Electronic Supplementary Information

Rhodium(III)-Catalyzed Synthesis of Spirocyclic Isoindole *N*-oxides and Isobenzofuranones via C-H Activation and Spiroannulation

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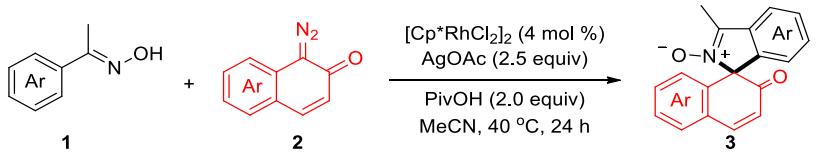
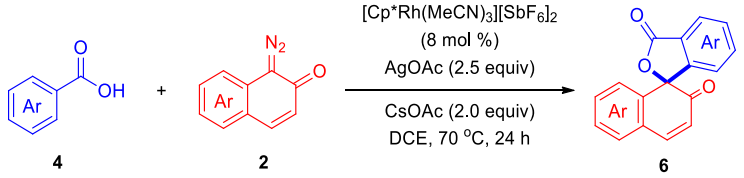
Ⅰ. General Information

All chemicals were obtained from commercial sources and were used as received unless otherwise noted. Oximes[1] and Diazo compounds[2] were prepared by following literature reports. All reactions were carried out using Schlenk techniques or in a nitrogen-filled glovebox. NMR spectra were recorded on a 400 or 600 MHz NMR spectrometer in the solvent indicated. The chemical shift is given in dimensionless δ values and is frequency referenced relative to TMS in ^{1}H and ^{13}C NMR spectroscopy. HRMS data were obtained on a Thermo Scientific LTQ Orbitrap Discovery spectrometer (Bremen, Germany). Column chromatography was performed on silica gel (200-300 mesh) using ethyl acetate (EA)/petroleum ether (PE).

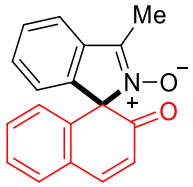
II. Experimental Procedures and Characterizations



Synthesis of Product 3: A mixture of oximes **1** (0.2 mmol), diazo compounds **2** (0.3 mmol), $[Cp^*RhCl_2]_2$ (4 mol %), AgOAc (2.5 equiv), PivOH (2.0 equiv) and MeCN (2.0 mL) were charged into a pressure tube. The reaction mixture was stirred under N_2 at 40 ºC for 24 h. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (1:1) to afford the product **3**.



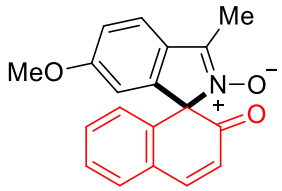
Synthesis of Product 6: A mixture of Benzoic Acids **4** (0.2 mmol), diazo compounds **2** (0.24 mmol), $[Cp^*Rh(MeCN)_3][SbF_6]_2$ (8 mol %), AgOAc (2.5 equiv), CsOAc (2.0 equiv), DCE (2 mL) were charged into a pressure tube. The reaction mixture was stirred under N_2 at 70 ºC for 24 h. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (5:1) to afford the product **6**.



3-Methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3aa**)

3aa was obtained according to the general procedure in 72% yield (39.4 mg), brown solid, R_f = 0.2 (PE/EA = 1/1).

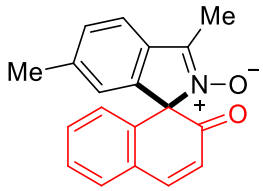
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (d, J = 10.0 Hz, 1H), 7.45 (d, J = 7.5 Hz, 1H), 7.41 - 7.35 (m, 3H), 7.34 - 7.30 (m, 1H), 7.21 - 7.18 (m, 1H), 7.00 (d, J = 7.6 Hz, 1H), 6.90 (d, J = 7.7 Hz, 1H), 6.30 (d, J = 10.0 Hz, 1H), 2.55 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.0, 146.9, 138.6, 136.0, 135.6, 131.3, 130.4, 130.3, 129.6, 129.4, 128.4, 126.7, 124.4, 120.8, 120.2, 120.1, 88.8, 9.8. HRMS (ESI) calculated for $C_{18}H_{13}NNaO_2^+$ $[M+Na]^+$: 298.0838, found: 298.0841.



6-Methoxy-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ba**)

3ba was obtained according to the general procedure in 65% yield (39.7 mg), brown solid, R_f = 0.3 (PE/EA = 1/1).

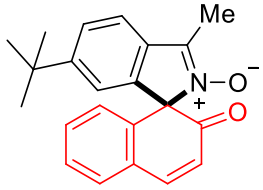
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, J = 10.0 Hz, 1H), 7.43 (d, J = 7.5 Hz, 1H), 7.38 (t, J = 7.5 Hz, 1H), 7.34 – 7.27 (m, 2H), 6.91 (d, J = 7.7 Hz, 1H), 6.87 (dd, J = 8.5, 2.4 Hz, 1H), 6.55 (d, J = 2.3 Hz, 1H), 6.28 (d, J = 9.9 Hz, 1H), 3.70 (s, 3H), 2.51 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.0, 160.5, 146.9, 146.8, 140.4, 135.8, 131.3, 130.4, 130.3, 129.6, 128.5, 126.9, 124.4, 121.2, 114.20, 108.3, 88.2 55.7, 9.8. HRMS (ESI) calculated for $C_{19}H_{15}NNaO_3^+$ $[M+Na]^+$: 328.0944, found: 328.0947.



3,6-Dimethyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ca**)

3ca was obtained according to the general procedure in 84% yield (48.4 mg), yellow solid, R_f = 0.2 (PE/EA = 1/1).

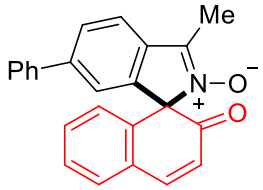
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, J = 10.0 Hz, 1H), 7.45 (d, J = 7.6 Hz, 1H), 7.39 (t, J = 7.5 Hz, 1H), 7.32 (t, J = 7.6 Hz, 1H), 7.30 – 7.25 (m, 1H), 7.16 (d, J = 7.9 Hz, 1H), 6.91 (d, J = 7.8 Hz, 1H), 6.79 (s, 1H), 6.30 (d, J = 10.0 Hz, 1H), 2.53 (s, 3H), 2.25 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.2, 147.2, 146.9, 139.0, 138.9, 135.8, 133.2, 131.3, 130.5, 130.4, 130.1, 129.5, 126.8, 124.5, 121.6, 120.1, 88.6, 21.7, 9.8. HRMS (ESI) calculated for $C_{19}H_{15}NNaO_2^+$ $[M+Na]^+$: 312.0995, found: 312.0994.



6-(Tert-butyl)-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3da**)

3da was obtained according to the general procedure in 79% yield (52.1 mg), yellow solid, R_f = 0.3 (PE/EA = 1/1).

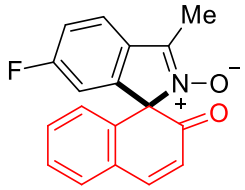
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (d, J = 10.0 Hz, 1H), 7.45 (dd, J = 7.6, 1.4 Hz, 1H), 7.41 – 7.37 (m, 2H), 7.33 – 7.29 (m, 2H), 6.97 (d, J = 1.7 Hz, 1H), 6.90 (d, J = 7.7 Hz, 1H), 6.30 (d, J = 10.0 Hz, 1H), 2.52 (s, 3H), 1.19 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.5, 152.5, 147.0, 147.0, 138.8, 135.9, 133.3, 131.3, 130.5, 130.4, 129.6, 126.9, 126.6, 124.5, 119.9, 117.7, 88.8, 35.2, 31.3, 9.8. HRMS (ESI) calculated for $C_{22}H_{21}NNaO_2^+$ $[M+Na]^+$: 354.1465, found: 354.1463.



3-Methyl-2'-oxo-6-phenyl-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ea**)

3ea was obtained according to the general procedure in 92% yield (64.6 mg), brown solid, R_f = 0.1 (PE/EA = 1/1).

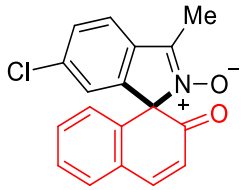
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, J = 9.9 Hz, 1H), 7.61 – 7.56 (m, 1H), 7.47 – 7.45 (m, 2H), 7.43 – 7.36 (m, 5H), 7.35 – 7.30 (m, 2H), 7.17 (s, 1H), 6.95 (d, J = 7.7 Hz, 1H), 6.33 (d, J = 9.9 Hz, 1H), 2.57 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.1, 147.1, 147.1, 146.7, 141.8, 140.1, 139.4, 135.7, 135.1, 131.4, 130.6, 130.5, 129.7, 129.0, 128.5, 128.0, 127.2, 127.0, 124.5, 120.5, 120.5, 119.7, 88.8, 9.9. HRMS (ESI) calculated for $C_{24}H_{17}NNaO_2^+$ $[M+Na]^+$: 374.1151, found: 374.1150.



6-Fluoro-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3fa**)

3fa was obtained according to the general procedure in 36% yield (21.1 mg), yellow solid, R_f = 0.1 (PE/EA = 1/1).

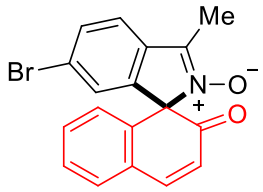
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (d, J = 10.0 Hz, 1H), 7.46 (d, J = 7.5 Hz, 1H), 7.42 (t, J = 7.5 Hz, 1H), 7.37 – 7.31 (m, 2H), 7.09 – 7.06 (m, 1H), 6.89 (d, J = 7.7 Hz, 1H), 6.74 (dd, J = 7.7, 2.4 Hz, 1H), 6.31 (d, J = 9.9 Hz, 1H), 2.53 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.4, 163.9, 162.2, 147.2, 146.0, 140.3, 135.0, 132.1, 131.0 (d, $J_{C\text{-}F}$ = 133.7 Hz), 130.4, 129.9, 126.9, 124.3, 121.4 (d, $J_{C\text{-}F}$ = 8.2 Hz), 116.6 (d, $J_{C\text{-}F}$ = 23.1 Hz), 109.5 (d, $J_{C\text{-}F}$ = 25.8 Hz), 88.3, 9.9. HRMS (ESI) calculated for $C_{18}H_{12}FNNaO_2^+$ $[M+Na]^+$: 316.0744, found: 316.0746.



6-Chloro-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ga**)

3ga was obtained according to the general procedure in 74% yield (45.7 mg), brown solid, R_f = 0.3 (ethyl acetate).

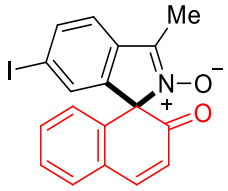
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (d, J = 10.0 Hz, 1H), 7.48 – 7.42 (m, 1H), 7.43 – 7.38 (m, 1H), 7.36 – 7.28 (m, 3H), 6.95 (d, J = 1.8 Hz, 1H), 6.87 (d, J = 7.7 Hz, 1H), 6.29 (d, J = 10.0 Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.4, 147.2, 146.0, 139.8, 134.9, 134.6, 134.5, 131.5, 130.6, 130.4, 129.9, 129.8, 126.9, 124.3, 121.5, 121.0, 88.3, 9.8. HRMS (ESI) calculated for $C_{18}H_{12}ClNNaO_2^+$ $[M+Na]^+$: 332.0449, found: 332.0457.



6-Bromo-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ha**)

3ha was obtained according to the general procedure in 72% yield (51 mg), yellow solid, R_f = 0.2 (PE/EA = 1/1).

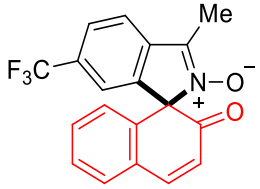
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, J = 10.0 Hz, 1H), 7.52 (dd, J = 8.1, 1.8 Hz, 1H), 7.48 (dd, J = 7.6, 1.4 Hz, 1H), 7.44 – 7.41 (m, 1H), 7.37 – 7.33 (m, 1H), 7.27 (s, 1H), 7.11 (d, J = 1.8 Hz, 1H), 6.89 (dd, J = 7.7, 1.1 Hz, 1H), 6.32 (d, J = 10.0 Hz, 1H), 2.53 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.3, 147.3, 146.2, 139.9, 135.1, 134.8, 132.7, 131.5, 130.6, 130.4, 130.0, 126.9, 124.3, 124.2, 122.4, 121.3, 88.3, 9.8. HRMS (ESI) calculated for $C_{18}H_{12}BrNNaO_2^+$ $[M+Na]^+$: 375.9944, found: 375.9954.



6-Iodo-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ia**)

3ia was obtained according to the general procedure in 68% yield (54.2 mg), yellow solid, R_f = 0.1 (PE/EA = 1/1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 (dd, J = 8.1, 1.5 Hz, 1H), 7.66 (d, J = 10.0 Hz, 1H), 7.47 (dd, J = 7.6, 1.4 Hz, 1H), 7.43 – 7.40 (m, 1H), 7.36 – 7.32 (m, 1H), 7.27 (d, J = 1.5 Hz, 1H), 7.14 (d, J = 8.1 Hz, 1H), 6.88 (dd, J = 7.8, 1.1 Hz, 1H), 6.31 (d, J = 10.0 Hz, 1H), 2.51 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.4, 147.3, 146.3, 140.0, 138.6, 135.7, 134.9, 131.5, 130.6, 130.4, 130.0, 129.7, 126.9, 124.3, 121.5, 93.52, 88.2, 9.8. HRMS (ESI) calculated for $C_{18}H_{12}INNaO_2^+$ $[M+Na]^+$: 423.9805, found: 423.9814.

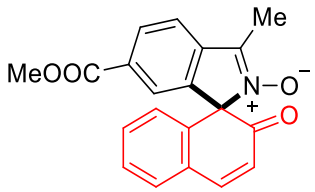


3-Methyl-2'-oxo-6-(trifluoromethyl)-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ja**)

3ja was obtained according to the general procedure in 80% yield (55 mg), brown solid, R_f = 0.2 (ethyl acetate).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (d, J = 10.0 Hz, 1H), 7.64 (dd, J = 8.0, 1.6 Hz, 1H), 7.52 – 7.48 (m, 2H), 7.45 – 7.42 (m, 1H), 7.36 – 7.33 (m, 1H), 7.17 (s, 1H), 6.86 (d, J = 7.7 Hz, 1H), 6.32 (d, J = 10.0 Hz, 1H), 2.55 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.2, 147.5, 145.8, 139.7, 138.9, 134.5, 131.6, 130.8, 130.5, 130.2 (q, $J_{C\text{-}F}$ = 98.6 Hz), 130.2, 127.0, 126.9 (q, $J_{C\text{-}F}$ = 39.4 Hz), 124.3, 123.8 (q, $J_{C\text{-}F}$ =

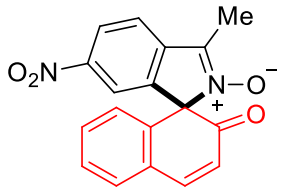
272.5 Hz), 120.2, 117.7 (q, $J_{C\text{-}F}$ = 38.7 Hz), 88.6, 9.8. HRMS (ESI) calculated for $C_{19}H_{12}F_3NNaO_2^+$ $[M+Na]^+$: 366.0712, found: 366.0722.



6-(Methoxycarbonyl)-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ka**)

3ka was obtained according to the general procedure in 91% yield (60.7 mg), yellow solid, R_f = 0.2 (PE/EA = 1/1).

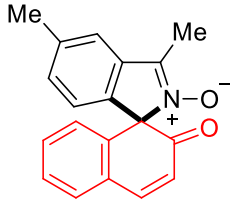
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 (d, J = 8.0 Hz, 1H), 7.68 (d, J = 9.9 Hz, 1H), 7.59 (s, 1H), 7.47 – 7.43 (m, 2H), 7.39 (t, J = 7.6 Hz, 1H), 7.30 (t, J = 7.6 Hz, 1H), 6.84 (d, J = 7.7 Hz, 1H), 6.30 (d, J = 9.8 Hz, 1H), 3.83 (s, 3H), 2.53 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.4, 166.2, 147.4, 146.1, 140.5, 138.5, 134.9, 131.4, 131.2, 130.6, 130.5, 129.9, 129.8, 126.8, 124.3, 121.7, 119.8, 88.7, 52.4, 9.8. HRMS (ESI) calculated for $C_{20}H_{15}NNaO_4^+$ $[M+Na]^+$: 356.0893, found: 356.0893.



3-Methyl-6-nitro-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3la**)

3la was obtained according to the general procedure in 84% yield (53.7 mg), yellow solid, R_f = 0.3 (PE/EA = 1/1).

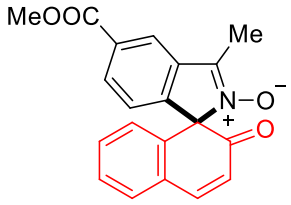
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.28 (d, J = 8.3 Hz, 1H), 7.79 (s, 1H), 7.73 (d, J = 9.9 Hz, 1H), 7.54 – 7.50 (m, 2H), 7.45 (t, J = 7.6 Hz, 1H), 7.34 (t, J = 7.6 Hz, 1H), 6.83 (d, J = 7.8 Hz, 1H), 6.33 (d, J = 10.0 Hz, 1H), 2.55 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 188.6, 147.7, 147.5, 145.4, 142.5, 139.0, 133.9, 131.7, 131.0, 130.6, 130.5, 126.9, 125.7, 124.2, 120.0, 116.4, 88.7, 9.9. HRMS (ESI) calculated for $C_{18}H_{12}N_2NaO_4^+$ $[M+Na]^+$: 343.0689, found: 343.0693.



3,5-Mimethyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ma**)

3ma was obtained according to the general procedure in 73% yield (42.2 mg), white solid, R_f = 0.2 (PE/EA = 1/1).

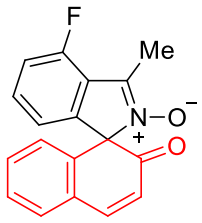
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, J = 10.0 Hz, 1H), 7.43 (dd, J = 7.5, 1.5 Hz, 1H), 7.39 – 7.35 (m, 1H), 7.32 – 7.28 (m, 1H), 7.21 (s, 1H), 7.00 (d, J = 7.7 Hz, 1H), 6.92 – 6.84 (m, 2H), 6.28 (d, J = 10.0 Hz, 1H), 2.52 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.3, 147.0, 146.9, 139.7, 136.1, 135.9, 135.9, 131.3, 130.5, 130.3, 129.5, 129.2, 126.8, 124.5, 120.9, 120.6, 88.7, 21.7, 9.8. HRMS (ESI) calculated for $C_{19}H_{15}NNaO_2^+$ $[M+Na]^+$: 312.0995, found: 312.0997.



5-(Methoxycarbonyl)-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3na**)

3na was obtained according to the general procedure in 75% yield (50.0 mg), brown solid, R_f = 0.1 (PE/EA = 1/1).

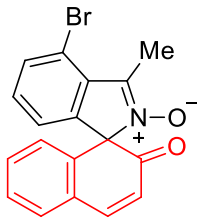
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.88 (d, J = 7.7 Hz,, 1H), 7.67 (d, J = 10.0 Hz, 1H), 7.46 (d, J = 7.5 Hz, 1H), 7.40 (t, J = 7.5 Hz, 1H), 7.36 – 7.29 (m, 1H), 7.05 (d, J = 7.9 Hz, 1H), 6.86 (d, J = 7.7 Hz, 1H), 6.30 (d, J = 10.0 Hz, 1H), 3.92 (s, 3H), 2.56 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.3, 166.1, 147.3, 146.2, 142.6, 136.7, 134.8, 131.6, 131.5, 130.6, 130.5, 129.9, 129.8, 126.9, 124.3, 121.0, 120.9, 88.7, 52.6, 9.8. HRMS (ESI) calculated for $C_{20}H_{15}NNaO_4^+$ $[M+Na]^+$: 356.0893, found: 356.0893.



4-Fluoro-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3oa**)

3oa was obtained according to the general procedure in 75% yield (43.9 mg), yellow solid, R_f = 0.2 (PE/EA = 1/1).

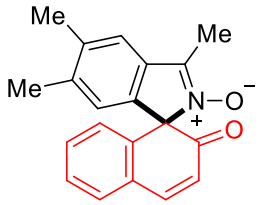
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, J = 10.0 Hz, 1H), 7.45 (dd, J = 7.6, 1.4 Hz, 1H), 7.42 – 7.39 (m, 1H), 7.35 – 7.32 (m, 1H), 7.18 – 7.13 (m, 1H), 7.05 – 7.01 (m, 1H), 6.91 (d, J = 7.7 Hz, 1H), 6.79 (d, J = 7.5 Hz, 1H), 6.29 (d, J = 10.0 Hz, 1H), 2.66 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.5, 155.2 (d, $J_{C\text{-}F}$ = 254.1 Hz), 147.2, 144.5, 140.3, 135.2, 131.5, 130.5, 130.4, 130.0 (d, $J_{C\text{-}F}$ = 6.9 Hz), 129.9, 126.9, 124.3, 123.6 (d, $J_{C\text{-}F}$ = 14.7 Hz), 117.1 (d, $J_{C\text{-}F}$ = 3.7 Hz), 116.9 (d, $J_{C\text{-}F}$ = 20.0 Hz), 88.6, 11.6. HRMS (ESI) calculated for $C_{18}H_{12}FNNaO_2^+$ $[M+Na]^+$: 316.0744, found: 316.0745.



4-Bromo-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3pa**)

3pa was obtained according to the general procedure in 43% yield (30.7 mg), brown solid, R_f = 0.3 (PE/EA = 1/1).

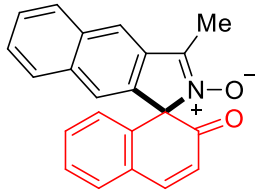
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, J = 10.0 Hz, 1H), 7.48 (dd, J = 8.1, 1.0 Hz, 1H), 7.45 (dd, J = 7.6, 1.5 Hz, 1H), 7.43 – 7.39 (m, 1H), 7.36 – 7.32 (m, 1H), 7.02 (t, J = 7.8 Hz, 1H), 6.94 – 6.88 (m, 2H), 6.30 (d, J = 10.1 Hz, 1H), 2.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.4, 147.2, 140.5, 135.3, 134.8, 134.5, 131.5, 131.2, 130.6, 130.4, 129.9, 129.3, 127.0, 124.3, 119.9, 114.6, 87.9, 12.6. HRMS (ESI) calculated for $C_{18}H_{12}BrNNaO_2^+$ $[M+Na]^+$: 375.9944, found: 375.9945.



3,5,6-Trimethyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3qa**)

3qa was obtained according to the general procedure in 63% yield (38.5 mg), brown solid, R_f = 0.2 (PE/EA = 1/1).

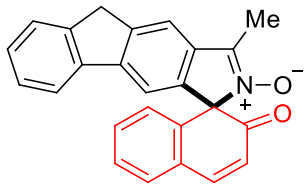
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, J = 10.0 Hz, 1H), 7.44 (d, J = 7.5 Hz, 1H), 7.37 (t, J = 7.5 Hz, 1H), 7.30 (t, J = 7.6 Hz, 1H), 7.17 (s, 1H), 6.90 (d, J = 7.7 Hz, 1H), 6.75 (s, 1H), 6.29 (d, J = 10.0 Hz, 1H), 2.52 (s, 3H), 2.26 (s, 3H), 2.14 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.4, 147.2, 146.9, 138.2, 137.6, 136.5, 136.1, 133.6, 131.3, 130.5, 130.3, 129.5, 126.8, 124.6, 121.9, 121.4, 88.5, 20.2, 20.1, 9.8. HRMS (ESI) calculated for $C_{20}H_{17}NNaO_2^+$ $[M+Na]^+$: 326.1151, found: 326.1152.



3-Methyl-2'-oxo-2'*H*-spiro[benzo[*f*]isoindole-1,1'-naphthalene] 2-oxide (**3ra**)

3ra was obtained according to the general procedure in 94% yield (61.1 mg), brown solid, R_f = 0.2 (ethyl acetate).

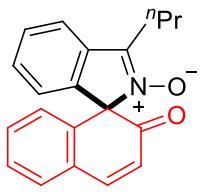
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (d, J = 8.2 Hz, 1H), 7.79 (s, 1H), 7.70 (d, J = 10.1 Hz, 1H), 7.65 (d, J = 8.0 Hz, 1H), 7.51 – 7.37 (m, 5H), 7.30 (t, J = 7.8 Hz, 1H), 6.94 (d, J = 7.8 Hz, 1H), 6.33 (d, J = 10.0 Hz, 1H), 2.63 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.5, 147.1, 147.0, 136.4, 135.8, 133.8, 133.7, 133.0, 131.3, 130.5, 130.5, 129.7, 128.6, 128.5, 127.4, 127.0, 126.9, 124.4, 120.2, 119.1, 87.9, 9.9. HRMS (ESI) calculated for $C_{22}H_{15}NNaO_2^+$ $[M+Na]^+$: 348.0995, found: 348.0995.



1-Methyl-2'-oxo-2'*H*,9*H*-spiro[indeno[1,2-*f*]isoindole-3,1'-naphthalene] 2-oxide (**3sa**)

3sa was obtained according to the general procedure in 76% yield (55.6 mg), yellow solid, R_f = 0.2 (ethyl acetate).

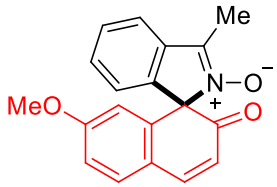
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (d, J = 10.0 Hz, 1H), 7.61 (d, J = 7.1 Hz, 1H), 7.57 (s, 1H), 7.50 (dd, J = 7.5, 2.8 Hz, 2H), 7.41 (t, J = 7.5 Hz, 1H), 7.35 – 7.27 (m, 4H), 6.95 (d, J = 7.7 Hz, 1H), 6.36 (d, J = 10.0 Hz, 1H), 3.90 (s, 2H), 2.59 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.3, 147.3, 147.1, 144.8, 143.4, 142.5, 140.6, 138.1, 136.1, 134.7, 131.4, 130.6, 130.5, 129.7, 127.6, 127.1, 127.0, 125.2, 124.7, 120.2, 117.0, 112.7, 88.4, 37.1, 9.9. HRMS (ESI) calculated for $C_{25}H_{17}NNaO_2^+$ $[M+Na]^+$: 386.1151, found: 386.1155.



2'-Oxo-3-propyl-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ta**)

3ta was obtained according to the general procedure in 72% yield (44.0 mg), yellow solid, R_f = 0.2 (PE/EA = 1/1).

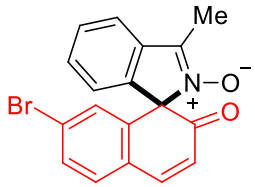
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 (d, J = 9.9 Hz, 1H), 7.44 – 7.30 (m, 5H), 7.17 (t, J = 7.6 Hz, 1H), 6.97 (d, J = 7.6 Hz, 1H), 6.88 (d, J = 7.7 Hz, 1H), 6.28 (d, J = 10.0 Hz, 1H), 2.98 – 2.94 (m, 2H), 1.94 -1.89 (m, 2H), 1.12 (t, J = 7.4 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 190.1, 150.3, 146.9, 138.8, 136.1, 135.9, 131.3, 130.5, 130.4, 129.6, 129.4, 128.2, 126.8, 124.5, 120.9, 120.2, 88.7, 26.0, 19.3, 14.4. HRMS (ESI) calculated for $C_{20}H_{17}NNaO_2^+$ $[M+Na]^+$: 326.1151, found: 326.1153.



7'-Methoxy-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ab**)

3ab was obtained according to the general procedure in 72% yield (43.7 mg), brown solid, R_f = 0.3 (PE/EA = 1/1).

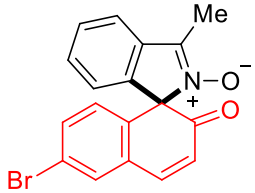
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.59 (d, J = 9.9 Hz, 1H), 7.39 – 7.33 (m, 3H), δ 7.19 (t, J = 7.3 Hz, 1H)., 6.99 (d, J = 7.5 Hz, 1H), 6.87 (dd, J = 8.5, 2.6 Hz, 1H), 6.41 (d, J = 2.6 Hz, 1H), 6.15 (d, J = 9.9 Hz, 1H), 3.71 (s, 3H), 2.54 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.7, 162.1, 147.1, 146.9, 138.8, 137.6, 135.7, 132.0, 129.3, 128.4, 123.4, 121.6, 120.7, 120.2, 113.9, 113.5, 88.7, 55.5, 9.7. HRMS (ESI) calculated for $C_{19}H_{15}NNaO_3^+$ $[M+Na]^+$: 328.0944, found: 328.0946.



7'-Bromo-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ac**)

3ac was obtained according to the general procedure in 30% yield (21.5 mg), brown solid, R_f = 0.2 (PE/EA = 1/1).

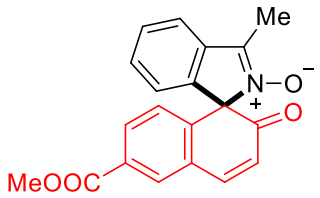
^{1}H NMR (600 MHz, Acetone-d_6) δ 7.92 (d, J = 10.0 Hz, 1H), 7.69 – 7.64 (m, 1H), 7.60 (d, J = 8.2 Hz, 1H), 7.56 (d, J = 7.7 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 7.25 (t, J = 7.6 Hz, 1H), 7.13 – 7.10 (m, 1H), 7.07 (d, J = 7.6 Hz, 1H), 6.33 (d, J = 10.0 Hz, 1H), 2.44 (s, 3H). ^{13}C NMR (151 MHz, Acetone- d_6) δ 188.9, 146.1, 138.3, 138.1, 136.7, 132.6, 131.9, 129.9, 129.7, 129.5, 127.9, 124.7, 124.4, 120.4, 120.0, 87.8, 8.6. HRMS (ESI) calculated for $C_{18}H_{12}BrNNaO_2^+$ $[M+Na]^+$: 375.9944, found: 375.9948.



6'-Bromo-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ad**)

3ad was obtained according to the general procedure in 56% yield (39.6 mg), brown solid, R_f = 0.2 (PE/EA = 1/1).

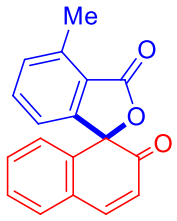
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.56 (m, 2H), 7.44 – 7.37 (m, 3H), 7.22 (t, J = 7.4 Hz, 1H), 6.98 (d, J = 7.5 Hz, 1H), 6.76 (d, J = 8.2 Hz, 1H), 6.34 (d, J = 9.9 Hz, 1H), 2.53 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 189.2, 147.2, 145.2, 137.9, 135.8, 134.2, 133.9, 132.8, 132.2, 129.6, 128.6, 128.4, 125.5, 123.5, 120.8, 120.3, 88.2, 9.7. HRMS (ESI) calculated for $C_{18}H_{12}BrNNaO_2^+$ $[M+Na]^+$: 375.9944, found: 375.9947.



6'-(Methoxycarbonyl)-3-methyl-2'-oxo-2'*H*-spiro[isoindole-1,1'-naphthalene] 2-oxide (**3ae**)

3ae was obtained according to the general procedure in 21% yield (14.3 mg), brown solid, R_f = 0.2 (PE/EA = 1/1).

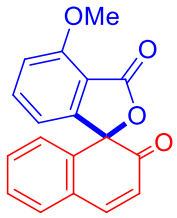
^{1}H NMR (600 MHz, Acetone-d_6) δ 8.24 (s, 1H), 8.06 (d, J = 10.0 Hz, 1H), 7.97 (dd, J = 8.1, 1.8 Hz, 1H), 7.56 (d, J = 7.7 Hz, 1H), 7.44 (t, J = 7.6 Hz, 1H), 7.23 (t, J = 7.6 Hz, 1H), 7.08 – 7.06 (m, 2H), 6.39 (d, J = 10.1 Hz, 1H), 3.91 (s, 3H), 2.44 (s, 3H). ^{13}C NMR (151 MHz, Acetone-d_6) δ 189.2, 165.3, 146.3, 140.8, 137.9, 136.8, 131.4, 131.2, 131.1, 130.9, 129.5, 127.8, 127.2, 124.9, 120.4, 119.9, 88.3, 51.8, 8.5. HRMS (ESI) calculated for $C_{20}H_{15}NNaO_4^+$ $[M+Na]^+$: 356.0893, found: 356.0887.



4-Methyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6aa**)

6aa was obtained according to the general procedure in 76% yield (42.8 mg), yellow solid, R_f = 0.2 (PE/EA = 5/1).

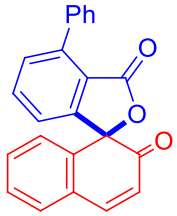
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, J = 10.0 Hz, 1H), 7.45 (d, J = 7.4 Hz, 1H), 7.40 (t, J = 7.4 Hz, 1H), 7.37 – 7.32 (m, 2H), 7.26 – 7.24 (m, 1H), 7.22 (d, J = 7.7 Hz, 1H), 6.99 (d, J = 7.7 Hz, 1H), 6.26 (d, J = 10.0 Hz, 1H), 2.74 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 193.0, 170.4, 148.8, 146.5, 140.9, 138.2, 134.1, 131.7, 130.9, 130.2, 129.5, 129.4, 126.5, 123.9, 121.2, 118.5, 86.7, 17.4. HRMS (ESI) calculated for $C_{18}H_{12}NaO_3^+$ $[M+Na]^+$: 299.0679, found: 299.0680.



4-Methoxy-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ba**)

6ba was obtained according to the general procedure in 75% yield (44.1 mg), yellow solid, R_f = 0.2 (PE/EA = 5/1).

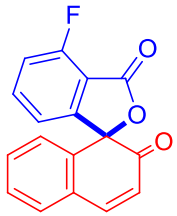
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, J = 10.0 Hz, 1H), 7.44 (t, J = 7.9 Hz, 2H), 7.41 – 7.38 (m, 1H), 7.36 – 7.32 (m, 1H), 7.24 (d, J = 7.7 Hz, 1H), 6.91 (d, J = 8.3 Hz, 1H), 6.72 (d, J = 7.6 Hz, 1H), 6.26 (d, J = 10.0 Hz, 1H), 4.00 (s, 3H). 13C NMR (151 MHz, $CDCl_3$) δ 192.6, 168.2, 159.1, 150.8, 146.5, 138.0, 136.7, 130.9, 130.2, 129.5, 129.5, 126.6, 123.9, 112.8, 111.8, 111.1, 86.4, 56.2. HRMS (ESI) calculated for $C_{18}H_{12}NaO_4^+$ $[M+Na]^+$: 315.0628, found: 315.0626.



4-Phenyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ca**)

6ca was obtained according to the general procedure in 86% yield (58.4 mg), yellow solid, R_f = 0.3 (PE/EA = 5/1).

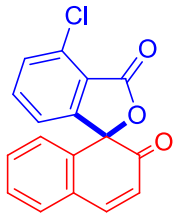
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, J = 10.0 Hz, 1H), 7.61 (d, J = 7.3 Hz, 2H), 7.53 – 7.40 (m, 7H), 7.36 (t, J = 7.6 Hz, 1H), 7.26 – 7.24 (m, 1H), 7.15 (d, J = 7.7 Hz, 1H), 6.30 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 193.1, 169.2, 149.7, 146.7, 143.9, 138.3, 136.1, 134.3, 131.8, 131.1, 130.4, 129.8, 129.7, 128.7, 128.2, 126.8, 124.0, 120.1, 119.9, 86.4. HRMS (ESI) calculated for $C_{23}H_{14}NaO_3^+$ $[M+Na]^+$: 361.0835, found: 361.0834.



4-Fluoro-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6da**)

6da was obtained according to the general procedure in 72% yield (40.2 mg), yellow solid, R_f = 0.2 (PE/EA = 5/1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, J = 10.0 Hz, 1H), 7.53 – 7.50 (m, 1H), 7.48 (d, J = 7.1 Hz, 1H), 7.44 (td, J = 7.5, 1.3 Hz, 1H), 7.38 (td, J = 7.5, 1.5 Hz, 1H), 7.24 (d, J = 7.7 Hz, 1H), 7.15 (t, J = 8.4 Hz, 1H), 6.97 (d, J = 7.7 Hz, 1H), 6.29 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.9, 166.2, 159.7 (d, $J_{C\text{-}F}$ = 267.1 Hz), 150.7, 146.8, 137.2 (d, $J_{C\text{-}F}$ = 4.6 Hz),137.1, 130.8 (d, $J_{C\text{-}F}$ = 112.5 Hz), 129.9, 129.4, 126.7, 123.7, 117.2 (d, $J_{C\text{-}F}$ = 18.8 Hz), 117.2 (d, $J_{C\text{-}F}$ = 4.5 Hz), 111.9 (d, $J_{C\text{-}F}$ = 15.0 Hz) ,99.9, 86.9. HRMS (ESI) calculated for $C_{17}H_9FNaO_3^+$ $[M+Na]^+$: 303.0428, found: 303.0427.

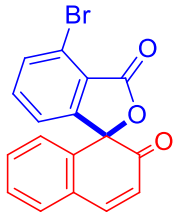


4-Chloro-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ea**)

6ea was obtained according to the general procedure in 74% yield (43.8 mg), yellow solid, R_f = 0.2 (PE/EA = 5/1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.68 (d, J = 10.0 Hz, 1H), 7.48 (d, J = 7.5 Hz, 1H), 7.45 – 7.40 (m, 3H),

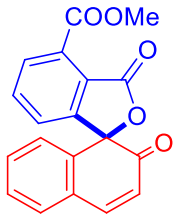
7.36 – 7.33 (m, 1H), 7.19 (d, J = 7.6 Hz, 1H), 7.08 (dd, J = 5.9, 2.5 Hz, 1H), 6.26 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 167.2, 150.7, 146.9, 137.3, 135.6, 134.2, 131.6, 131.2, 130.5, 130.0, 129.6, 126.8, 123.8, 120.9, 119.8, 86.1. HRMS (ESI) calculated for $C_{17}H_9ClNaO_3^+$ $[M+Na]^+$: 319.0132, found: 319.0137.



4-Bromo-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6fa**)

6fa was obtained according to the general procedure in 76% yield (52.0 mg), yellow solid, R_f = 0.3 (PE/EA = 5/1).

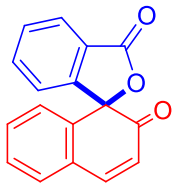
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.70 (d, J = 10.0 Hz, 1H), 7.65 (d, J = 7.8 Hz, 1H), 7.49 (dd, J = 7.6, 1.4 Hz, 1H), 7.43 (td, J = 7.5, 1.3 Hz, 1H), 7.38 – 7.34 (m, 2H), 7.20 (d, J = 7.7 Hz, 1H), 7.14 (d, J = 7.7 Hz, 1H), 6.27 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 167.7, 150.7, 147.0, 137.2, 135.6, 134.8, 131.2, 130.6, 130.0, 129.6, 126.7, 123.7, 122.3, 121.8, 120.4, 85.8. HRMS (ESI) calculated for $C_{17}H_9BrNaO_3^+$ $[M+Na]^+$: 362.9627, found: 362.9634.



Methyl 2',3-dioxo-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-4-carboxylate (**6ga**)

6ga was obtained according to the general procedure in 40% yield (25.9 mg), yellow solid, R_f = 0.2 (PE/EA = 5/1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79 (d, J = 7.5 Hz, 1H), 7.68 (d, J = 10.0 Hz, 1H), 7.56 (t, J = 7.7 Hz, 1H), 7.48 (d, J = 7.5 Hz, 1H), 7.43 (t, J = 7.5 Hz, 1H), 7.36 (t, J = 7.5 Hz, 1H), 7.30 (d, J = 7.9 Hz, 1H), 7.22 (d, J = 7.8 Hz, 1H), 6.29 (d, J = 9.7 Hz, 1H), 4.04 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.4, 167.2, 165.8, 149.7, 146.9, 137.5, 134.4, 132.6, 131.2, 130.7, 130.5, 129.9, 129.6, 126.9, 124.0, 123.9, 121.9, 86.5, 53.1. HRMS (ESI) calculated for $C_{19}H_{12}NaO_5^+$ $[M+Na]^+$: 343.0577, found: 343.0579.

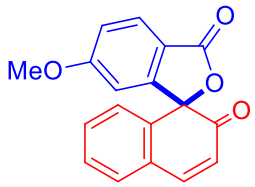


2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ha**)

6ha was obtained according to the general procedure in 61% yield (32.2 mg), yellow solid, R_f = 0.3 (PE/EA = 5/1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 – 7.91 (m, 1H), 7.68 (d, J = 9.9 Hz, 1H), 7.56 – 7.50 (m, 2H), 7.48 (d, J = 7.5 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 7.34 (t, J = 7.6 Hz, 1H), 7.20 (d, J = 7.2 Hz, 2H), 6.28 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.8, 170.4, 148.7, 146.8, 137.9, 134.6, 131.1, 130.4, 130.1, 129.8, 129.6, 126.9, 126.6, 124.0, 123.9, 121.3, 87.7. HRMS (ESI) calculated for $C_{17}H_{10}NaO_3^+$

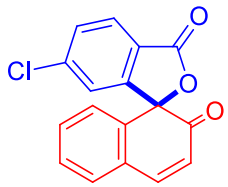
$[M+Na]^+$: 285.0522, found: 285.0525.



6-Methoxy-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ia**)

6ia was obtained according to the general procedure in 39% yield (22.5 mg), yellow solid, R_f = 0.3 (PE/EA = 5/1).

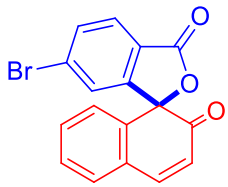
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (d, J = 8.5 Hz, 1H), 7.65 (d, J = 10.0 Hz, 1H), 7.46 (d, J = 7.4 Hz, 1H), 7.41 (td, J = 7.5, 1.3 Hz, 1H), 7.35 (td, J = 7.5, 1.5 Hz, 1H), 7.22 (d, J = 7.8 Hz, 1H), 7.00 (dd, J = 8.5, 2.2 Hz, 1H), 6.58 (d, J = 2.1 Hz, 1H), 6.28 (d, J = 10.0 Hz, 1H), 3.77 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.8, 170.1, 164.9, 151.3, 146.6, 138.1, 131.1, 130.3, 129.7, 129.6, 128.5, 126.8, 124.0, 116.9, 116.2, 105.9, 86.9, 56.0. HRMS (ESI) calculated for $C_{18}H_{12}NaO_4^+$ $[M+Na]^+$: 315.0628, found: 315.0628.



6-Chloro-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ja**)

6ja was obtained according to the general procedure in 48% yield (28.7 mg), brown solid, R_f = 0.2 (PE/EA = 5/1).

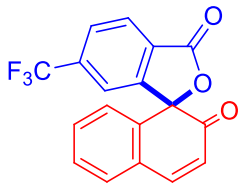
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, J = 8.1 Hz, 1H), 7.69 (d, J = 10.0 Hz, 1H), 7.54 – 7.47 (m, 2H), 7.45 (t, J = 7.5 Hz, 1H), 7.38 (t, J = 7.6 Hz, 1H), 7.21 (d, J = 7.7 Hz, 1H), 7.15 (s, 1H), 6.30 (d, J = 9.9 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 169.2, 150.1, 146.9, 141.3, 137.1, 131.3, 130.9, 130.6, 130.1, 129.5, 128.1, 126.8, 123.8, 122.6, 121.8, 86.9. HRMS (ESI) calculated for $C_{17}H_9ClNaO_3^+$ $[M+Na]^+$: 319.0132, found: 319.0137.



6-Bromo-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6ka**)

6ka was obtained according to the general procedure in 42% yield (28.6 mg), brown solid, R_f = 0.2 (PE/EA = 5/1).

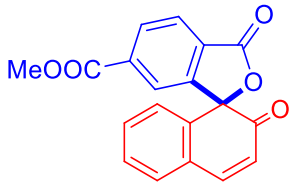
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, J = 8.1 Hz, 1H), 7.69 (d, J = 10.0 Hz, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.49 (d, J = 7.5 Hz, 1H), 7.47 – 7.43 (m, 1H), 7.41 – 7.36 (m, 1H), 7.31 (s, 1H), 7.21 (d, J = 7.7 Hz, 1H), 6.30 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 169.3, 150.2, 146.9, 137.1, 133.8, 131.3, 130.6, 130.1, 129.7, 129.5, 128.2, 126.7, 124.7, 123.8, 123.1, 86.9. HRMS (ESI) calculated for $C_{17}H_9BrNaO_3^+$ $[M+Na]^+$: 362.9627, found: 362.9631.



6-(Trifluoromethyl)-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6la**)

6la was obtained according to the general procedure in 42% yield (27.5 mg), yellow solid, R_f = 0.2 (PE/EA = 5/1).

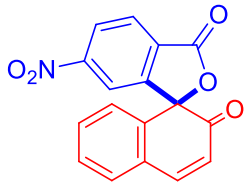
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11 (d, J = 8.0 Hz, 1H), 7.81 (d, J = 8.0 Hz, 1H), 7.73 (d, J = 10.0 Hz, 1H), 7.52 (d, J = 7.5 Hz, 1H), 7.47 (t, J = 7.5 Hz, 1H), 7.43 – 7.36 (m, 2H), 7.20 (d, J = 7.7 Hz, 1H), 6.32 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.8, 168.8, 149.1, 147.2, 136.7, 136.4 (q, $J_{C\text{-}F}$ = 33.2 Hz), 131.4, 130.8, 130.3, 129.6, 127.7, 127.5 (q, $J_{C\text{-}F}$ = 4.6 Hz), 126.9, 123.7, 123.0 (q, $J_{C\text{-}F}$ = 273.7 Hz), 118.6 (q, $J_{C\text{-}F}$ = 3.9 Hz), 87.3. HRMS (ESI) calculated for $C_{18}H_9F_3NaO_3^+$ $[M+Na]^+$: 353.0396, found: 353.0396.



Methyl 2',3-dioxo-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-6-carboxylate (**6ma**)

6ma was obtained according to the general procedure in 30% yield (19.4 mg), brown solid, R_f = 0.2 (PE/EA = 5/1).

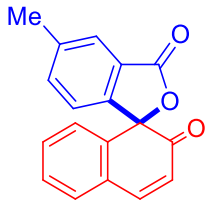
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.19 (dd, J = 8.0, 1.3 Hz, 1H), 8.04 (d, J = 8.0 Hz, 1H), 7.80 (s, 1H), 7.72 (d, J = 10.0 Hz, 1H), 7.50 (d, J = 7.5 Hz, 1H), 7.46 – 7.43 (m, 1H), 7.38 – 7.34 (m, 1H), 7.19 (d, J = 7.7 Hz, 1H), 6.31 (d, J = 10.0 Hz, 1H), 3.90 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 169.3, 165.4, 148.8, 147.1, 137.2, 136.0, 131.4, 131.2, 130.7, 130.1, 129.7, 127.9, 127.0, 126.8, 123.9, 122.7, 87.5, 52.9. HRMS (ESI) calculated for $C_{19}H_{12}NaO_5^+$ $[M+Na]^+$: 343.0577, found: 343.0580.



6-Nitro-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6na**)

6na was obtained according to the general procedure in 38% yield (23.6 mg), brown solid, R_f = 0.2 (PE/EA = 5/1).

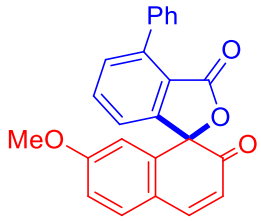
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.40 (d, J = 8.2 Hz, 1H), 8.16 (d, J = 8.3 Hz, 1H), 7.97 (s, 1H), 7.76 (d, J = 10.0 Hz, 1H), 7.55 (d, J = 7.6 Hz, 1H), 7.49 (t, J = 7.5 Hz, 1H), 7.40 (t, J = 7.6 Hz, 1H), 7.19 (d, J = 7.7 Hz, 1H), 6.33 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 191.3, 167.9, 151.8, 149.7, 147.5, 136.2, 131.5, 130.9, 130.6, 129.6, 129.4, 128.2, 126.9, 125.7, 123.6, 117.1, 87.1. HRMS (ESI) calculated for $C_{17}H_9NNaO_5^+$ $[M+Na]^+$: 330.0373, found: 330.0374.



5-Methyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6oa**)

6oa was obtained according to the general procedure in 49% yield (27.2 mg), yellow solid, R_f = 0.3 (PE/EA = 5/1).

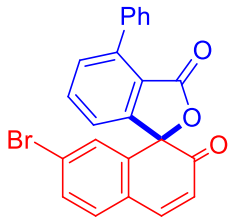
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.65 (d, J = 10.0 Hz, 1H), 7.45 (d, J = 7.4 Hz, 1H), 7.42 – 7.38 (m, 1H), 7.35 – 7.30 (m, 2H), 7.19 (d, J = 7.7 Hz, 1H), 7.06 (d, J = 7.9 Hz, 1H), 6.27 (d, J = 10.0 Hz, 1H), 2.41 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 193.0, 170.5, 146.6, 146.2, 140.6, 138.1, 135.7, 131.0, 130.3, 129.6, 129.5, 126.9, 126.5, 124.2, 123.9, 121.0, 87.6, 21.3. HRMS (ESI) calculated for $C_{18}H_{12}NaO_3^+$ [M+Na]$^+$: 299.0679, found: 299.0681.



7'-Methoxy-4-phenyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6cb**)

6cb was obtained according to the general procedure in 85% yield (62.8 mg), brown solid, R_f = 0.2 (PE/EA = 5/1).

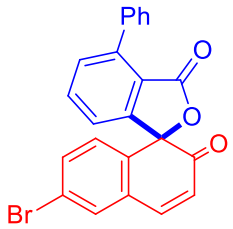
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.64 – 7.59 (m, 3H), 7.52 (t, J = 7.6 Hz, 1H), 7.50 – 7.43 (m, 4H), 7.41 (d, J = 8.4 Hz, 1H), 7.18 (d, J = 7.7 Hz, 1H), 6.90 (dd, J = 8.4, 2.6 Hz, 1H), 6.78 (d, J = 2.7 Hz, 1H), 6.15 (d, J = 9.9 Hz, 1H), 3.75 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 193.0, 169.3, 162.1, 149.8, 146.9, 143.9, 140.7, 136.1, 134.3, 132.2, 131.8, 129.8, 128.7, 128.2, 122.6, 121.1, 120.1, 119.6, 114.7, 112.7, 86.6, 55.7. HRMS (ESI) calculated for $C_{24}H_{16}NaO_4^+$ [M+Na]$^+$: 391.0941, found: 391.0944.



7'-Bromo-4-phenyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6cc**)

6cc was obtained according to the general procedure in 77% yield (64.2 mg), yellow solid, R_f = 0.3 (PE/EA = 5/1).

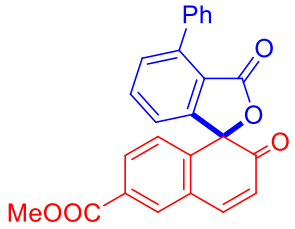
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 – 7.59 (m, 3H), 7.56 – 7.53 (m, 2H), 7.51 – 7.46 (m, 4H), 7.39 (s, 1H), 7.34 (d, J = 8.1 Hz, 1H), 7.15 (d, J = 7.7 Hz, 1H), 6.32 (d, J = 10.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.1, 168.8, 148.9, 145.7, 144.2, 140.0, 135.9, 134.5, 133.0, 132.1, 131.6, 129.9, 129.7, 128.8, 128.6, 128.2, 125.9, 124.3, 120.0, 119.6, 85.8. HRMS (ESI) calculated for $C_{23}H_{13}BrNaO_3^+$ [M+Na]$^+$: 438.9940, found: 438.9941.



6'-Bromo-4-phenyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-2',3-dione (**6cd**)

6cd was obtained according to the general procedure in 84% yield (69.7 mg), brown solid, R_f = 0.2 (PE/EA = 5/1).

^{1}H NMR (600 MHz, Acetone-d_6) δ 7.99 (d, J = 10.1 Hz, 1H), 7.94 (d, J = 2.1 Hz, 1H), 7.72 (t, J = 7.7 Hz, 1H), 7.64 – 7.61 (m, 3H), 7.57 (d, J = 7.5 Hz, 1H), 7.52 – 7.46 (m, 3H), 7.37 – 7.34 (m, 1H), 7.32 (d, J = 8.3 Hz, 1H), 6.42 (d, J = 10.1 Hz, 1H). ^{13}C NMR (151 MHz, Acetone-d_6) δ 193.1, 169.2, 150.3, 146.6, 144.2, 138.0, 137.1, 135.7, 134.4, 133.9, 133.1, 132.8, 130.5, 129.5, 129.3, 128.8, 125.6, 123.9, 121.1, 120.5, 86.4. HRMS (ESI) calculated for $C_{23}H_{13}BrNaO_3^+$ $[M+Na]^+$: 438.9940, found: 438.9947.



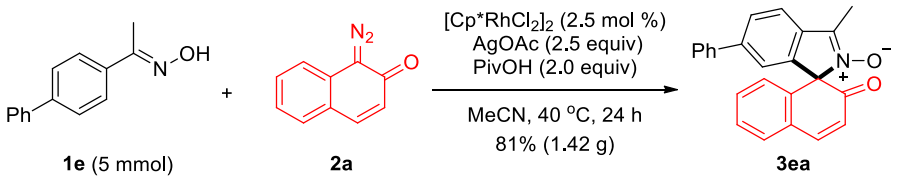
Methyl 2',3-dioxo-4-phenyl-2'*H*,3*H*-spiro[isobenzofuran-1,1'-naphthalene]-6'-carboxylate (**6ce**)

6ce was obtained according to the general procedure in 75% yield (59.0 mg), brown solid, R_f = 0.3 (PE/EA = 5/1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (s, 1H), 8.01 (dd, J = 8.1, 1.8 Hz, 1H), 7.73 (d, J = 10.0 Hz, 1H), 7.61 – 7.58 (m, 2H), 7.53 (t, J = 7.6 Hz, 1H), 7.50 – 7.44 (m, 4H), 7.35 (d, J = 8.1 Hz, 1H), 7.14 (d, J = 7.7 Hz, 1H), 6.37 (d, J = 10.0 Hz, 1H), 3.94 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 192.2, 168.8, 165.7, 148.9, 145.8, 144.2, 142.7, 135.9, 134.5, 132.1, 131.8, 131.7, 131.3, 130.0, 129.7, 128.8, 128.2, 126.9, 124.9, 120.0, 119.6, 86.2, 52.7. HRMS (ESI) calculated for $C_{25}H_{16}NaO_5^+$ $[M+Na]^+$: 419.0890, found: 419.0896.

III. Synthetic Applications

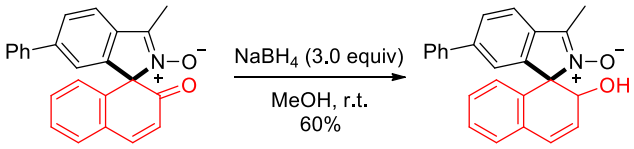
(a) Reaction on a gram scale



1e (5 mmol) **2a** **3ea**

A mixture of oximes (**1e**, 5 mmol), diazo compounds (**2a**, 7.5 mmol), $[Cp^*RhCl_2]_2$ (2.5 mol %), AgOAc (2.5 equiv), PivOH (2.0 equiv) and MeCN (50 mL) were charged into a oven-dried seal-tube. The reaction mixture was stirred under N_2 at 40 ºC for 24 h. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (1:1) to afford the product **3ea** (1.42g, 81%) as a brown solid.

(b) Derivatization of 3ea

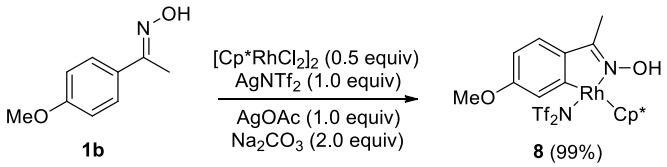


3ea **7**

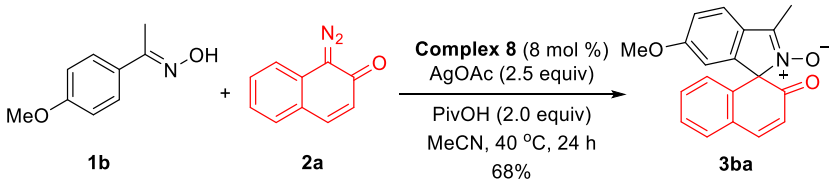
To a solution of **3ea** (35.1 mg, 0.1 mmol) in methanol (1mL) was added $NaBH_4$ (11.4 mg, 0.3 mmol) at room temperature. The mixture was stirred until the complete consumption of the start matreal. Then the mixture was quenched by water and the aqueous layer was extracted with EtOAc for three times, and the combined organic layers were washed with brine, dried, and concentrated. The residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (1:1) to give the product **7** (21.2 mg, 60%) as a white solid. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.64 (dd, J = 7.9, 1.7 Hz, 1H), 7.59 (d, J = 7.8 Hz, 1H), 7.52 (d, J = 1.6 Hz, 1H), 7.50 – 7.48 (m, 2H), 7.44 – 7.41 (m, 2H), 7.35 – 7.32 (m, 1H), 7.31 – 7.29 (m, 1H), 7.28 – 7.25 (m, 1H), 7.11 (td, J = 7.5, 1.5 Hz, 1H), 6.70 – 6.65 (m, 2H), 6.07 (dd, J = 9.9, 2.0 Hz, 1H), 5.55 (s, 1H), 5.39 (s, 1H), 2.44 (s, 3H). ^{13}C NMR (151 MHz, DMSO-d_6) δ 143.8, 139.7, 139.6, 138.8, 133.7, 133.1, 132.7, 129.0, 128.7, 128.3, 127.6, 127.1, 127.0, 126.6, 126.4, 124.5, 119.7, 119.4, 87.4, 70.4, 9.5. HRMS (ESI) calculated for $C_{24}H_{19}NNaO_2^+$ $[M+Na]^+$: 376.1308, found: 376.1306.

IV. Mechanistic Studies

(a) Catalytic reactivity of rhodacyclic complex 8

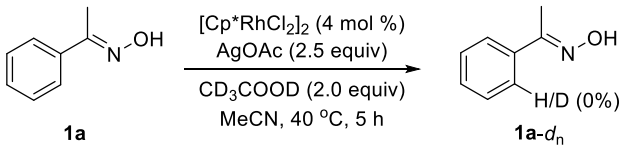


Preparation of cyclometalated complex 8: A mixture of oxime **1b** (8.3 mg 0.05 mmol), $[Cp^*RhCl_2]_2$ (15.5 mg, 0.025 mmol), $AgNTf_2$ (19.4 mg, 0.05 mmol), AgOAc (8.4 mg, 0.05 mmol), Na_2CO_3 (10.6 mg, 0.1 mmol) and 1,2-dichloroethane (1.0 mL) were charged into a pressure tube. The reaction mixture was stirred for 12 h at 60 °C under air. The reaction mixture was filtered through a pad of Celite washing with acetonitrile, and then concentrated under reduced pressure. The residue was washed with n-hexane to give **8**.[3]

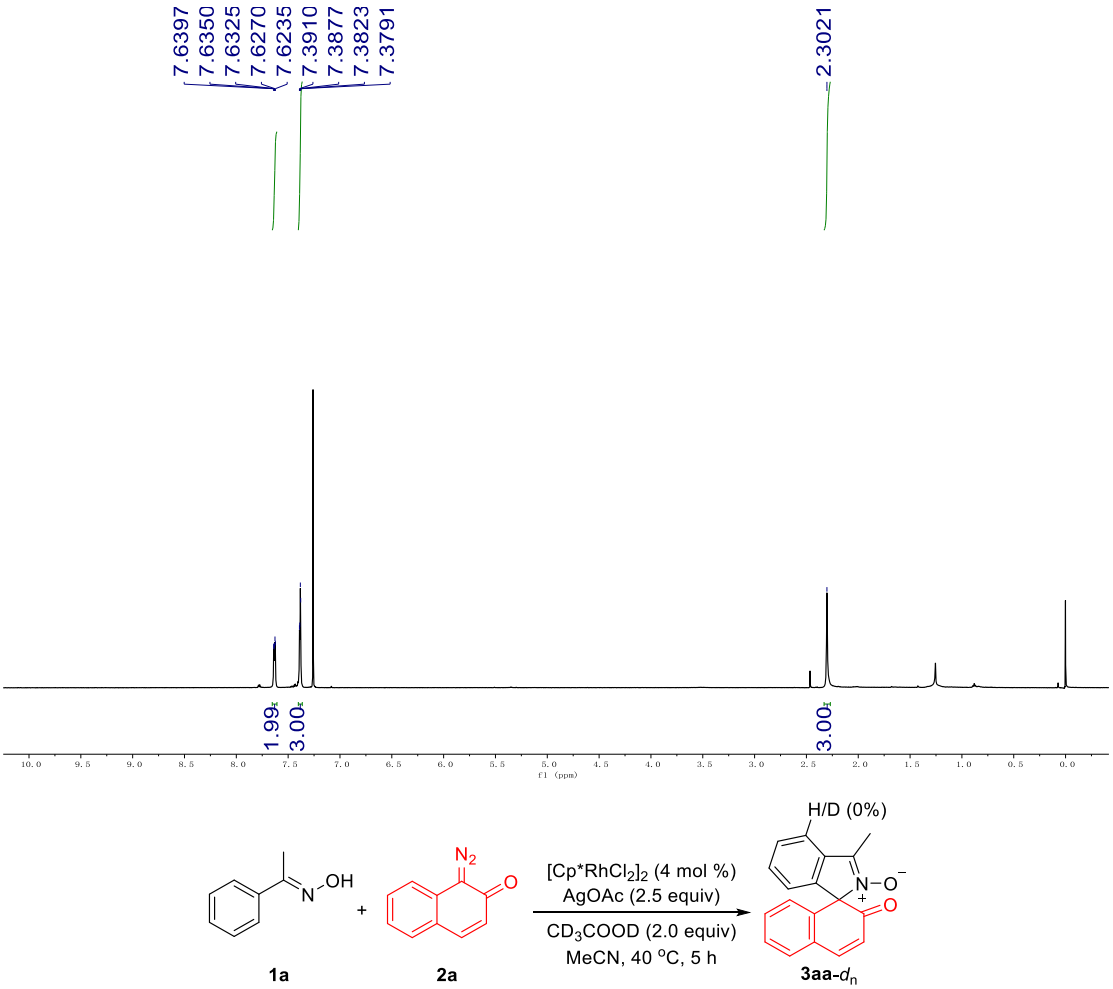


Catalytic reactivity of complex 8: A mixture of oximes (**1b**, 0.2 mmol), diazo compounds (**2a**, 0.3 mmol), **8** (8 mol %), AgOAc (2.5 equiv), PivOH (2.0 equiv) and MeCN (2 mL) were charged into a oven-dried seal-tube. The reaction mixture was stirred under N_2 at 40 °C for 24 h. After the solvent was removed under reduced pressure, the residue was purified by silica gel chromatography using petroleum ether/ethyl acetate (1:1) to afford the product **3ba** (41.5 mg, 68%) as a brown solid.

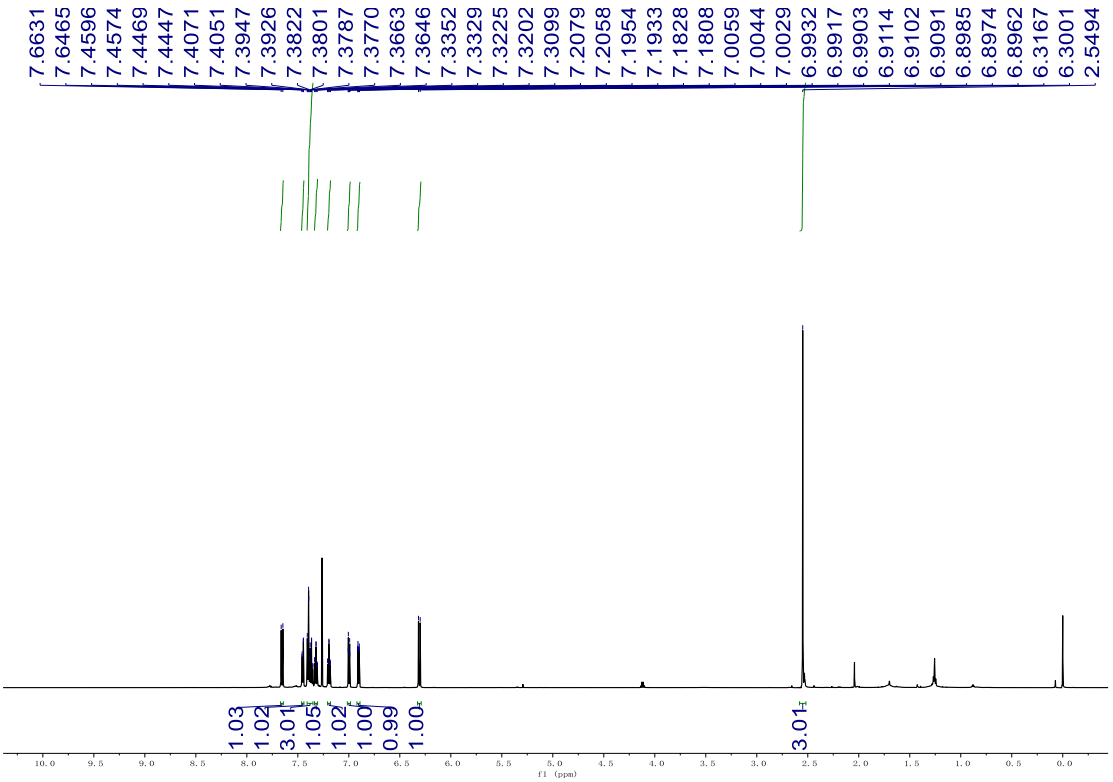
(b) H/D Exchange Experiment



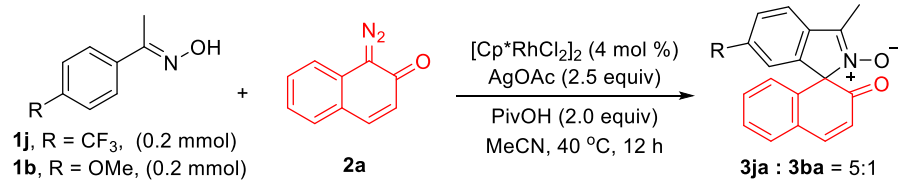
To a pressure tube equipped with a stir bar was charged oximes **1a** (0.1 mmol), $[Cp^*RhCl_2]_2$ (4 mol %), AgOAc (2.5 equiv), CD_3COOD (2.0 equiv), and MeCN (1.0 mL). The reaction mixture was stirred under N_2 at 40 °C for 5 h. After that, the solvent was removed under reduced pressure and the residue was purified by silica gel chromatography using PE/EA to afford the recovered **1a**-d_n. The extent of deuteration was obtained by ^1H NMR analysis.



To a pressure tube equipped with a stir bar was charged oximes **1a** (0.1 mmol), **2a** (0.15 mmol), $[Cp^*RhCl_2]_2$ (4 mol %), AgOAc (2.5 equiv), CD_3COOD (2.0 equiv), and MeCN (1.0 mL). The reaction mixture was stirred under N_2 at 40 ºC for 5 h. After that, the solvent was removed under reduced pressure and the residue was purified by silica gel chromatography using PE/EA to afford the product **3aa**-d_n. The extent of deuteration was obtained by ^1H NMR analysis.



(c) Competition experiment

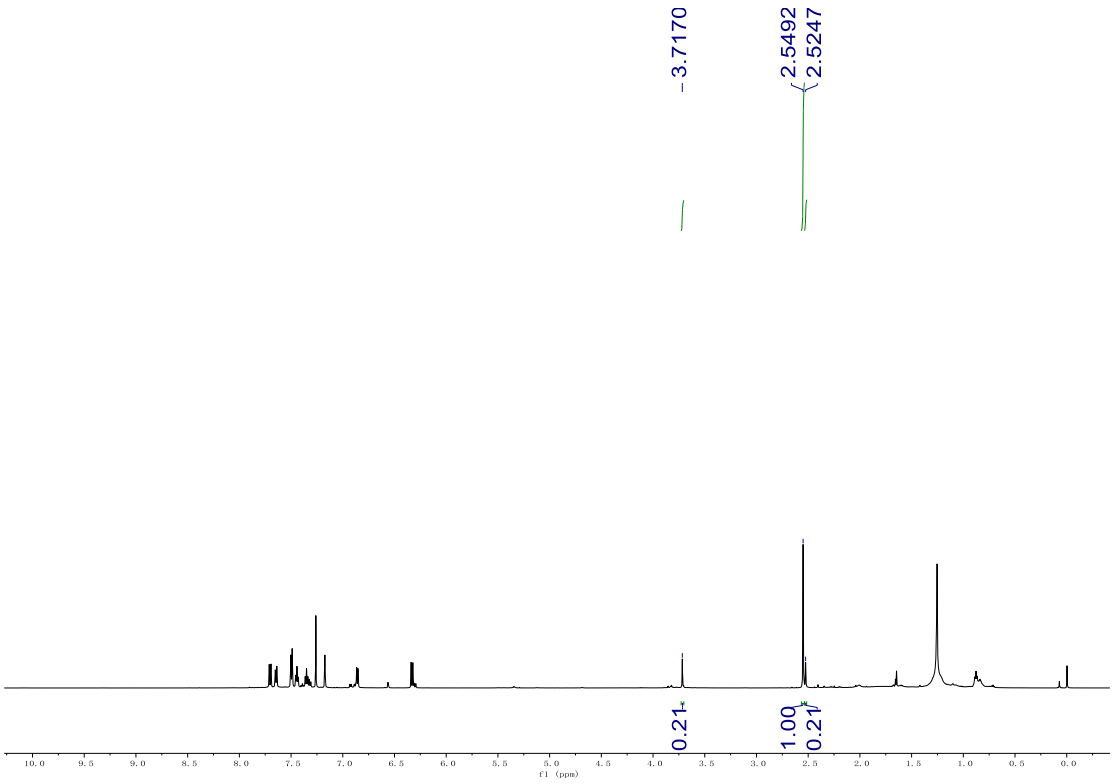


1j, R = CF_3, (0.2 mmol)
1b, R = OMe, (0.2 mmol)

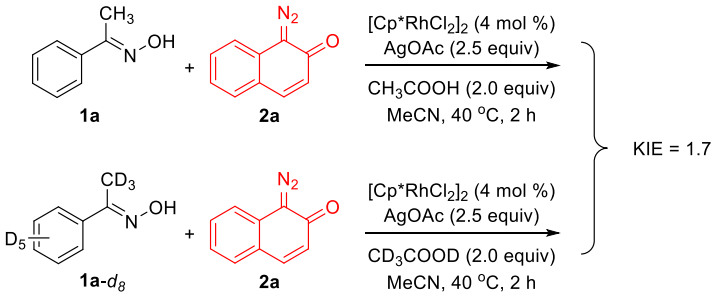
2a

3ja : 3ba = 5:1

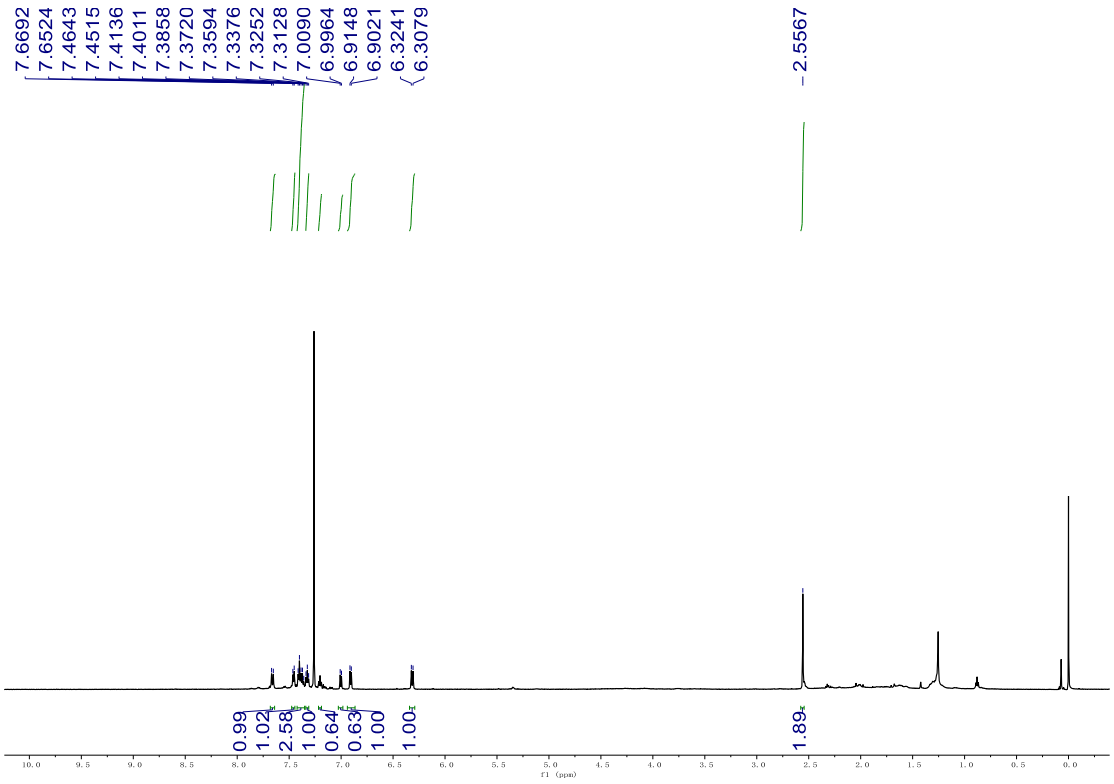
A pressure tube was charged with **1j** (0.2 mmol), **1b** (0.2 mmol), **2a** (0.2 mmol), $[Cp^*RhCl_2]_2$ (4 mol %), AgOAc (2.5 equiv), PivOH (2.0 equiv), and MeCN (2.0 mL). The reaction mixture was stirred at 40 °C for 12 h under N_2. After that, the solvent was removed under reduced pressure and the residue was purified by silica gel chromatography using PE/EA to afford 3ja and 3ba.The ratio of **3ja**:**3ba** = 5:1 was determined on the basis of ^1H NMR analysis.



(d) Parallel kinetic isotope effects



A pressure tube equipped with a stir bar was charged with **1a** (0.1 mmol), **2a** (0.15 mmol), $[Cp^*RhCl_2]_2$ (4 mol %), AgOAc (2.5 equiv), CH_3COOH (2.0 equiv), and MeCN (1.0 mL). To another tube was added **1a-*d*$_8$** (0.1 mmol), **2a** (0.15 mmol), $[Cp^*RhCl_2]_2$ (4 mol %), AgOAc (2.5 equiv), CD_3COOD (2.0 equiv), and MeCN (1.0 mL). The two reaction mixtures were stirred side by side at 40 °C for 2 h under N_2. The reactions tubes were quenched at 0 °C and these two mixtures were rapidly combined. The solvent was rapidly removed under reduced pressure. The resulting residue was purified by silica gel chromatography using PE/EA to afford the mixed products. The KIE (k_H/k_D = 1.7) value was determined on the basis of ^{1}H NMR analysis.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7.3376
7.3252
7.3128
7.0090
6.9964
6.9148
6.9021
6.3241
6.3079
2.5567
0.99
1.02
2.58
1.00
0.64
0.63
1.00
1.00
1.89
f1 (ppm)

V X-Ray Crystallographic Data

The data of **3aa** (CCDC 1992558) can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

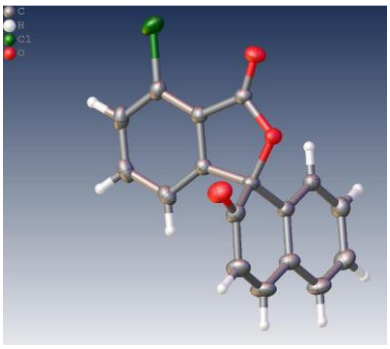


Table S1. Crystal data and structure refinement for **6ea**

Empirical formula	$C_{17}H_9ClO_3$
Formula weight	296.69
Temperature/K	153.0
Crystal system	triclinic
Space group	P-1
a/Å	8.2844(11)
b/Å	8.6986(12)
c/Å	10.7524(15)
α/°	68.114(4)
β/°	78.900(4)
γ/°	68.561(4)
Volume/Å^3	667.83(16)
Z	1
ρcalc g/cm^3	0.738
μ/mm^{-1}	0.146
F(000)	152.0
Crystal size/mm^3	0.15 × 0.11 × 0
Radiation	MoKα (λ = 0.71073)
Index ranges	-10 ≤ h ≤ 10, -11 ≤ k ≤ 11, -13 ≤ l ≤ 13
Reflections collected	41128
Independent reflections	3060 [R_{int} = 0.0450, R_{sigma} = 0.0184]
Data/restraints/parameters	3060/0/190
Goodness-of-fit on F2	1.184

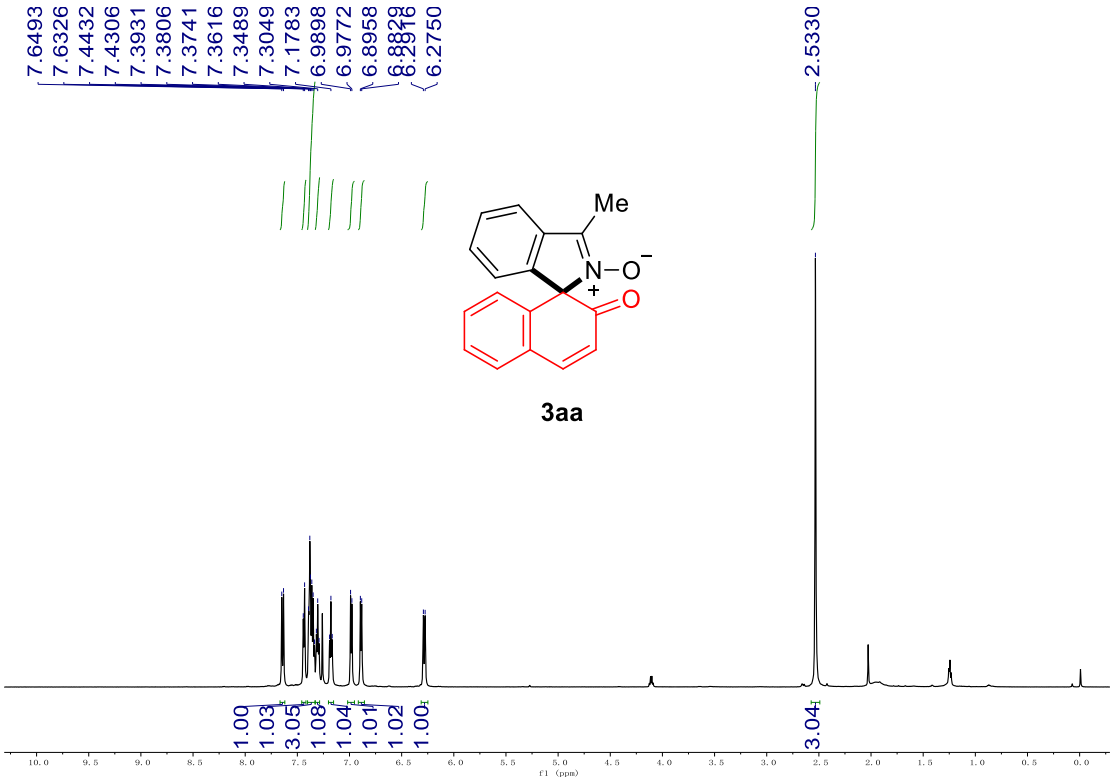
Final R indexes [I>=2σ (I)]	R_1 = 0.0387, wR_2 = 0.1130
Final R indexes [all data]	R_1 = 0.0485, wR_2 = 0.1287
Largest diff. peak/hole / e $Å^{-3}$	0.37/-0.47

VI. References

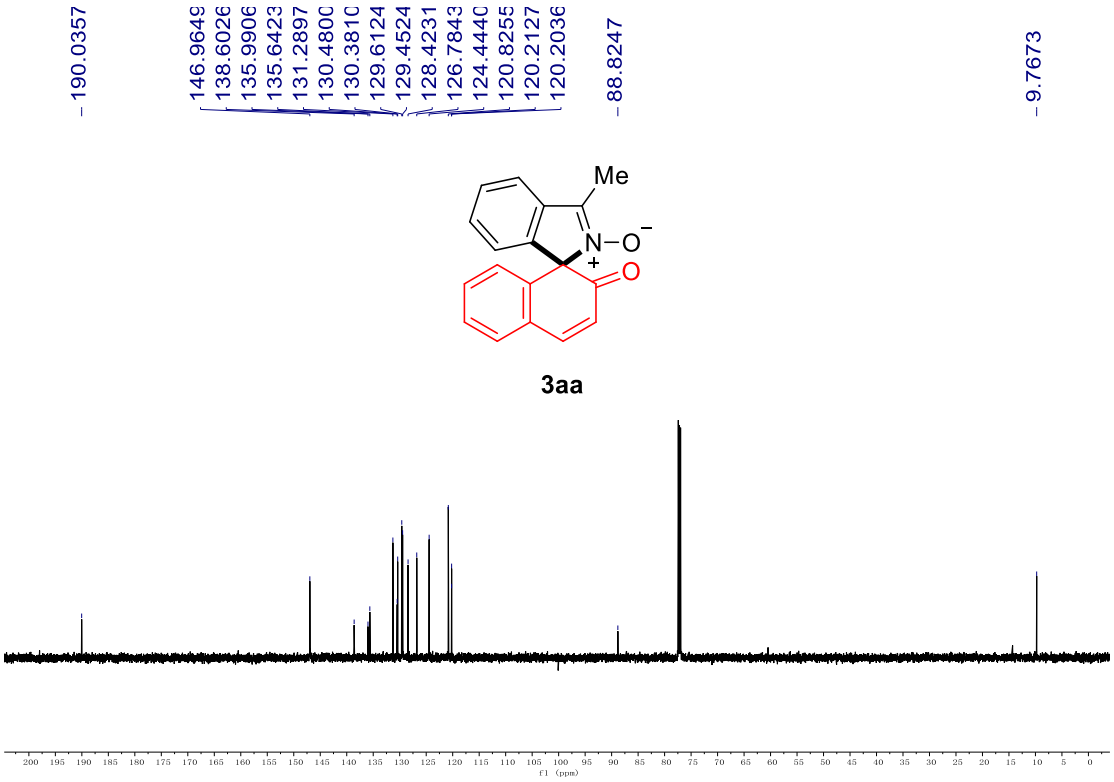
(1) Q. Wang, F. Wang, X. Yang, X. Zhou and X. Li, *Org. Lett.,* 2016, **18**, 6144.
(2) Z. Liu, J. Wu and S. Yang, *Org. Lett.,* 2017, **19**, 5434.
(3) H. Kim and S. Chang, *Angew. Chem. Int. Ed*., 2017, **56**, 3344-3348.

VII. NMR Spectra

3aa

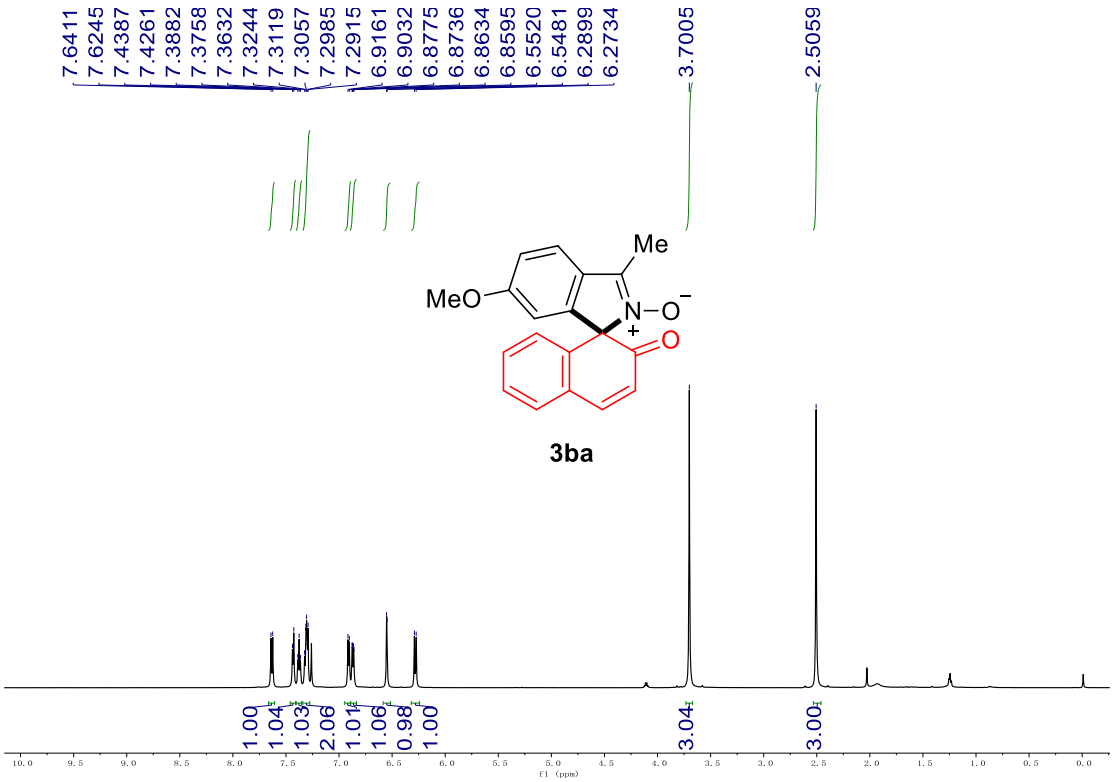


3aa



3aa

3ba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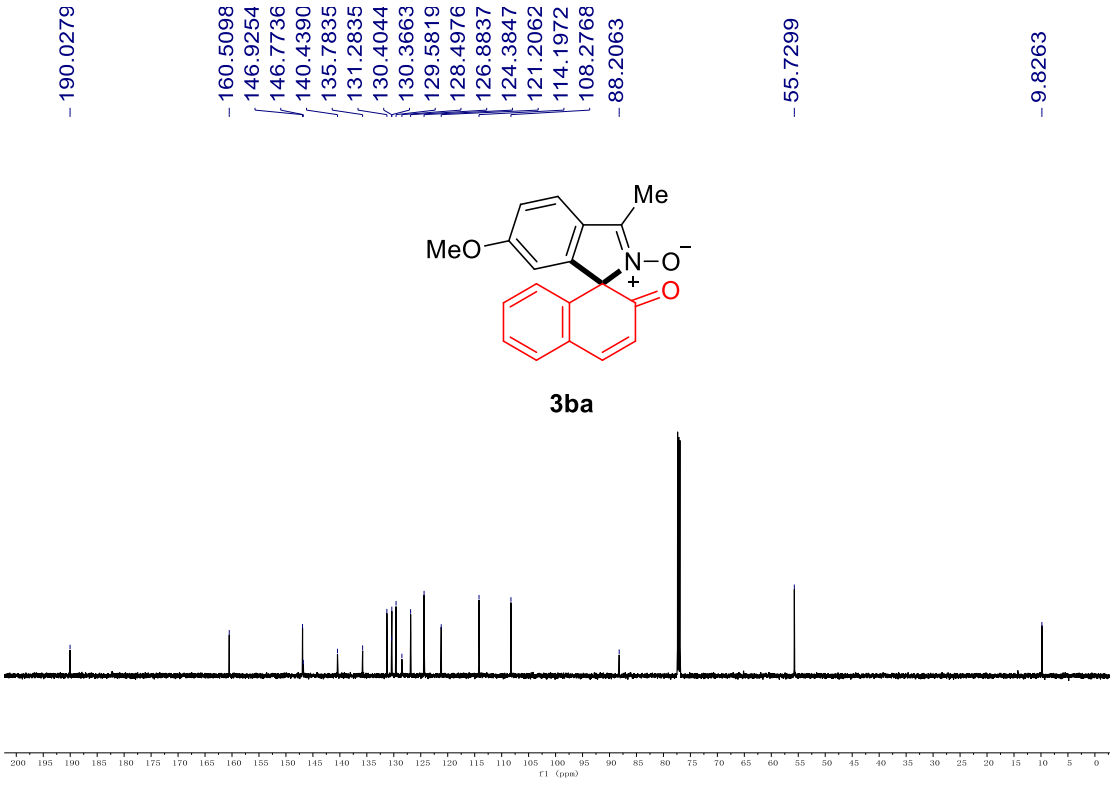
7.2985
7.2915
6.9161
6.9032
6.8775
6.8736
6.8634
6.8595
6.5520
6.5481
6.2899
6.2734
3.7005
2.5059
Me
MeO
N–O
+
O
3ba
1.00
1.04
1.03
2.06
1.01
1.06
0.98
1.00
3.04
3.00
f1 (ppm)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128.4976
126.8837
124.3847
121.2062
114.1972
108.2768
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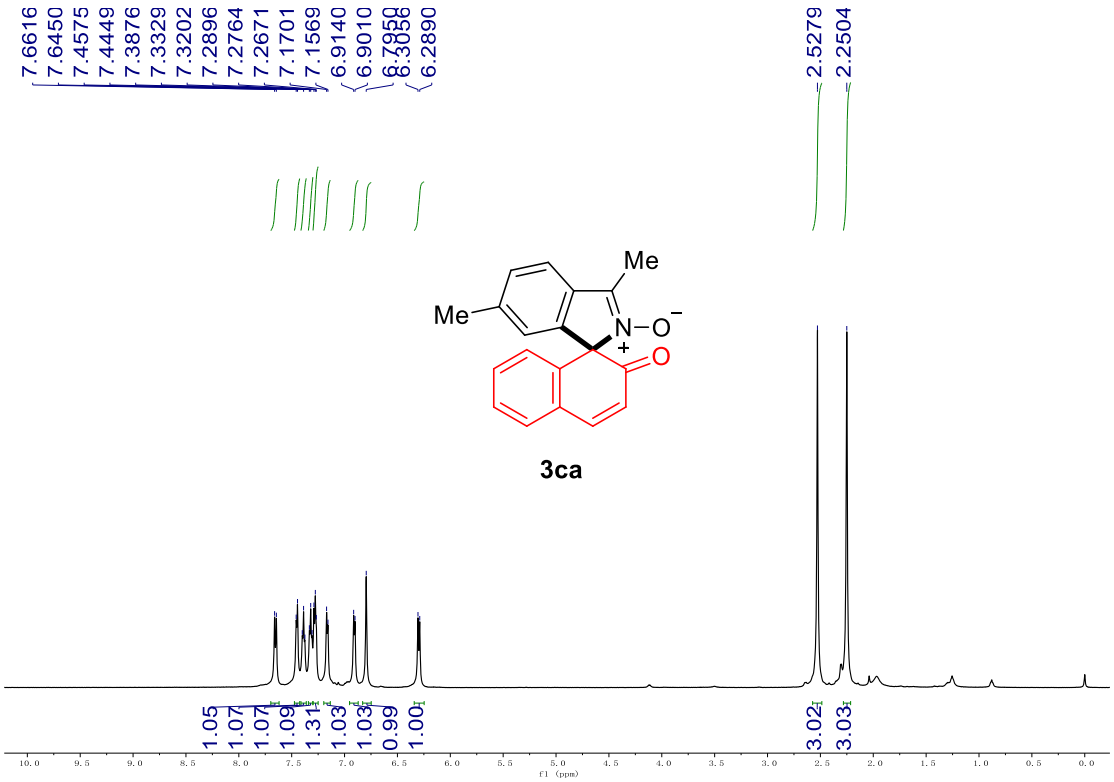
55.7299
9.8263
Me
MeO
N–O
+
O
3ba
f1 (ppm)

3ca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7.1701
7.1569
6.9140
6.9010
6.7950
6.3058
6.2890
2.5279
2.2504
Me
Me
N–O
O
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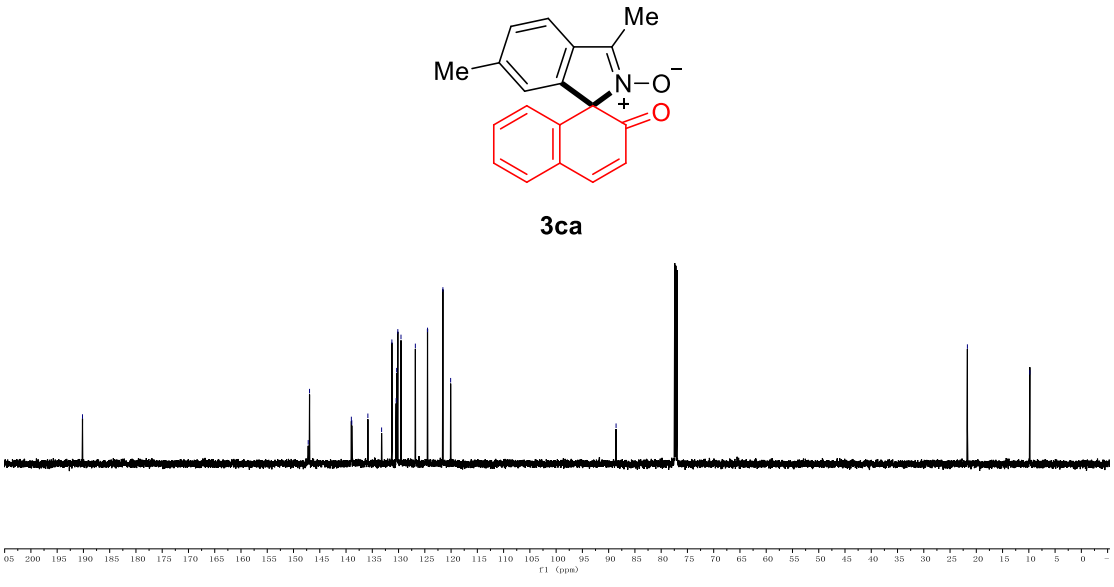
1.07
1.07
1.09
1.31
1.03
1.03
0.99
1.00
3.02
3.03

3ca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130.1391
129.5362
126.8020
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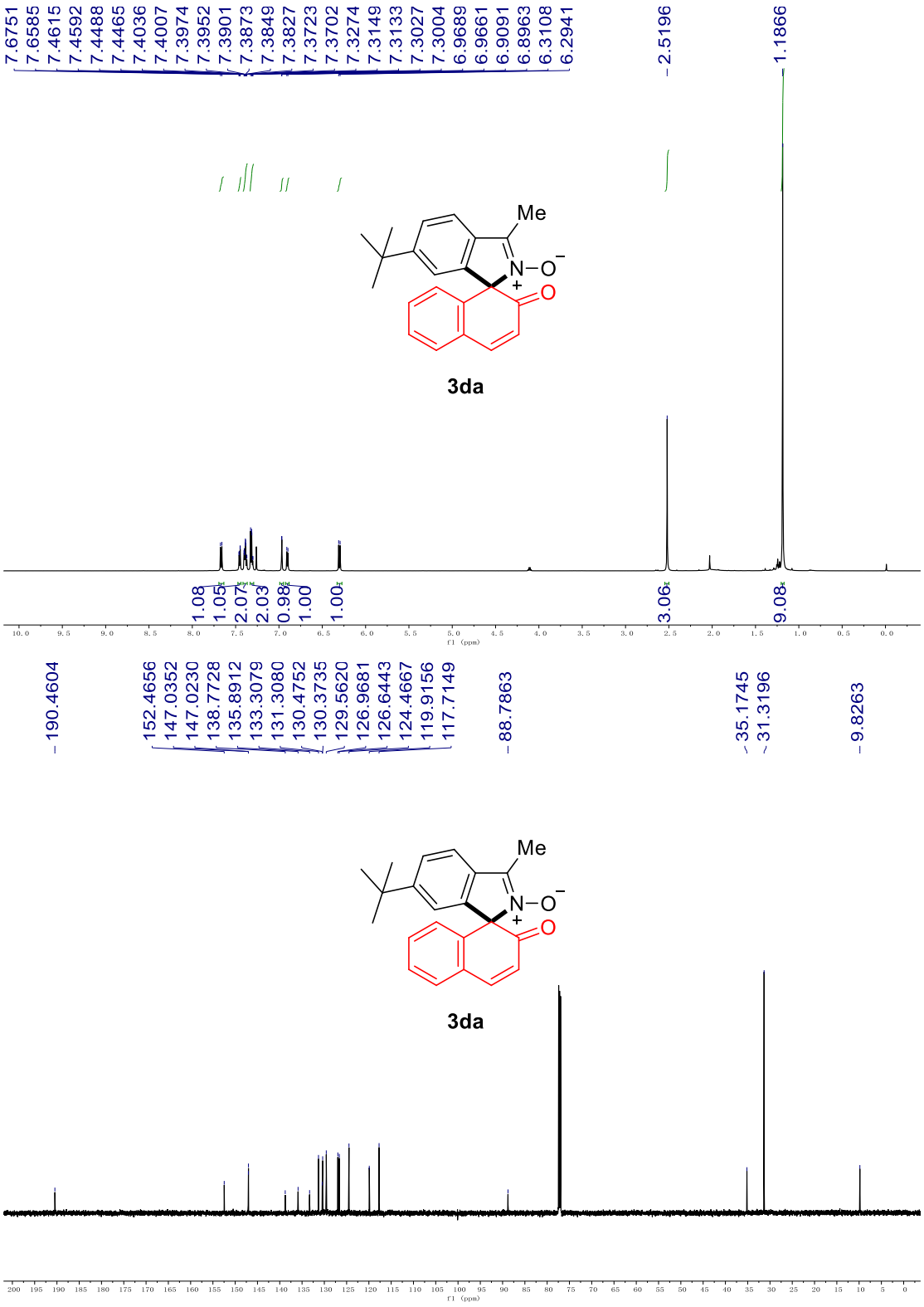
21.6940
9.8139
Me
Me
N–O
O

3ca

3da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7.3027
7.3004
6.9689
6.9661
6.9091
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6.3108
6.2941
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1.1866
Me
N–O
O
3da
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1.05
2.07
2.03
0.98
1.00
1.00
3.06
9.08
f1 (ppm)
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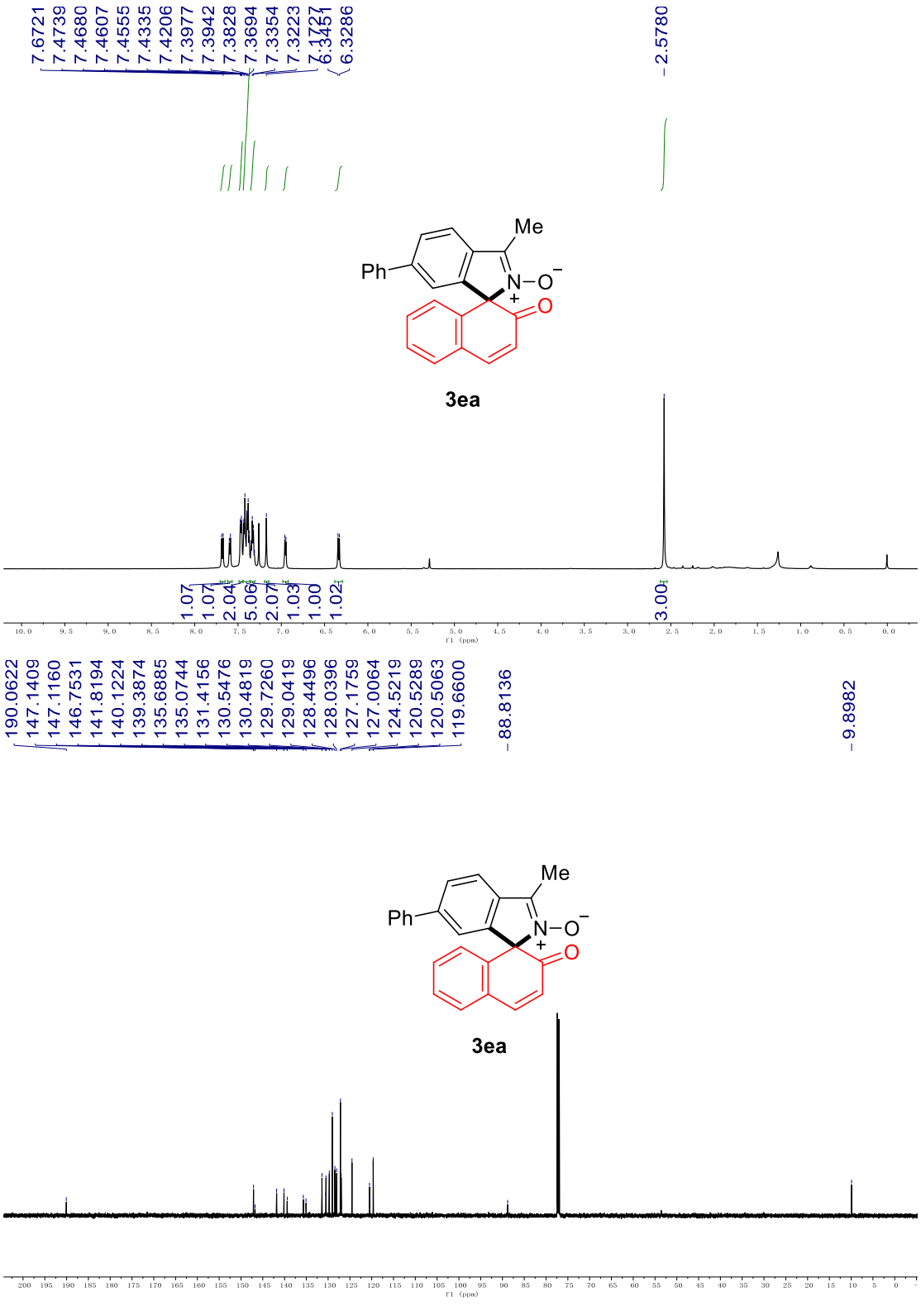
N–O
O
3da
f1 (ppm)

3ea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7.3354
7.3223
7.1727
6.3451
6.3286
2.5780
Me
Ph
N−O
+
O
3ea
1.07
1.07
2.04
5.06
2.07
1.03
1.00
1.02
3.00
f1 (ppm)
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Ph
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+
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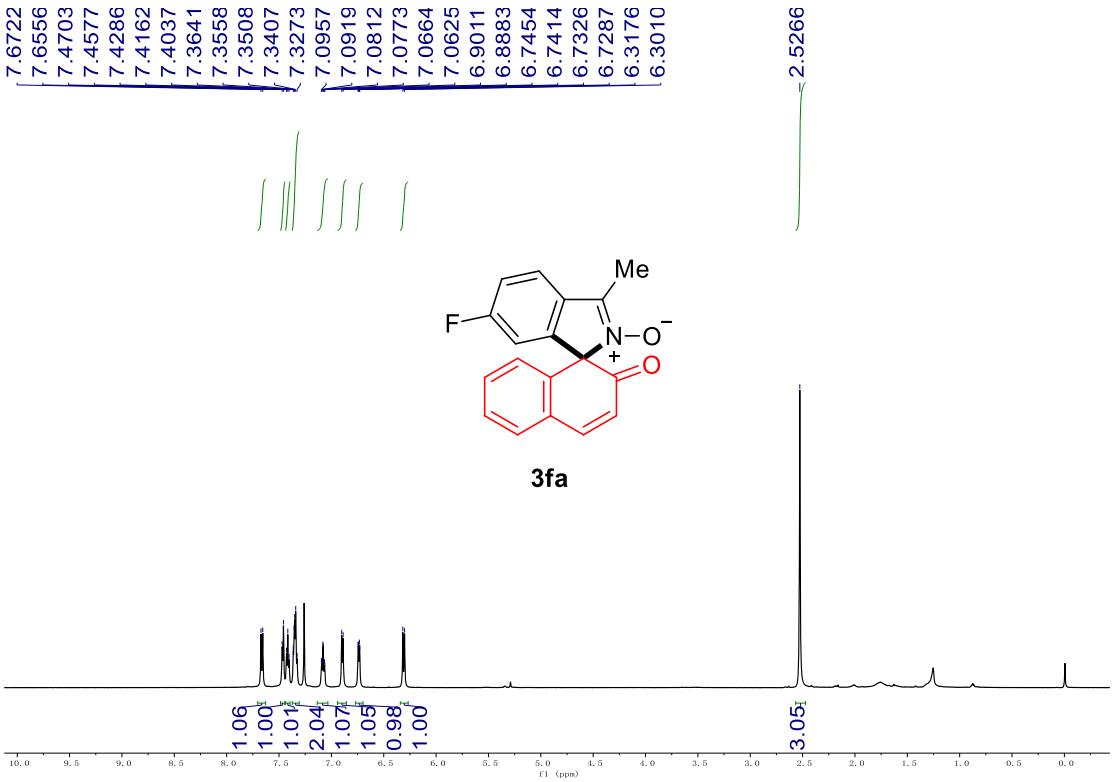
3ea
f1 (ppm)

3fa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6.7326
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Me
F
N–O
+
O
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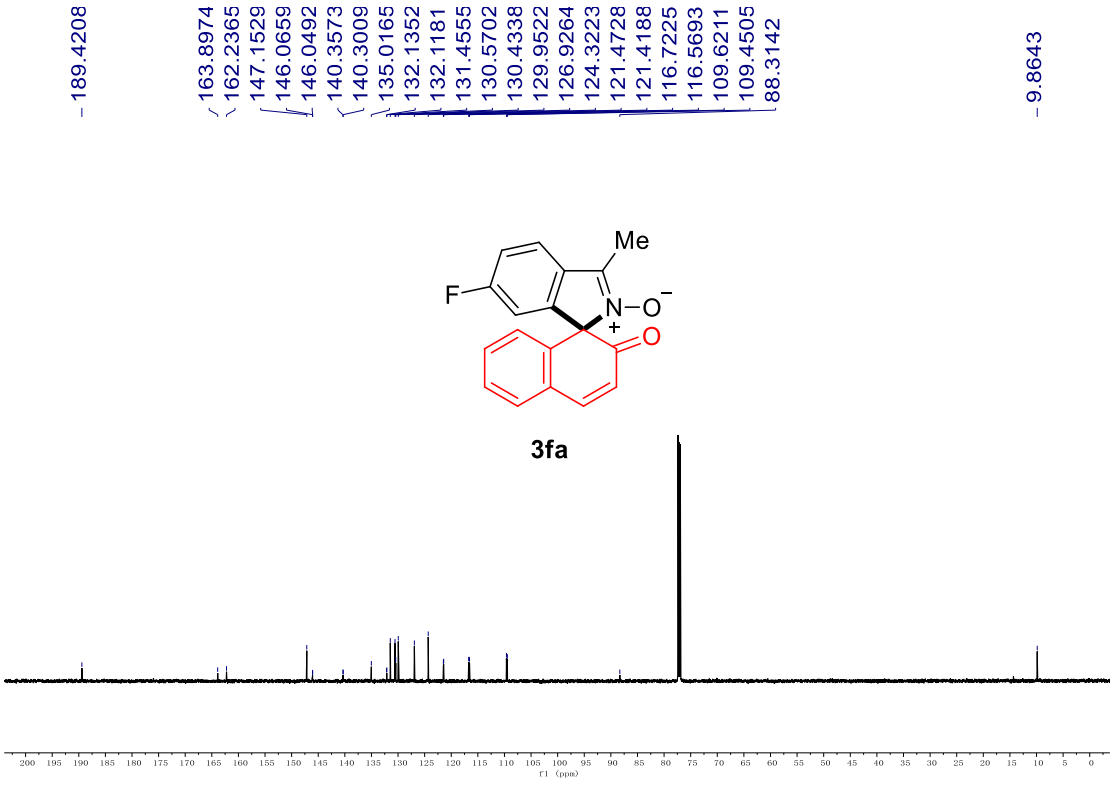
1.00
3.05
f1 (ppm)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+
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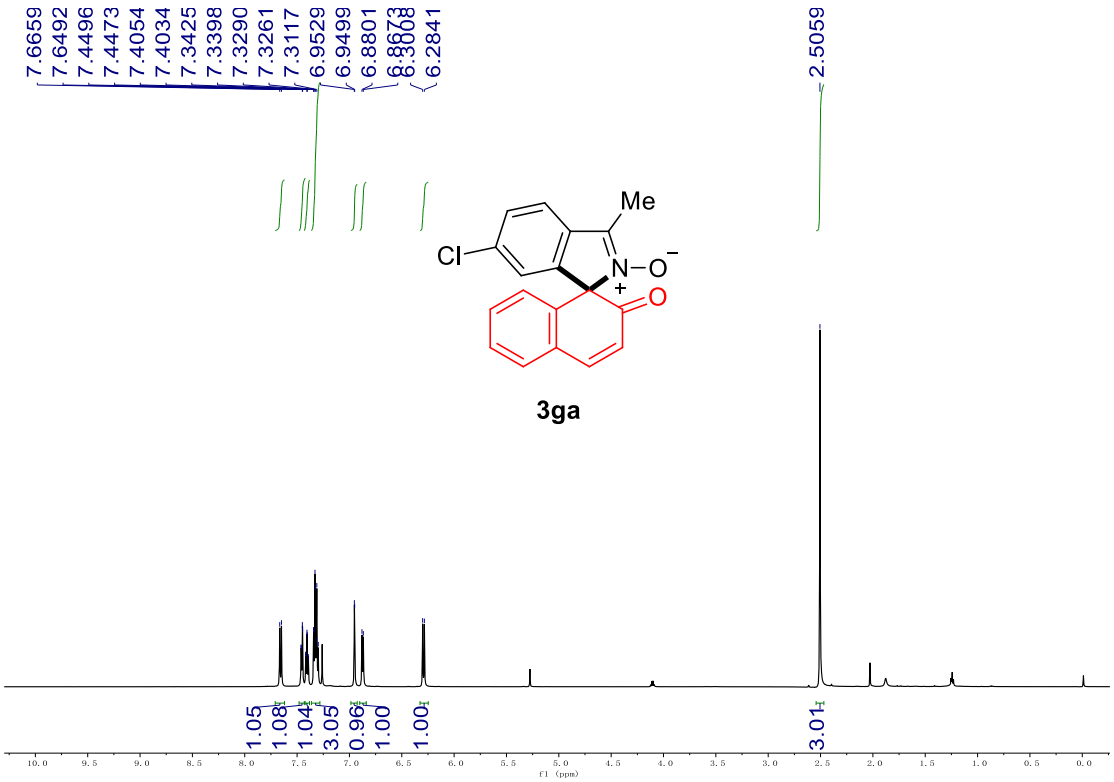
f1 (ppm)

3ga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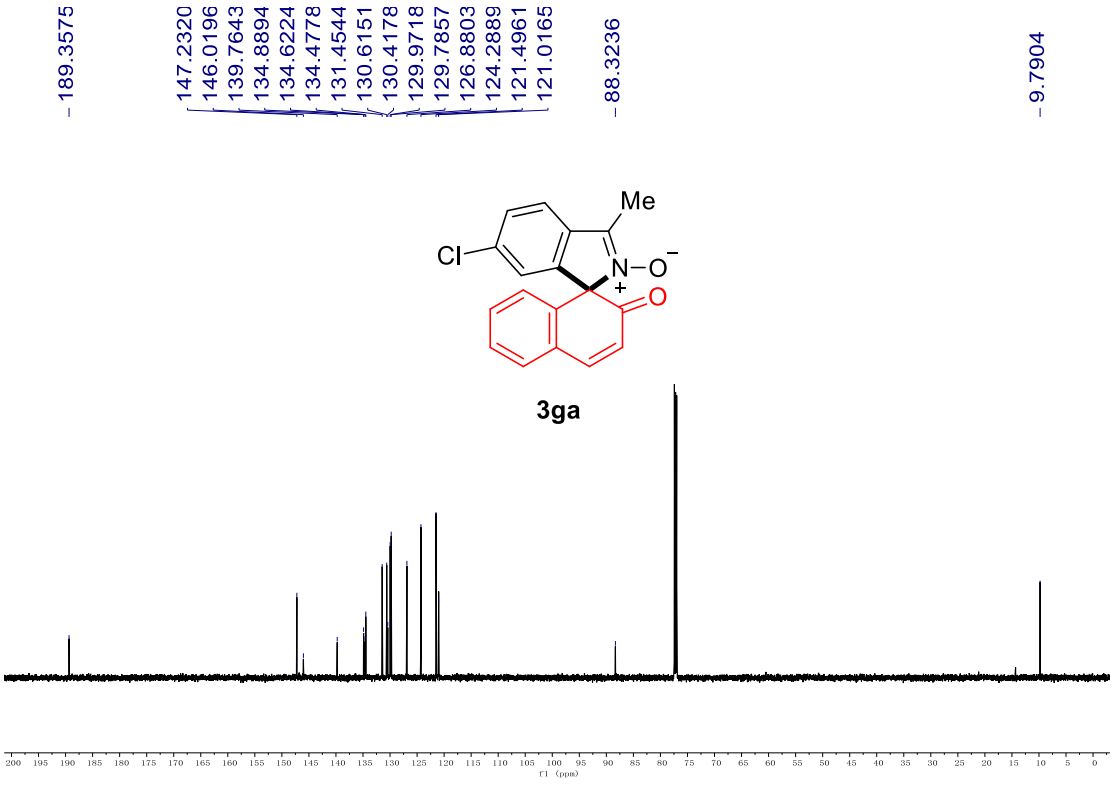
f1 (ppm)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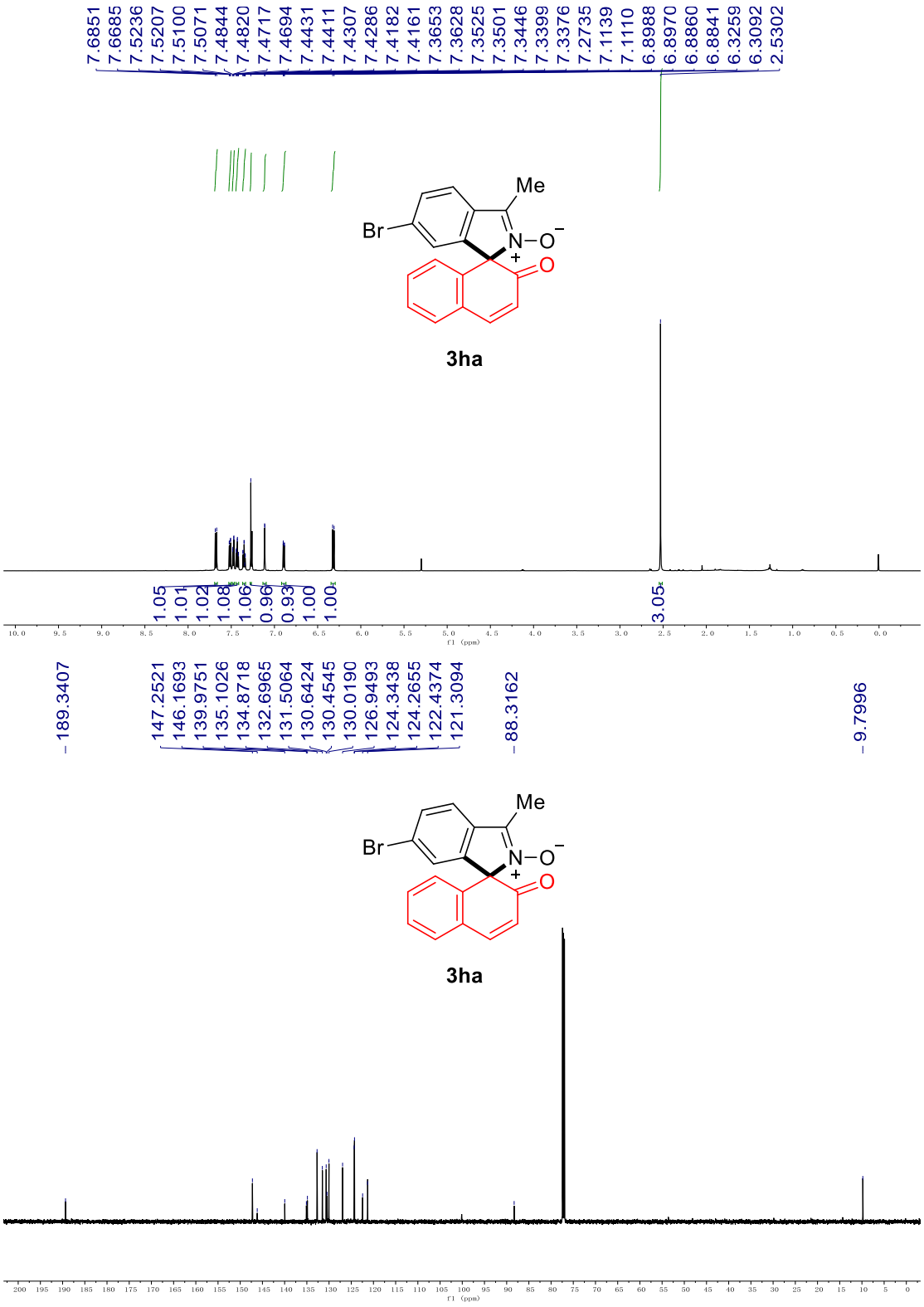
3ha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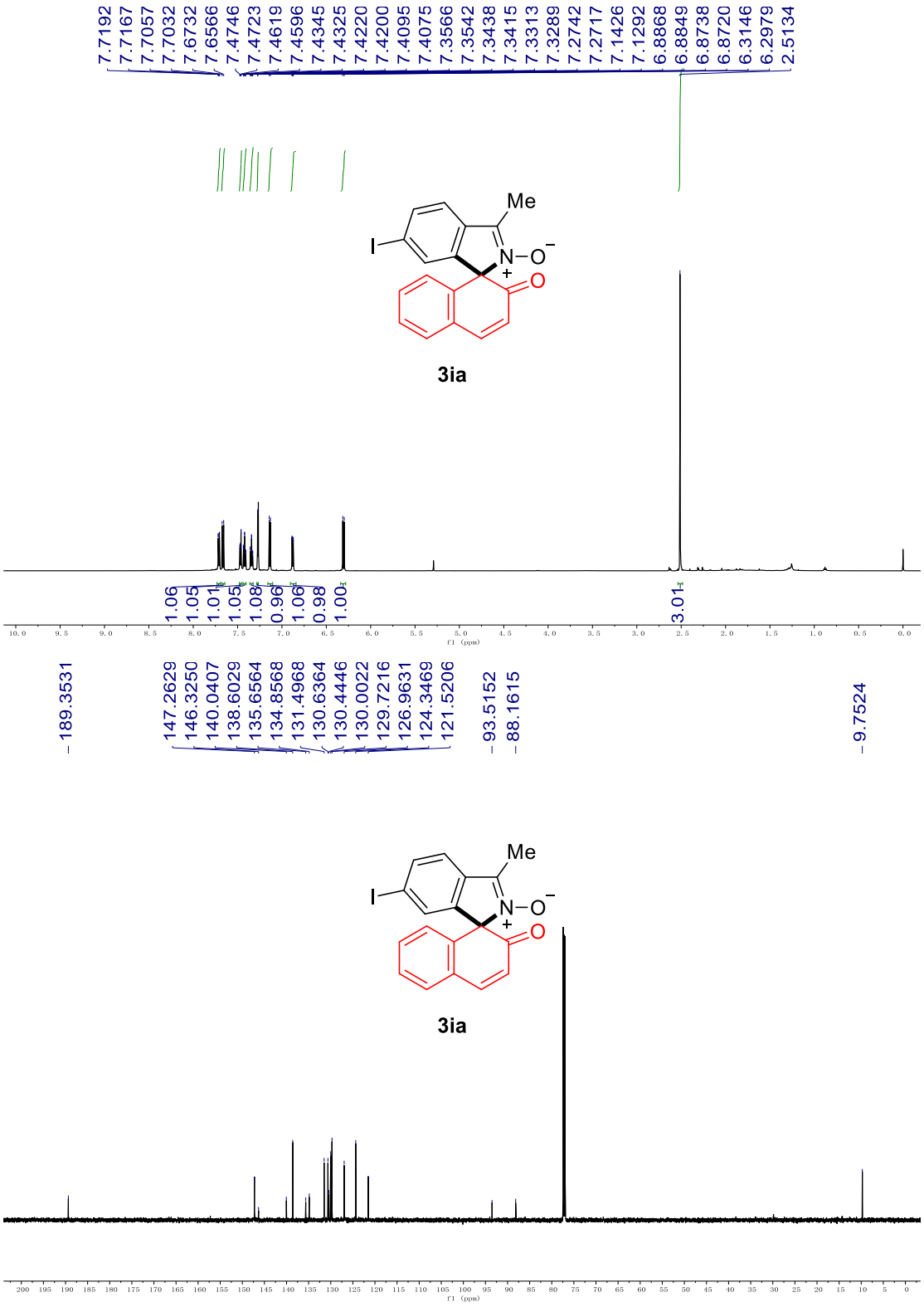
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I
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+
O
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N–O
+
O
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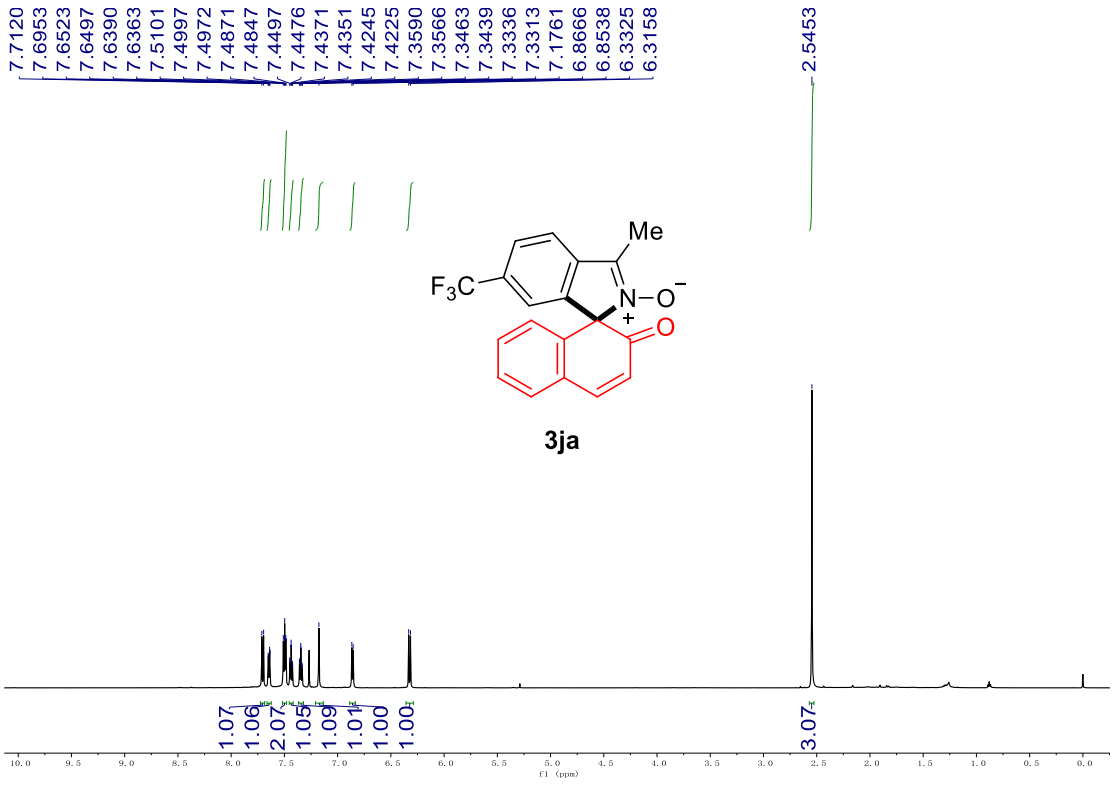
f1 (ppm)

3ja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7.3463
7.3439
7.3336
7.3313
7.1761
6.8666
6.8538
6.3325
6.3158
2.5453
Me
F3C
N–O
+
O
3ja
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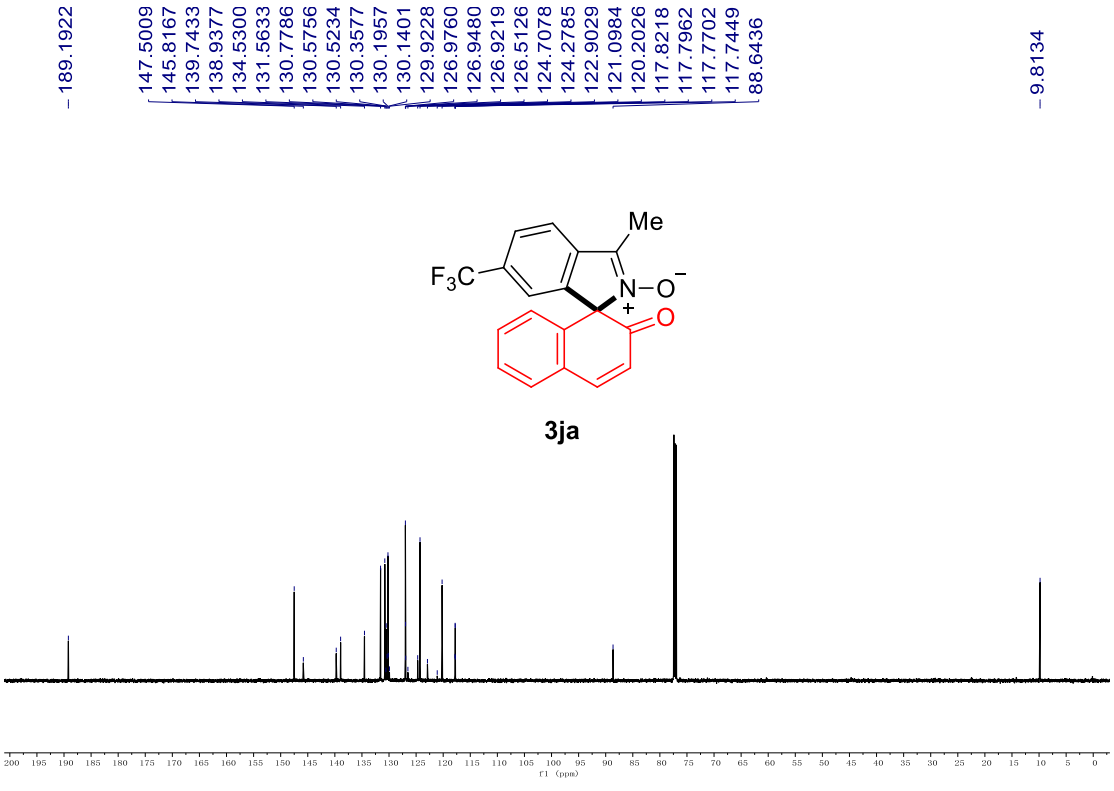
1.06
2.07
1.05
1.09
1.01
1.00
1.00
3.07
f1 (ppm)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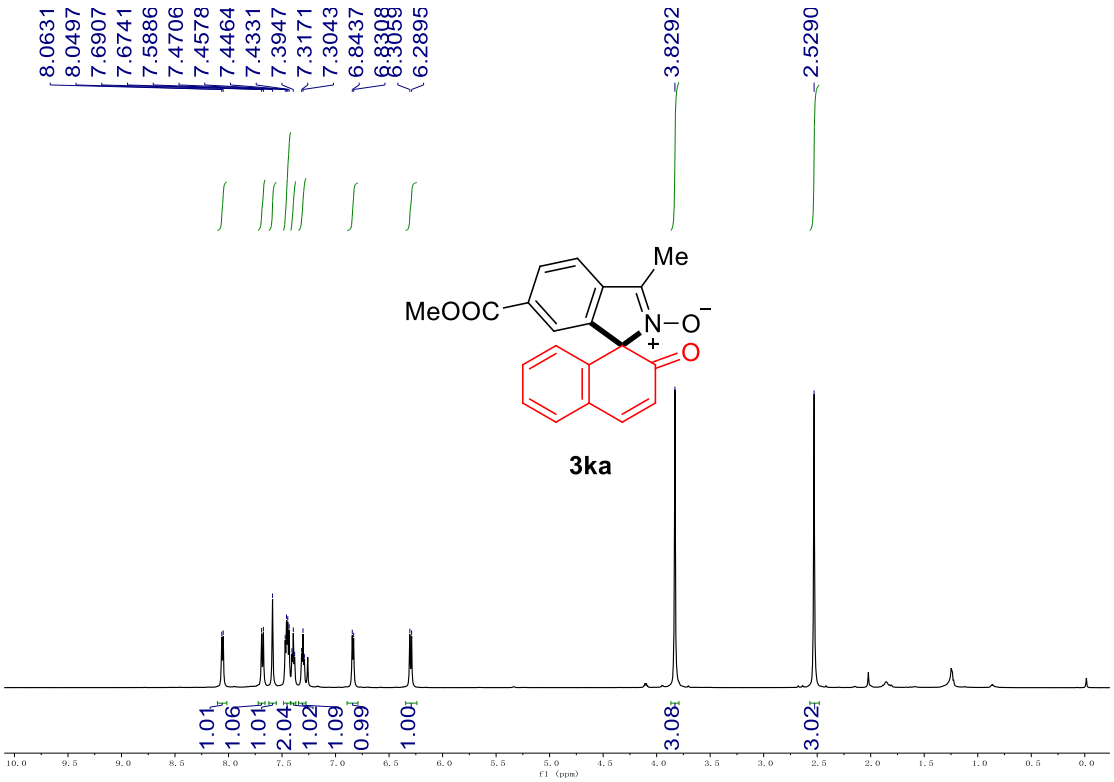
130.1957
130.1401
129.9228
126.9760
126.9480
126.9219
126.5126
124.7078
124.2785
122.9029
121.0984
120.2026
117.8218
117.7962
117.7702
117.7449
88.6436
9.8134
Me
F3C
N–O
+
O
3ja
f1 (ppm)

3ka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7.6907
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7.5886
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7.4578
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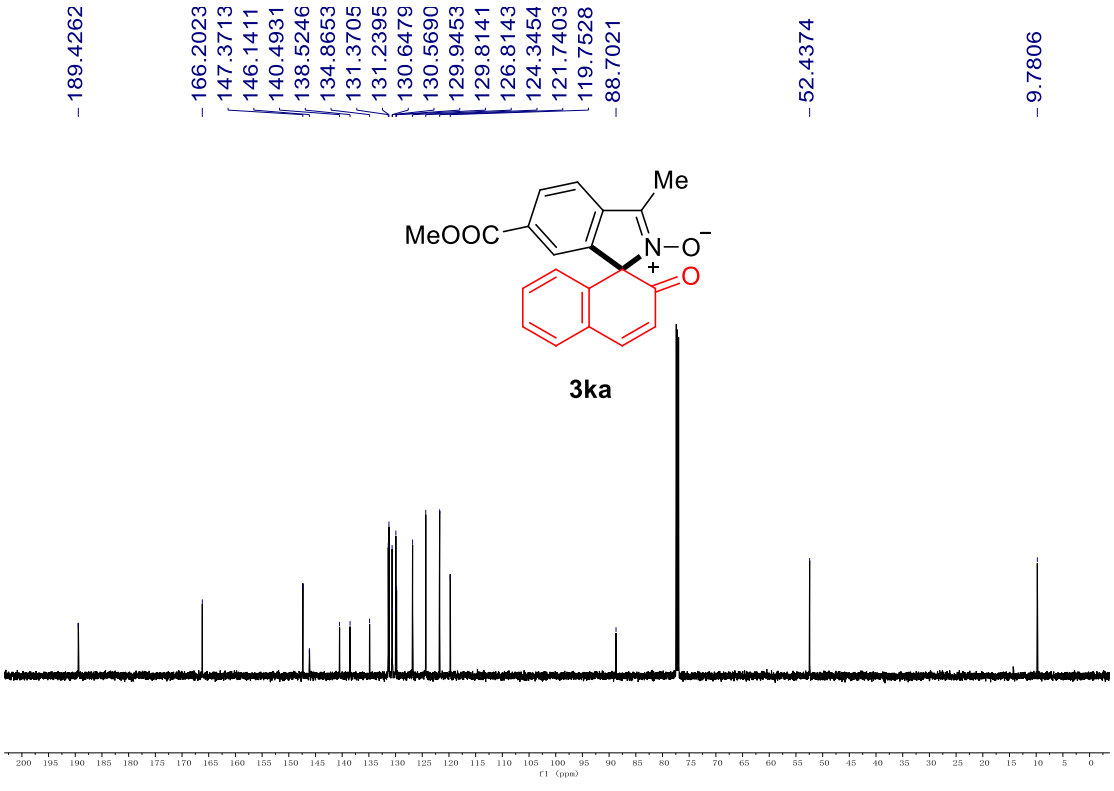
7.4331
7.3947
7.3171
7.3043
6.8437
6.2895
3.8292
2.5290
Me
MeOOC
N–O
O
3ka
1.01
1.06
1.01
2.04
1.02
1.09
0.99
1.00
3.08
3.02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138.5246
134.8653
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131.2395
130.6479
130.5690
129.9453
129.8141
126.8143
124.3454
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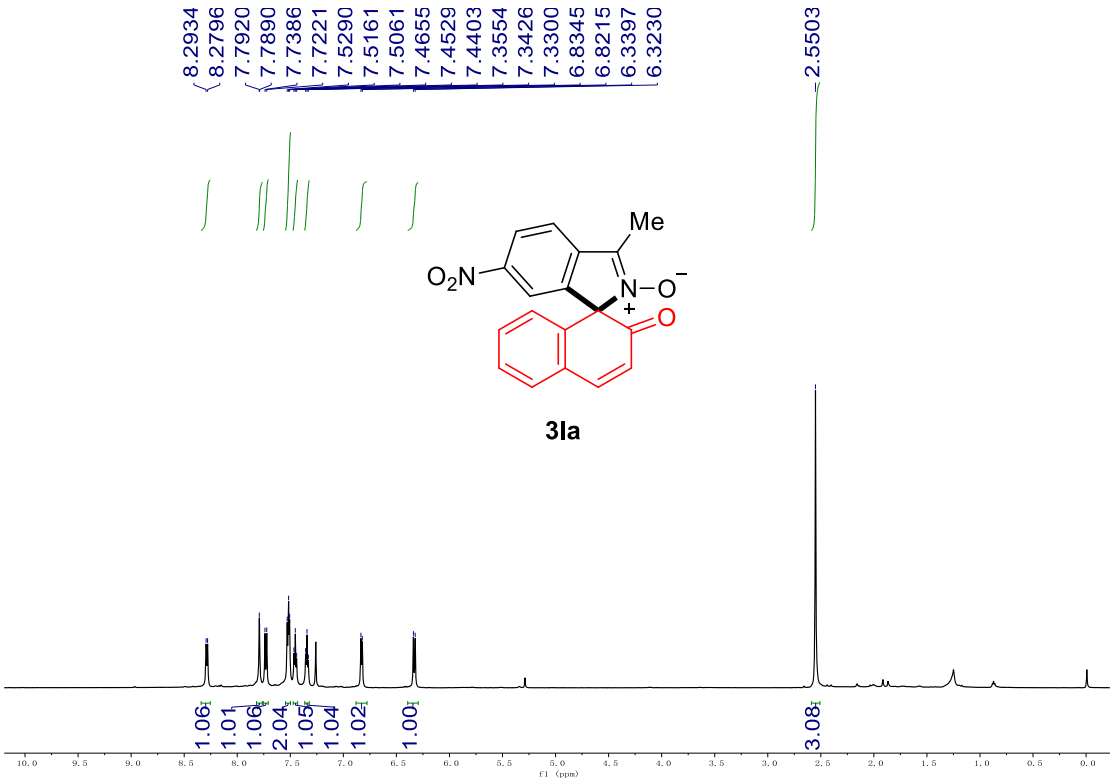
119.7528
88.7021
52.4374
9.7806
Me
MeOOC
N–O
O
3ka

3la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8.2796
7.7920
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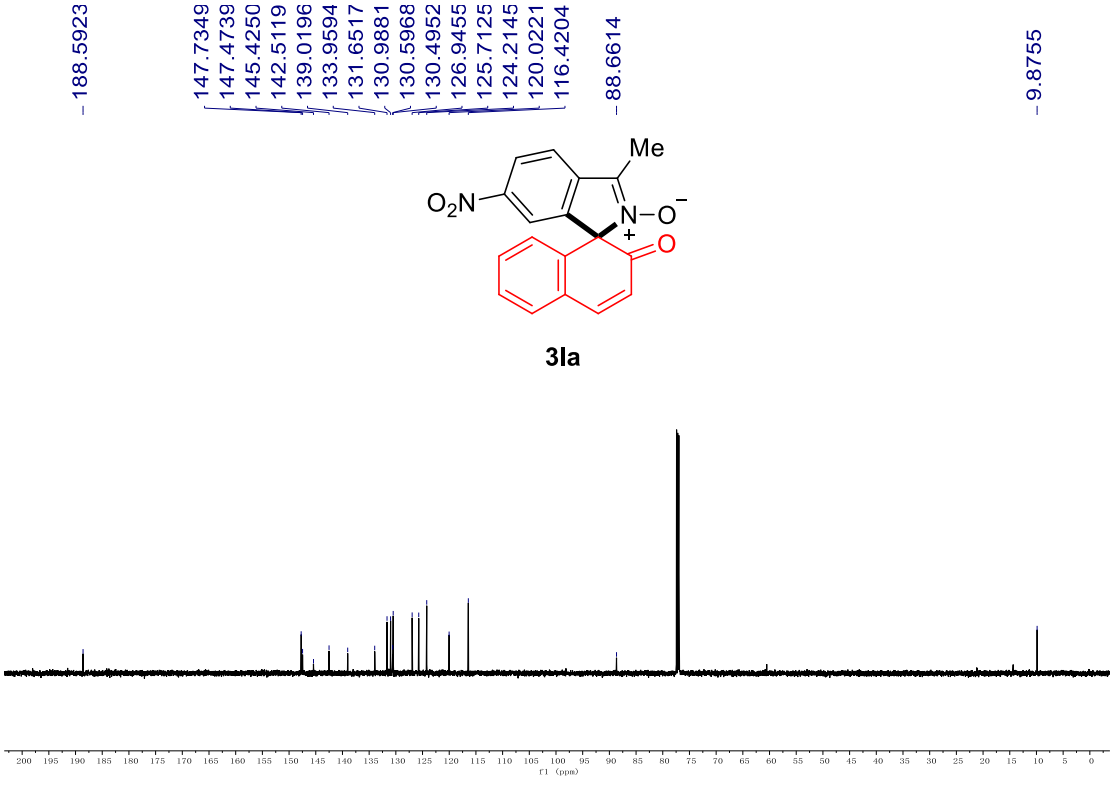
7.7221
7.5290
7.5161
7.5061
7.4655
7.4529
7.4403
7.3554
7.3426
7.3300
6.8345
6.8215
6.3397
6.3230
2.5503
Me
O2N
N−O
+
O
1.06
1.01
1.06
2.04
1.05
1.04
1.02
1.00
3.08
f1 (ppm)

3la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147.4739
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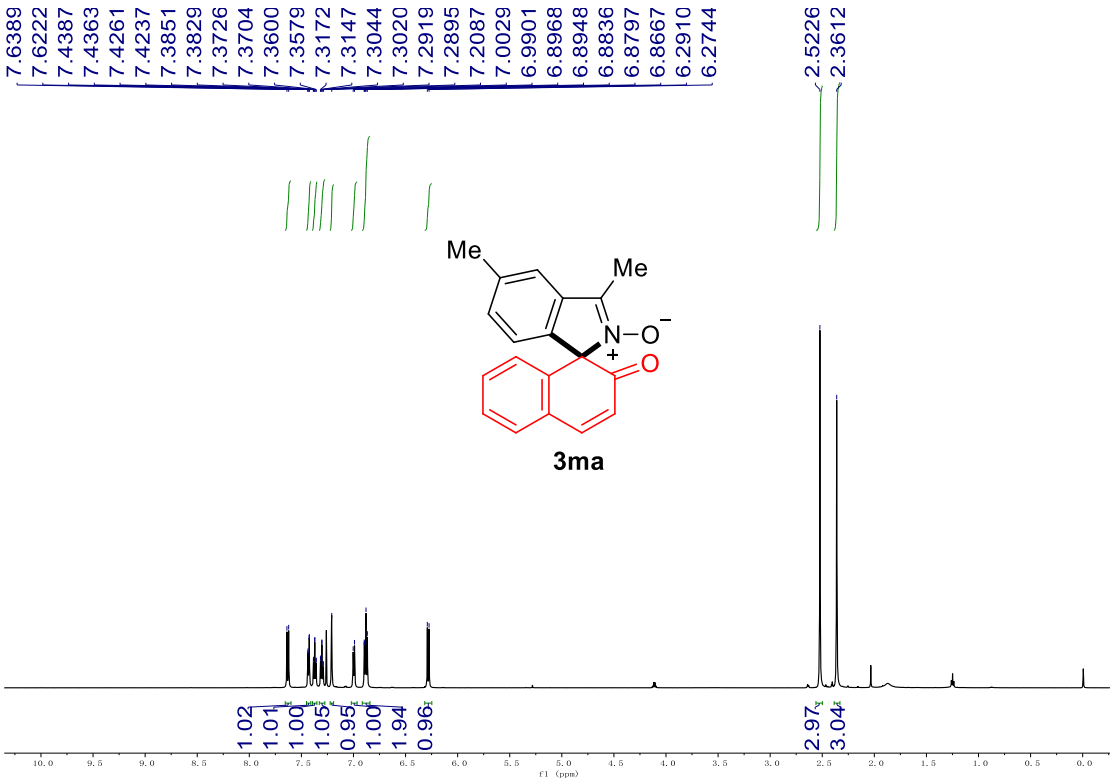
133.9594
131.6517
130.9881
130.5968
130.4952
126.9455
125.7125
124.2145
120.0221
116.4204
88.6614
9.8755
Me
O2N
N−O
+
O
f1 (ppm)

3ma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7.4387
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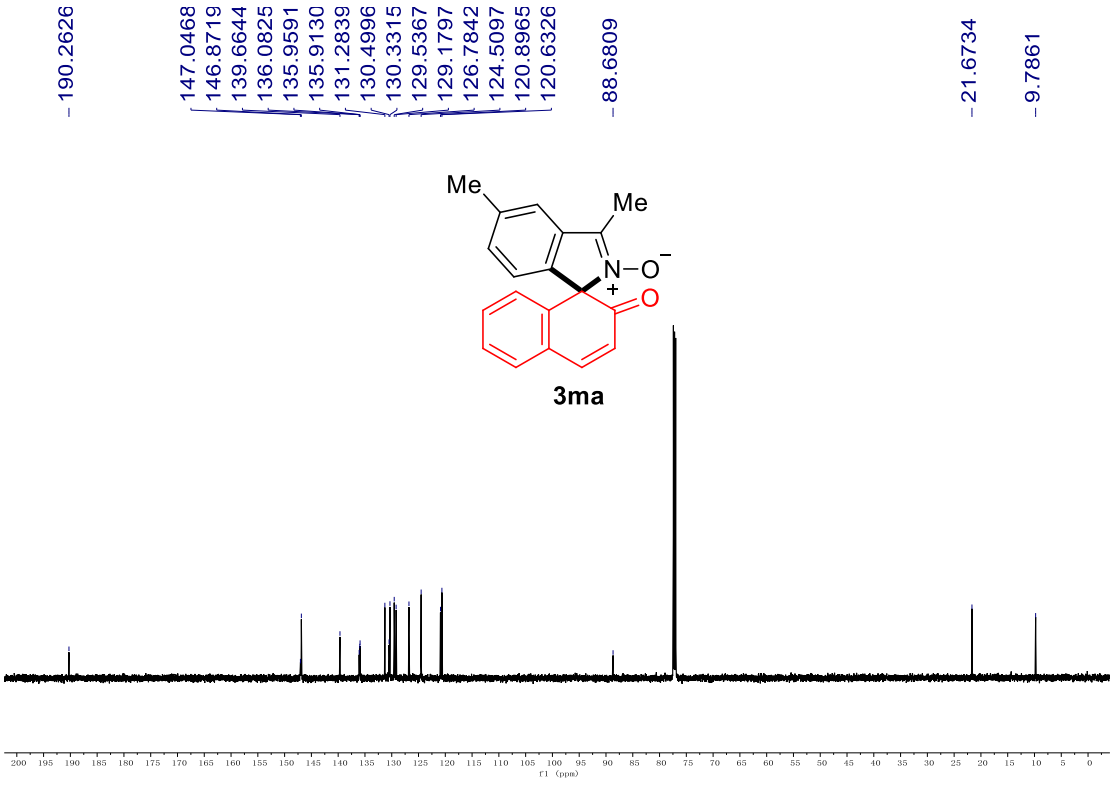
7.4261
7.4237
7.3851
7.3829
7.3726
7.3704
7.3600
7.3579
7.3172
7.3147
7.3044
7.3020
7.2919
7.2895
7.2087
7.0029
6.9901
6.8968
6.8948
6.8836
6.8797
6.8667
6.2910
6.2744
2.5226
2.3612
Me
Me
N−O
+
O
3ma
1.02
1.01
1.00
1.05
0.95
1.00
1.94
0.96
2.97
3.04
f1 (ppm)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139.6644
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135.9591
135.9130
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130.4996
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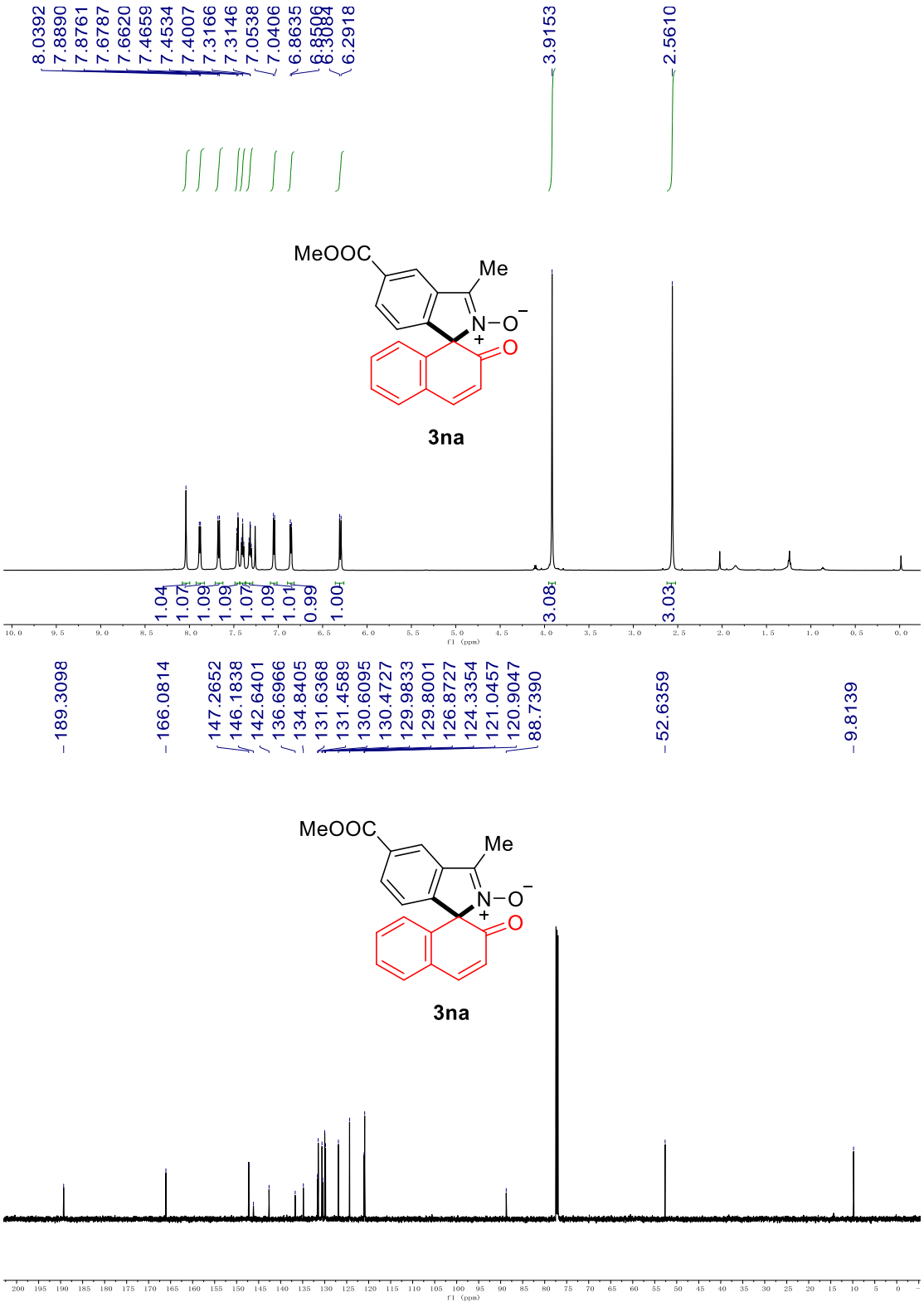
129.5367
129.1797
126.7842
124.5097
120.8965
120.6326
88.6809
21.6734
9.7861
Me
Me
N−O
+
O
3ma
f1 (ppm)

3na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7.8761
7.6787
7.6620
7.4659
7.4534
7.4007
7.3166
7.3146
7.0538
7.0406
6.8635
6.3084
6.2918
3.9153
2.5610
MeOOC
Me
N−O
O
3na
1.04
1.07
1.09
1.09
1.07
1.09
1.01
0.99
1.00
3.08
3.03
f1 (ppm)
189.3098
166.0814
147.2652
146.1838
142.6401
136.6966
134.8405
131.6368
131.4589
130.6095
130.4727
129.9833
129.8001
126.8727
124.3354
121.0457
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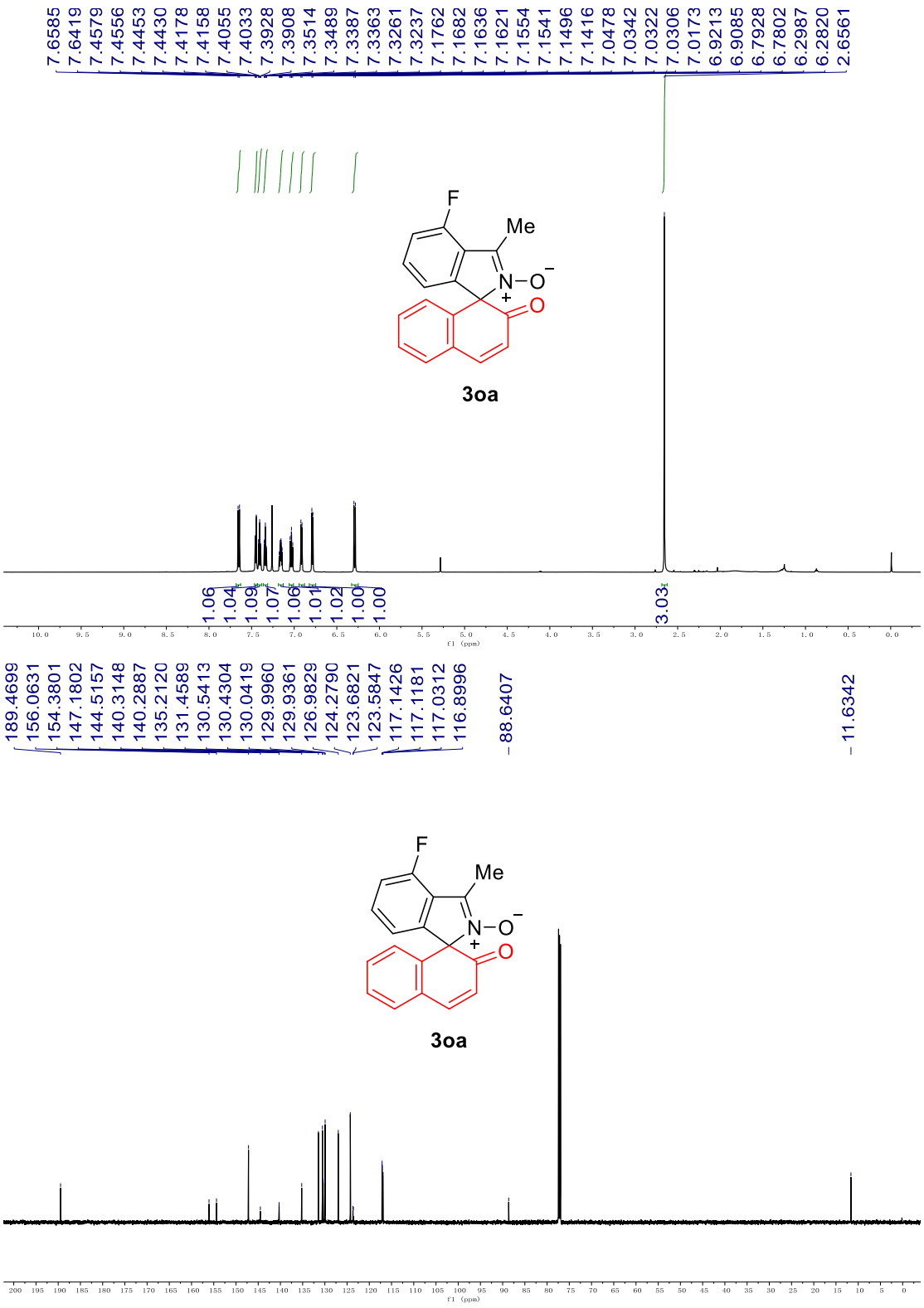
88.7390
52.6359
9.8139
MeOOC
Me
N−O
O
3na
f1 (ppm)

3oa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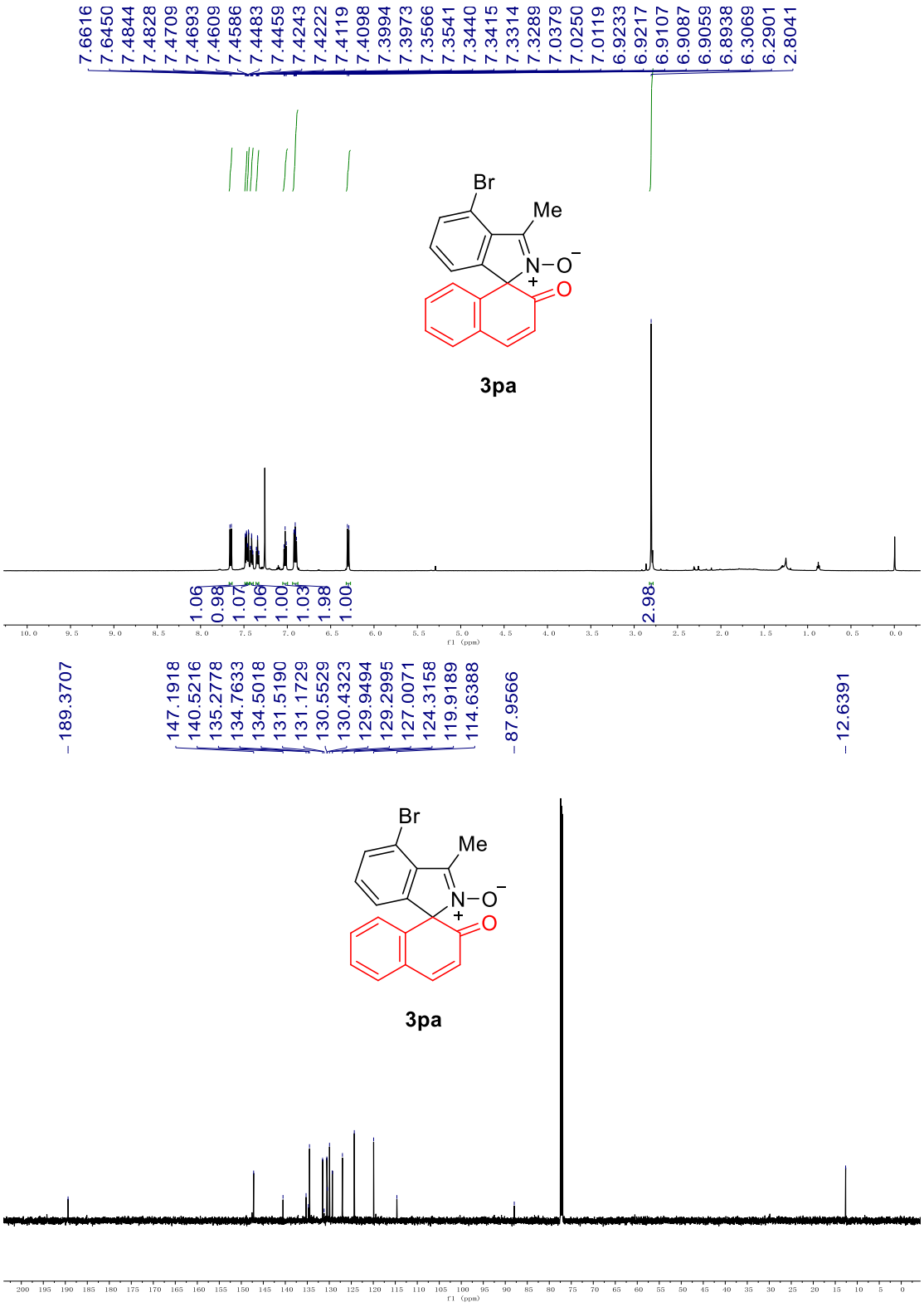
7.4430
7.4178
7.4158
7.4055
7.4033
7.3928
7.3908
7.3514
7.3489
7.3387
7.3363
7.3261
7.3237
7.1762
7.1682
7.1636
7.1621
7.1554
7.1541
7.1496
7.1416
7.0478
7.0342
7.0322
7.0306
7.0173
6.9213
6.9085
6.7928
6.7802
6.2987
6.2820
2.6561
F
Me
N−O
+
O
3oa
1.06
1.04
1.09
1.07
1.06
1.01
1.02
1.00
1.00
3.03
f1 (ppm)
189.4699
156.0631
154.3801
147.1802
144.5157
140.3148
140.2887
135.2120
131.4589
130.5413
130.4304
130.0419
129.9960
129.9361
126.9829
124.2790
123.6821
123.5847
117.1426
117.1181
117.0312
116.8996
88.6407
11.6342
F
Me
N−O
+
O
3oa
f1 (ppm)

3pa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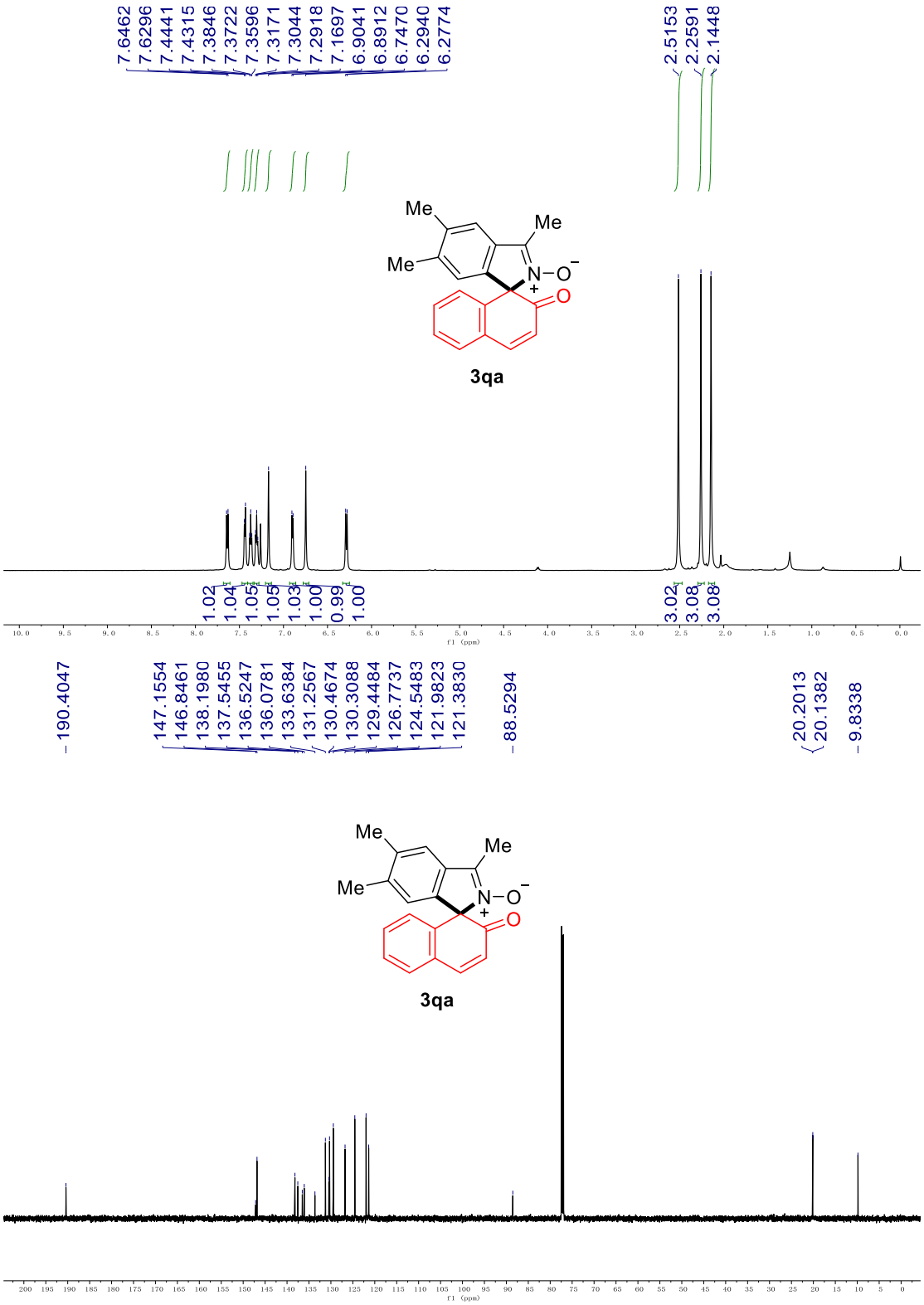
7.6450
7.4844
7.4828
7.4709
7.4693
7.4609
7.4586
7.4483
7.4459
7.4243
7.4222
7.4119
7.4098
7.3994
7.3973
7.3566
7.3541
7.3440
7.3415
7.3314
7.3289
7.0379
7.0250
7.0119
6.9233
6.9217
6.9107
6.9087
6.9059
6.8938
6.3069
6.2901
2.8041
Br
Me
N−O
+
O
3pa
1.06
0.98
1.07
1.06
1.00
1.03
1.98
1.00
2.98
f1 (ppm)
189.3707
147.1918
140.5216
135.2778
134.7633
134.5018
131.5190
131.1729
130.5529
130.4323
129.9494
129.2995
127.0071
124.3158
119.9189
114.6388
87.9566
12.6391
Br
Me
N−O
+
O
3pa
f1 (ppm)

3qa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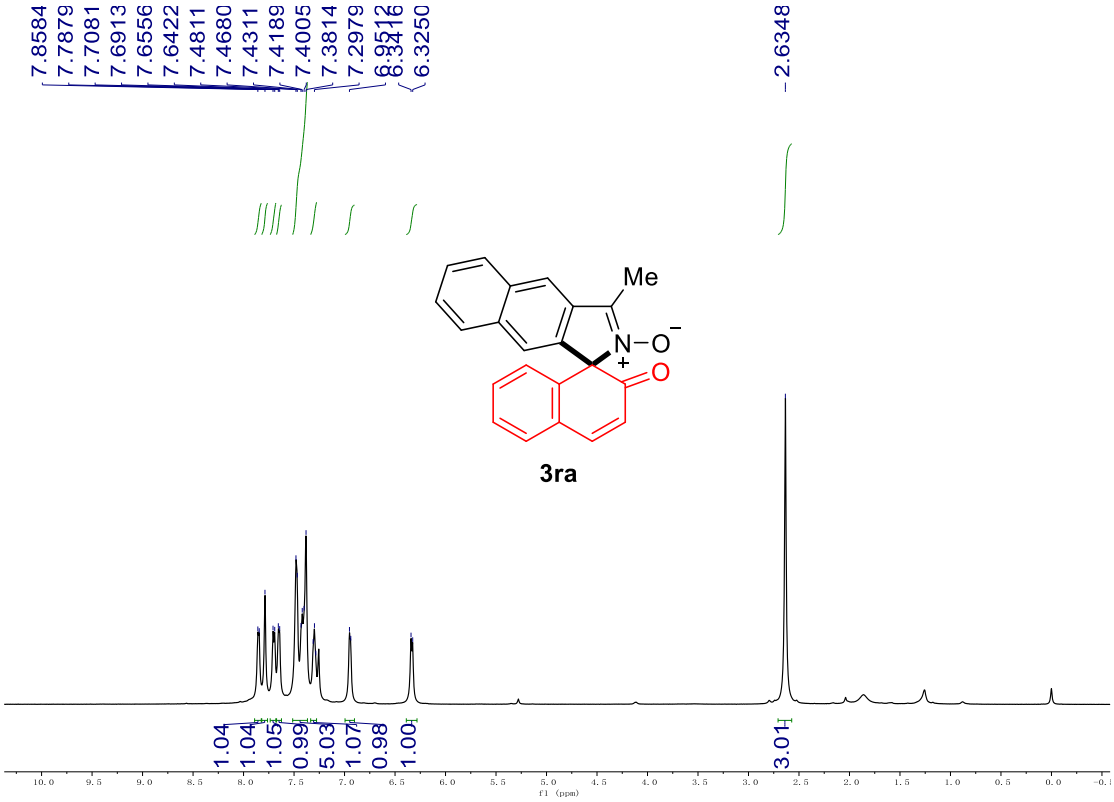
7.6296
7.4441
7.4315
7.3846
7.3722
7.3596
7.3171
7.3044
7.2918
7.1697
6.9041
6.8912
6.7470
6.2940
6.2774
2.5153
2.2591
2.1448
Me
Me
Me
N−O
+
O
3qa
1.02
1.04
1.05
1.05
1.03
1.00
0.99
1.00
3.02
3.08
3.08
f1 (ppm)
190.4047
147.1554
146.8461
138.1980
137.5455
136.5247
136.0781
133.6384
131.2567
130.4674
130.3088
129.4484
126.7737
124.5483
121.9823
121.3830
88.5294
20.2013
20.1382
9.8338
Me
Me
Me
N−O
+
O
3qa
f1 (ppm)

3ra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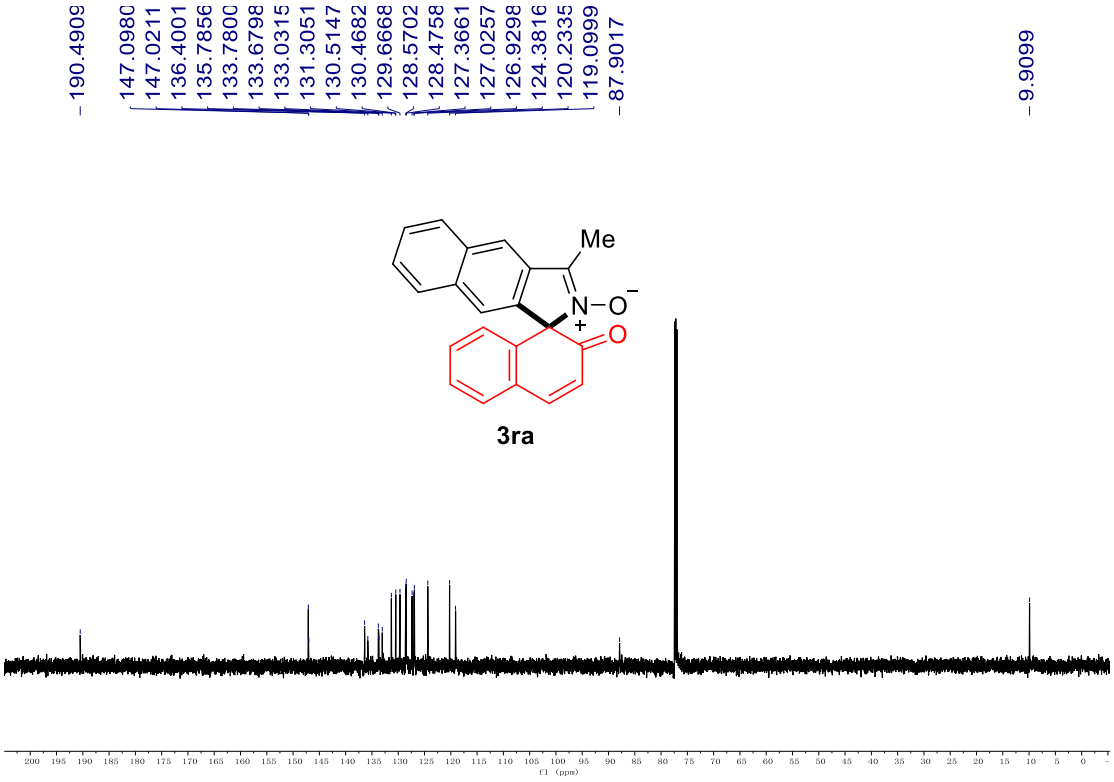
7.7879
7.7081
7.6913
7.6556
7.6422
7.4811
7.4680
7.4311
7.4189
7.4005
7.3814
7.2979
6.9512
6.3416
6.3250
2.6348
Me
N−O
O
3ra
1.04
1.04
1.05
0.99
5.03
1.07
0.98
1.00
3.01
f1 (ppm)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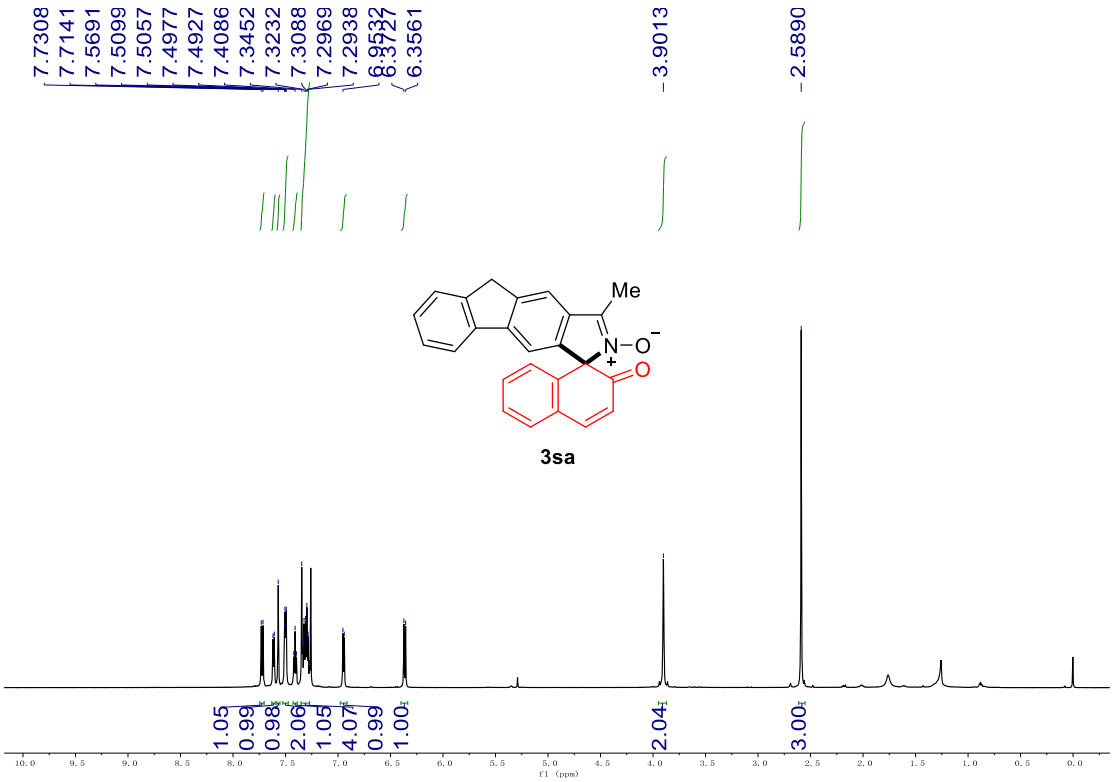
133.0315
131.3051
130.5147
130.4682
129.6668
128.5702
128.4758
127.3661
127.0257
126.9298
124.3816
120.2335
119.0999
87.9017
9.9099
Me
N−O
O
3ra
f1 (ppm)

3sa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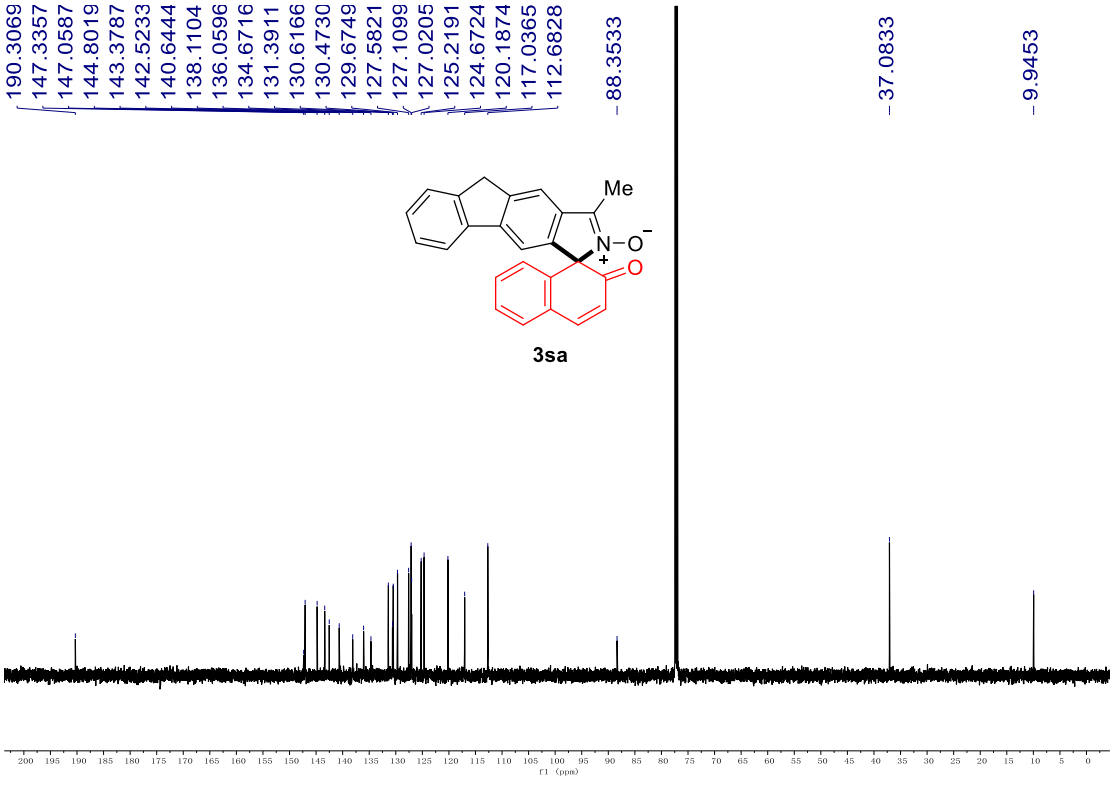
7.7141
7.5691
7.5099
7.5057
7.4977
7.4927
7.4086
7.3452
7.3232
7.3088
7.2969
7.2938
6.9532
6.3561
3.9013
2.5890
Me
N−O
O
3sa
1.05
0.99
0.98
2.06
1.05
4.07
0.99
1.00
2.04
3.00
f1 (ppm)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140.6444
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136.0596
134.6716
131.3911
130.6166
130.4730
129.6749
127.5821
127.1099
127.0205
125.2191
124.6724
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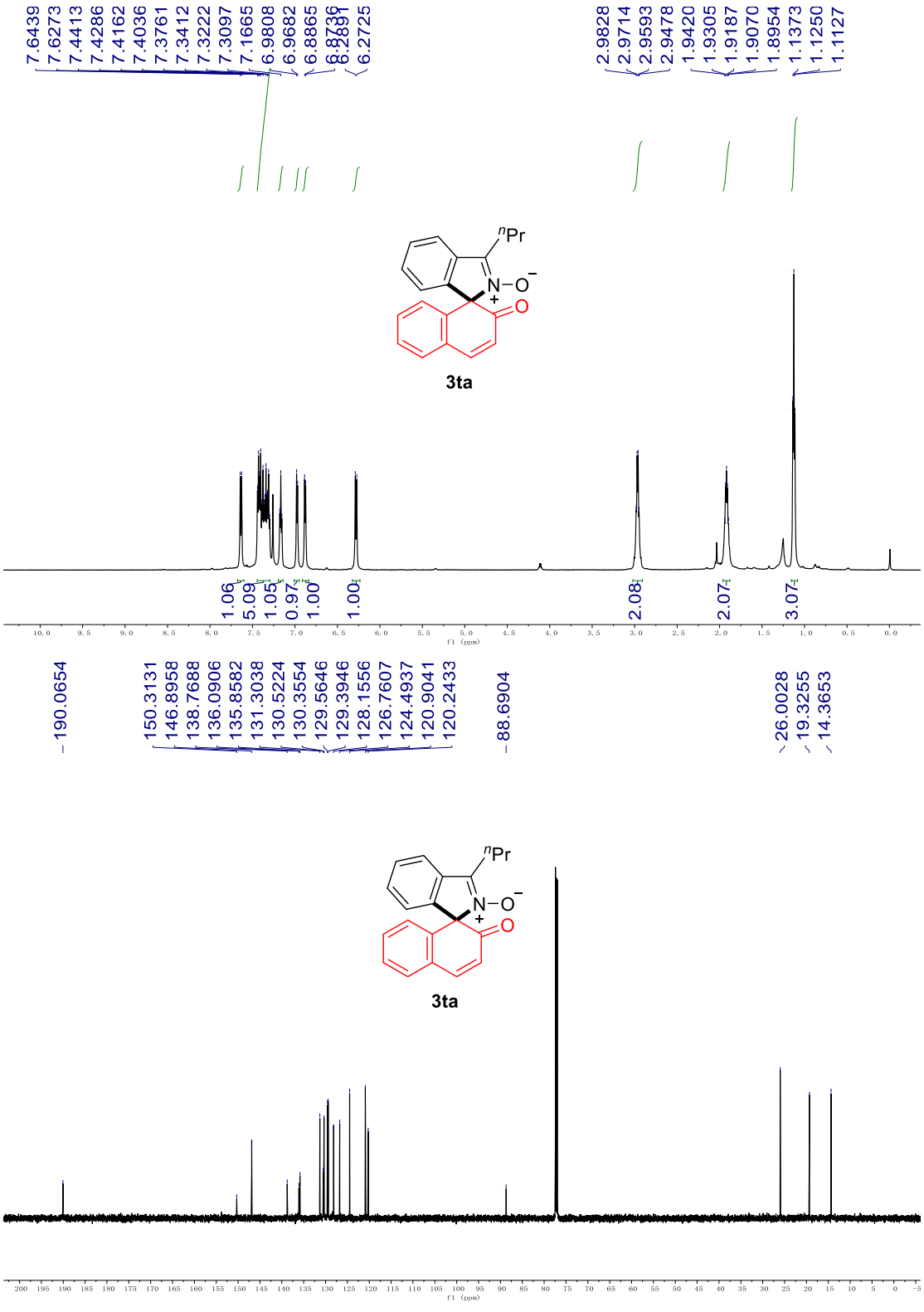
112.6828
88.3533
37.0833
9.9453
Me
N−O
O
3sa
f1 (ppm)

3ta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7.3222
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6.9808
6.9682
6.8865
6.8736
6.8591
6.2725
2.9828
2.9714
2.9593
2.9478
1.9420
1.9305
1.9187
1.9070
1.8954
1.1373
1.1250
1.1127
nPr
N−O
O
3ta
1.06
5.09
1.05
0.97
1.00
1.00
2.08
2.07
3.07
f1 (ppm)
190.0654
150.3131
146.8958
138.7688
136.0906
135.8582
131.3038
130.5224
130.3554
129.5646
129.3946
128.1556
126.7607
124.4937
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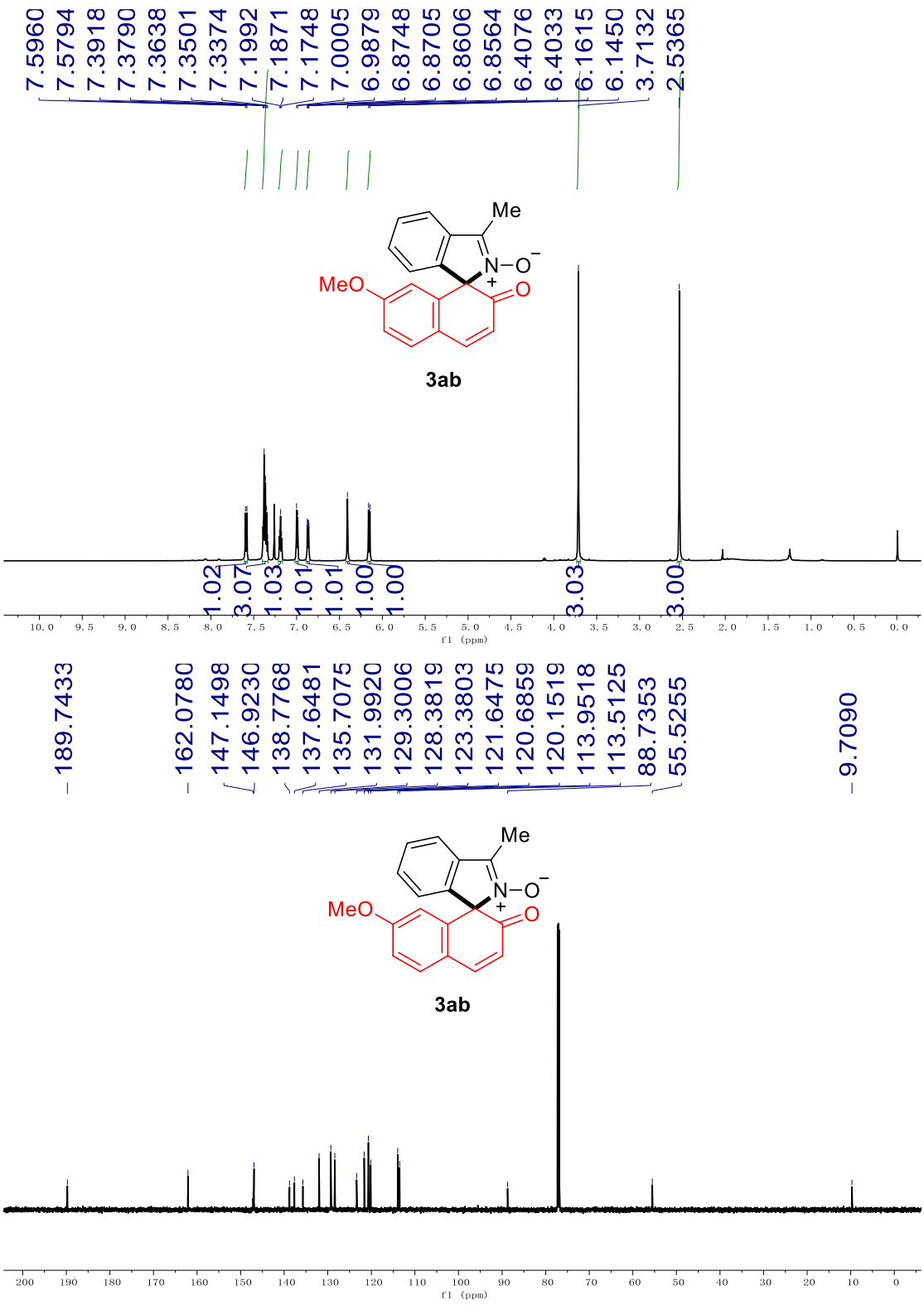
26.0028
19.3255
14.3653
nPr
N−O
O
3ta
f1 (ppm)

3ab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7.0005
6.9879
6.8748
6.8705
6.8606
6.8564
6.4076
6.4033
6.1615
6.1450
3.7132
2.5365
Me
N–O
MeO
O
3ab
1.02
3.07
1.03
1.01
1.01
1.00
1.00
3.03
3.00
f1 (ppm)
189.7433
162.0780
147.1498
146.9230
138.7768
137.6481
135.7075
131.9920
129.3006
128.3819
123.3803
121.6475
120.6859
120.1519
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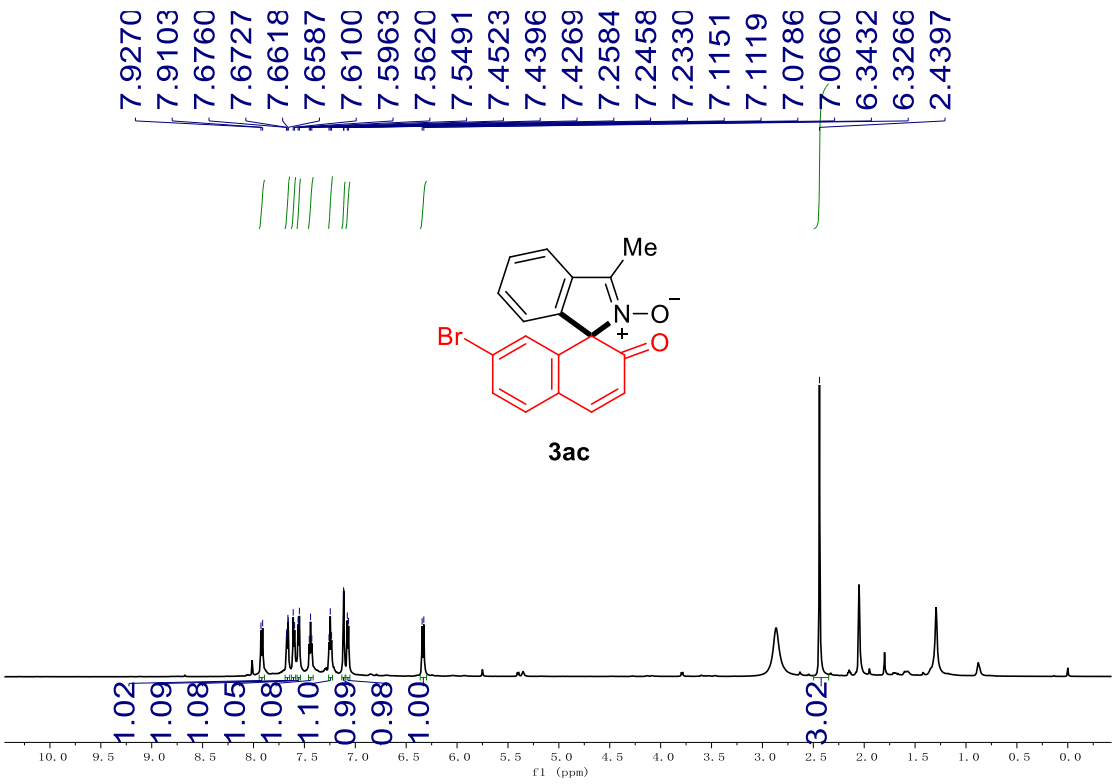
113.5125
88.7353
55.5255
9.7090
Me
N–O
MeO
O
3ab
f1 (ppm)

3ac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7.6727
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7.6587
7.6100
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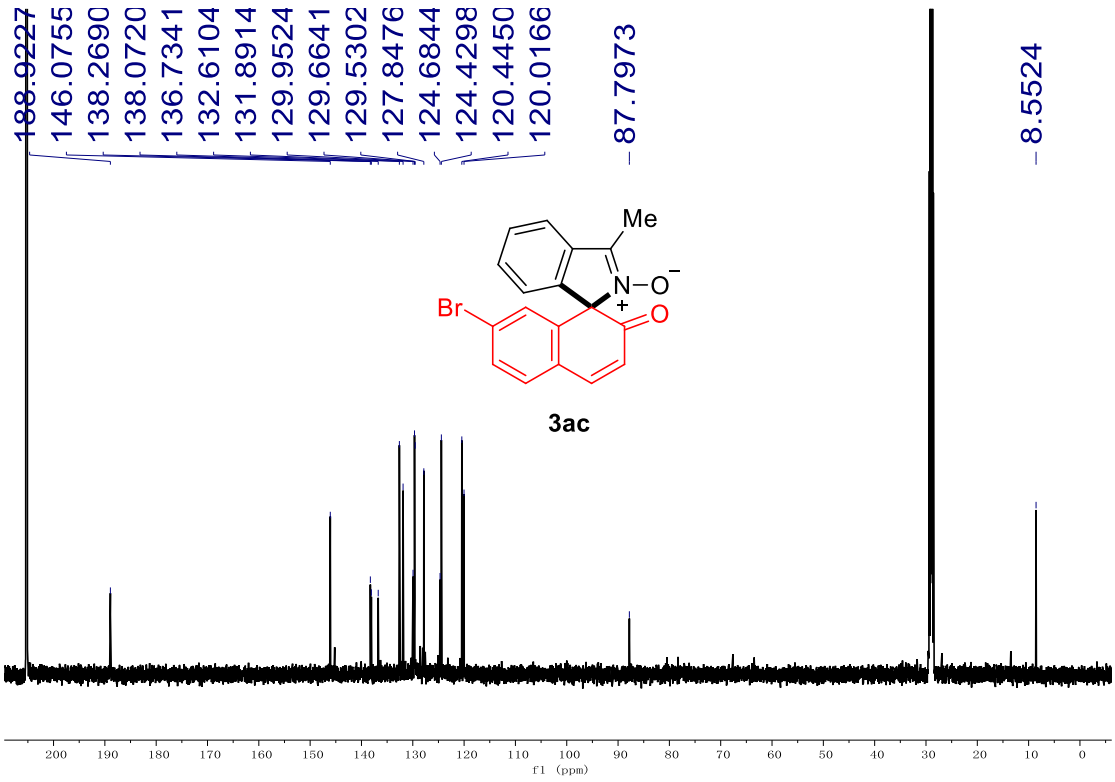
7.5620
7.5491
7.4523
7.4396
7.4269
7.2584
7.2458
7.2330
7.1151
7.1119
7.0786
7.0660
6.3432
6.3266
2.4397
Me
N–O
Br
O
3ac
1.02
1.09
1.08
1.05
1.08
1.10
0.99
0.98
1.00
3.02
f1 (ppm)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127.8476
124.6844
124.4298
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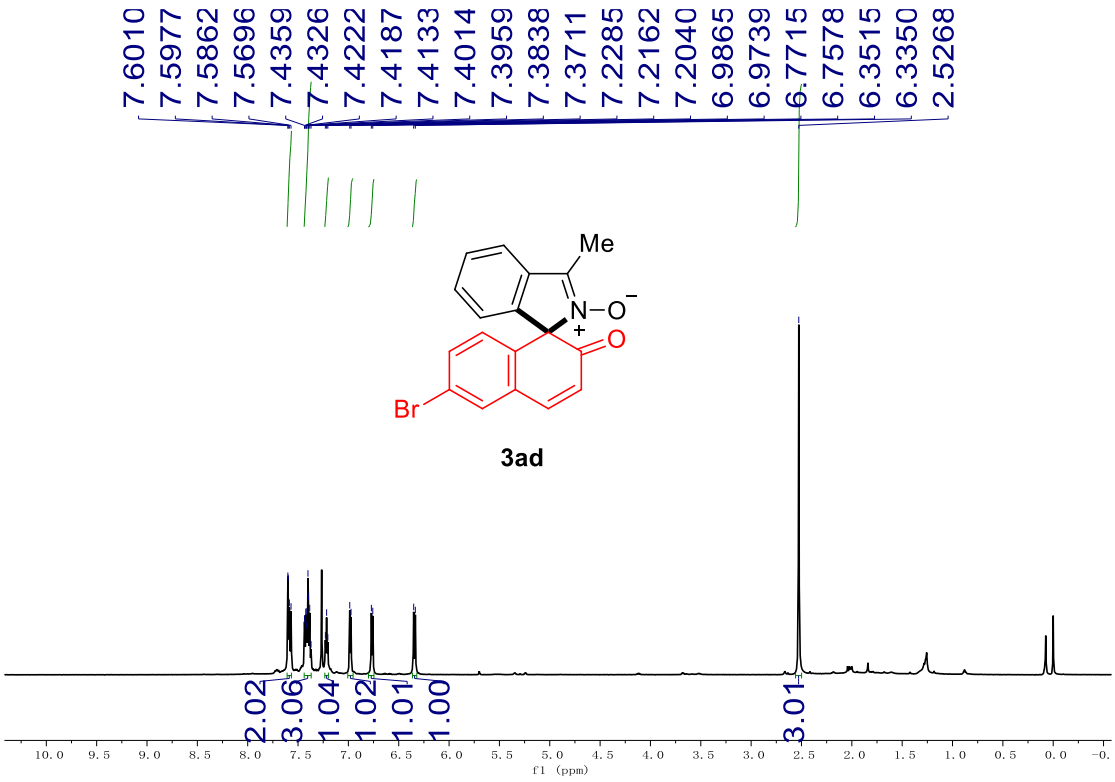
120.0166
87.7973
8.5524
Me
N–O
Br
O
3ac
f1 (ppm)

3ad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7.5862
7.5696
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7.4222
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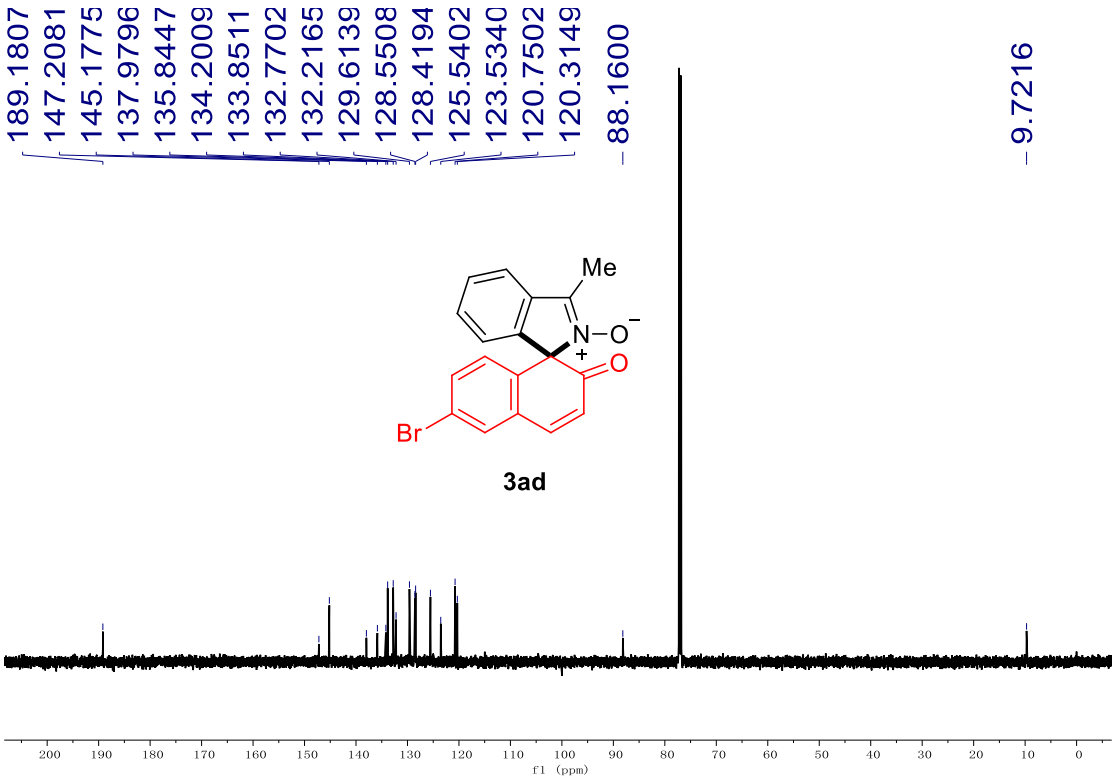
7.4133
7.4014
7.3959
7.3838
7.3711
7.2285
7.2162
7.2040
6.9865
6.9739
6.7715
6.7578
6.3515
6.3350
2.5268
Me
N–O
O
Br
3ad
2.02
3.06
1.04
1.02
1.01
1.00
3.01
f1 (ppm)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137.9796
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133.8511
132.7702
132.2165
129.6139
128.5508
128.4194
125.5402
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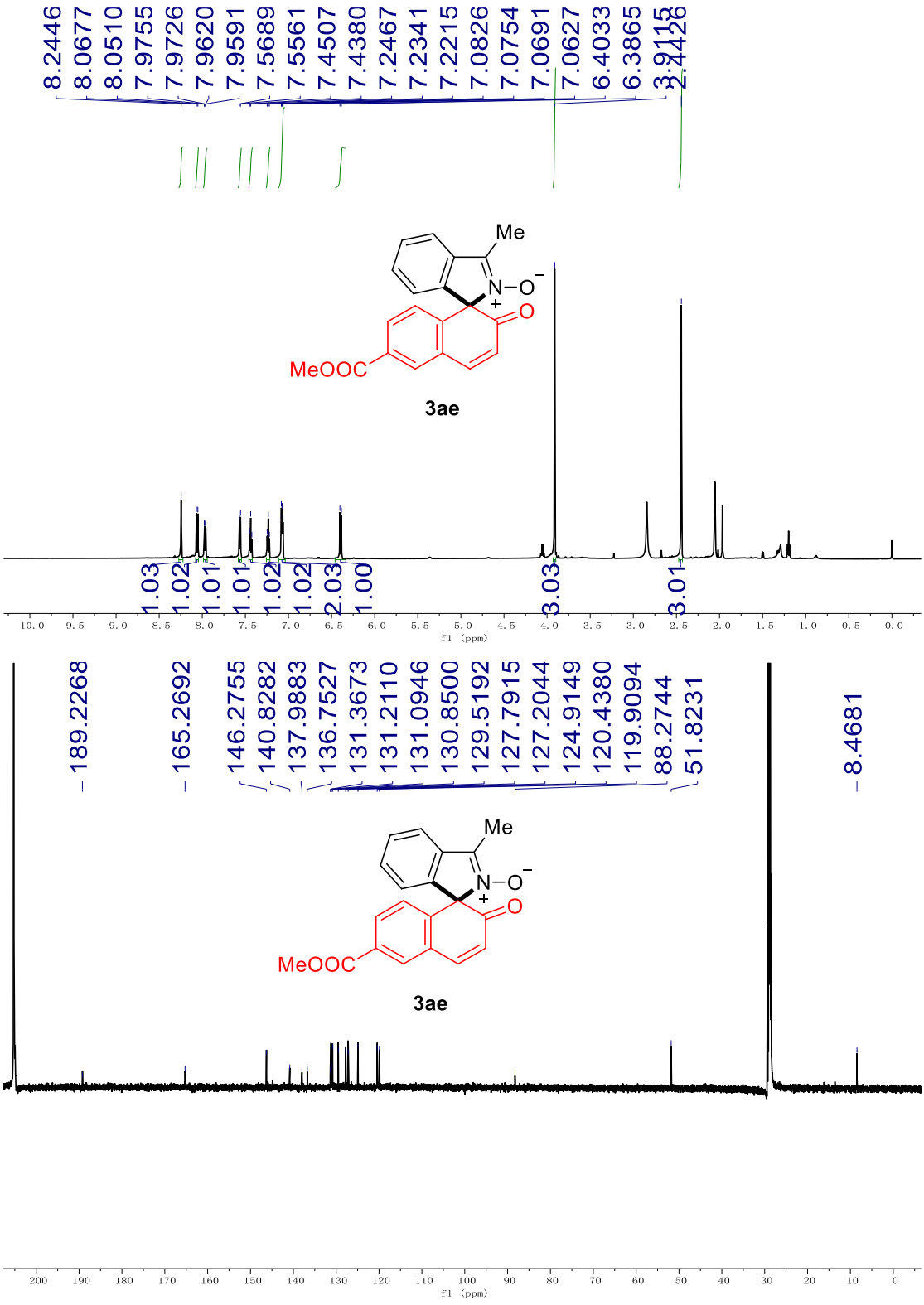
120.3149
88.1600
9.7216
Me
N–O
O
Br
3ad
f1 (ppm)

3ae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8.0510
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7.9591
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7.5561
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7.4380
7.2467
7.2341
7.2215
7.0826
7.0754
7.0691
7.0627
6.4033
6.3865
3.9115
2.4426
Me
N–O
O
MeOOC
3ae
1.03
1.02
1.01
1.01
1.02
1.02
2.03
1.00
3.03
3.01
f1 (ppm)
189.2268
165.2692
146.2755
140.8282
137.9883
136.7527
131.3673
131.2110
131.0946
130.8500
129.5192
127.7915
127.2044
124.9149
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119.9094
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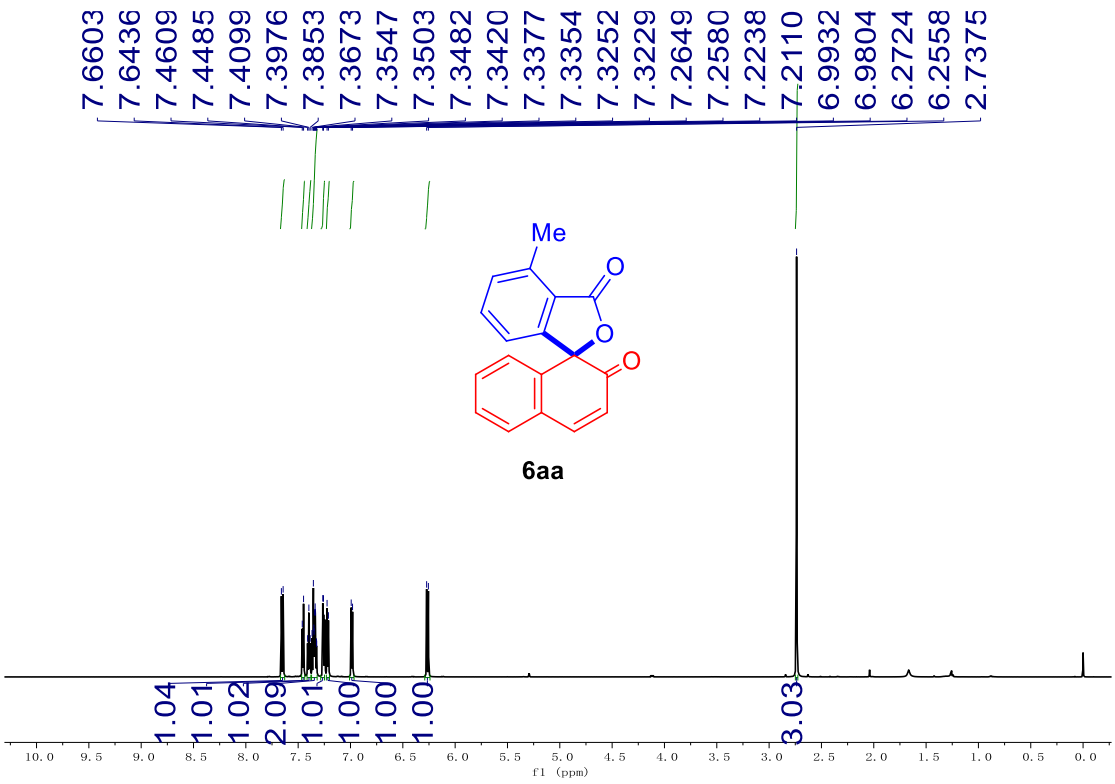
8.4681
Me
N–O
O
MeOOC
3ae
f1 (ppm)

6aa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7.4609
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7.4099
7.3976
7.3853
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7.3547
7.3503
7.3482
7.3420
7.3377
7.3354
7.3252
7.3229
7.2649
7.2580
7.2238
7.2110
6.9932
6.9804
6.2724
6.2558
2.7375
Me
O
O
O
6aa
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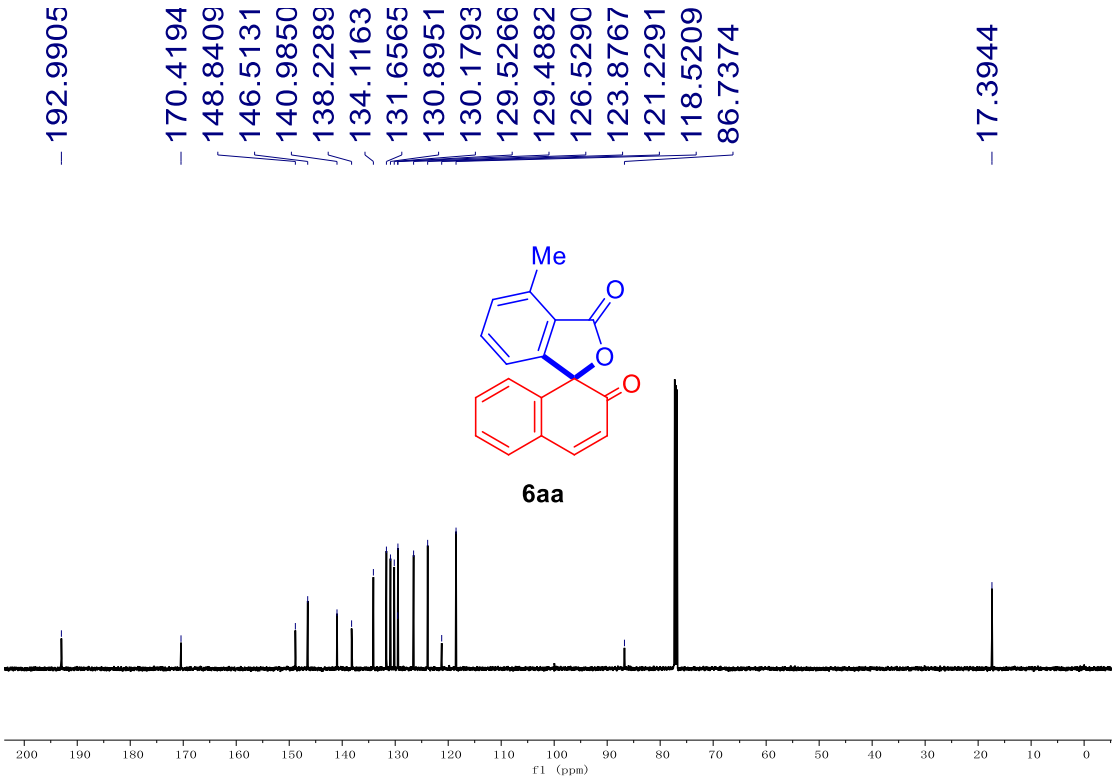
1.01
1.02
2.09
1.01
1.00
1.00
1.00
3.03
f1 (ppm)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140.9850
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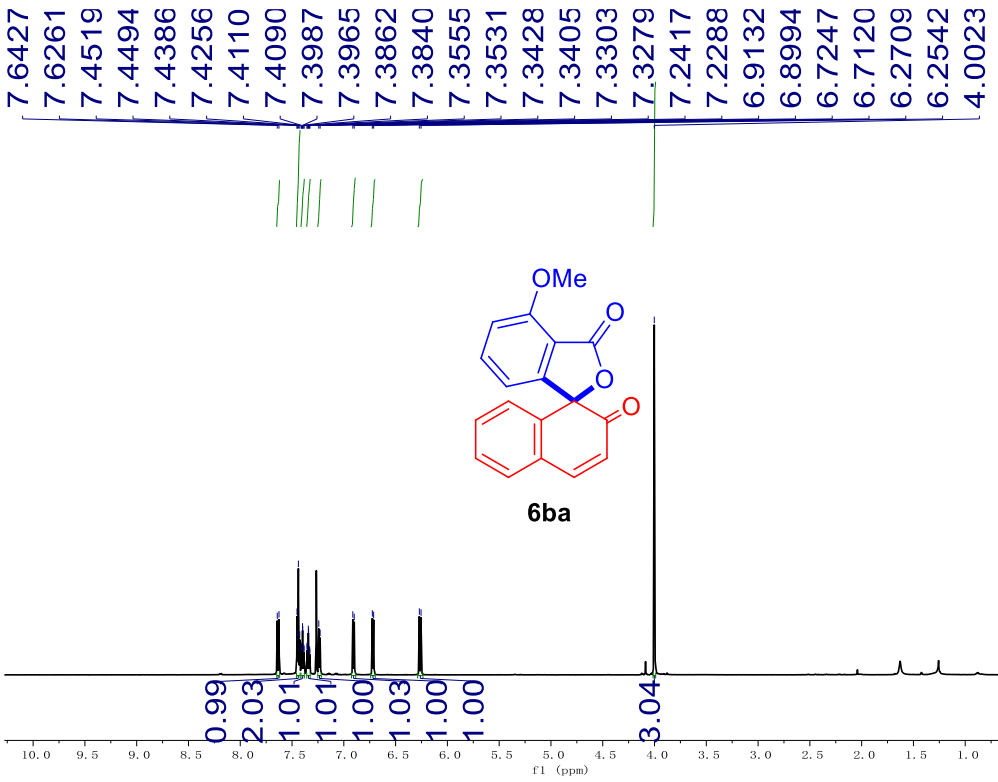
130.8951
130.1793
129.5266
129.4882
126.5290
123.8767
121.2291
118.5209
86.7374
17.3944
Me
O
O
O
6aa
f1 (ppm)

6ba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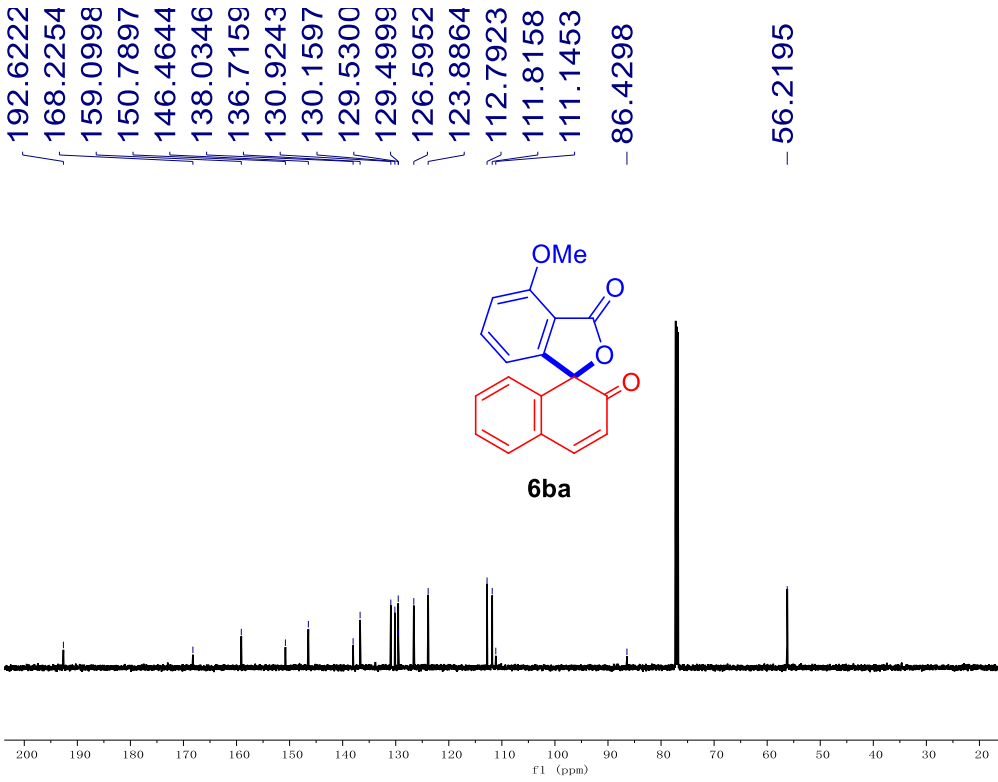
7.6261
7.4519
7.4494
7.4386
7.4256
7.4110
7.4090
7.3987
7.3965
7.3862
7.3840
7.3555
7.3531
7.3428
7.3405
7.3303
7.3279
7.2417
7.2288
6.9132
6.8994
6.7247
6.7120
6.2709
6.2542
4.0023
OMe
O
O
O
6ba
0.99
2.03
1.01
1.01
1.00
1.03
1.00
1.00
3.04
f1 (ppm)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159.0998
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146.4644
138.0346
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130.9243
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129.4999
126.5952
123.8864
112.7923
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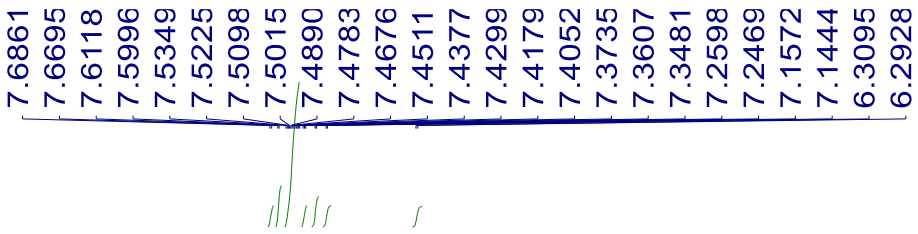
111.1453
86.4298
56.2195
OMe
O
O
O
6ba
f1 (ppm)

6ca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7.6118
7.5996
7.5349
7.5225
7.5098
7.5015
7.4890
7.4783
7.4676
7.4511
7.4377
7.4299
7.4179
7.4052
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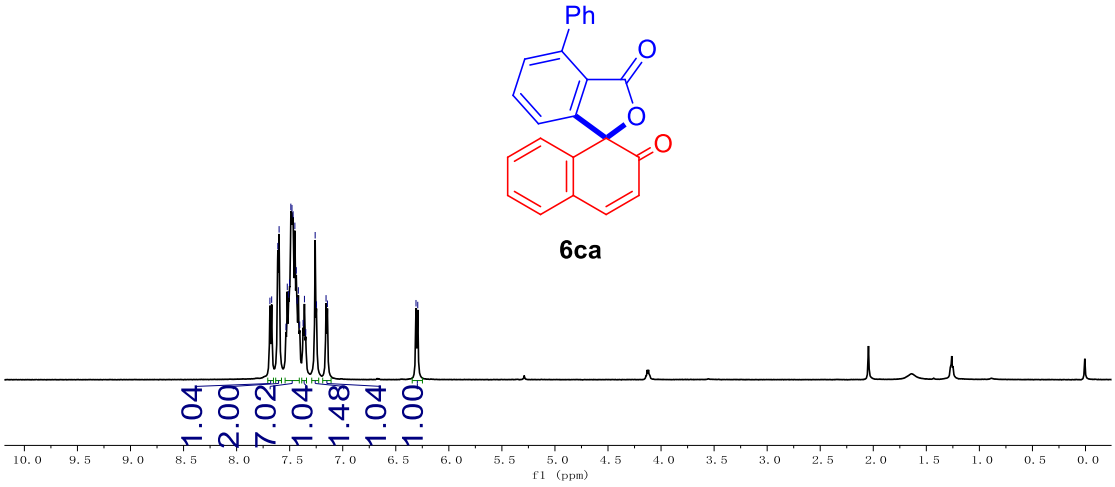
7.3607
7.3481
7.2598
7.2469
7.1572
7.1444
6.3095
6.2928


Ph
O
O
O
6ca
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2.00
7.02
1.04
1.48
1.04
1.00
f1 (ppm)


193.0689
169.1581
149.7115
146.7494
143.9469
138.2524
136.0648
134.3456
131.8302
131.1035
130.3780
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129.7158
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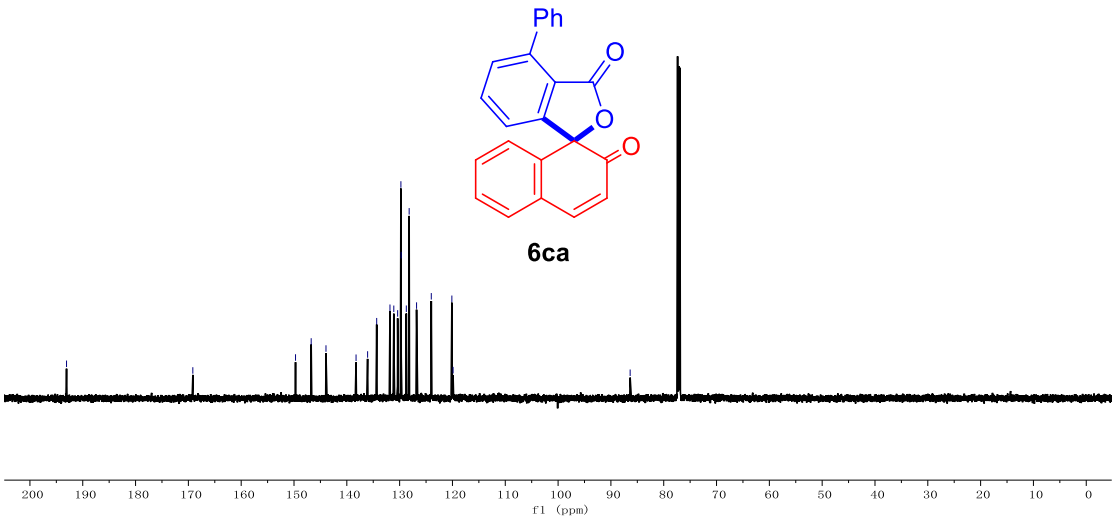
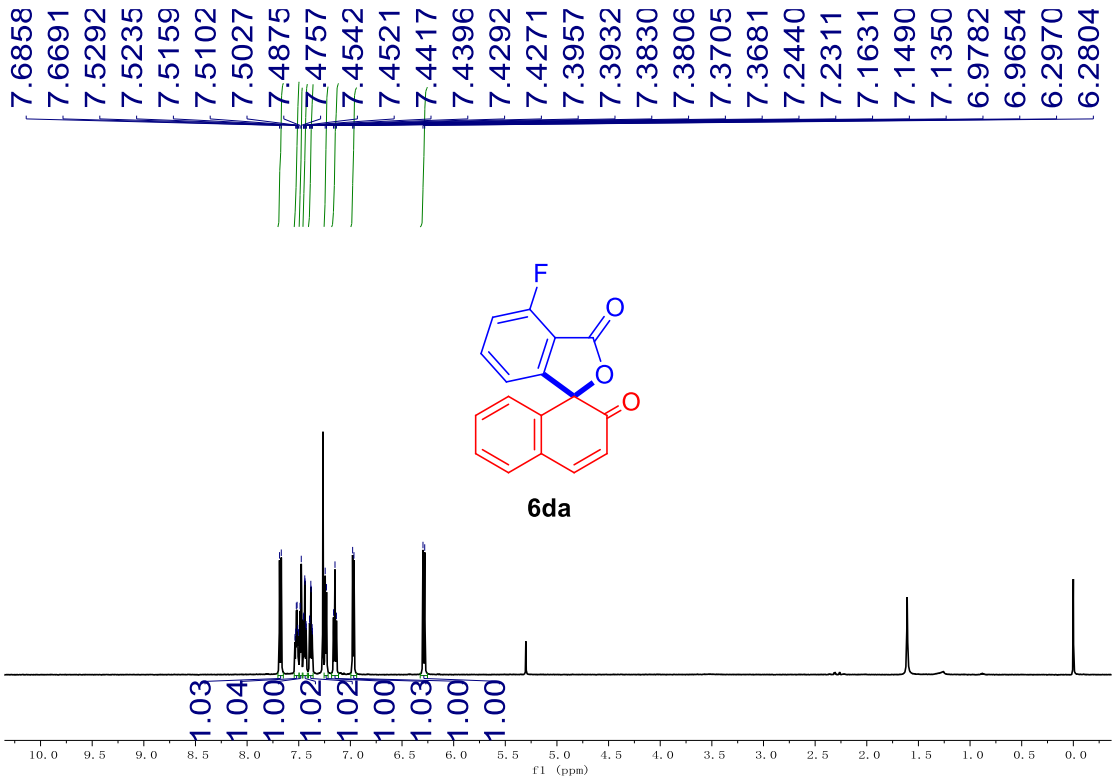
126.7947
124.0090
120.1123
119.8821
86.3566


Ph
O
O
O
6ca
f1 (ppm)

6da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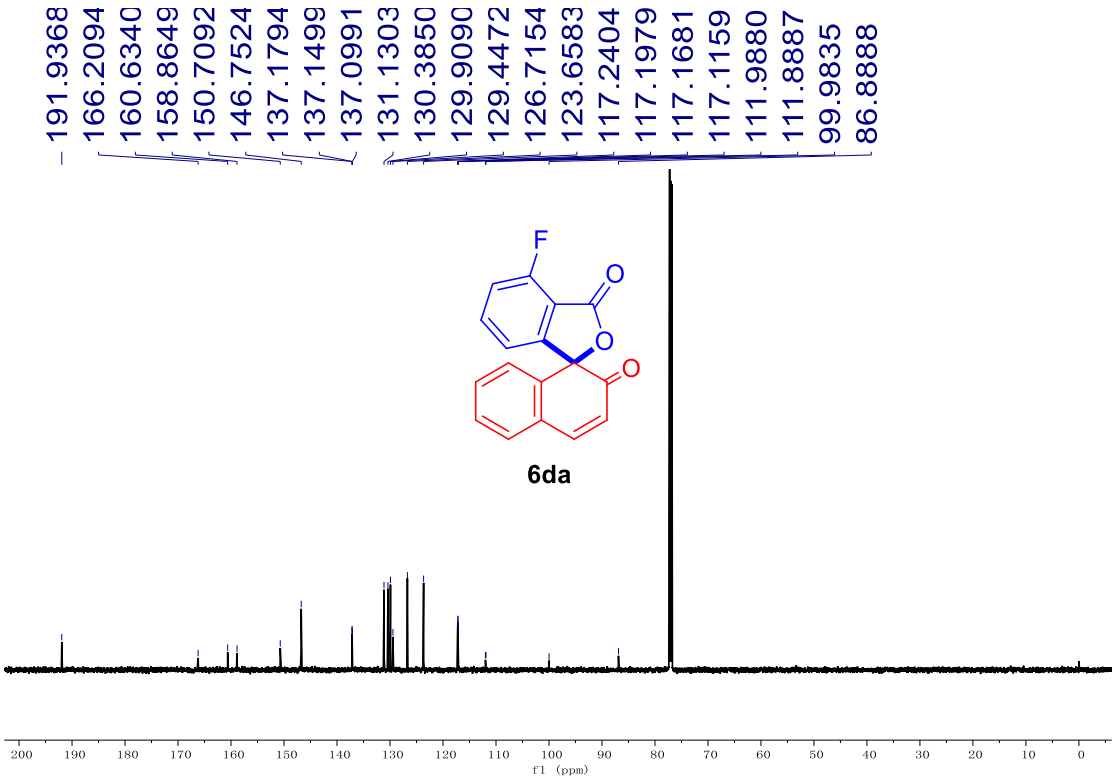
7.5159
7.5102
7.5027
7.4875
7.4757
7.4542
7.4521
7.4417
7.4396
7.4292
7.4271
7.3957
7.3932
7.3830
7.3806
7.3705
7.3681
7.2440
7.2311
7.1631
7.1490
7.1350
6.9782
6.9654
6.2970
6.2804
F
O
O
O
6da
1.03
1.04
1.00
1.02
1.02
1.00
1.03
1.00
1.00
f1 (ppm)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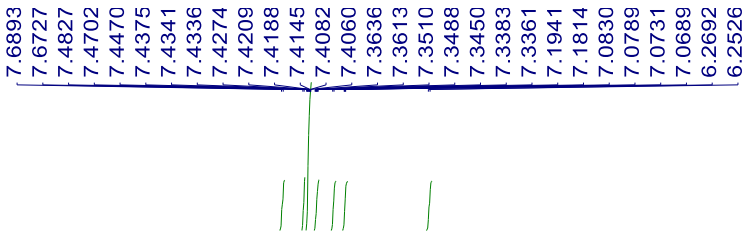
160.6340
158.8649
150.7092
146.7524
137.1794
137.1499
137.0991
131.1303
130.3850
129.9090
129.4472
126.7154
123.6583
117.2404
117.1979
117.1681
117.1159
111.9880
111.8887
99.9835
86.8888
F
O
O
O
6da
f1 (ppm)

6ea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7.4827
7.4702
7.4470
7.4375
7.4341
7.4336
7.4274
7.4209
7.4188
7.4145
7.4082
7.4060
7.3636
7.3613
7.3510
7.3488
7.3450
7.3383
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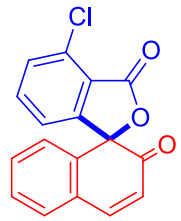
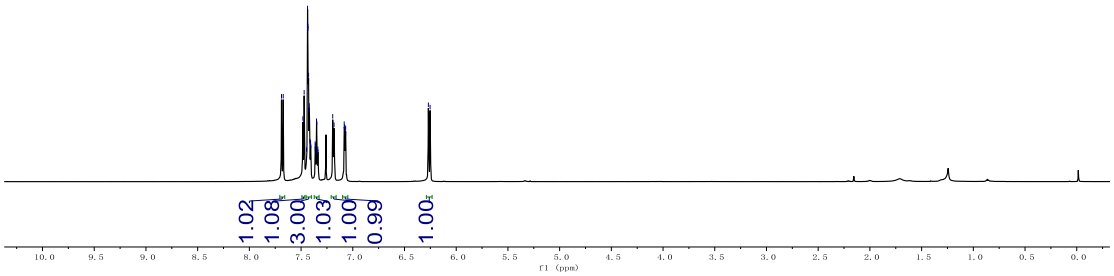
7.1941
7.1814
7.0830
7.0789
7.0731
7.0689
6.2692
6.2526


Cl
O
O
O

6ea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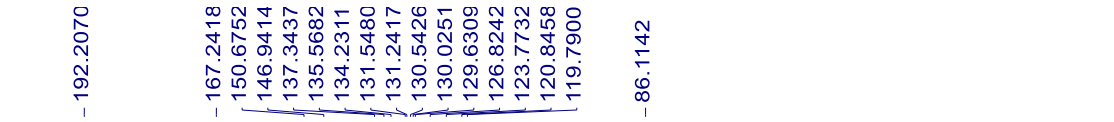
1.08
3.00
1.03
1.00
0.99
1.00
f1 (ppm)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137.3437
135.5682
134.2311
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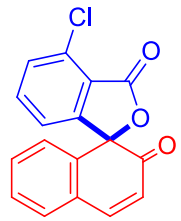
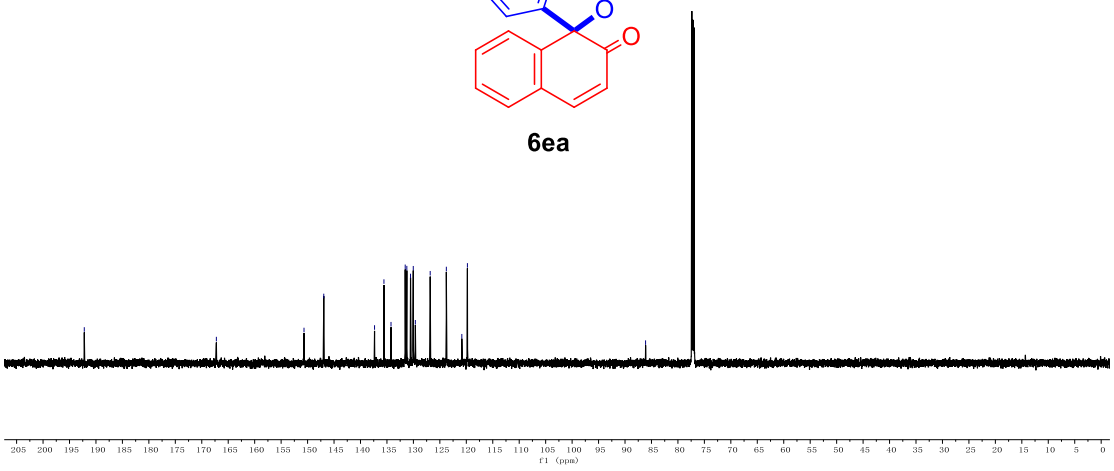
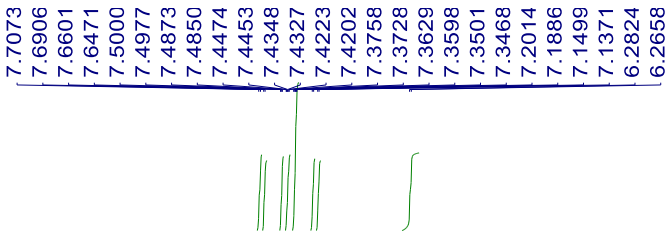
126.8242
123.7732
120.8458
119.7900
86.1142


Cl
O
O
O

6ea


f1 (ppm)

6fa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7.6601
7.6471
7.5000
7.4977
7.4873
7.4850
7.4474
7.4453
7.4348
7.4327
7.4223
7.4202
7.3758
7.3728
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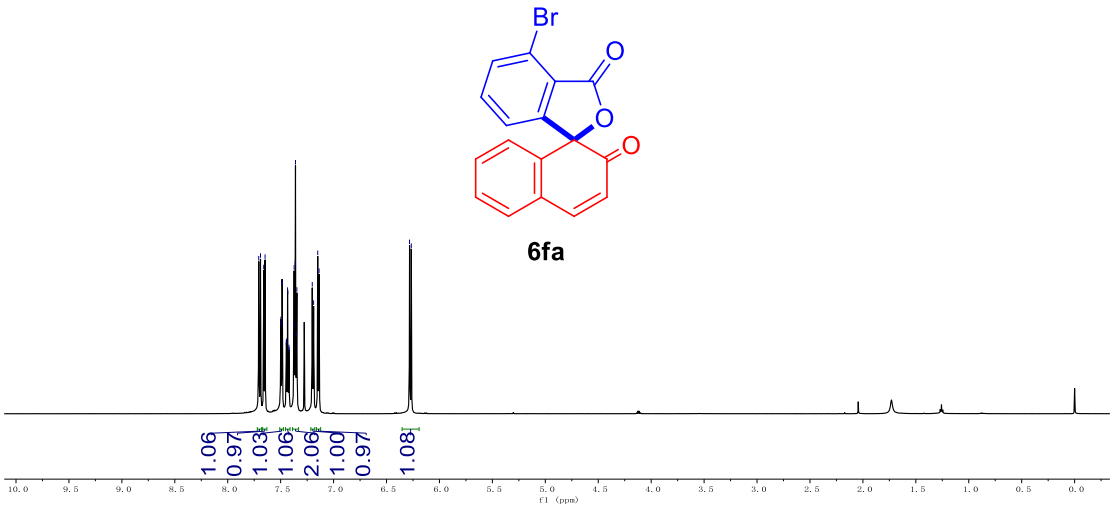
7.3501
7.3468
7.2014
7.1886
7.1499
7.1371
6.2824
6.2658


Br
O
O
O
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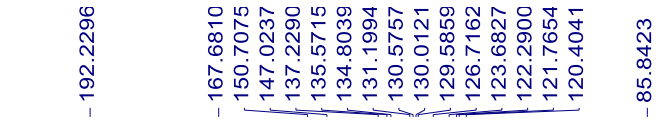
0.97
1.03
1.06
2.06
1.00
0.97
1.08
f1 (ppm)

6fa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135.5715
134.8039
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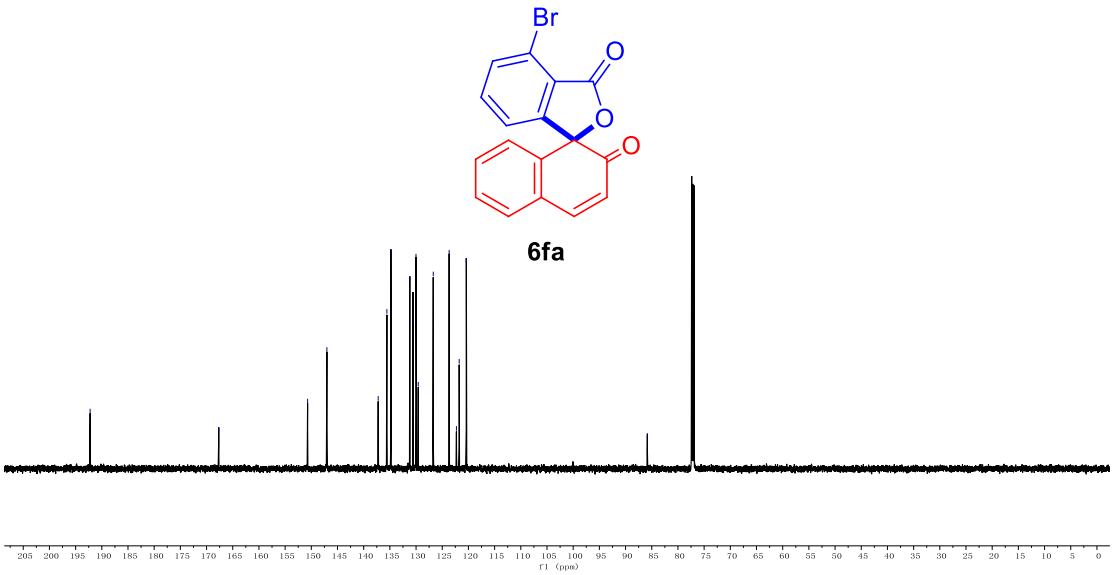
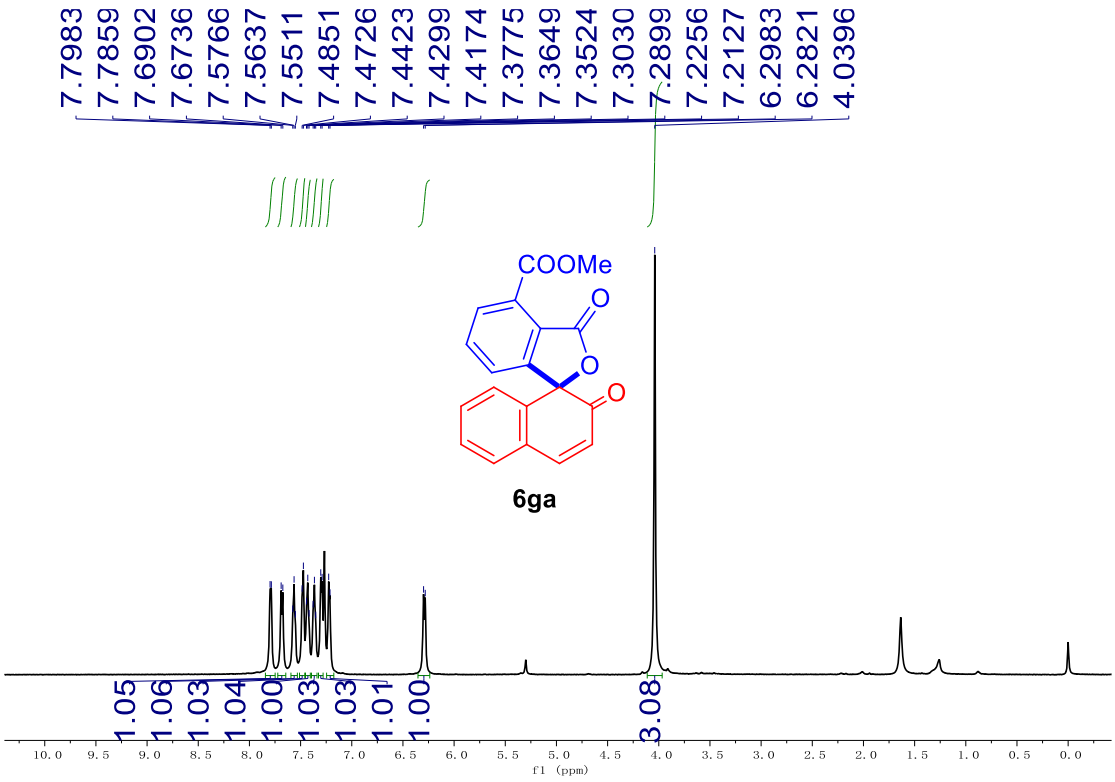
130.0121
129.5859
126.7162
123.6827
122.2900
121.7654
120.4041
85.8423


Br
O
O
O
f1 (ppm)

6fa

6ga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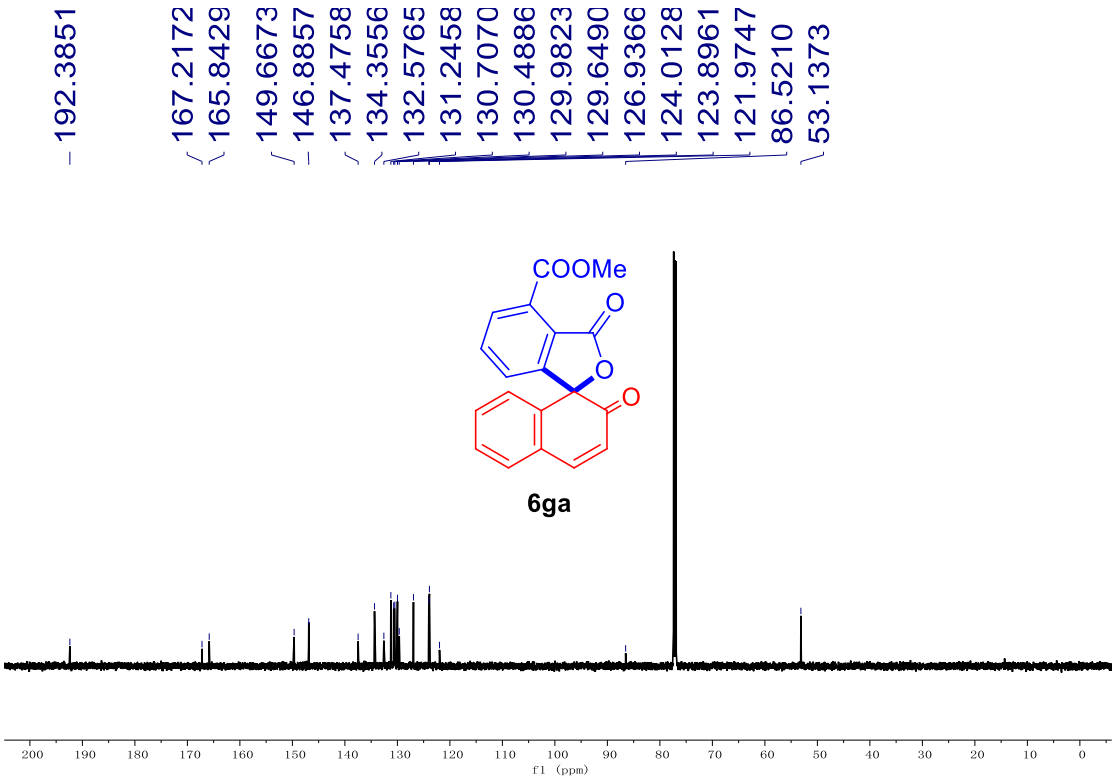
7.6902
7.6736
7.5766
7.5637
7.5511
7.4851
7.4726
7.4423
7.4299
7.4174
7.3775
7.3649
7.3524
7.3030
7.2899
7.2256
7.2127
6.2983
6.2821
4.0396
COOMe
O
O
O
6ga
1.05
1.06
1.03
1.04
1.00
1.03
1.03
1.01
1.00
3.08
f1 (ppm)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134.3556
132.5765
131.2458
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129.9823
129.6490
126.9366
124.0128
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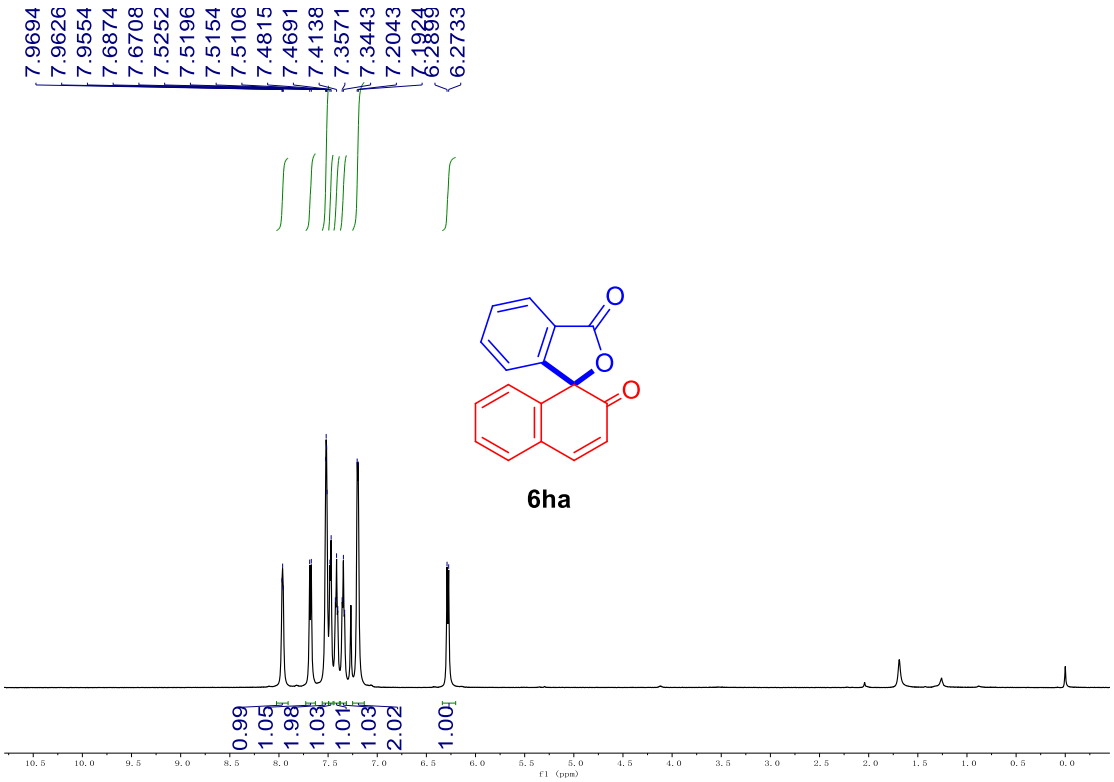
121.9747
86.5210
53.1373
COOMe
O
O
O
6ga
f1 (ppm)

6ha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7.5196
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7.5106
7.4815
7.4691
7.4138
7.3571
7.3443
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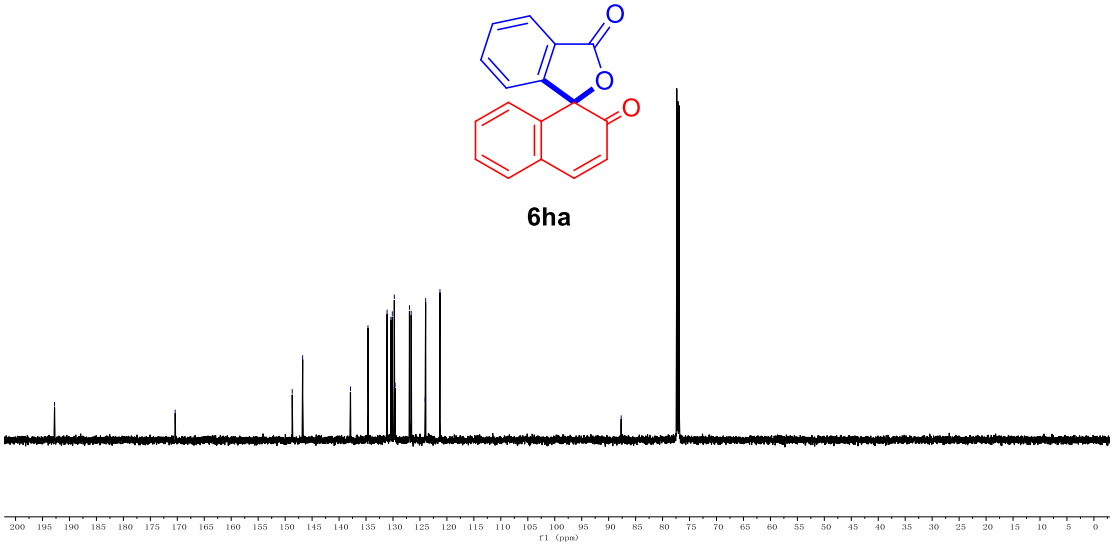
6.2733
6ha
0.99
1.05
1.98
1.03
1.01
1.03
2.02
1.00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129.6075
126.9389
126.6190
124.0333
123.9446
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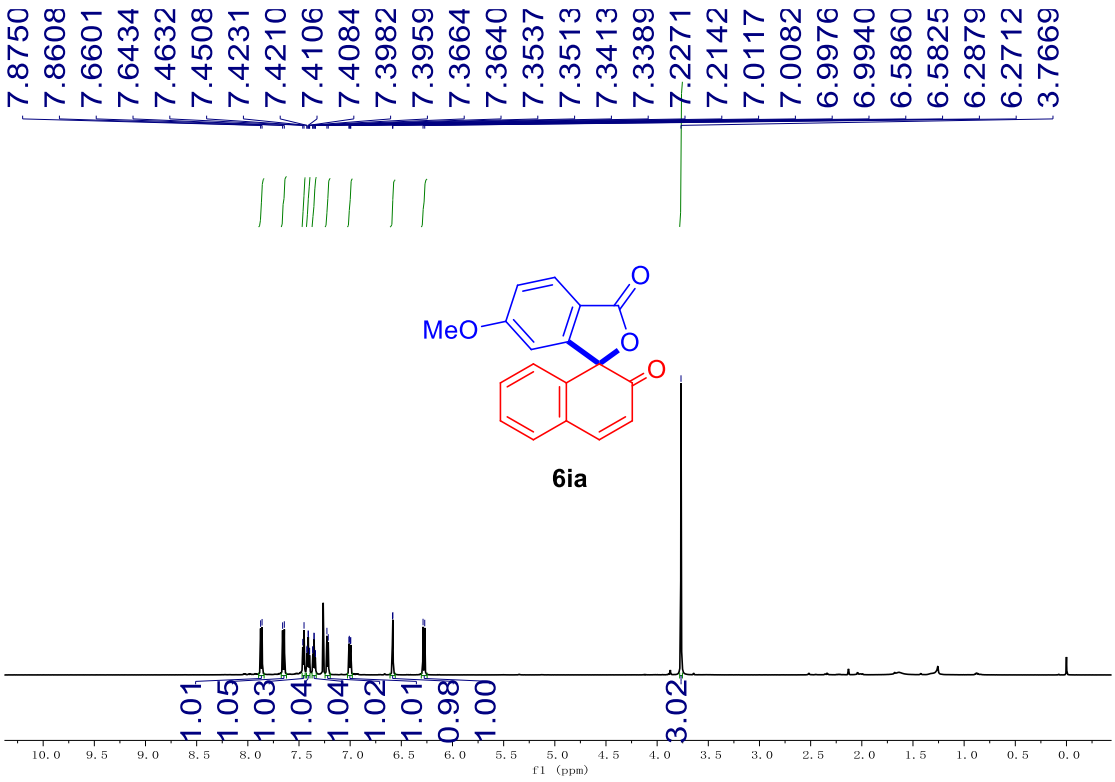
87.6779
6ha

6ia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7.4632
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7.4106
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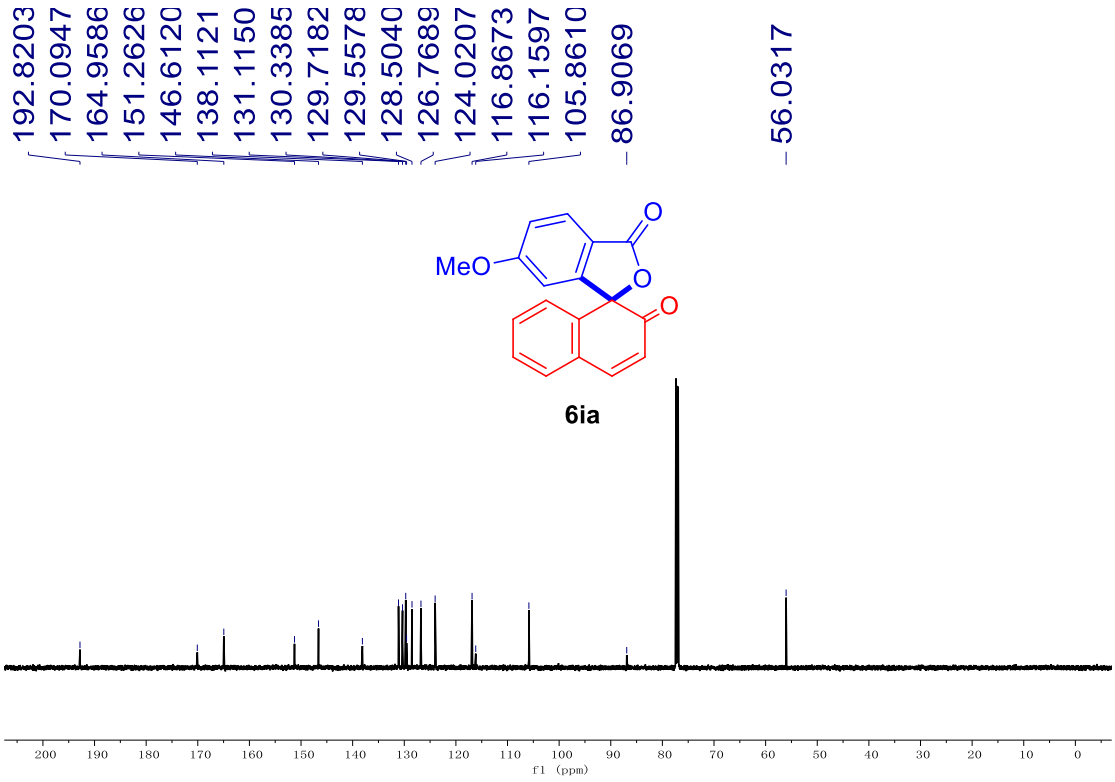
7.3982
7.3959
7.3664
7.3640
7.3537
7.3513
7.3413
7.3389
7.2271
7.2142
7.0117
7.0082
6.9976
6.9940
6.5860
6.5825
6.2879
6.2712
3.7669
MeO
O
O
O
6ia
1.01
1.05
1.03
1.04
1.04
1.02
1.01
0.98
1.00
3.02
f1 (ppm)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126.7689
124.0207
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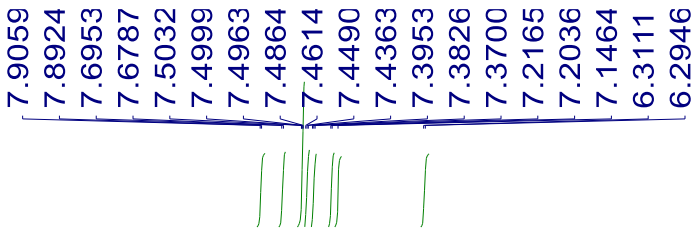
56.0317
MeO
O
O
O
6ia
f1 (ppm)

6ja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7.5032
7.4999
7.4963
7.4864
7.4614
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7.3953
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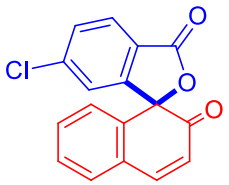
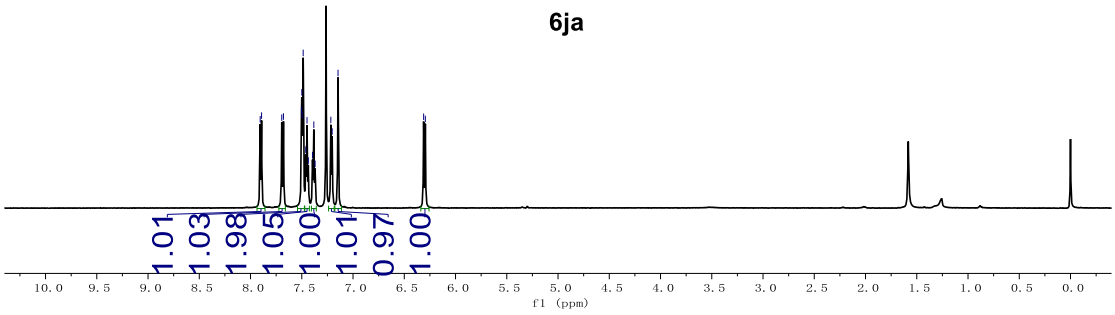
7.2165
7.2036
7.1464
6.3111
6.2946


Cl
O
O
O

6ja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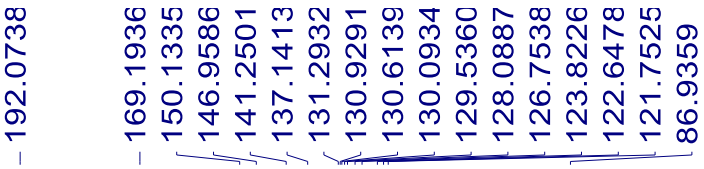
1.03
1.98
1.05
1.00
1.01
0.97
1.00
f1 (ppm)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137.1413
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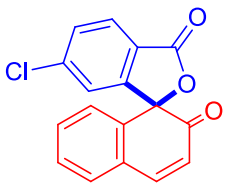
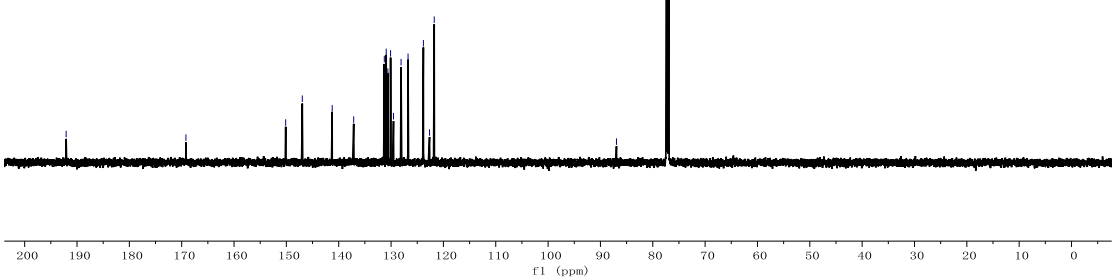
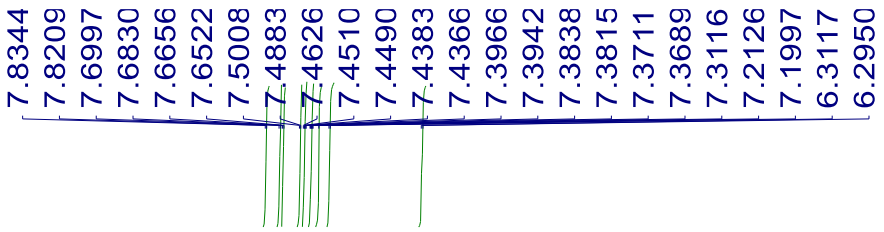
128.0887
126.7538
123.8226
122.6478
121.7525
86.9359


Cl
O
O
O

6ja


f1 (ppm)

6ka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7.4626
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7.4490
7.4383
7.4366
7.3966
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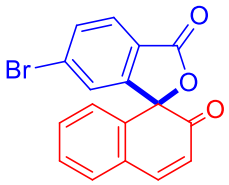
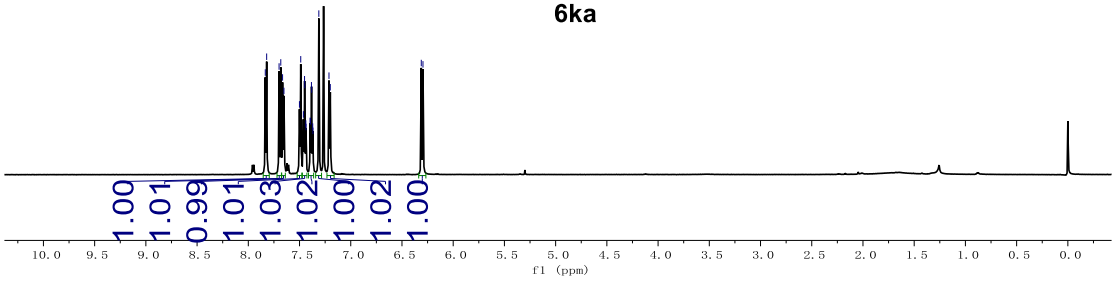
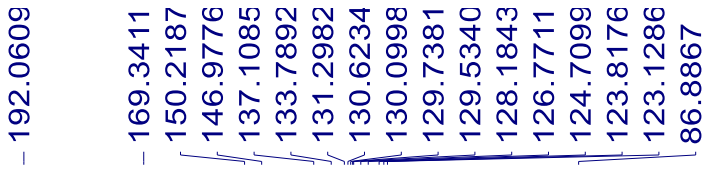
7.3689
7.3116
7.2126
7.1997
6.3117
6.2950


Br
O
O
O

6ka


1.00
1.01
0.99
1.01
1.03
1.02
1.00
1.02
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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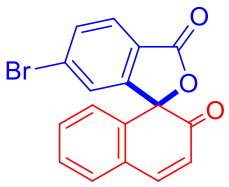
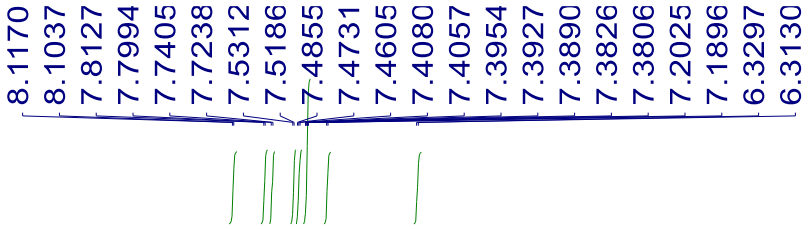
146.9776
137.1085
133.7892
131.2982
130.6234
130.0998
129.7381
129.5340
128.1843
126.7711
124.7099
123.8176
123.1286
86.8867


Br
O
O
O

6ka

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

6la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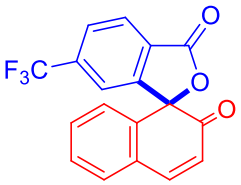
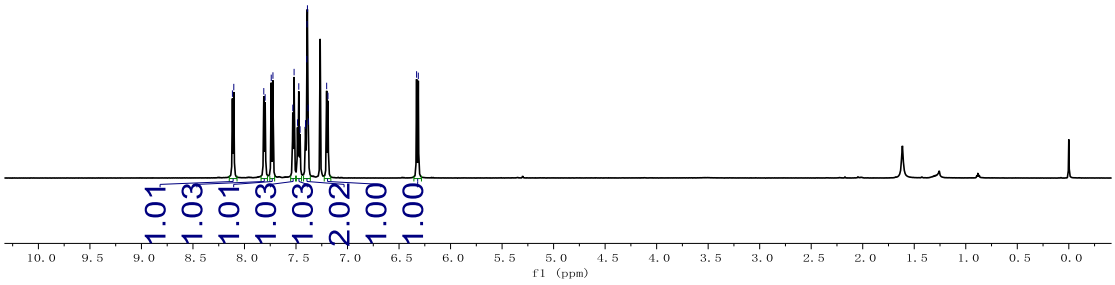
8.1037
7.8127
7.7994
7.7405
7.7238
7.5312
7.5186
7.4855
7.4731
7.4605
7.4080
7.4057
7.3954
7.3927
7.3890
7.3826
7.3806
7.2025
7.1896
6.3297
6.3130


F3C
O
O
O

6la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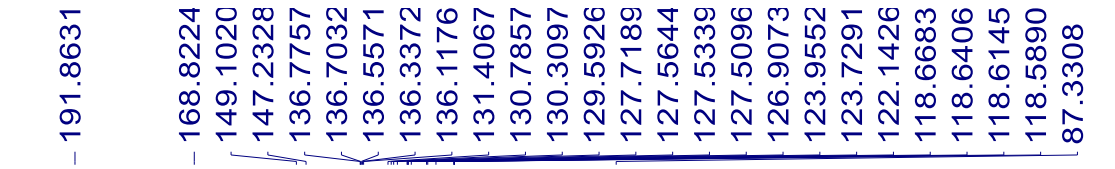
1.03
1.01
1.03
1.03
2.02
1.00
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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168.8224
149.1020
147.2328
136.7757
136.7032
136.5571
136.3372
136.1176
131.4067
130.7857
130.3097
129.5926
127.7189
127.5644
127.5339
127.5096
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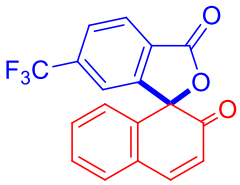
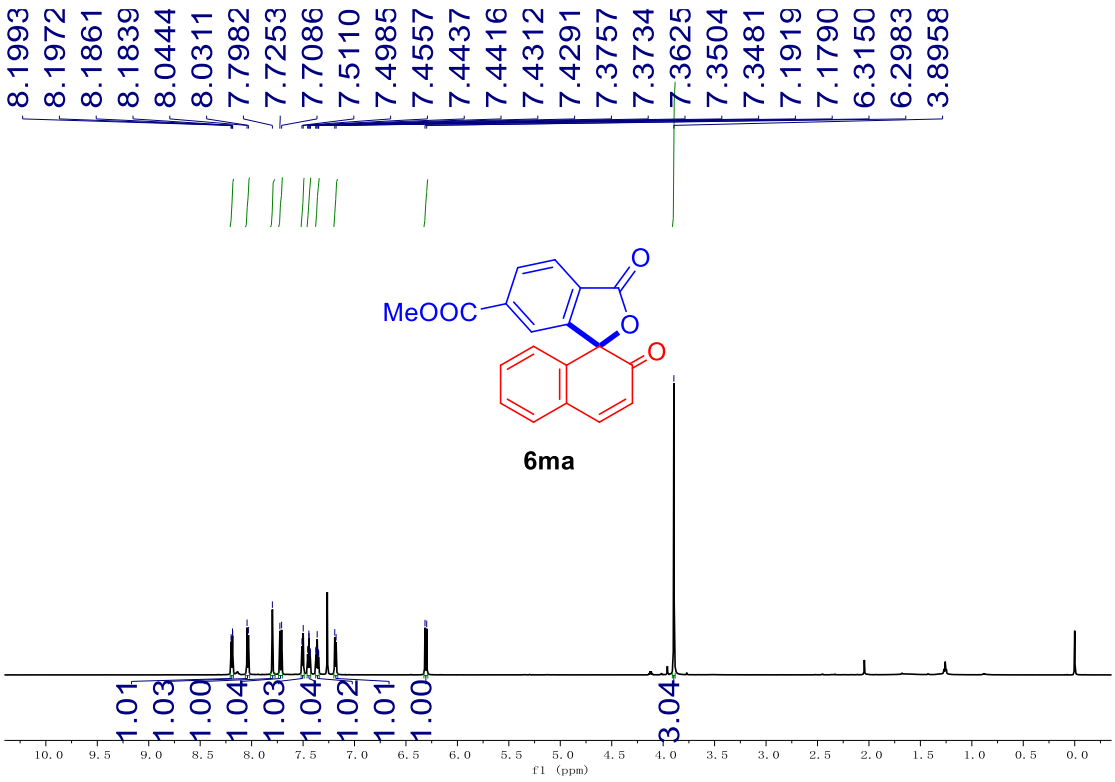
123.9552
123.7291
122.1426
118.6683
118.6406
118.6145
118.5890
87.3308


F3C
O
O
O

6la

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

6ma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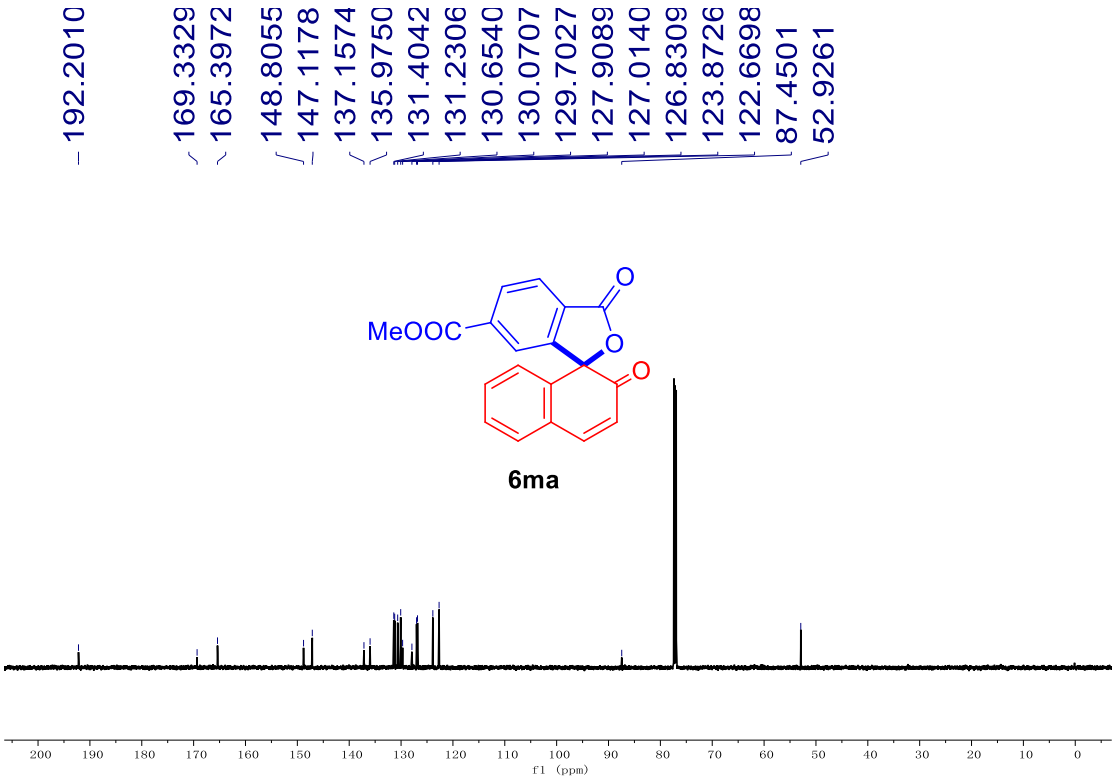
8.1972
8.1861
8.1839
8.0444
8.0311
7.7982
7.7253
7.7086
7.5110
7.4985
7.4557
7.4437
7.4416
7.4312
7.4291
7.3757
7.3734
7.3625
7.3504
7.3481
7.1919
7.1790
6.3150
6.2983
3.8958
MeOOC
O
O
O
6ma
1.01
1.03
1.00
1.04
1.03
1.04
1.02
1.01
1.00
3.04
f1 (ppm)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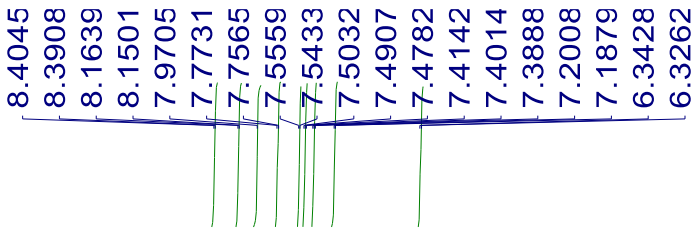
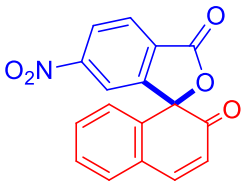
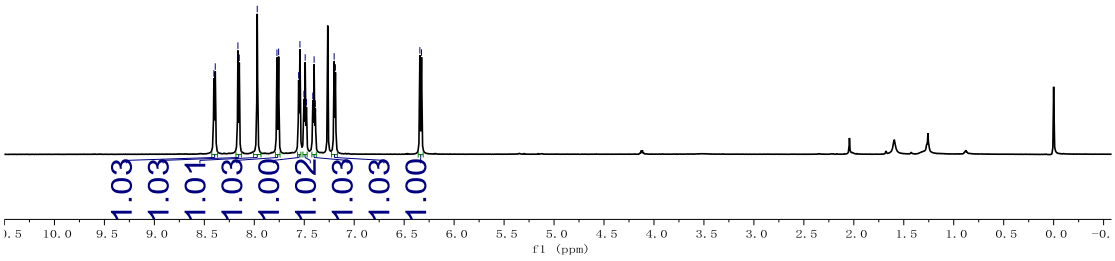
165.3972
148.8055
147.1178
137.1574
135.9750
131.4042
131.2306
130.6540
130.0707
129.7027
127.9089
127.0140
126.8309
123.8726
122.6698
87.4501
52.9261
MeOOC
O
O
O
6ma
f1 (ppm)

6na


8.4045
8.3908
8.1639
8.1501
7.9705
7.7731
7.7565
7.5559
7.5433
7.5032
7.4907
7.4782
7.4142
7.4014
7.3888
7.2008
7.1879
6.3428
6.3262


O2N
O
O
O

6na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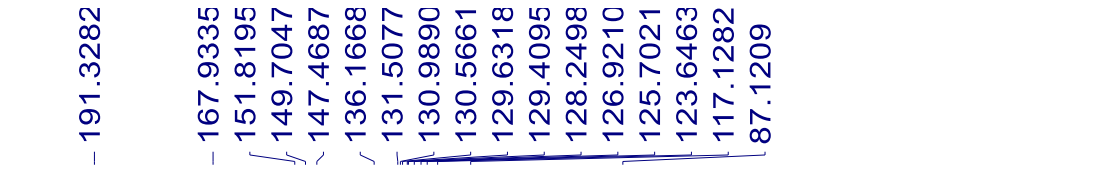
1.03
1.01
1.03
1.00
1.02
1.03
1.03
1.00
f1 (ppm)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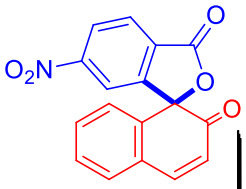
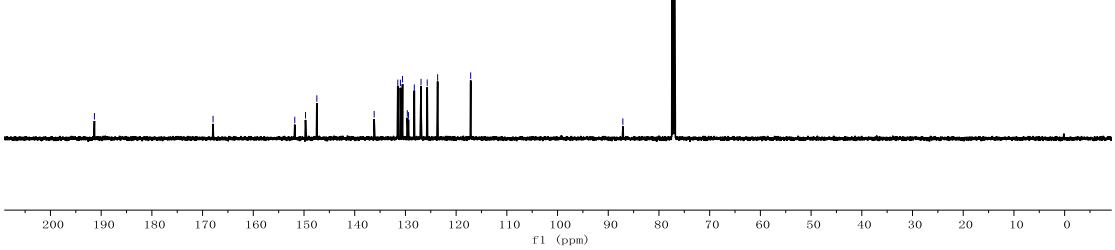
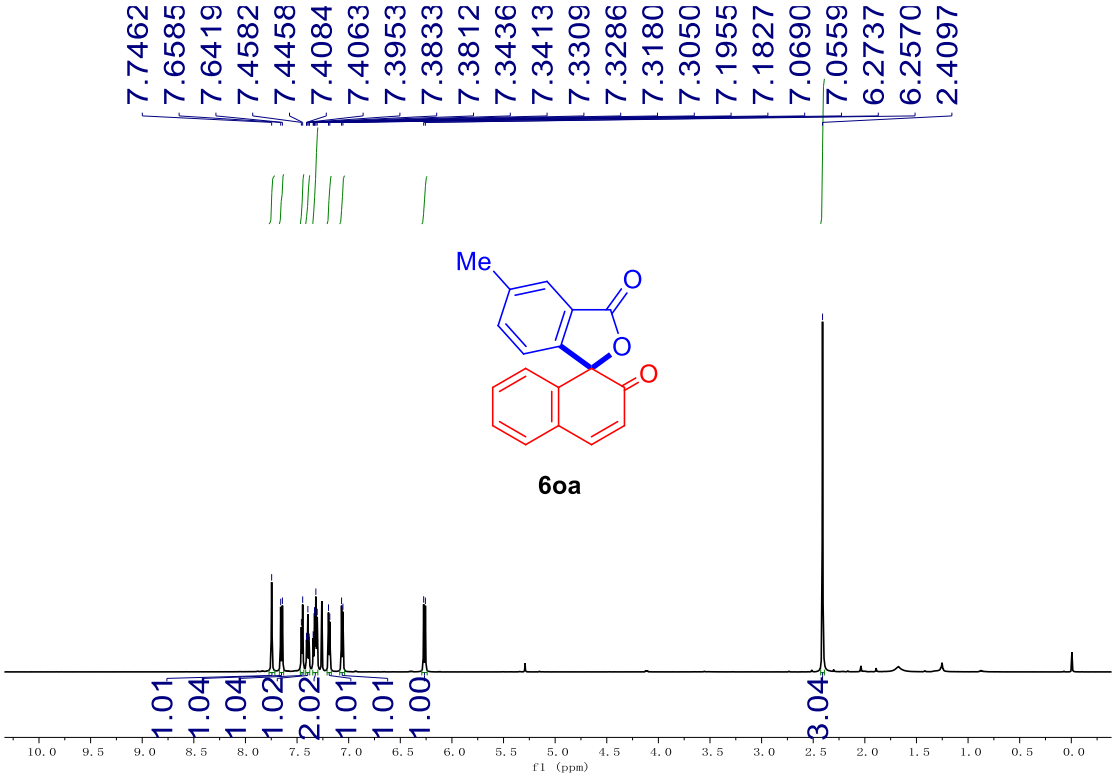
167.9335
151.8195
149.7047
147.4687
136.1668
131.5077
130.9890
130.5661
129.6318
129.4095
128.2498
126.9210
125.7021
123.6463
117.1282
87.1209


O2N
O
O
O

6na


f1 (ppm)

6oa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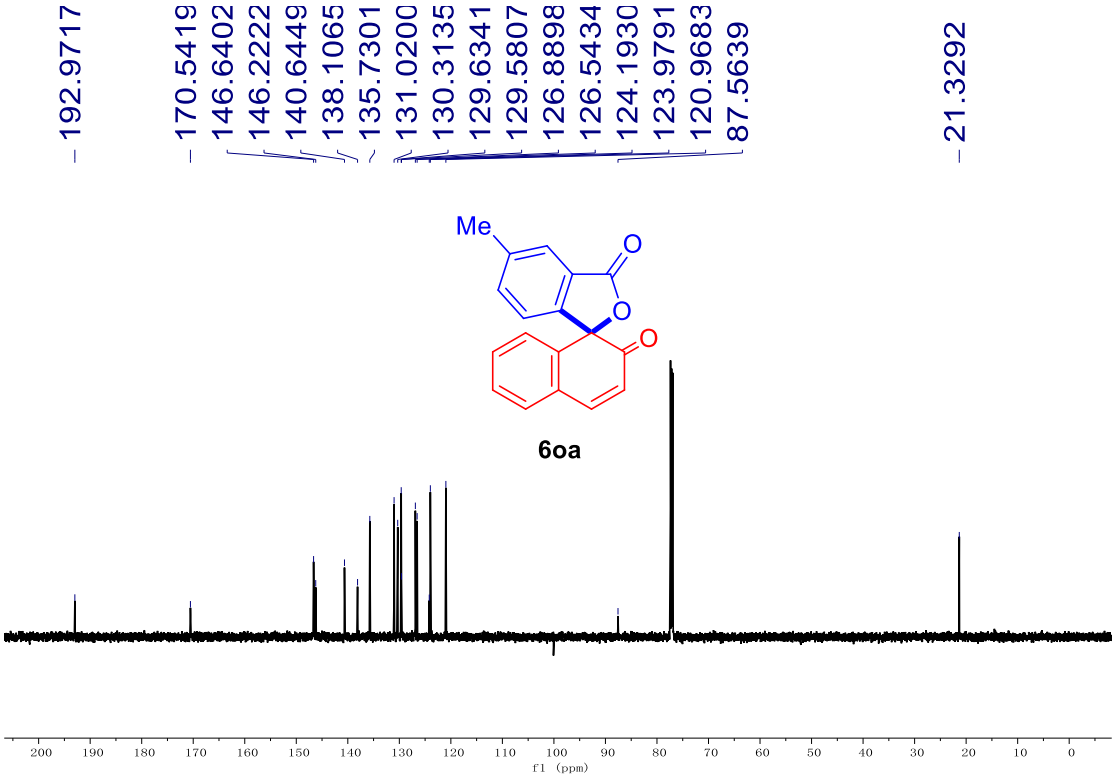
7.4458
7.4084
7.4063
7.3953
7.3833
7.3812
7.3436
7.3413
7.3309
7.3286
7.3180
7.3050
7.1955
7.1827
7.0690
7.0559
6.2737
6.2570
2.4097
Me
O
O
O
1.01
1.04
1.04
1.02
2.02
1.01
1.01
1.00
3.04
f1 (ppm)

6oa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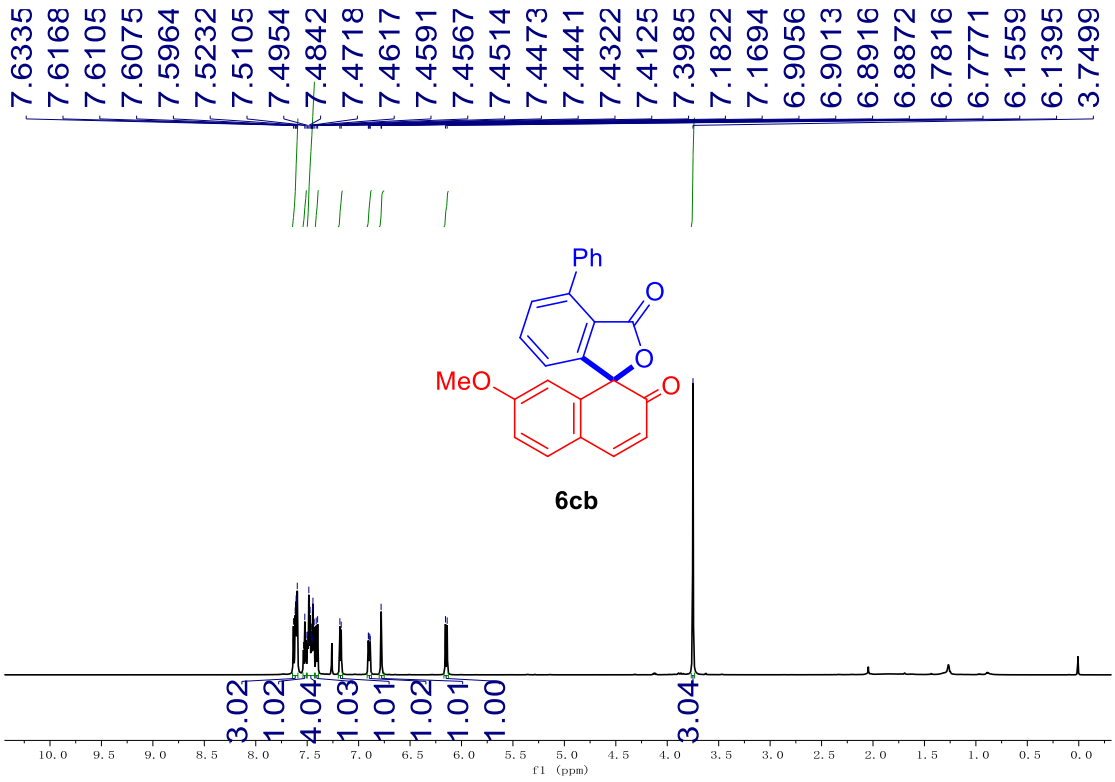
170.5419
146.6402
146.2222
140.6449
138.1065
135.7301
131.0200
130.3135
129.6341
129.5807
126.8898
126.5434
124.1930
123.9791
120.9683
87.5639
21.3292
Me
O
O
O
f1 (ppm)

6cb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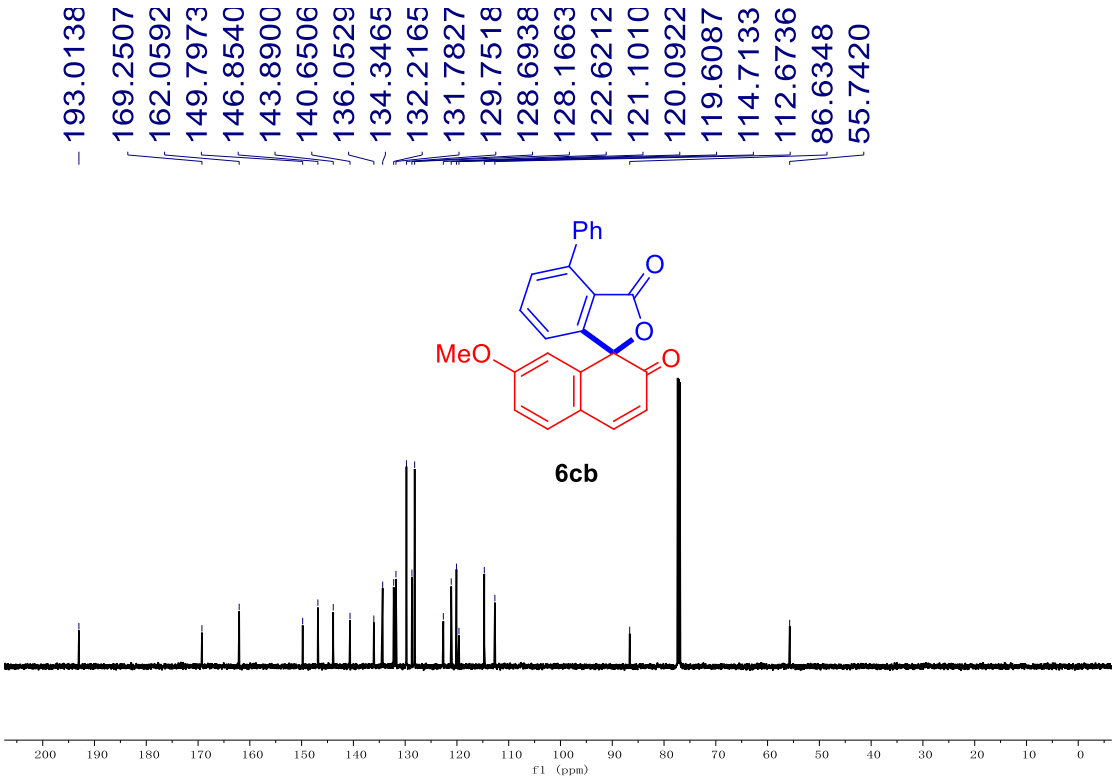
7.6168
7.6105
7.6075
7.5964
7.5232
7.5105
7.4954
7.4842
7.4718
7.4617
7.4591
7.4567
7.4514
7.4473
7.4441
7.4322
7.4125
7.3985
7.1822
7.1694
6.9056
6.9013
6.8916
6.8872
6.7816
6.7771
6.1559
6.1395
3.7499
Ph
O
O
MeO
O
6cb
3.02
1.02
4.04
1.03
1.01
1.02
1.01
1.00
3.04
f1 (ppm)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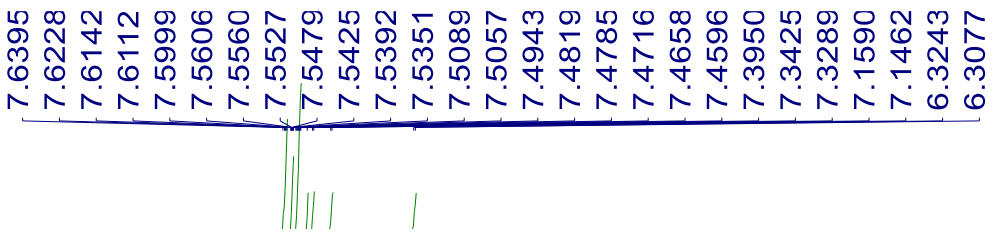
146.8540
143.8900
140.6506
136.0529
134.3465
132.2165
131.7827
129.7518
128.6938
128.1663
122.6212
121.1010
120.0922
119.6087
114.7133
112.6736
86.6348
55.7420
Ph
O
O
MeO
O
6cb
f1 (ppm)

6cc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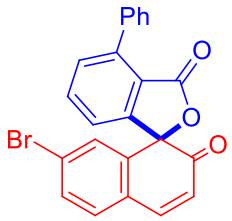
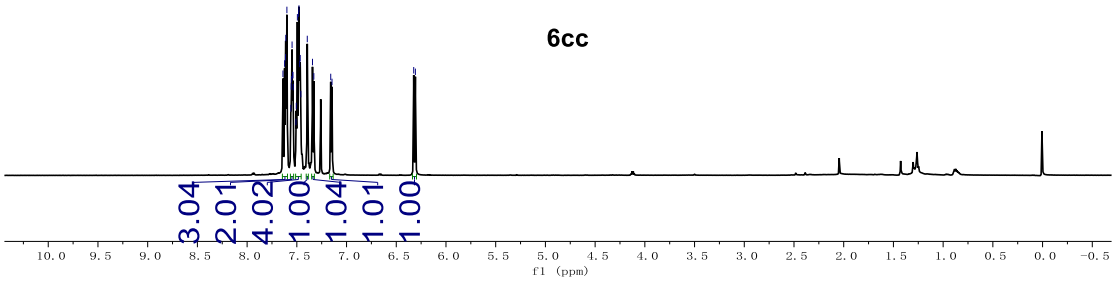
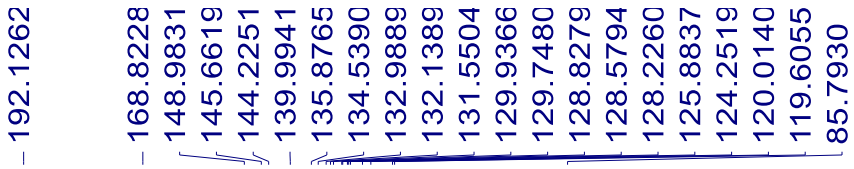
7.6228
7.6142
7.6112
7.5999
7.5606
7.5560
7.5527
7.5479
7.5425
7.5392
7.5351
7.5089
7.5057
7.4943
7.4819
7.4785
7.4716
7.4658
7.4596
7.3950
7.3425
7.3289
7.1590
7.1462
6.3243
6.3077


Ph
O
O
Br
O

6cc


3.04
2.01
4.02
1.00
1.04
1.01
1.00
f1 (ppm)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139.9941
135.8765
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132.9889
132.1389
131.5504
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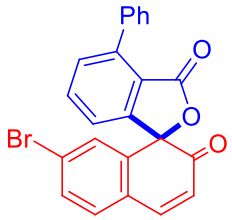
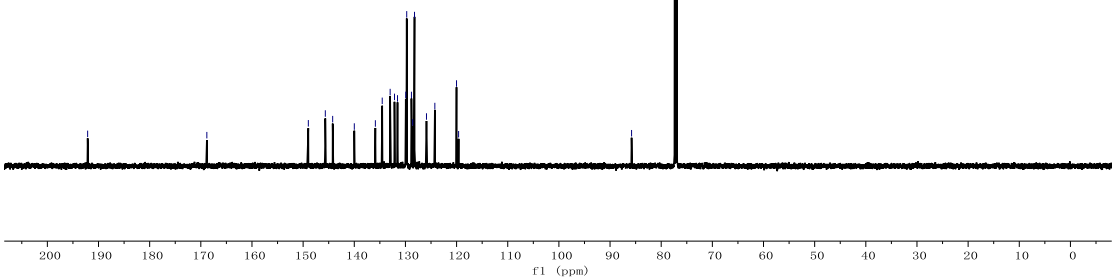
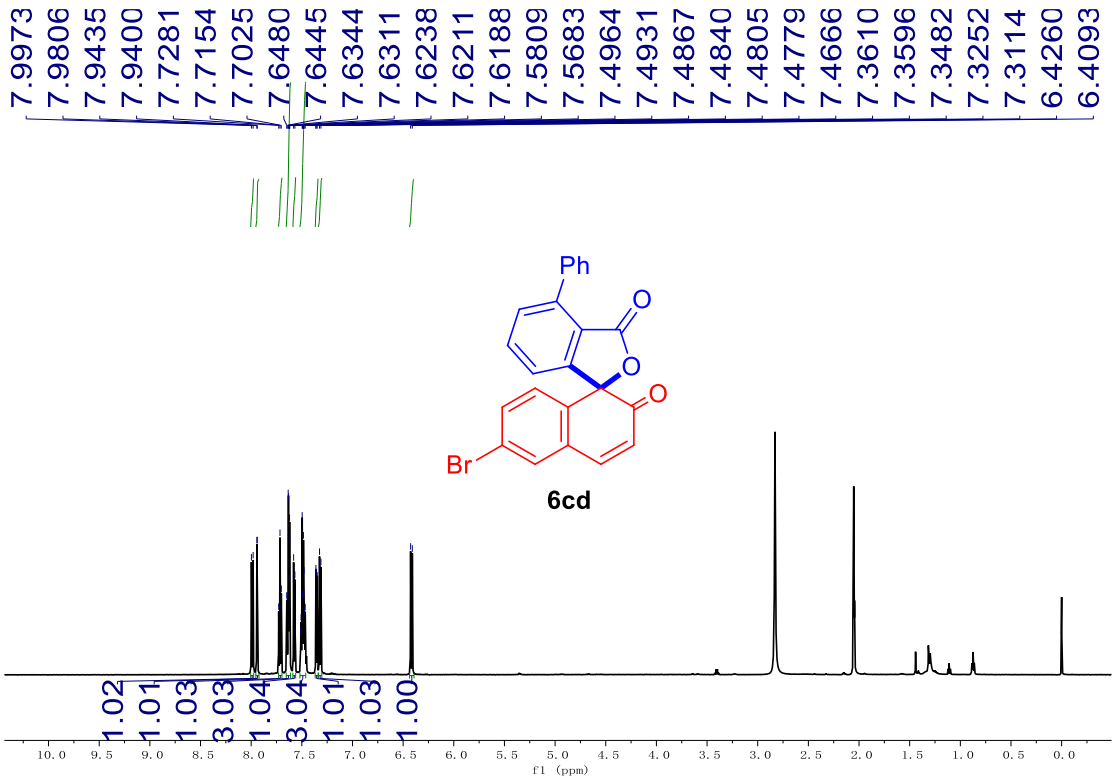
128.2260
125.8837
124.2519
120.0140
119.6055
85.7930


Ph
O
O
Br
O

6cc


f1 (ppm)

6cd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7.7281
7.7154
7.7025
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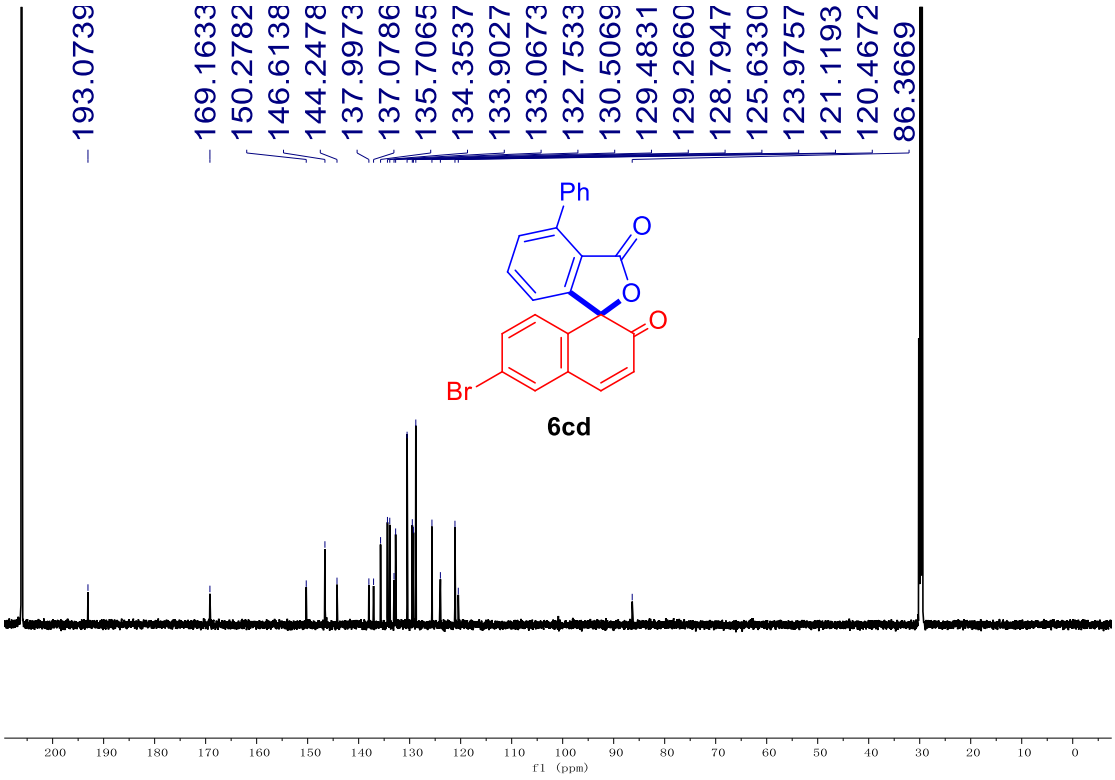
7.6445
7.6344
7.6311
7.6238
7.6211
7.6188
7.5809
7.5683
7.4964
7.4931
7.4867
7.4840
7.4805
7.4779
7.4666
7.3610
7.3596
7.3482
7.3252
7.3114
6.4260
6.4093
Ph
O
O
O
Br
6cd
1.02
1.01
1.03
3.03
1.04
3.04
1.01
1.03
1.00
f1 (ppm)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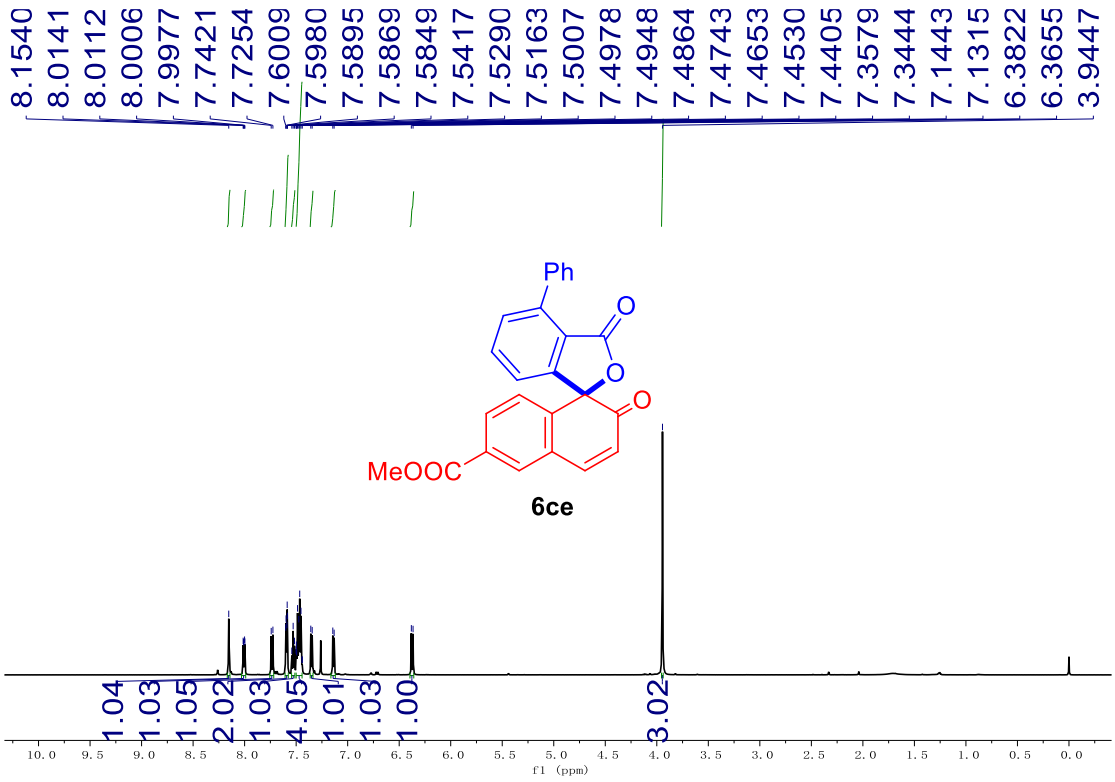
133.0673
132.7533
130.5069
129.4831
129.2660
128.7947
125.6330
123.9757
121.1193
120.4672
86.3669
Ph
O
O
O
Br
6cd
f1 (ppm)

6ce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8.0112
8.0006
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7.7421
7.7254
7.6009
7.5980
7.5895
7.5869
7.5849
7.5417
7.5290
7.5163
7.5007
7.4978
7.4948
7.4864
7.4743
7.4653
7.4530
7.4405
7.3579
7.3444
7.1443
7.1315
6.3822
6.3655
3.9447
Ph
O
O
O
MeOOC
6ce
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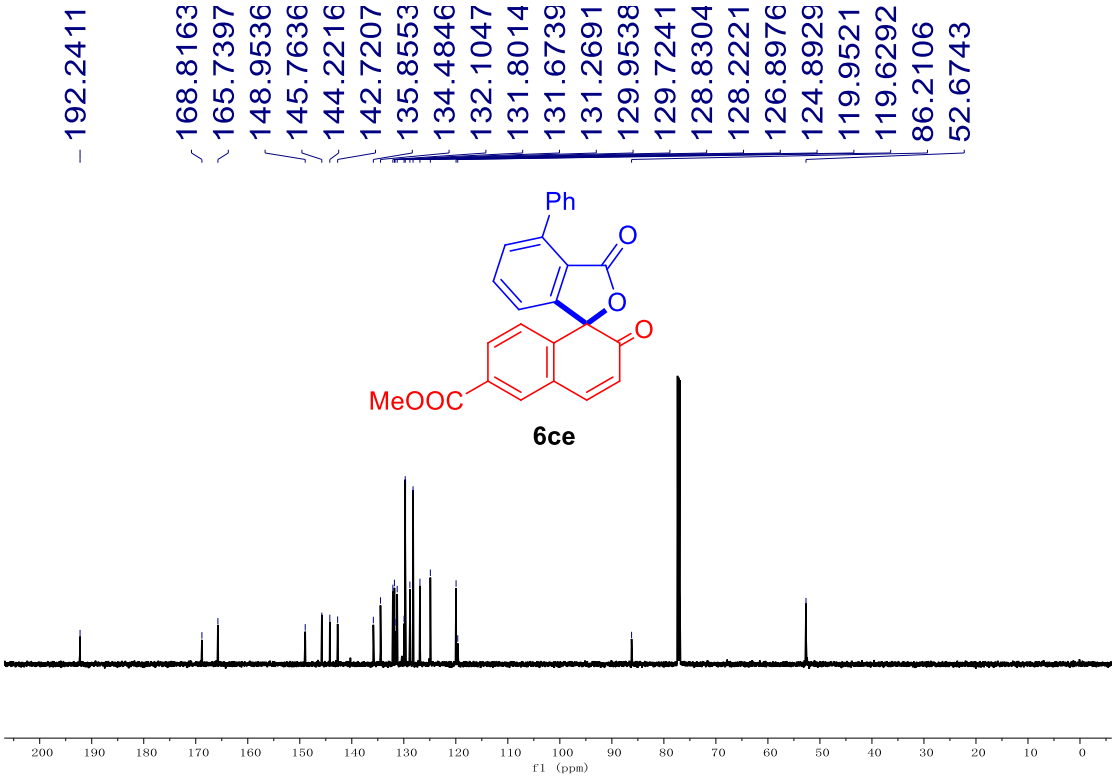
1.03
1.05
2.02
1.03
4.05
1.01
1.03
1.00
3.02
f1 (ppm)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142.7207
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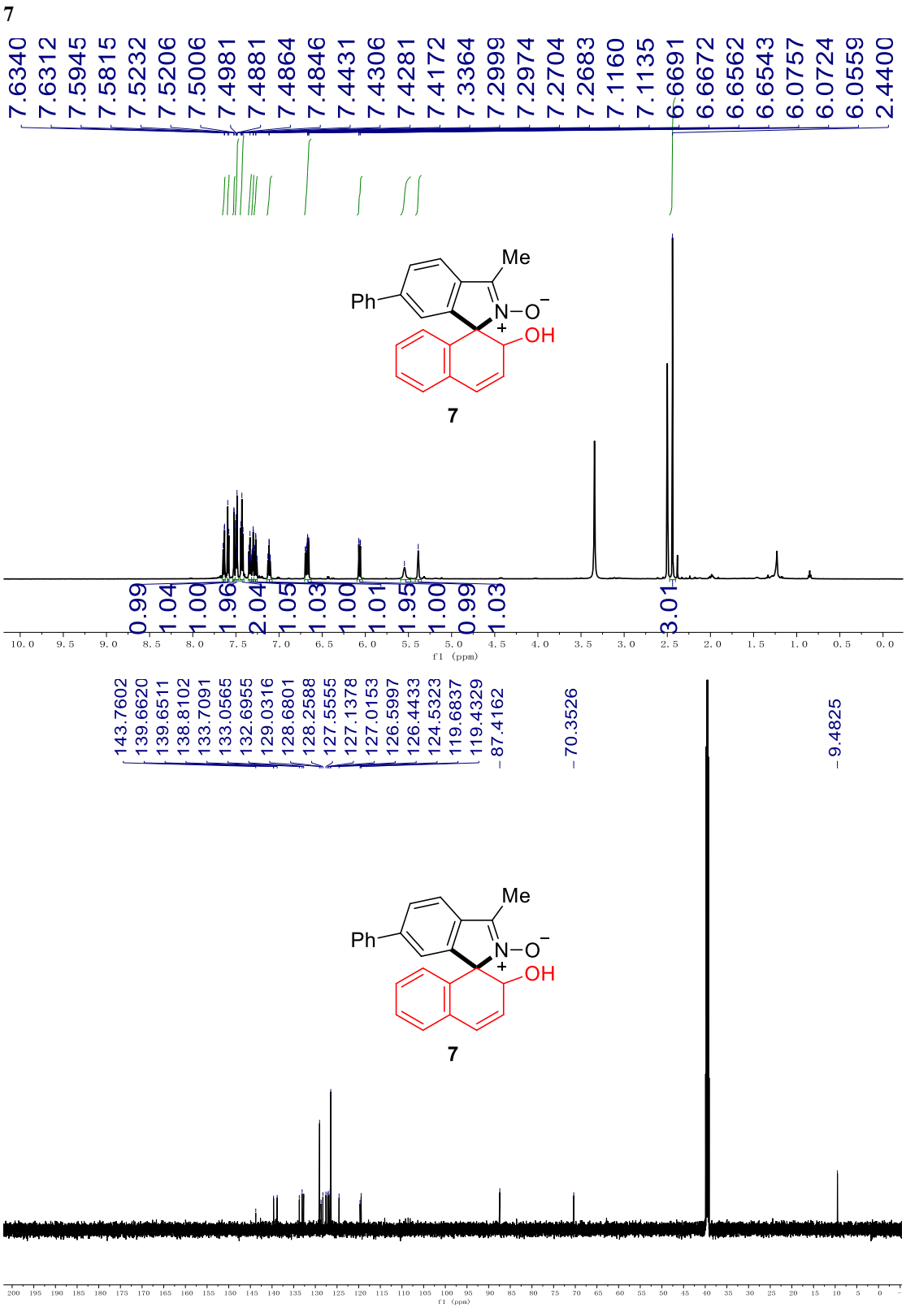
132.1047
131.8014
131.6739
131.2691
129.9538
129.7241
128.8304
128.2221
126.8976
124.8929
119.9521
119.6292
86.2106
52.6743
Ph
O
O
O
MeOOC
6ce
f1 (ppm)

7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7.6312
7.5945
7.5815
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7.5206
7.5006
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7.4881
7.4864
7.4846
7.4431
7.4306
7.4281
7.4172
7.3364
7.2999
7.2974
7.2704
7.2683
7.1160
7.1135
6.6691
6.6672
6.6562
6.6543
6.0757
6.0724
6.0559
2.4400
Me
Ph
N−O
+
OH
7
0.99
1.04
1.00
1.96
2.04
1.05
1.03
1.00
1.01
1.95
1.00
0.99
1.03
3.01
f1 (ppm)
143.7602
139.6620
139.6511
138.8102
133.7091
133.0565
132.6955
129.0316
128.6801
128.2588
127.5555
127.1378
127.0153
126.5997
126.4433
124.5323
119.6837
119.4329
87.4162
70.3526
9.4825
Me
Ph
N−O
+
OH
7
f1 (ppm)