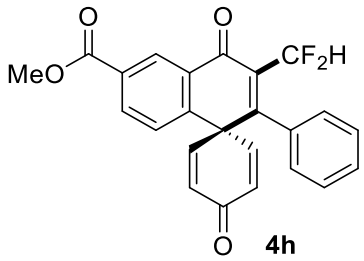
$CDCl_3$) δ -115.74. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{17}F_2O_2$ $[M+H]^+$: 363.1191, found: 363.1194.



4g

3'-(Difluoromethyl)-5'-methyl-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4g)

The general procedure was followed using **1b'** (103.8 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4g** (57.3 mg, 50%) as a white solid. **M.p.**: 152–155 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.55 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.46 (dd, *J* = 7.9, 7.9 Hz, 1H), 7.40–7.35 (m, 1H), 7.32 (dd, *J* = 8.3, 6.7 Hz, 2H), 7.22 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.10–7.03 (m, 2H), 6.74–6.65 (m, 2H), 6.53–6.27 (m, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.1, 179.7, 156.1 (t, *J* = 4.4 Hz), 147.5, 140.7, 135.6, 133.4, 132.9, 132.8, 132.4 (t, *J* = 20.6 Hz), 130.7, 129.5, 127.8, 127.8, 127.1, 127.1, 111.5 (t, *J* = 240.8 Hz), 50.9. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.46. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}{}^{35}ClF_2O_2$ $[M+H]^+$: 383.0645, found: 383.0645.

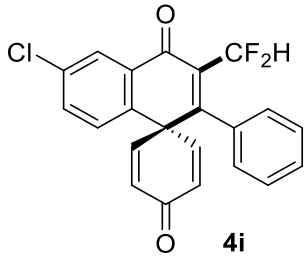


4h

Methyl-3'-(difluoromethyl)-4,4'-dioxo-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-6'-carboxylate (4h)

The general procedure was followed using **1t** (111.0 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4h** (54.8 mg, 45%) as a white solid. **M.p.**: 175–178 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.93 (d, *J* = 1.8 Hz, 1H), 8.23 (dd, *J* = 8.3, 1.9 Hz, 1H), 7.41–7.32 (m, 1H), 7.11 (d, *J* = 7.2 Hz, 1H), 6.42 (t, *J* = 53.3 Hz, 1H), 6.39 (d, *J* = 10.0 Hz, 1H), 3.98 (s, 1H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.0, 179.9, 165.5, 158.6 (t, *J* = 4.4 Hz), 146.7,

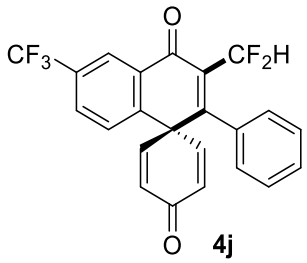
141.8, 134.1, 132.9, 131.3 (t, J = 20.7 Hz), 131.2, 130.2, 129.6, 129.4, 128.7, 127.8, 127.7, 111.6 (t, J = 240.6 Hz), 60.4, 52.7, 50.8. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.94. **HR-MS** (ESI) *m/z* calc. for $C_{24}H_{17}F_2O_4$ $[M+H]^+$: 407.1089, found: 407.1091.



4i

6'-Chloro-3'-(difluoromethyl)-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4i)

The general procedure was followed using **1r** (103.8 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4i** (59.6 mg, 52%) as a white solid. **M.p.**: 161–165 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.25 (d, J = 2.3 Hz, 1H), 7.56 (dd, J = 8.5, 2.4 Hz, 1H), 7.39 (ddd, J = 6.6, 3.8, 1.1 Hz, 1H), 7.34 (dd, J = 10.3, 4.7 Hz, 2H), 7.22 (d, J = 8.5 Hz, 1H), 7.13–7.07 (m, 2H), 6.78–6.68 (m, 2H), 6.37 (t, J = 53.3 Hz, 1H), 6.36 (dt, J = 11.0, 2.4 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 184.0, 179.5, 158.9 (t, J = 4.8 Hz), 146.9, 135.9, 135.9, 134.0, 132.9, 131.4, 131.2 (t, J = 20.9 Hz), 131.0, 130.0, 129.6, 127.8, 127.7, 127.6, 111.7 (t, J = 240.7 Hz), 50.5. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -116.00. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}{}^{35}ClF_2O_2$ $[M+H]^+$: 383.0645, found: 383.0645.

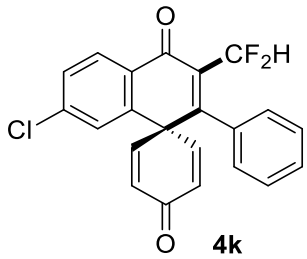


4j

3'-(Difluoromethyl)-2'-phenyl-6'-(trifluoromethyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4j)

The general procedure was followed using **1c'** (114.0 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4j** (54.9 mg, 44%) as a white solid. **M.p.**: 120–122 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.58 (d, J = 1.9 Hz, 1H), 7.83 (dd, J = 8.3, 2.1 Hz, 1H), 7.44–7.37 (m, 2H), 7.35 (dd, J = 8.3, 6.8 Hz, 2H), 7.13–7.06 (m, 2H), 6.76–6.67 (m, 2H), 6.53–6.26 (m,

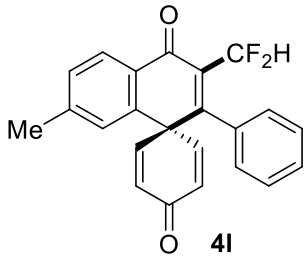
3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.8, 179.5, 158.9 (t, J = 4.6 Hz), 146.4, 141.2, 132.8, 131.4 (t, J = 20.4 Hz), 131.9 (q, J = 33.8 Hz), 131.3, 130.5, 130.0 (q, J = 3.2 Hz), 129.7, 129.3, 127.9, 127.7, 125.3 (q, J = 3.8 Hz), 123.2 (q, J = 272.8 Hz), 111.5 (t, J = 240.7 Hz), 50.7. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -63.06, -116.03. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{14}F_5O_2$ $[M+H]^+$: 417.0909, found: 417.0908.



4k

7'-Chloro-3'-(difluoromethyl)-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4k)

The general procedure was followed using **1v** (103.8 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4k** (57.3 mg, 50%) as a white solid. **M.p.**: 198–202 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.24 (d, J = 8.5 Hz, 1H), 7.52 (dd, J = 8.5, 1.9 Hz, 1H), 7.39 (dd, J = 8.4, 6.5 Hz, 1H), 7.34 (dd, J = 7.5, 7.5 Hz, 1H), 7.22 (d, J = 1.9 Hz, 2H), 7.12–7.07 (m, 2H), 6.73 (d, J = 10.0 Hz, 2H), 6.38 (t, J = 53.3 Hz, 1H), 6.37 (d, J = 10.0 Hz, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 179.7, 158.4 (t, J = 4.8 Hz), 146.7, 140.7, 139.5, 132.9, 131.2 (t, J = 20.7 Hz), 131.2, 129.9, 129.6, 129.6, 128.5, 128.2, 127.8, 127.8, 111.7 (t, J = 240.6 Hz), 50.5. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.97. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}{}^{35}ClF_2O_2$ $[M+H]^+$: 383.0645, found: 383.0649.

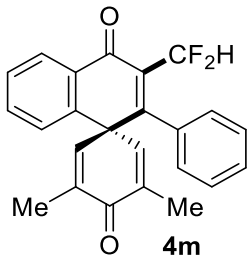


4l

3'-(Difluoromethyl)-7'-methyl-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4l)

The general procedure was followed using **1w** (97.8 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4l** (45.6 mg, 42%) as a white solid. **M.p.**: 197–200 °C. **^{1}H NMR** (600 MHz,

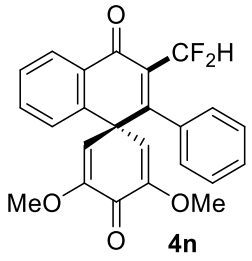
CDCl3) δ 8.92 (d, J = 1.9 Hz, 1H), 8.22 (dd, J = 8.2, 1.9 Hz, 1H), 7.39 (dd, J = 7.5, 7.5 Hz, 1H), 7.35–7.31 (m, 3H), 7.11–7.07 (m, 2H), 6.75–6.70 (m, 2H), 6.42–6.31 (m, 3H), 3.97 (s, 3H). **^{13}C NMR** (151 MHz, CDCl3) δ 184.6, 180.5, 158.1 (t, J = 4.7 Hz), 147.7, 145.1, 137.6, 133.3, 131.3 (t, J = 20.5 Hz), 130.7, 130.4, 129.3, 128.4, 128.0, 127.8, 127.4, 127.7, 111.8 (t, J = 240.2 Hz), 50.8, 21.9. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.94. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{17}F_2O_2$ $[M+H]^+$: 395.1089, found: 395.1100.



4m

3'-(Difluoromethyl)-3,5-dimethyl-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4m)

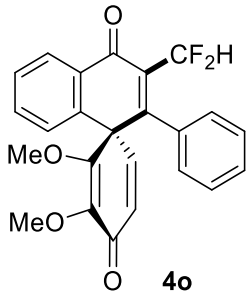
The general procedure was followed using **1d'** (102.0 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4m** (69.9 mg, 62%) as a white solid. **M.p.**: 126–130 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.30 (dd, J = 7.9, 1.1 Hz, 1H), 7.59 (ddd, J = 7.7, 7.7, 1.4 Hz, 1H), 7.55–7.50 (m, 1H), 7.38–7.27 (m, 3H), 7.22 (d, J = 7.8 Hz, 1H), 7.07–7.00 (m, 2H), 6.50 (s, 2H), 6.36 (d, J = 53.5 Hz, 1H), 1.84 (s, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 185.6, 180.9, 160.0 (t, J = 4.6 Hz), 142.3, 139.3, 137.5, 133.6, 133.4, 130.8 (t, J = 20.5 Hz), 130.0, 129.1, 128.8, 128.2, 127.8, 127.7, 127.3, 112.0 (t, J = 240.0 Hz), 50.5, 16.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.76. **HR-MS** (ESI) *m/z* calc. for $C_{24}H_{19}F_2O_2$ $[M+H]^+$: 375.1191, found: 375.1191.



4n

3'-(Difluoromethyl)-3,5-dimethoxy-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4n)

The general procedure was followed using **1z** (111.6 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4n** (73.44 mg, 60%) as a white solid. **M.p.**: 158–162 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.28 (dd, J = 7.9, 1.2 Hz, 1H), 7.58 (ddd, J = 7.7, 7.7, 1.5 Hz, 1H), 7.54–7.50 (m, 1H), 7.37–7.34 (m, 1H), 7.31 (dd, J = 7.4, 7.4 Hz, 2H), 7.27 (s, 1H), 7.09–7.02 (m, 2H), 6.39 (t, J = 53.5 Hz, 1H), 5.71 (d, J = 3.6 Hz, 2H), 3.65–3.62 (m, 6H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.8, 175.1, 160.9 (t, J = 4.6 Hz), 152.3, 140.8, 133.9, 133.1, 130.3 (t, J = 20.4 Hz), 129.6, 129.3, 128.7, 128.0, 127.8, 127.7, 127.6, 115.7, 112.1 (t, J = 240.1 Hz), 55.6, 48.1. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.74. **HR-MS** (ESI) *m/z* calc. for $C_{24}H_{19}F_2O_4$ $[M+H]^+$: 409.1246, found: 409.1250.

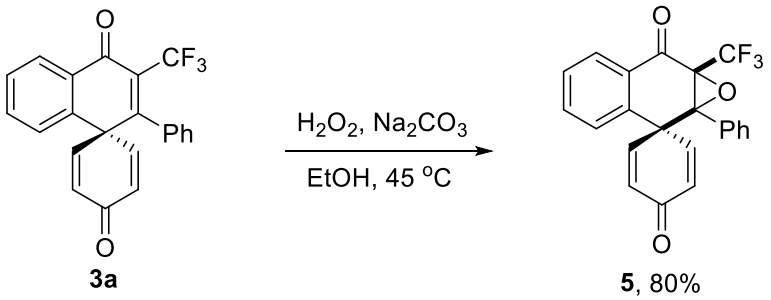


4o

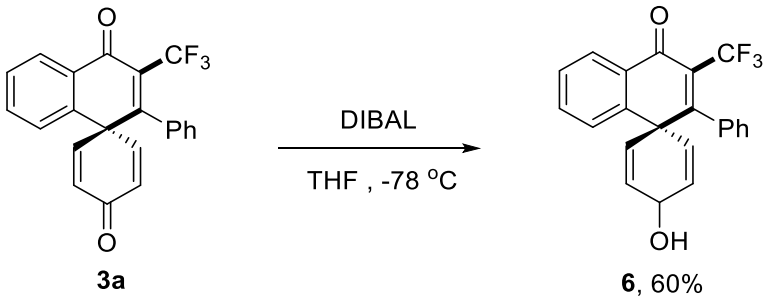
(*S*)-3'-(Difluoromethyl)-2,3-dimethoxy-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (4o)

The general procedure was followed using **1v'** (111.6 mg, 0.30 mmol) and **2b** (110 mg, 0.9 mmol). Purification by column chromatography on silica gel (*n*-hexane/EtOAc = 3:1) yielded **4o** (64.9 mg, 53%) as a white solid. **M.p.**: 143–148 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.30 (dd, J = 7.8, 1.3 Hz, 1H), 7.60 (ddd, J = 7.6, 7.6, 1.5 Hz, 1H), 7.57–7.52 (m, 1H), 7.42–7.35 (m, 2H), 7.35–7.28 (m, 1H), 7.27–7.25 (m, 1H), 7.20 (d, J = 6.8 Hz, 1H), 7.12 (d, J = 7.6 Hz, 1H), 6.43 (t, J = 53.4 Hz, 1H), 6.33 (dd, J = 56.1, 9.7 Hz, 1H), 3.85 (s, 3H), 3.43 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 183.9, 180.9, 159.5, 158.5 (t, J = 4.8 Hz), 142.4, 140.1, 138.6, 133.8, 133.0, 131.9 (t, J = 20.4 Hz), 130.6, 129.5, 129.4, 129.0, 128.0, 127.9, 127.8, 127.7, 127.5, 126.7, 111.8 (t, J = 240.2 Hz), 61.2, 60.6, 55.2. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -115.04 (d, J = 320.1 Hz, 1F), -116.48 (d, J = 320.3 Hz, 1F). **HR-MS** (ESI) *m/z* calc. for $C_{24}H_{19}F_2O_4$ $[M+H]^+$: 409.1246, found: 409.1248.

4. Derivation of naphthoquinone 3a to 5, 6, 7 and 8:

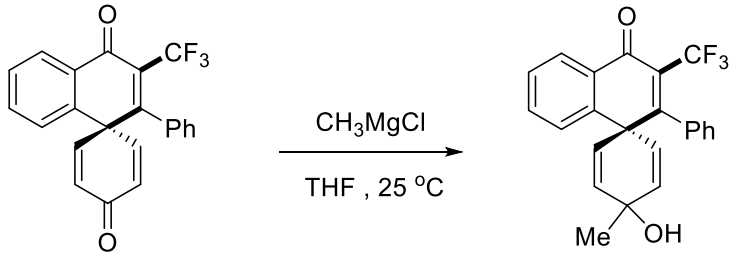


Trifluoromethylated spiro[5.5]trienone **3a** (0.3 mmol, 109.8 mg) was placed into a flask, EtOH (5 mL) was added and the solution was stirred for 5 min, before Na_2CO_3 (48 mg, 1.5 equiv) was added. Then H_2O_2 (30%, 1.5 equiv) was added to the biphasic solution and the reaction mixture was kept at 45 °C for 3 h. Then the mixture was diluted with CH_2Cl_2 and transferred to a round bottom flask. Silica was added to the flask and volatiles were evaporated under vacuum. The purification was performed by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1:5) gave product **8** (91.7 mg, 80% yield) as a white solid. **M.p.**: 150–154 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.99 (dd, J = 7.8, 1.5 Hz, 1H), 7.59 (ddd, J = 7.7, 7.7, 1.5 Hz, 1H), 7.55–7.50 (m, 1H), 7.40 (ddd, J = 6.4, 2.1, 2.1 Hz, 1H), 7.35–7.30 (m, 2H), 7.23–7.14 (m, 4H), 6.78 (dd, J = 10.0, 3.1 Hz, 1H), 6.45 (dd, J = 10.2, 1.7 Hz, 1H), 6.16 (dd, J = 10.0, 1.7 Hz, 1H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 188.4, 184.3, 148.0, 146.0, 130.1, 129.7, 129.6, 129.6, 129.4, 128.9, 128.2, 128.0, 127.4, 127.4, 127.0, 121.2 (q, J = 281.1 Hz), 70.1, 63.6 (q, J = 33.6 Hz), 51.6. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -65.61. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{14}F_3O_3$ $[M+H]^+$: 383.089; found: 383.089.



To a solution of trifluoromethylated spiro[5.5]trienones **3a** (0.3 mmol, 109.8 mg) in anhydrous THF at –78 °C, DIBAL (0.3 mmol, 1.0 eq.) was added. The solution was stirred for 3 hour at –78 °C. It was quenched by addition of saturated NH_4Cl (15 mL) solution and the aqueous phase was extracted with ethyl acetate (3 × 20 mL). The

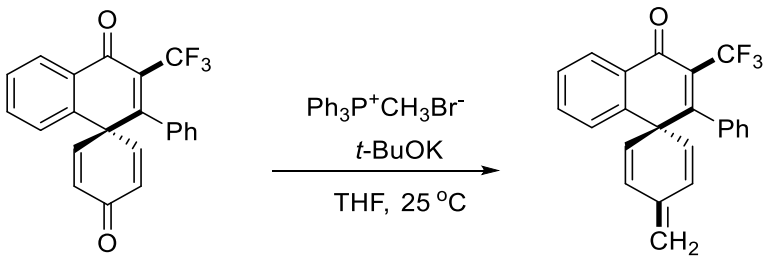
combined organic phases were dried over Na_2SO_4 and the solvent removed in vacuo. The crude compound was purified by silica gel chromatography (ethyl acetate/petroleum ether = 1:2) gave the corresponding alcohol **6** (66.6 mg, 60 %) as white solid. **M.p.**: 167–171 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.24 (dd, J = 7.9, 1.1 Hz, 1H), 7.59 (ddd, J = 7.9, 7.9, 1.4 Hz, 1H), 7.51–7.46 (m, 1H), 7.42–7.33 (m, 3H), 7.27–7.23 (m, 1H), 7.11 (dd, J = 7.9, 1.5 Hz, 2H), 6.10–6.02 (m, 2H), 5.69 (d, J = 9.4 Hz, 2H), 4.33 (dd, J = 7.3, 4.2 Hz, 1H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.1, 161.4 (q, J = 1.9 Hz), 143.1, 135.5, 133.6, 130.2, 129.5, 129.3, 128.8, 128.4 (q, J = 1.6 Hz), 128.3, 128.2 (q, J = 26.2 Hz), 127.0, 126.6, 122.1 (q, J = 277.8 Hz), 60.2, 49.2. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.50. **HR-MS** (ESI) *m/z* calc. for $C_{22}H_{16}F_3O_2$ $[M+H]^+$: 369.1097, found: 369.1102.



3a **7**, 50%

To a solution of trifluoromethylated spiro[5.5]trienones **3a** (0.3 mmol, 109.8 mg) in anhydrous THF at °C was added CH_3MgCl (0.6 mmol, 2.0 eq.). The solution was stirred for 3 hour. To quench the reaction, saturated NH_4Cl solution (15 mL) was added and the aqueous phase was extracted with ethyl acetate (3 × 20 mL). The combined organic phases were dried over Na_2SO_4 and the solvent removed in vacuo. The crude compound was purified by silica gel chromatography (ethyl acetate/petroleum ether = 1:2) gave the corresponding alcohol **7** (57.3 mg, 50% yield) as white solid. **M.p.**: 160–164 °C. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.42 (d, J = 7.7 Hz, 1H), 7.77 (d, J = 7.2 Hz, 1H), 7.68 (d, J = 7.2 Hz, 1H), 7.61–7.53 (m, 3H), 7.37 (d, J = 7.8 Hz, 1H), 7.31 (d, J = 6.2 Hz, 2H), 6.09 (d, J = 9.8 Hz, 2H), 5.77 (d, J = 9.8 Hz, 2H), 1.50 (s, 3H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.1, 161.4 (q, J = 1.7 Hz), 143.0, 135.5, 134.2, 133.6, 129.5, 129.1, 128.7, 128.2, 128.0, 127.9 (q, J = 24.5 Hz), 127.0, 126.7, 122.1 (q, J = 277.7 Hz), 63.4, 49.3, 28.0. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.48. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{18}F_3O_2$

$[M+H]^+$: 383.1253, found: 383.1255.

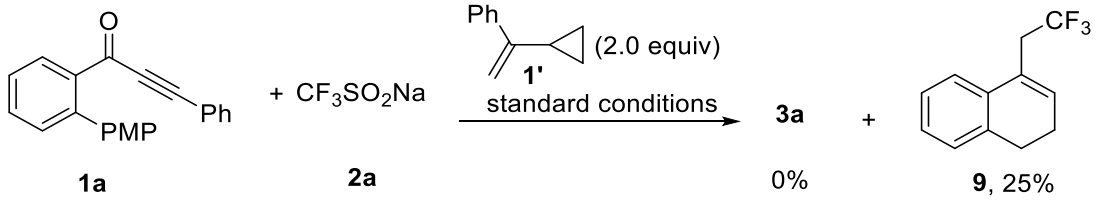


3a **8**, 56%

To a solution of methyltriphenylphosphoniumbromide (214.3 mg, 0.6 mmol, 2.0 eq.) in THF (10 mL) under Ar atmosphere, potassium *tert*-butoxide (67.3 mg, 0.6 mmol, 2.0 eq.) was added as a solution in THF (5 mL) at 25 ºC. The reaction mixture was stirred for 1 h at room temperature. Then trifluoromethylated spiro[5.5]trienones **3a** (0.3 mmol, 109.8 mg) was added and the reaction was stirred for 6 h at room temperature and quenched with saturated NH_4Cl aqueous solution (15 mL). The solvent was removed in vacuo and the resulting mixture was extracted with diethyl ether (3 × 15 mL). The combined organic layer was washed with brine, and dried over anhydrous sodium sulfate. Evaporation of the solvent followed by purification by flash chromatography on silica gel (*n*-hexane/EtOAc = 5:1) gave compound **8** (61.2 mg, 56%) as a white solid. **M.p.**: 143–146 ºC. **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.23 (dd, J = 7.9, 1.5 Hz, 1H), 7.58 (ddd, J = 7.6, 7.6, 1.5 Hz, 1H), 7.48–7.43 (m, 1H), 7.40 (d, J = 7.9 Hz, 1H), 7.34–7.23 (m, 3H), 7.08 (ddd, J = 6.9, 6.9, 1.5 Hz, 2H), 6.32 (d, J = 9.7 Hz, 2H), 5.53 (d, J = 9.5 Hz, 2H), 4.93 (s, 2H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.3, 163.2, 144.2, 136.2, 135.0, 133.6, 129.5, 129.5, 129.2, 129.1, 128.2, 128.0, 128.0 (q, J = 47.4 Hz), 127.3 (q, J = 1.4 Hz), 127.1, 126.8, 122.3 (q, J = 277.8 Hz), 116.4, 50.9. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.28. **HR-MS** (ESI) *m/z* calc. for $C_{23}H_{16}F_3O$ $[M+H]^+$: 365.1148, found 365.1153.

5. Mechanistic Studies

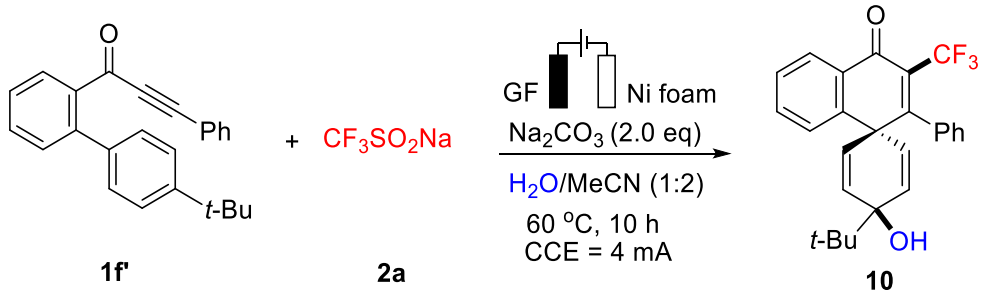
5.1 Radical Clock Experiment



1a 2a 0% 9, 25%

The electrocatalysis was carried out in an undivided cell under air with a graphite felt (GF) anode (10 mm × 15 mm × 6 mm) and a nickel cathode (10 mm × 15 mm × 0.25 mm). 1-(4'-methoxy-[1,1'-biphenyl]-2-yl)-3-phenylprop-2-yn-1-one **1a** (0.3 mmol, 1.0 equiv), CF_3SO_2Na **2a** (0.9 mmol, 3.0 equiv) and Na_2CO_3 (0.6 mmol, 3.0 equiy) were dissolved in a mixture of H_2O/MeCN (1 : 2, 4 mL), then (1-cyclopropylvinyl) benzene 1' (86.5 mg, 0.6 mmol, 0.2 eq.) was added as the radical-trapping reagent. Electrocatalysis was performed at 60 °C with a constant current of 4.0 mA maintained for 10 h. The GF anode was washed with ethyl acetate (3 × 5 mL) in an ultrasonic bath and the solvent was transferred to a round bottom flask. Silica was added to the flask and all volatiles were evaporated under vacuum. Purification was performed by flash column chromatography on silica gel (*n*-hexane/EtOAc = 20:1) gave the corresponding products **9** (31.8 mg, 25%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.30 (m, 4H), 5.94 (t, J = 7.2 Hz, 1H), 4.51–4.47 (m, 1H), 4.27–4.24 (m, 1H), 3.37–3.32 (m, 2H), 2.74–2.70 (m, 2H). ^{19}F NMR (565 MHz, $CDCl_3$) δ -63.65. The NMR data was consistent with the literature.[3]

5.2 Trapping Experiment for Key Intermediate



1f' 2a 10

The electrocatalysis was carried out in an undivided cell under air with a graphite felt (GF) anode (10 mm × 15 mm × 6 mm) and a nickel cathode (10 mm × 15 mm × 2 mm). 1-(4'-(*tert*-butyl)-[1,1'-biphenyl]-2-yl)-3-phenylprop-2-yn-1-one **1f'** (0.3 mmol, 1.0

equiv), CF_3SO_2Na **2a** (0.9 mmol, 3.0 equiv) and Na_2CO_3 (0.6 mmol, 3.0 equiy) were dissolved in a mixture of H_2O/MeCN (1 : 2 mL). Electrocatalysis was performed at 60 °C with a constant current of 4.0 mA maintained for 10 h. The GF anode was washed with ethyl acetate (3 × 5 mL) in an ultrasonic bath and the solvent was transfered to a round bottom flask. Silica was added to the flask and all volatiles were evaporated under vacuum. Purification was performed by flash column chromatography on silica gel (*n*-hexane/EtOAc = 4:1) gave the corresponding products **10** (38.2 mg, 30%) as a white solid. **M.p.**: 149–152 ºC. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 8.13 (d, J = 7.5 Hz, 1H), 7.51 (dd, J = 5.7, 1.5 Hz, 2H), 7.37 (ddd, J = 8.1, 6.2, 2.3 Hz, 1H), 7.20 (m, 3H), 6.97 (dd, J = 6.6, 2.9 Hz, 2H), 6.03 (dd, J = 6.5, 6.5 Hz, 2H), 5.74 (t, J = 6.4 Hz, 2H), 0.47 (s, 9H). **^{13}C NMR** (151 MHz, $CDCl_3$) δ 180.5, 162.6 (q, J = 1.9 Hz), 142.5, 135.5, 133.7, 130.5, 130.2, 129.5, 128.2, 128.1 (q, J = 26.4 Hz), 128.0, 127.6 (q, J = 1.4 Hz), 127.3, 126.8, 122.2 (q, J = 277.9 Hz), 70.1, 49.6, 36.8, 24.4. **^{19}F NMR** (565 MHz, $CDCl_3$) δ -56.15. **HRMS** (ESI) (*m/z*): calcd for $C_{24}H_{24}F_3O_2$ ($[M+H]^+$): 425.1723, found: 425.1728.

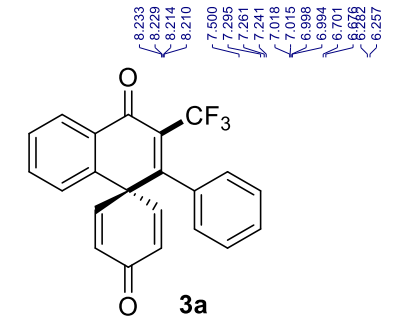
6. References

[1] Y. Zhang, D. Guo, S. Ye, Z. Liu, G. Zhu, *Org. Lett*. **2017**, *19*, 1302–1305. .

[2] Y. Yang, J. Zhang, B. Hu, M. Ji, S. Ye, G. Zhu, *Org. Lett.* **2018**, *20*, 2988–2992.

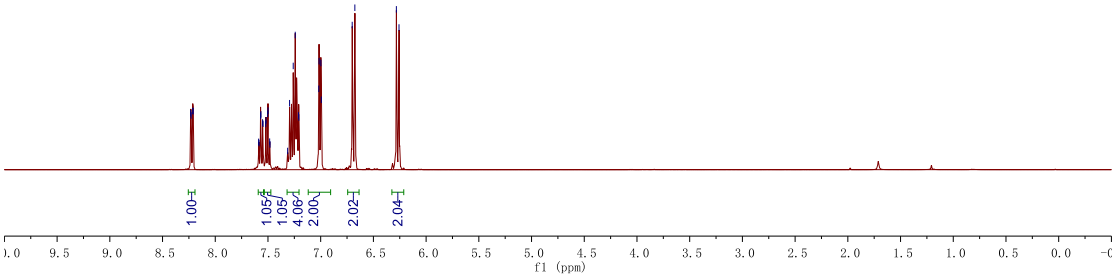
[3] Q. -F. Bao, Y. Xia, M. Li, Y.-Z. Wang, Y.-M. Liang, *Org. Lett.* **2020**, *22*, 7757–7761.

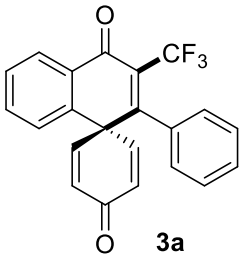
7. NMR Spectra



3a

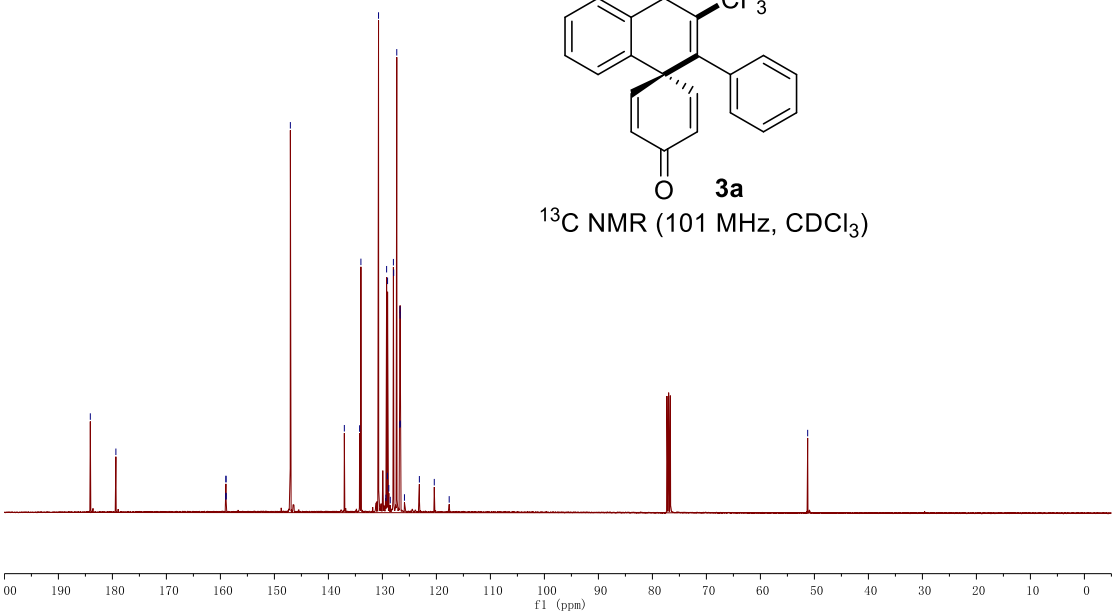
^{1}H NMR (400 MHz, $CDCl_3$)





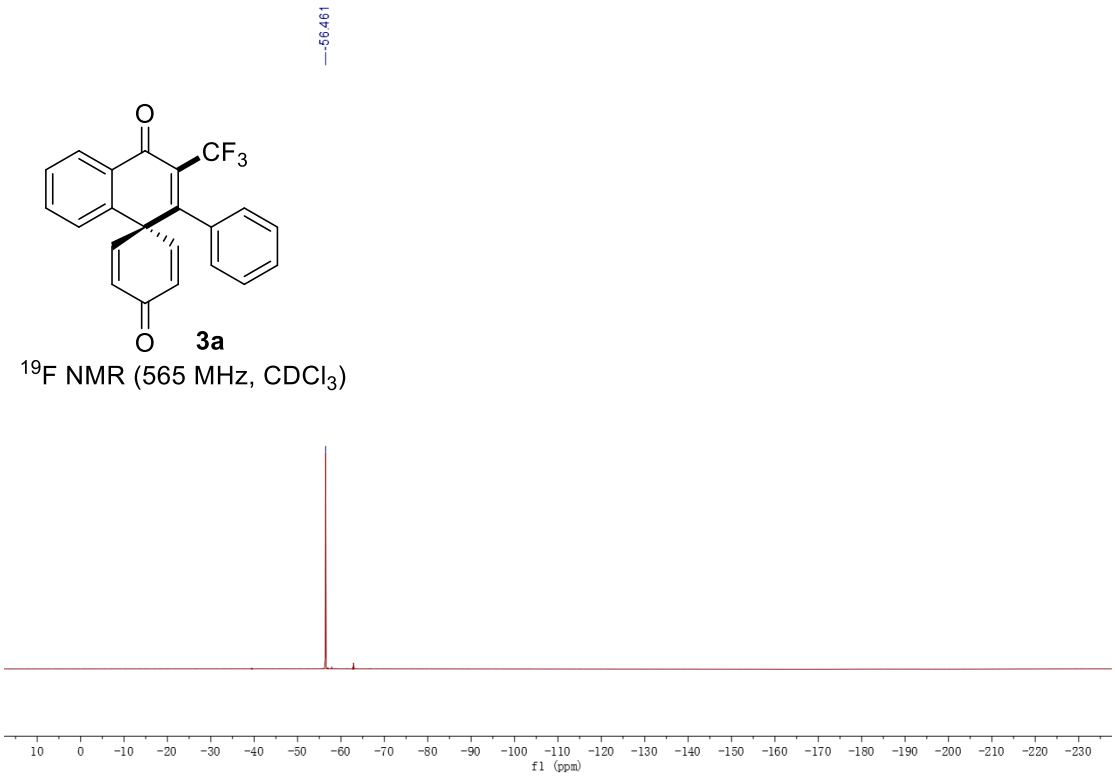
3a

^{13}C NMR (101 MHz, $CDCl_3$)



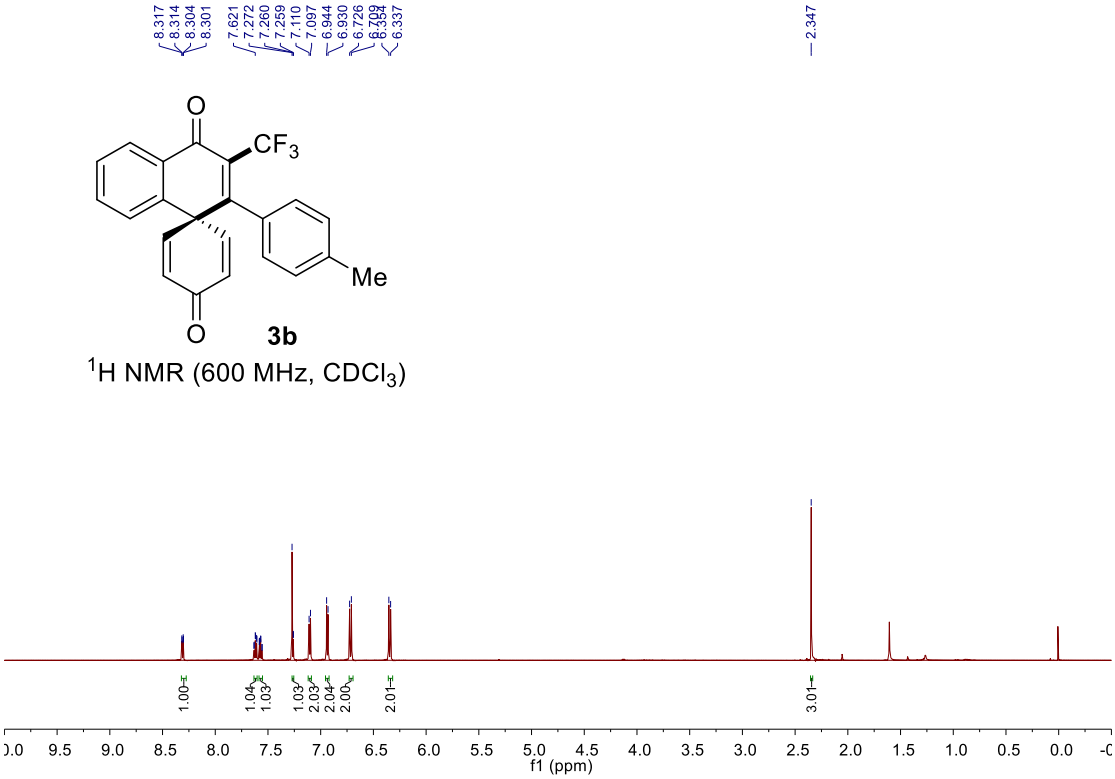
3a

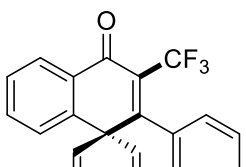
^{19}F NMR (565 MHz, $CDCl_3$)

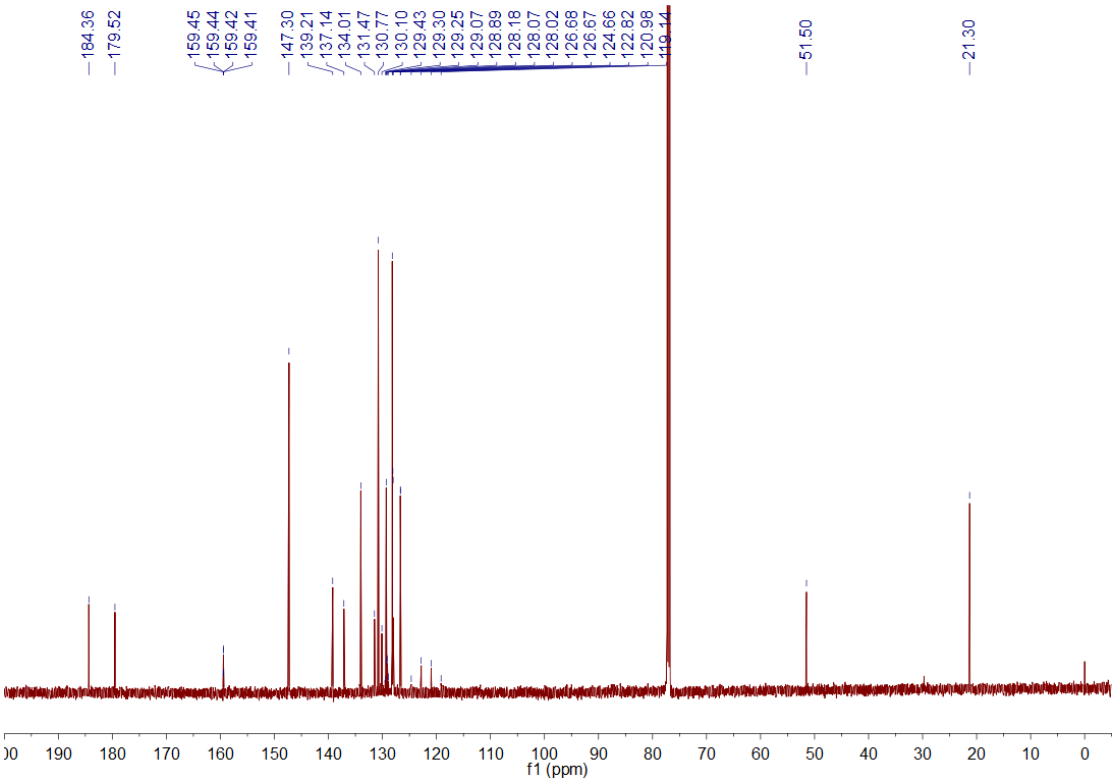


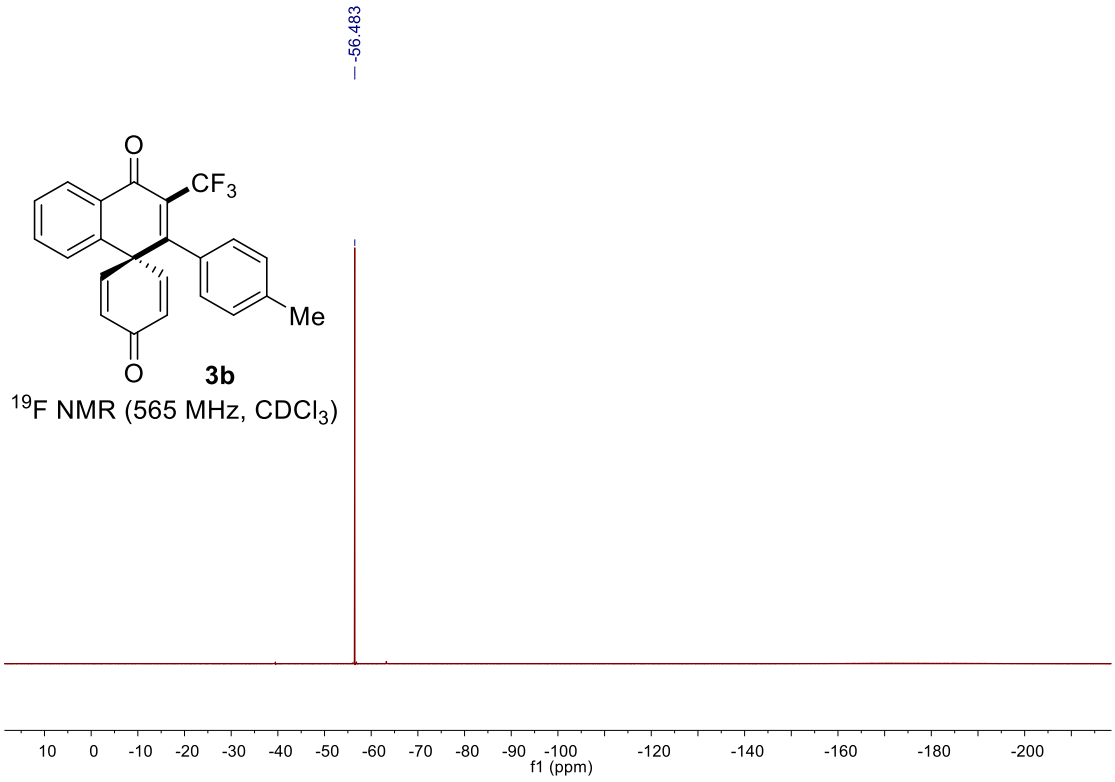
3b

^{1}H NMR (600 MHz, $CDCl_3$)



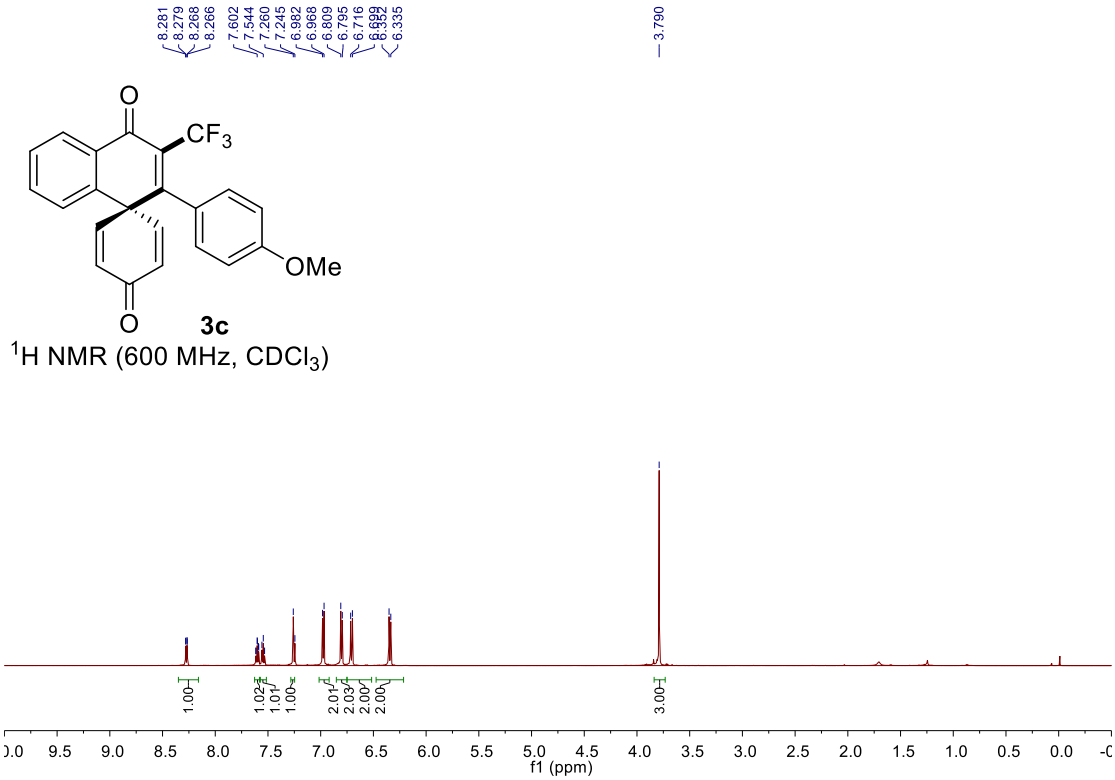






3b

^{19}F NMR (565 MHz, $CDCl_3$)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8.266
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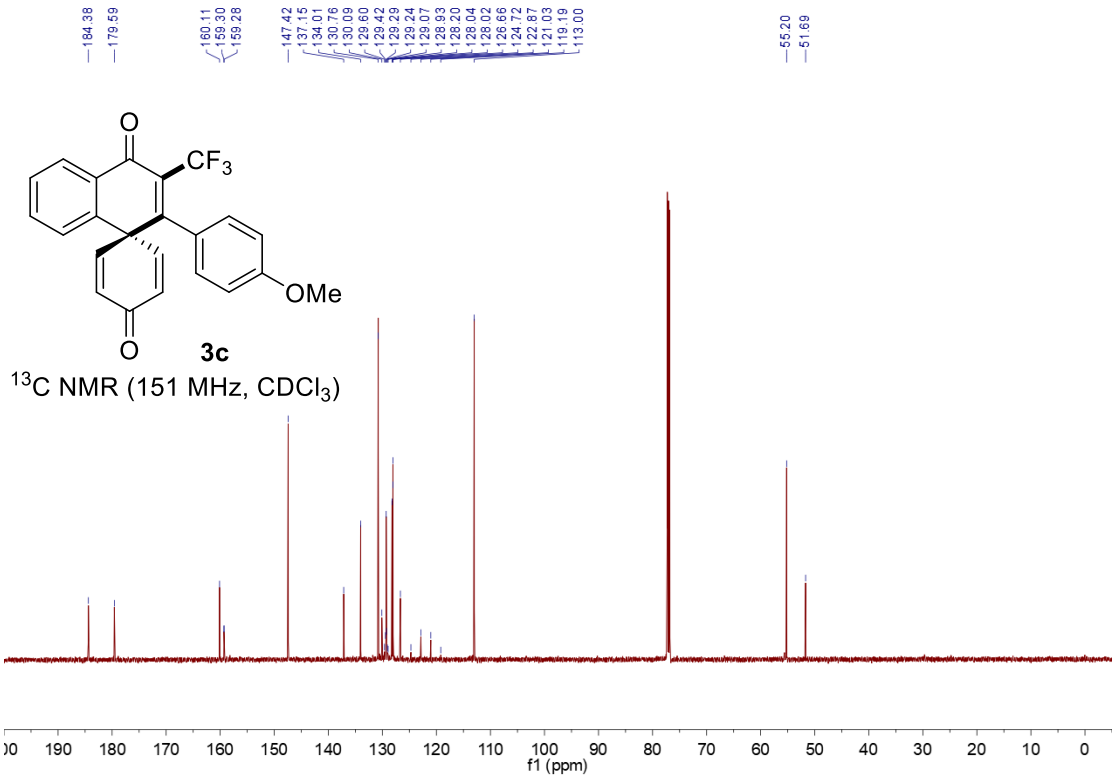
7.544
7.260
7.245
6.982
6.968
6.809
6.795
6.716
6.699
6.352
6.335
3.790
O
CF3
OMe
O
3c
1H NMR (600 MHz, CDCl3)
1.00
1.02
1.01
1.00
2.01
2.03
2.00
2.00
3.00
f1 (ppm)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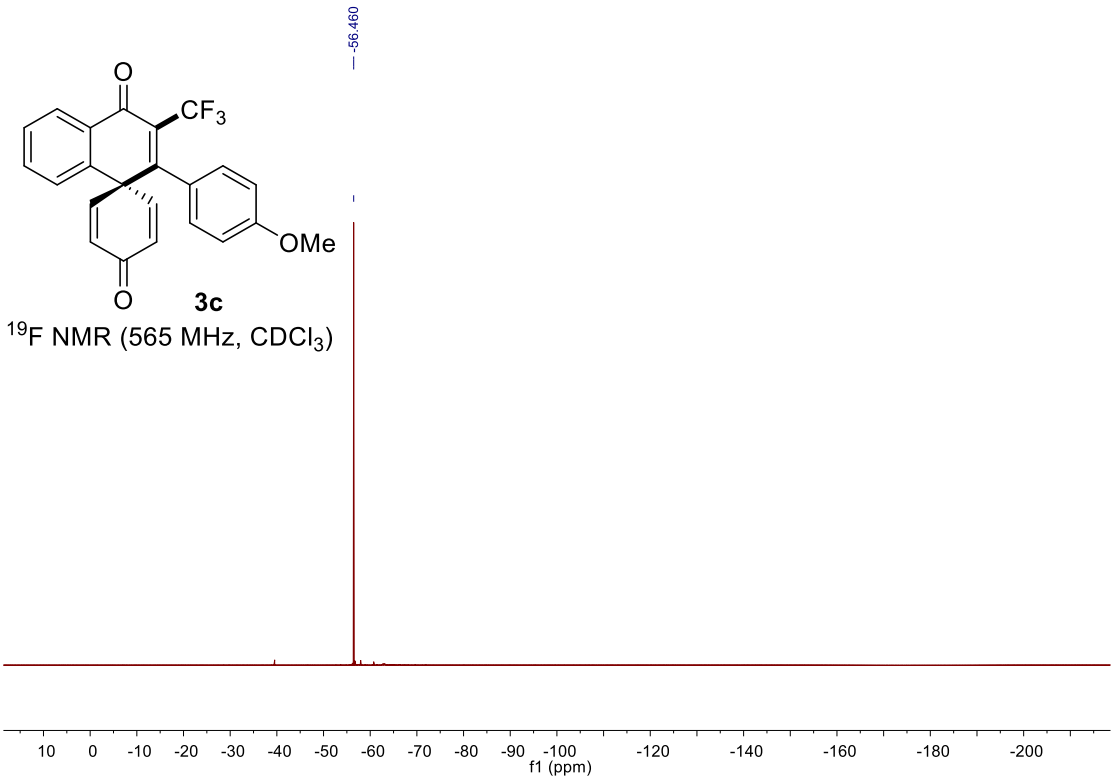
179.59
160.11
159.30
159.28
147.42
137.15
134.01
130.76
130.09
129.60
129.42
129.29
129.24
129.07
128.93
128.20
128.04
128.02
126.66
124.72
122.87
121.03
119.19
113.00
55.20
51.69
O
CF3
OMe
O
3c
13C NMR (151 MHz, CDCl3)
f1 (ppm)

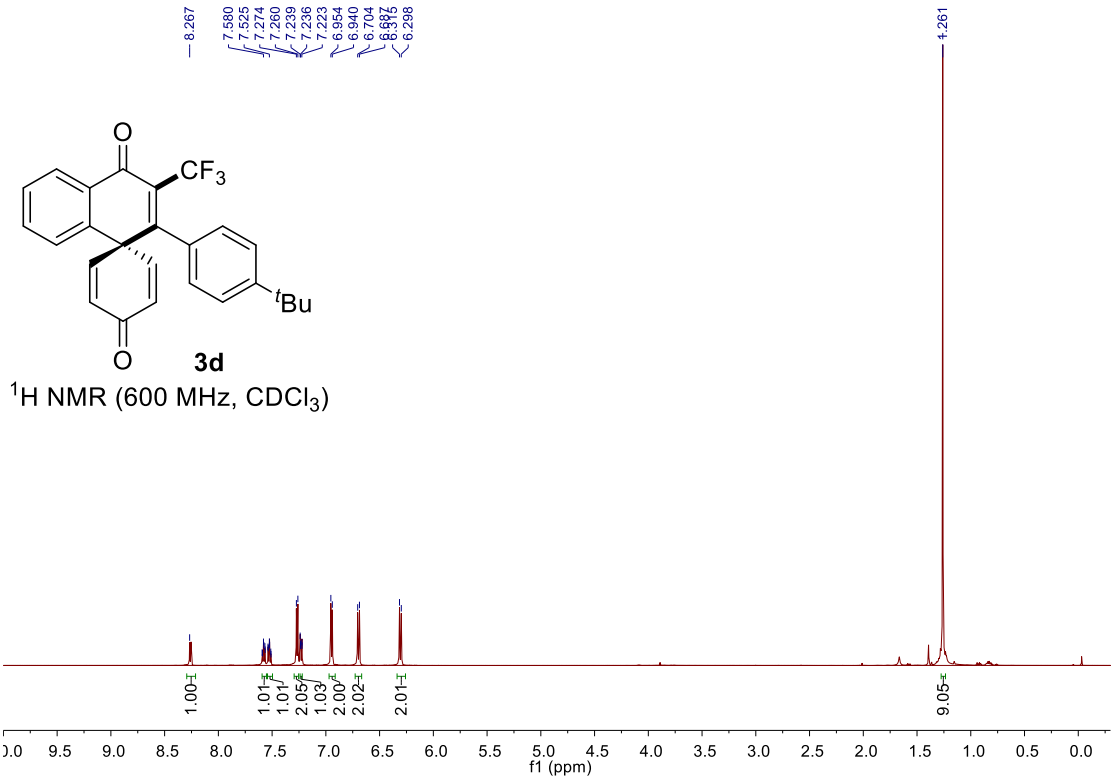
3c

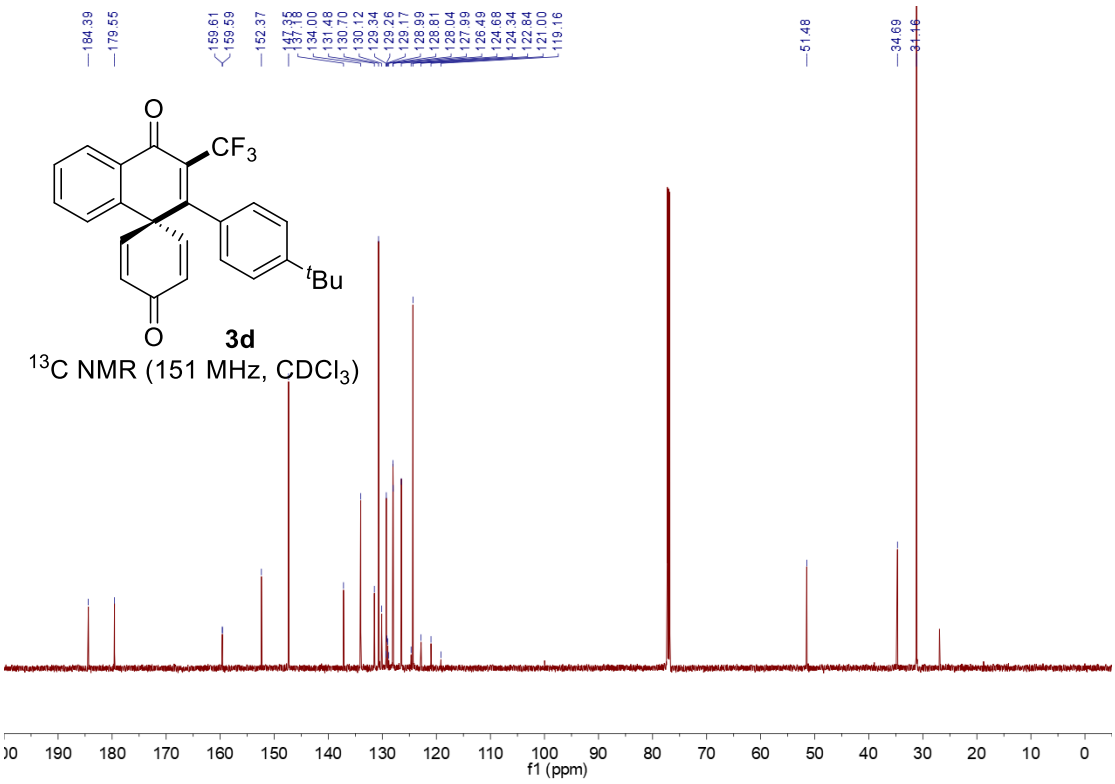
^{19}F NMR (565 MHz, $CDCl_3$)



3d

^{1}H NMR (600 MHz, $CDCl_3$)

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159.61
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147.35
137.18
134.00
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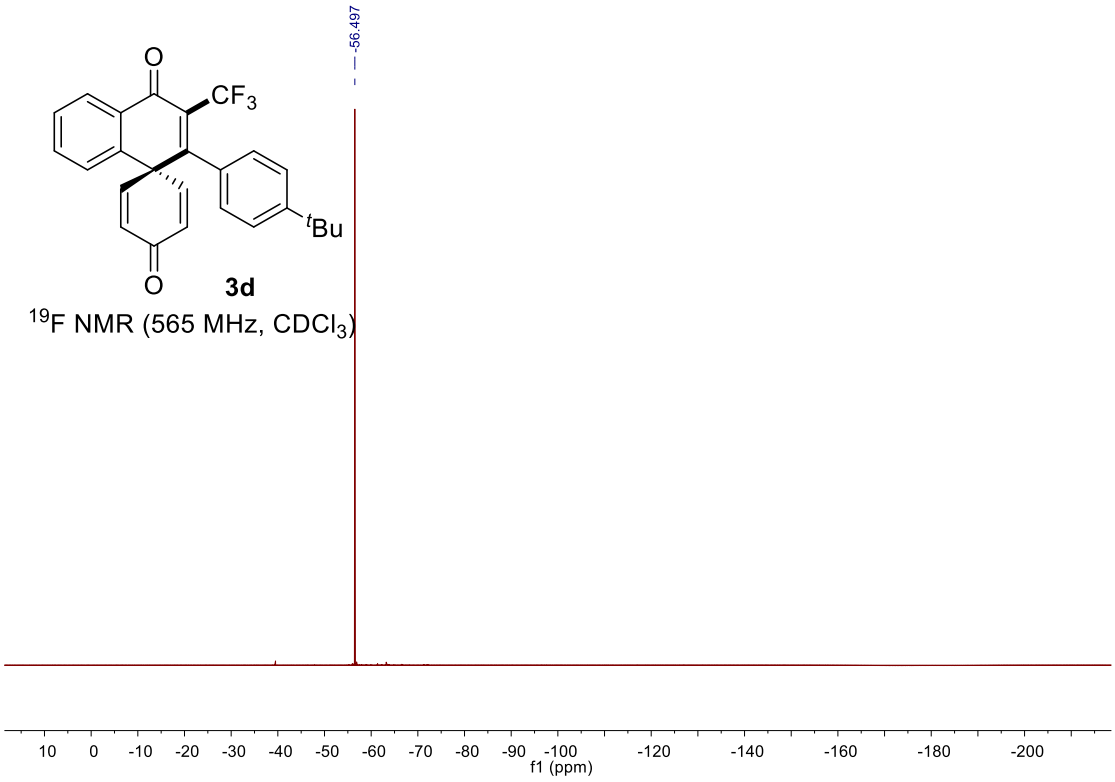
130.70
130.12
129.34
129.26
129.17
128.99
128.81
128.04
127.99
126.49
124.68
124.34
122.84
121.00
119.16
51.48
34.69
31.16
O
CF3
tBu
O
3d
13C NMR (151 MHz, CDCl3)
f1 (ppm)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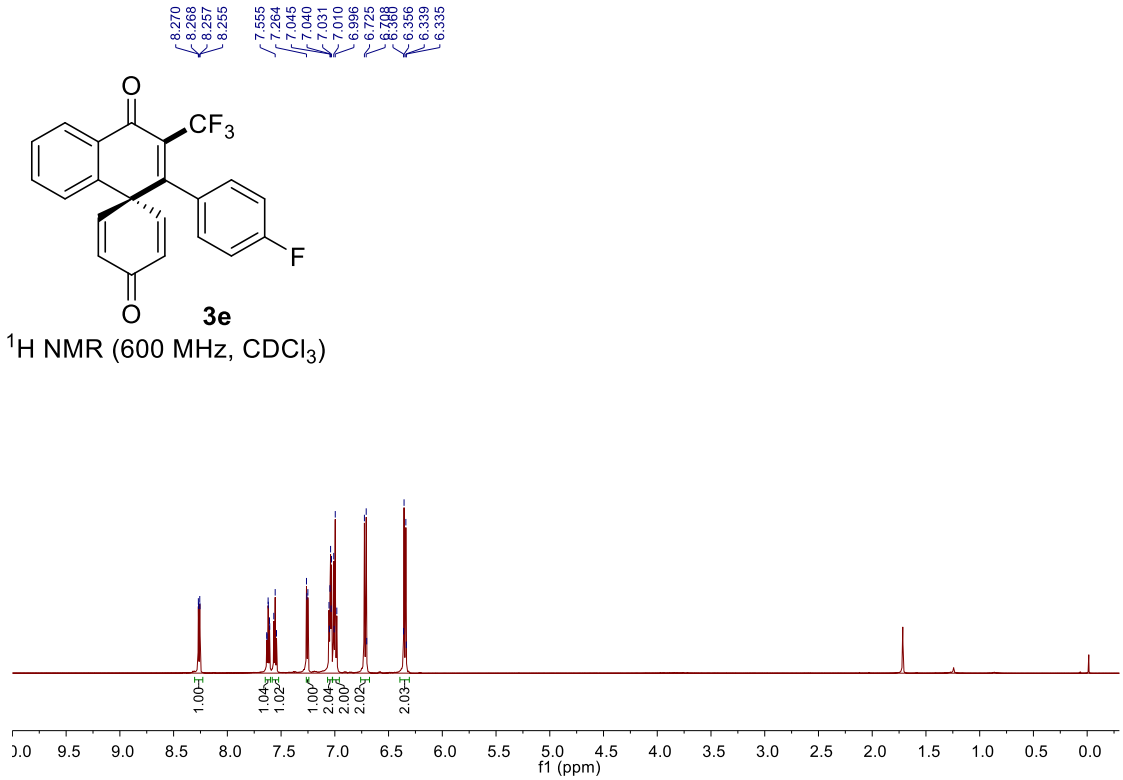
f1 (ppm)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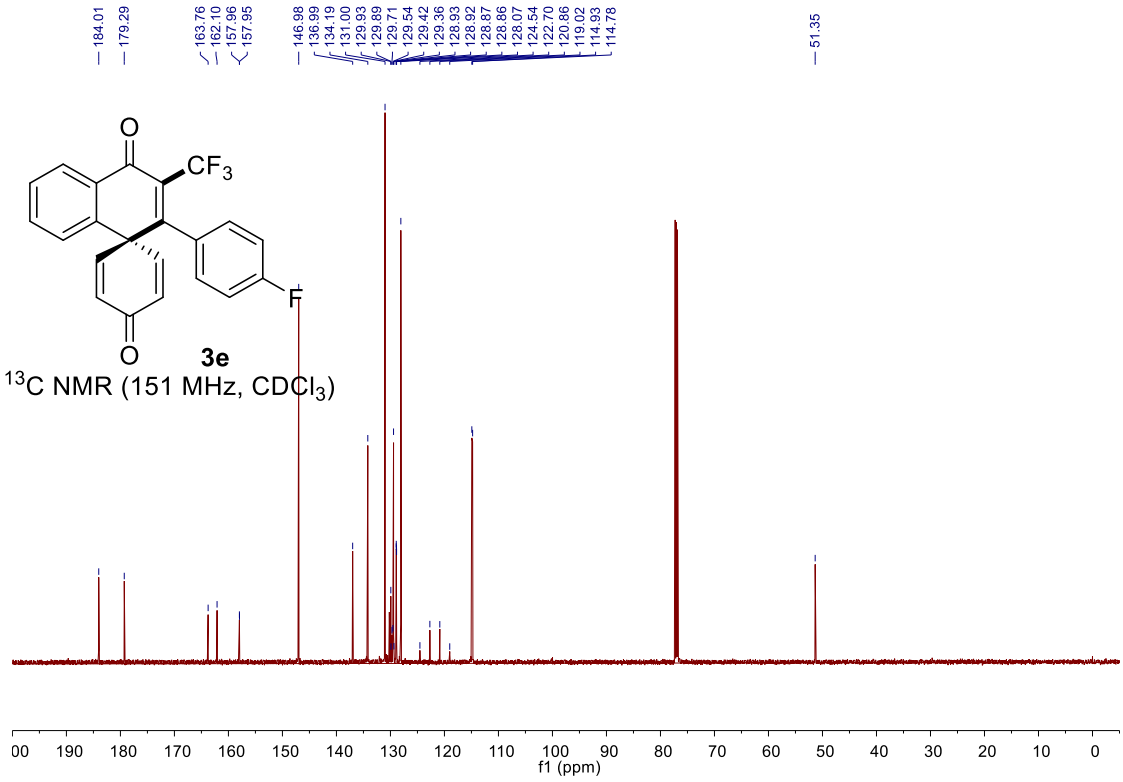
8.255
7.555
7.264
7.045
7.040
7.031
7.010
6.996
6.725
6.708
6.360
6.356
6.339
6.335
O
CF3
F
O
3e
1H NMR (600 MHz, CDCl3)
1.00
1.04
1.02
1.00
2.04
2.00
2.02
2.03
f1 (ppm)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157.96
157.95
146.98
136.99
134.19
131.00
129.93
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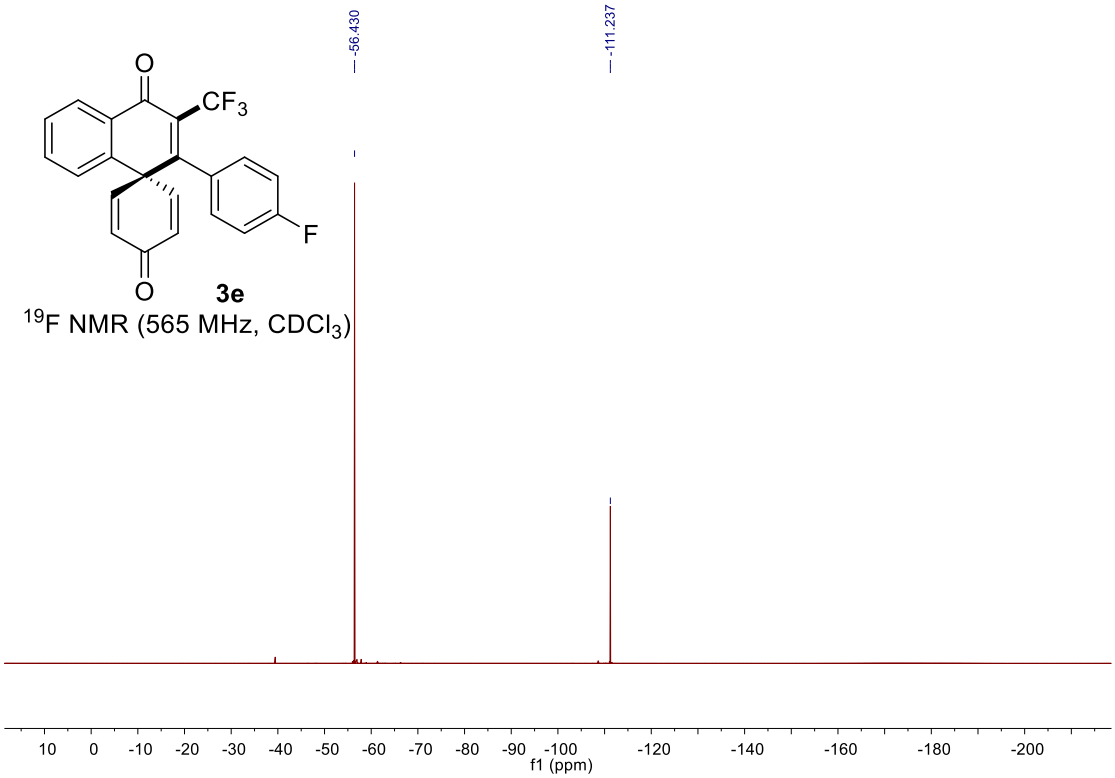
129.71
129.54
129.42
129.36
128.93
128.92
128.87
128.86
128.07
124.54
122.70
120.86
119.02
114.93
114.78
51.35
O
CF3
F
O
3e
13C NMR (151 MHz, CDCl3)
f1 (ppm)

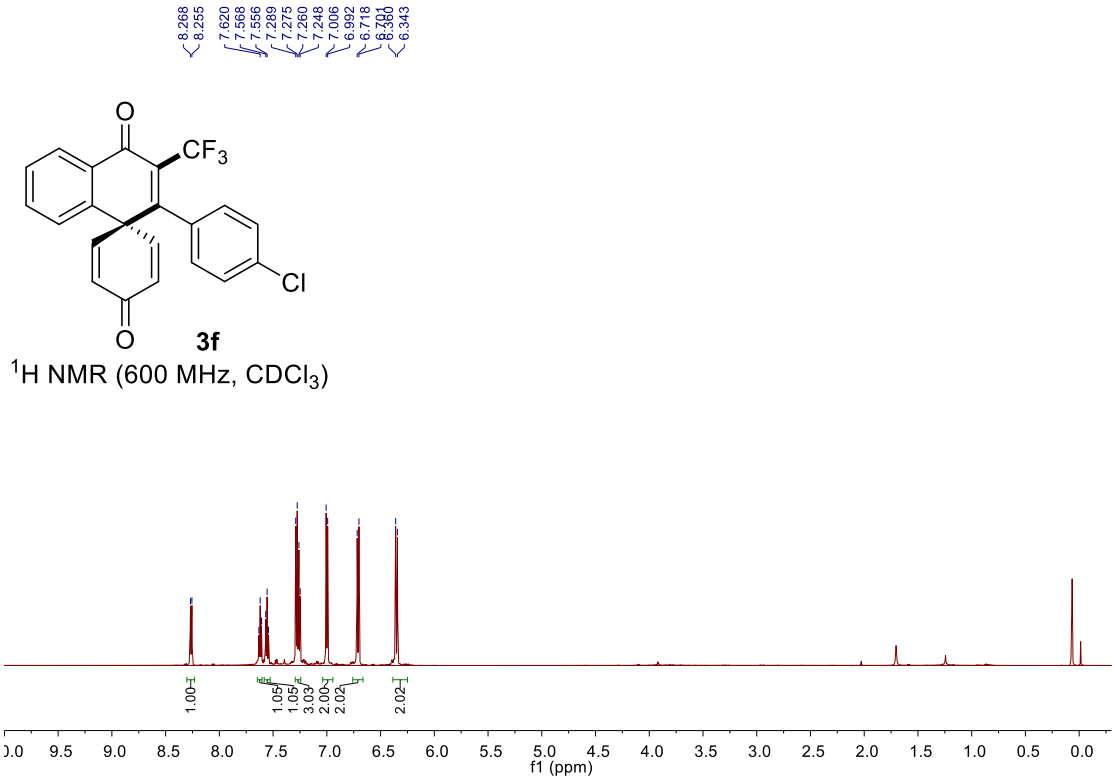
3e

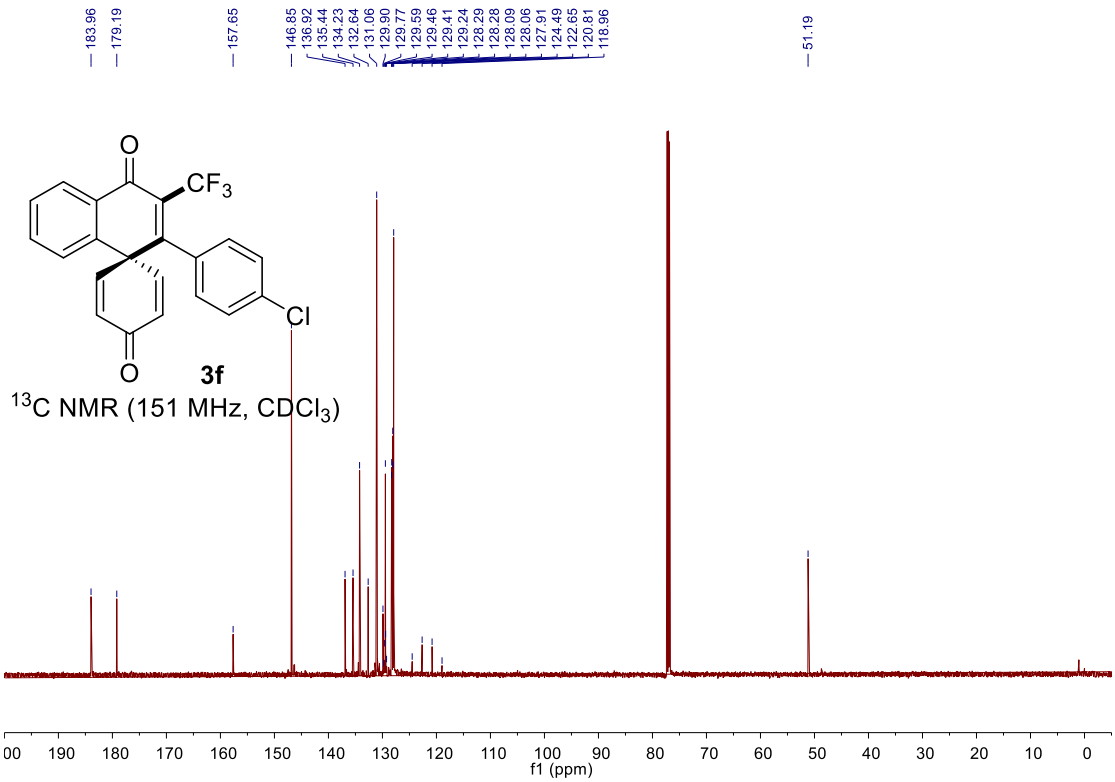
^{19}F NMR (565 MHz, $CDCl_3$)



3f

^{1}H NMR (600 MHz, $CDCl_3$)

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129.41
129.24
128.29
128.28
128.09
128.06
127.91
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122.65
120.81
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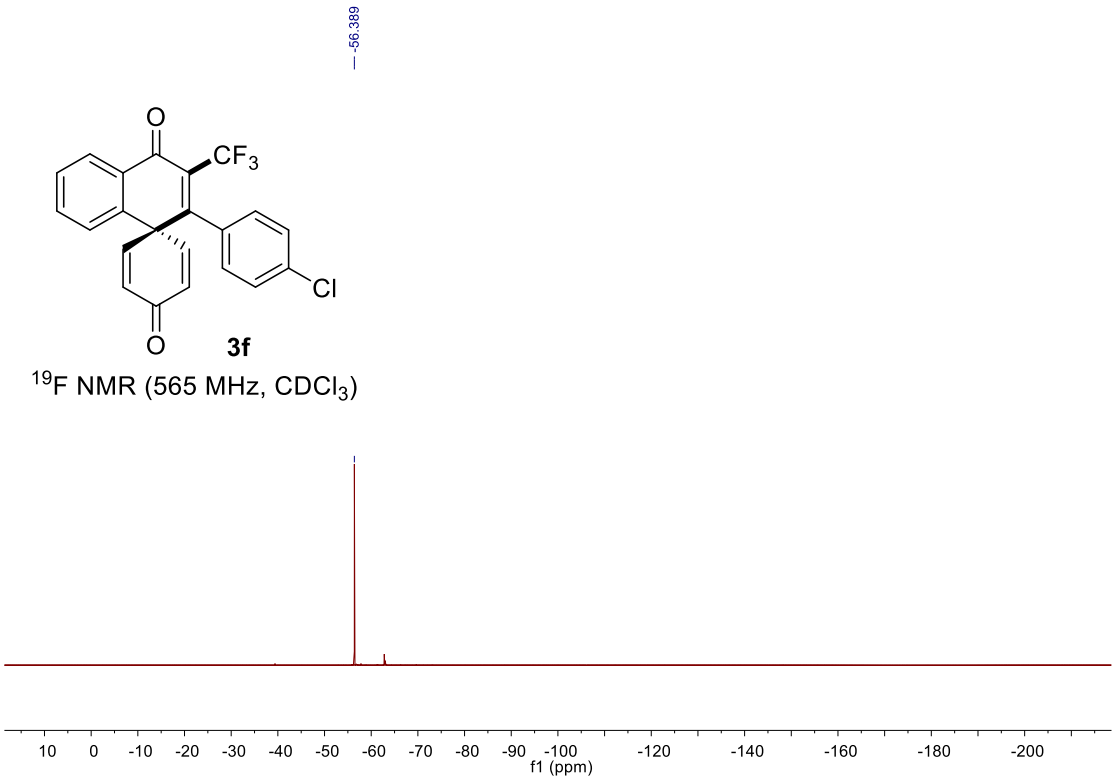
51.19
O
CF3
Cl
O
3f
13C NMR (151 MHz, CDCl3)
f1 (ppm)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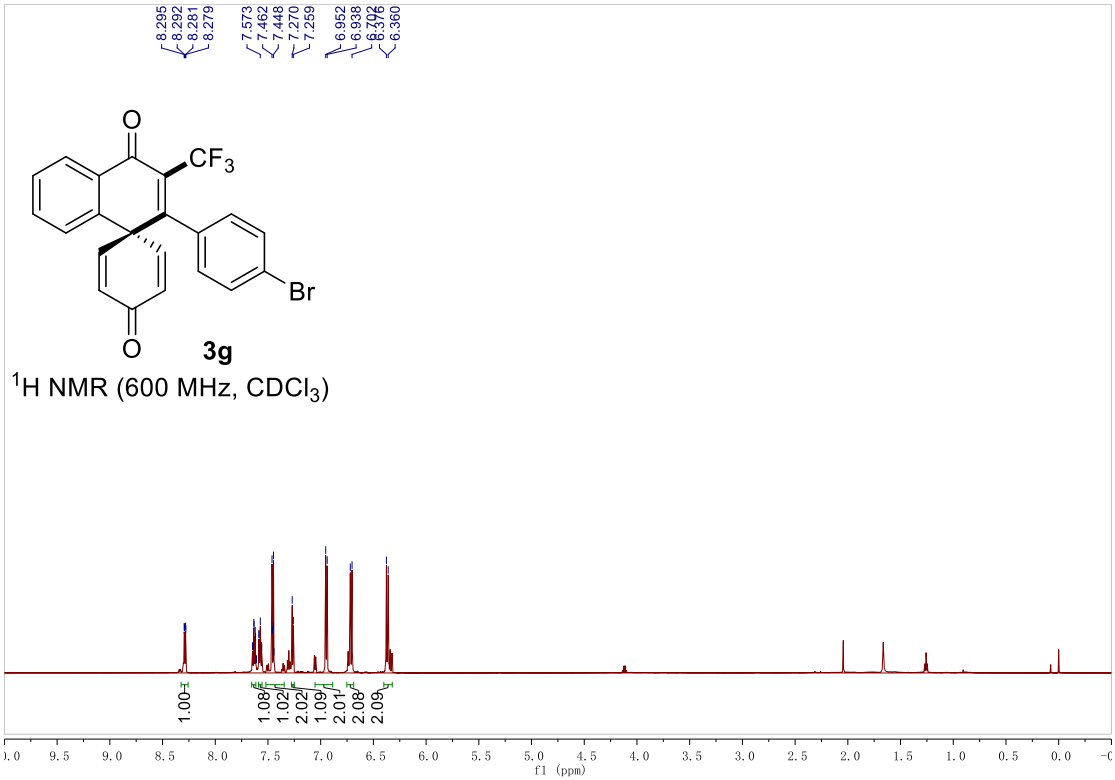
f1 (ppm)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6.952
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6.360
O
CF3
Br
O
3g
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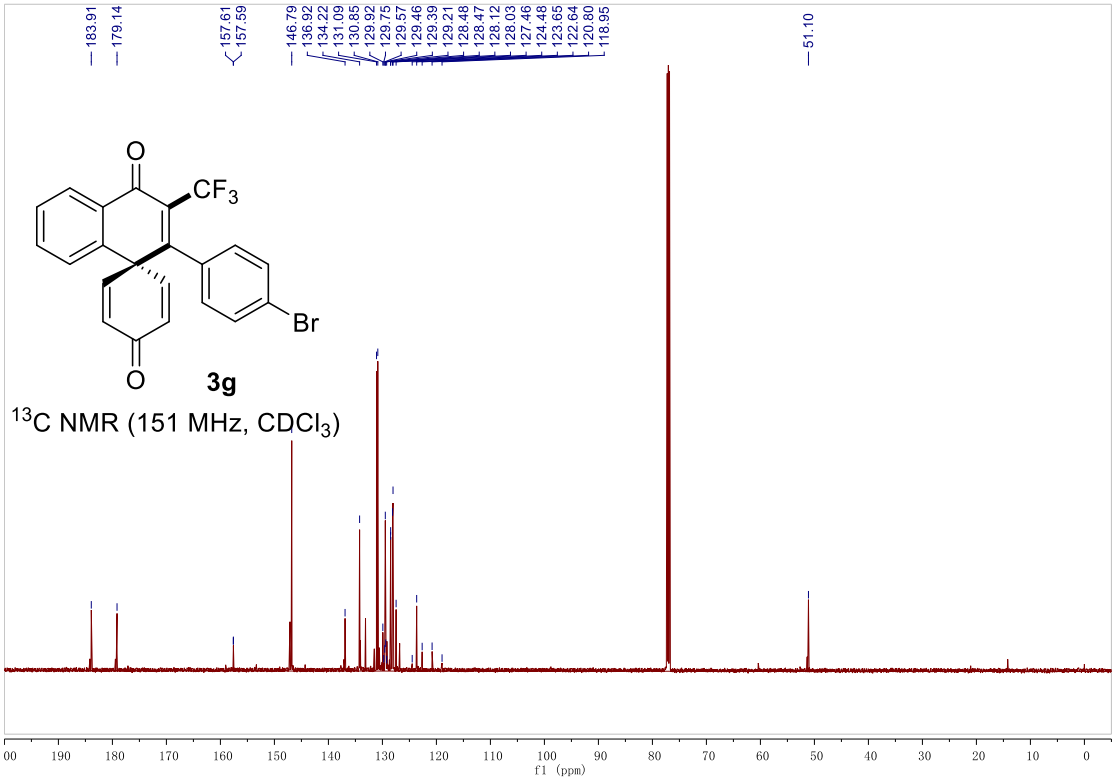
1.08
1.02
2.02
1.09
2.01
2.08
2.09
f1 (ppm)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136.92
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129.92
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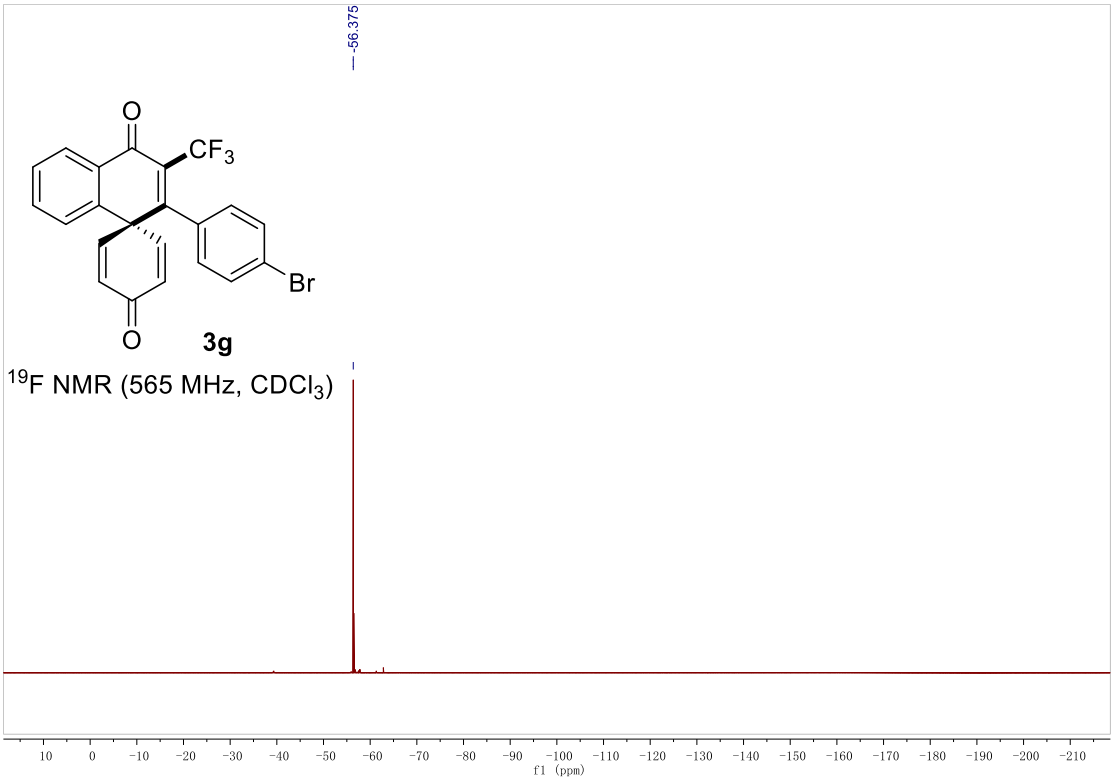
f1 (ppm)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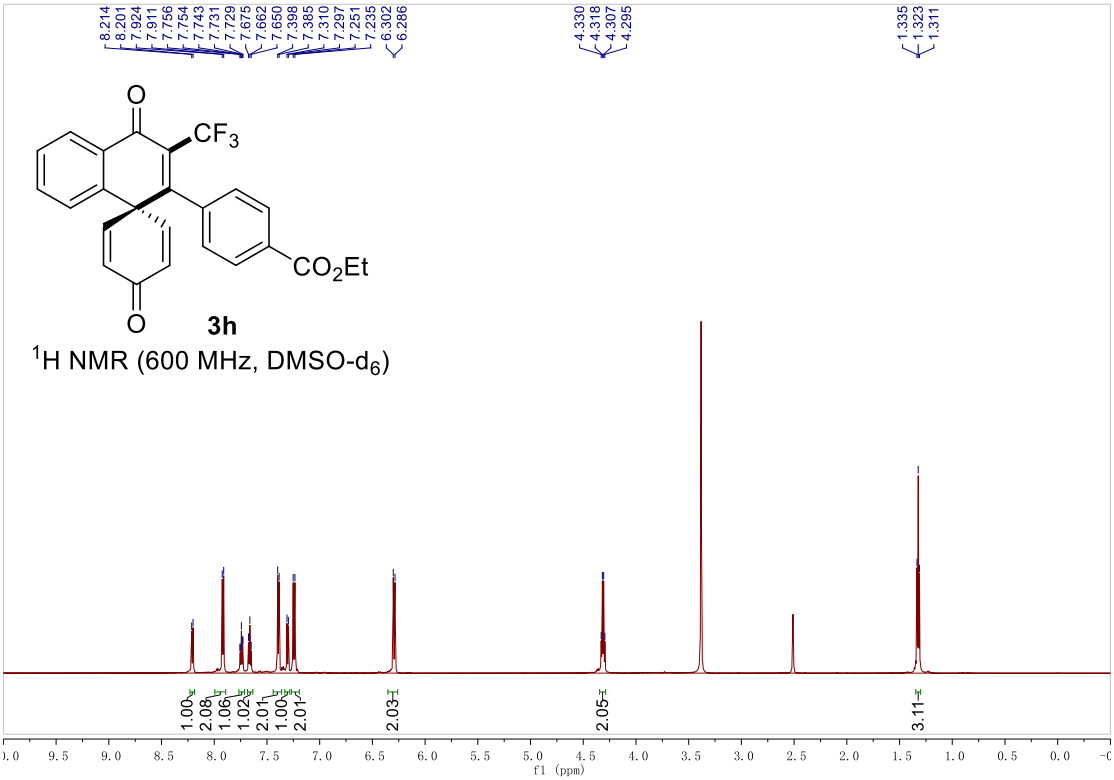
CF3
Br
O
3g
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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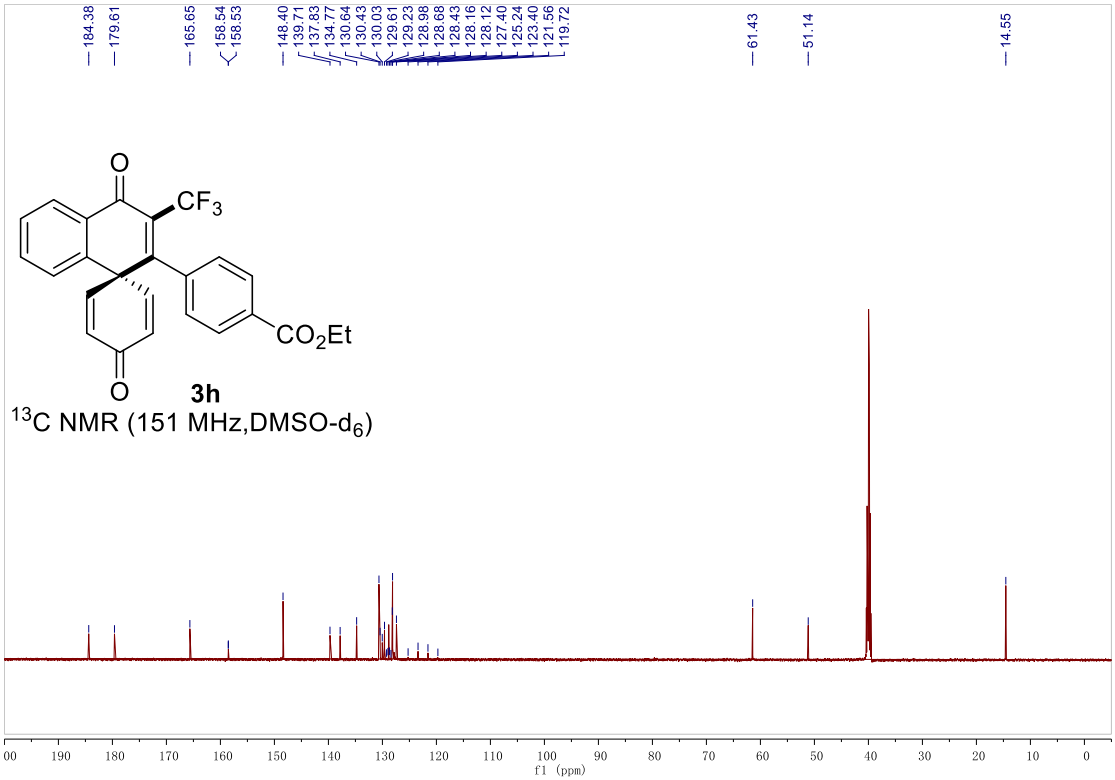
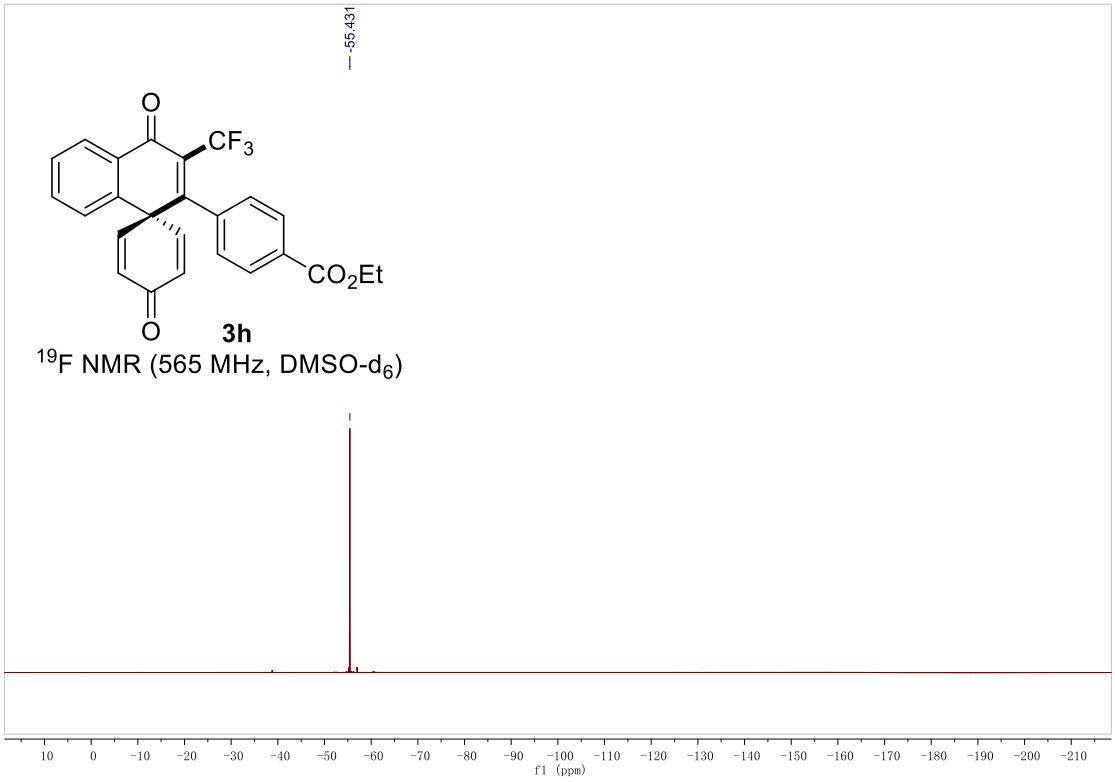
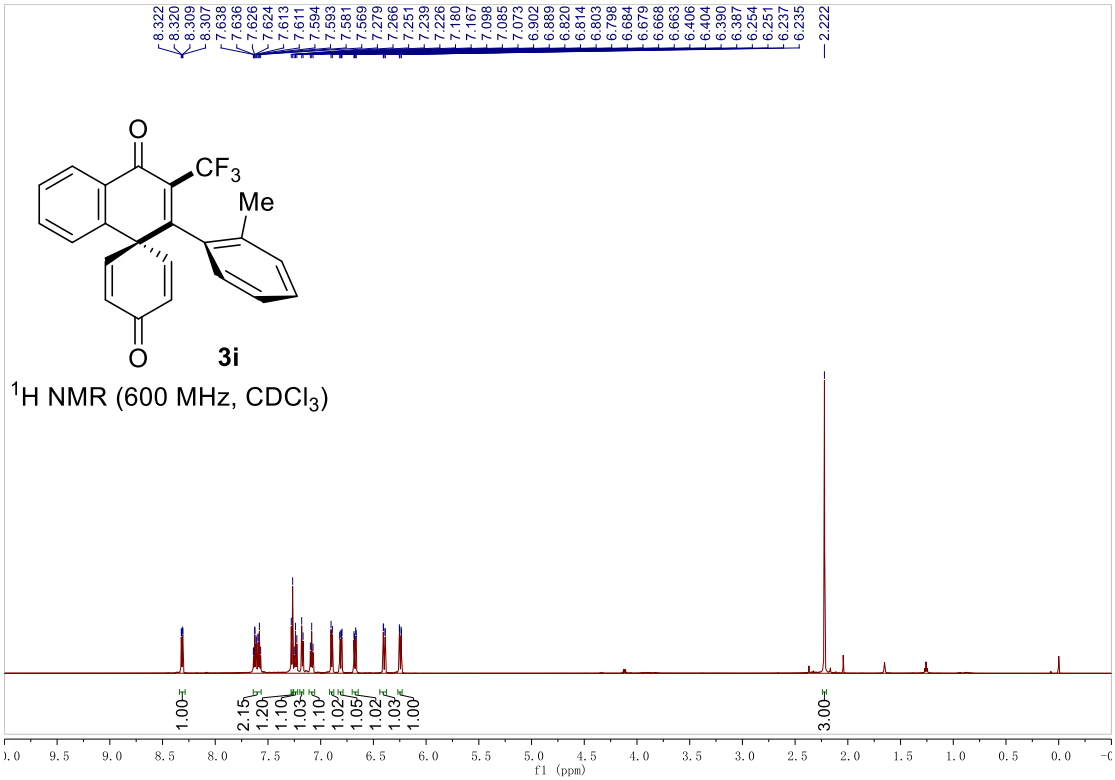
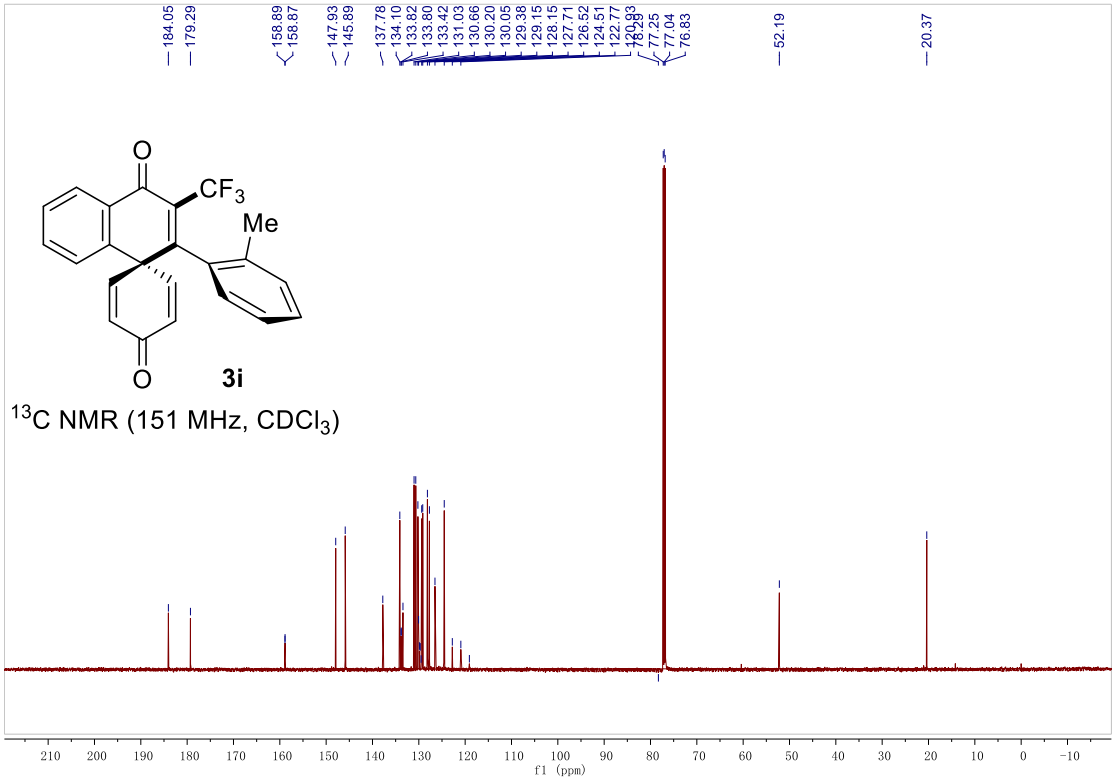
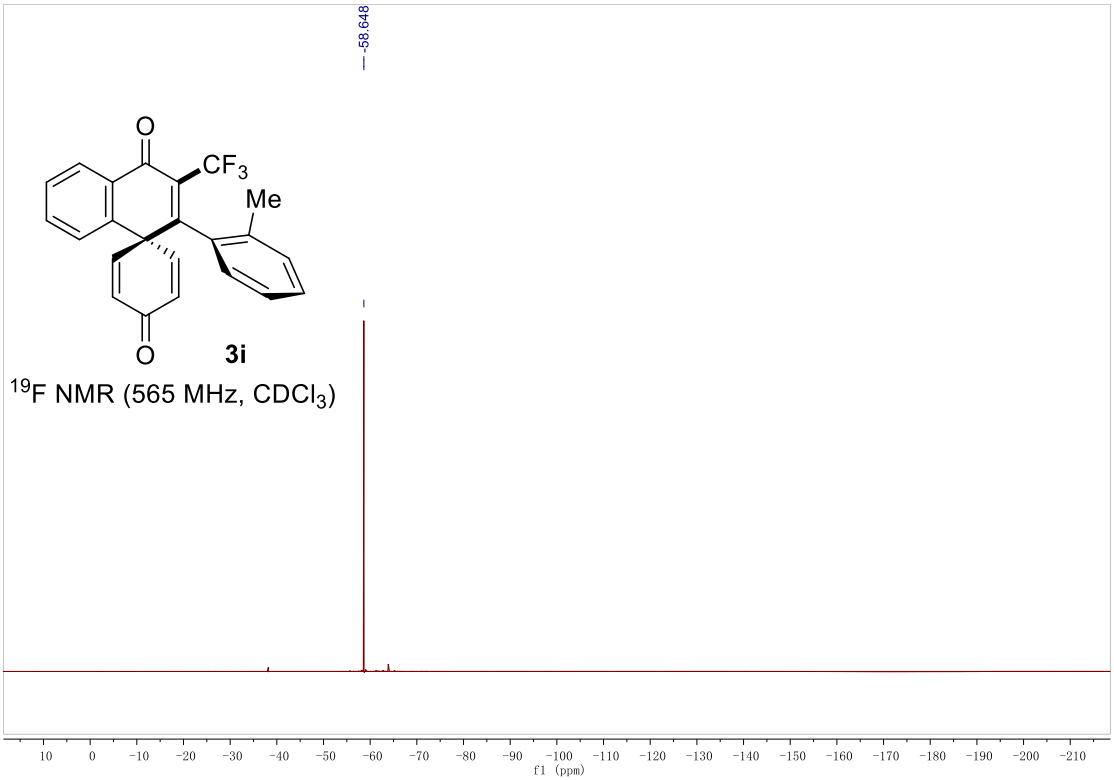
7.385
7.310
7.297
7.251
7.235
6.302
6.286
4.330
4.318
4.307
4.295
1.335
1.323
1.311
O
CF3
CO2Et
O
3h
1H NMR (600 MHz, DMSO-d6)
1.00
2.08
1.06
1.02
2.01
1.00
2.01
2.03
2.05
3.11
f1 (ppm)

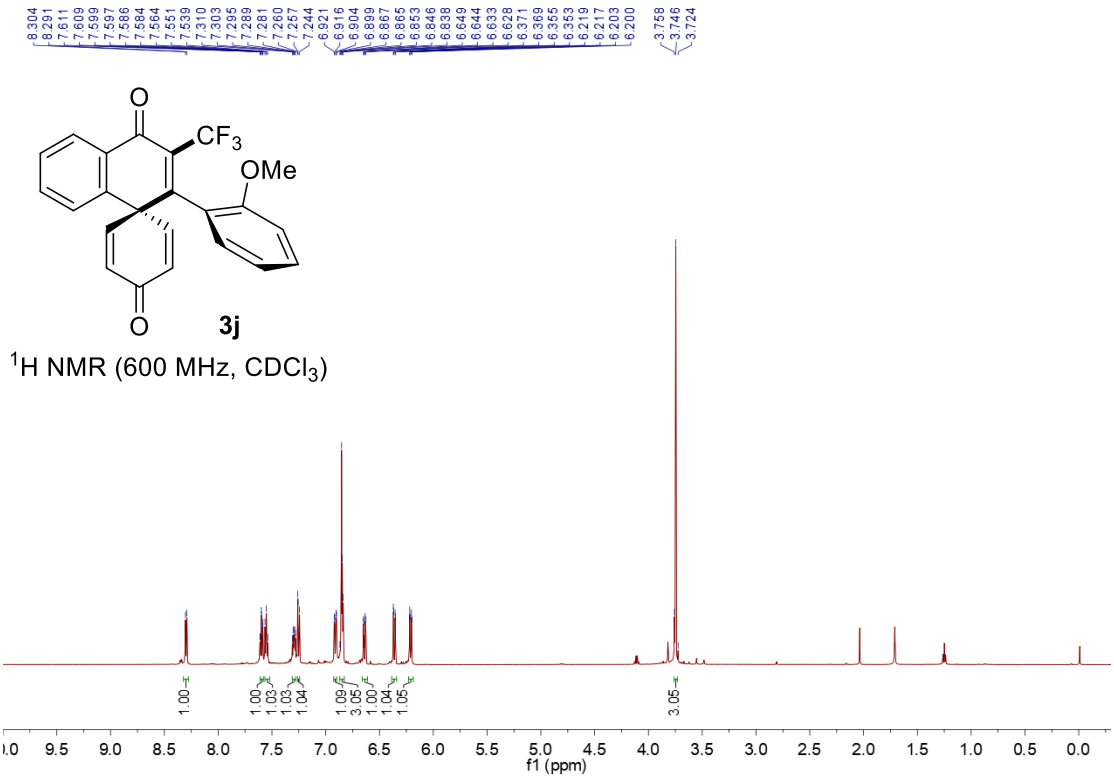

184.38
179.61
165.65
158.54
158.53
148.40
139.71
137.83
134.77
130.64
130.43
130.03
129.61
129.23
128.98
128.68
128.43
128.16
128.12
127.40
125.24
123.40
121.56
119.72
61.43
51.14
14.55
O
CF3
CO2Et
O
3h
13C NMR (151 MHz,DMSO-d6)
f1 (ppm)

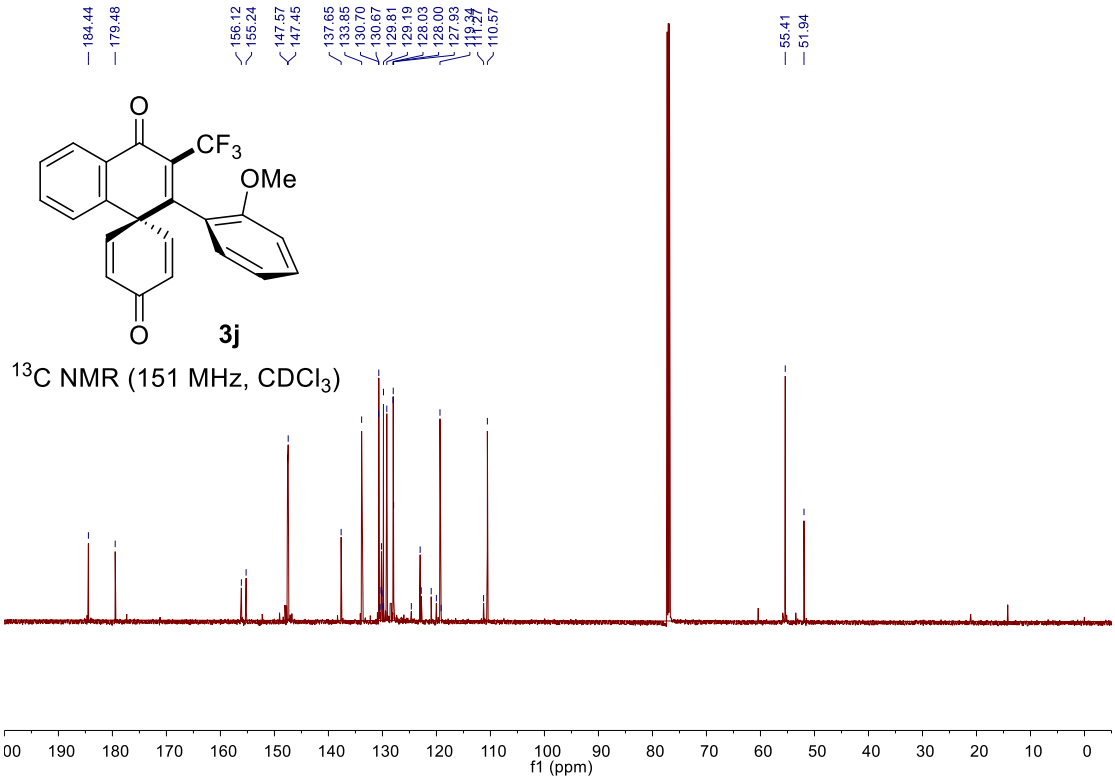

-55.431
O
CF3
CO2Et
O
3h
19F NMR (565 MHz, DMSO-d6)
f1 (ppm)

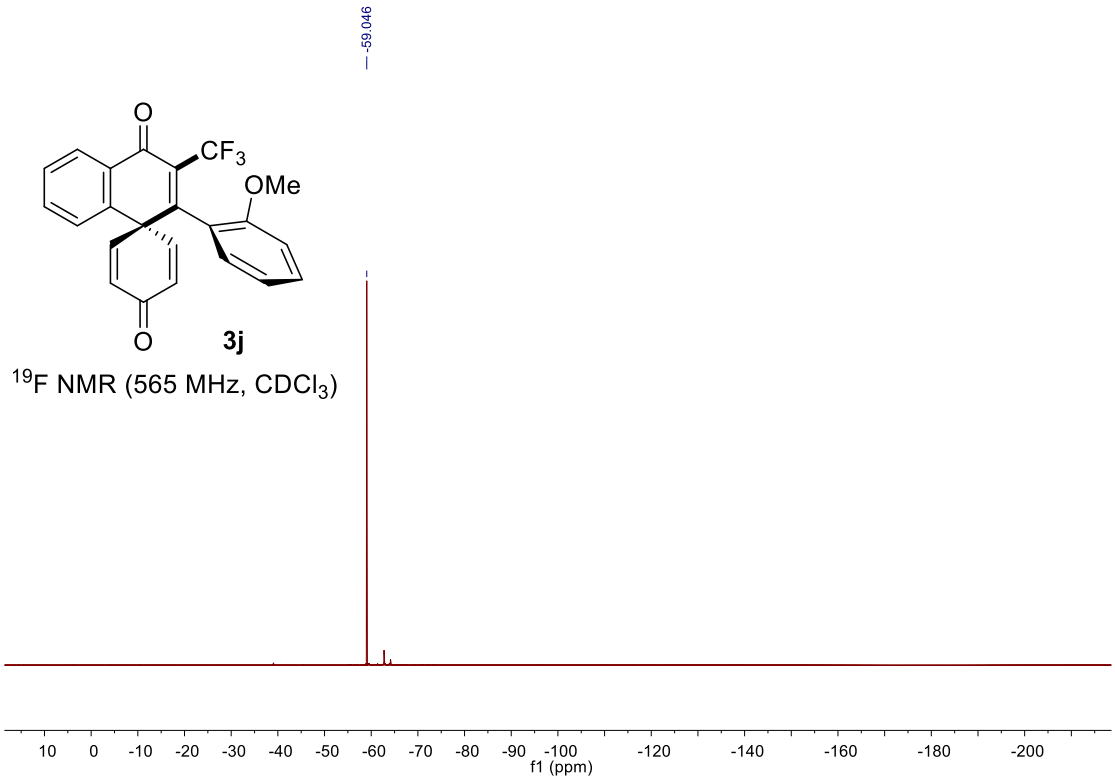

8.322
8.320
8.309
8.307
7.638
7.636
7.626
7.624
7.613
7.611
7.594
7.593
7.581
7.569
7.279
7.266
7.251
7.239
7.226
7.180
7.167
7.098
7.085
7.073
6.902
6.889
6.820
6.814
6.803
6.798
6.684
6.679
6.668
6.663
6.406
6.404
6.390
6.387
6.254
6.251
6.237
6.235
2.222
O
CF3
Me
O
3i
1H NMR (600 MHz, CDCl3)
1.00
2.15
1.20
1.10
1.03
1.10
1.02
1.05
1.02
1.03
1.00
3.00
f1 (ppm)

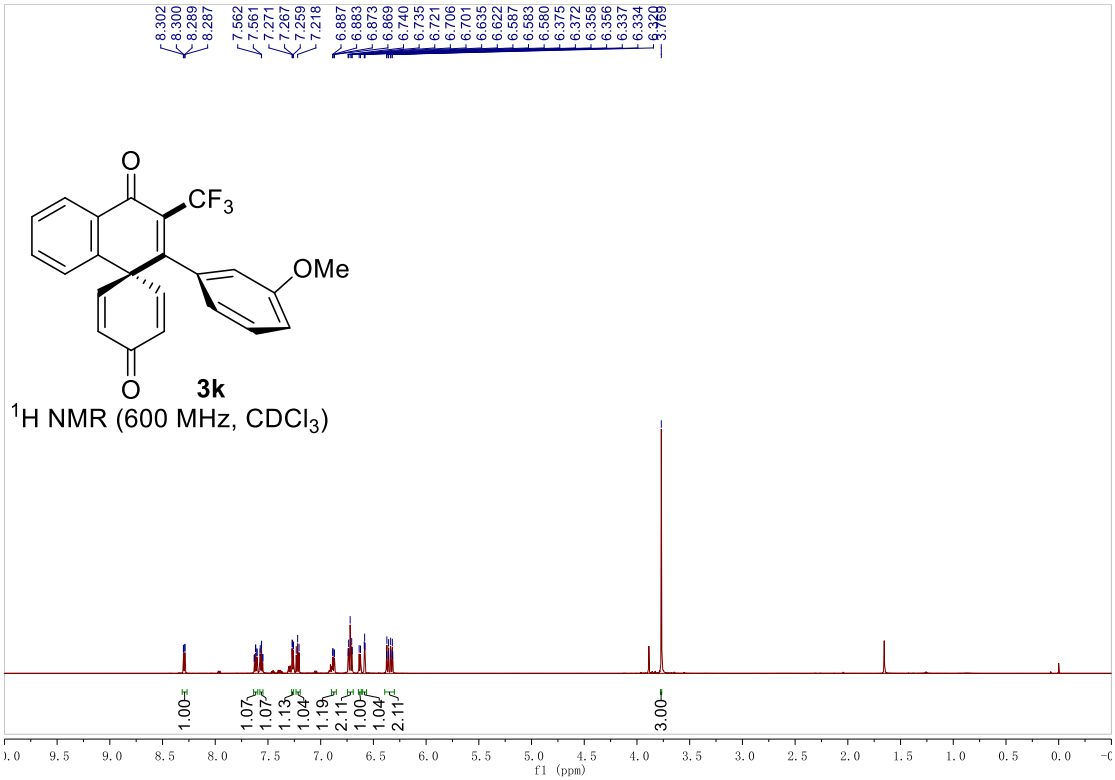

184.05
179.29
158.89
158.87
147.93
145.89
137.78
134.10
133.82
133.80
133.42
131.03
130.66
130.20
130.05
129.38
129.15
128.15
127.71
126.52
124.51
122.77
77.25
77.04
76.83
52.19
20.37
O
CF3
Me
O
3i
13C NMR (151 MHz, CDCl3)
f1 (ppm)

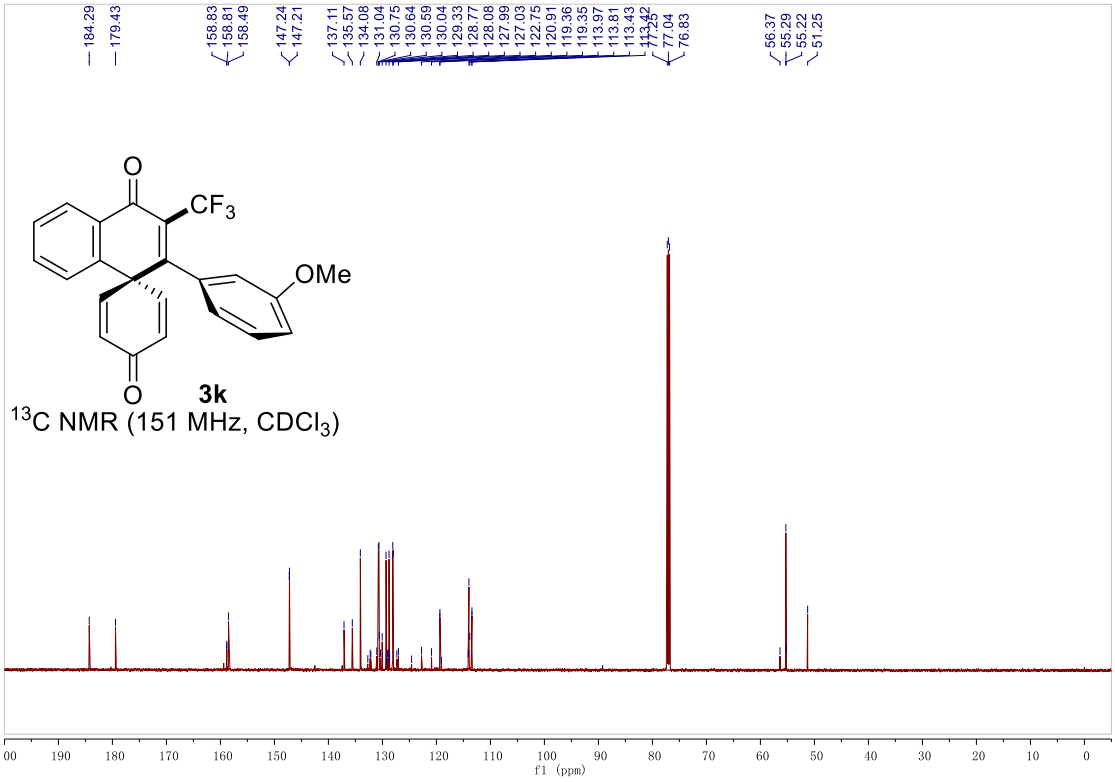

-58.648
O
CF3
Me
O
3i
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

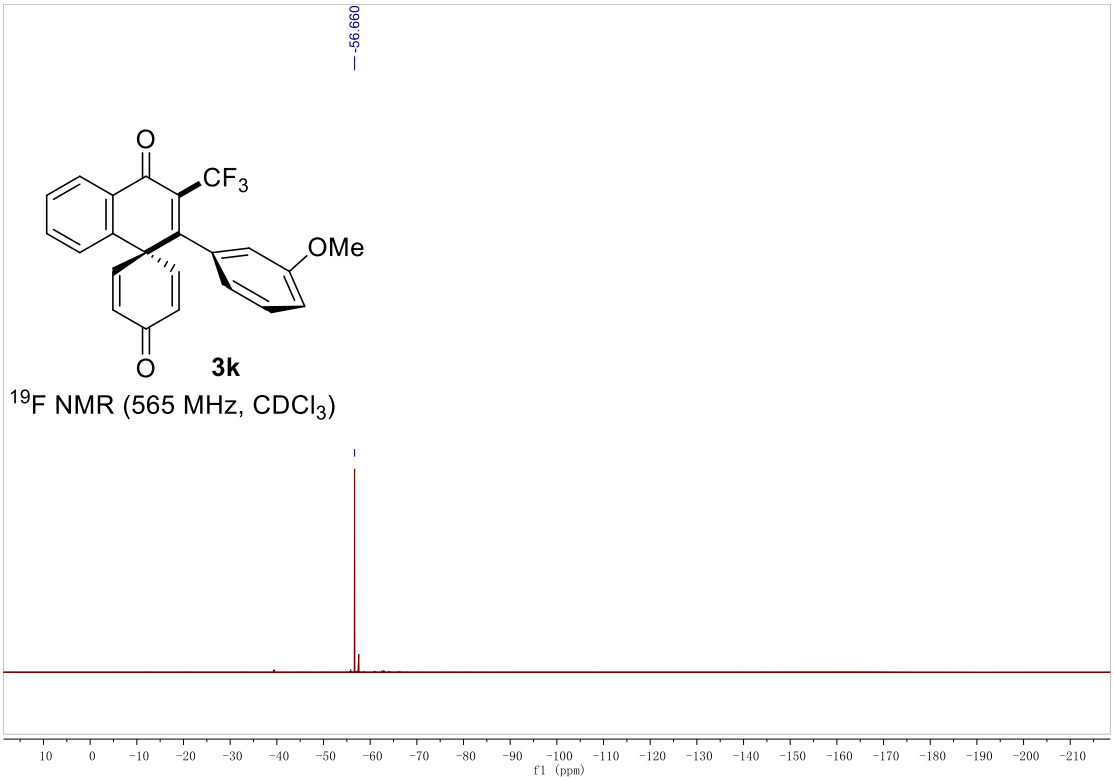

8.304
8.291
7.611
7.609
7.599
7.597
7.586
7.584
7.564
7.551
7.539
7.310
7.303
7.295
7.289
7.281
7.260
7.257
7.244
6.921
6.916
6.904
6.899
6.867
6.865
6.853
6.846
6.838
6.649
6.644
6.633
6.628
6.371
6.369
6.355
6.353
6.219
6.217
6.203
6.200
3.758
3.746
3.724
O
CF3
OMe
O
3j
1H NMR (600 MHz, CDCl3)
1.00
1.00
1.03
1.03
1.04
1.09
3.05
1.00
1.04
1.05
3.05
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 0.0
f1 (ppm)

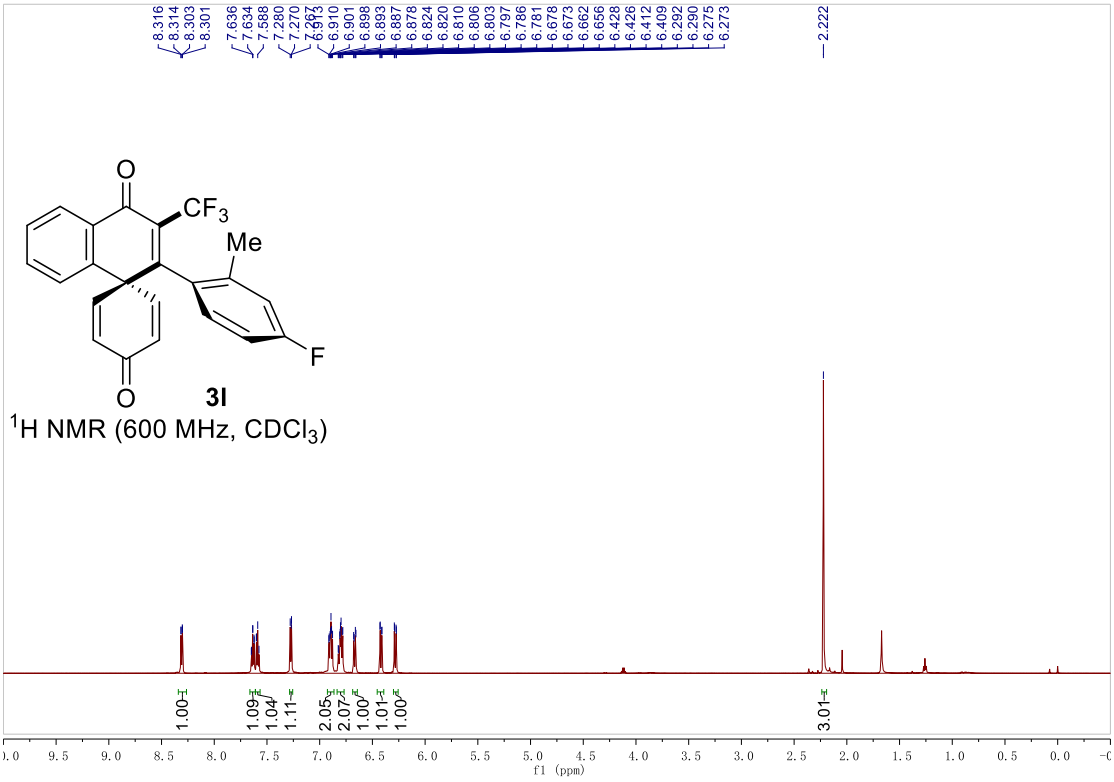

184.44
179.48
156.12
155.24
147.57
147.45
137.65
133.85
130.70
130.67
129.81
129.19
128.03
128.00
127.93
55.41
51.94
O
CF3
OMe
O
3j
13C NMR (151 MHz, CDCl3)
f1 (ppm)

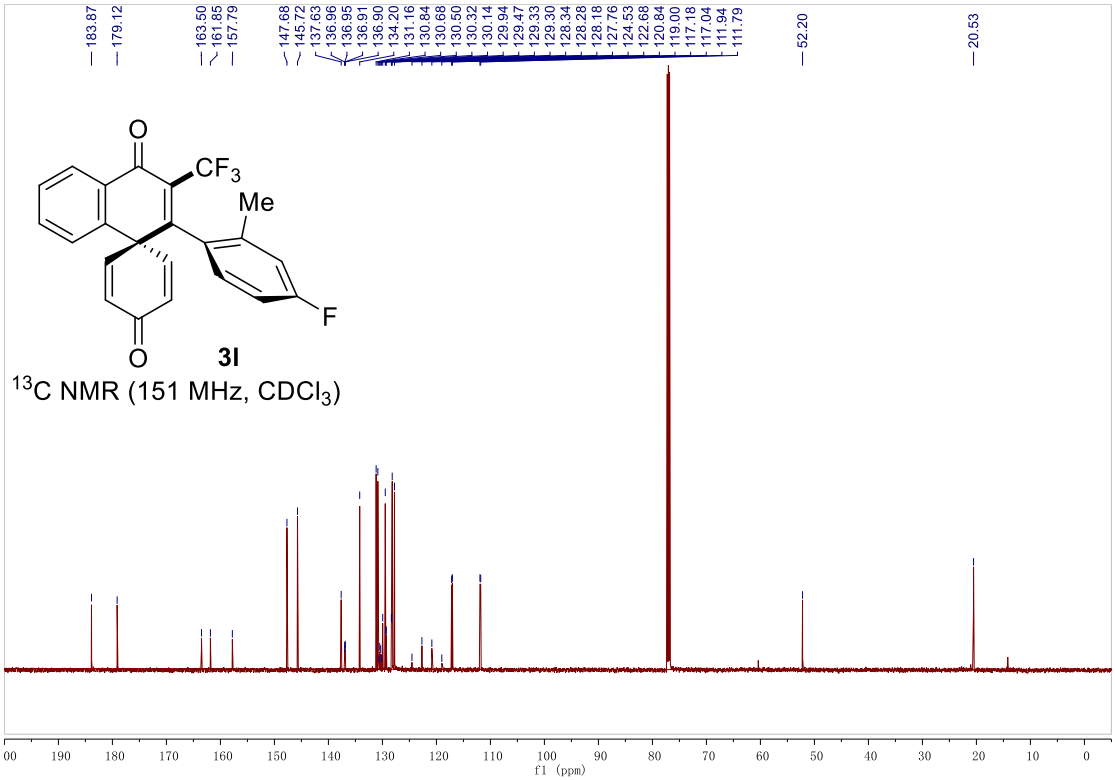

59.046
O
CF3
OMe
O
3j
19F NMR (565 MHz, CDCl3)
f1 (ppm)

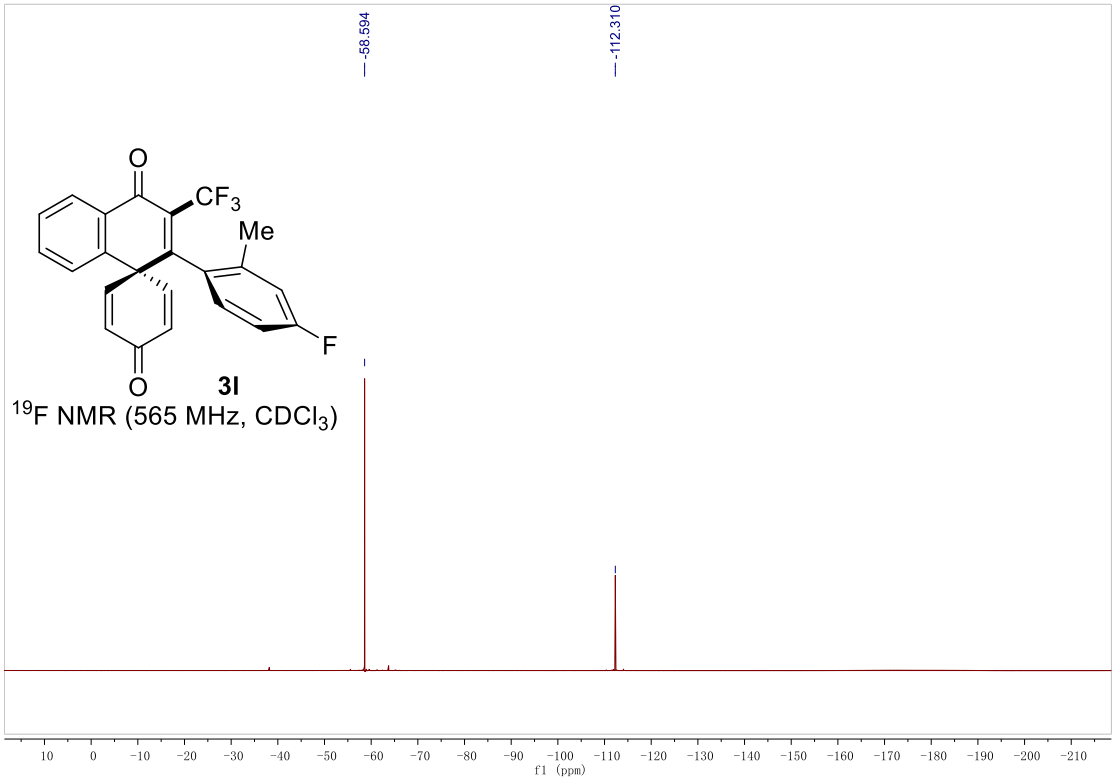

8.302
8.300
8.289
8.287
7.562
7.561
7.271
7.267
7.259
7.218
6.887
6.883
6.873
6.869
6.740
6.735
6.721
6.706
6.701
6.635
6.622
6.587
6.583
6.580
6.375
6.372
6.358
6.356
6.337
6.334
6.320
3.769
O
CF3
OMe
O
3k
1H NMR (600 MHz, CDCl3)
1.00
1.07
1.07
1.13
1.04
1.19
2.11
1.00
1.04
2.11
3.00
f1 (ppm)

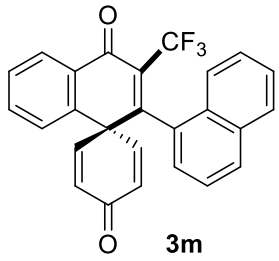

184.29
179.43
158.83
158.81
158.49
147.24
147.21
137.11
135.57
134.08
131.04
130.75
130.64
130.59
130.04
129.33
128.77
128.08
127.99
127.03
122.75
120.91
119.36
119.35
113.97
113.81
113.43
113.42
77.25
77.04
76.83
56.37
55.29
55.22
51.25
O
CF3
OMe
O
3k
13C NMR (151 MHz, CDCl3)
f1 (ppm)


-56.660
O
CF3
OMe
O
3k
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


8.316
8.314
8.303
8.301
7.636
7.634
7.588
7.280
7.270
6.910
6.901
6.898
6.893
6.887
6.878
6.824
6.820
6.810
6.806
6.803
6.797
6.786
6.781
6.678
6.673
6.662
6.656
6.428
6.426
6.412
6.409
6.292
6.290
6.275
6.273
2.222
O
CF3
Me
F
O
3l
1H NMR (600 MHz, CDCl3)
1.00
1.09
1.04
1.11
2.05
2.07
1.00
1.01
1.00
3.01
f1 (ppm)

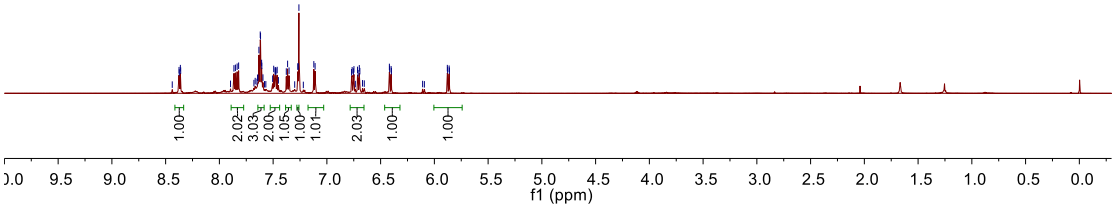

183.87
179.12
163.50
161.85
157.79
147.68
145.72
137.63
136.96
136.95
136.91
136.90
134.20
131.16
130.84
130.68
130.50
130.32
130.14
129.94
129.47
129.33
129.30
128.34
128.28
128.18
127.76
124.53
122.68
120.84
119.00
117.18
117.04
111.94
111.79
52.20
20.53
O
CF3
Me
F
O
3l
13C NMR (151 MHz, CDCl3)
f1 (ppm)


-58.594
-112.310
O
CF3
Me
F
O
3l
19F NMR (565 MHz, CDCl3)
f1 (ppm)



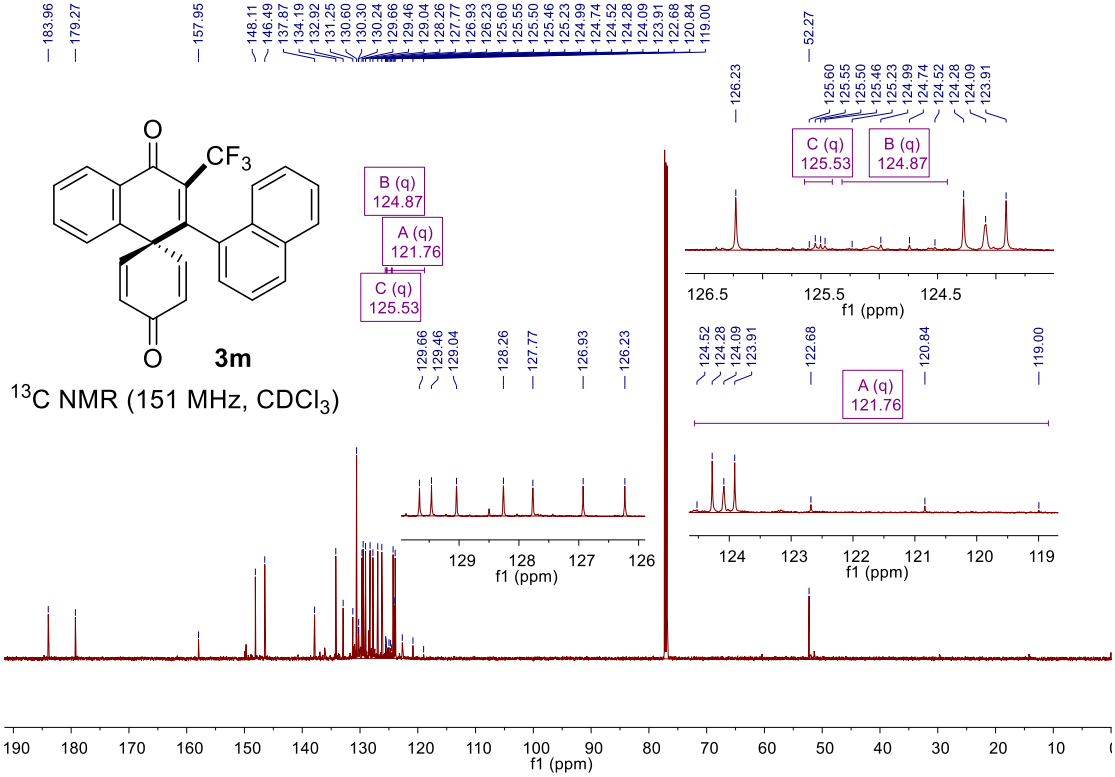
3m

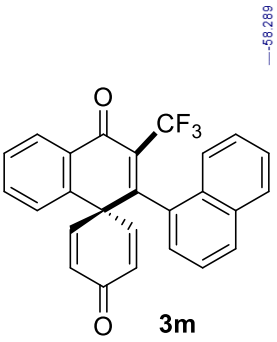
^{1}H NMR (600 MHz, $CDCl_3$)



3m

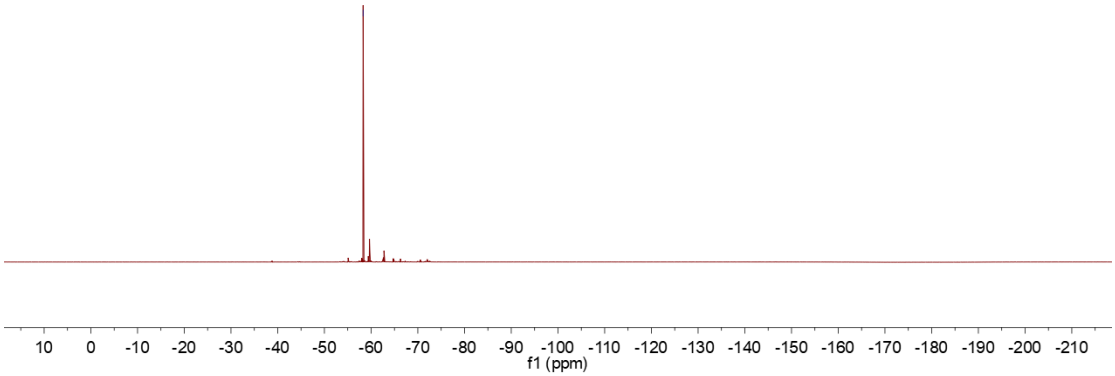
^{13}C NMR (151 MHz, $CDCl_3$)

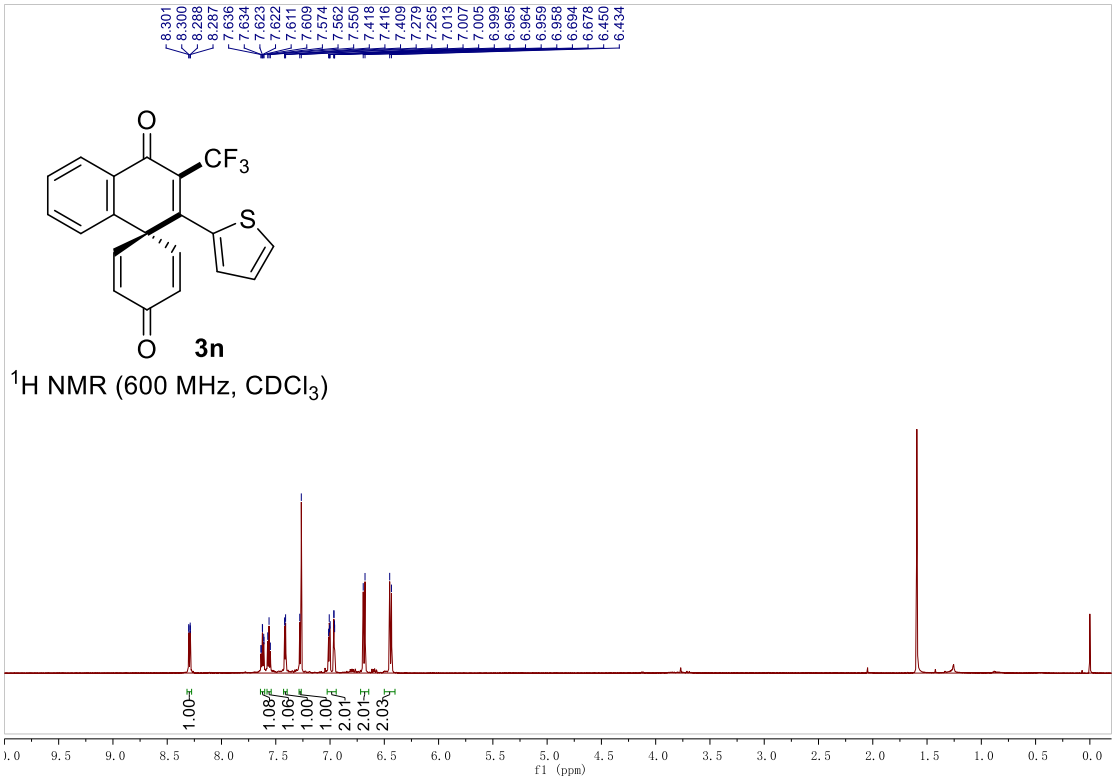




3m

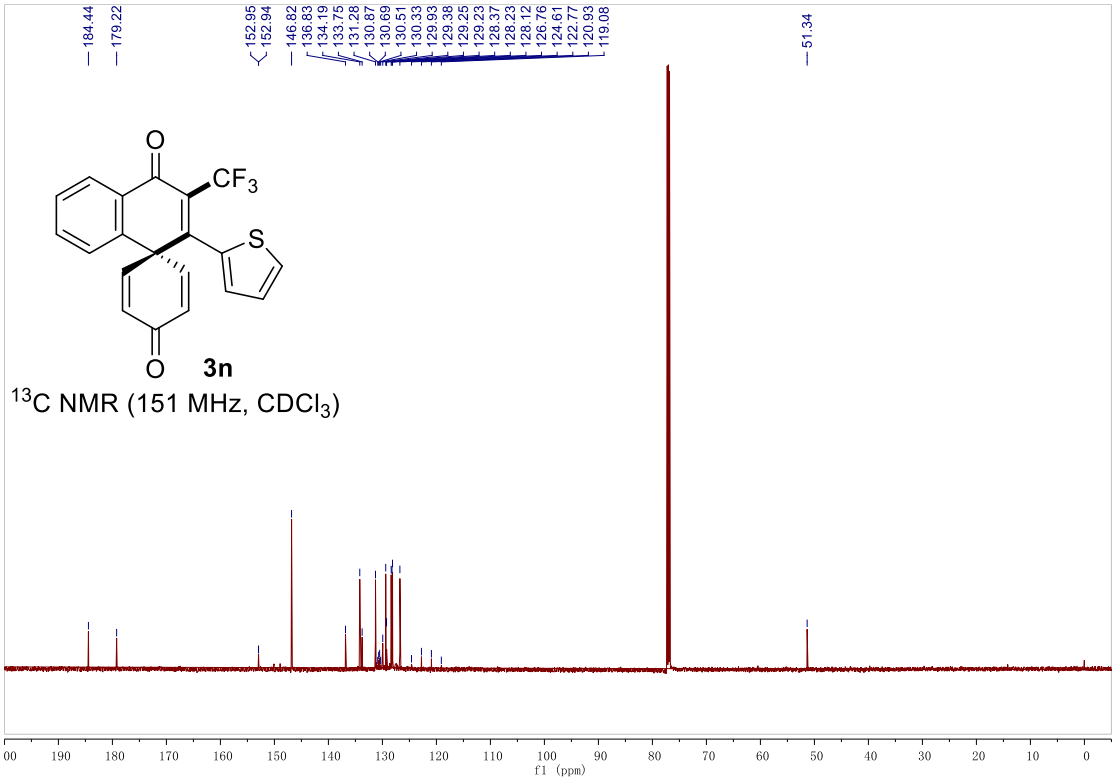
^{19}F NMR (565 MHz, $CDCl_3$)



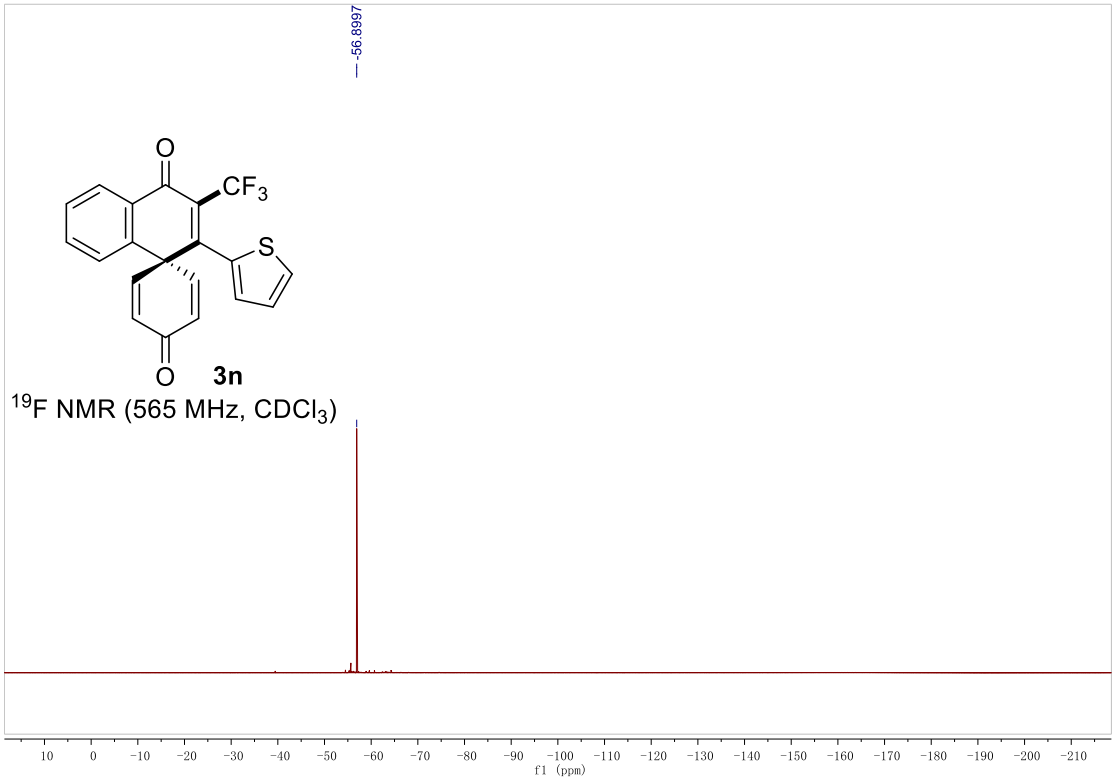


3n

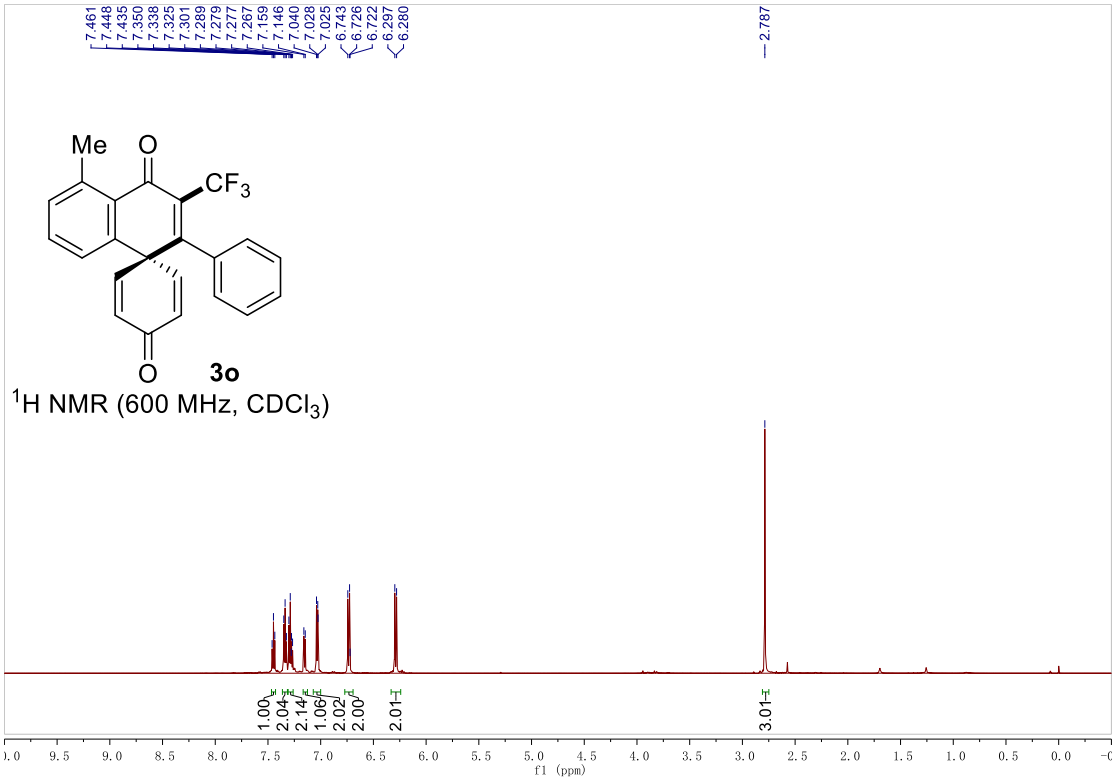
^{1}H NMR (600 MHz, $CDCl_3$)

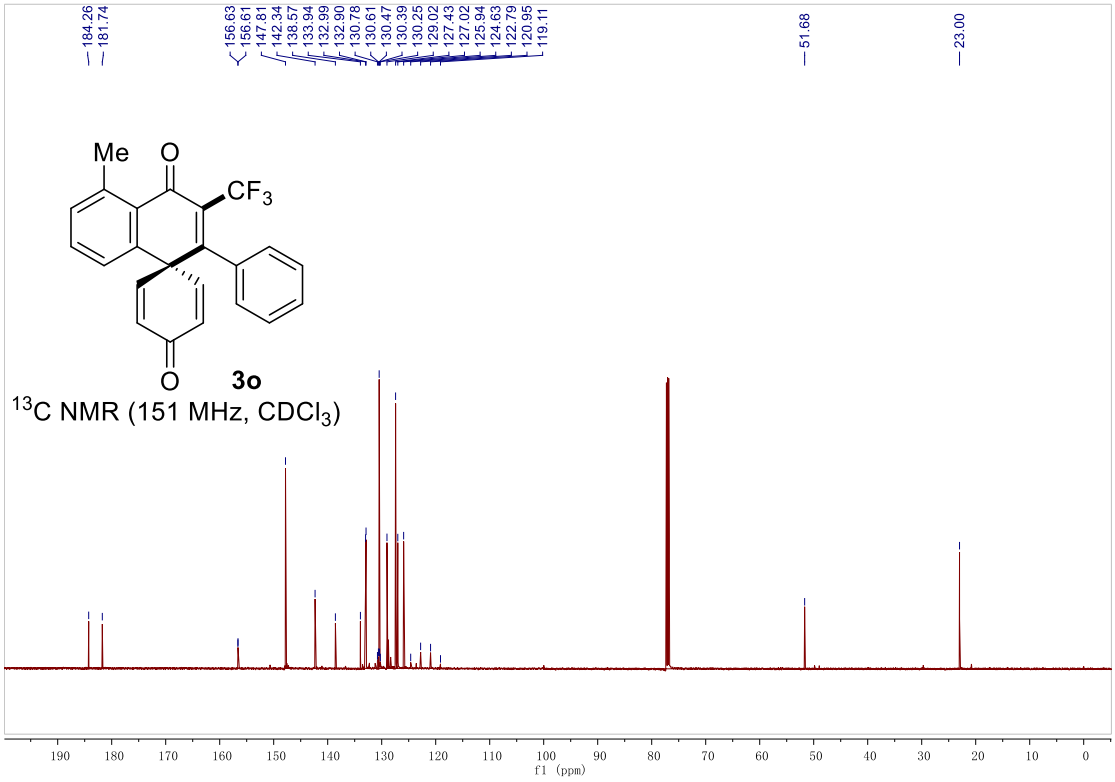


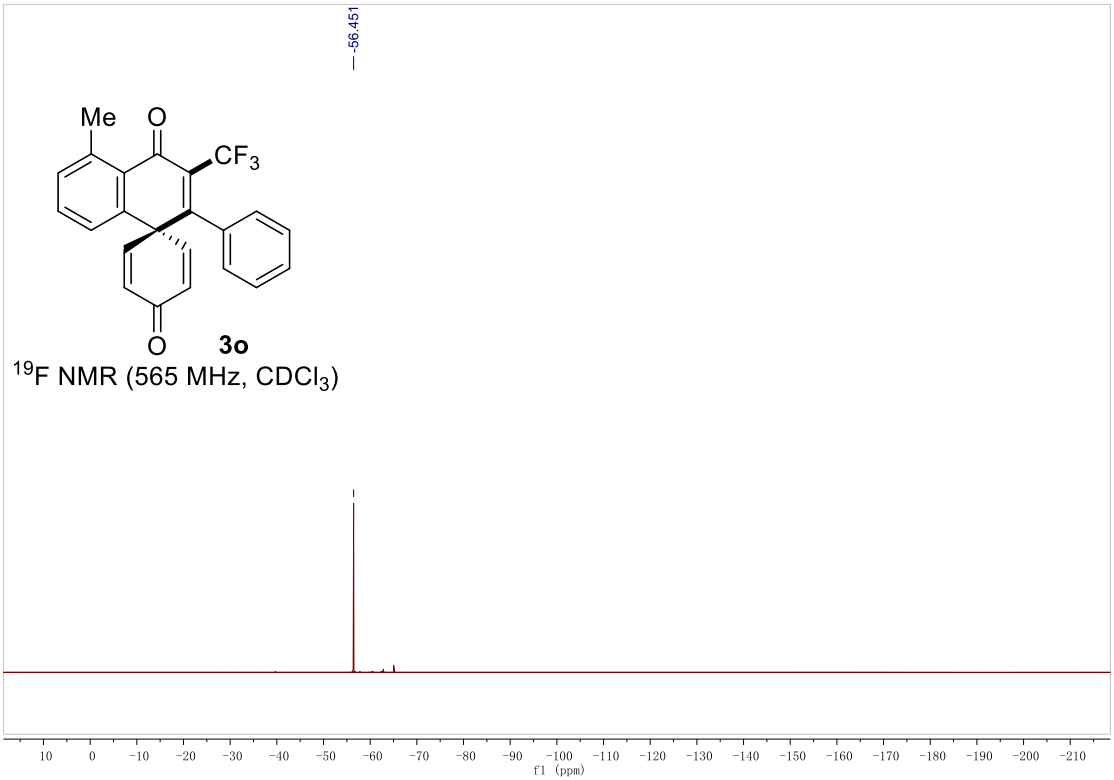
^{13}C NMR (151 MHz, $CDCl_3$)

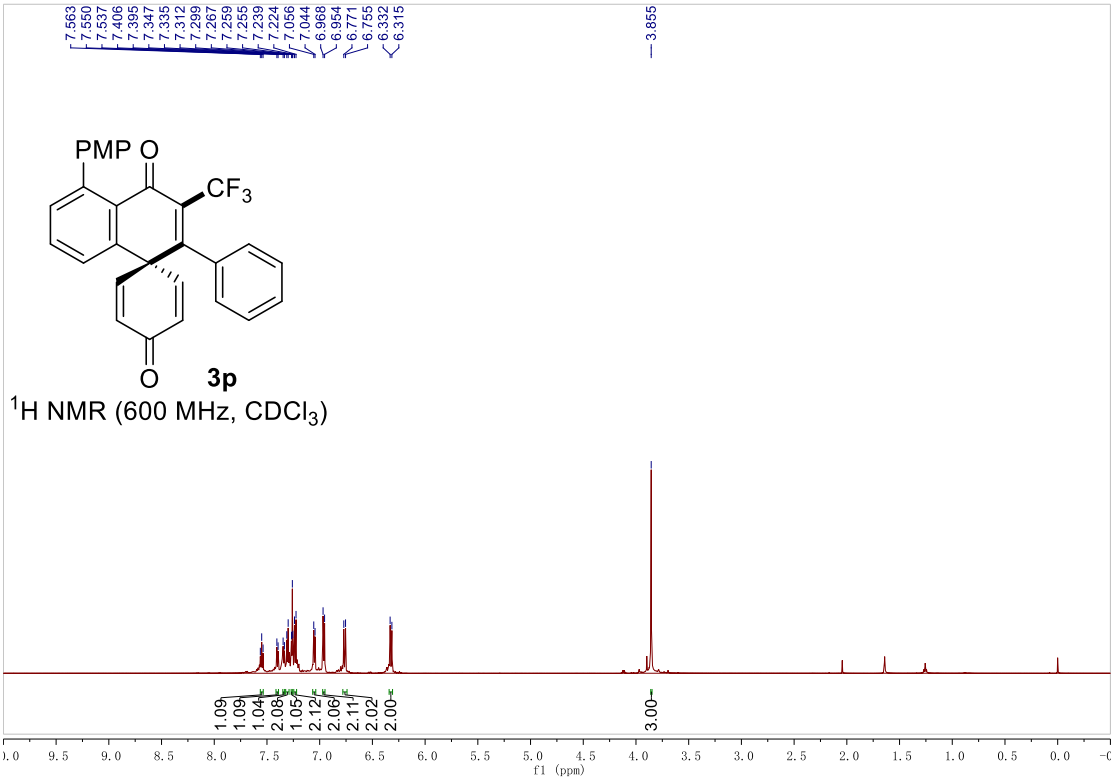


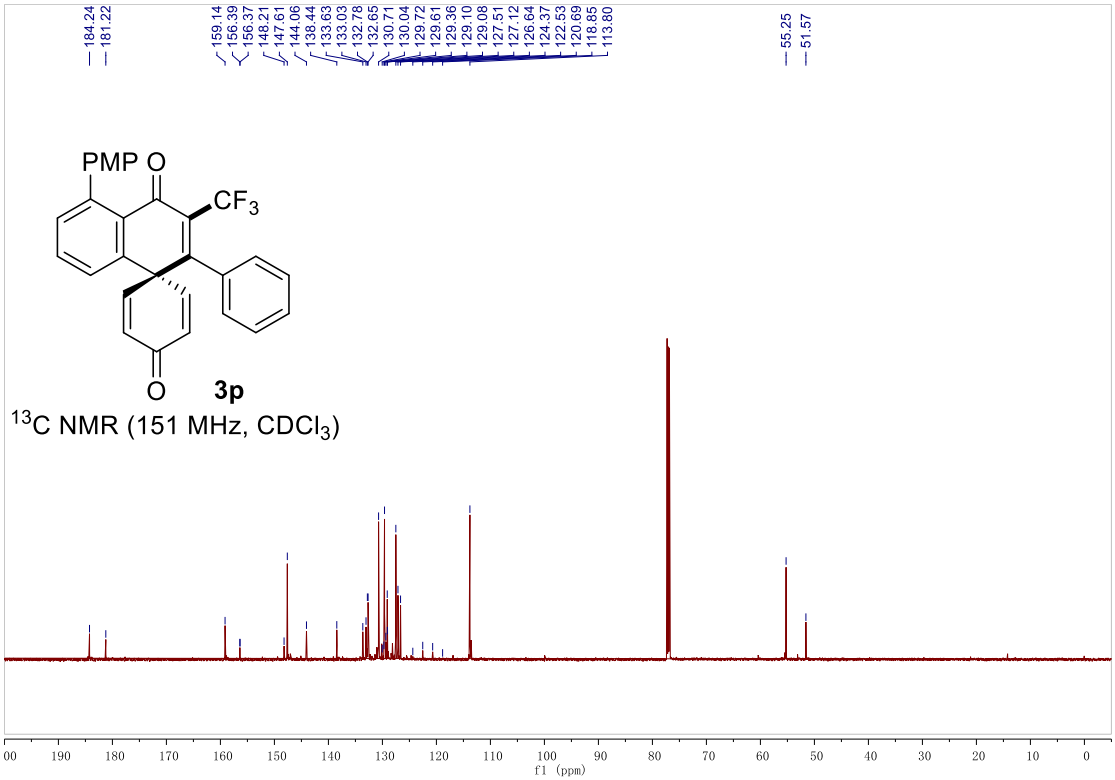
^{19}F NMR (565 MHz, $CDCl_3$)

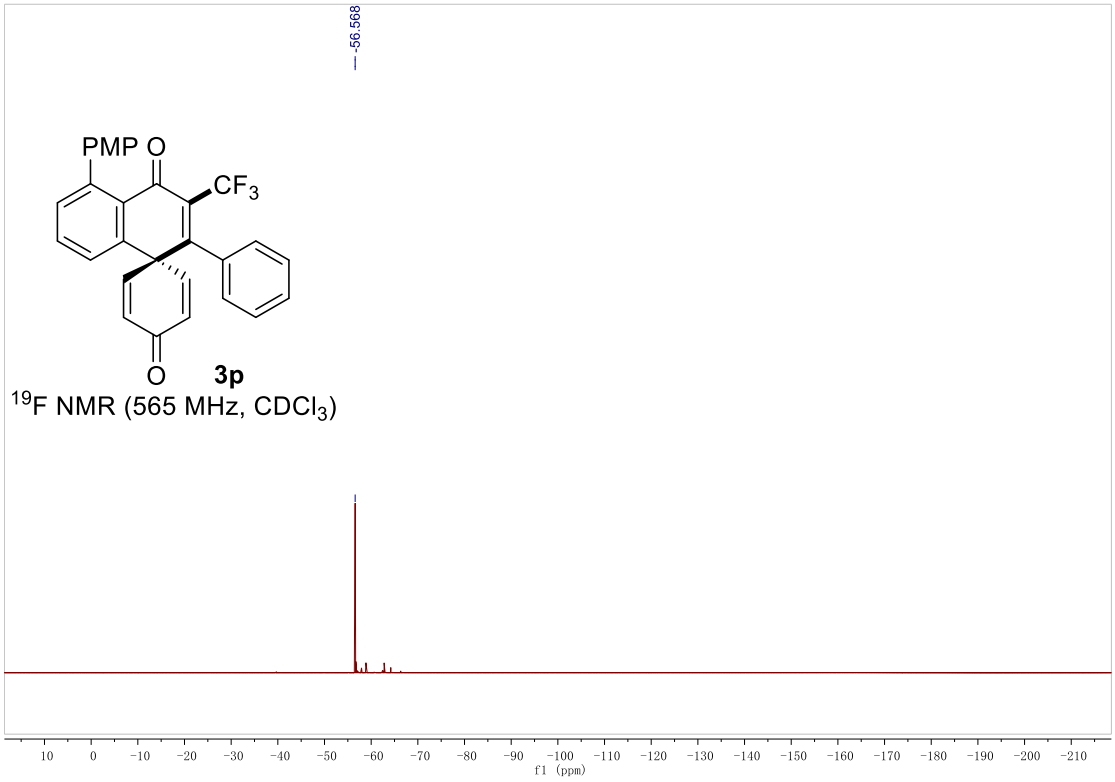

7.461
7.448
7.435
7.350
7.338
7.325
7.301
7.289
7.279
7.277
7.267
7.159
7.146
7.040
7.028
7.025
6.743
6.726
6.722
6.297
6.280
2.787
Me
O
CF3
O
3o
1H NMR (600 MHz, CDCl3)
1.00
2.04
2.14
1.06
2.02
2.00
2.01
3.01
f1 (ppm)

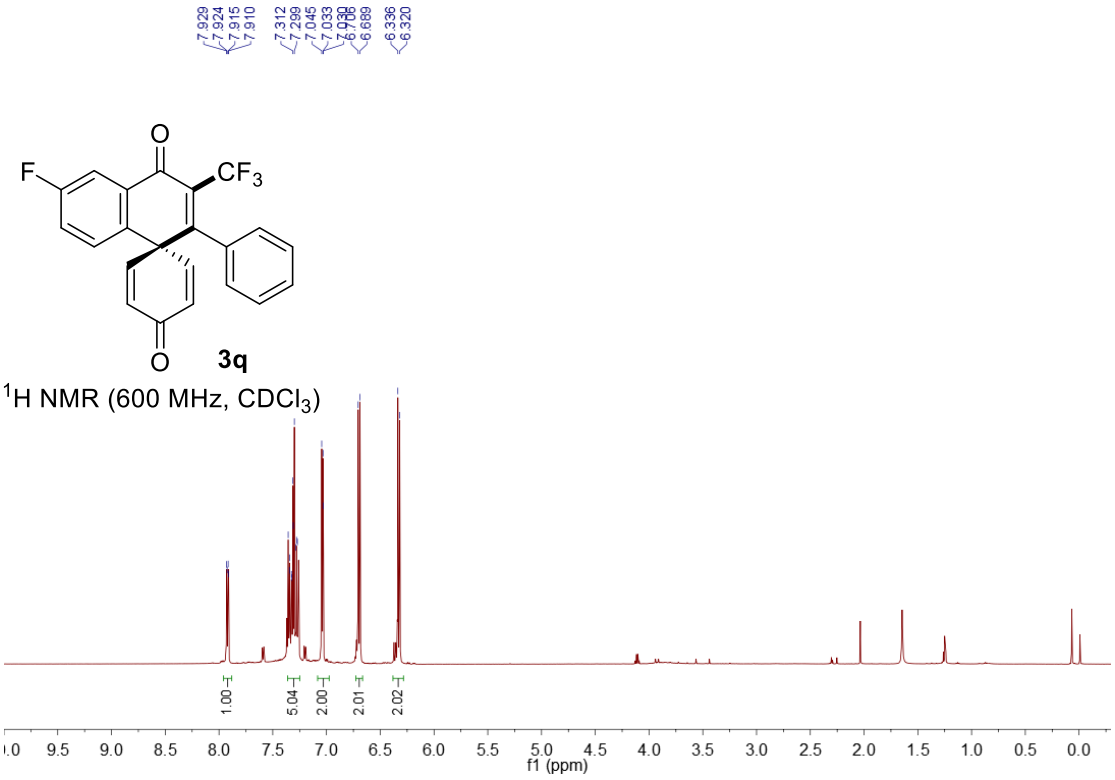

184.26
181.74
156.63
156.61
147.81
142.34
138.57
133.94
132.99
132.90
130.78
130.61
130.47
130.39
130.25
129.02
127.43
127.02
125.94
124.63
122.79
120.95
119.11
51.68
23.00
Me
O
CF3
O
3o
13C NMR (151 MHz, CDCl3)
f1 (ppm)

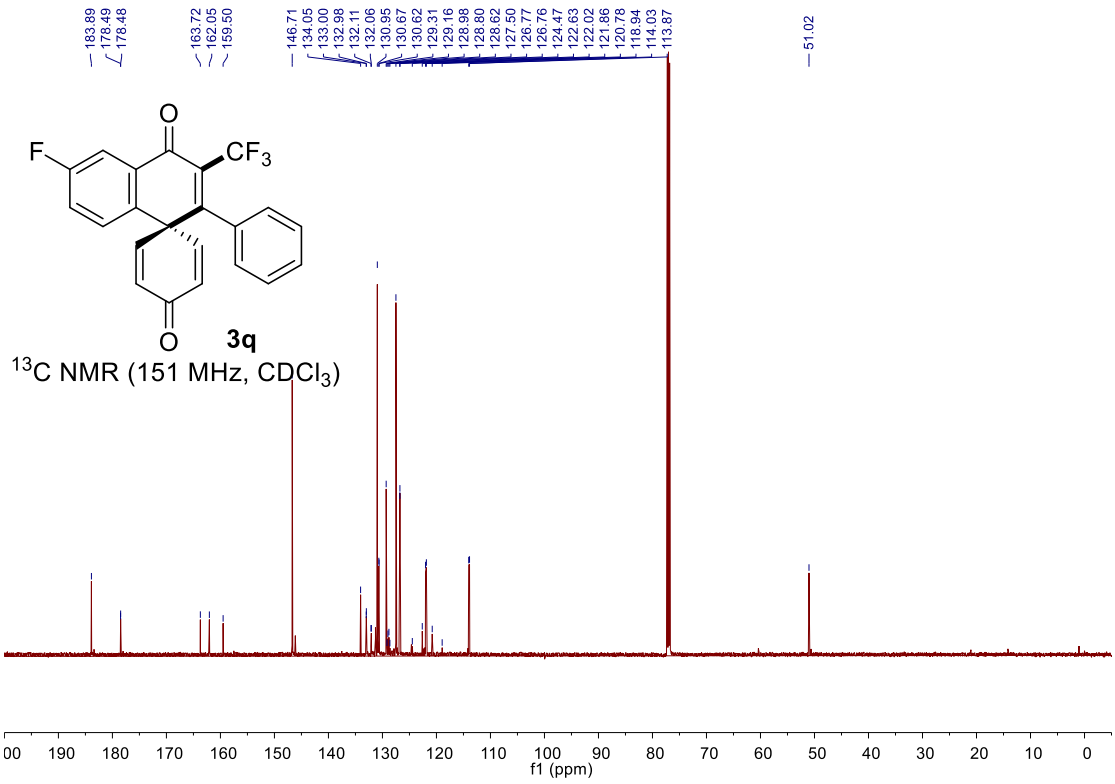

-56.451
Me
O
CF3
O
3o
19F NMR (565 MHz, CDCl3)
f1 (ppm)

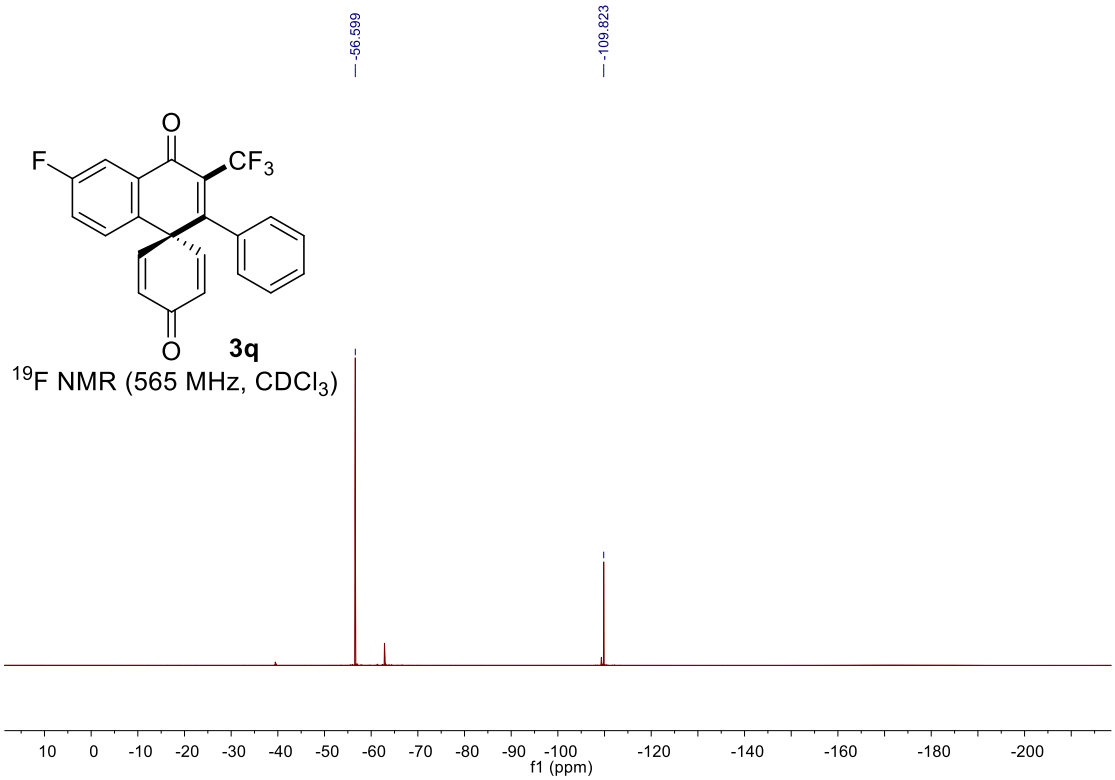

7.563
7.550
7.537
7.406
7.395
7.347
7.335
7.312
7.299
7.267
7.259
7.255
7.239
7.224
7.056
7.044
6.968
6.954
6.771
6.755
6.332
6.315
3.855
PMP O
CF3
O
3p
1H NMR (600 MHz, CDCl3)
1.09
1.09
1.04
2.08
1.05
2.12
2.06
2.11
2.02
2.00
3.00
f1 (ppm)

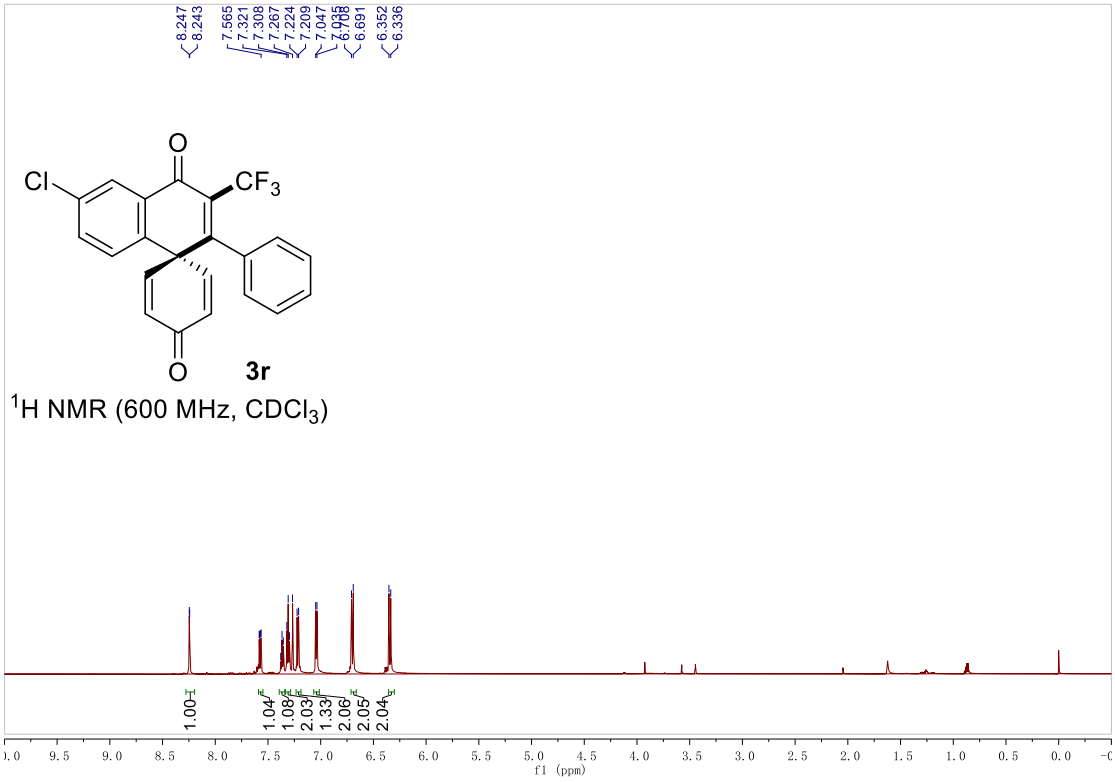

184.24
181.22
159.14
156.39
156.37
148.21
147.61
144.06
138.44
133.63
133.03
132.78
132.65
130.71
130.04
129.72
129.61
129.36
129.10
129.08
127.51
127.12
126.64
124.37
122.53
120.69
118.85
113.80
55.25
51.57
PMP O
CF3
O
3p
13C NMR (151 MHz, CDCl3)
f1 (ppm)

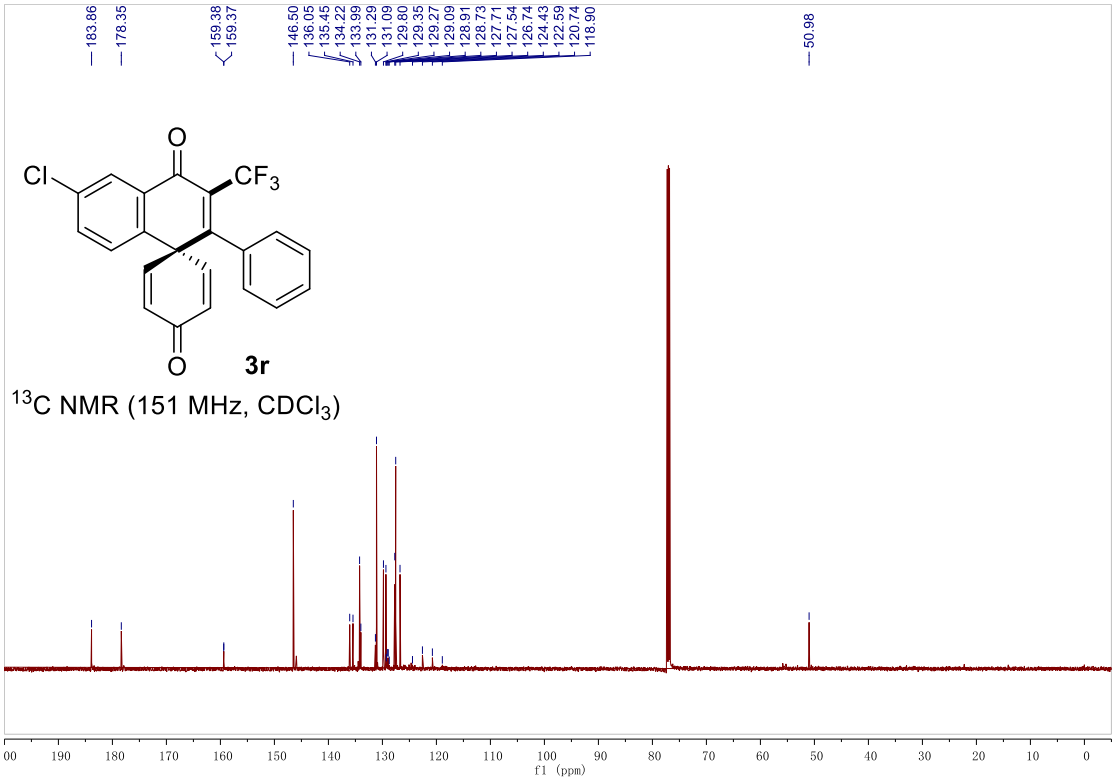

-56.568
PMP O
CF3
O
3p
19F NMR (565 MHz, CDCl3)
f1 (ppm)

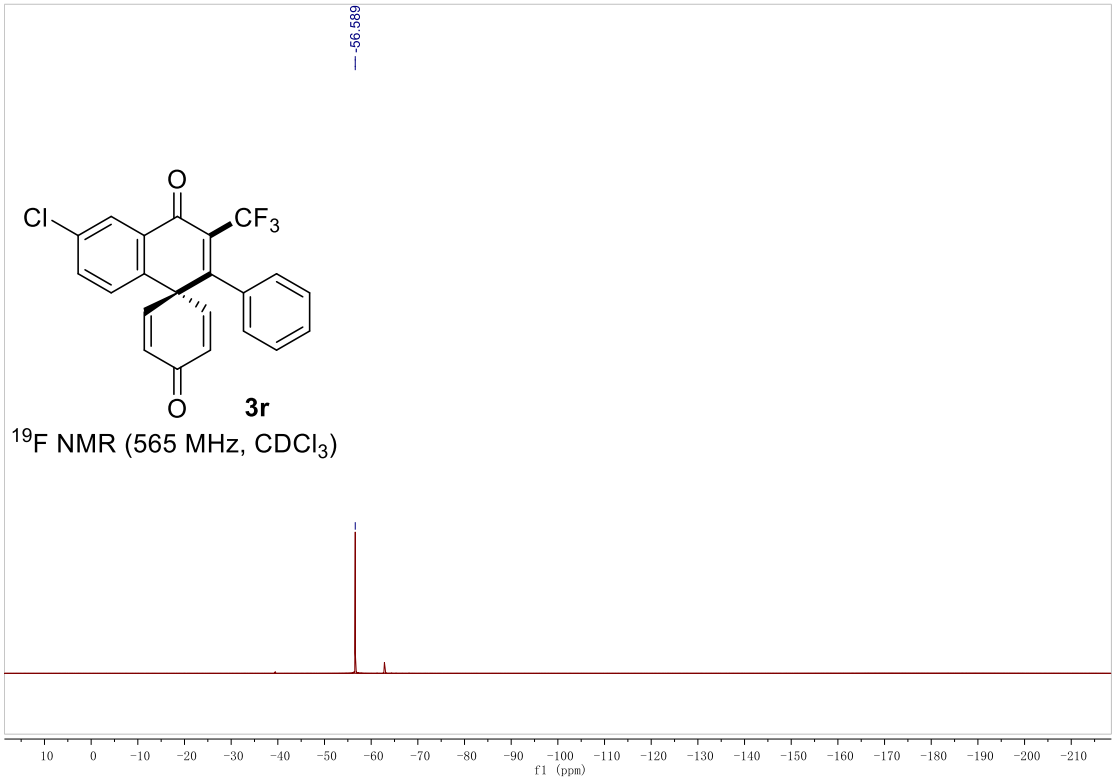

7.929
7.924
7.915
7.910
7.312
7.299
7.045
7.033
6.689
6.336
6.320
F
CF3
O
O
3q
1H NMR (600 MHz, CDCl3)
1.00
5.04
2.00
2.01
2.02
f1 (ppm)

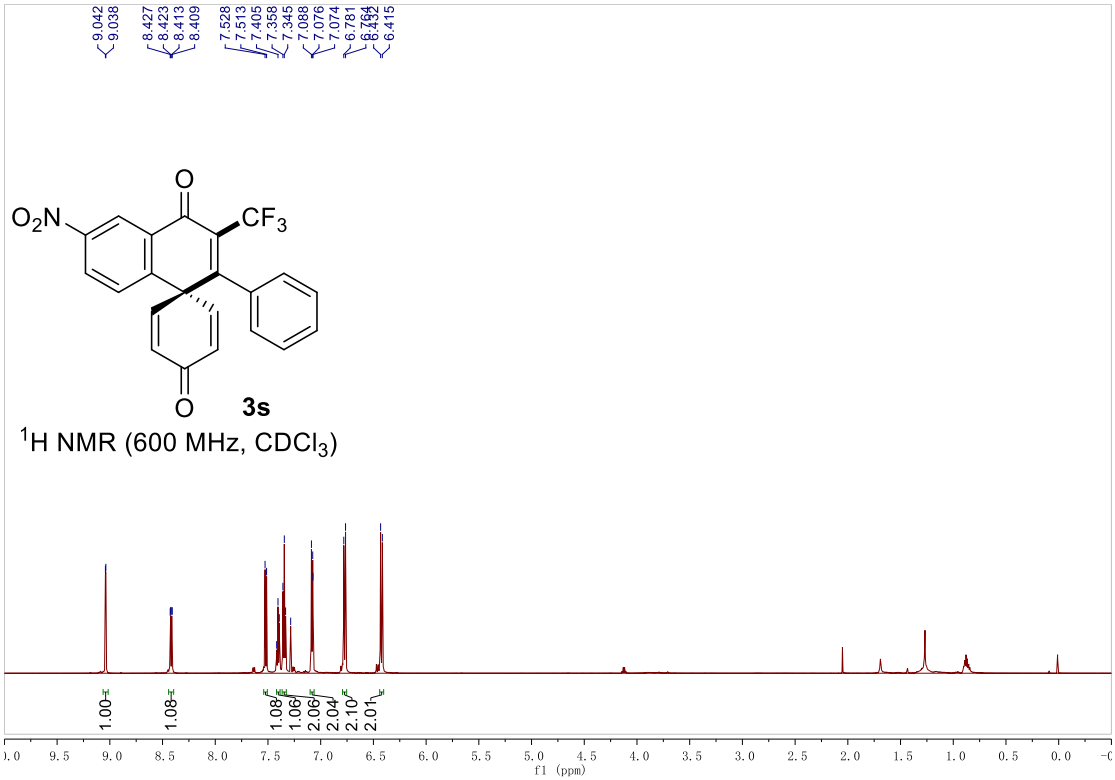

183.89
178.49
178.48
163.72
162.05
159.50
146.71
134.05
133.00
132.98
132.11
132.06
130.95
130.67
130.62
129.31
129.16
128.98
128.80
128.62
127.50
126.77
126.76
124.47
122.63
122.02
121.86
120.78
118.94
114.03
113.87
51.02
F
CF3
O
O
3q
13C NMR (151 MHz, CDCl3)
f1 (ppm)

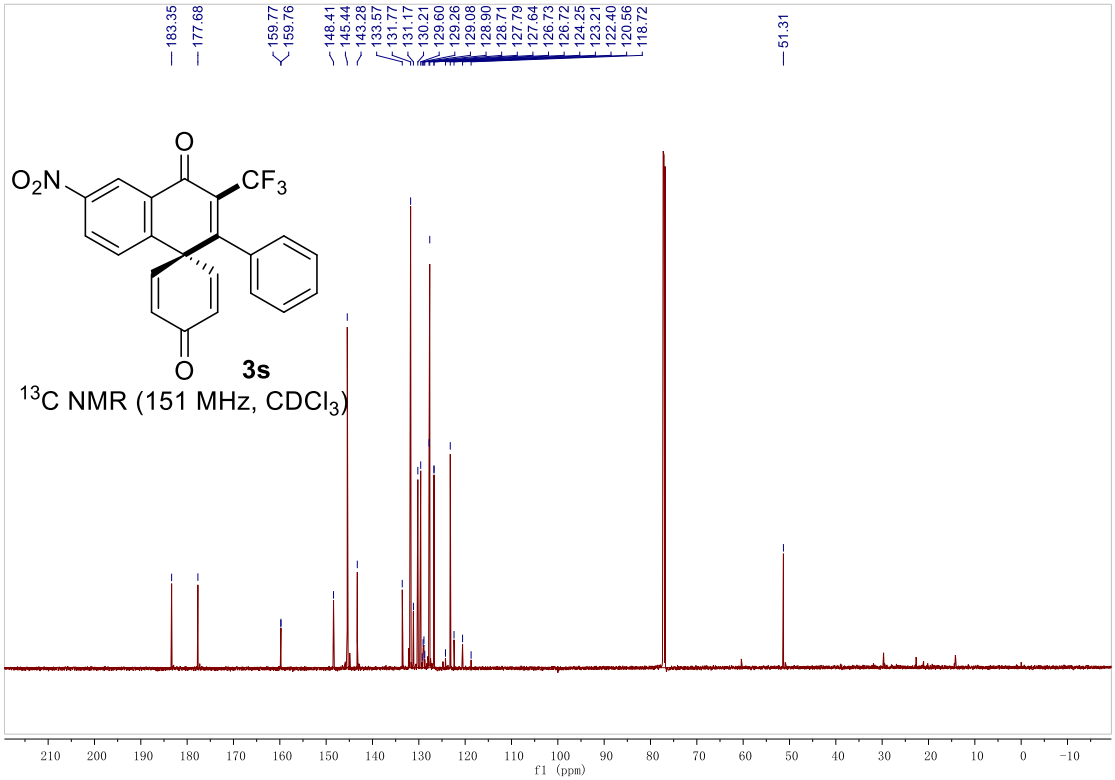

-56.599
-109.823
F
CF3
O
O
3q
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)

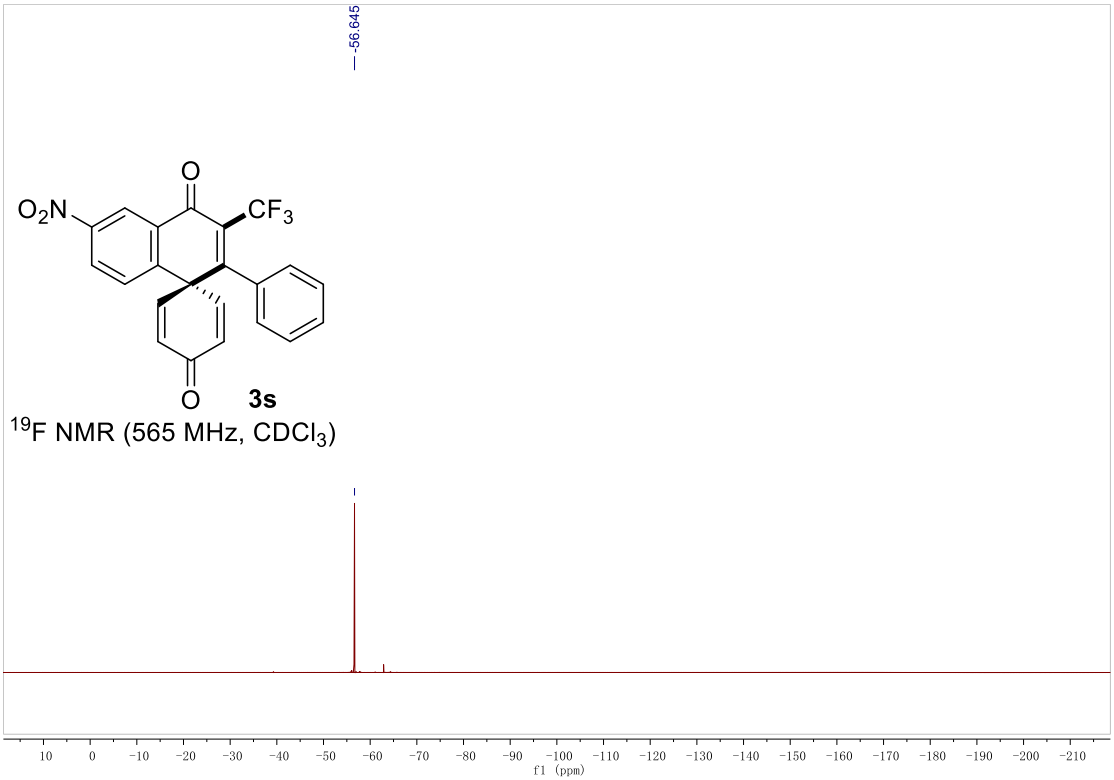

8.247
8.243
7.565
7.321
7.308
7.267
7.224
7.209
7.047
7.035
7.008
6.691
6.352
6.336
Cl
CF3
O
O
3r
1H NMR (600 MHz, CDCl3)
1.00
1.04
1.08
2.03
1.33
2.06
2.05
2.04
f1 (ppm)

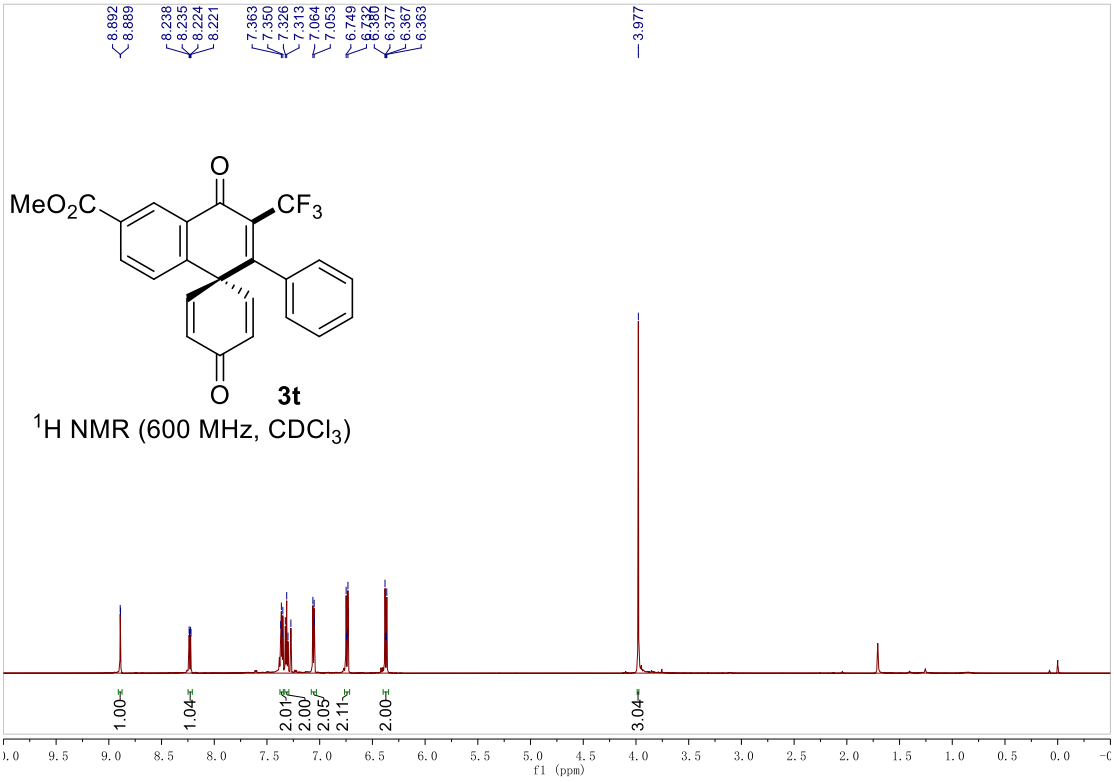

183.86
178.35
159.38
159.37
146.50
136.05
135.45
134.22
133.99
131.29
131.09
129.80
129.35
129.27
129.09
128.91
128.73
127.71
127.54
126.74
124.43
122.59
120.74
118.90
50.98
Cl
CF3
O
O
3r
13C NMR (151 MHz, CDCl3)
f1 (ppm)

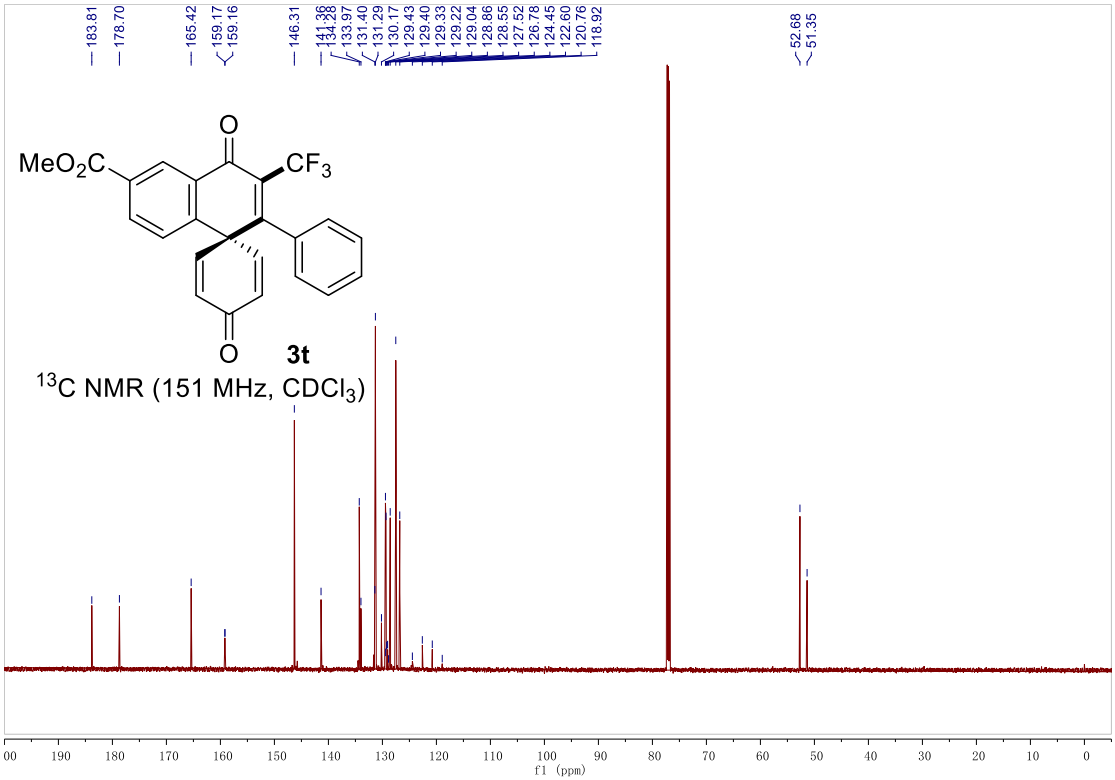

-56.589
Cl
CF3
O
O
3r
19F NMR (565 MHz, CDCl3)
f1 (ppm)

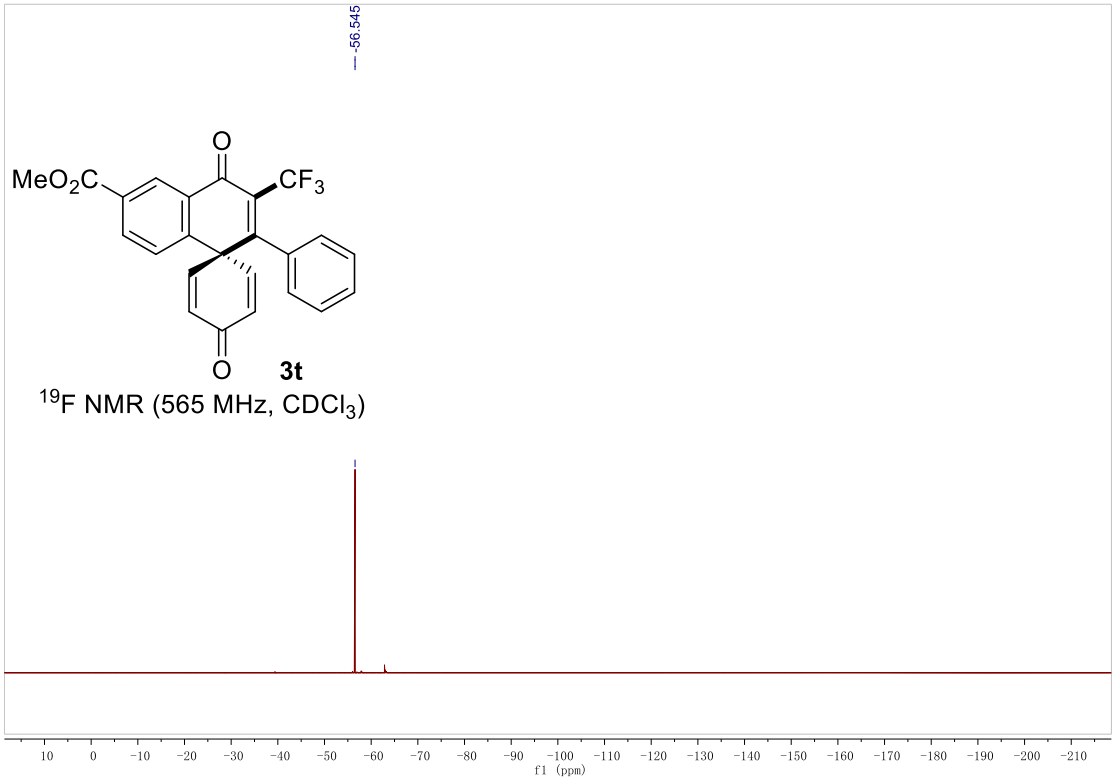

9.042
9.038
8.427
8.423
8.413
8.409
7.528
7.513
7.405
7.358
7.345
7.088
7.076
7.074
6.781
6.415
O
O2N
CF3
O
3s
1H NMR (600 MHz, CDCl3)
1.00
1.08
1.08
1.06
2.06
2.04
2.10
2.01
f1 (ppm)


183.35
177.68
159.77
159.76
148.41
145.44
143.28
133.57
131.77
131.17
130.21
129.60
129.26
129.08
128.90
128.71
127.79
127.64
126.73
126.72
124.25
123.21
122.40
120.56
118.72
51.31
O
O2N
CF3
O
3s
13C NMR (151 MHz, CDCl3)
f1 (ppm)


-56.645
O
O2N
CF3
O
3s
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


8.892
8.889
8.238
8.235
8.224
8.221
7.363
7.350
7.326
7.313
7.064
7.053
6.749
6.377
6.367
6.363
3.977
MeO2C
CF3
O
3t
1H NMR (600 MHz, CDCl3)
1.00
1.04
2.01
2.00
2.05
2.11
2.00
3.04
f1 (ppm)


183.81
178.70
165.42
159.17
159.16
146.31
141.36
134.28
133.97
131.40
131.29
130.17
129.43
129.40
129.33
129.22
129.04
128.86
128.55
127.52
126.78
124.45
122.60
120.76
118.92
52.68
51.35
MeO2C
CF3
O
O
3t
13C NMR (151 MHz, CDCl3)
f1 (ppm)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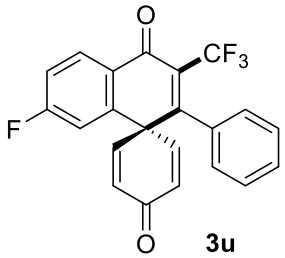
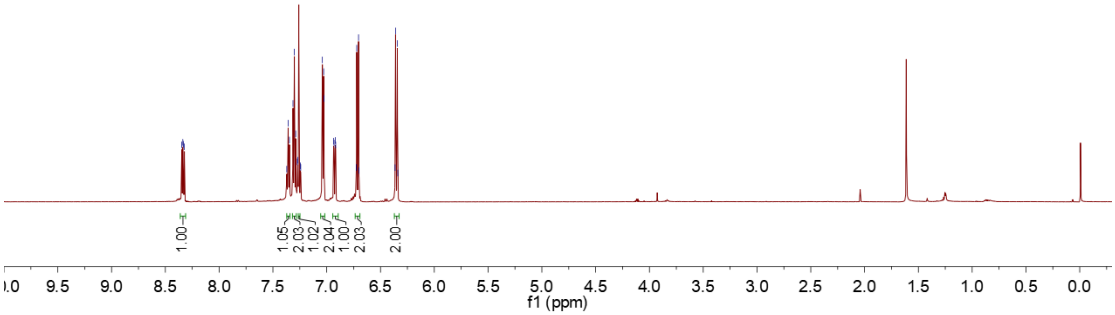
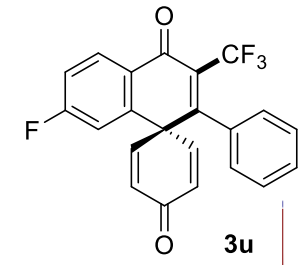
MeO2C
CF3
O
O
3t
19F NMR (565 MHz, CDCl3)
f1 (ppm)



3u

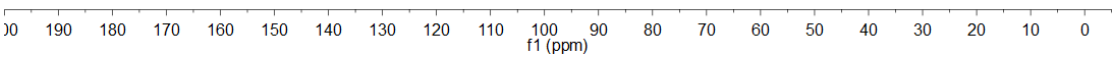
^{1}H NMR (600 MHz, $CDCl_3$)

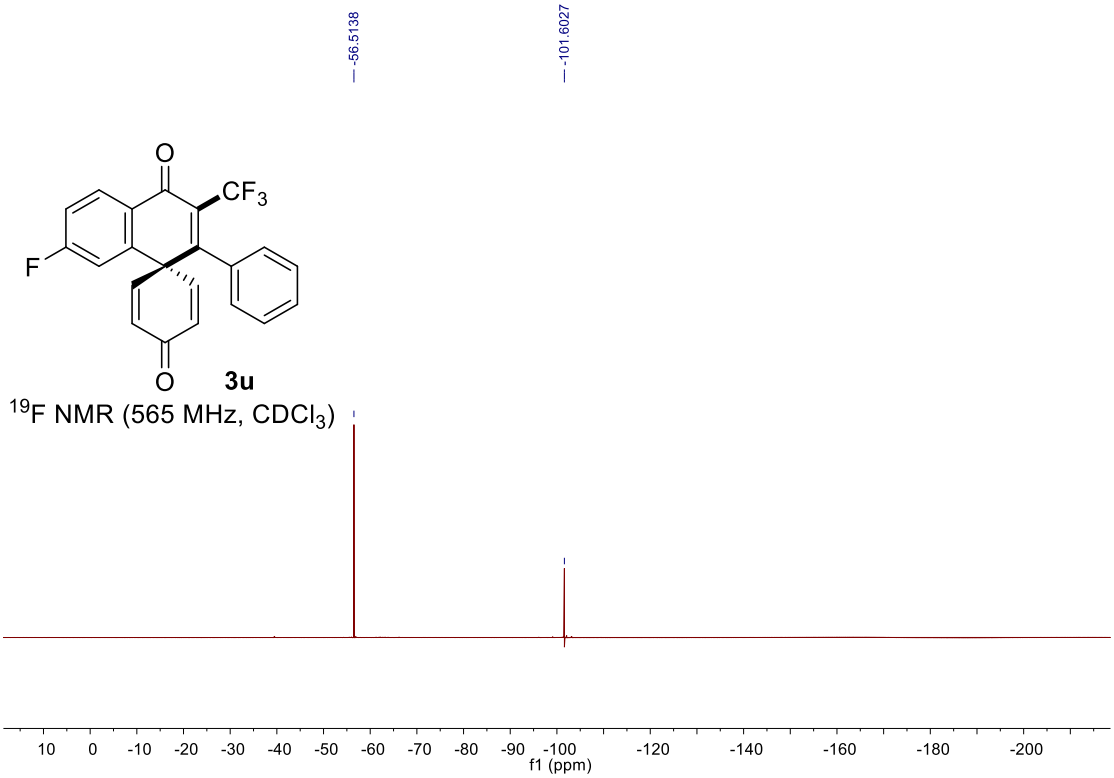


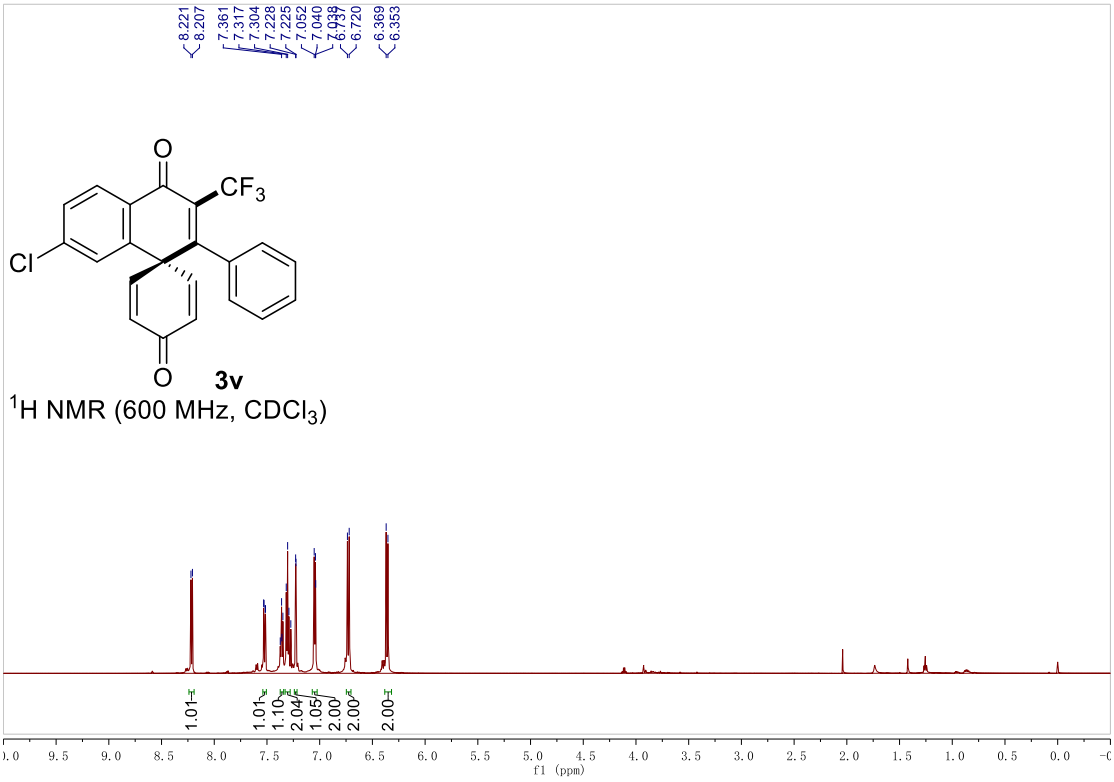


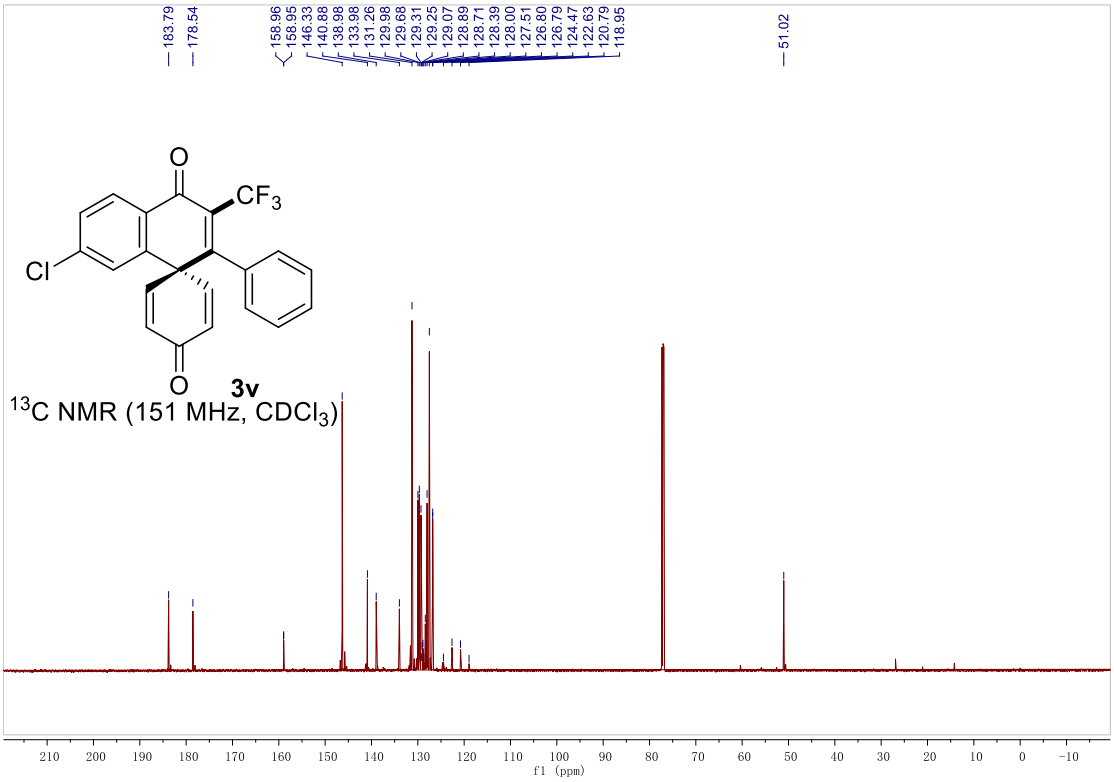
3u

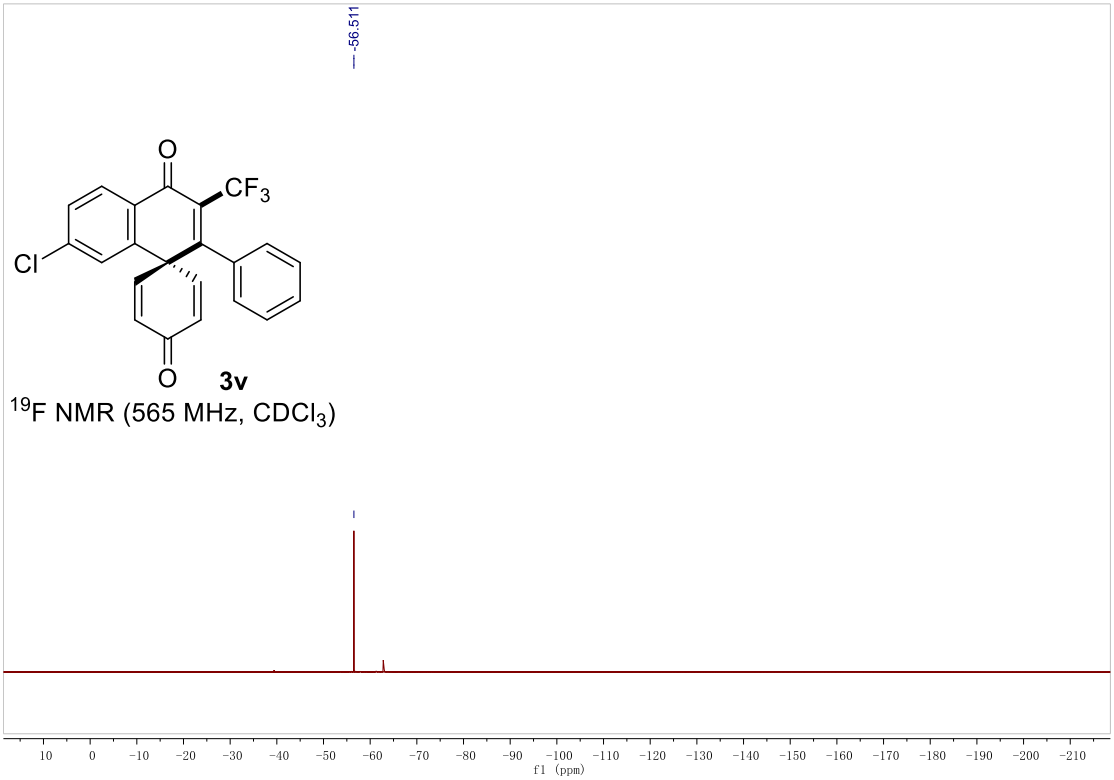
^{13}C NMR (151 MHz, $CDCl_3$)

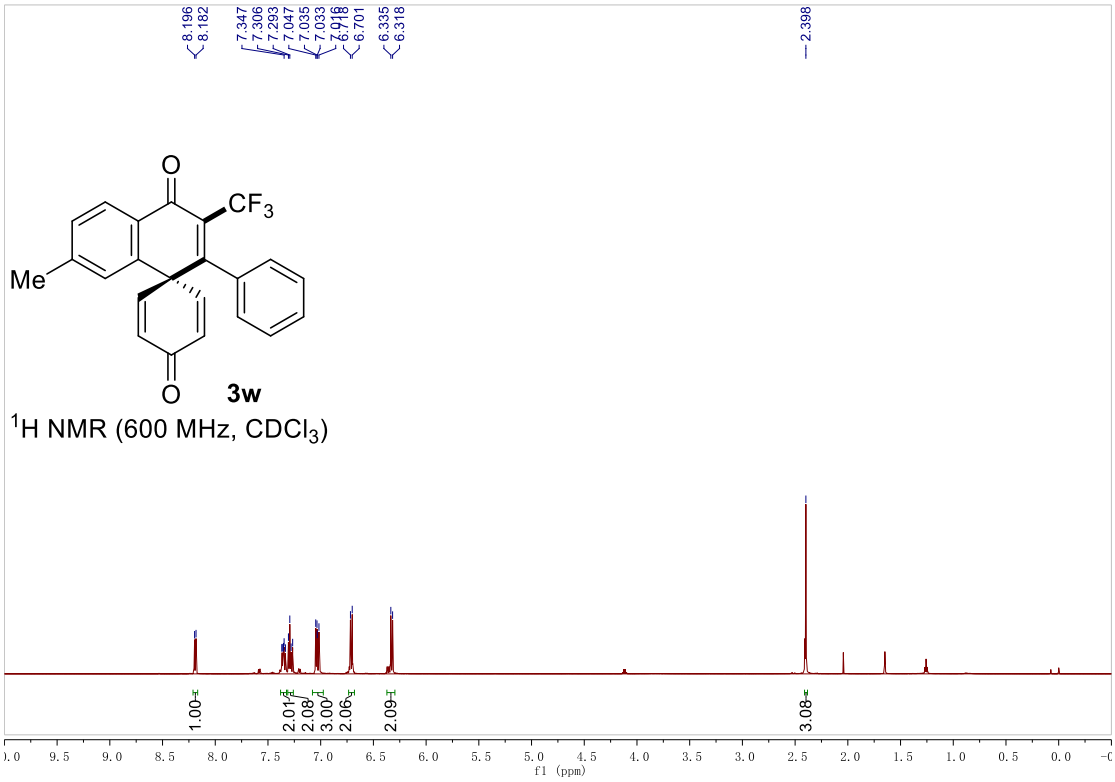


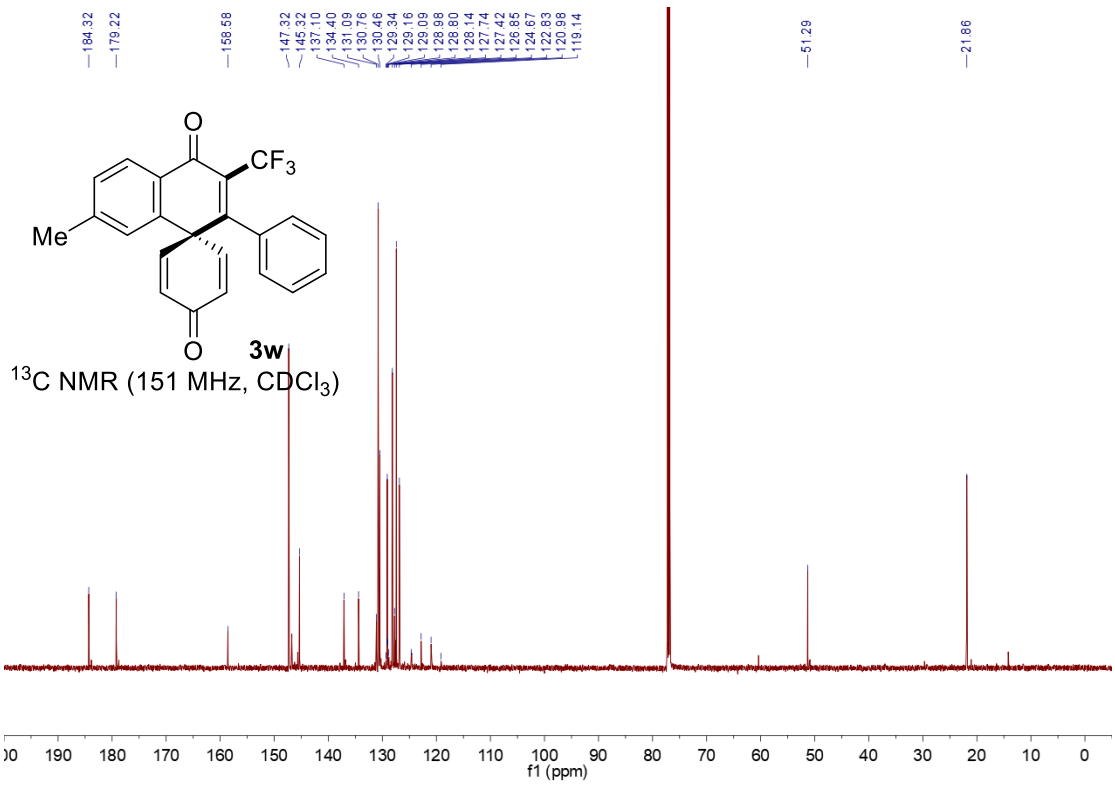

-56.5138
-101.6027
O
CF3
F
O
3u
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -120 -140 -160 -180 -200
f1 (ppm)


8.221
8.207
7.361
7.317
7.304
7.228
7.225
7.052
7.040
7.038
7.037
6.720
6.369
6.353
O
CF3
Cl
O
3v
1H NMR (600 MHz, CDCl3)
1.01
1.01
1.10
2.04
1.05
2.00
2.00
2.00
f1 (ppm)


183.79
178.54
158.96
158.95
146.33
140.88
138.98
133.98
131.26
129.98
129.68
129.31
129.25
129.07
128.89
128.71
128.39
128.00
127.51
126.80
126.79
124.47
122.63
120.79
118.95
51.02
O
CF3
Cl
O
3v
13C NMR (151 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

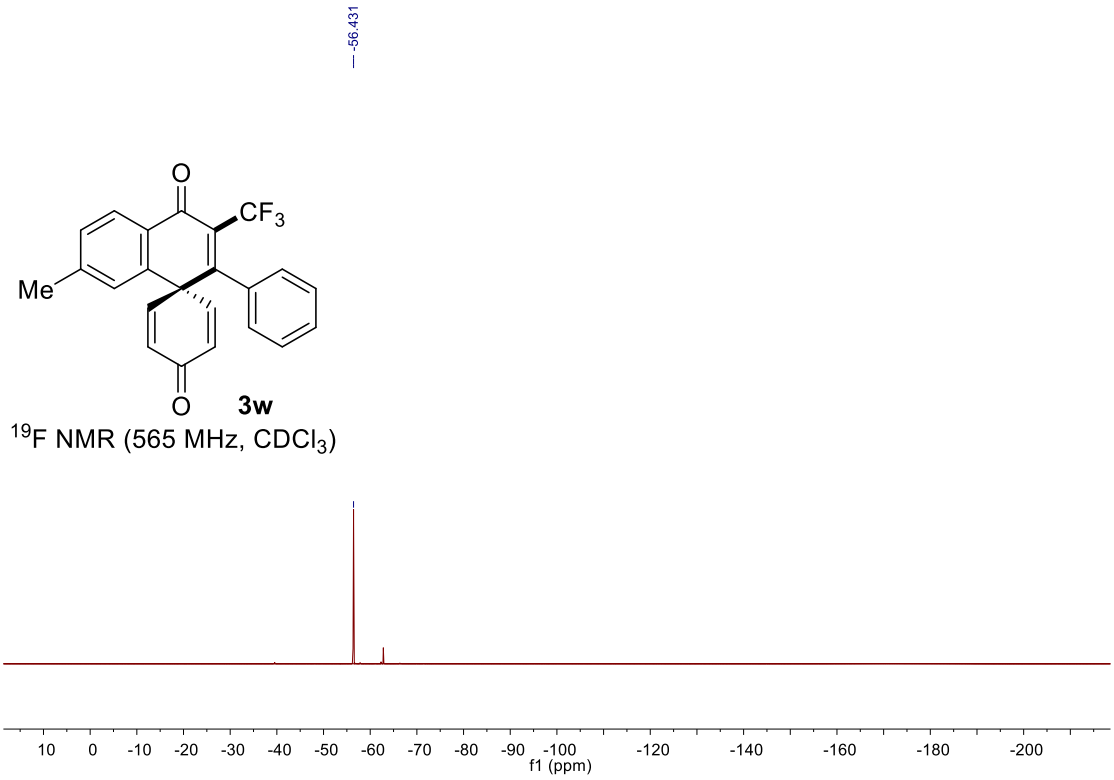

-56.511
O
CF3
Cl
O
3v
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

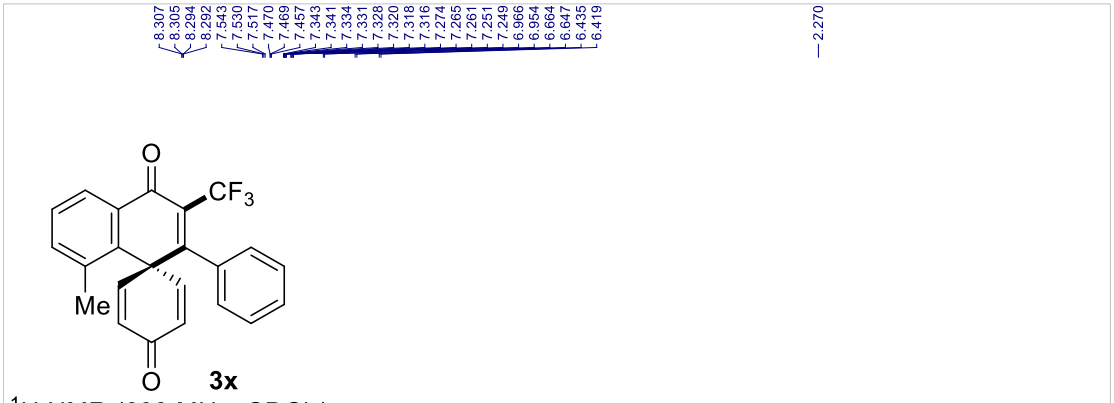

8.196
8.182
7.347
7.306
7.293
7.047
7.035
7.033
7.016
6.718
6.701
6.335
6.318
2.398
O
CF3
Me
O
3w
1H NMR (600 MHz, CDCl3)
1.00
2.01
2.08
3.00
2.06
2.09
3.08
f1 (ppm)


184.32
179.22
158.58
147.32
145.32
137.10
134.40
131.09
130.76
130.46
129.34
129.16
129.09
128.98
128.80
128.14
127.74
127.42
126.85
124.67
122.83
120.98
119.14
51.29
21.86
O
CF3
Me
O
3w
13C NMR (151 MHz, CDCl3)
f1 (ppm)

3w

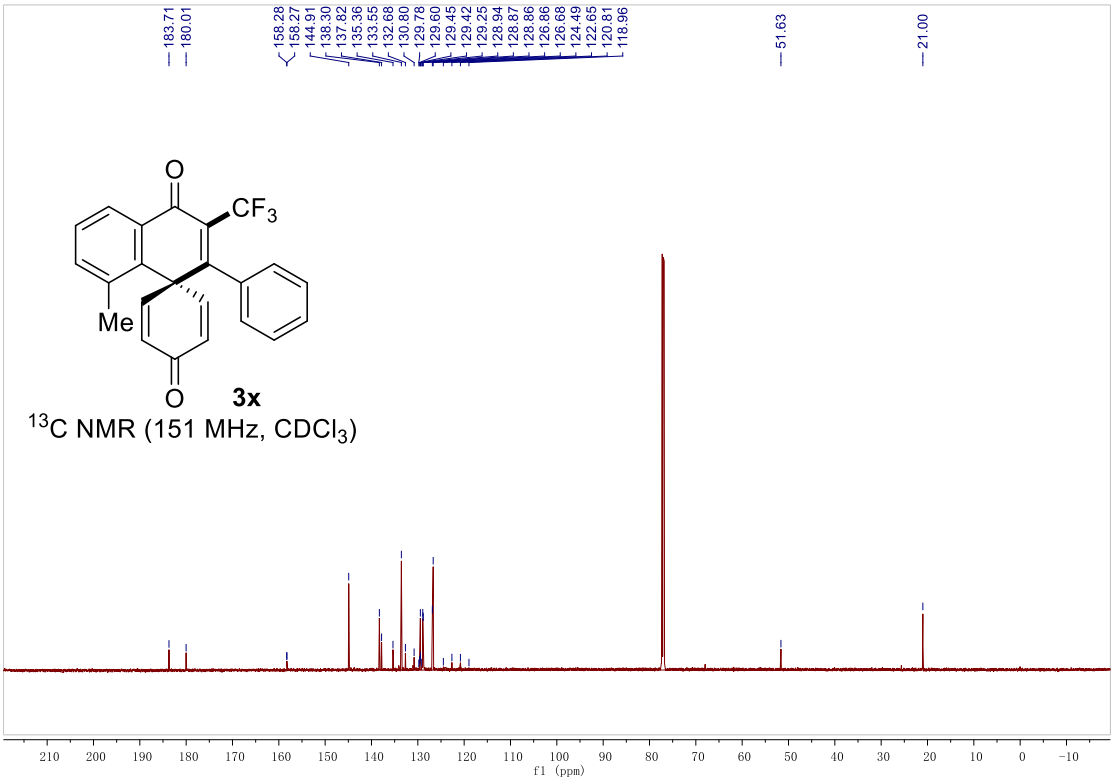
^{19}F NMR (565 MHz, $CDCl_3$)

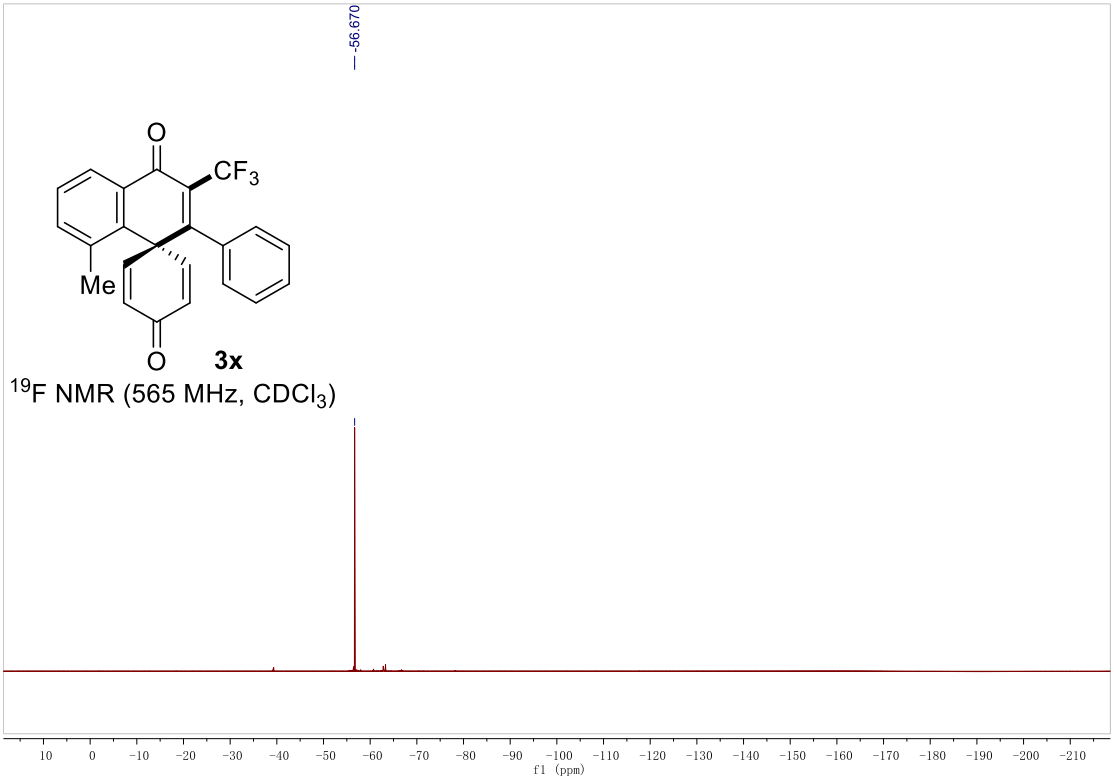


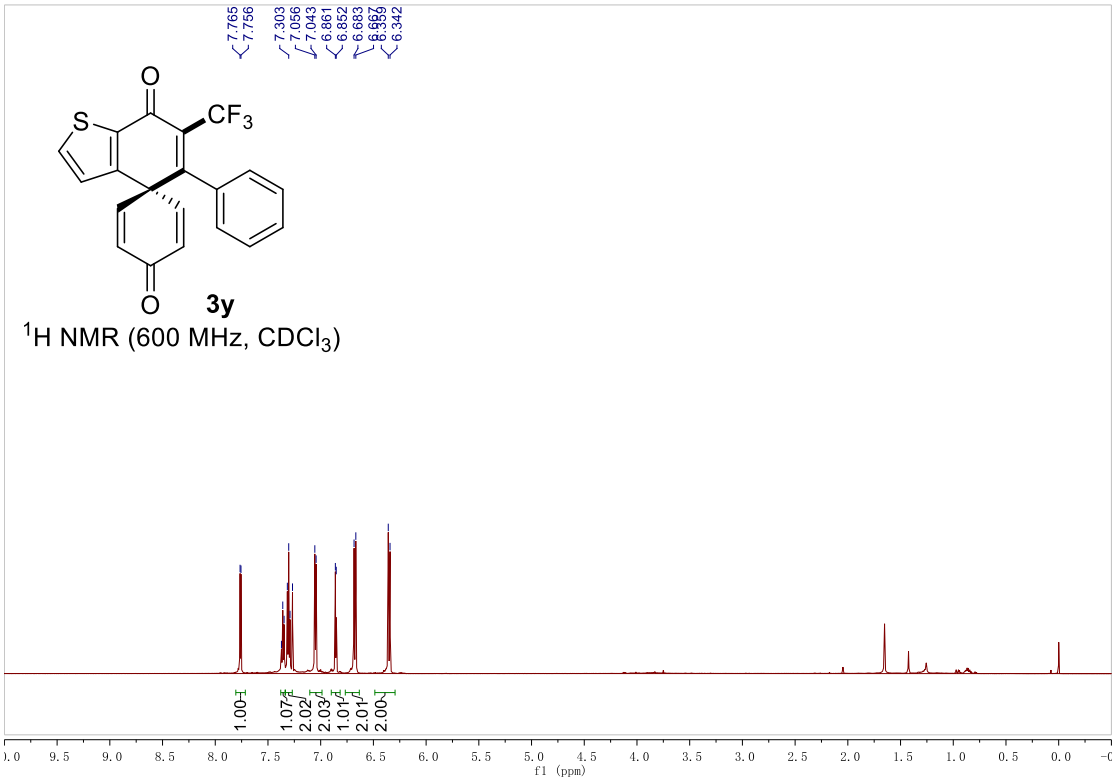


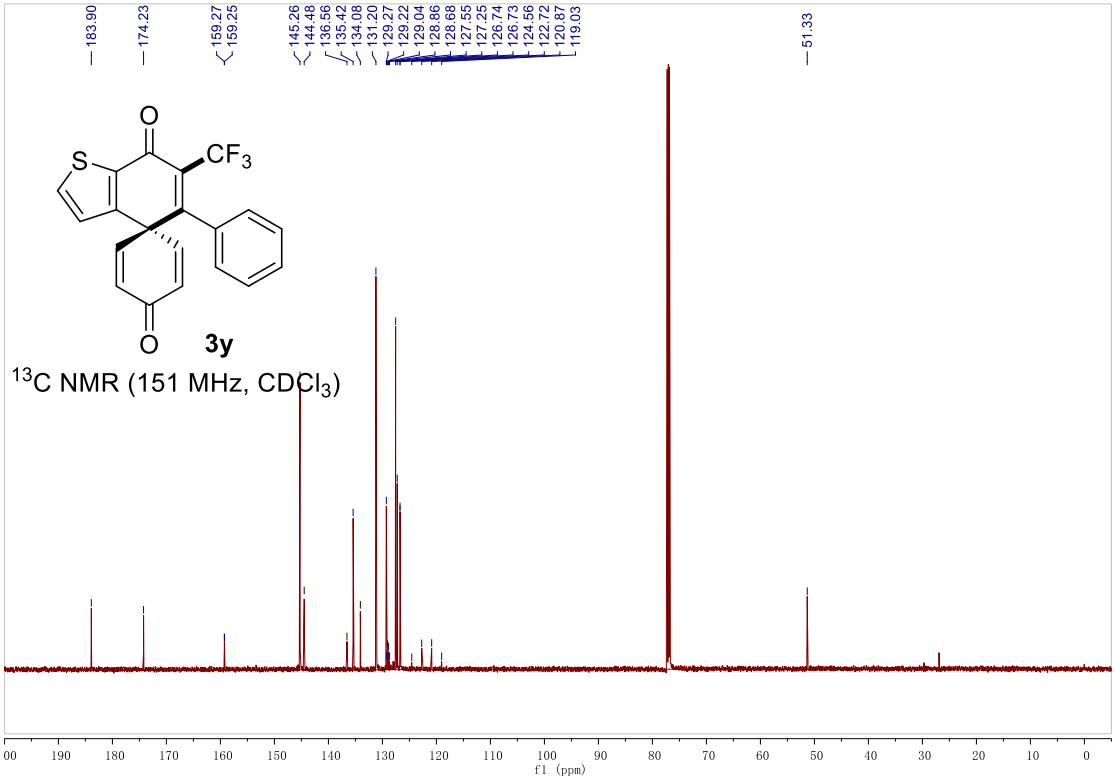
3x

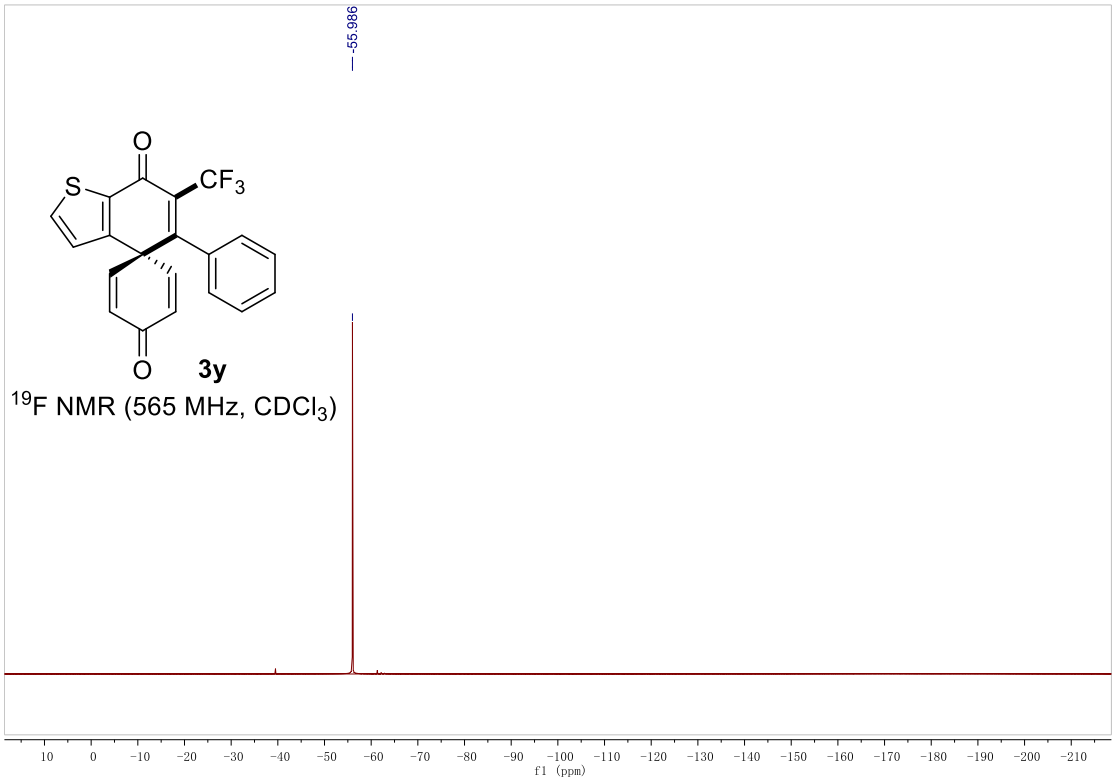
^{1}H NMR (600 MHz, $CDCl_3$)

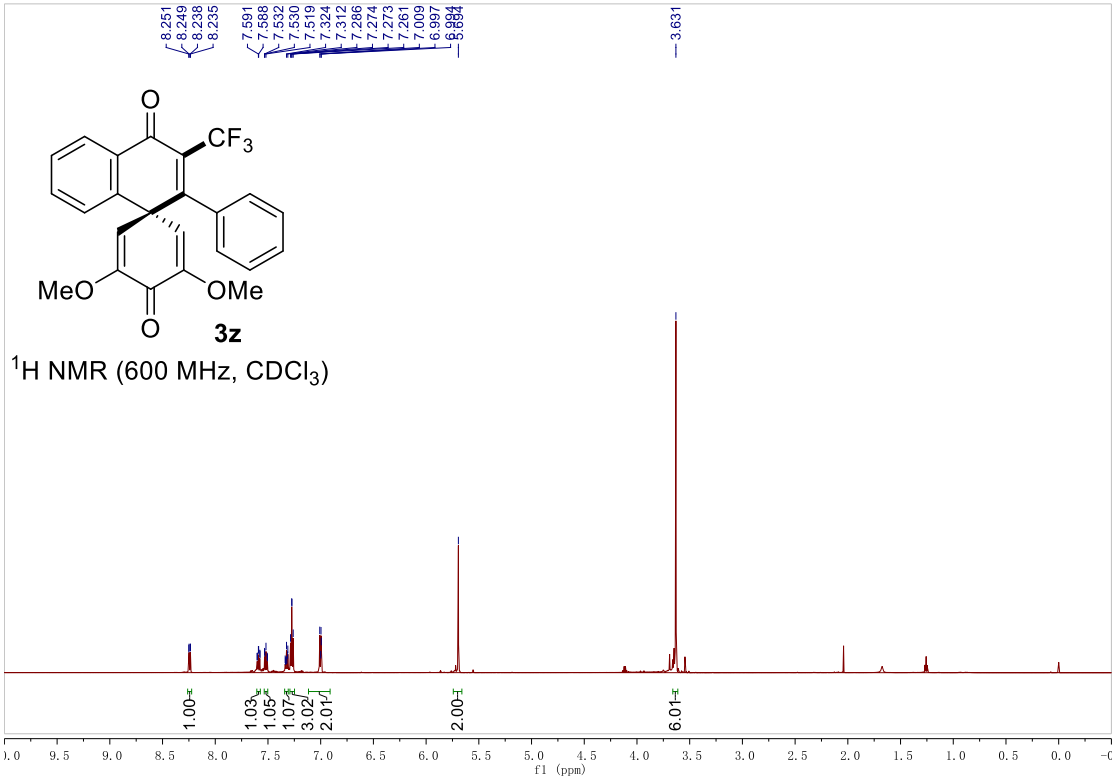

183.71
180.01
158.28
158.27
144.91
138.30
137.82
135.36
133.55
132.68
130.80
129.78
129.60
129.45
129.42
129.25
128.94
128.87
128.86
126.86
126.68
124.49
122.65
120.81
118.96
51.63
21.00
O
CF3
Me
O
3x
13C NMR (151 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

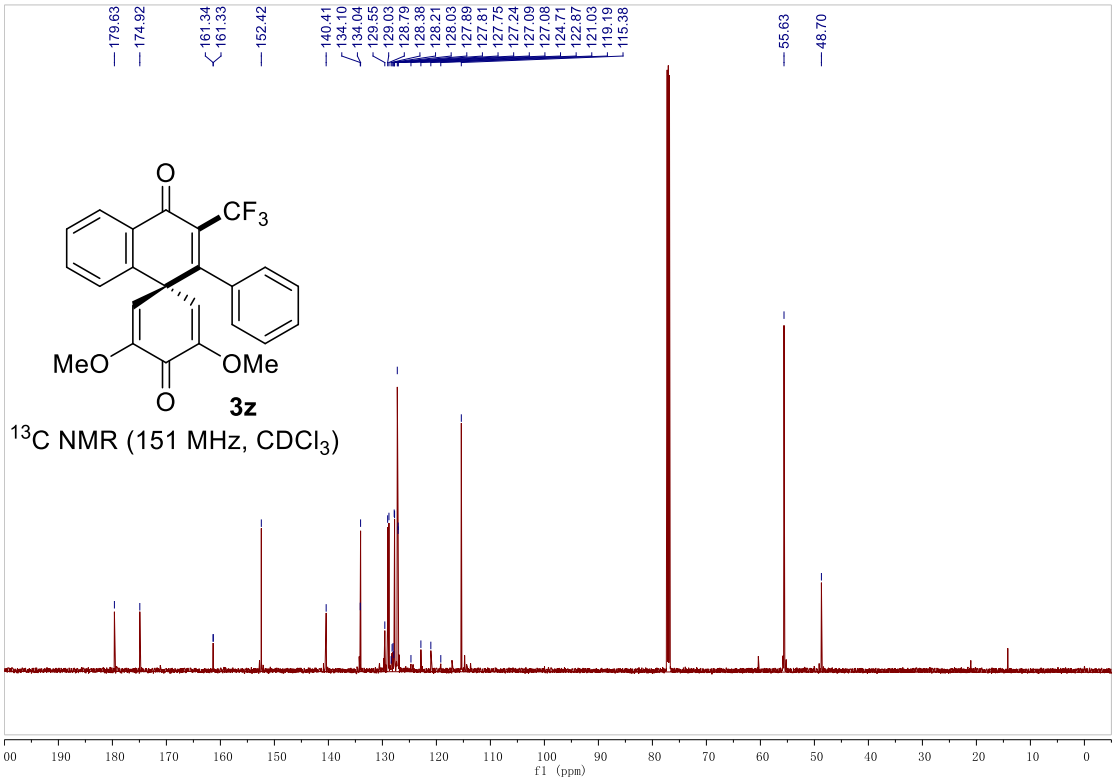

-56.670
O
CF3
Me
O
3x
19F NMR (565 MHz, CDCl3)
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

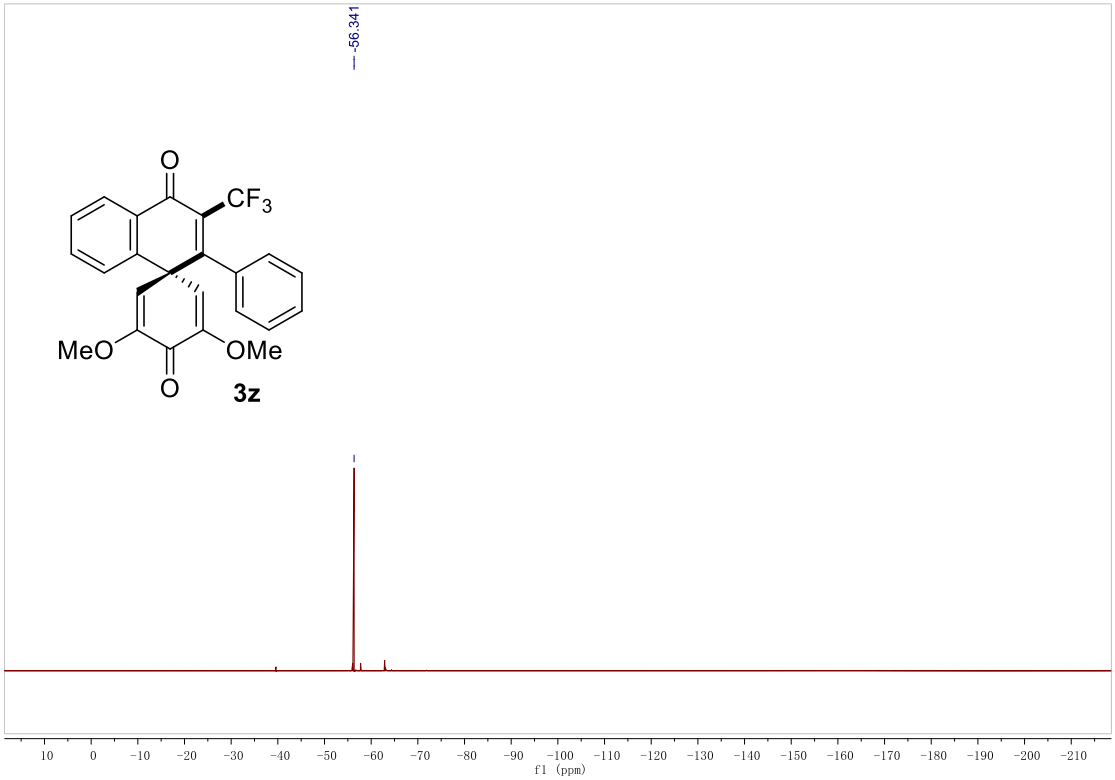

7.765
7.756
7.303
7.056
7.043
6.861
6.852
6.683
6.667
6.359
6.342
O
S
CF3
O
3y
1H NMR (600 MHz, CDCl3)
1.00
1.07
2.02
2.03
1.01
2.01
2.00
f1 (ppm)

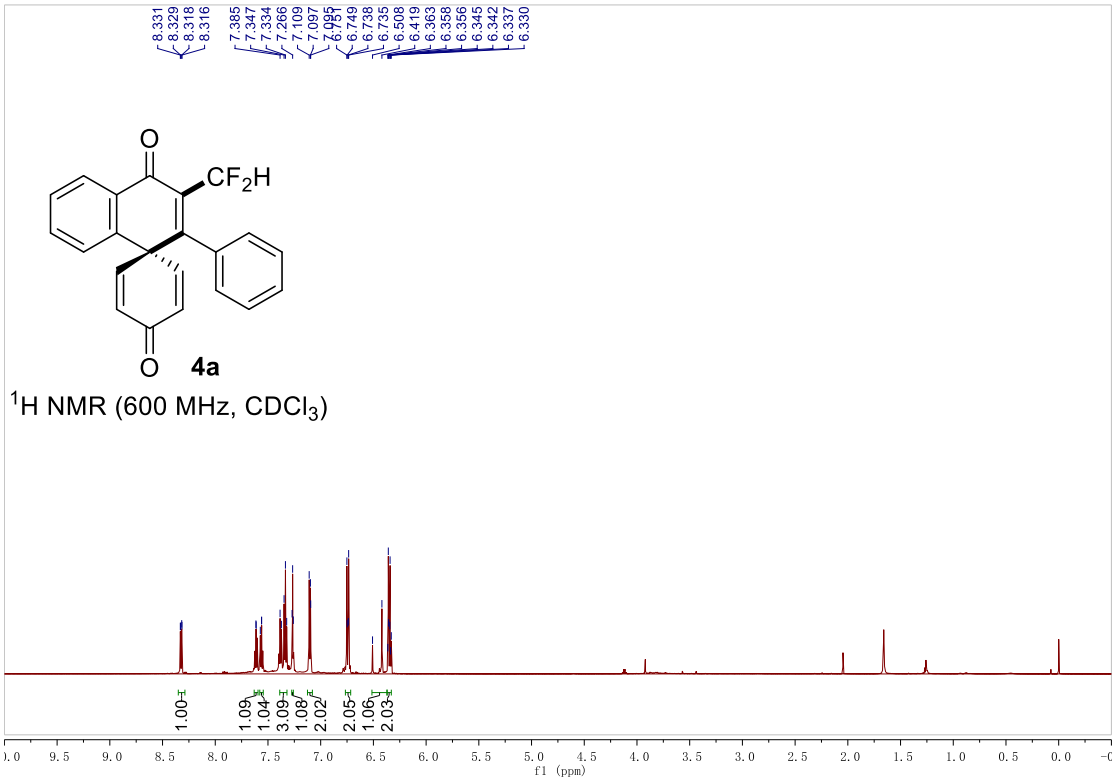

183.90
174.23
159.27
159.25
145.26
144.48
136.56
135.42
134.08
131.20
129.27
129.22
129.04
128.86
128.68
127.55
127.25
126.74
126.73
124.56
122.72
120.87
119.03
51.33
O
S
CF3
O
3y
13C NMR (151 MHz, CDCl3)
f1 (ppm)

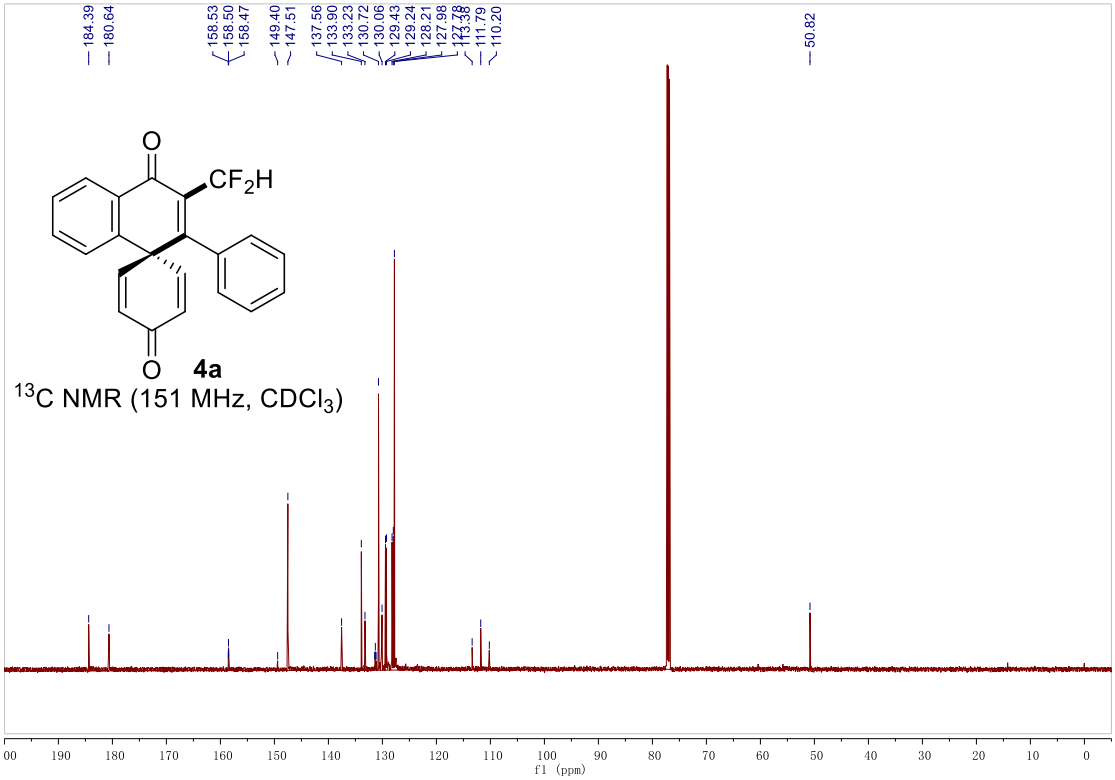

-55.986
O
S
CF3
O
3y
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

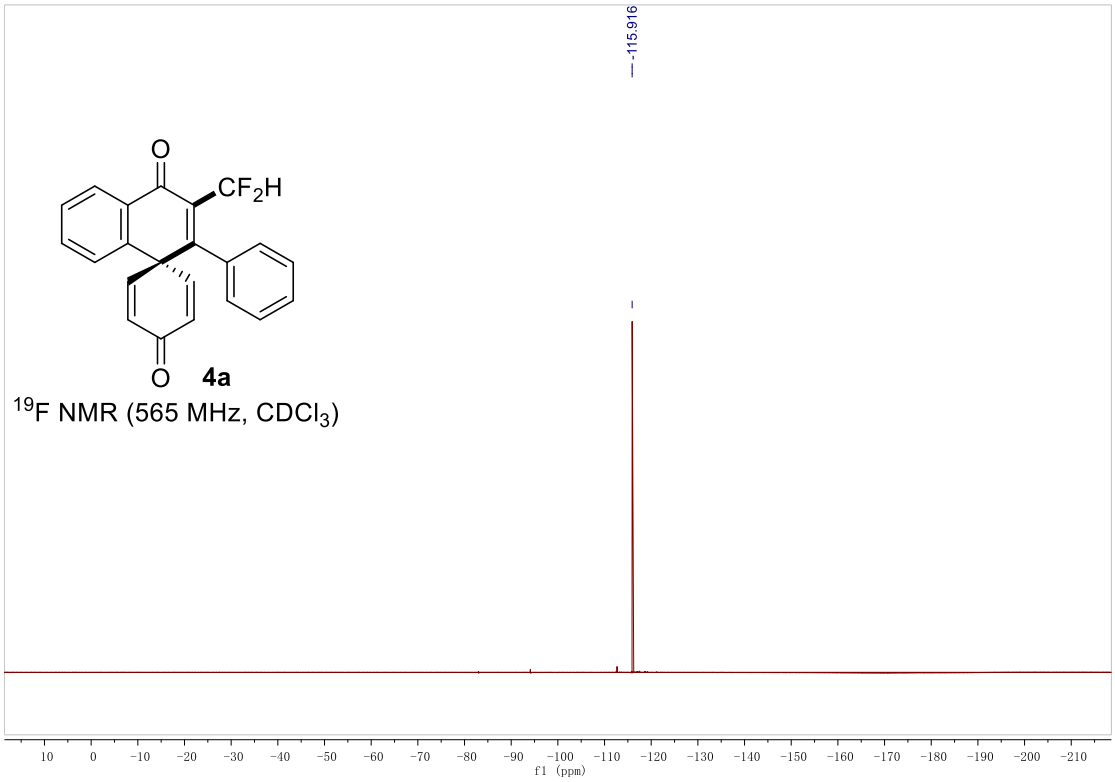

8.251
8.249
8.238
8.235
7.591
7.588
7.532
7.530
7.519
7.324
7.312
7.286
7.274
7.273
7.261
7.009
6.997
6.994
6.984
3.631
O
CF3
MeO
OMe
O
3z
1H NMR (600 MHz, CDCl3)
1.00
1.03
1.05
1.07
3.02
2.01
2.00
6.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

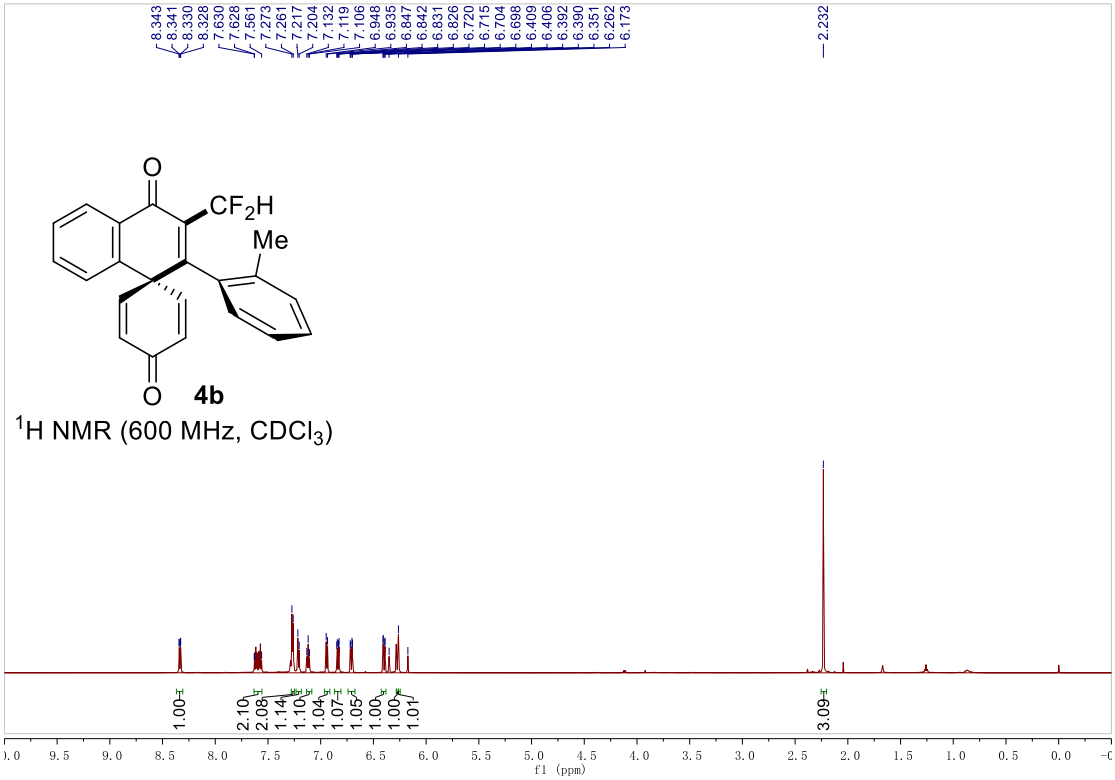

179.63
174.92
161.34
161.33
152.42
140.41
134.10
134.04
129.55
129.03
128.79
128.38
128.21
128.03
127.89
127.81
127.75
127.24
127.09
127.08
124.71
122.87
121.03
119.19
115.38
55.63
48.70
O
CF3
MeO
OMe
O
3z
13C NMR (151 MHz, CDCl3)
f1 (ppm)

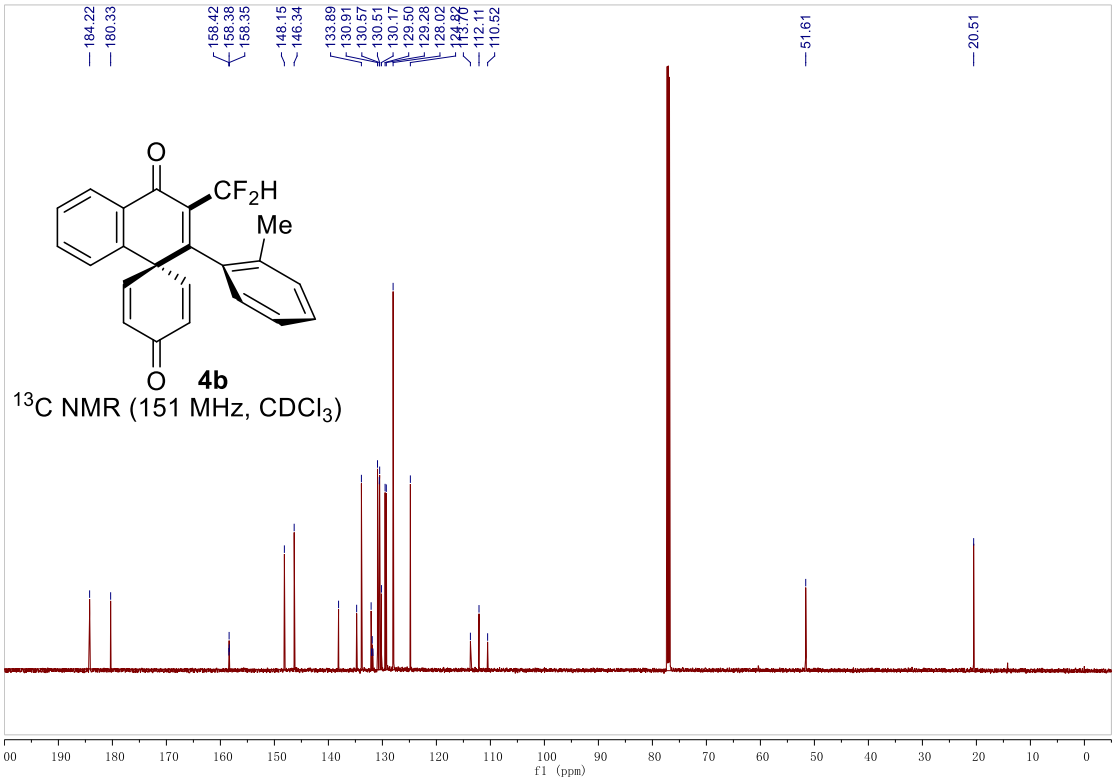

-56.341
O
CF3
MeO
OMe
O
3z
f1 (ppm)

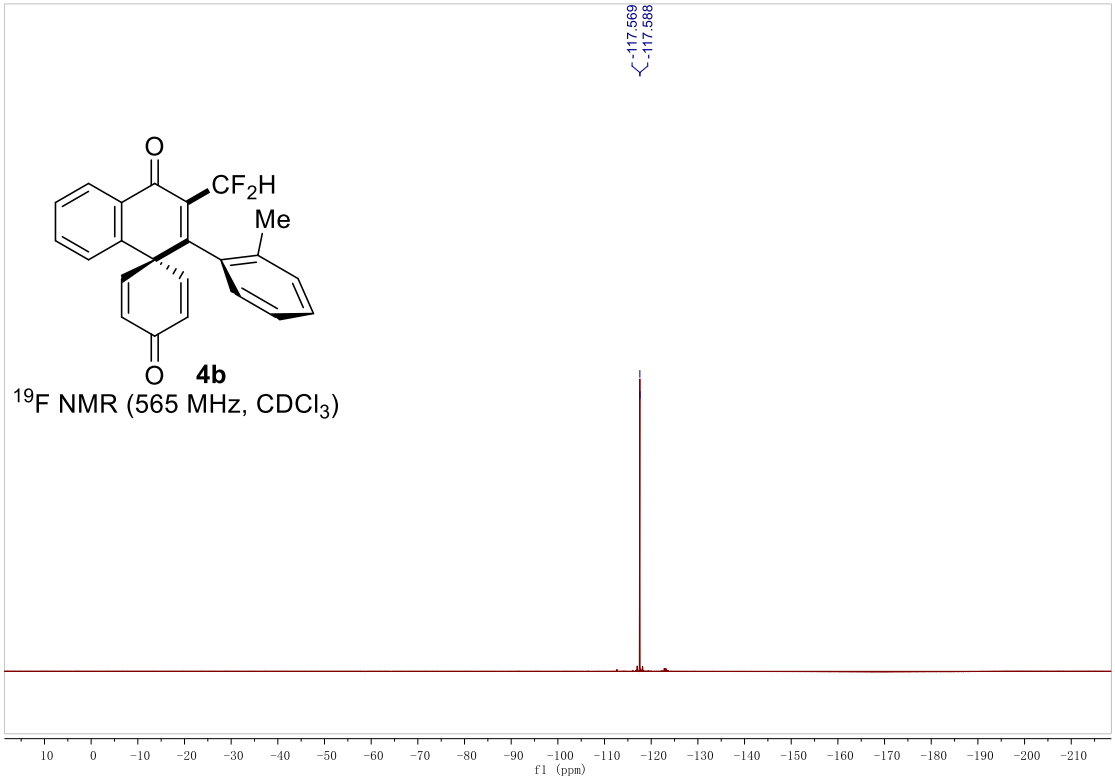

8.331
8.329
8.318
8.316
7.385
7.347
7.334
7.266
7.109
7.097
6.749
6.738
6.735
6.508
6.419
6.363
6.358
6.356
6.345
6.342
6.337
6.330
O
CF2H
O
4a
1H NMR (600 MHz, CDCl3)
1.00
1.09
1.04
3.09
1.08
2.02
2.05
1.06
2.03
f1 (ppm)

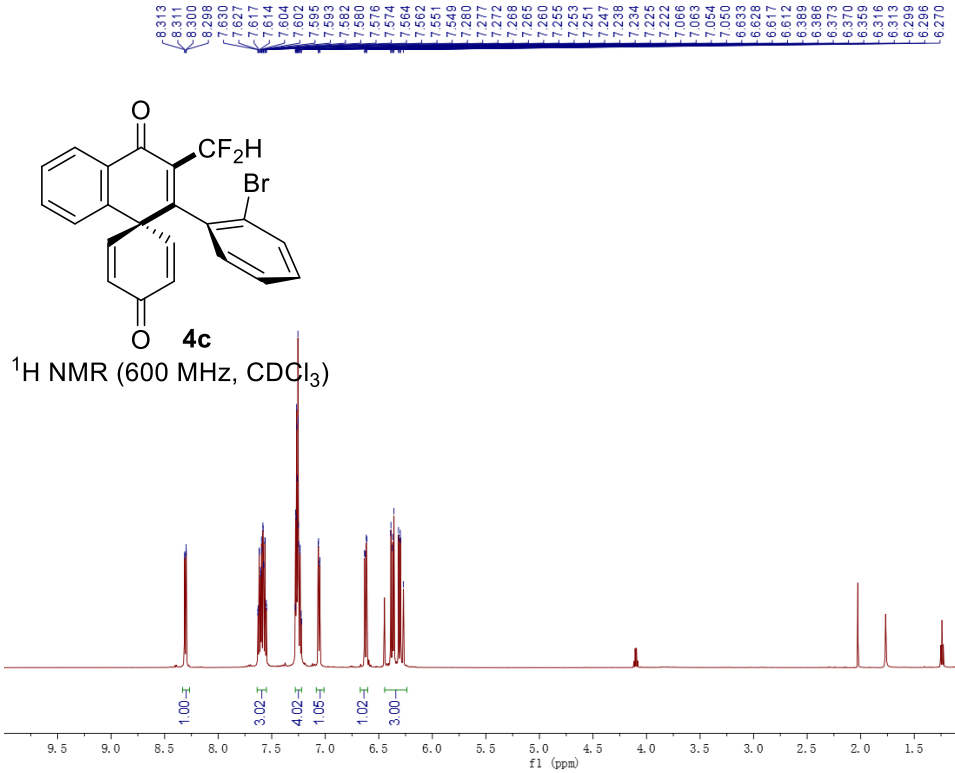

184.39
180.64
158.53
158.50
158.47
149.40
147.51
137.56
133.90
133.23
130.72
130.06
129.43
129.24
128.21
127.98
113.38
111.79
110.20
50.82
O
CF2H
O
4a
13C NMR (151 MHz, CDCl3)
f1 (ppm)

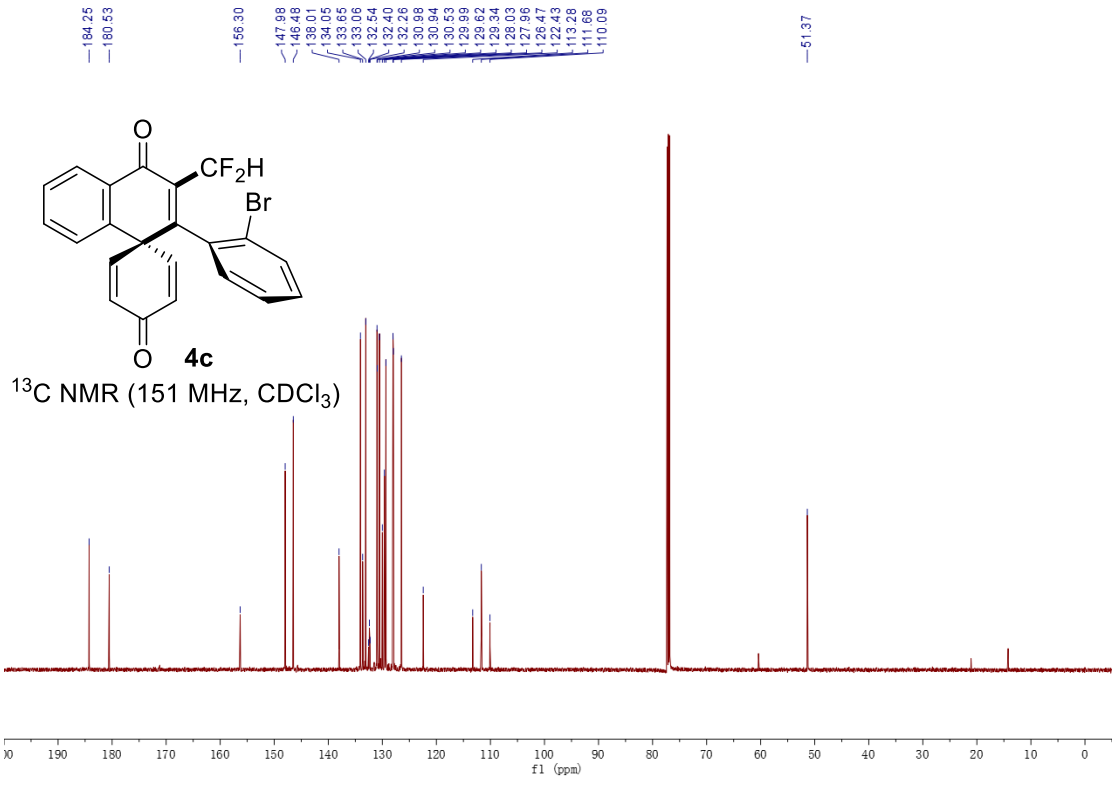

-115.916
O
CF2H
O
4a
19F NMR (565 MHz, CDCl3)
f1 (ppm)


8.343
8.341
8.330
8.328
7.630
7.628
7.561
7.273
7.261
7.217
7.204
7.132
7.119
7.106
6.948
6.935
6.847
6.842
6.831
6.826
6.720
6.715
6.704
6.698
6.409
6.406
6.392
6.390
6.351
6.262
6.173
2.232
O
CF2H
Me
O
4b
1H NMR (600 MHz, CDCl3)
1.00
2.10
2.08
1.14
1.10
1.04
1.07
1.05
1.00
1.00
1.01
3.09
f1 (ppm)


184.22
180.33
158.42
158.38
158.35
148.15
146.34
133.89
130.91
130.57
130.51
130.17
129.50
129.28
128.02
124.82
124.70
112.11
110.52
51.61
20.51
O
CF2H
Me
O
4b
13C NMR (151 MHz, CDCl3)
f1 (ppm)

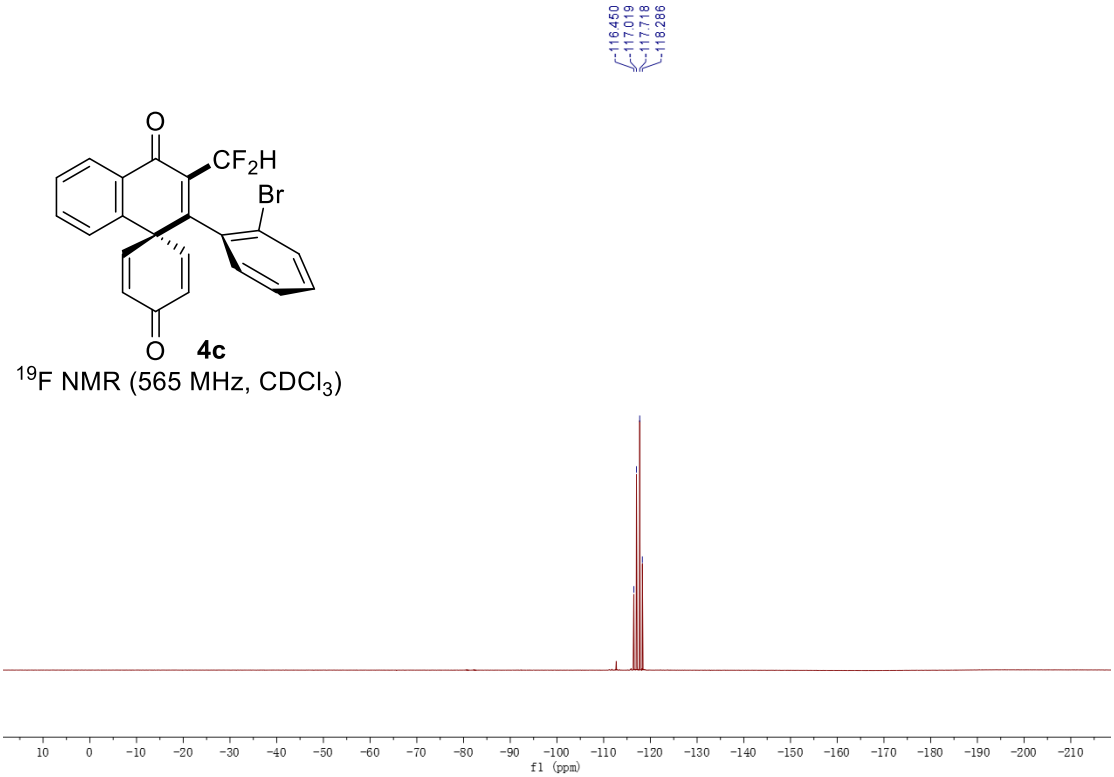

-117.569
-117.588
O
CF2H
Me
O
4b
19F NMR (565 MHz, CDCl3)
f1 (ppm)

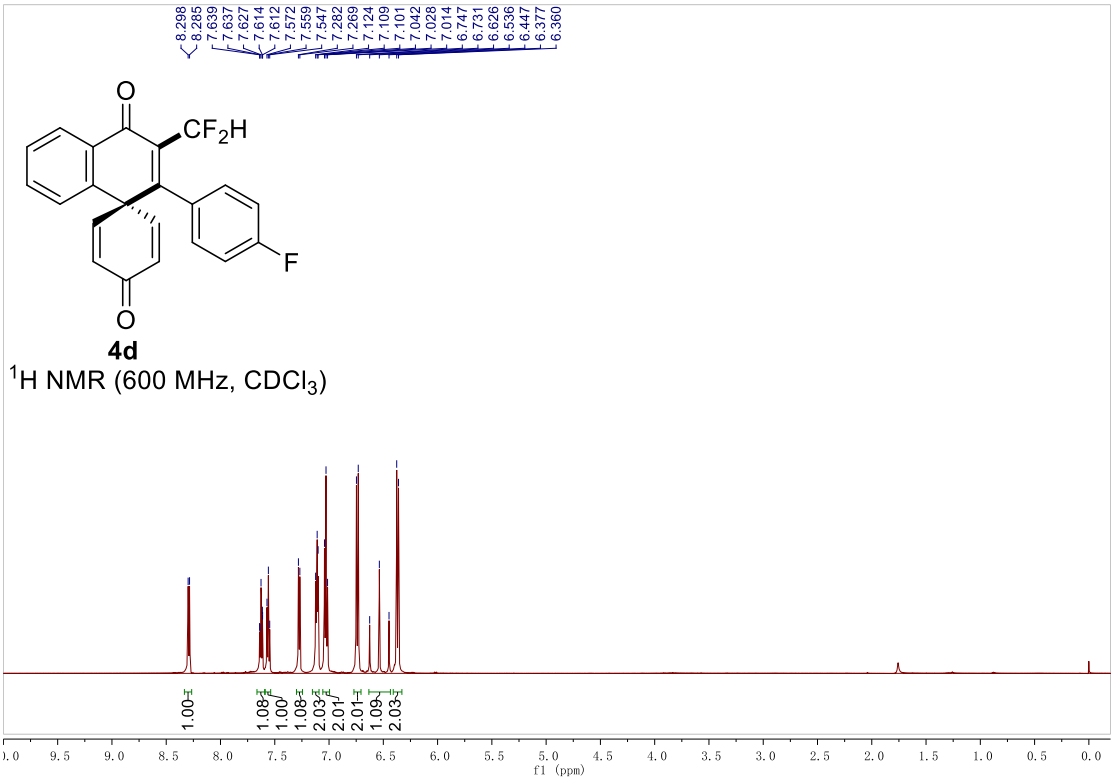

8.313
8.311
8.300
8.298
7.630
7.627
7.617
7.614
7.604
7.602
7.595
7.593
7.582
7.580
7.576
7.574
7.564
7.562
7.551
7.549
7.280
7.277
7.272
7.268
7.265
7.260
7.255
7.253
7.251
7.247
7.238
7.234
7.225
7.222
7.066
7.063
7.054
7.050
6.633
6.628
6.617
6.612
6.389
6.386
6.373
6.370
6.359
6.316
6.313
6.299
6.296
6.270
O
CF2H
Br
O
4c
1H NMR (600 MHz, CDCl3)
1.00
3.02
4.02
1.05
1.02
3.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


184.25
180.53
156.30
147.98
146.48
138.01
134.05
133.65
133.06
132.54
132.40
132.26
130.98
130.94
130.53
129.99
129.62
129.34
128.03
127.96
126.47
122.43
113.28
111.68
110.09
51.37
O
CF2H
Br
O
4c
13C NMR (151 MHz, CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4c

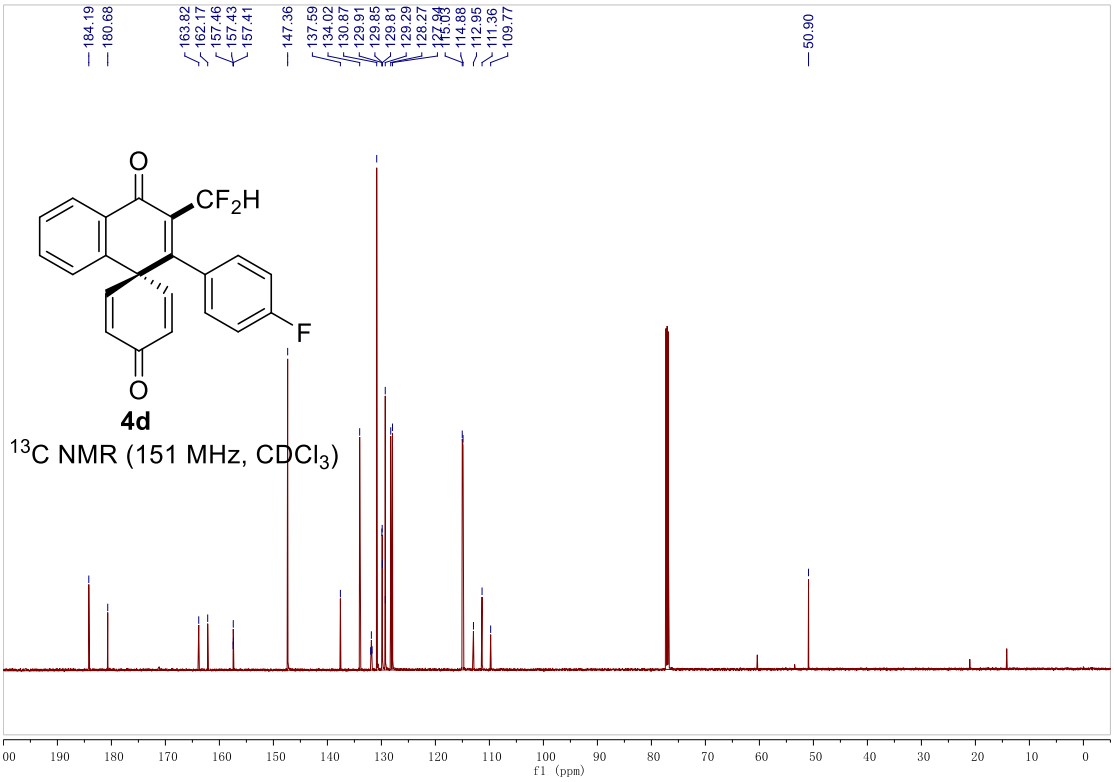
^{19}F NMR (565 MHz, $CDCl_3$)

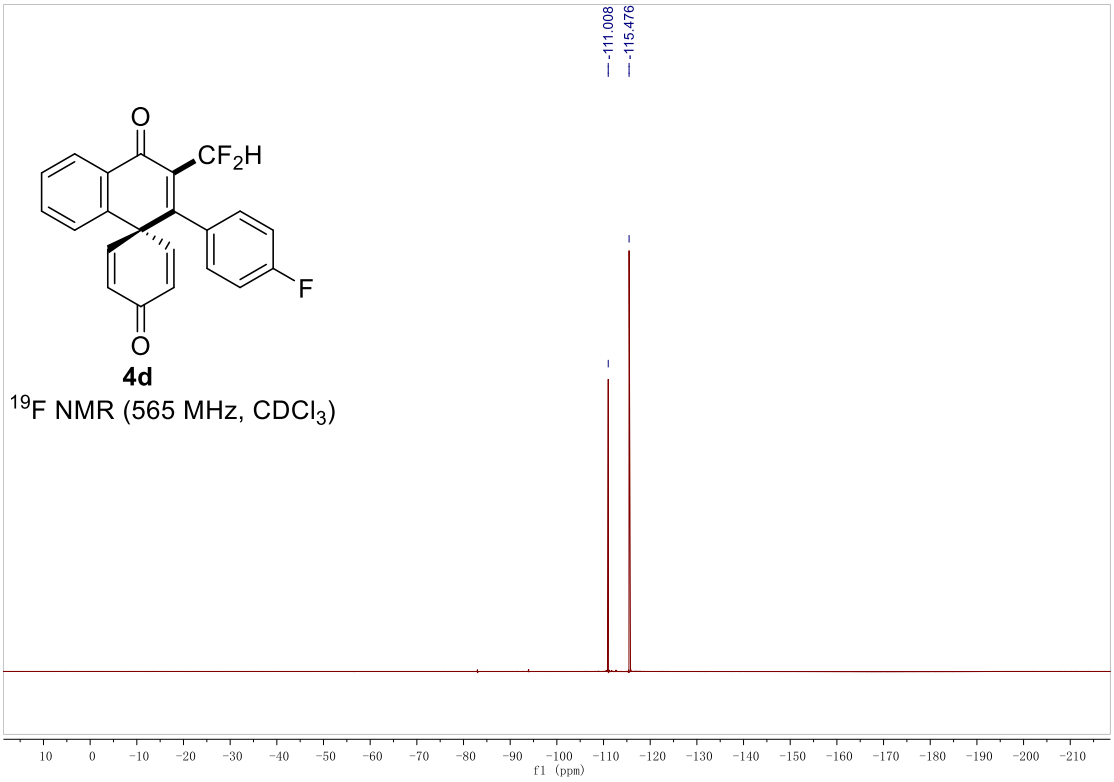


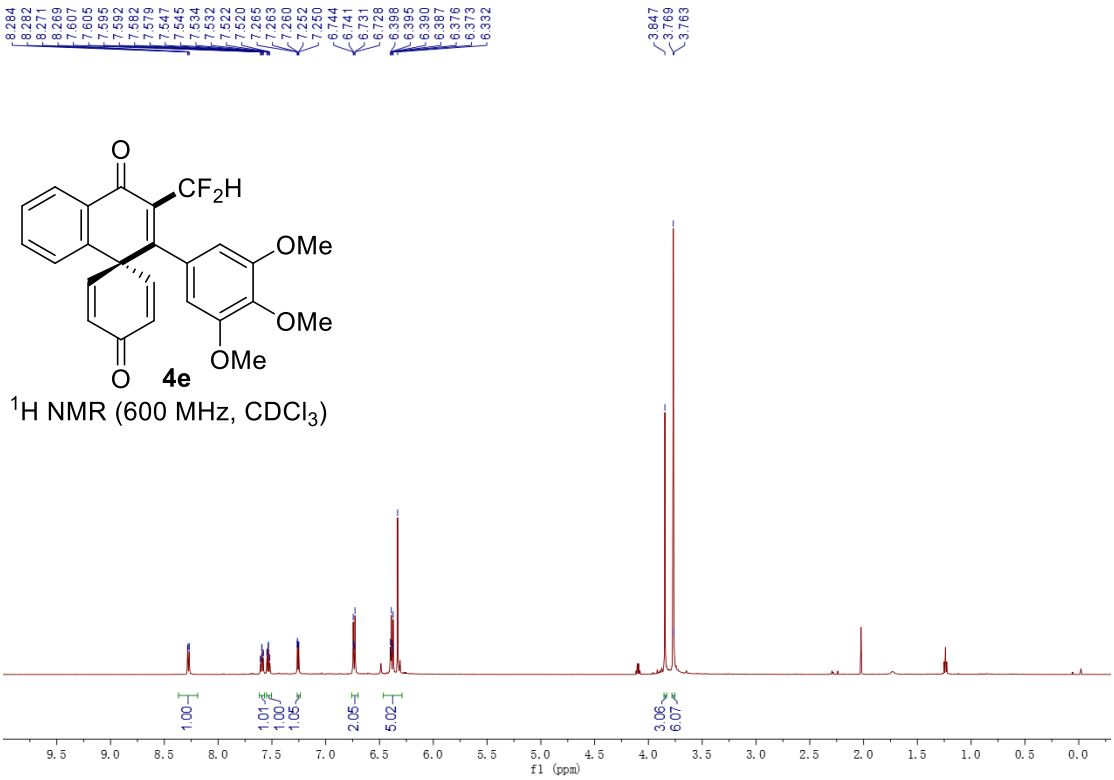


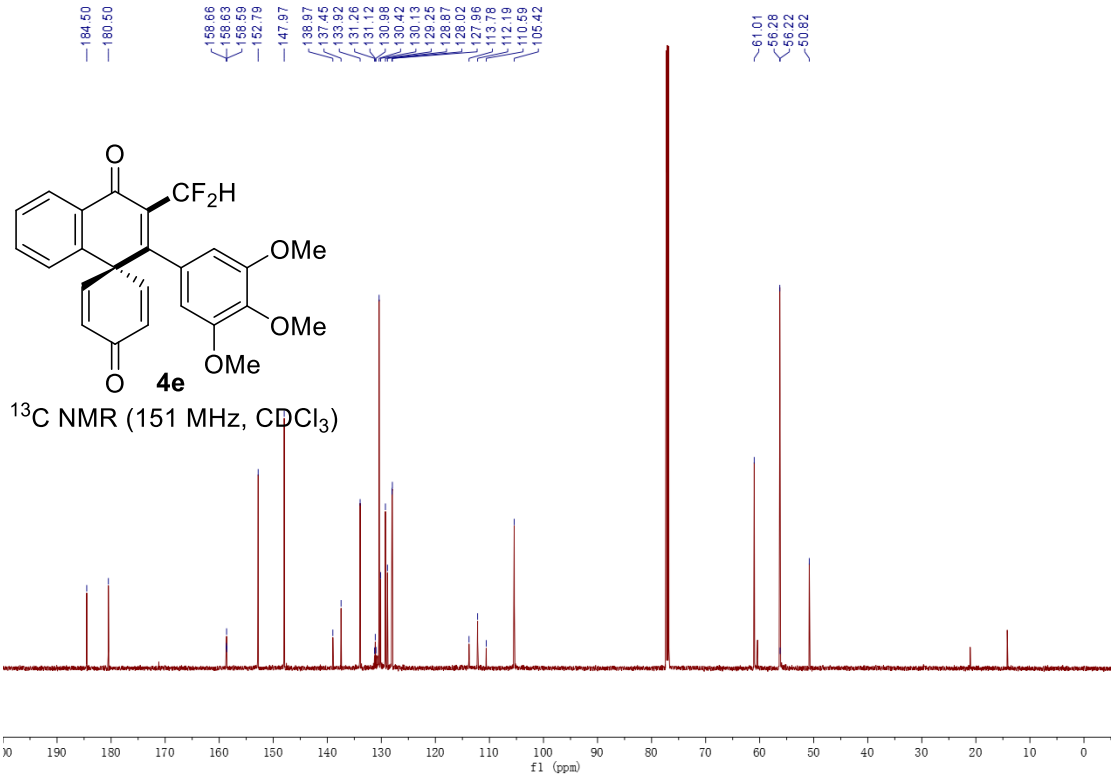
4d

^{1}H NMR (600 MHz, $CDCl_3$)


184.19
180.68
163.82
162.17
157.46
157.43
157.41
147.36
137.59
134.02
130.87
129.91
129.85
129.81
129.29
128.27
114.88
112.95
111.36
109.77
50.90
O
CF2H
F
O
4d
13C NMR (151 MHz, CDCl3)
f1 (ppm)

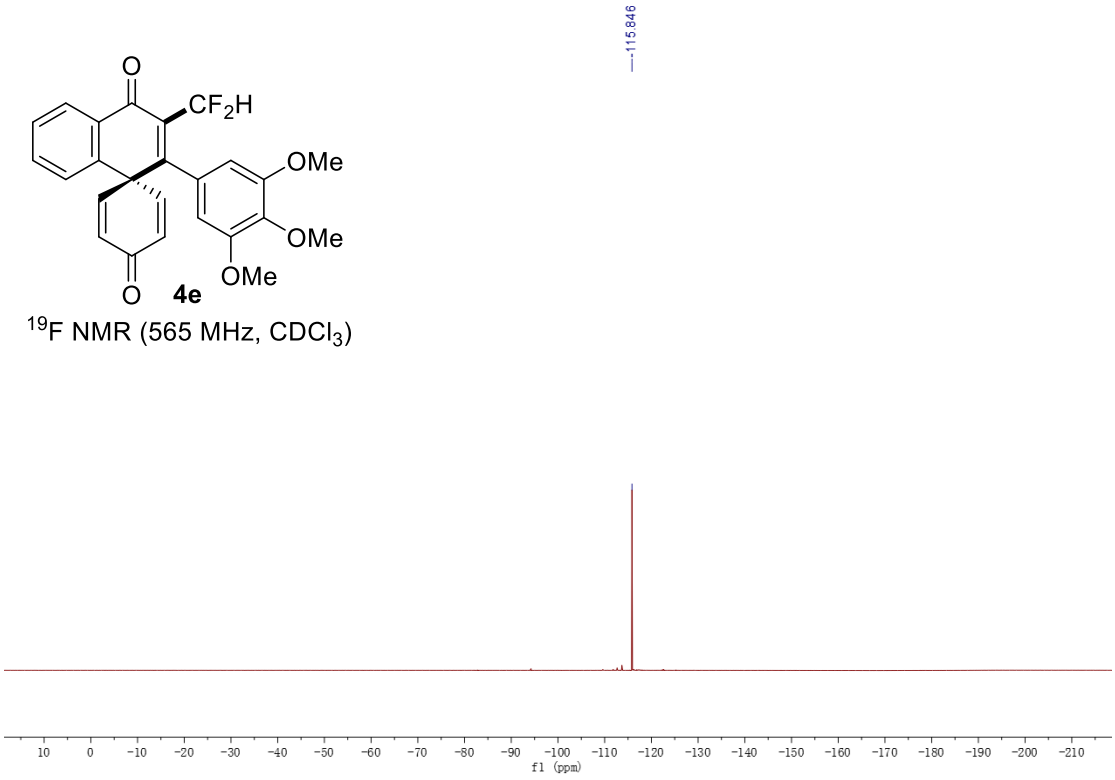

-111.008
-115.476
O
CF2H
F
O
4d
19F NMR (565 MHz, CDCl3)
f1 (ppm)

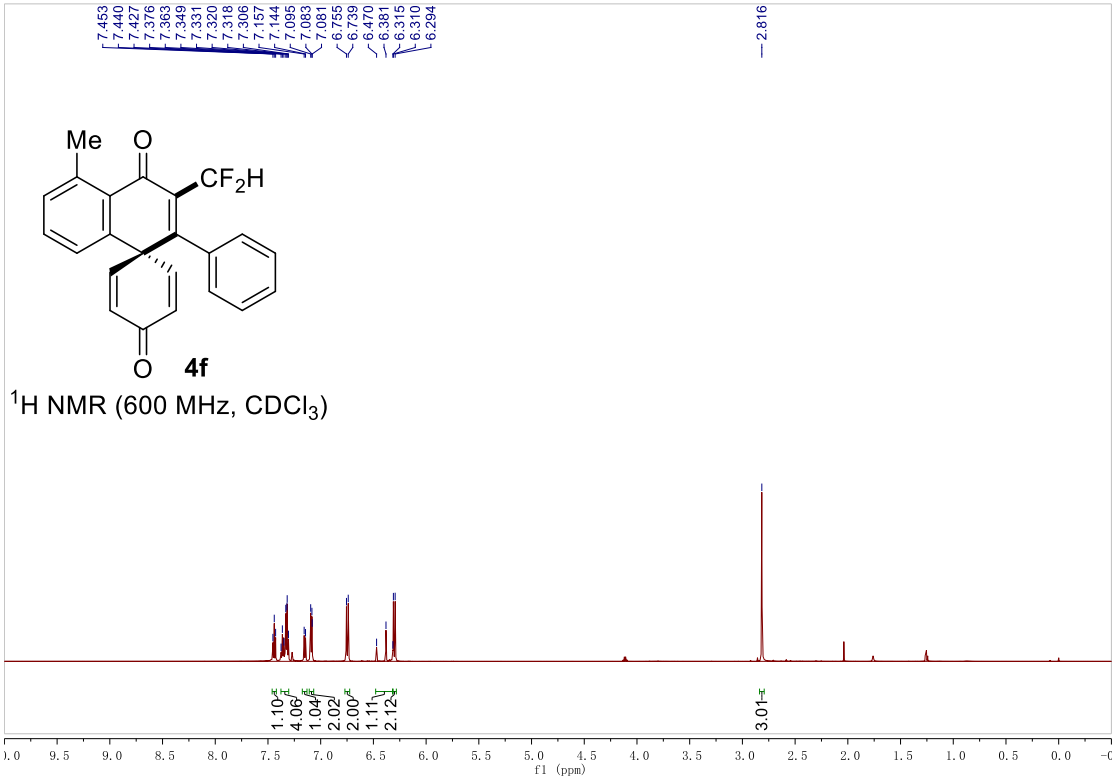

8.284
8.282
8.271
8.269
7.607
7.605
7.595
7.592
7.582
7.579
7.547
7.545
7.534
7.532
7.522
7.520
7.265
7.263
7.260
7.252
7.250
6.744
6.741
6.731
6.728
6.398
6.395
6.390
6.387
6.376
6.373
6.332
3.847
3.769
3.763
O
CF2H
OMe
OMe
OMe
O
4e
1H NMR (600 MHz, CDCl3)
1.00
1.01
1.00
1.05
2.05
5.02
3.06
6.07
9.5 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
f1 (ppm)


184.50
180.50
158.66
158.63
158.59
152.79
147.97
138.97
137.45
133.92
131.26
131.12
130.98
130.42
130.13
129.25
128.87
128.02
127.96
113.78
112.19
110.59
105.42
61.01
56.28
56.22
50.82
O
CF2H
OMe
OMe
OMe
O
4e
13C NMR (151 MHz, CDCl3)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4e

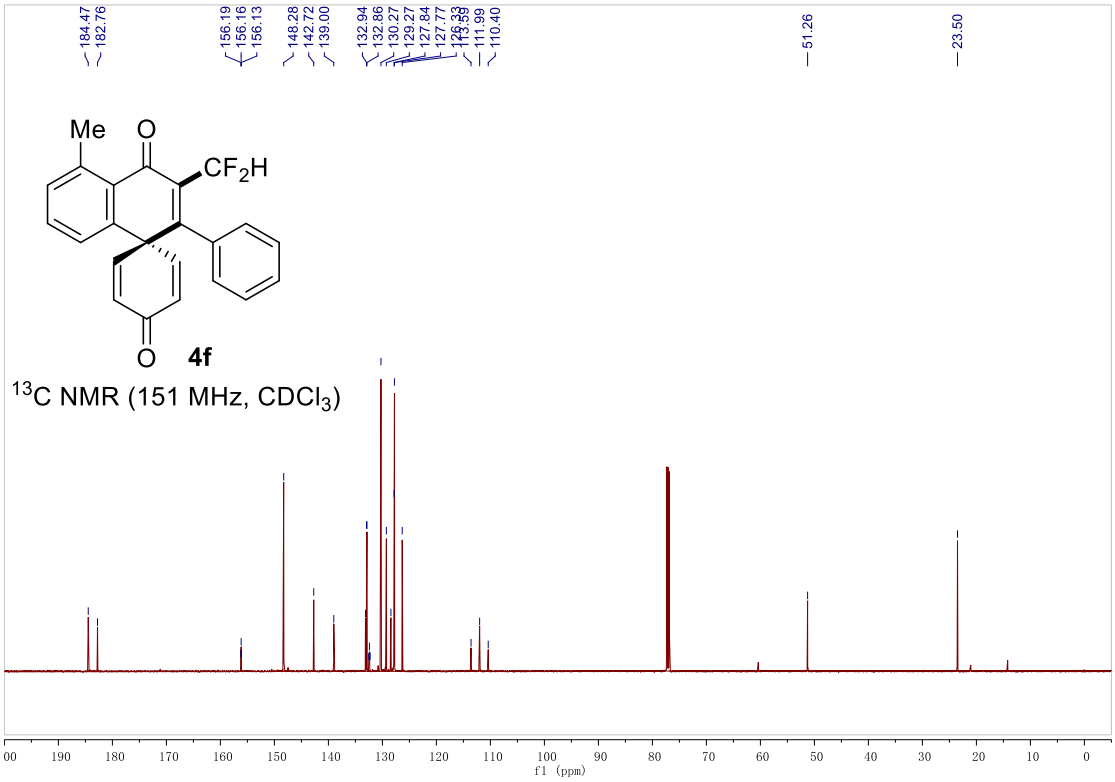
^{19}F NMR (565 MHz, $CDCl_3$)

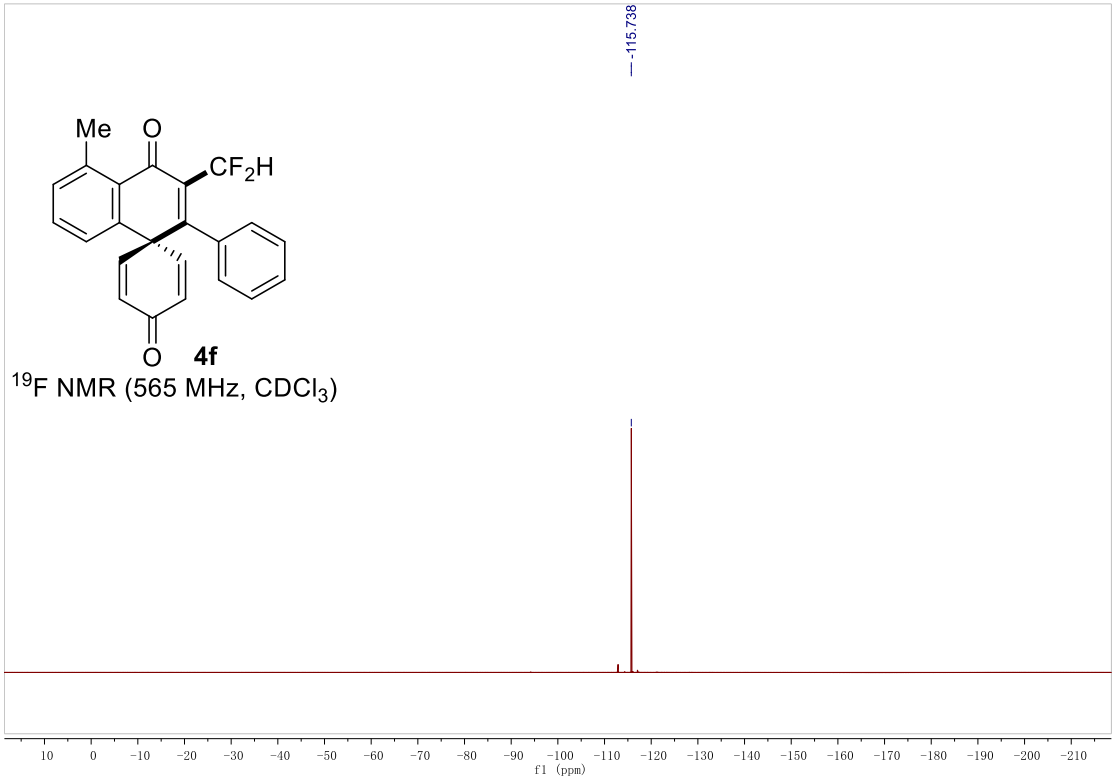


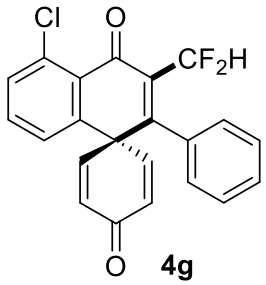


4f

^{1}H NMR (600 MHz, $CDCl_3$)

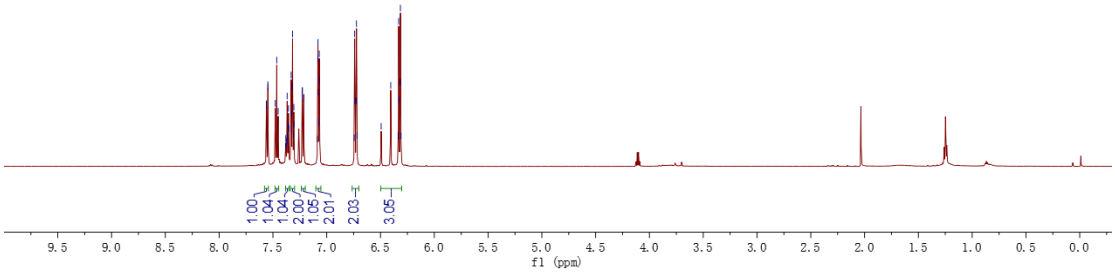

184.47
182.76
156.19
156.16
156.13
148.28
142.72
139.00
132.94
132.86
130.27
129.27
127.84
127.77
111.99
110.40
51.26
23.50
Me
O
CF2H
O
4f
13C NMR (151 MHz, CDCl3)
f1 (ppm)

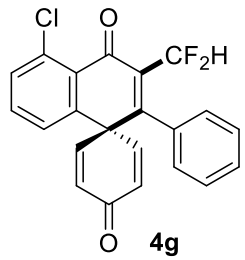

-115.738
Me
O
CF2H
O
4f
19F NMR (565 MHz, CDCl3)
f1 (ppm)



4g

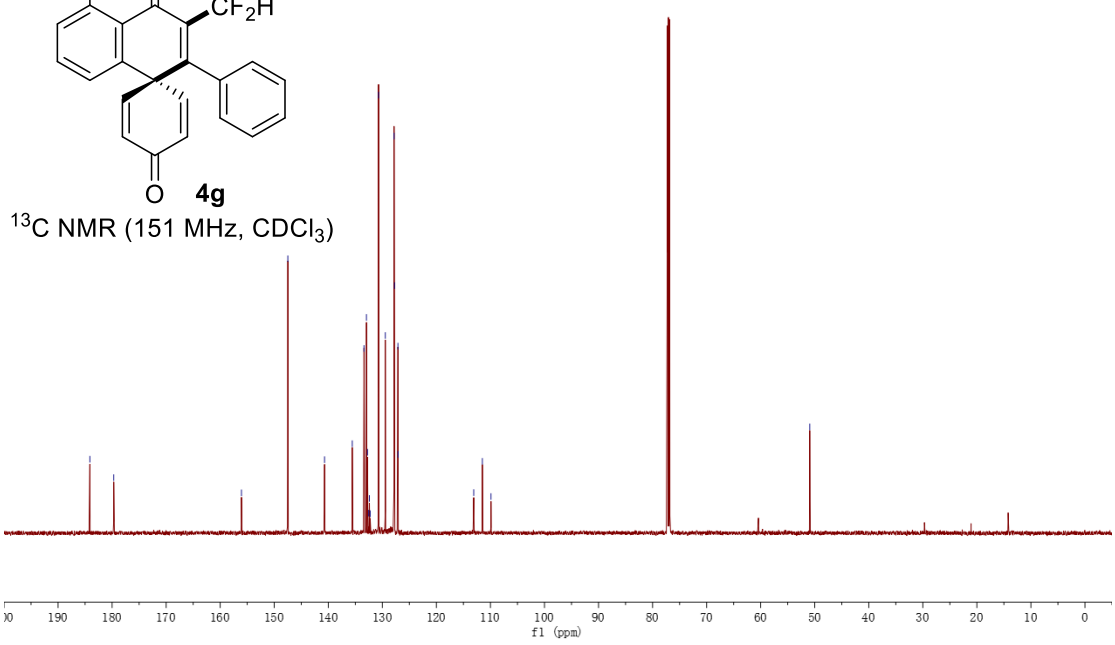
^{1}H NMR (600 MHz, $CDCl_3$)





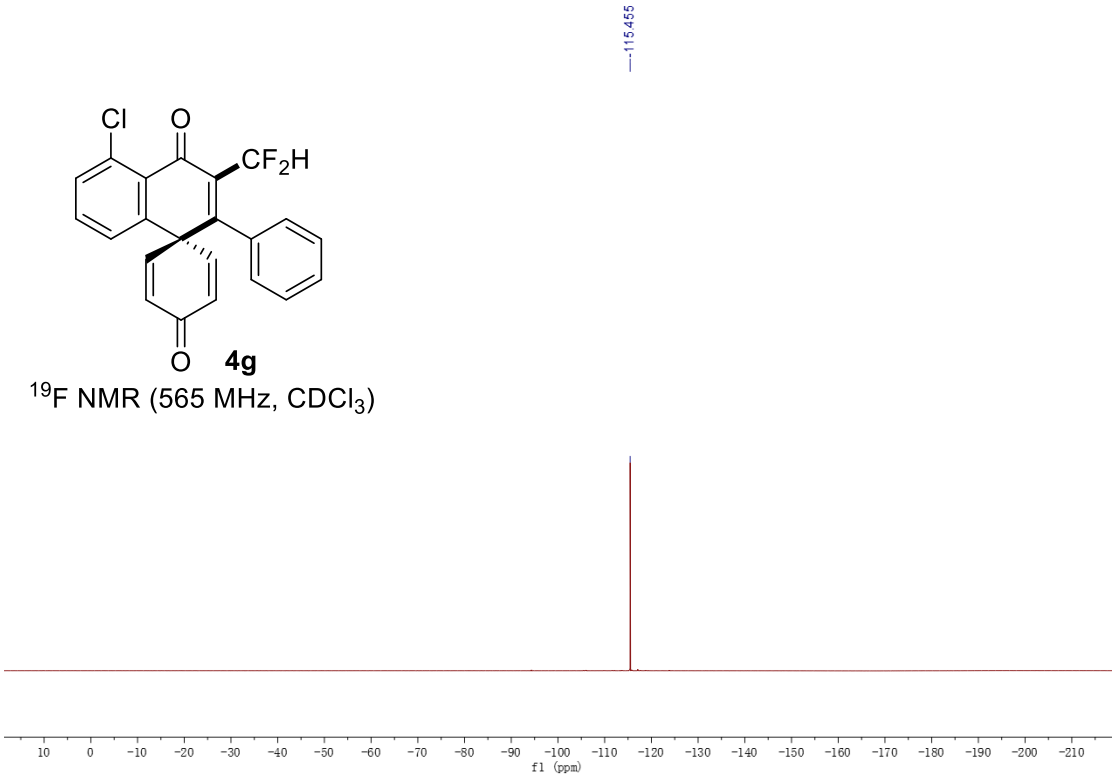
4g

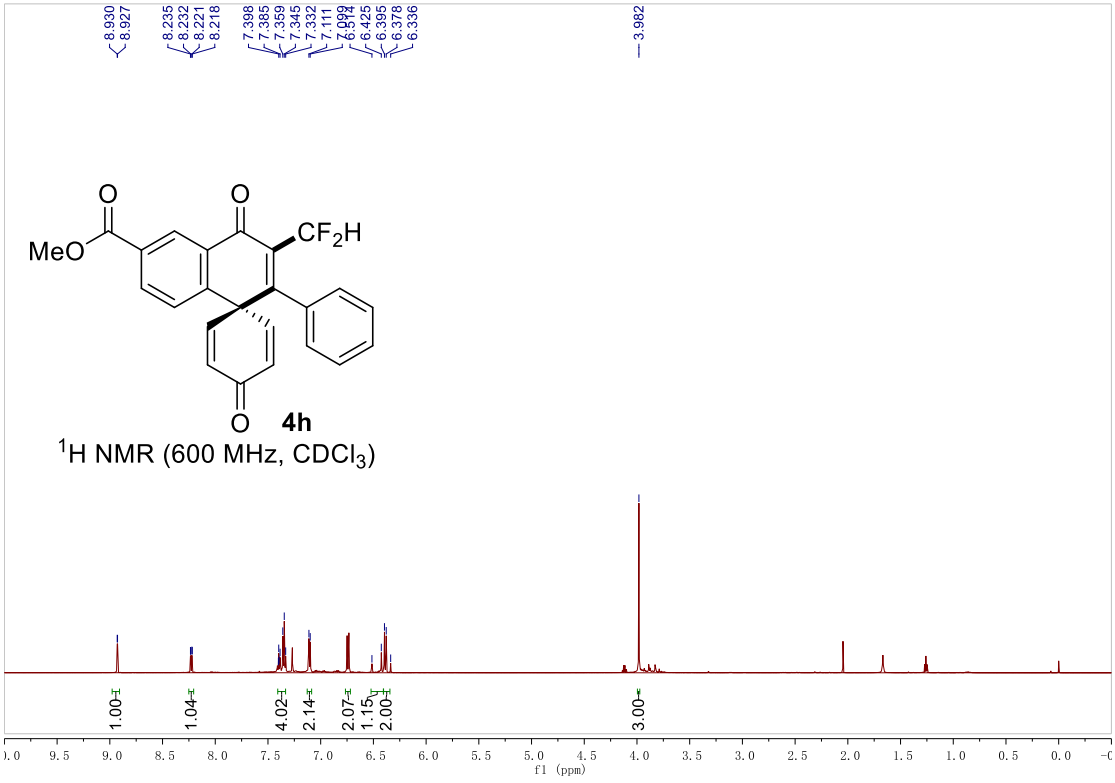
^{13}C NMR (151 MHz, $CDCl_3$)



4g

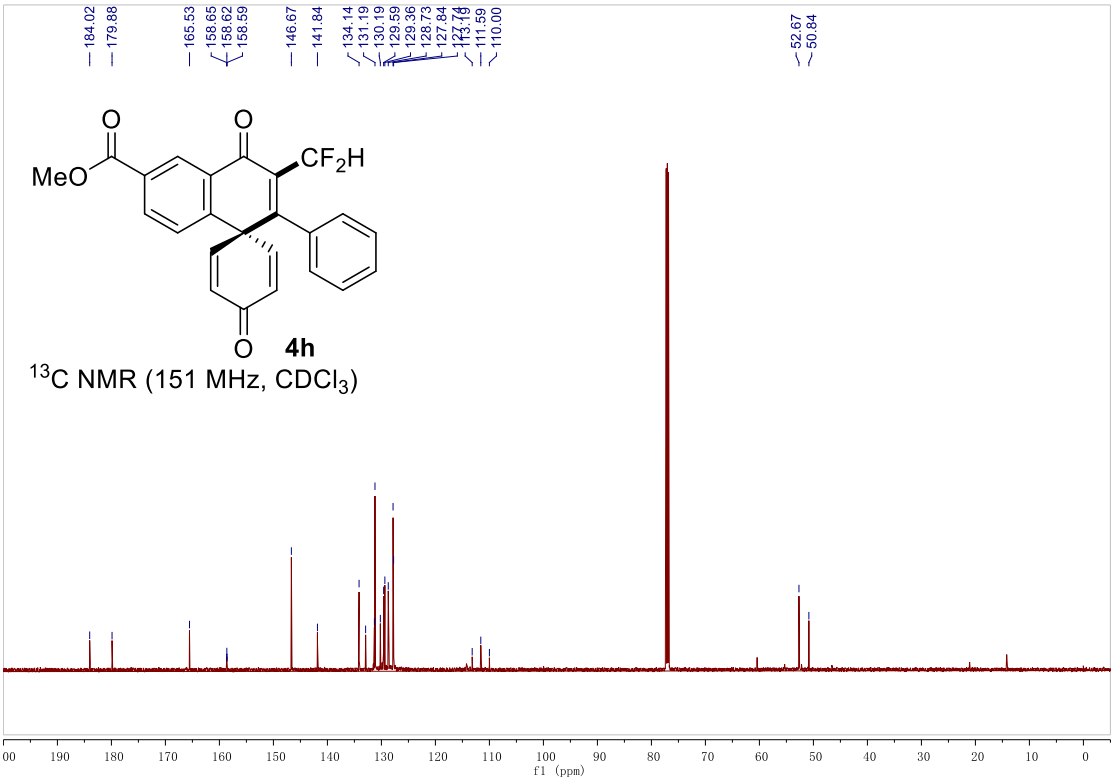
^{19}F NMR (565 MHz, $CDCl_3$)

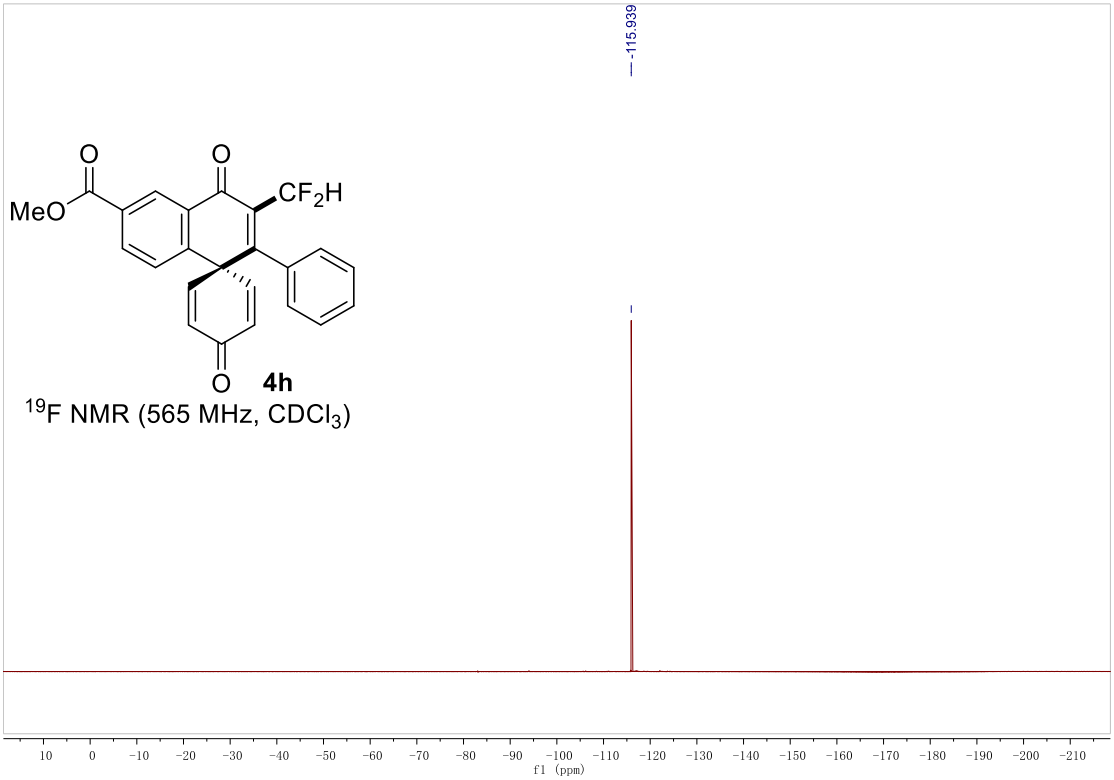


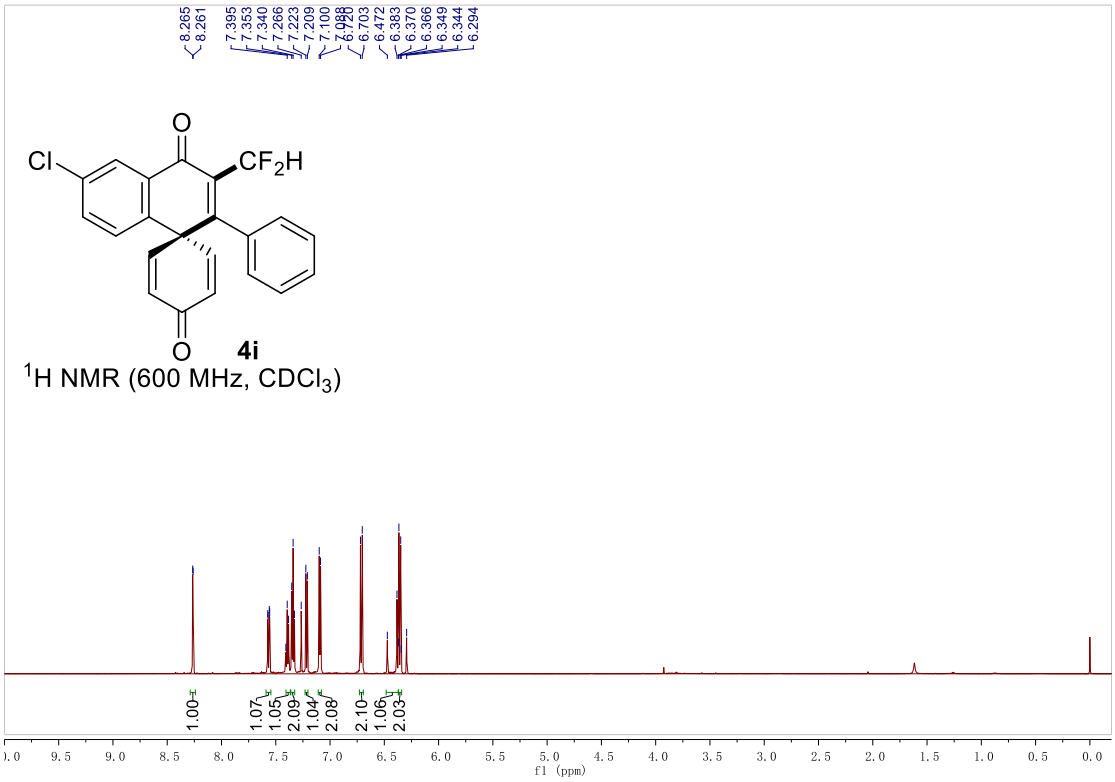


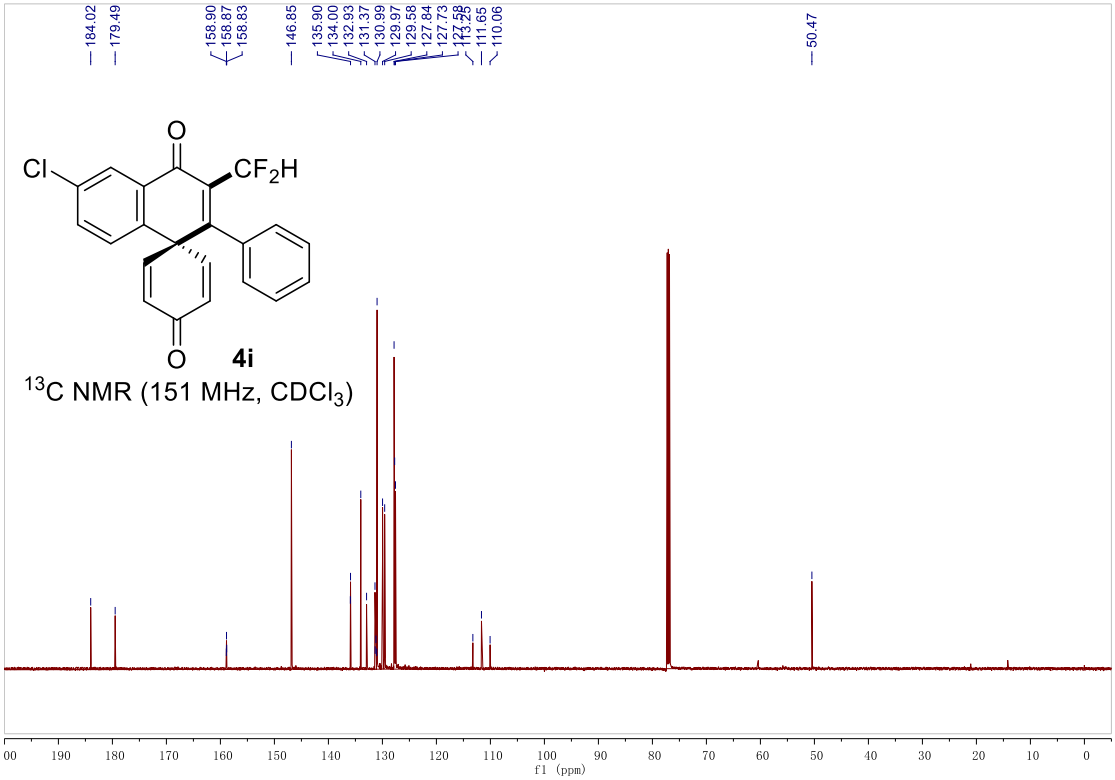
4h

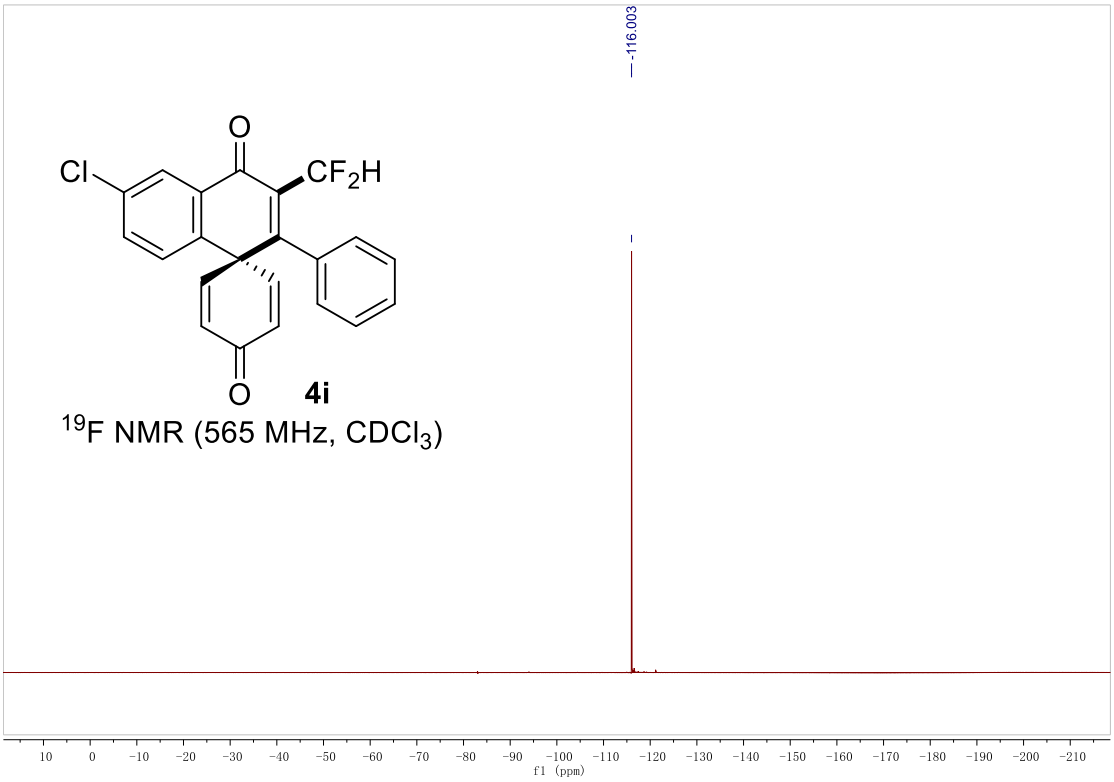
^{1}H NMR (600 MHz, $CDCl_3$)


184.02
179.88
165.53
158.65
158.62
158.59
146.67
141.84
134.14
131.19
130.19
129.59
129.36
128.73
127.84
127.74
113.19
111.59
110.00
52.67
50.84
MeO
CF2H
4h
13C NMR (151 MHz, CDCl3)
f1 (ppm)


-115.939
MeO
CF2H
4h
19F NMR (565 MHz, CDCl3)
f1 (ppm)


8.265
8.261
7.395
7.353
7.340
7.266
7.223
7.209
7.100
6.703
6.472
6.383
6.370
6.366
6.349
6.344
6.294
Cl
CF2H
O
O
4i
1H NMR (600 MHz, CDCl3)
1.00
1.07
1.05
2.09
1.04
2.08
2.10
1.06
2.03
f1 (ppm)


184.02
179.49
158.90
158.87
158.83
146.85
135.90
134.00
132.93
131.37
130.99
129.97
129.58
127.84
127.73
127.58
113.25
111.65
110.06
50.47
Cl
CF2H
O
O
4i
13C NMR (151 MHz, CDCl3)
f1 (ppm)


Cl
CF2H
O
O
4i
19F NMR (565 MHz, CDCl3)
-116.003
f1 (ppm)


8.585
8.582
7.839
7.835
7.825
7.822
7.421
7.416
7.414
7.408
7.403
7.400
7.393
7.391
7.389
7.361
7.358
7.350
7.348
7.339
7.336
7.334
7.109
7.105
7.103
7.101
7.095
7.092
7.089
6.742
6.738
6.735
6.724
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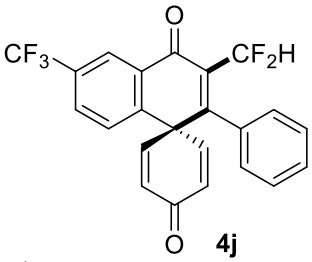
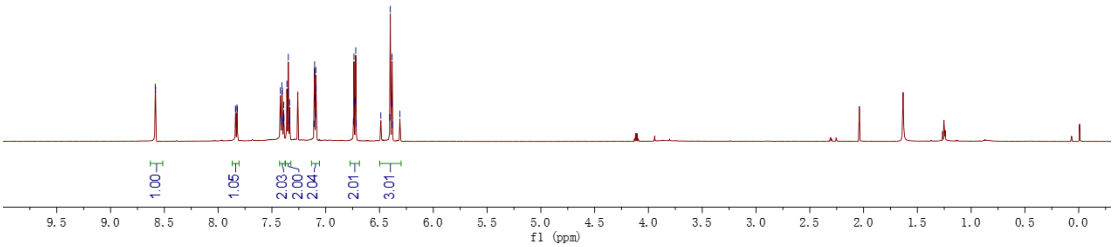
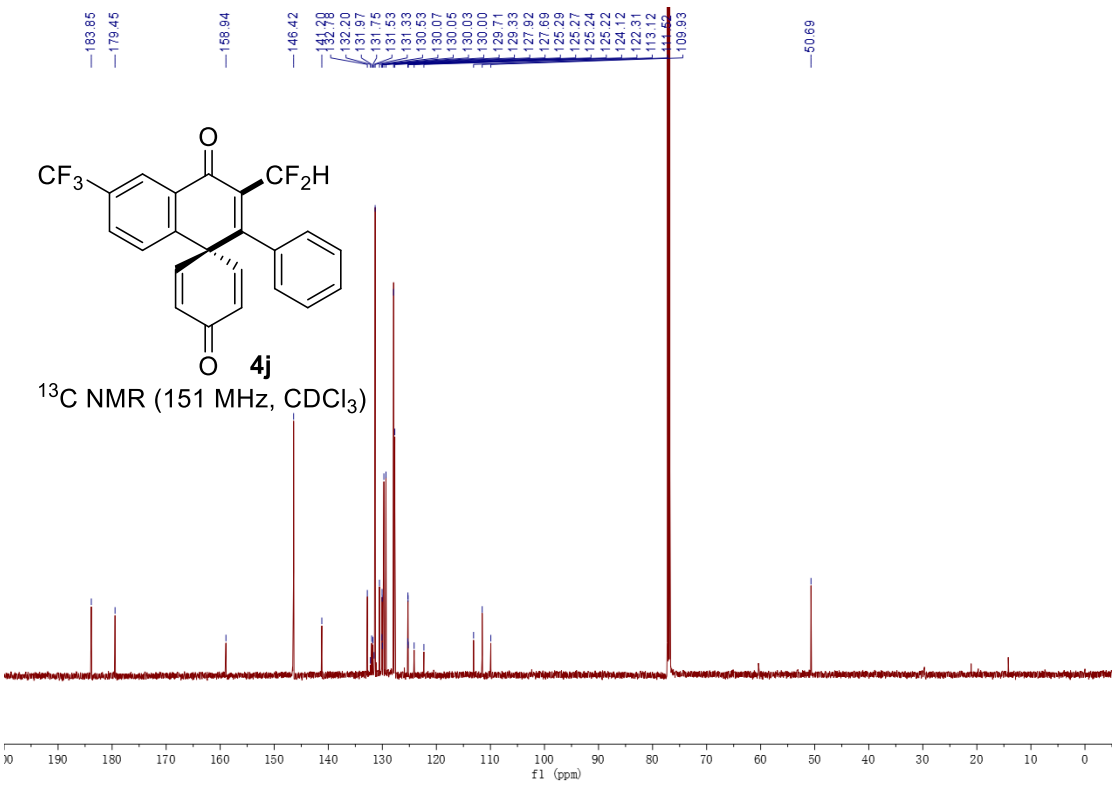
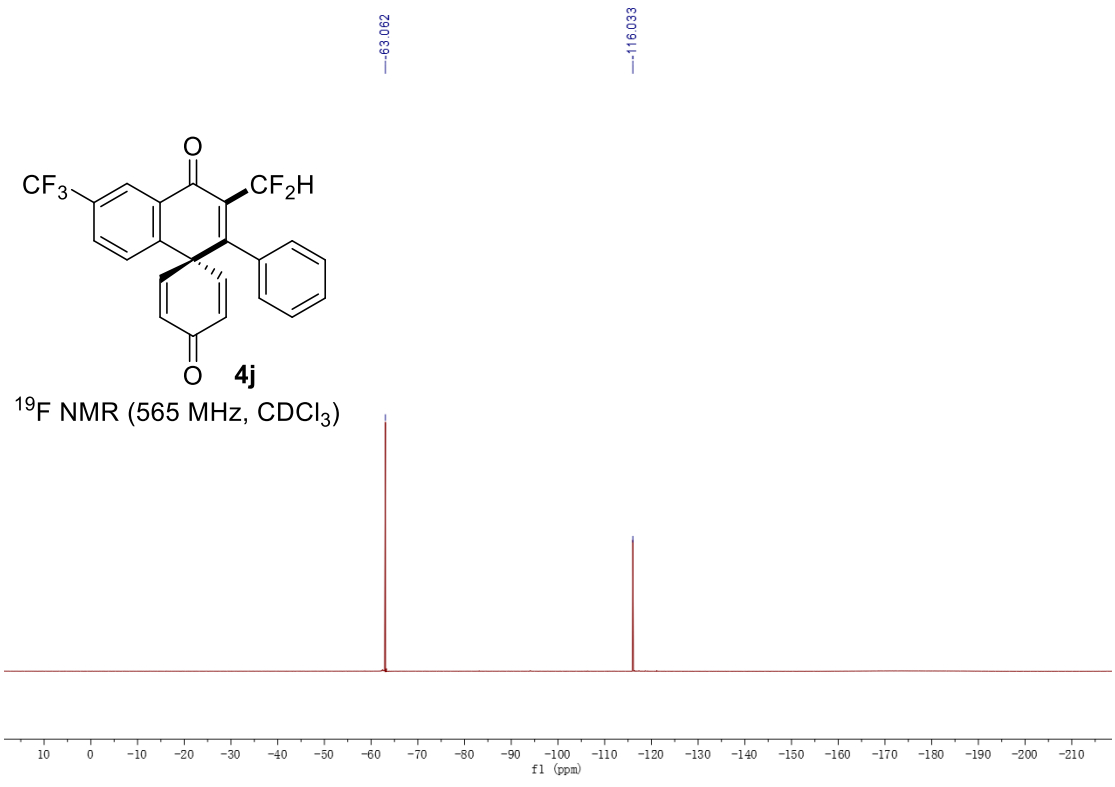
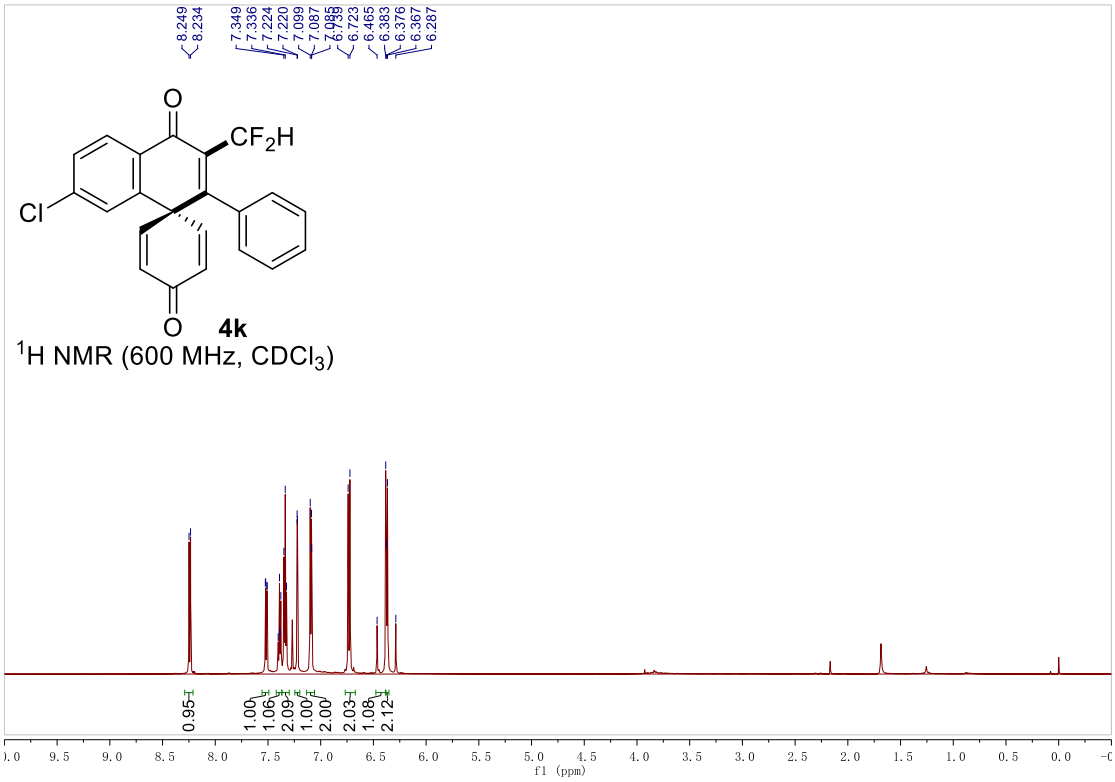
6.405
6.399
6.386
6.383
6.378
6.310

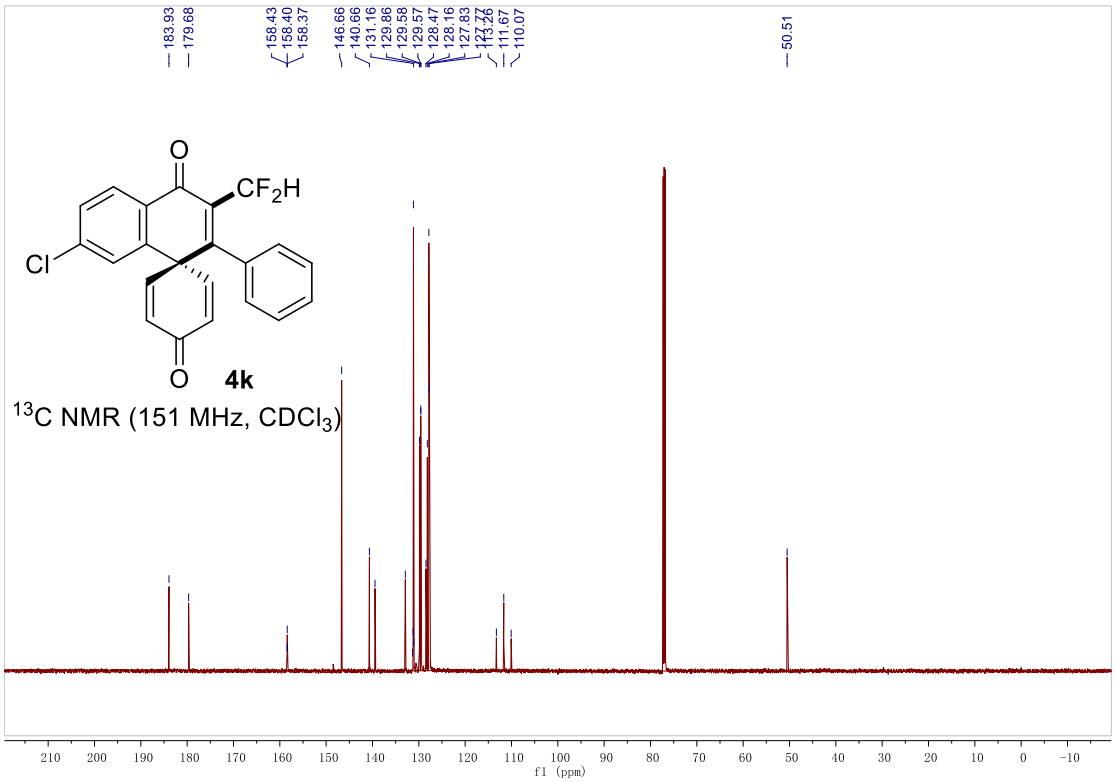

CF3
CF2H
O
O
4j
1H NMR (600 MHz, CDCl3)

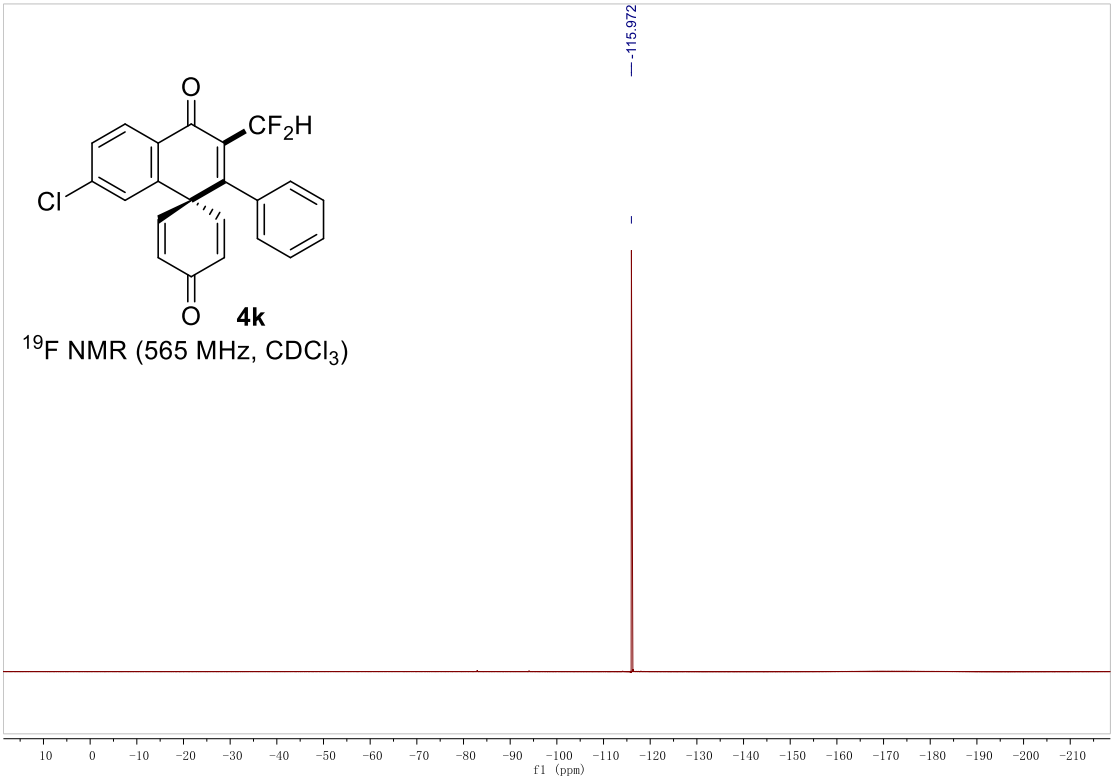

1.00
1.05
2.03
2.00
2.04
2.01
3.01
f1 (ppm)


183.85
179.45
158.94
146.42
141.20
132.78
132.20
131.97
131.75
131.53
131.33
130.53
130.07
130.05
130.03
130.00
129.71
129.33
127.92
127.69
125.29
125.27
125.24
125.22
124.12
122.31
113.12
111.62
108.93
50.69
O
CF3
CF2H
O
4j
13C NMR (151 MHz, CDCl3)
f1 (ppm)

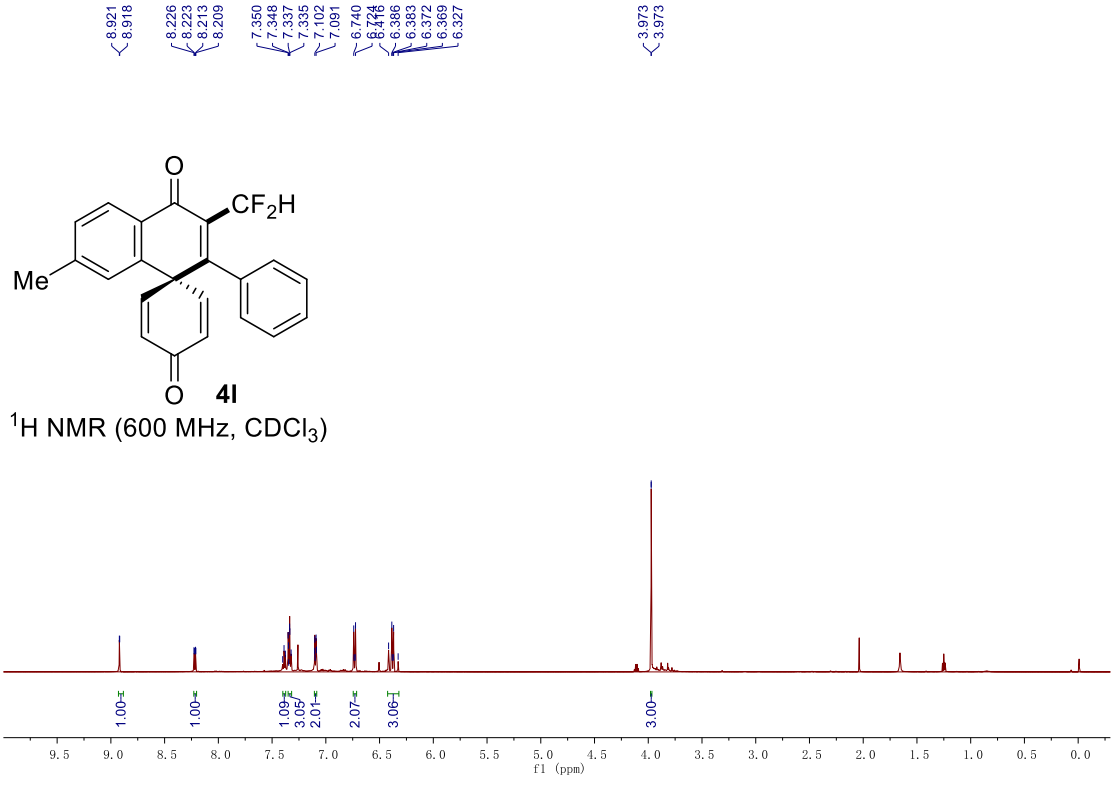

63.062
116.033
O
CF3
CF2H
O
4j
19F NMR (565 MHz, CDCl3)
f1 (ppm)


8.249
8.234
7.349
7.336
7.224
7.220
7.099
7.087
6.723
6.465
6.383
6.376
6.367
6.287
O
CF2H
Cl
O
4k
1H NMR (600 MHz, CDCl3)
0.95
1.00
1.06
2.09
1.00
2.00
2.03
1.08
2.12
f1 (ppm)

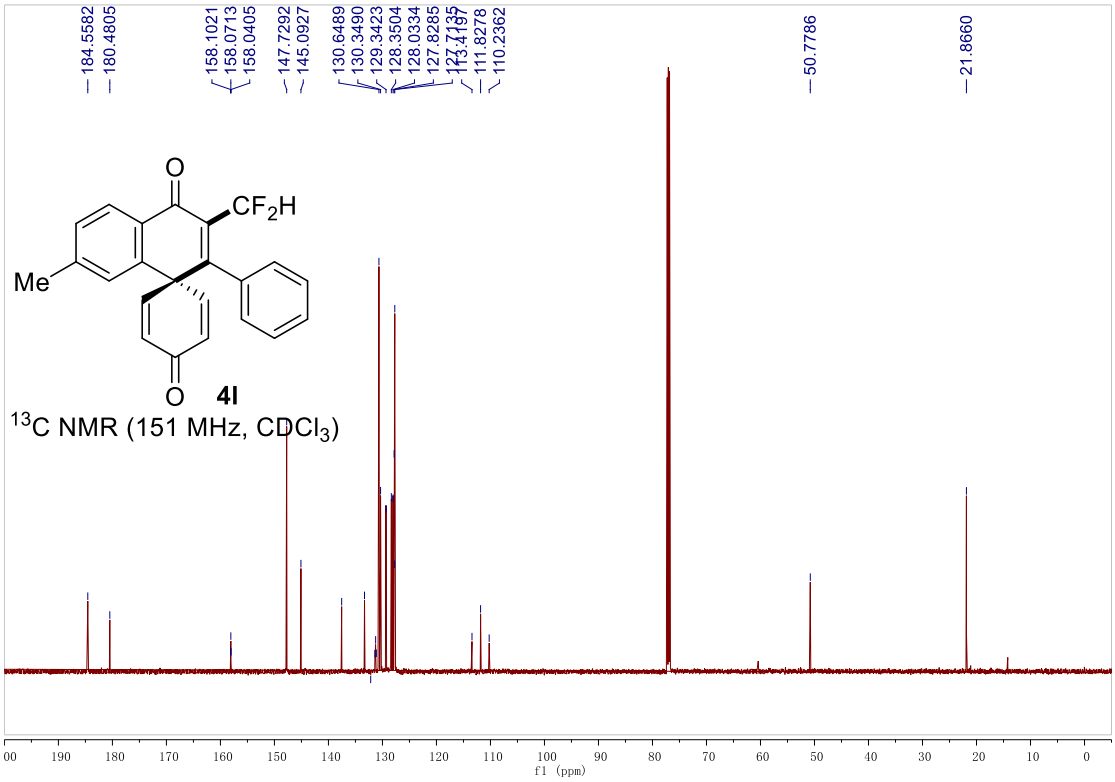

183.93
179.68
158.43
158.40
158.37
146.66
140.66
131.16
129.86
129.58
129.57
128.47
128.16
127.83
111.67
110.07
50.51
O
CF2H
Cl
O
4k
13C NMR (151 MHz, CDCl3)
f1 (ppm)

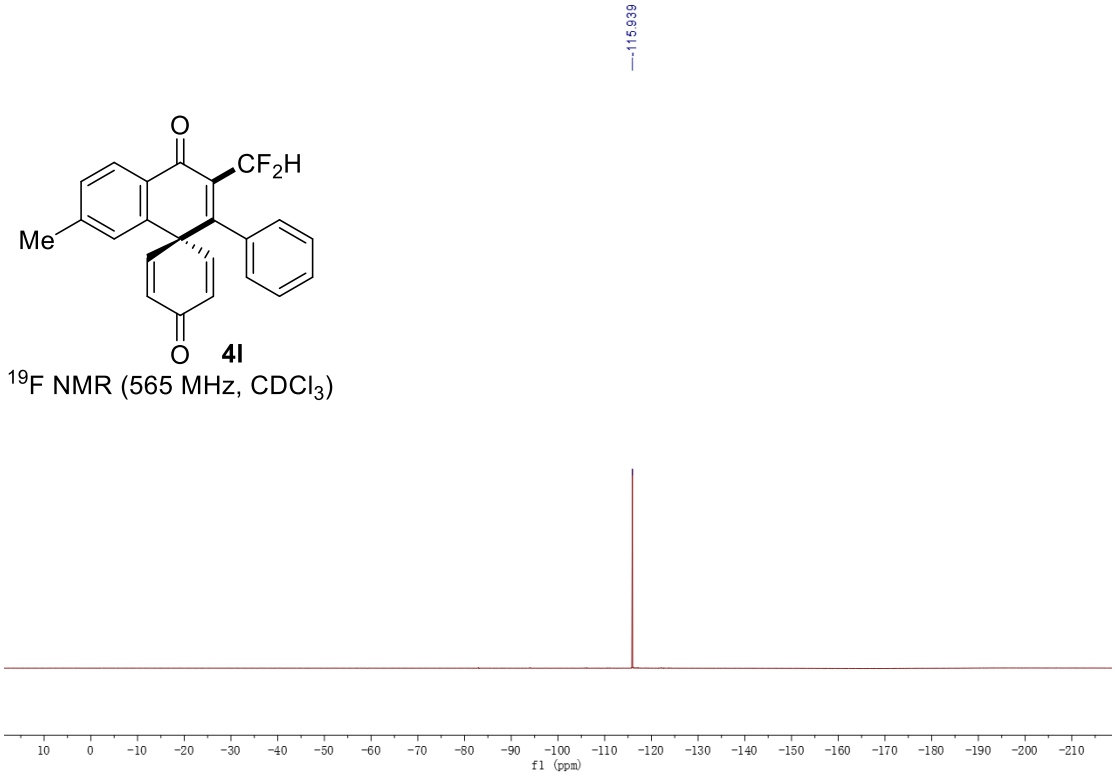


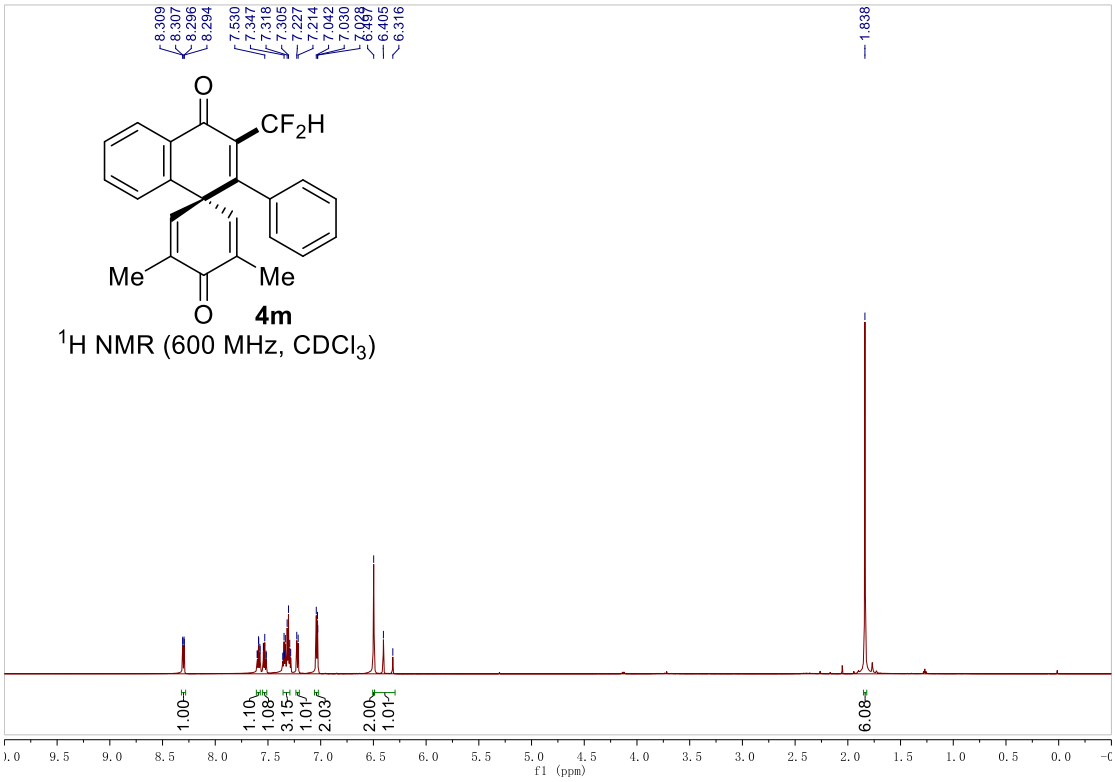
^{19}F NMR (565 MHz, $CDCl_3$)

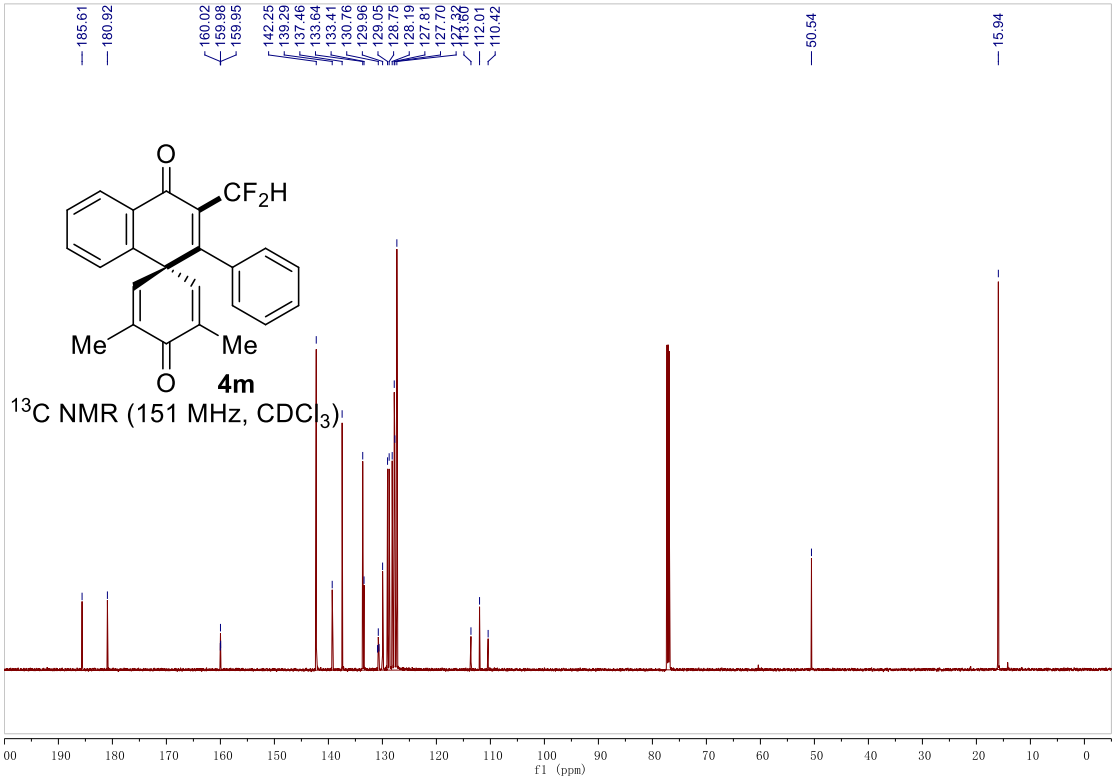


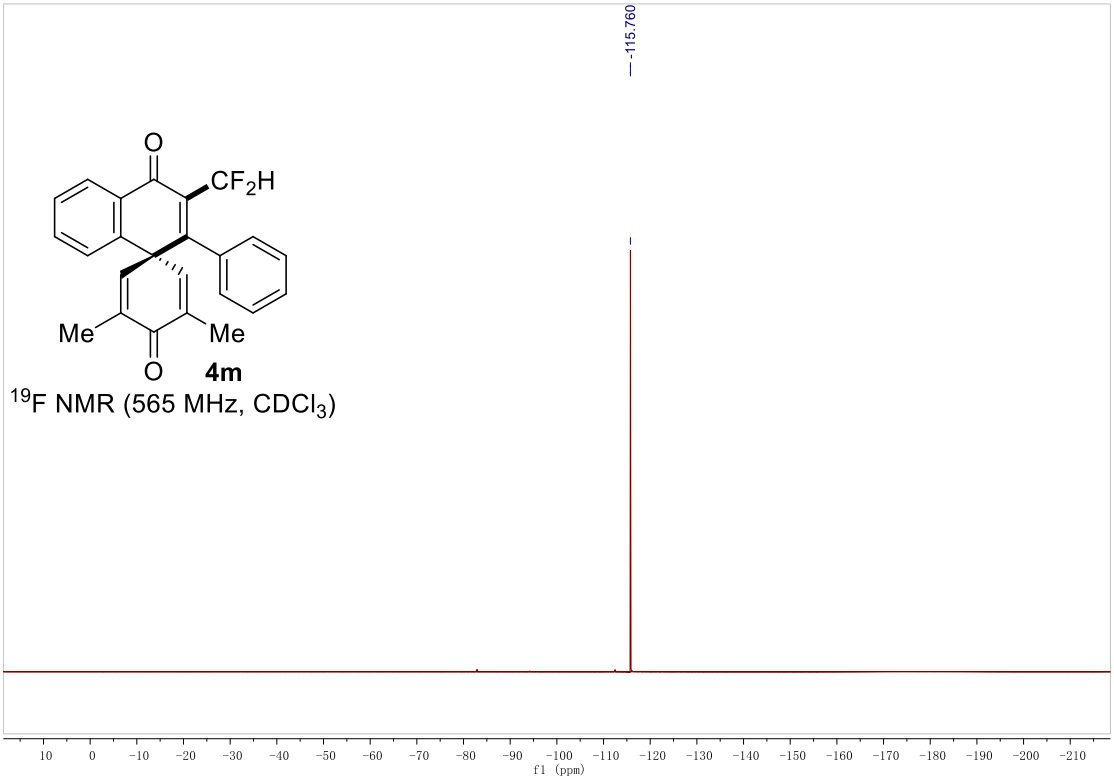
^{1}H NMR (600 MHz, $CDCl_3$)

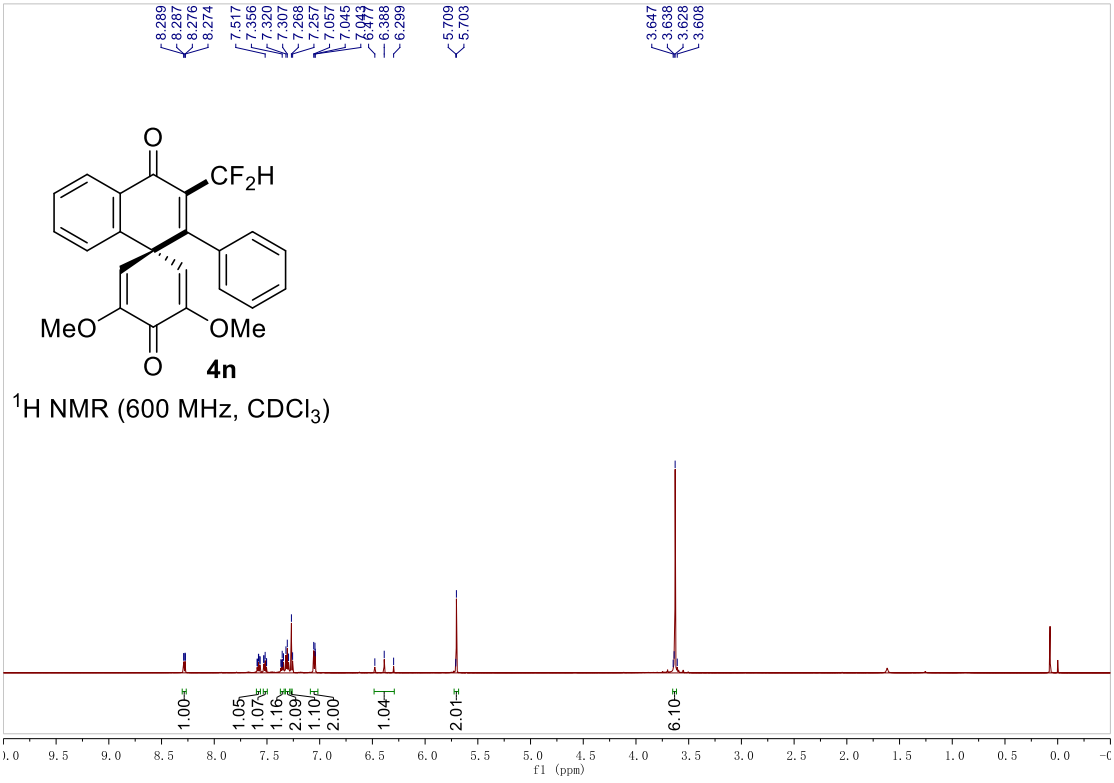

184.5582
180.4805
158.1021
158.0713
158.0405
147.7292
145.0927
130.6489
130.3490
129.3423
128.3504
128.0334
127.8285
123.4197
111.8278
110.2362
50.7786
21.8660
O
CF2H
Me
O
4l
13C NMR (151 MHz, CDCl3)
f1 (ppm)

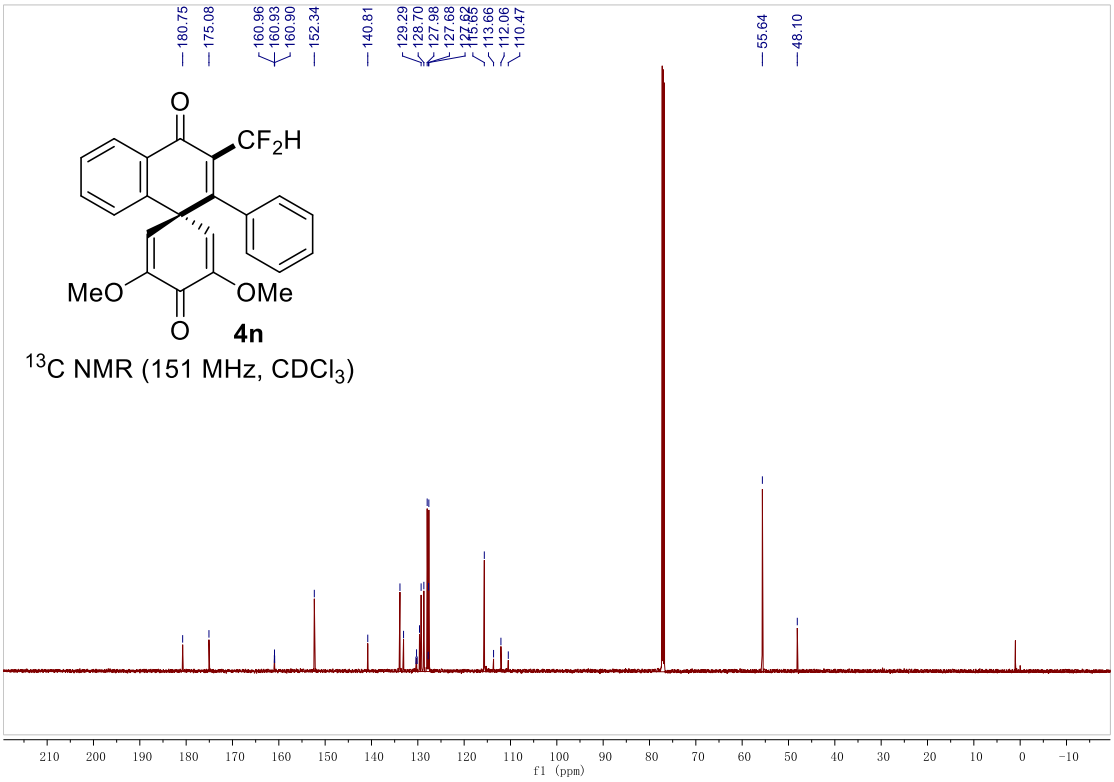

115.939
O
CF2H
Me
O
4l
19F NMR (565 MHz, CDCl3)
f1 (ppm)

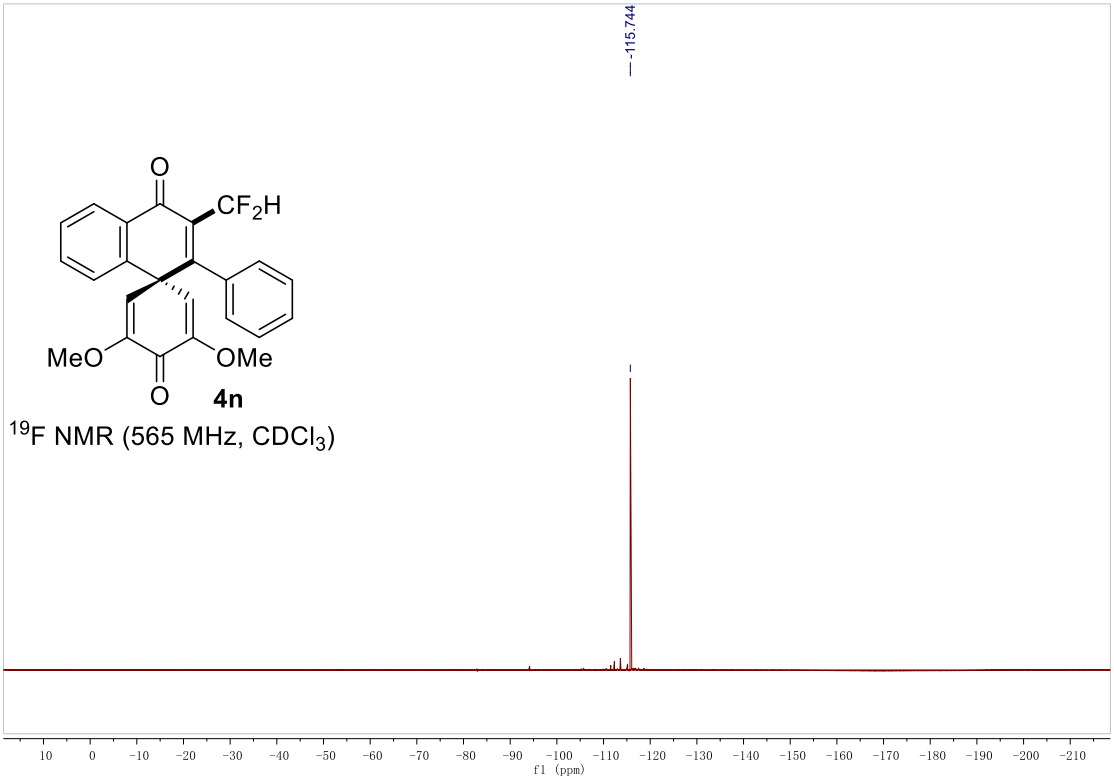

8.309
8.307
8.296
8.294
7.530
7.347
7.318
7.305
7.227
7.214
7.042
7.030
6.405
6.316
1.838
O
CF2H
Me
Me
O
4m
1H NMR (600 MHz, CDCl3)
1.00
1.10
1.08
3.15
1.01
2.03
2.00
1.01
6.08
f1 (ppm)

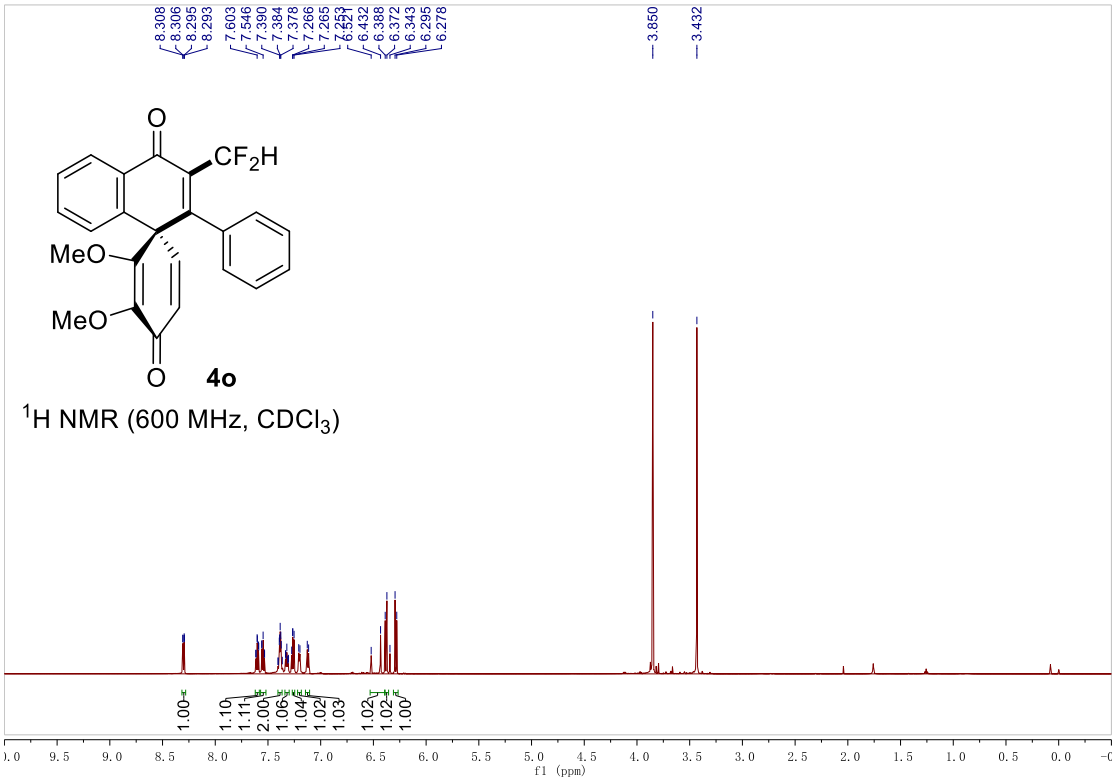

185.61
180.92
160.02
159.98
159.95
142.25
139.29
137.46
133.64
133.41
130.76
129.96
129.05
128.75
128.19
127.81
127.70
112.01
110.42
50.54
15.94
O
CF2H
Me
Me
O
4m
13C NMR (151 MHz, CDCl3)
f1 (ppm)

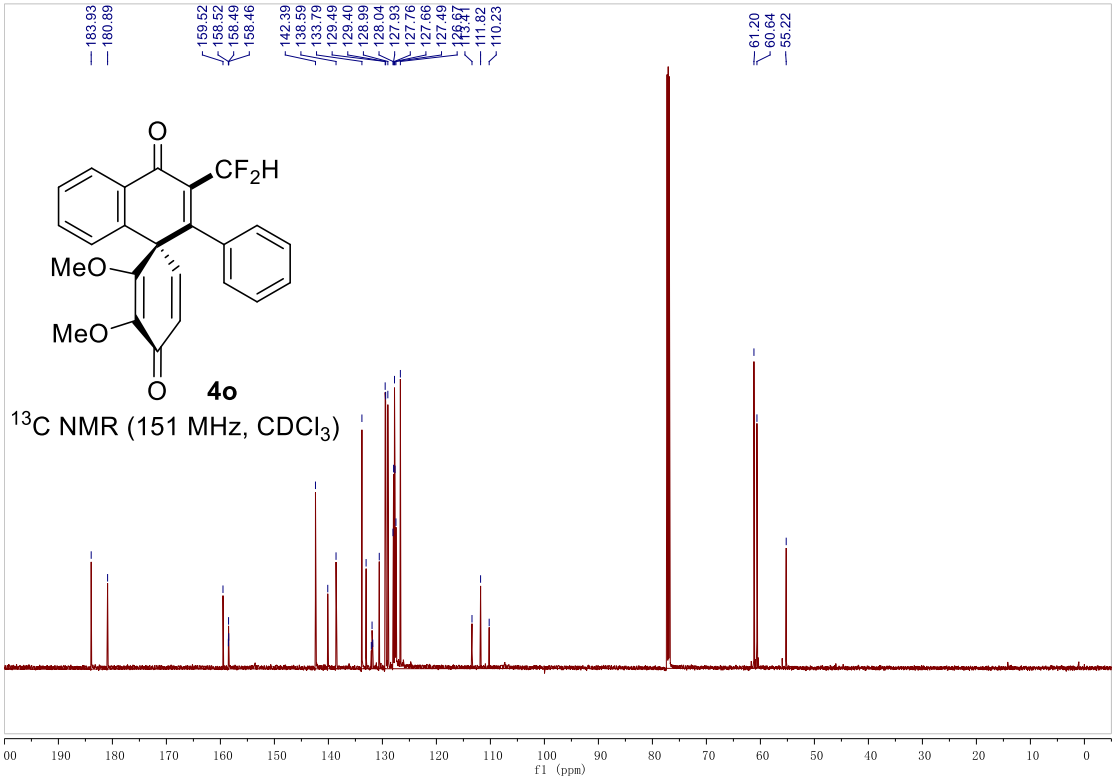

-115.760
O
CF2H
Me
Me
O
4m
19F NMR (565 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

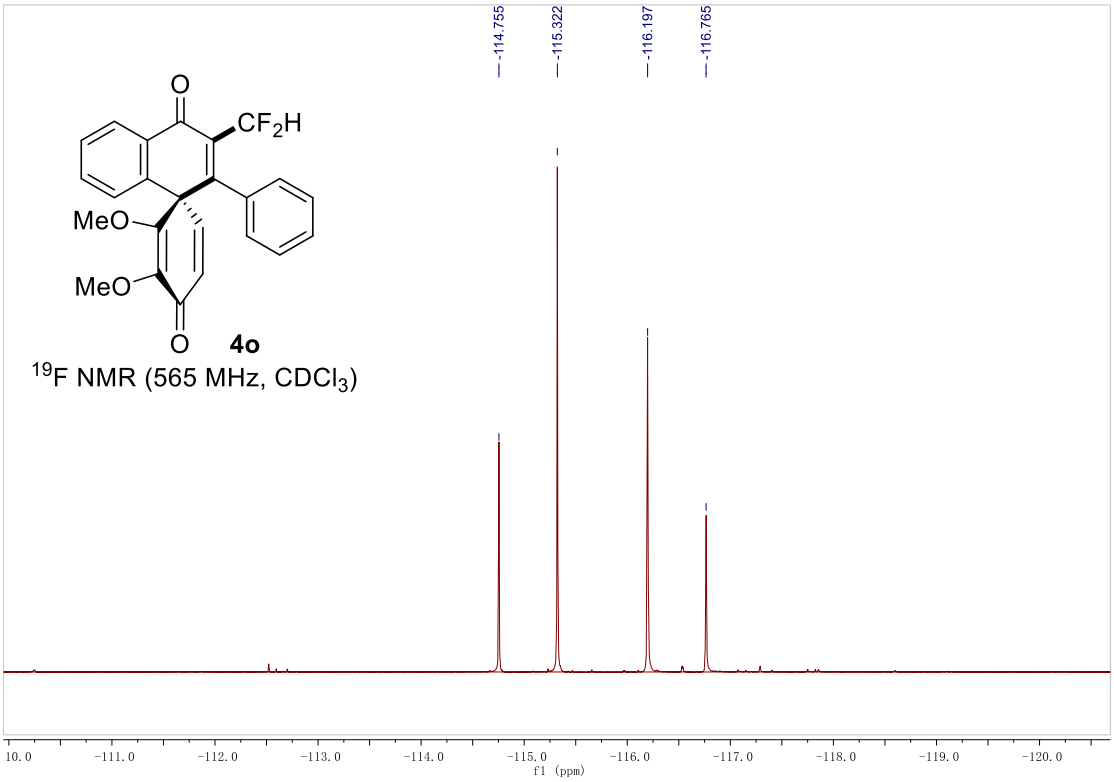

8.289
8.287
8.276
8.274
7.517
7.356
7.320
7.307
7.268
7.257
7.057
7.045
7.043
6.477
6.388
6.299
5.709
5.703
3.647
3.638
3.628
3.608
O
CF2H
MeO
OMe
O
4n
1H NMR (600 MHz, CDCl3)
1.00
1.05
1.07
1.16
2.09
1.10
2.00
1.04
2.01
6.10
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

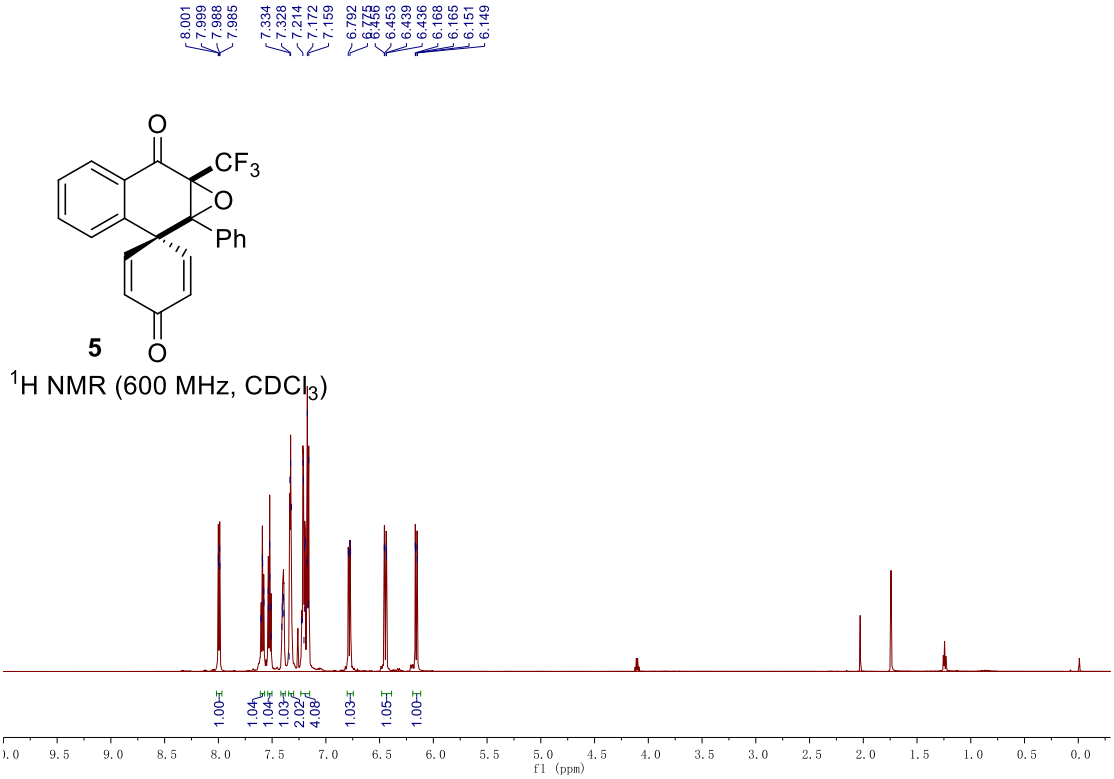

180.75
175.08
160.96
160.93
160.90
152.34
140.81
129.29
128.70
127.98
127.68
113.66
112.06
110.47
55.64
48.10
O
CF2H
MeO
OMe
O
4n
13C NMR (151 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

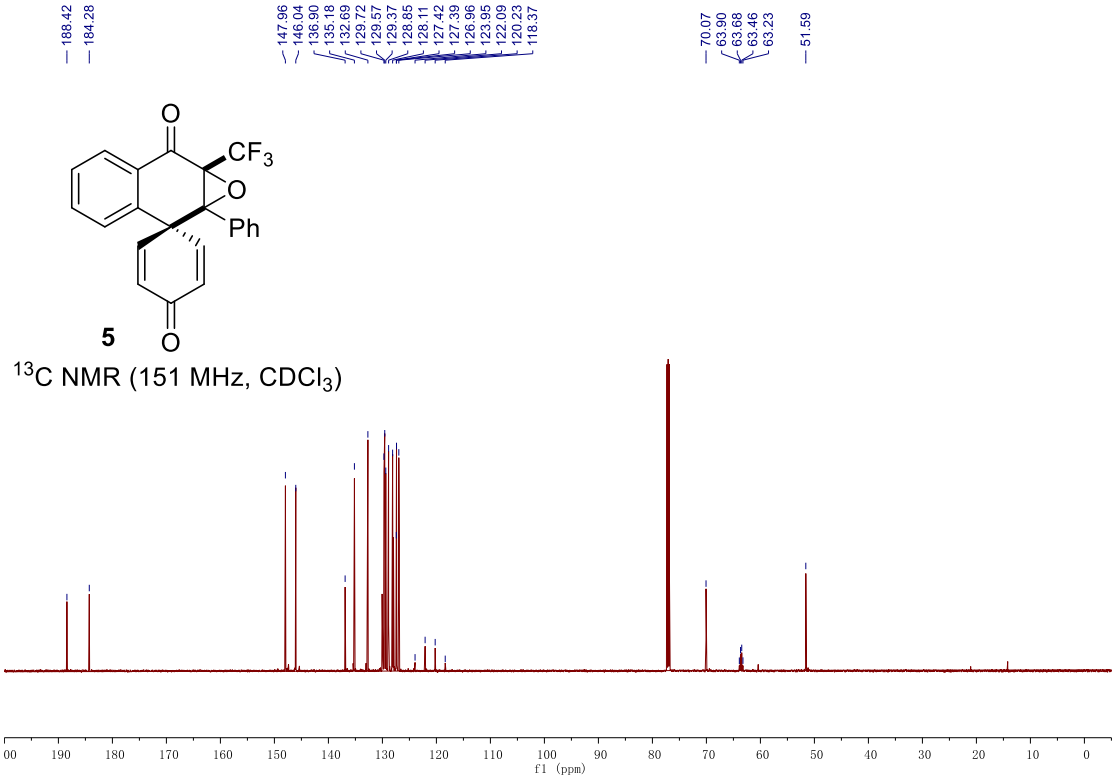

−115.744
O
CF2H
MeO
OMe
O
4n
19F NMR (565 MHz, CDCl3)
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

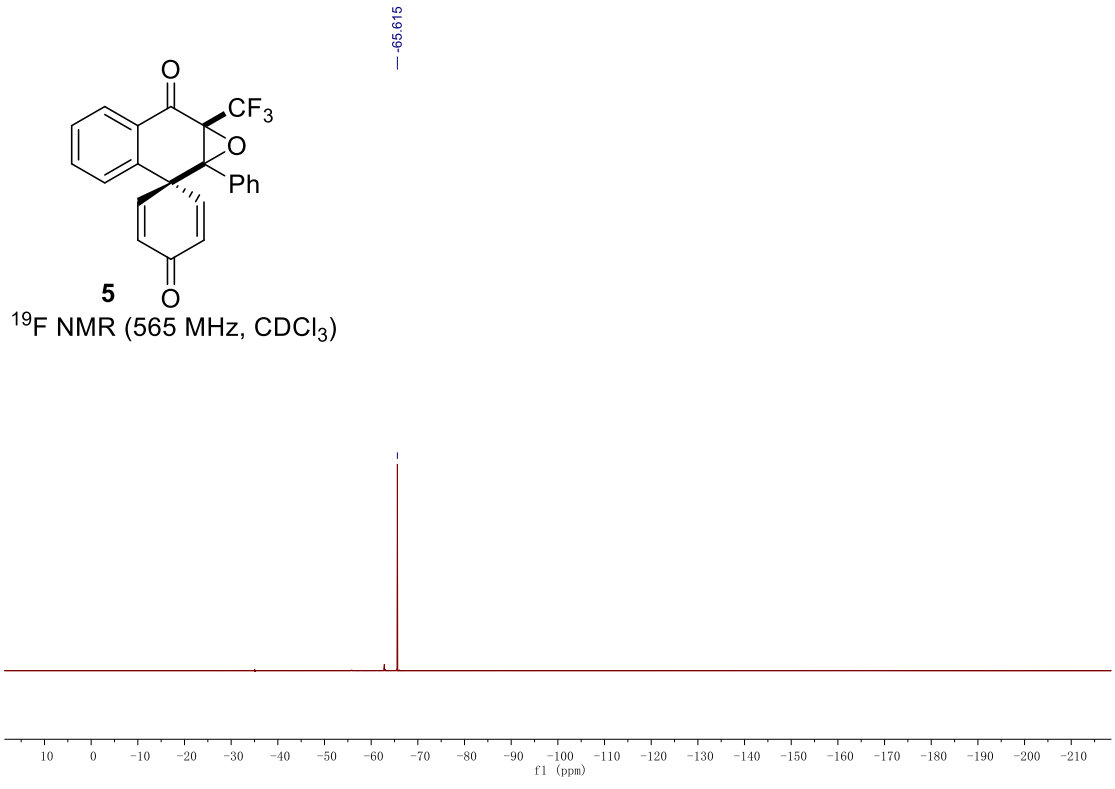

8.308
8.306
8.295
8.293
7.603
7.546
7.390
7.384
7.378
7.266
7.265
6.432
6.388
6.372
6.343
6.295
6.278
3.850
3.432
O
CF2H
MeO
MeO
O
4o
1H NMR (600 MHz, CDCl3)
1.00
1.10
1.11
2.00
1.06
1.04
1.02
1.03
1.02
1.02
1.00
f1 (ppm)

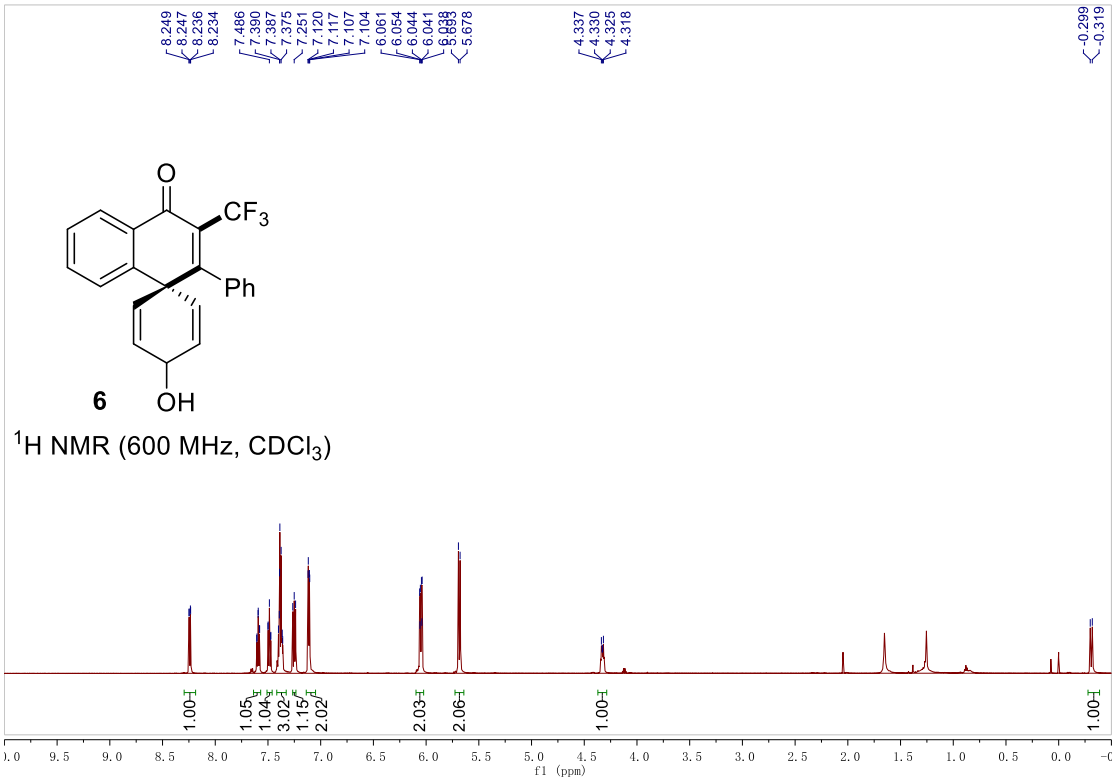

183.93
180.89
159.52
158.52
158.49
158.46
142.39
138.59
133.79
129.49
129.40
128.99
128.04
127.93
127.76
127.66
127.49
113.41
111.82
110.23
61.20
60.64
55.22
O
CF2H
MeO
MeO
O
4o
13C NMR (151 MHz, CDCl3)
f1 (ppm)

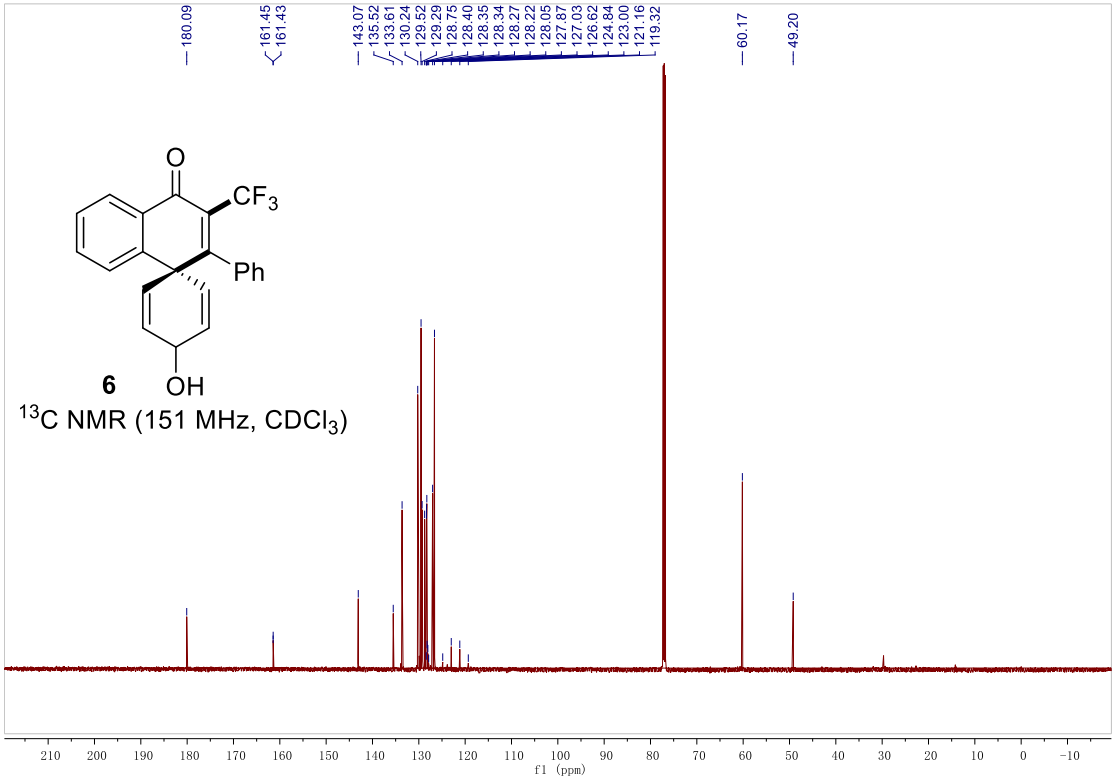

-114.755
-115.322
-116.197
-116.765
O
CF2H
MeO
MeO
O
4o
19F NMR (565 MHz, CDCl3)
10.0
-111.0
-112.0
-113.0
-114.0
-115.0
-116.0
-117.0
-118.0
-119.0
-120.0
f1 (ppm)

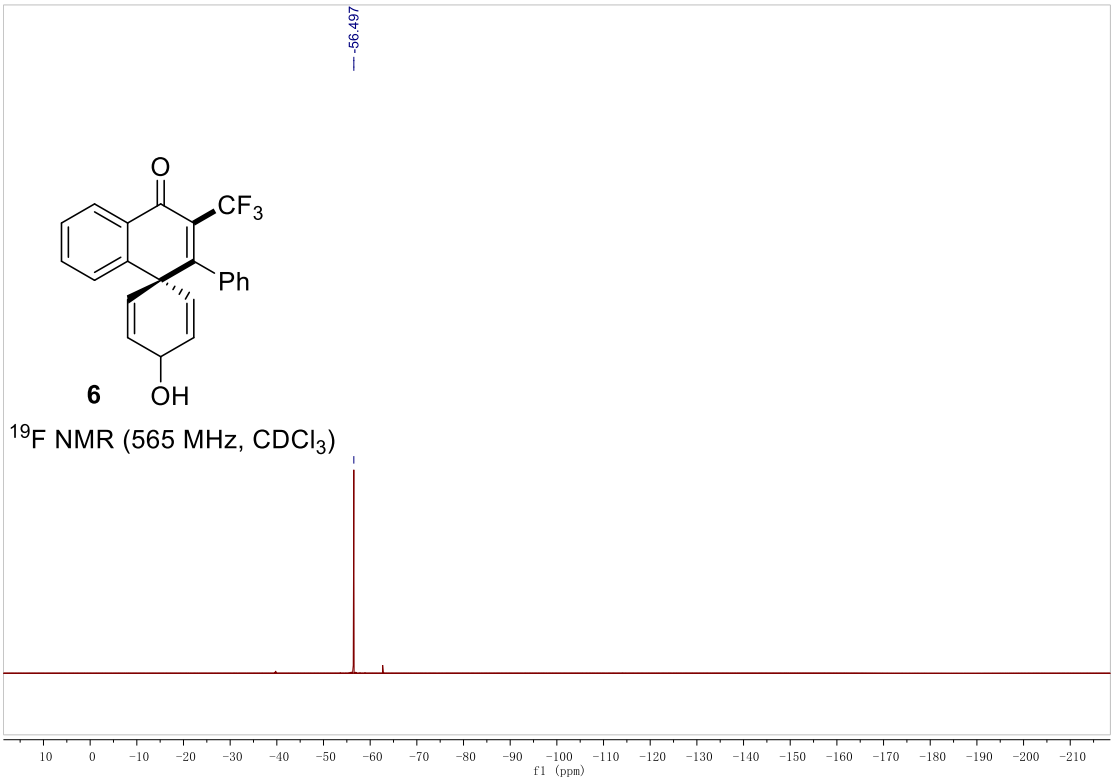

8.001
7.999
7.988
7.985
7.334
7.328
7.214
7.172
7.159
6.792
6.775
6.455
6.453
6.439
6.436
6.168
6.165
6.151
6.149
O
CF3
O
Ph
O
5
1H NMR (600 MHz, CDCl3)
1.00
1.04
1.04
1.03
2.02
4.08
1.03
1.05
1.00
f1 (ppm)

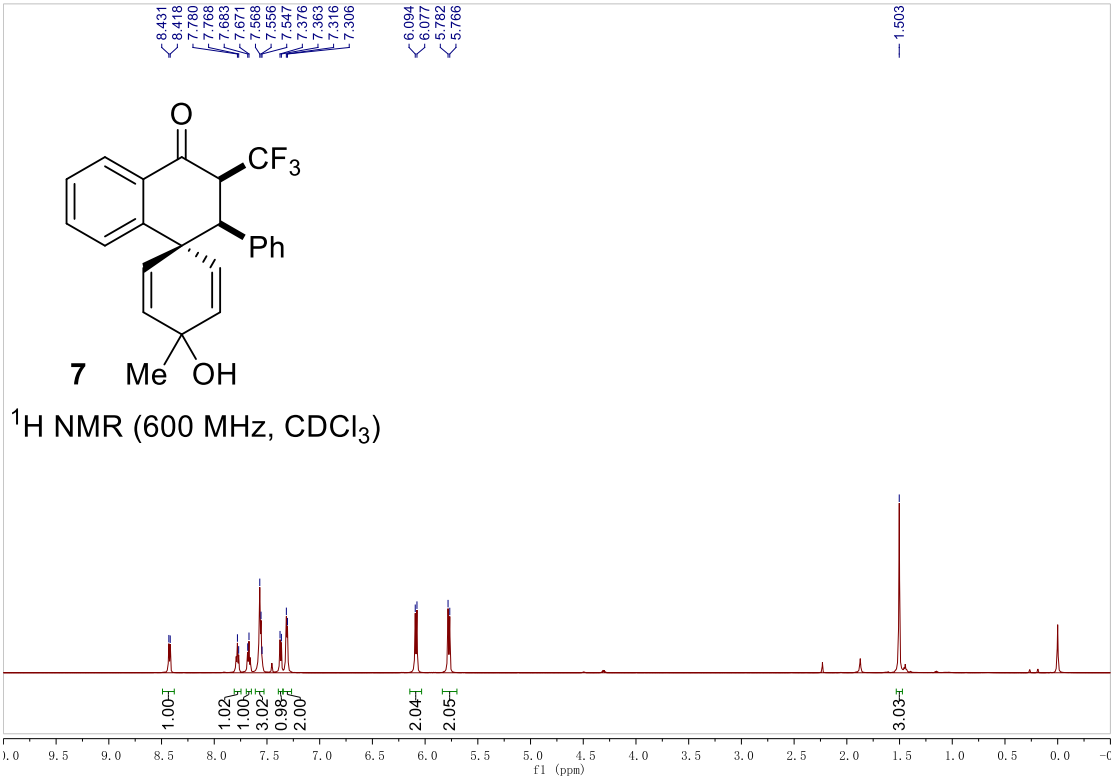

188.42
184.28
147.96
146.04
136.90
135.18
132.69
129.72
129.57
129.37
128.85
128.11
127.42
127.39
126.96
123.95
122.09
120.23
118.37
70.07
63.90
63.68
63.46
63.23
51.59
O
CF3
O
Ph
O
5
13C NMR (151 MHz, CDCl3)
f1 (ppm)

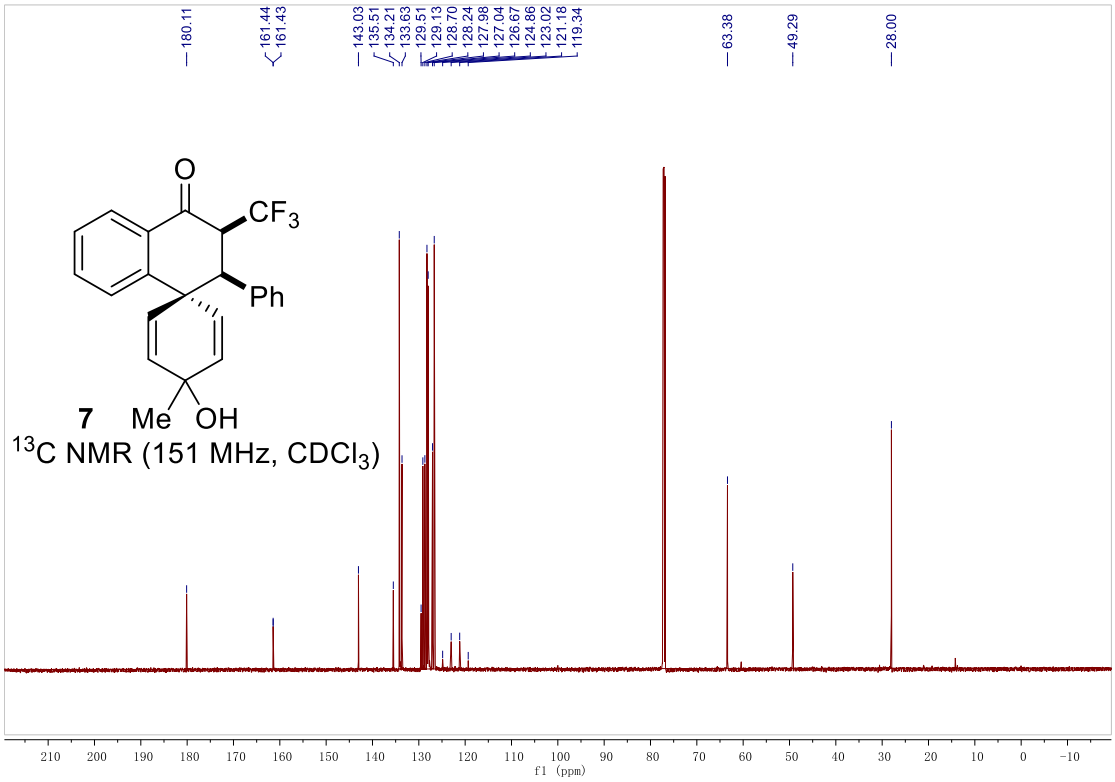

-65.615
O
CF3
O
Ph
O
5
19F NMR (565 MHz, CDCl3)
f1 (ppm)

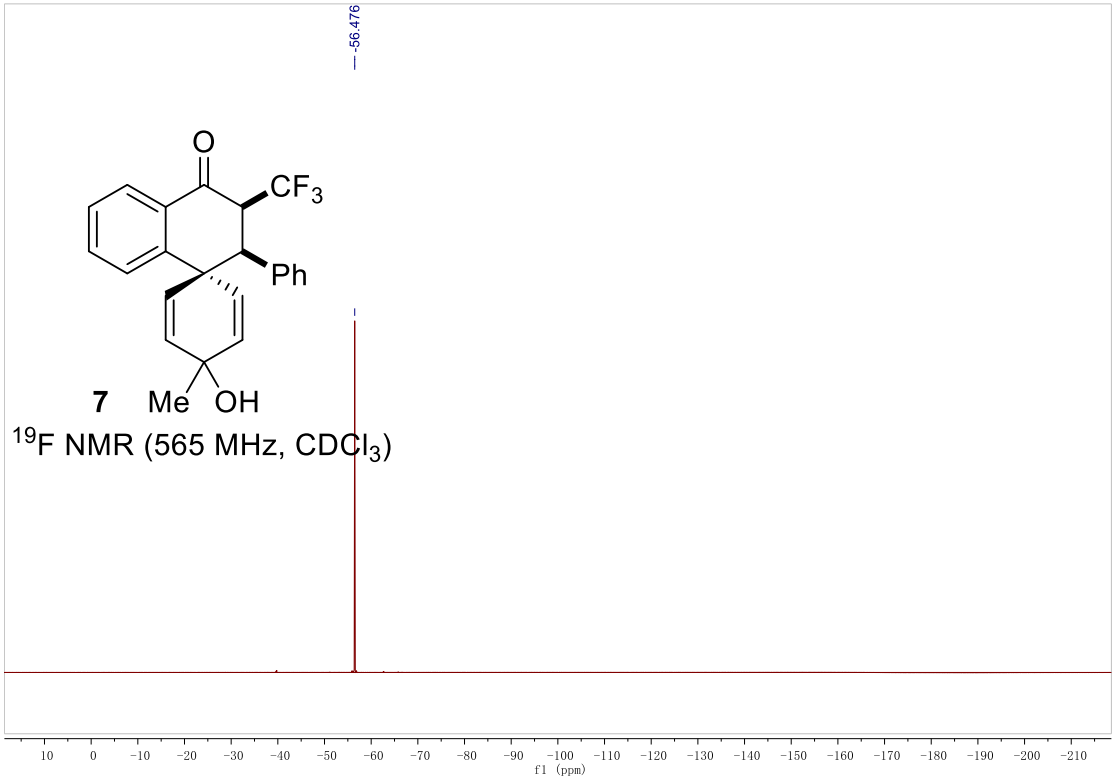

8.249
8.247
8.236
8.234
7.486
7.390
7.387
7.375
7.251
7.120
7.117
7.107
7.104
6.061
6.054
6.044
6.041
5.678
4.337
4.330
4.325
4.318
-0.299
-0.319
O
CF3
Ph
OH
6
1H NMR (600 MHz, CDCl3)
1.00
1.05
1.04
3.02
1.15
2.02
2.03
2.06
1.00
1.00
f1 (ppm)

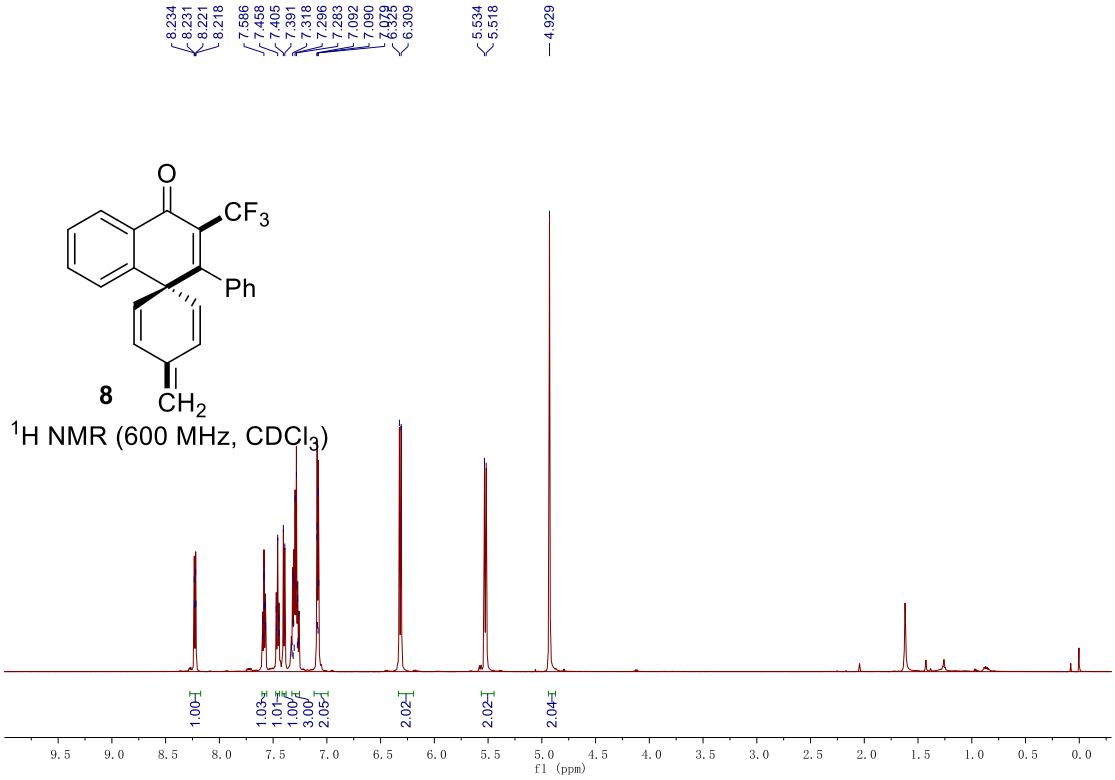

180.09
161.45
161.43
143.07
135.52
133.61
130.24
129.52
129.29
128.75
128.40
128.35
128.34
128.27
128.22
128.05
127.87
127.03
126.62
124.84
123.00
121.16
119.32
60.17
49.20
O
CF3
Ph
OH
6
13C NMR (151 MHz, CDCl3)
f1 (ppm)

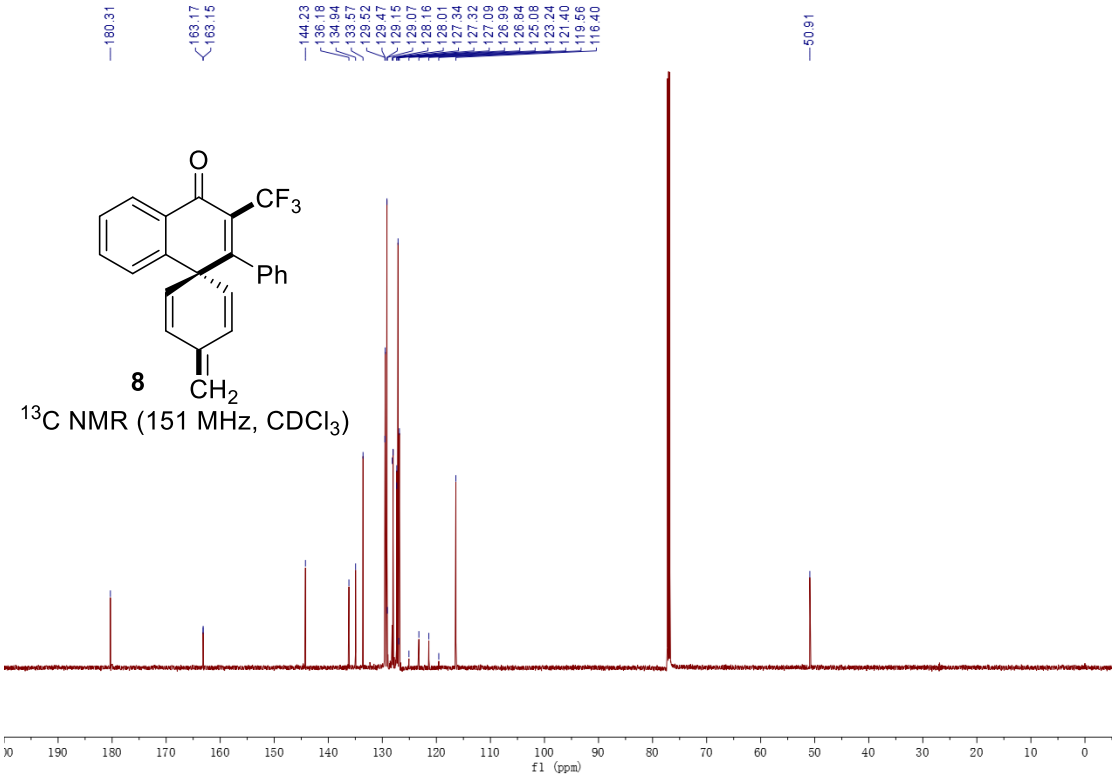

-56.497
O
CF3
Ph
6
OH
19F NMR (565 MHz, CDCl3)
f1 (ppm)


8.431
8.418
7.780
7.768
7.683
7.671
7.568
7.556
7.547
7.376
7.363
7.316
7.306
6.094
6.077
5.782
5.766
1.503
O
CF3
Ph
7
Me
OH
1H NMR (600 MHz, CDCl3)
1.00
1.02
1.00
3.02
0.98
2.00
2.04
2.05
3.03
f1 (ppm)


180.11
161.44
161.43
143.03
135.51
134.21
133.63
129.51
129.13
128.70
128.24
127.98
127.04
126.67
124.86
123.02
121.18
119.34
63.38
49.29
28.00
O
CF3
Ph
7 Me OH
13C NMR (151 MHz, CDCl3)
f1 (ppm)

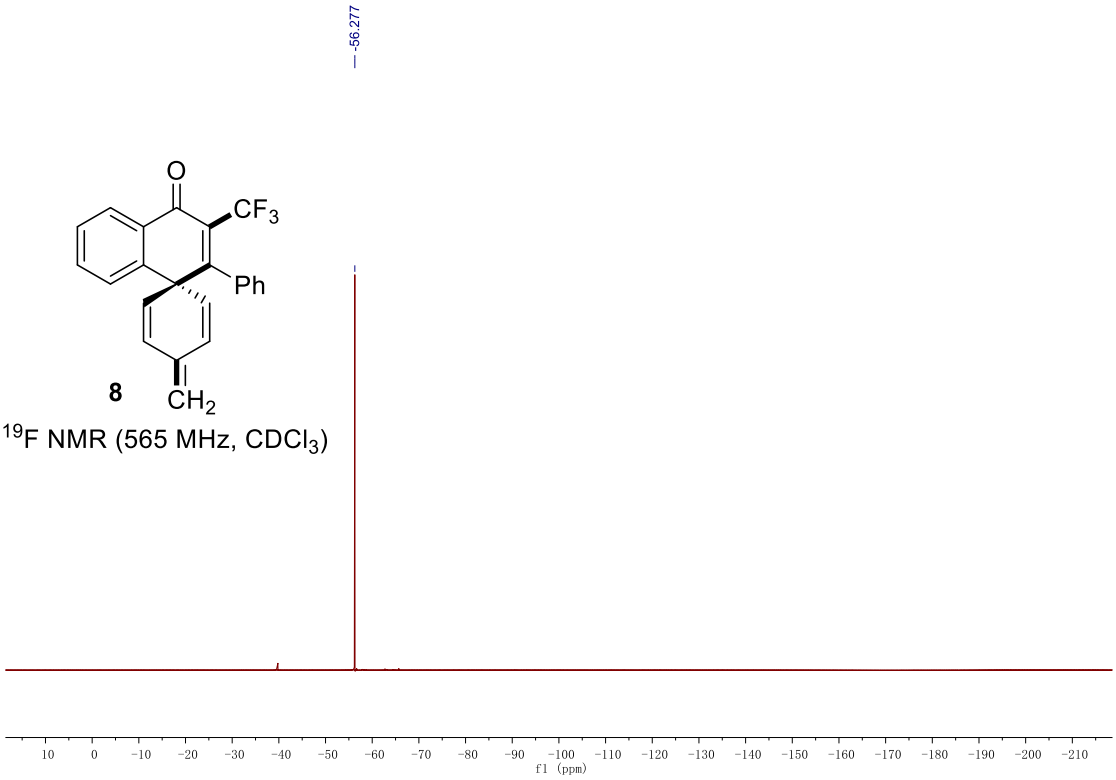

-56.476
O
CF3
Ph
7 Me OH
19F NMR (565 MHz, CDCl3)
f1 (ppm)

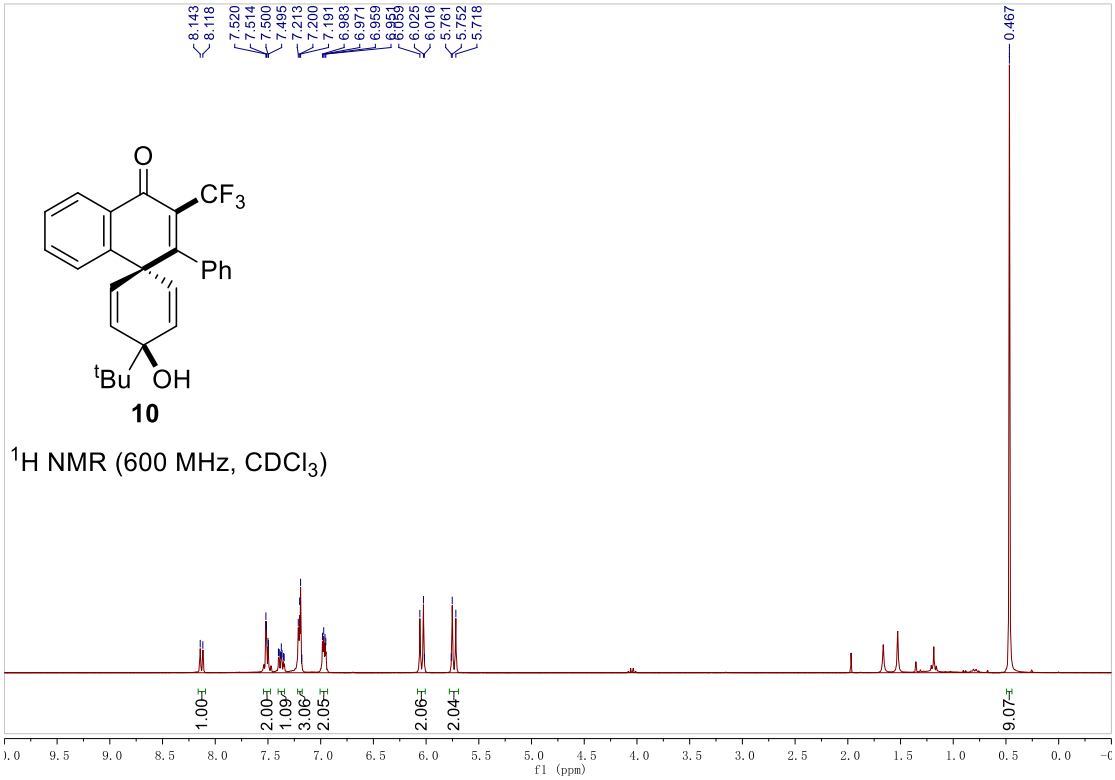

8.234
8.231
8.221
8.218
7.586
7.458
7.405
7.391
7.318
7.296
7.283
7.092
7.090
6.309
5.534
5.518
4.929
O
CF3
Ph
CH2
8
1H NMR (600 MHz, CDCl3)
1.00
1.03
1.01
1.00
3.00
2.05
2.02
2.02
2.04
f1 (ppm)


180.31
163.17
163.15
144.23
136.18
134.94
133.57
129.52
129.47
129.15
129.07
128.16
128.01
127.34
127.32
127.09
126.99
126.84
125.08
123.24
121.40
119.56
116.40
50.91
O
CF3
Ph
CH2
8
13C NMR (151 MHz, CDCl3)
f1 (ppm)

8

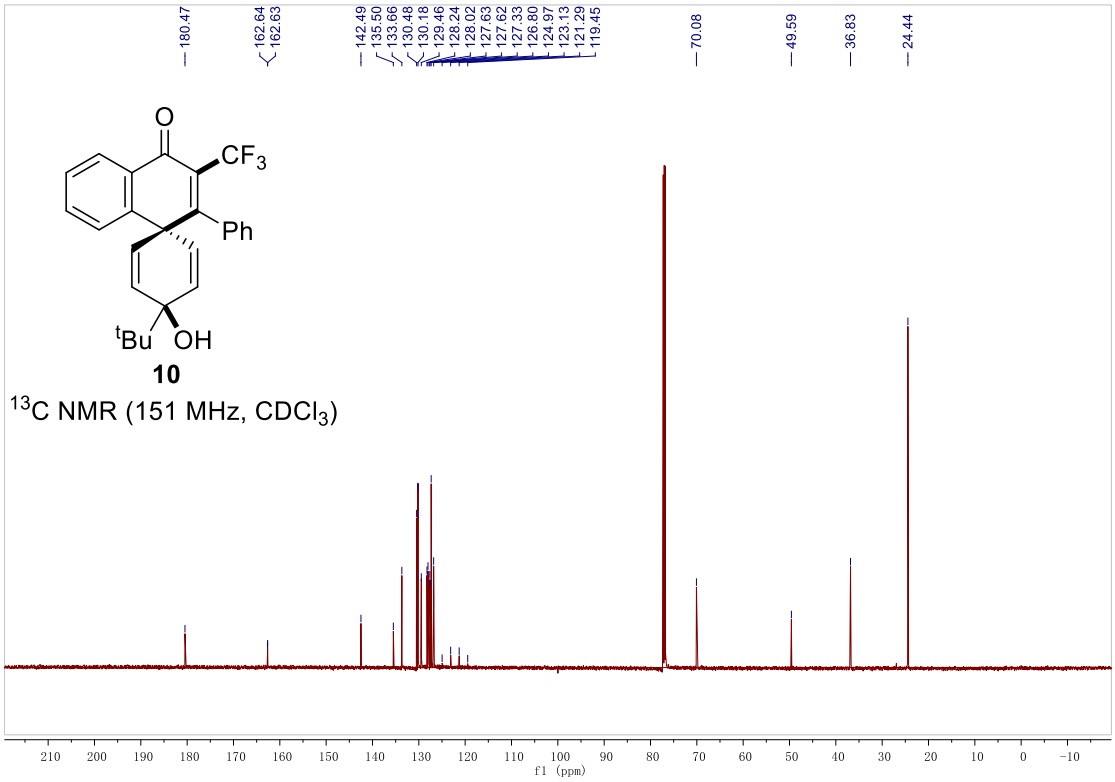
^{19}F NMR (565 MHz, $CDCl_3$)

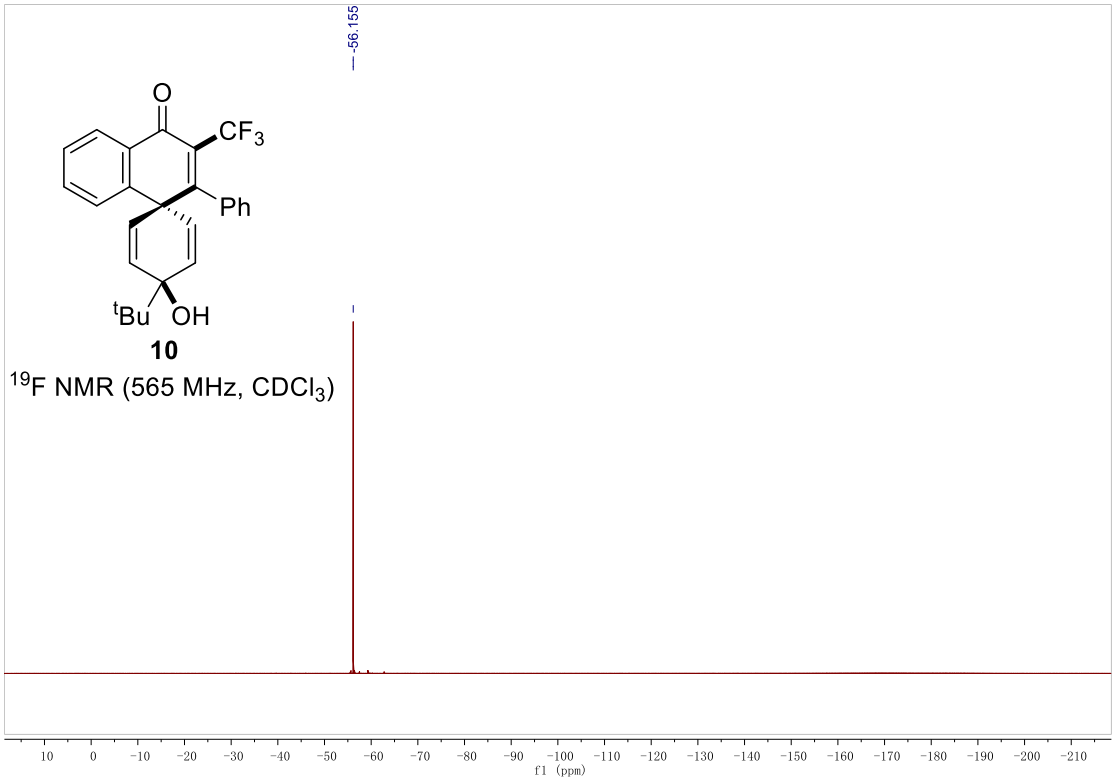




10

^{1}H NMR (600 MHz, $CDCl_3$)


180.47
162.64
162.63
142.49
135.50
133.66
130.48
130.18
129.46
128.24
128.02
127.63
127.62
127.33
126.80
124.97
123.13
121.29
119.45
70.08
49.59
36.83
24.44
O
CF3
Ph
tBu
OH
10
13C NMR (151 MHz, CDCl3)
f1 (ppm)


-56.155
O
CF3
Ph
tBu
OH
10
19F NMR (565 MHz, CDCl3)
f1 (ppm)