Supporting Information for

Radical spirocyclization of biaryl ynones for the construction of NO_2-containing spiro[5.5]trienones

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1. General considerations

All reactions were carried out under air. ^{1}H NMR and ^{13}C NMR spectra were measured on a Bruker Avance NMR spectrometer (600 MHz/151 MHz/565 NMR) in $CDCl_3$ as solvent and recorded in ppm relative to internal tetramethylsilane standard. ^{1}H NMR data are reported as follows: δ, chemical shift; coupling constants (J are given in Hertz, Hz) and integration. Abbreviations to denote the multiplicity of a particular signal were s (singlet), d (doublet), t (triplet), q (quartet), dd (doublet of doublets) and m (multiplet).

2. General procedure for the synthesis of 3.

Conditions: A 15 mL sealable reaction tube was charged with 1-(4'-methoxy-[1,1'-biphenyl]-2-yl)-3-phenylprop-2-yn-1-one (**1a**, 0.2 mmol), $NaNO_2$ (**2**, 0.4 mmol), $K_2S_2O_8$ (0.8 mmol), MeCN (2 mL), and a magnetic stir bar. Then the mixtures were allowed to react at 110 ^oC for 36 h under air. After completion of the reaction, the mixture was concentrated to yield the crude product, which was further purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 7:1) to give the desired product **3a**.

3. Mechanism study

Free-radical inhibition and trapping experiment

A 15 mL Schlenk flask was charged with 1-(4'-methoxy-[1,1'-biphenyl]-2-yl)-3-phenylprop-2-yn-1-one (**1a**), $NaNO_2$ (**2**, 0.4 mmol), $K_2S_2O_8$ (0.8 mmol), MeCN (2 mL), BHT (0.4 mmol), and a magnetic stir bar. The reaction mixture was stirred at 110 °C for 36 h. After completion of the reaction, TLC detection showed that the reaction was completely inhibited and no desired product **3a** was found, indicating a radical pathway involved in the reaction. Meanwhile, the key intermediate nitro radical is captured by radical acceptor (acrylamide) and its corresponding product nitrative oxindole was isolated in 61% yield.

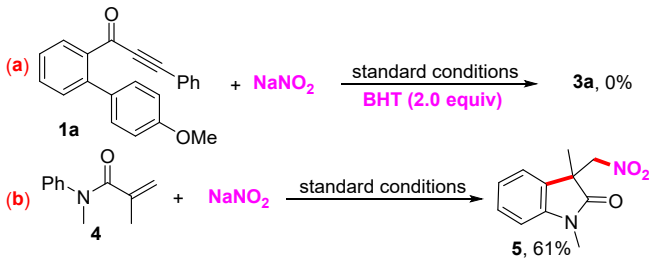


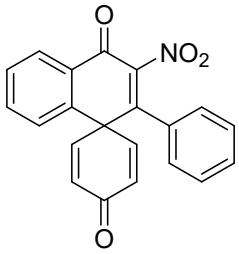
Figure S1. Control Experiments.

The product purified by flash column chromatography on silica gel (PE/AcOEt = 5:1) to afford the **5** as a yellow liquid (26.74 mg, 61% yield).[1] ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 (td, *J* = 7.8, 1.1 Hz, 1H), 7.17 – 7.12 (m, 1H), 7.01 (td, *J* = 7.6, 0.7 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 4.84 (d, *J* = 13.5 Hz, 1H), 4.69 (d, *J* = 13.5 Hz, 1H), 3.21 (s, 3H), 1.34 (s, 3H).^{13}C NMR (151 MHz, $CDCl_3$) δ 177.2, 143.5, 129.2, 129.1, 122.9, 122.4, 108.8, 79.0, 47.0, 26.6, 21.7.

Reference 1: Y.-M. Li, X.-H. Wei, X.-A. Li, S.-D. Yang, *Chem. Commun.* **2013**, *49*, 11701-11703.

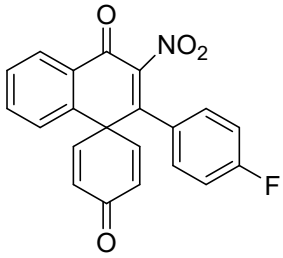
4. Characterization data of products

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



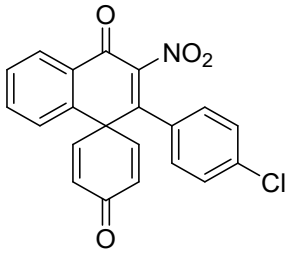
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3a** as a yellow solid (56.95 mg, 83% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.34 (d, *J* = 7.9 Hz, 1H), 7.71 – 7.67 (m, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.35 – 7.31 (m, 3H), 7.18 – 7.13 (m, 2H), 6.79 (d, *J* = 10.0 Hz, 2H), 6.42 (d, *J* = 10.0 Hz, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.8, 174.2, 149.9, 147.3, 146.0, 137.8, 134.8, 131.3, 130.4, 130.1, 129.6, 129.4, 128.7, 128.3, 128.2, 127.5, 50.4.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3b** as a yellow solid (62.07 mg, 86% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.70 (td, *J* = 7.9, 1.4 Hz, 1H), 7.64 – 7.58 (m, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.17 (ddd, *J* = 7.9, 5.0, 2.5 Hz, 2H), 7.08 – 6.98 (m, 2H), 6.84 – 6.73 (m, 2H), 6.51 – 6.32 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.6, 174.1, 163.6 (d, *J* = 251.9 Hz), 150.2, 146.3, 145.8, 137.7, 134.9, 131.5, 129.8 (d, *J* = 8.7 Hz), 129.7, 129.3, 128.7, 128.3, 126.0 (d, *J* = 3.3 Hz), 115.7 (d, *J* = 22.0 Hz), 50.4.

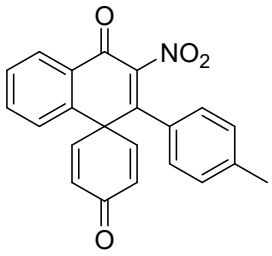
2'-(4-Chlorophenyl)-3'-nitro-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3c)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3c** as a yellow solid (66.48 mg, 88% yield). ^{1}H

NMR (600 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.70 (td, *J* = 7.9, 1.3 Hz, 1H), 7.62 (dd, *J* = 11.2, 4.0 Hz, 1H), 7.32 (dd, *J* = 8.0, 6.0 Hz, 3H), 7.11 (d, *J* = 8.5 Hz, 2H), 6.78 (d, *J* = 10.0 Hz, 2H), 6.45 (d, *J* = 10.0 Hz, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.6, 174.0, 150.0, 146.0, 145.7, 137.7, 136.8, 135.0, 131.5, 129.7, 129.3, 129.0, 128.75, 128.73, 128.4, 128.3, 50.2.

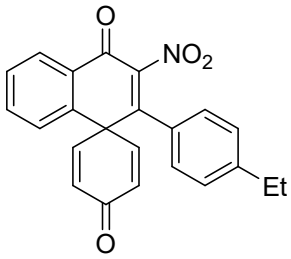
3'-Nitro-2'-(*p*-tolyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3d)



V

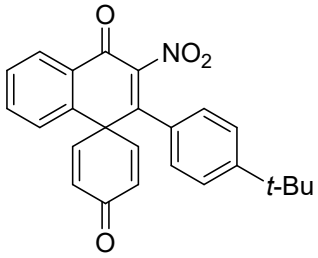
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3d** as a yellow solid (55.68 mg, 78% yield). ^1H NMR (600 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.68 (td, *J* = 7.8, 1.5 Hz, 1H), 7.64 – 7.56 (m, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 8.2 Hz, 2H), 6.84 – 6.75 (m, 2H), 6.47 – 6.36 (m, 2H), 2.32 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.0, 174.3, 150.0, 147.7, 146.2, 140.6, 137.9, 134.8, 131.2, 129.5, 129.4, 128.9, 128.7, 128.2, 127.36, 127.31, 50.5, 21.3.

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



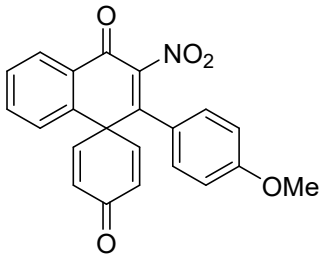
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3e** as a yellow solid (61.55 mg, 83% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.68 (td, *J* = 7.9, 1.3 Hz, 1H), 7.59 (dd, *J* = 11.2, 4.0 Hz, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.14 (d, *J* = 8.1 Hz, 2H), 7.07 (d, *J* = 8.2 Hz, 2H), 6.80 (t, *J* = 6.3 Hz, 2H), 6.42 (t, *J* = 6.4 Hz, 2H), 2.62 (q, *J* = 7.6 Hz, 2H), 1.21 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.0, 174.3, 150.0, 147.7, 146.6, 146.2, 137.9, 134.7, 131.2, 129.5, 129.4, 128.7, 128.2, 127.7, 127.5, 127.4, 50.5, 28.5, 14.8.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3f** as a yellow solid (61.01 mg, 76% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.29 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.67 (td, *J* = 7.9, 1.4 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.32 (t, *J* = 7.7 Hz, 3H), 7.10 (d, *J* = 8.4 Hz, 2H), 6.83 – 6.78 (m, 2H), 6.46 – 6.40 (m, 2H), 1.27 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.1, 174.3, 153.5, 149.9, 147.8, 146.3, 137.9, 134.8, 131.2, 129.5, 129.4, 128.7, 128.1, 127.3, 127.1, 125.2, 50.5, 34.7, 31.0.

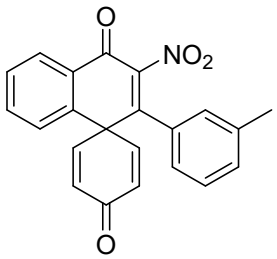
2'-(4-Methoxyphenyl)-3'-nitro-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3g)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 3:1) to afford the **3g** as a yellow solid (61.14 mg, 80% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.60 (td, *J* = 7.8, 1.4 Hz, 1H), 7.53 – 7.47 (m, 1H), 7.25 (d, *J* = 7.8 Hz, 1H), 7.07 – 6.99 (m, 2H), 6.77 – 6.73 (m, 2H), 6.73 – 6.68 (m, 2H), 6.39 – 6.32 (m, 2H), 3.71

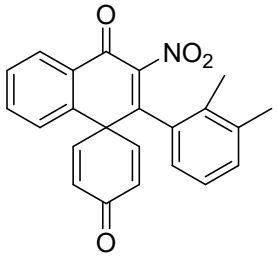
(s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.0, 174.3, 171.2, 161.0, 150.2, 147.5, 146.3, 137.8, 134.7, 131.2, 129.5, 129.4, 128.9, 128.6, 128.2, 122.3, 113.8, 60.4, 55.2, 50.7.

3'-Nitro-2'-(m-tolyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3h)



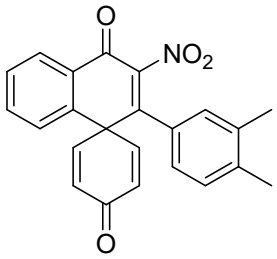
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3h** as a yellow solid (60.66 mg, 85% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 7.9, 1.1 Hz, 1H), 7.68 (td, *J* = 7.7, 1.4 Hz, 1H), 7.63 – 7.56 (m, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.19 (d, *J* = 7.3 Hz, 2H), 6.98 – 6.89 (m, 2H), 6.79 (d, *J* = 10.1 Hz, 2H), 6.42 (dd, *J* = 10.6, 2.2 Hz, 2H), 2.30 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.9, 174.3, 149.8, 147.6, 146.1, 138.0, 137.8, 134.8, 131.2, 131.1, 130.1, 129.5, 129.4, 128.7, 128.2, 128.1, 127.9, 124.6, 50.4, 21.4.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3i** as a yellow solid (58.61 mg, 79% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.34 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.69 (td, *J* = 7.9, 1.4 Hz, 1H), 7.64 – 7.59 (m, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.13 (d, *J* = 7.5 Hz, 1H), 7.00 (t, *J* = 7.7 Hz, 1H), 6.87 (dd, *J* = 10.0, 3.1 Hz, 1H), 6.82 – 6.75 (m, 2H), 6.42 (dd, *J* = 9.9, 1.6 Hz, 1H), 6.33 (dd, *J* = 10.0, 1.6 Hz, 1H), 2.22 (s, 3H), 2.15 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.8, 174.1, 149.9, 148.4, 146.7, 145.3, 138.5, 137.9, 134.8, 133.7, 131.6, 131.1, 130.9, 129.6, 129.4, 128.9, 128.6, 128.3, 126.2, 124.7, 51.4, 20.2, 18.5.

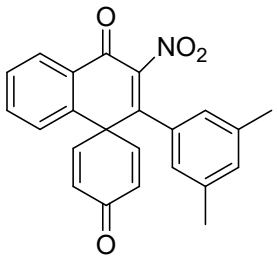
2'-(3,4-Dimethylphenyl)-3'-nitro-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3j)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3j** as a yellow solid (56.38 mg, 76% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.67 (td, *J* = 7.9,

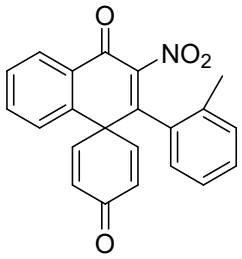
1.4 Hz, 1H), 7.62 – 7.56 (m, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.05 (d, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 10.0 Hz, 2H), 6.79 – 6.76 (m, 2H), 6.44 – 6.40 (m, 2H), 2.22 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.1, 174.3, 149.9, 147.8, 146.3, 139.3, 137.8, 136.7, 134.7, 131.1, 129.5, 129.5, 129.4, 128.6, 128.2, 128.2, 127.7, 124.8, 50.6, 19.8, 19.6.

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



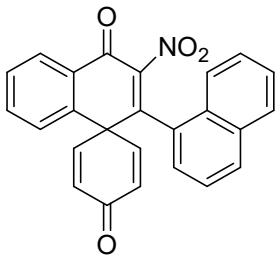
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3k** as a yellow solid (54.11 mg, 73% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.68 (td, *J* = 7.7, 1.5 Hz, 1H), 7.63 – 7.57 (m, 1H), 7.32 (dd, *J* = 7.9, 0.5 Hz, 1H), 6.99 (s, 1H), 6.79 – 6.75 (m, 2H), 6.73 (s, 2H), 6.45 – 6.36 (m, 2H), 2.25 (s, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.0, 174.3, 149.7, 147.8, 146.2, 137.9, 137.8, 134.7, 132.1, 131.1, 130.1, 129.5, 129.4, 128.6, 128.2, 125.0, 50.4, 21.2.

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



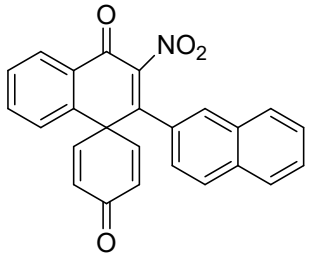
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3l** as a yellow solid (60.69 mg, 85% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.33 (dd, *J* = 7.9, 1.3 Hz, 1H), 7.69 (td, *J* = 7.7, 1.5 Hz, 1H), 7.64 – 7.57 (m, 1H), 7.35 (dd, *J* = 16.4, 4.5 Hz, 1H), 7.25 (td, *J* = 7.6, 1.2 Hz, 1H), 7.16 (d, *J* = 7.7 Hz, 1H), 7.09 (t, *J* = 7.5 Hz, 1H), 6.96 (dd, *J* = 7.7, 0.8 Hz, 1H), 6.89 (dd, *J* = 10.0, 3.1 Hz, 1H), 6.79 (dd, *J* = 9.9, 3.1 Hz, 1H), 6.44 (dd, *J* = 9.9, 1.6 Hz, 1H), 6.34 (dd, *J* = 10.0, 1.6 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.7, 174.1, 150.1, 147.6, 146.5, 145.2, 138.5, 135.3, 134.9, 131.2, 131.1, 130.9, 130.2, 129.6, 129.3, 128.8, 128.7, 128.5, 128.2, 124.9, 51.3, 20.6.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3m** as a yellow solid (62.87 mg, 80% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.39 (d, *J* = 7.8 Hz, 1H), 7.86 – 7.81 (m, 2H), 7.73 (dd, *J* = 6.2, 3.4 Hz, 1H), 7.71 – 7.67 (m, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.48 (dt, *J* = 6.4, 3.4 Hz, 2H), 7.40 – 7.36 (m, 1H), 7.33 (d, *J* = 7.9 Hz, 1H), 7.21 (d, *J* = 7.1 Hz, 1H), 6.89 (dd, *J* = 10.0, 3.1 Hz, 1H), 6.82 (dd, *J* = 9.9, 3.1 Hz, 1H), 6.45 (dd, *J* = 9.9, 1.4 Hz, 1H), 5.99 (dd, *J* = 10.0, 1.4 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.6, 174.1, 150.9, 146.8, 146.6, 145.6, 138.5, 134.9, 133.3, 131.1, 131.0, 130.8, 129.8, 129.7, 129.5, 128.9, 128.7, 128.4, 127.1, 126.8, 126.6, 126.5, 124.8, 124.0, 51.4.

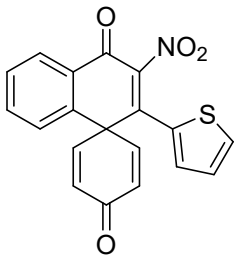
2'-(Naphthalen-2-yl)-3'-nitro-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3n)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3n** as a yellow solid (59.79 mg, 76% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.30 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.75 (d, *J* = 7.9 Hz, 1H), 7.72 (d, *J* = 8.3 Hz, 2H), 7.63 (td, *J* = 7.9, 1.4 Hz, 1H), 7.58 – 7.54 (m, 2H), 7.50 – 7.44 (m, 2H), 7.28 (d, *J* = 7.6 Hz, 1H), 7.19 – 7.16 (m, 1H), 6.79 (d, *J* = 10.1 Hz, 2H), 6.34 (d, *J* = 10.0 Hz, 2H). ^{13}C NMR (151 MHz,

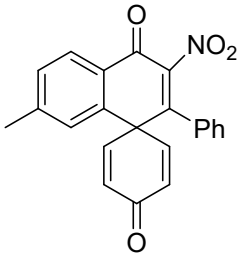
$CDCl_3$) δ 183.7, 174.2, 150.2, 147.4, 146.1, 137.9, 134.8, 133.5, 132.0, 131.3, 129.6, 129.5, 128.6, 128.4, 128.3, 128.2, 127.8, 127.8, 127.6, 127.5, 127.1, 124.2, 50.6.

3'-Nitro-2'-(thiophen-2-yl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3o)



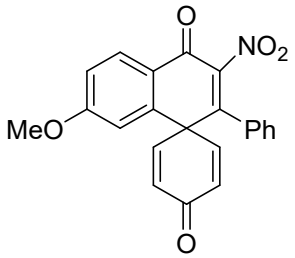
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3o** as a yellow solid (56.77 mg, 81% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.32 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.68 (td, *J* = 7.8, 1.5 Hz, 1H), 7.61 – 7.58 (m, 1H), 7.49 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.34 (d, *J* = 7.9 Hz, 1H), 7.21 (dd, *J* = 3.7, 1.1 Hz, 1H), 7.04 (dd, *J* = 5.1, 3.7 Hz, 1H), 6.77 – 6.75 (m, 2H), 6.58 – 6.53 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.1, 174.0, 149.4, 146.1, 140.7, 137.3, 134.8, 131.9, 130.9, 130.6, 130.2, 129.6, 129.3, 128.7, 128.2, 127.6, 50.2.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3p** as a yellow solid (55.67 mg, 78% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 8.0 Hz, 1H), 7.33 – 7.28 (m, 2H), 7.23 (t, *J* = 7.6 Hz, 2H), 7.09 – 7.05 (m, 2H), 7.01 (s, 1H), 6.73 – 6.68 (m, 2H), 6.33 (d, *J* = 10.0 Hz, 2H), 2.34 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.0, 174.0, 150.0, 146.8, 146.4, 146.2, 137.8, 131.2, 130.7, 130.28, 130.20, 128.9, 128.2, 128.1, 127.6, 127.0, 50.3, 21.9.

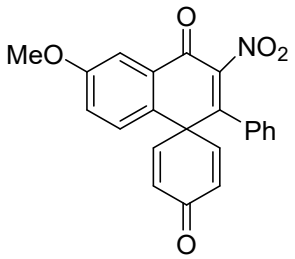
7'-Methoxy-3'-nitro-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3q)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 3:1) to afford the **3q** as a yellow solid (64.15 mg, 86% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.8 Hz, 1H), 7.30 (d, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 7.7 Hz, 2H), 7.09 – 7.06 (m, 2H), 7.03 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.74 – 6.70 (m, 2H), 6.65 (d, *J* = 2.4 Hz, 1H), 6.36 – 6.31 (m, 2H),

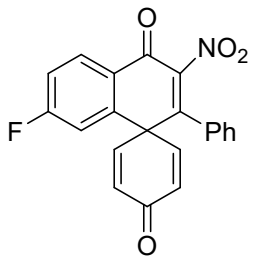
3.79 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 182.8, 172.1, 163.6, 149.0, 145.2, 145.1, 139.3, 130.1, 129.8, 129.2, 129.1, 127.1, 126.6, 121.6, 114.7, 112.2, 54.8, 49.3.

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



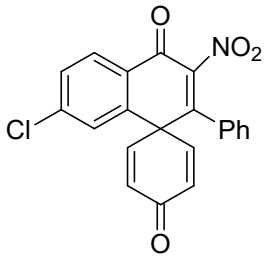
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 3:1) to afford the **3r** as a yellow solid (61.11 mg, 82% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (d, *J* = 1.3 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 7.7 Hz, 2H), 7.15 (s, 2H), 7.08 (d, *J* = 7.5 Hz, 2H), 6.69 – 6.65 (m, 2H), 6.32 (t, *J* = 6.4 Hz, 2H), 3.84 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.9, 174.2, 160.3, 149.9, 147.4, 146.1, 131.1, 130.7, 130.3, 130.2, 130.1, 129.8, 128.2, 127.5, 123.4, 109.7, 55.9, 50.0.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3s** as a yellow solid (54.20 mg, 75% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.37 (dd, *J* = 8.8, 5.7 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.35 – 7.29 (m, 3H), 7.15 (d, *J* = 7.5 Hz, 2H), 7.00 (dd, *J* = 9.0, 2.4 Hz, 1H), 6.79 (d, *J* = 10.0 Hz, 2H), 6.44 (d, *J* = 10.0 Hz, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.4, 173.1, 166.3 (d, *J* = 256.7 Hz), 149.7, 147.3, 145.3, 141.1 (d, *J* = 8.7 Hz), 131.6, 131.5 (d, *J* = 9.8 Hz), 130.5, 129.8, 128.3, 127.5, 126.0 (d, *J* = 2.1 Hz), 117.8 (d, *J* = 22.2 Hz), 115.5 (d, *J* = 23.8 Hz), 50.3.

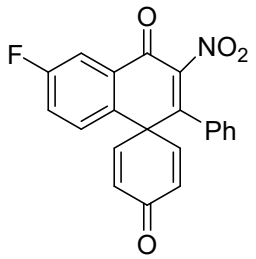
7'-Chloro-3'-nitro-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3t)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3t** as a yellow solid (53.53 mg, 71% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.27 (d, *J* = 8.5 Hz, 1H), 7.57 (dd, *J* = 8.5, 2.0

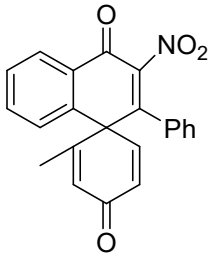
Hz, 1H), 7.42 – 7.38 (m, 1H), 7.35 – 7.31 (m, 2H), 7.28 (d, *J* = 1.9 Hz, 1H), 7.14 (dd, *J* = 5.2, 3.3 Hz, 2H), 6.82 – 6.75 (m, 2H), 6.48 – 6.43 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.4, 173.4, 149.7, 147.3, 145.1, 141.8, 139.6, 131.7, 130.5, 130.3, 129.8, 128.7, 128.3, 127.8, 127.5, 50.1.

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



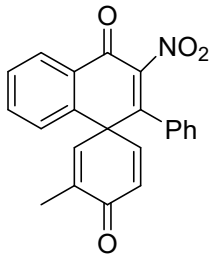
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3u** as a yellow solid (50.59 mg, 70% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (dd, *J* = 8.3, 2.8 Hz, 1H), 7.40 (ddd, *J* = 8.7, 5.1, 1.9 Hz, 2H), 7.35 – 7.30 (m, 3H), 7.18 – 7.11 (m, 2H), 6.80 – 6.72 (m, 2H), 6.48 – 6.36 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.5, 173.4, 162.8 (d, *J* = 256.7 Hz), 149.6, 147.9, 145.5, 133.7, 131.5 (d, *J* = 7.6 Hz), 131.3 (d, *J* = 8.2 Hz), 130.5, 129.8, 128.3, 127.4, 122.7 (d, *J* = 22.8 Hz), 114.2 (d, *J* = 23.3 Hz), 50.1.

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



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3v** as a yellow solid (54.41 mg, 72% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.35 (dd, *J* = 7.9, 1.0 Hz, 1H), 7.71 – 7.67 (m, 1H), 7.61 (t, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.8 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 7.8 Hz, 2H), 6.75 (d, *J* = 9.8 Hz, 1H), 6.41 (dd, *J* = 9.8, 1.4 Hz, 1H), 6.33 (s, 1H), 1.80 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.7, 174.3, 154.9, 151.0, 147.4, 146.0, 139.0, 135.1, 131.4, 130.6, 130.3, 130.0, 129.9, 129.5, 128.4, 128.1, 127.9, 127.1, 53.6, 20.2.

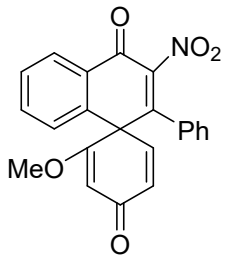
3-Methyl-3'-nitro-2'-phenyl-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3w)



The product purified by flash column chromatography on silica gel (PE/ AcOEt = 7:1) to afford the **3w** as a yellow solid (49.24 mg, 69% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.36 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.69 (td, *J* = 7.7,

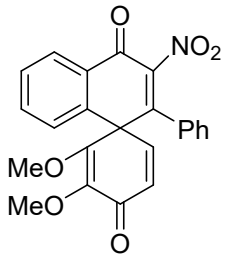
1.4 Hz, 1H), 7.63 – 7.60 (m, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.8 Hz, 2H), 7.22 (d, *J* = 7.7 Hz, 1H), 7.15 – 7.13 (m, 2H), 6.74 (d, *J* = 9.8 Hz, 1H), 6.41 (dd, *J* = 9.8, 1.5 Hz, 1H), 6.34 – 6.32 (m, 1H), 1.80 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 184.7, 174.3, 154.9, 151.0, 147.3, 146.0, 139.0, 135.1, 131.4, 130.6, 130.3, 130.0, 129.9, 129.5, 128.4, 128.1, 127.9, 127.1, 53.6, 20.2.

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



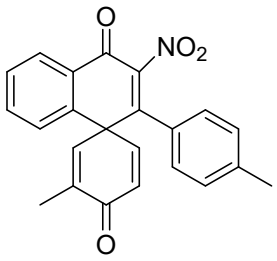
The product purified by flash column chromatography on silica gel (PE/ AcOEt = 3:1) to afford the **3x** as a yellow solid (63.51 mg, 85% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.67 (td, *J* = 7.9, 1.4 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.39 – 7.35 (m, 1H), 7.35 – 7.29 (m, 3H), 7.16 – 7.10 (m, 2H), 6.79 (dd, *J* = 9.8, 2.8 Hz, 1H), 6.43 (d, *J* = 9.8 Hz, 1H), 5.72 (d, *J* = 2.7 Hz, 1H), 3.65 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 179.1, 174.3, 152.9, 149.5, 148.4, 146.0, 139.4, 134.8, 131.1, 130.2, 130.0, 129.3, 129.1, 128.5, 128.17, 128.13, 127.6, 113.9, 55.4, 50.7.

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



The product purified by flash column chromatography on silica gel (PE/AcOEt = 1:1) to afford the **3y** as a yellow solid (63.68 mg, 79% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.31 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.67 (td, *J* = 7.8, 1.5 Hz, 1H), 7.62 – 7.57 (m, 1H), 7.42 – 7.38 (m, 1H), 7.35 – 7.31 (m, 3H), 7.22 – 7.18 (m, 2H), 6.45 (d, *J* = 9.7 Hz, 1H), 6.35 (d, *J* = 9.7 Hz, 1H), 3.88 (s, 3H), 3.47 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.5, 174.5, 158.5, 150.5, 147.6, 140.9, 140.1, 138.8, 134.7, 130.3, 130.1, 129.99, 129.97, 129.4, 128.3, 128.1, 127.3, 127.0, 61.4, 60.8, 54.9.

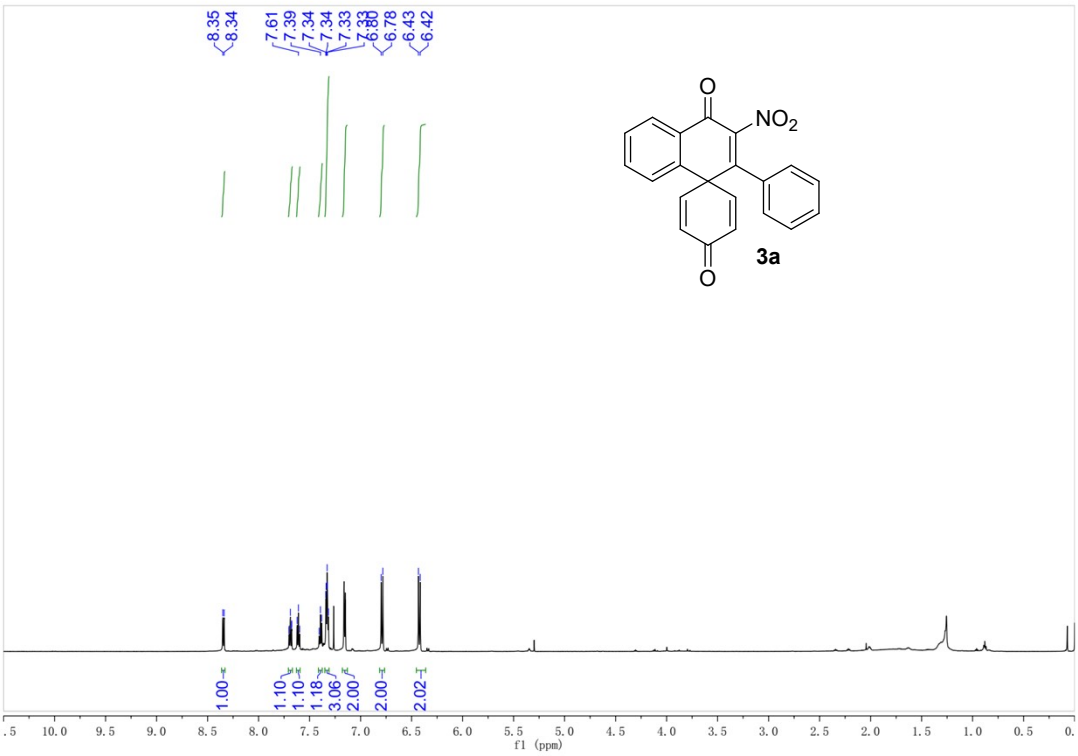
3-Methyl-3'-nitro-2'-(p-tolyl)-4'*H*-spiro[cyclohexane-1,1'-naphthalene]-2,5-diene-4,4'-dione (3z)

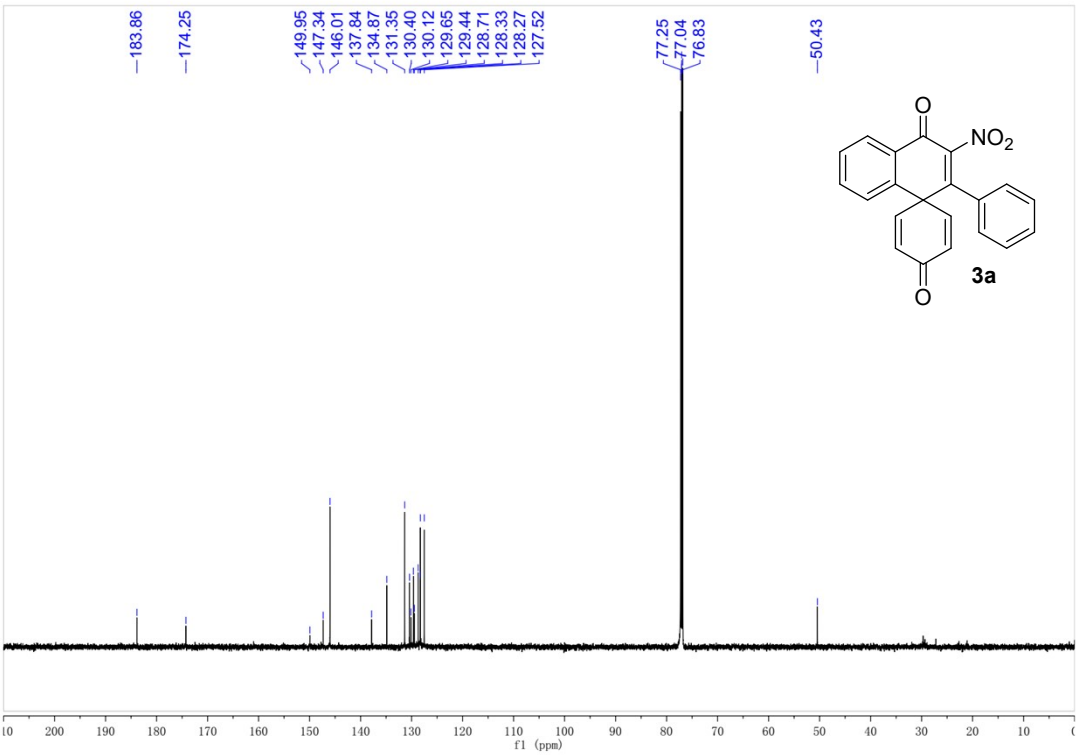


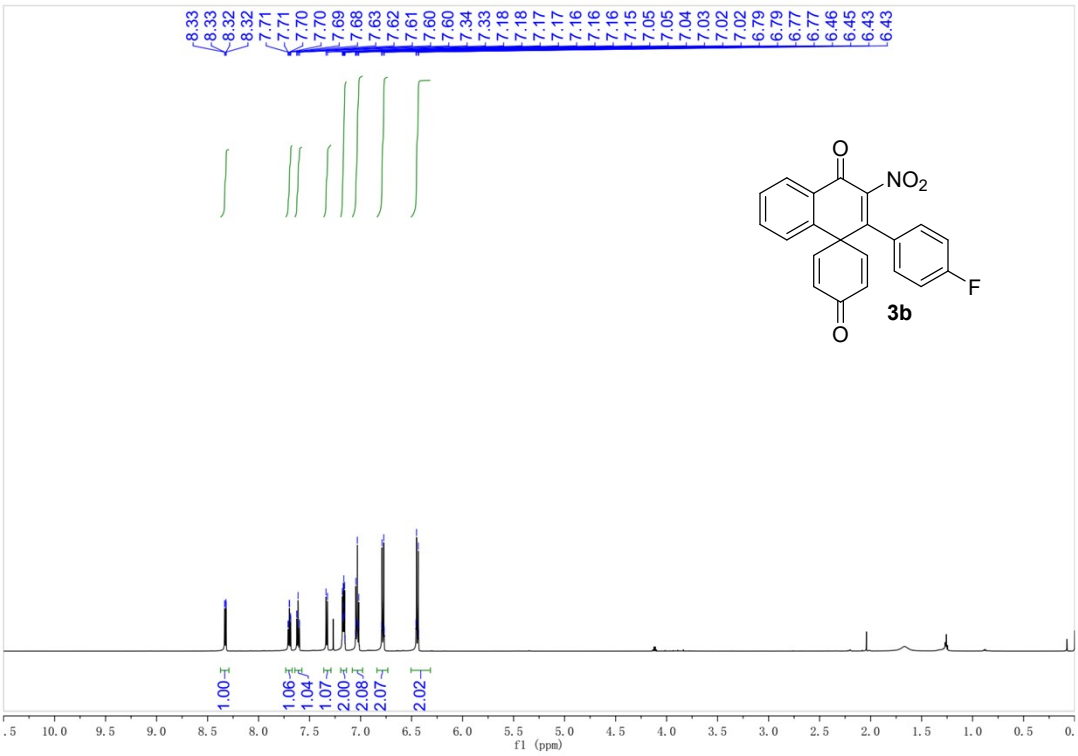
The product purified by flash column chromatography on silica gel (PE/AcOEt = 7:1) to afford the **3z** as a yellow solid (57.31 mg, 77% yield). ^{1}H

NMR (600 MHz, $CDCl_3$) δ 8.24 – 8.16 (m, 1H), 7.61 – 7.54 (m, 1H), 7.48 (dd, *J* = 11.1, 4.1 Hz, 1H), 7.21 (d, *J* = 7.9 Hz, 1H), 7.01 (d, *J* = 7.9 Hz, 2H), 6.92 (d, *J* = 8.1 Hz, 2H), 6.67 (dd, *J* = 9.8, 3.1 Hz, 1H), 6.48 (dd, *J* = 2.9, 1.4 Hz, 1H), 6.30 (d, *J* = 9.8 Hz, 1H), 2.22 (s, 3H), 1.81 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 183.7, 173.4, 148.8, 147.4, 144.7, 140.2, 139.3, 137.6, 137.4, 133.6, 130.0, 128.3, 128.2, 127.7, 127.6, 127.0, 126.36, 126.32, 49.7, 20.2, 14.7.

5. ^{1}H NMR and ^{13}C NMR of the products



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7.03
7.02
7.02
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6.45
6.43
6.43
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NO2
F
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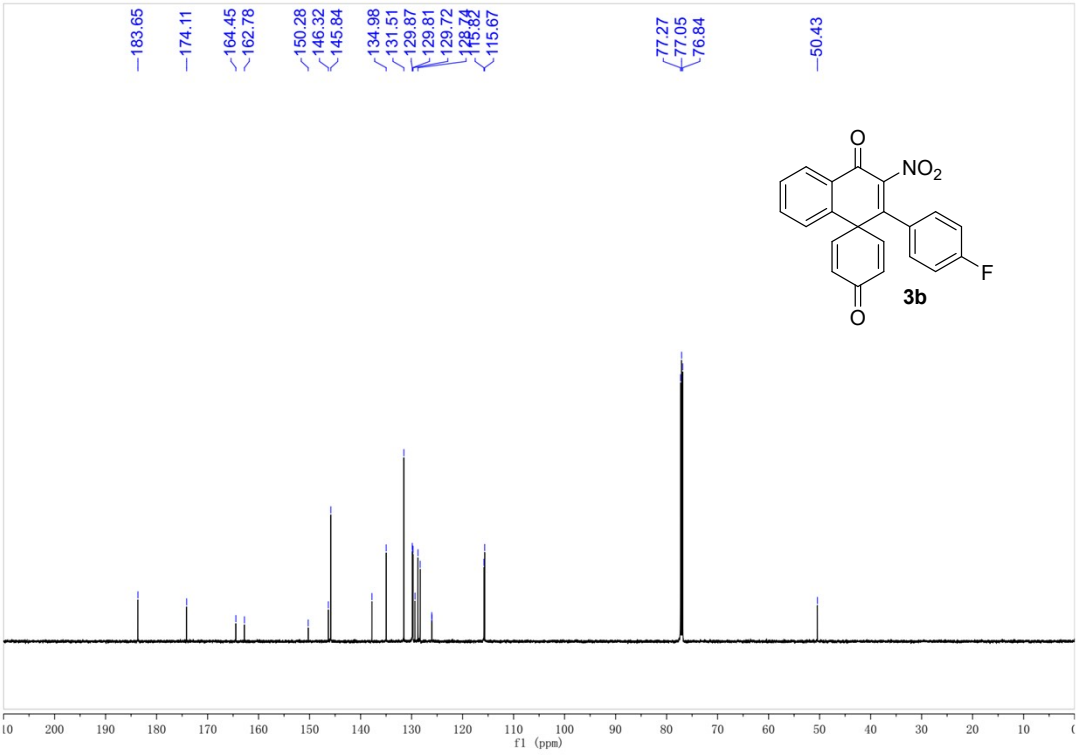
1.06
1.04
1.07
2.00
2.08
2.07
2.02
f1 (ppm)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O
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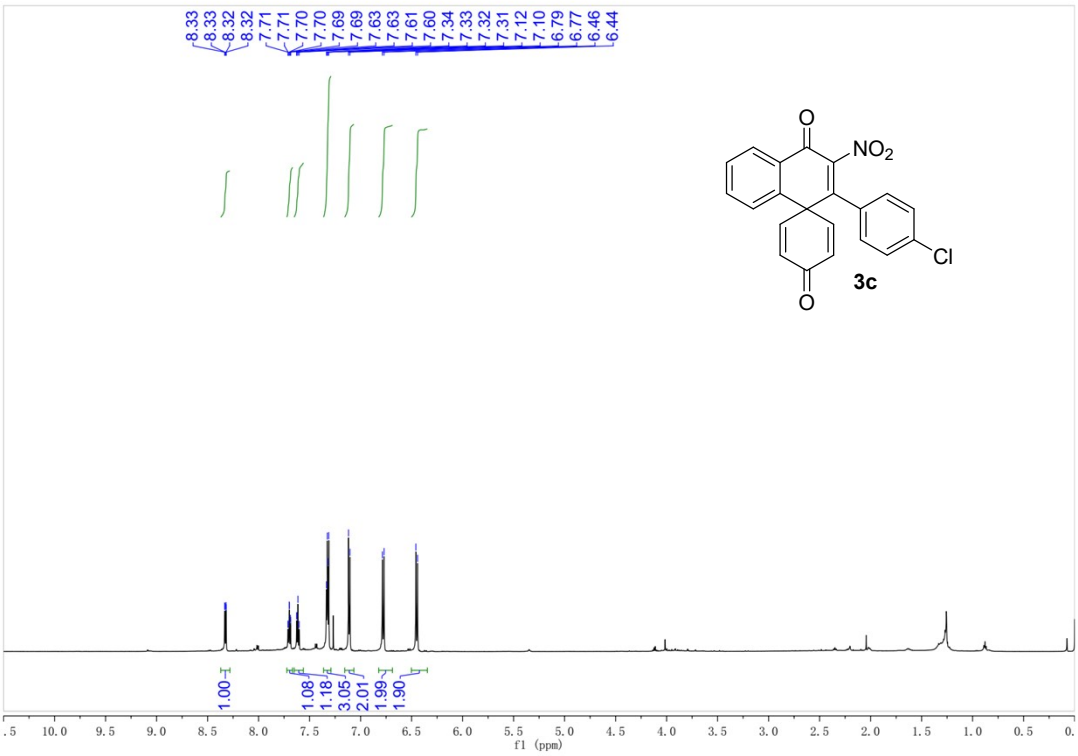
f1 (ppm)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6.46
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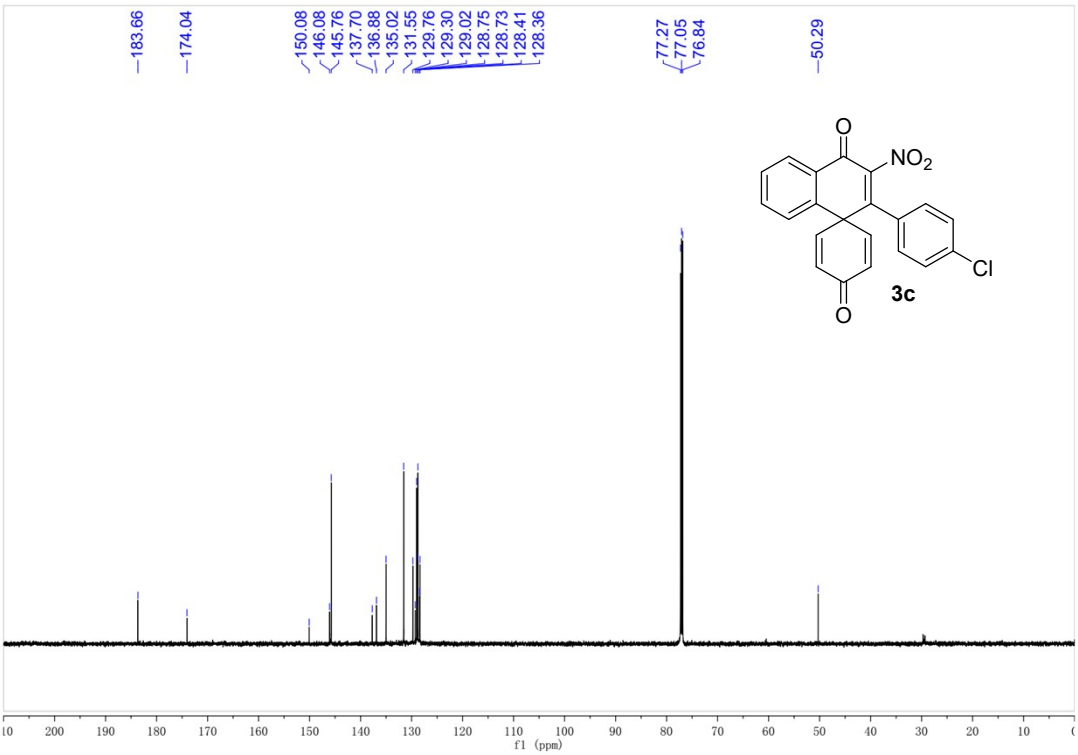
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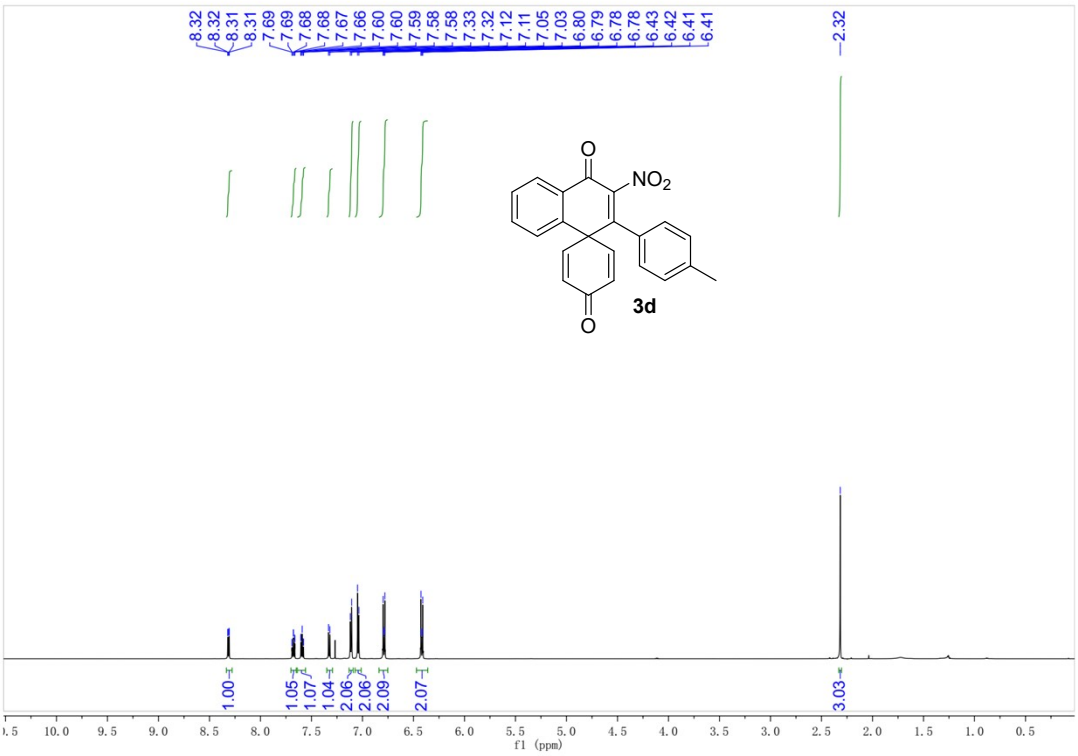
Cl
3c
O
f1 (ppm)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7.58
7.58
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7.05
7.03
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6.79
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6.78
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6.42
6.41
6.41
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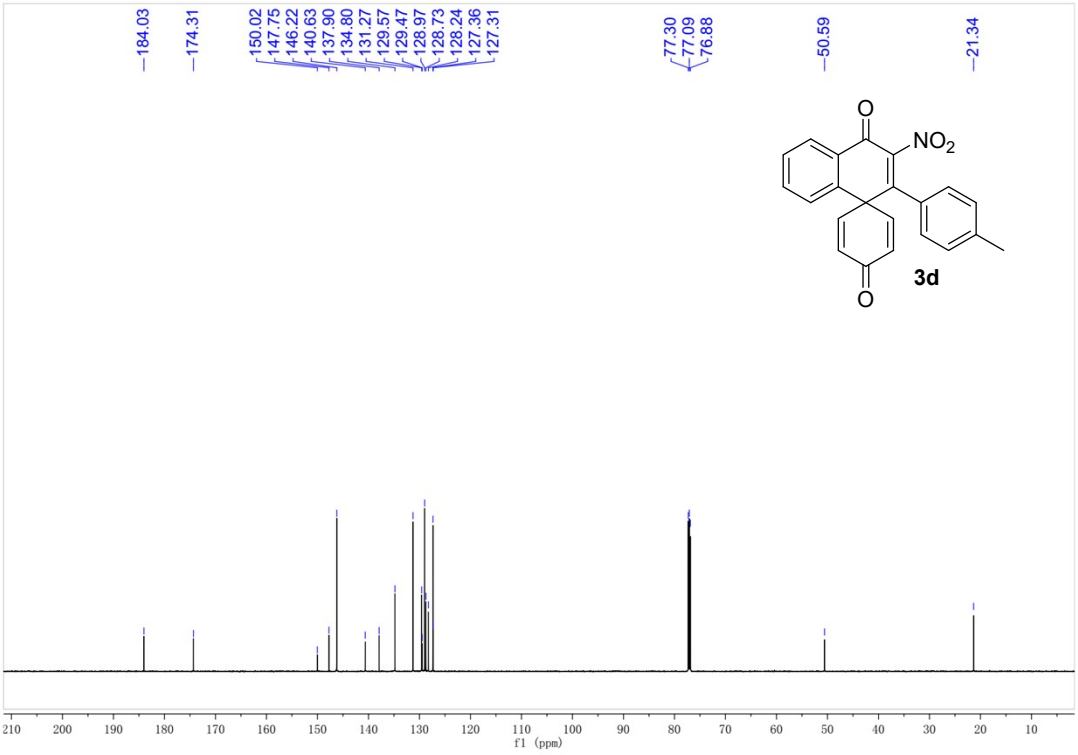
f1 (ppm)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76.88
50.59
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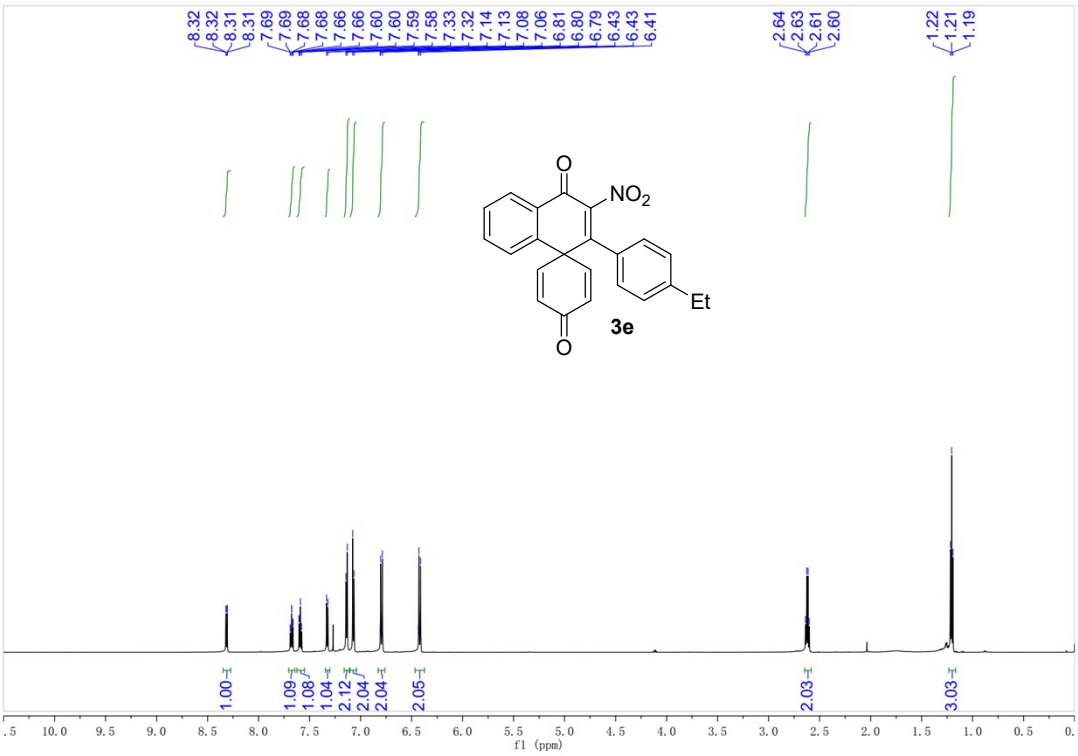
3d
O
f1 (ppm)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7.14
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7.08
7.06
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6.79
6.43
6.43
6.41
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2.63
2.61
2.60
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1.21
1.19
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NO2
Et
O
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3.03
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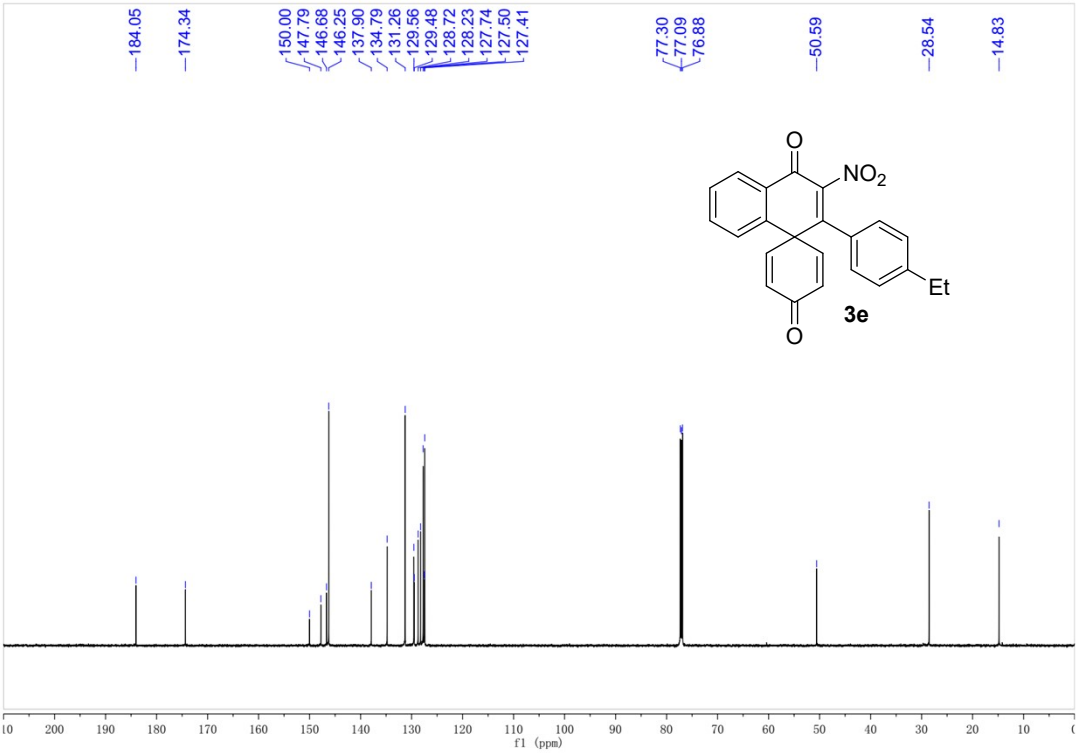
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127.50
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76.88
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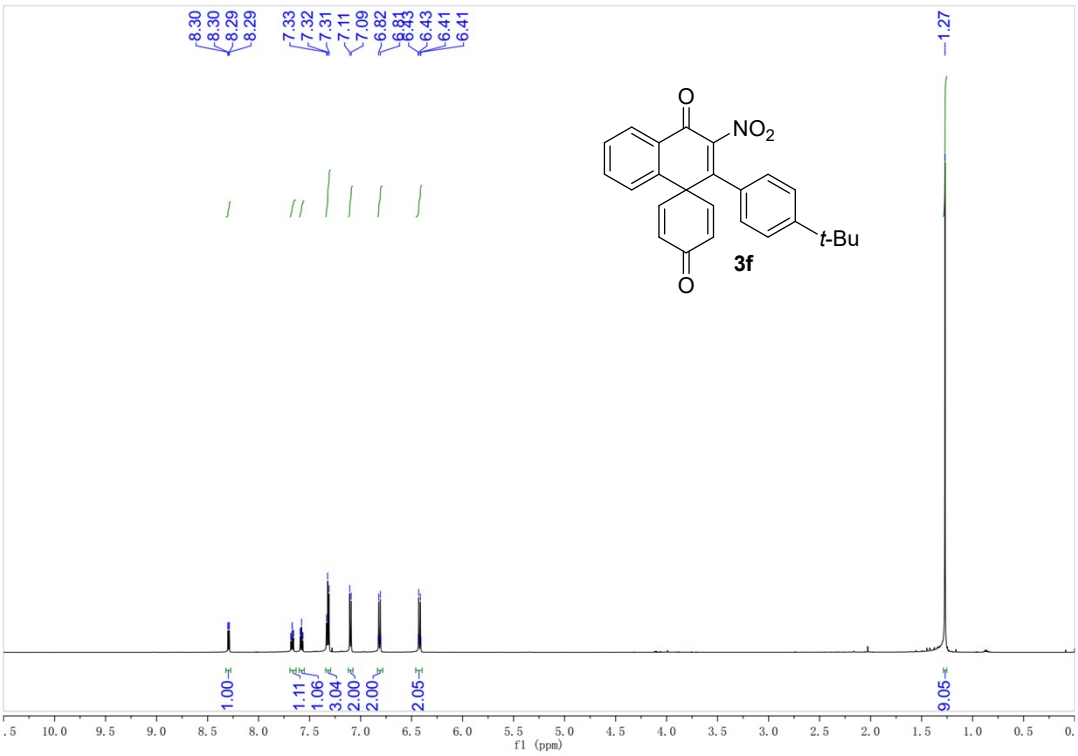
O
NO2
Et
O
3e
f1 (ppm)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6.43
6.41
6.41
1.27
O
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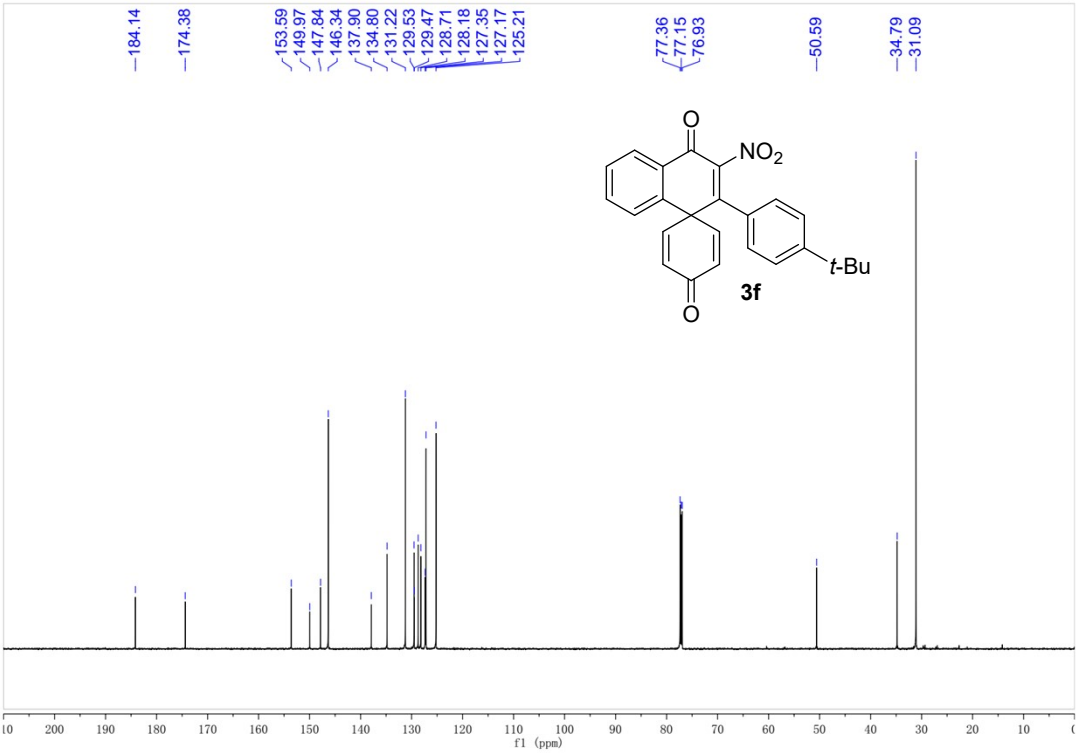
f1 (ppm)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127.17
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31.09
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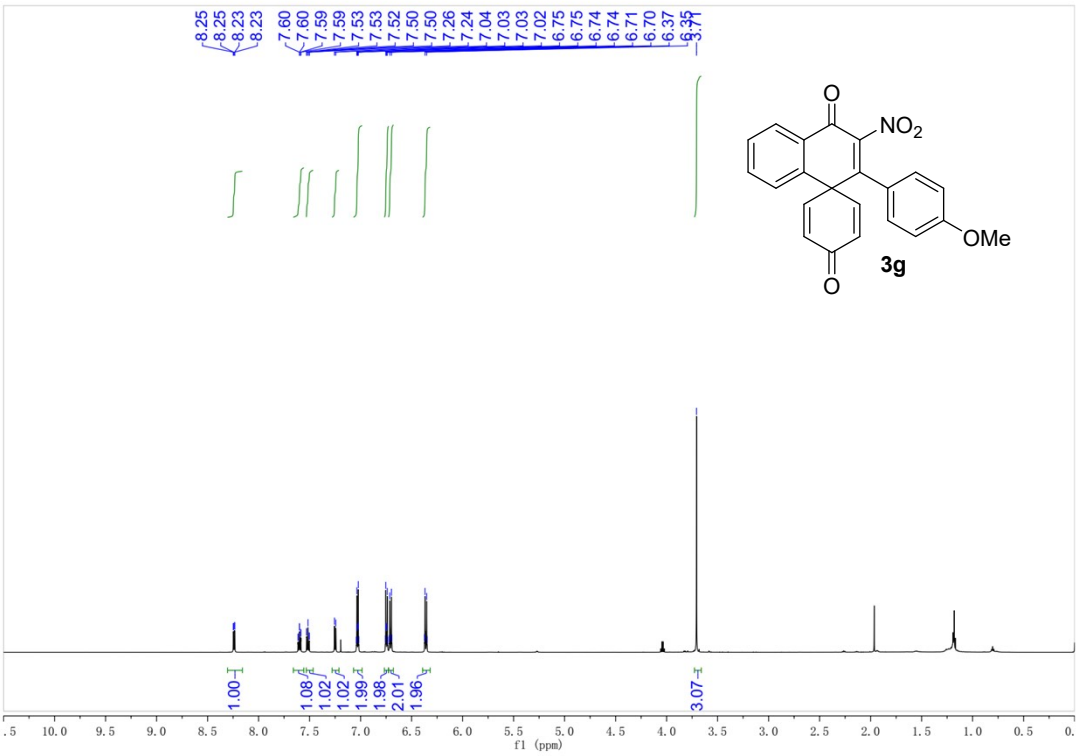
NO2
t-Bu
3f
O
f1 (ppm)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6.35
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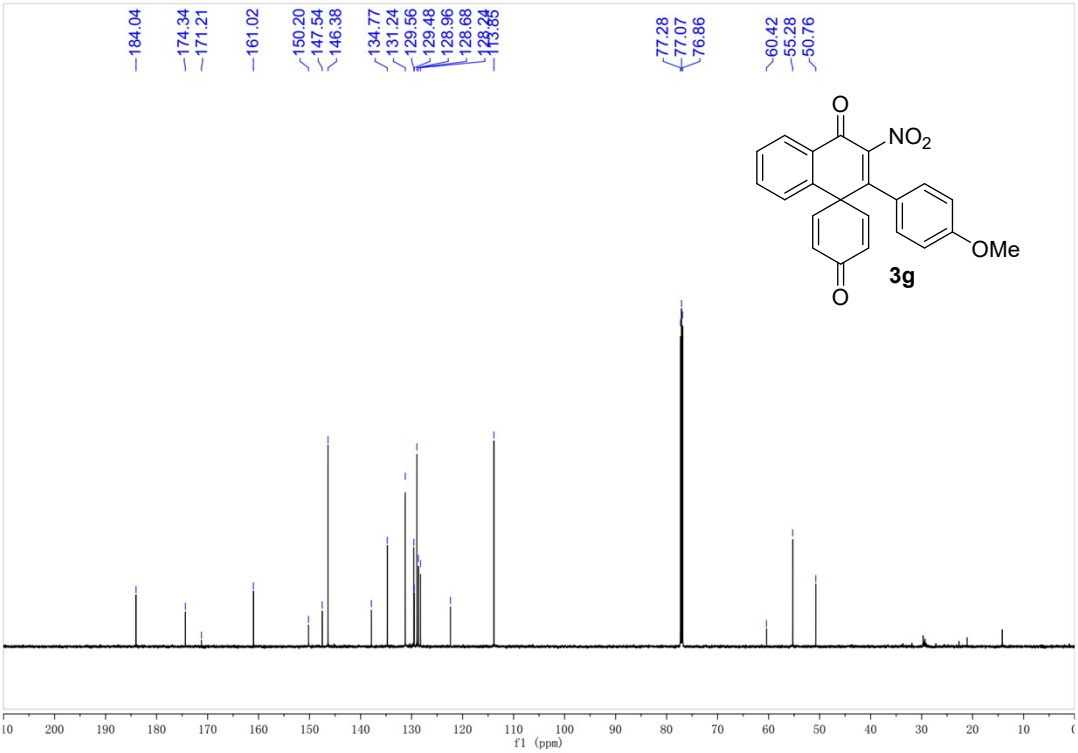
3.07
f1 (ppm)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128.24
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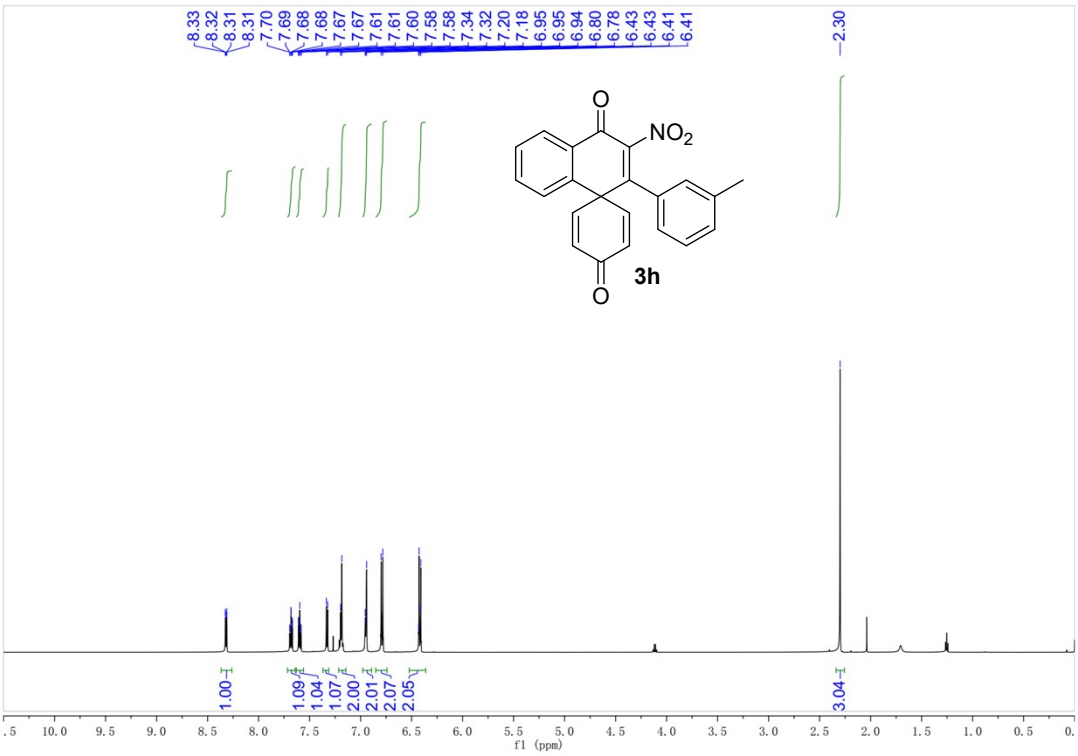
OMe
3g
O
f1 (ppm)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7.20
7.18
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6.95
6.94
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6.78
6.43
6.43
6.41
6.41
2.30
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1.04
1.07
2.00
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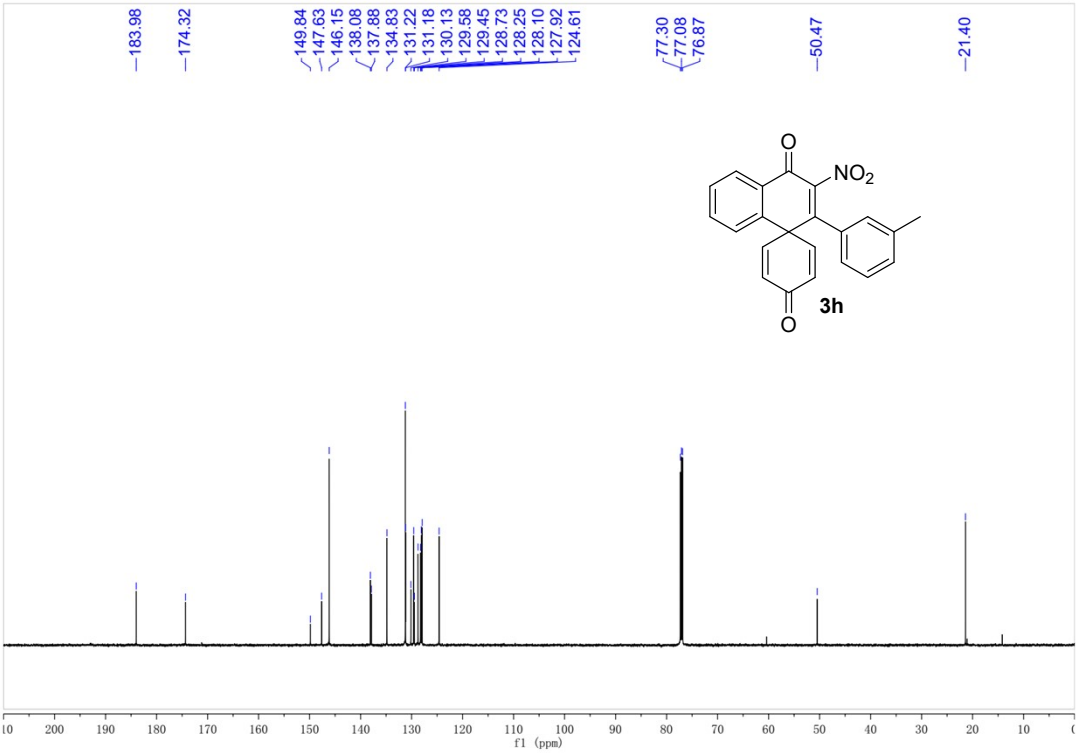
2.07
2.05
3.04
f1 (ppm)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128.25
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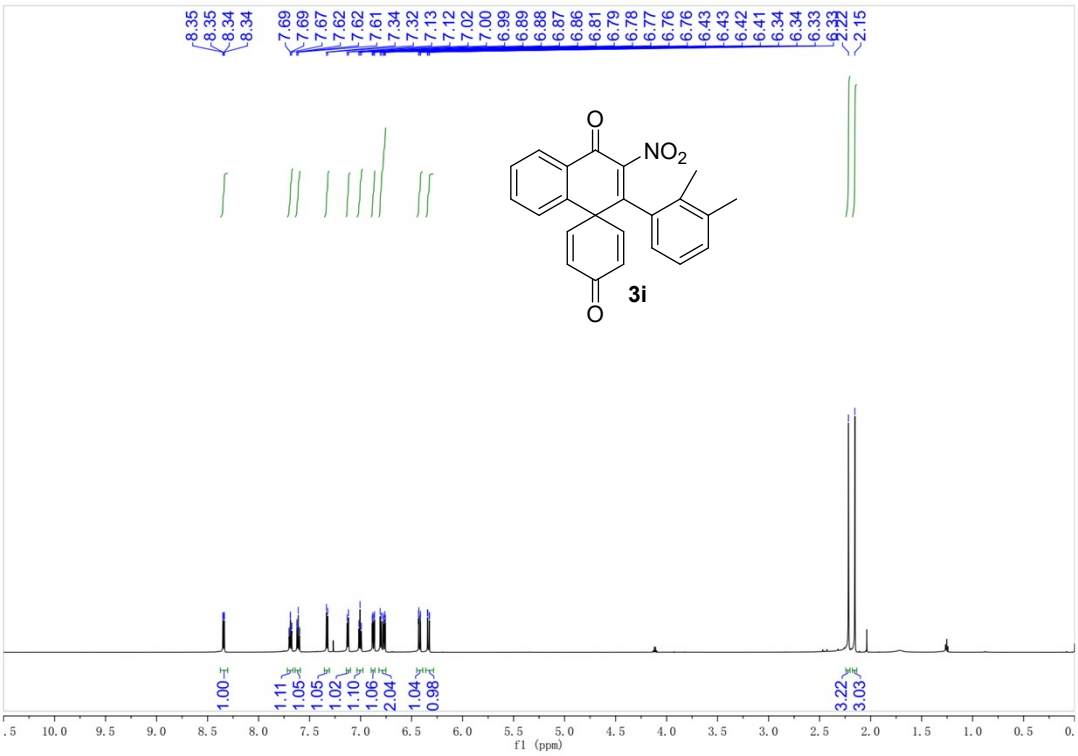
O
3h
f1 (ppm)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3i
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3.03
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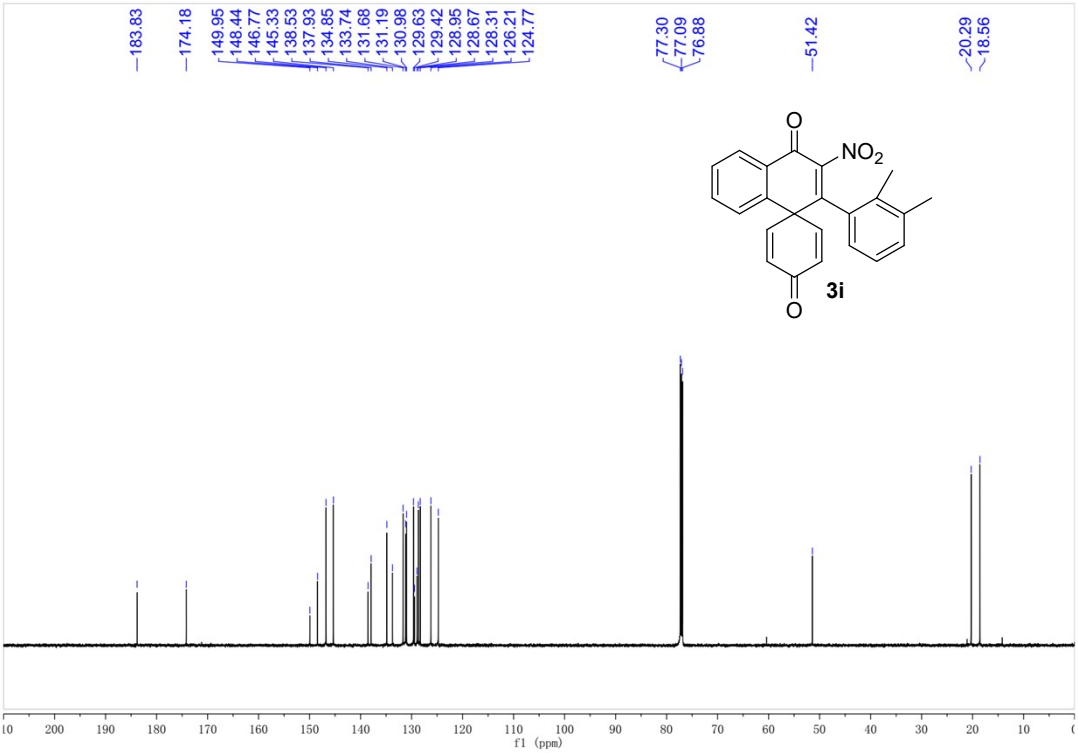
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128.31
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18.56
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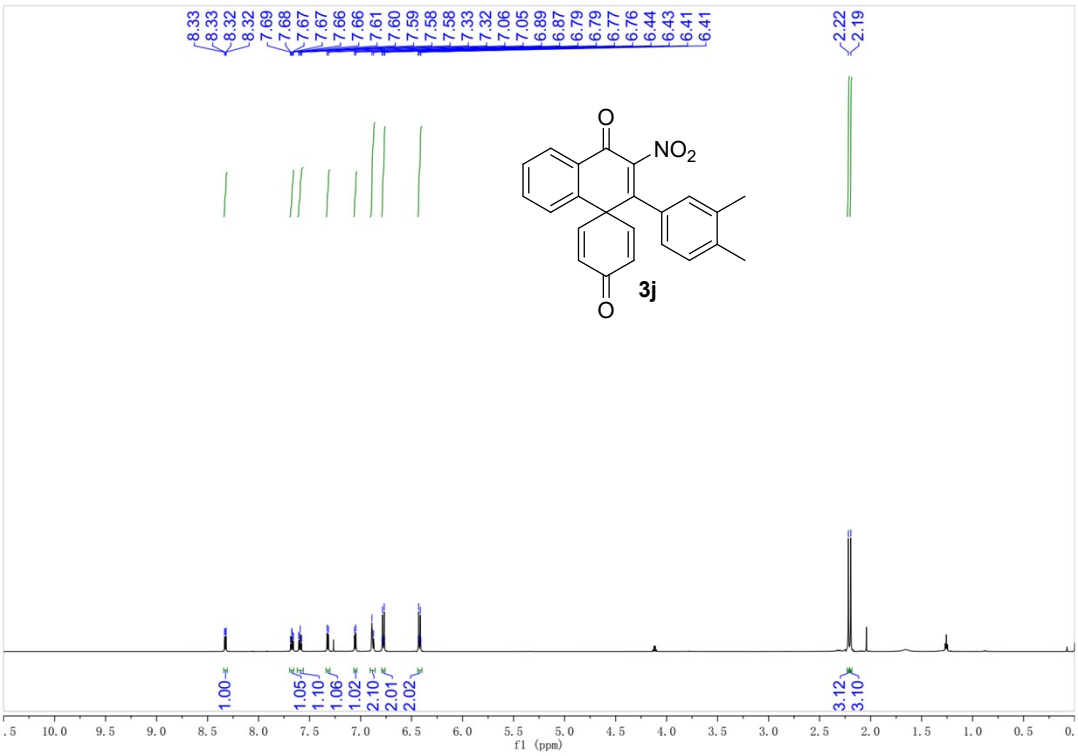
O
3i
f1 (ppm)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7.58
7.58
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7.32
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6.77
6.76
6.44
6.43
6.41
6.41
2.22
2.19
O
NO2
3j
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1.02
2.10
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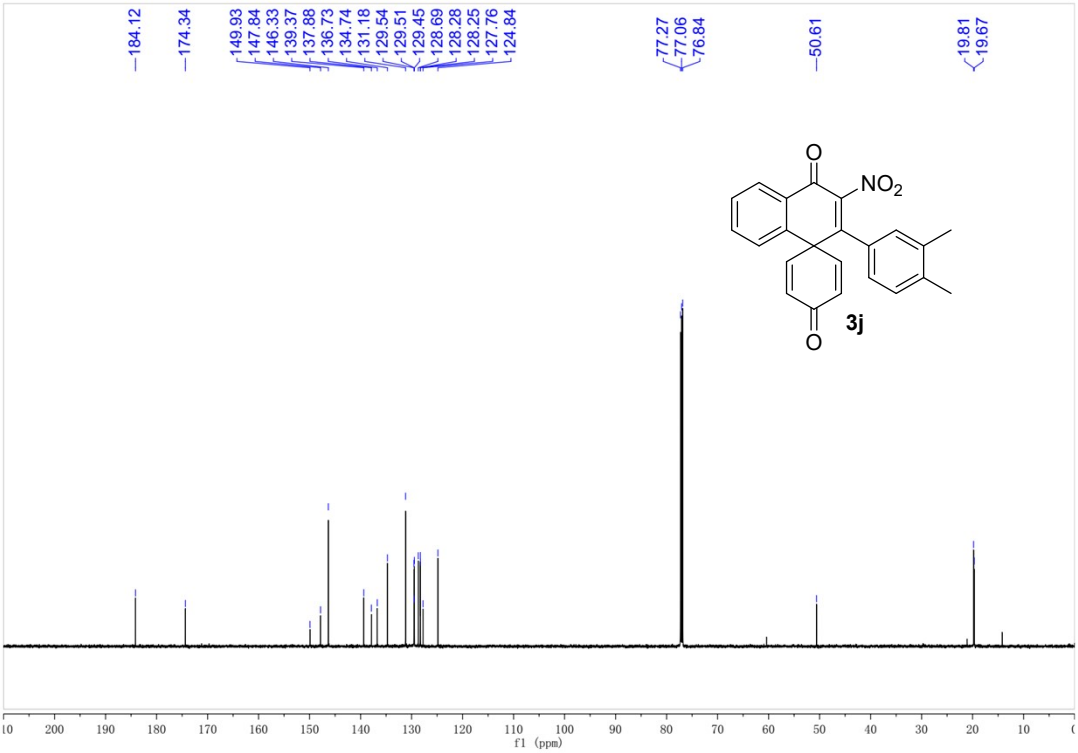
2.02
3.12
3.10
f1 (ppm)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128.25
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19.67
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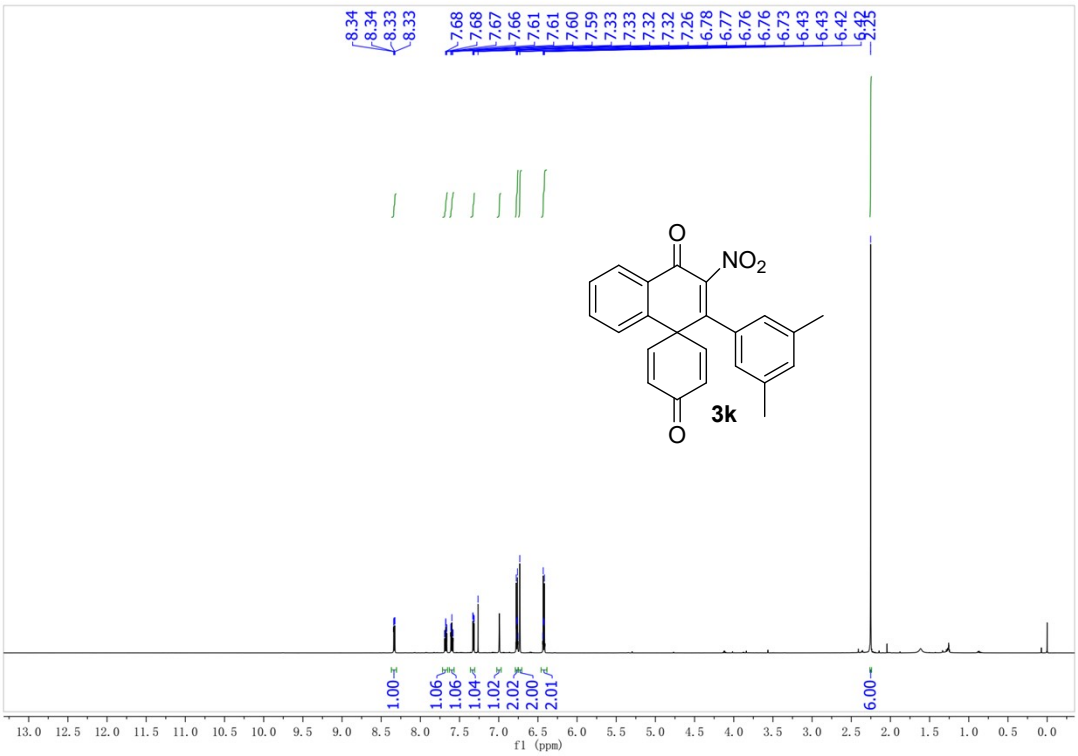
NO2
3j
O
f1 (ppm)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7.32
7.32
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6.77
6.76
6.76
6.73
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6.43
6.42
6.42
2.25
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2.00
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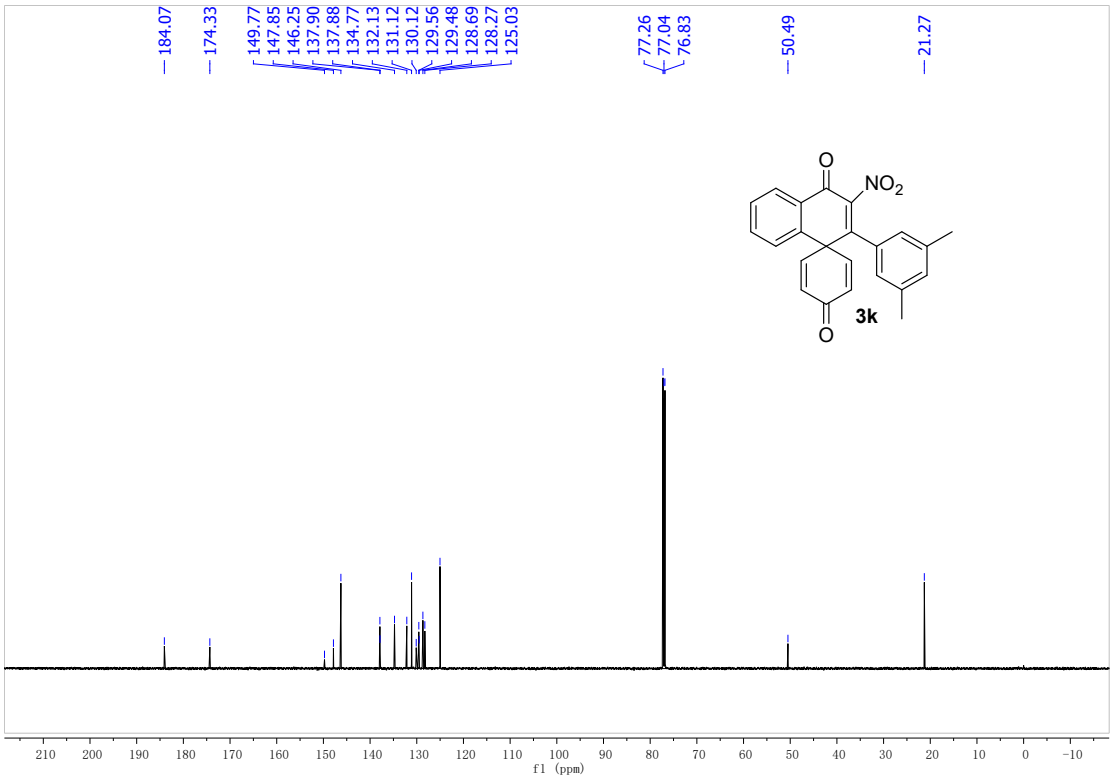
6.00
f1 (ppm)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77.04
76.83
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21.27
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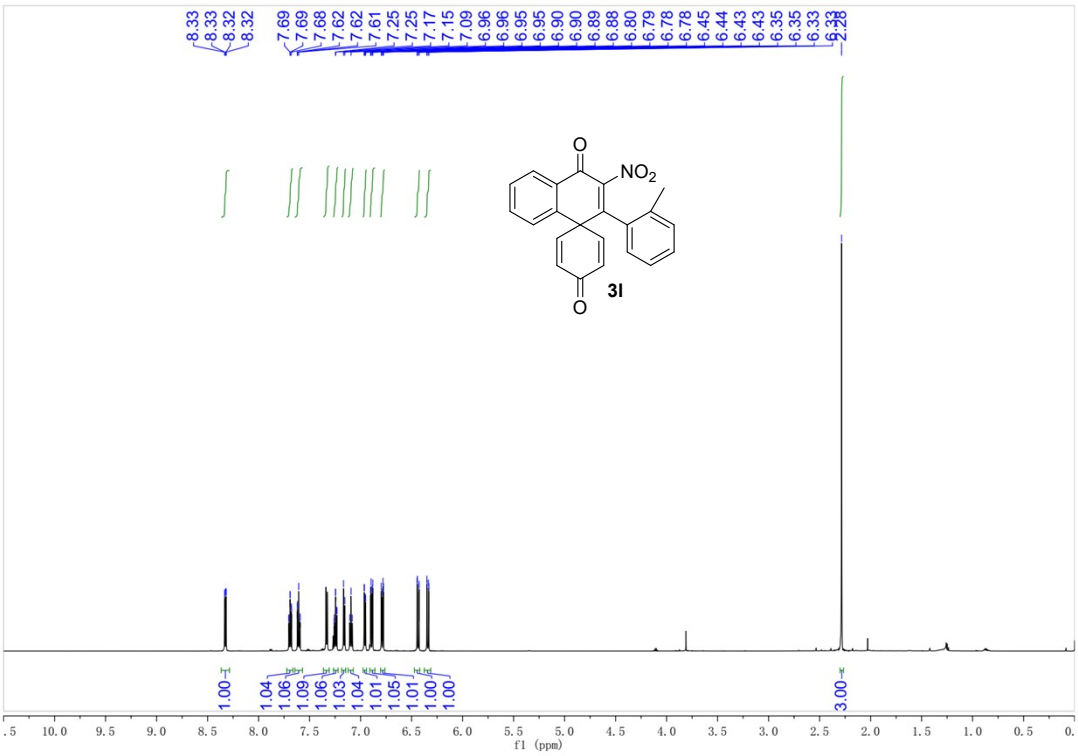
O
3k
f1 (ppm)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6.95
6.95
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6.80
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6.78
6.78
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6.35
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2.28
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O
3l
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1.00
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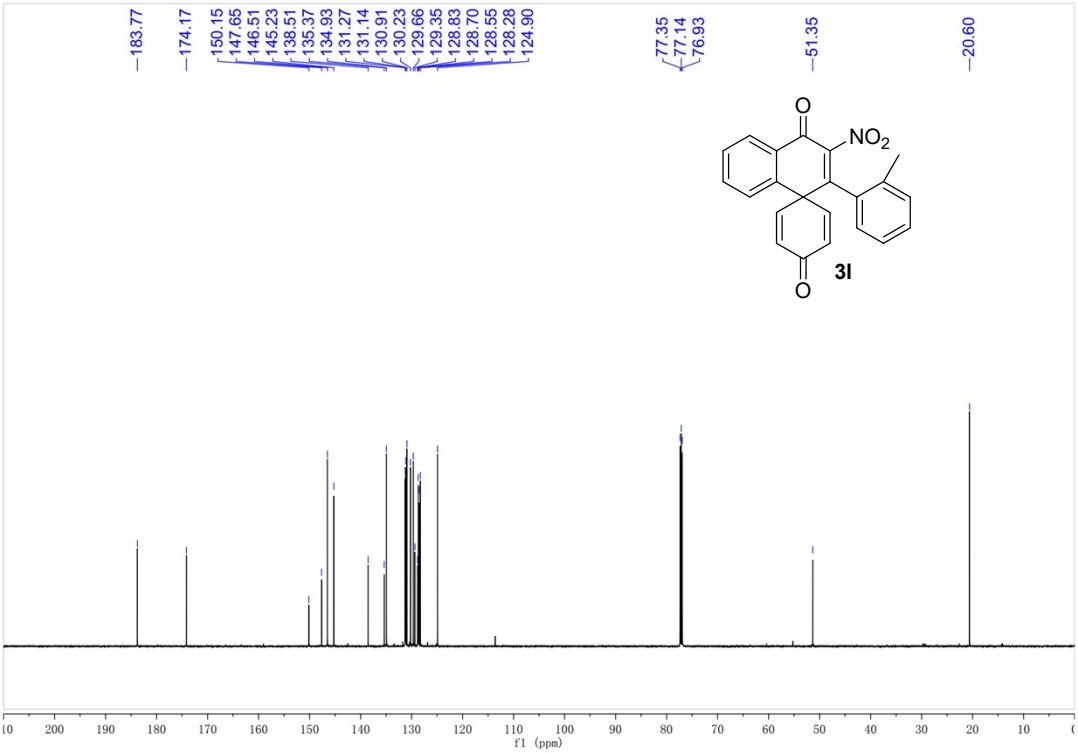
3.00
f1 (ppm)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128.70
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76.93
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20.60
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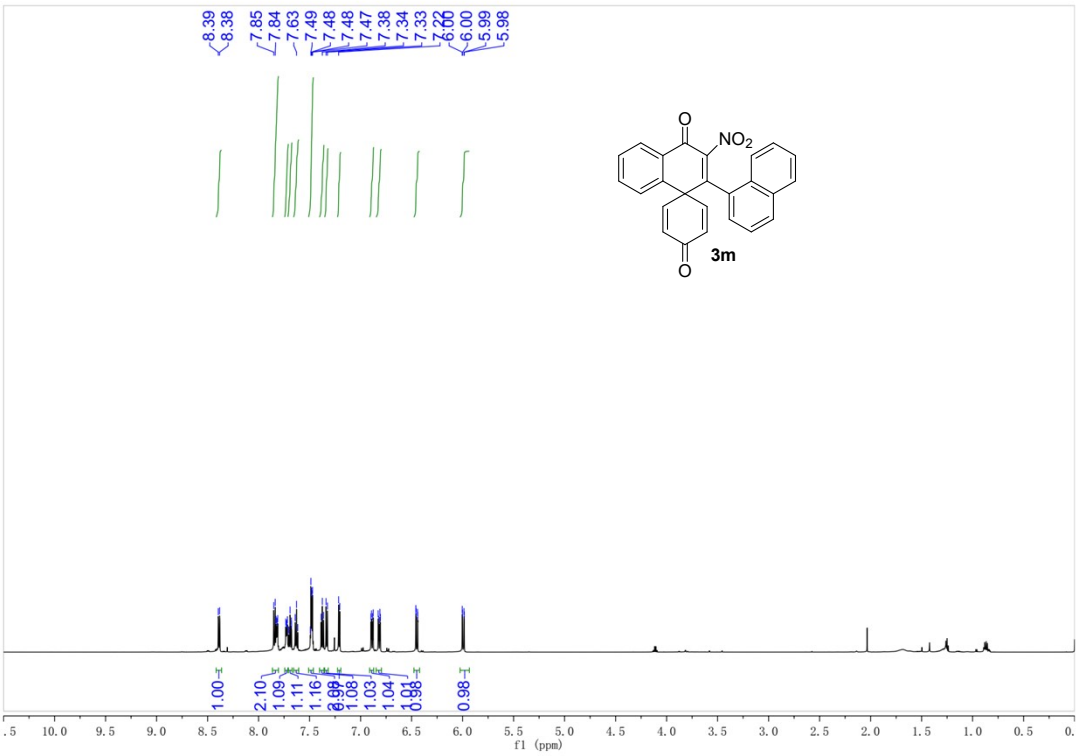
3l
f1 (ppm)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6.00
5.99
5.98
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3m
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1.04
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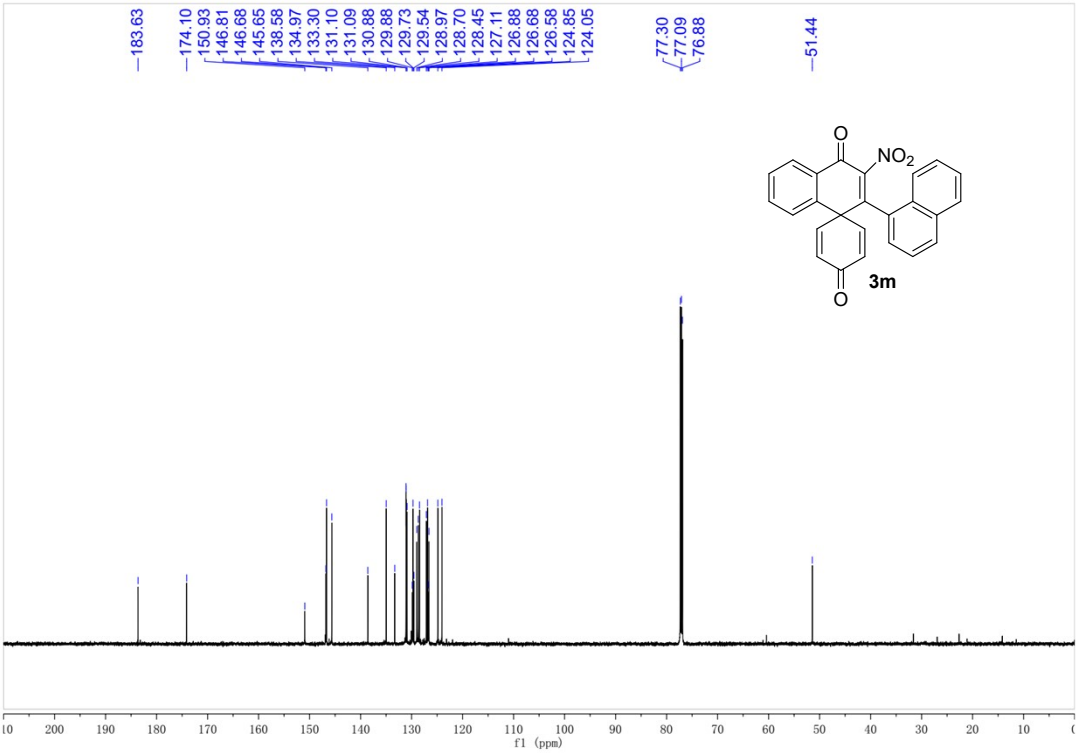
0.98
f1 (ppm)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128.70
128.45
127.11
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76.88
51.44
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O
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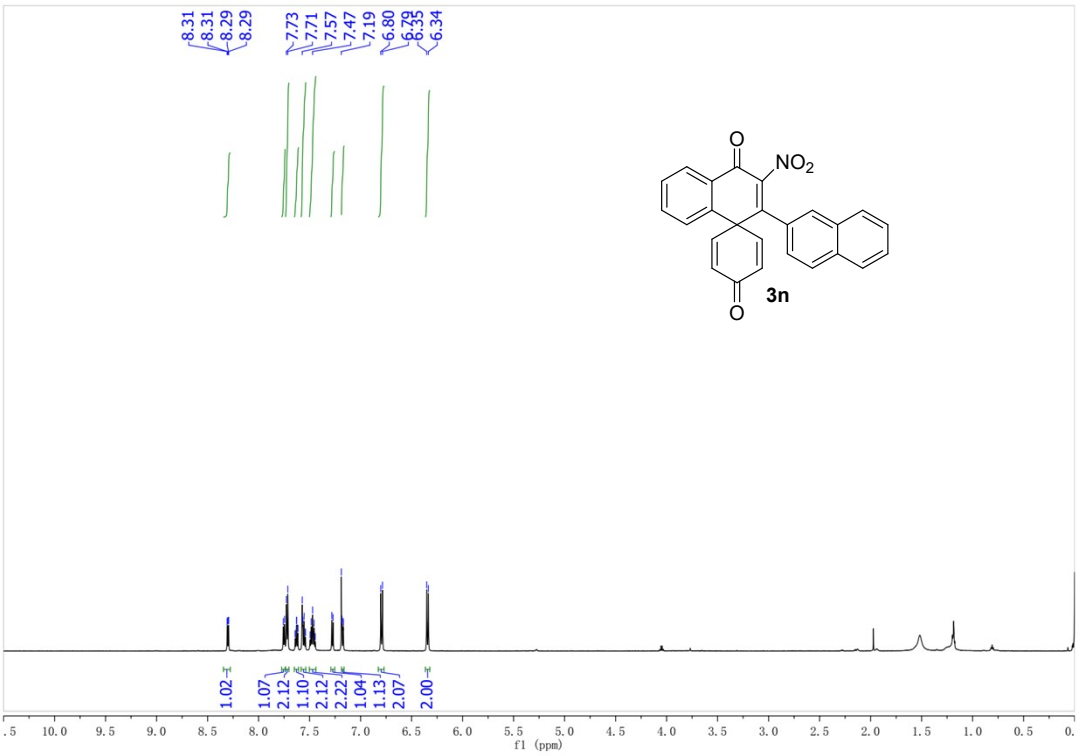
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6.34
O
NO2
O
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1.13
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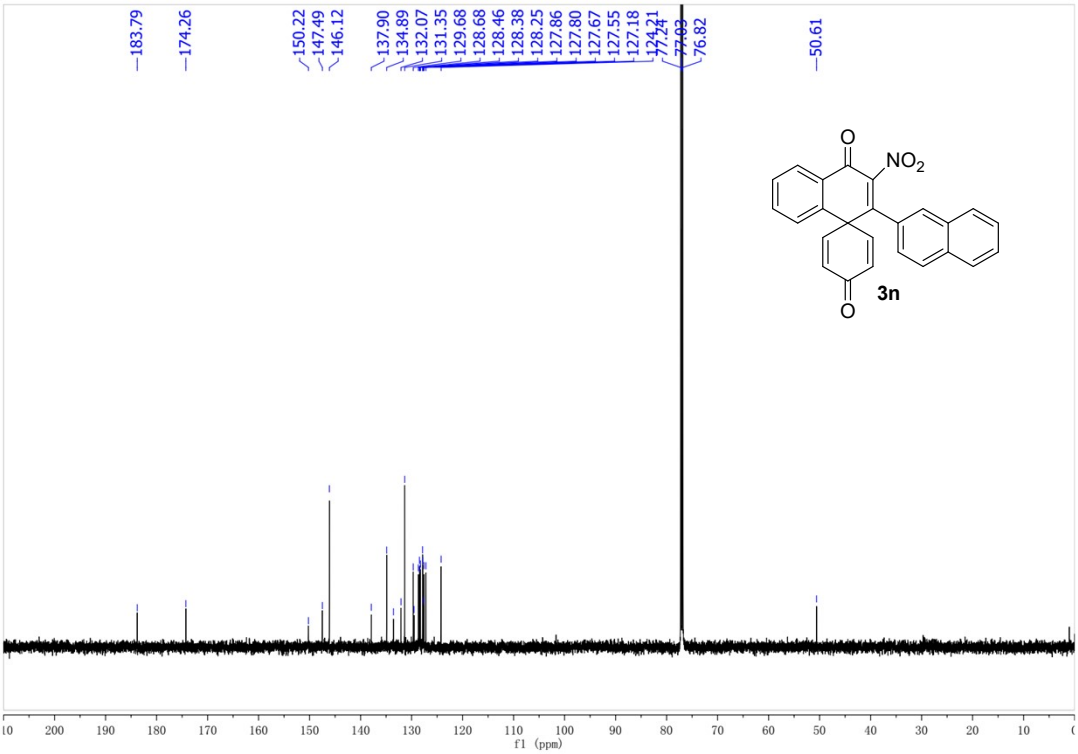
2.00
f1 (ppm)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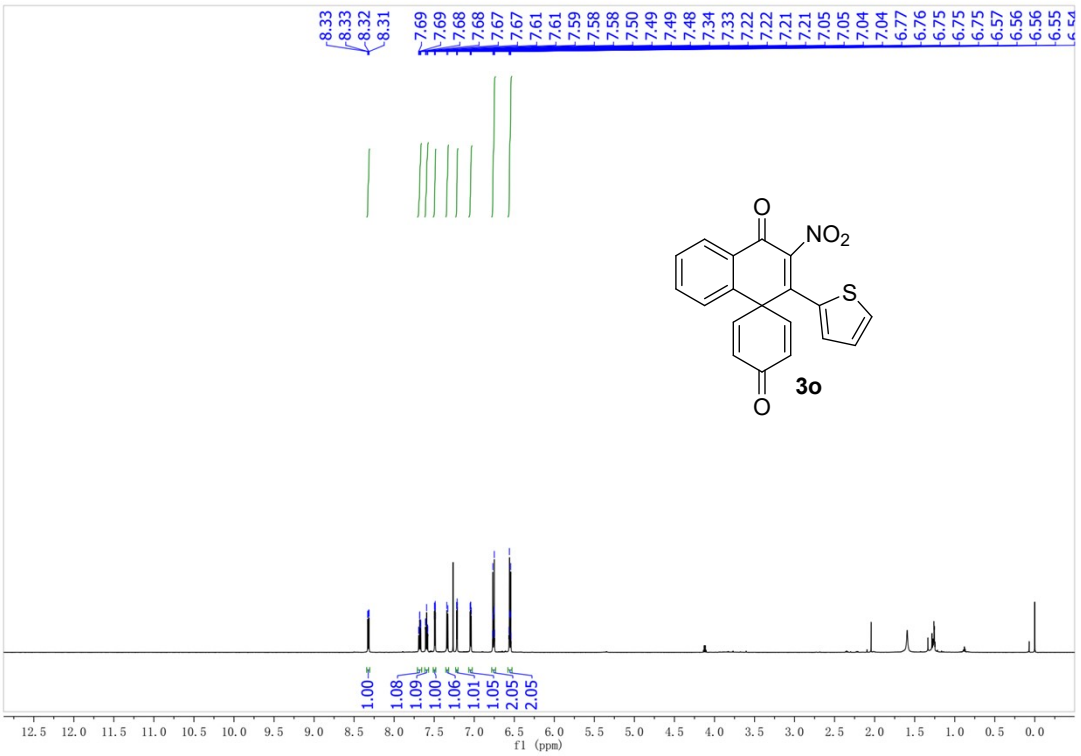
3n
f1 (ppm)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6.56
6.56
6.55
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S
O
3o
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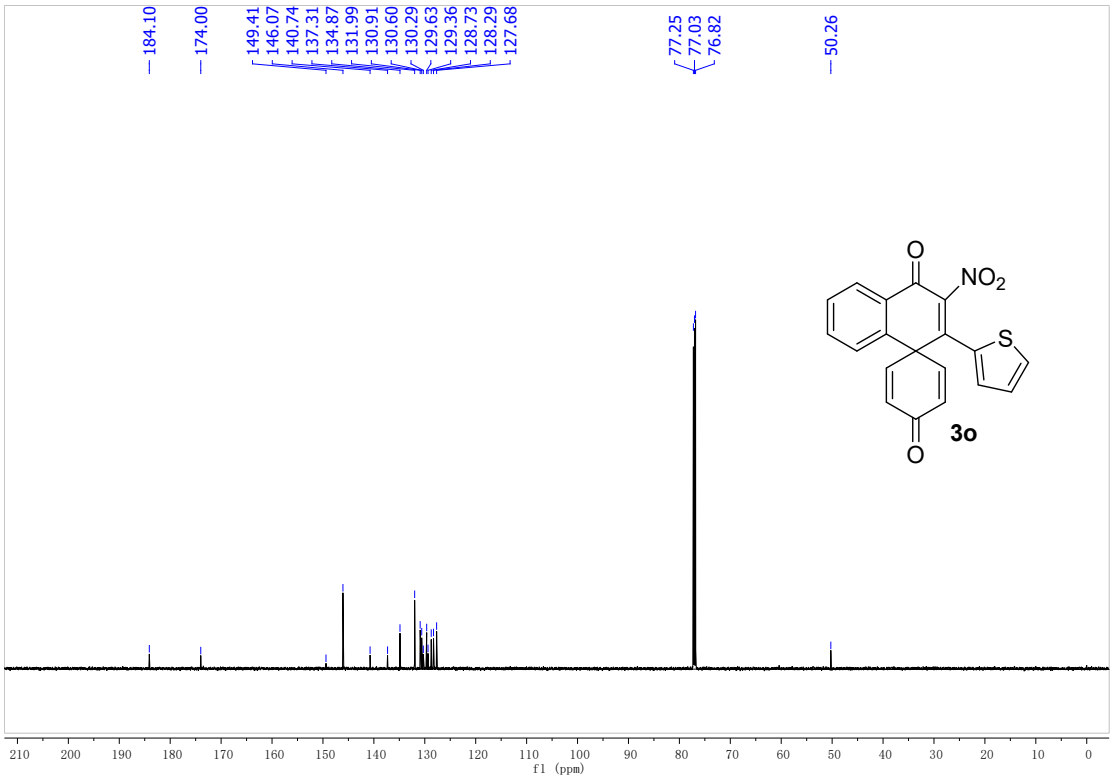
1.08
1.09
1.00
1.06
1.01
1.05
2.05
2.05
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.36
128.73
128.29
127.68
77.25
77.03
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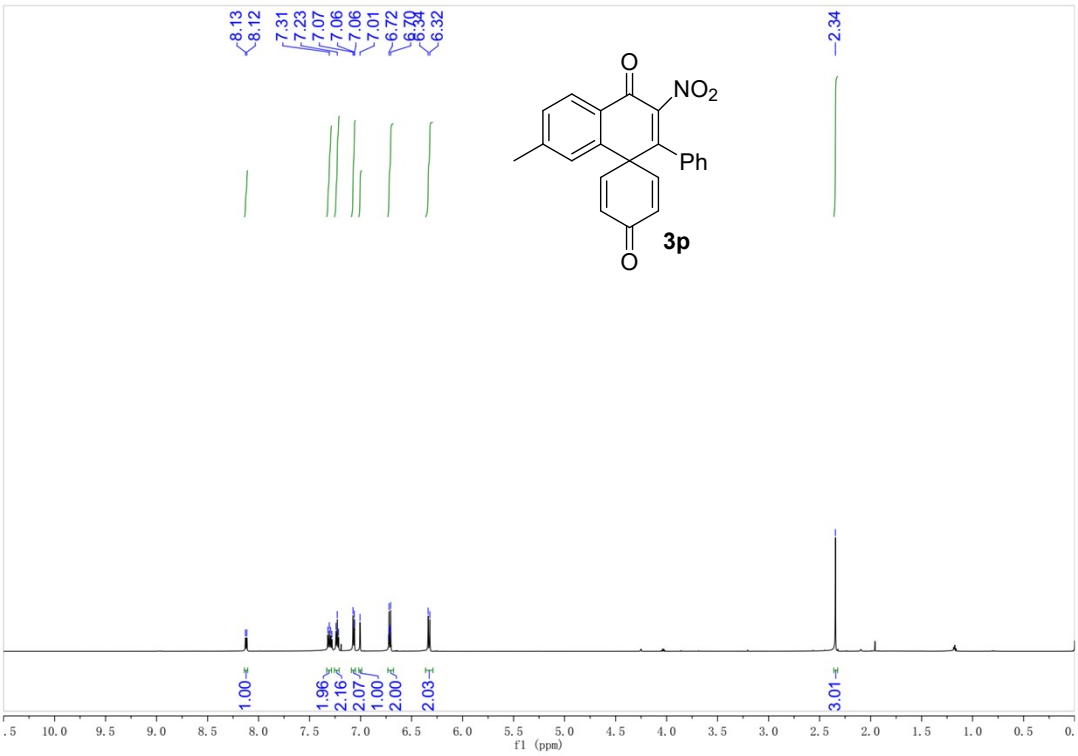
O
NO2
S
O
3o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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6.34
6.32
2.34
O
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Ph
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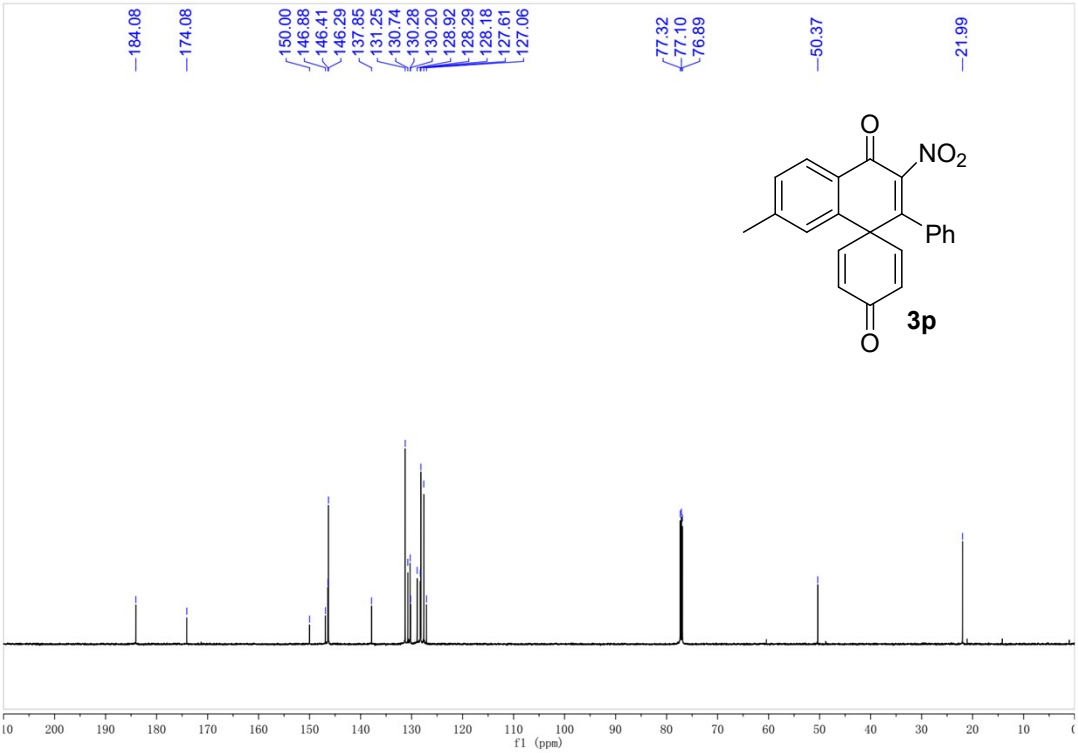
2.16
2.07
1.00
2.00
2.03
3.01
f1 (ppm)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128.18
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77.10
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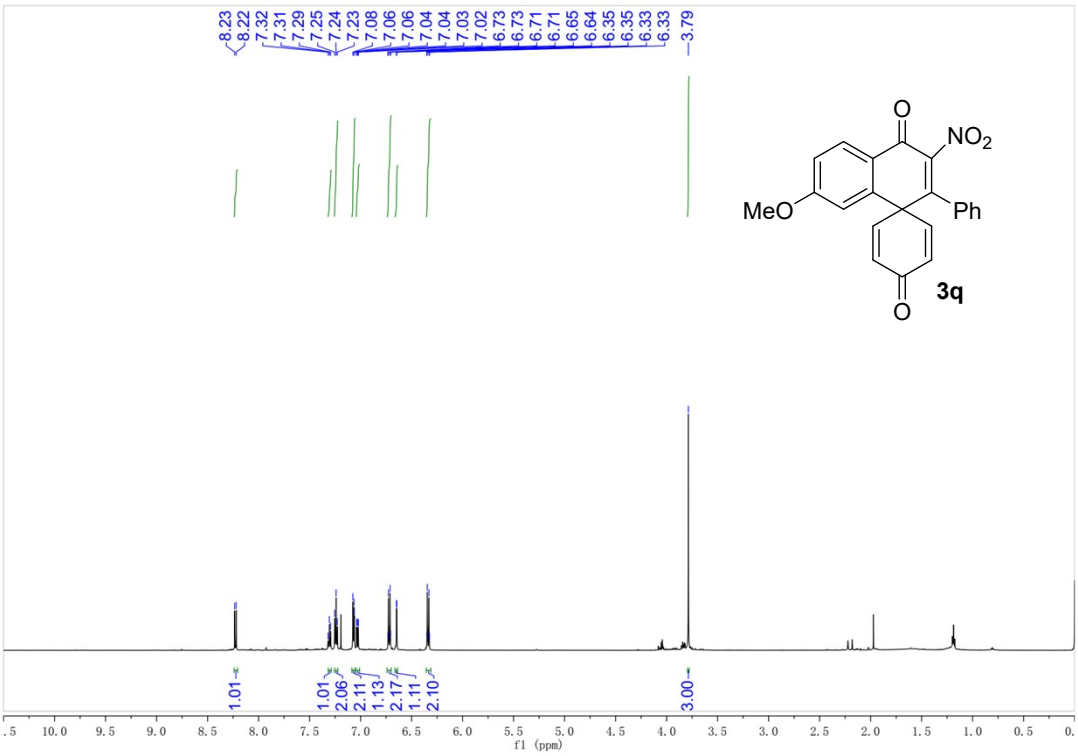
O
NO2
Ph
3p
O
f1 (ppm)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6.33
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O
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MeO
Ph
O
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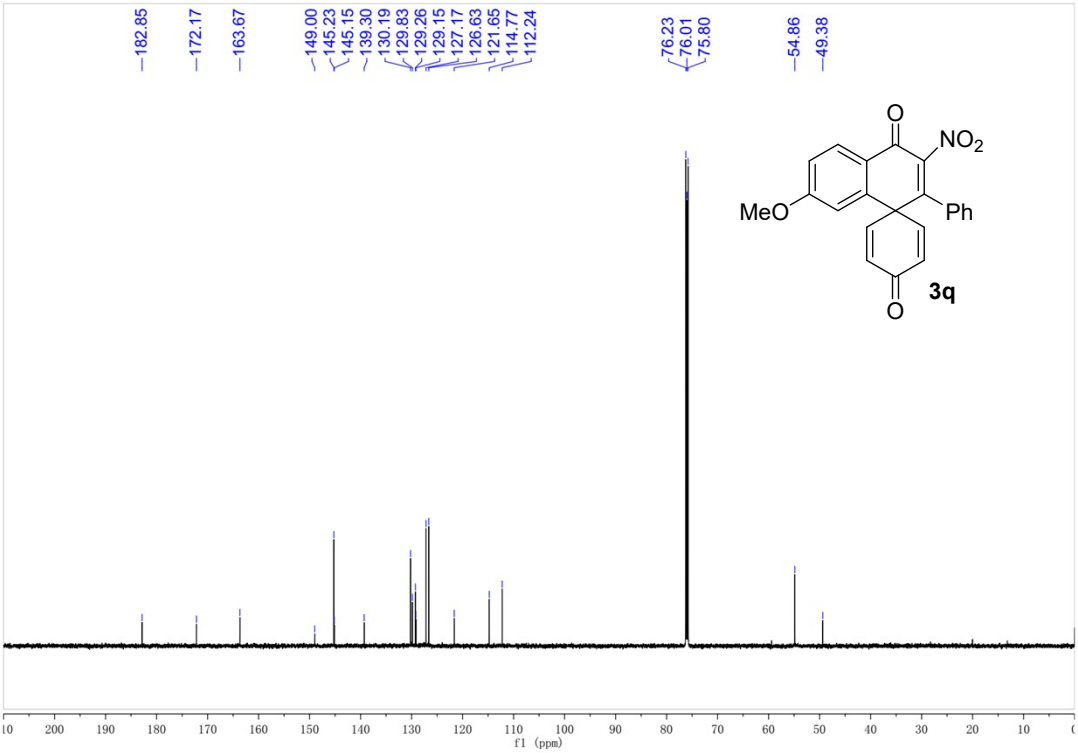
1.01
2.06
2.11
1.13
2.17
1.11
2.10
3.00
f1 (ppm)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130.19
129.83
129.26
129.15
127.17
126.63
121.65
114.77
112.24
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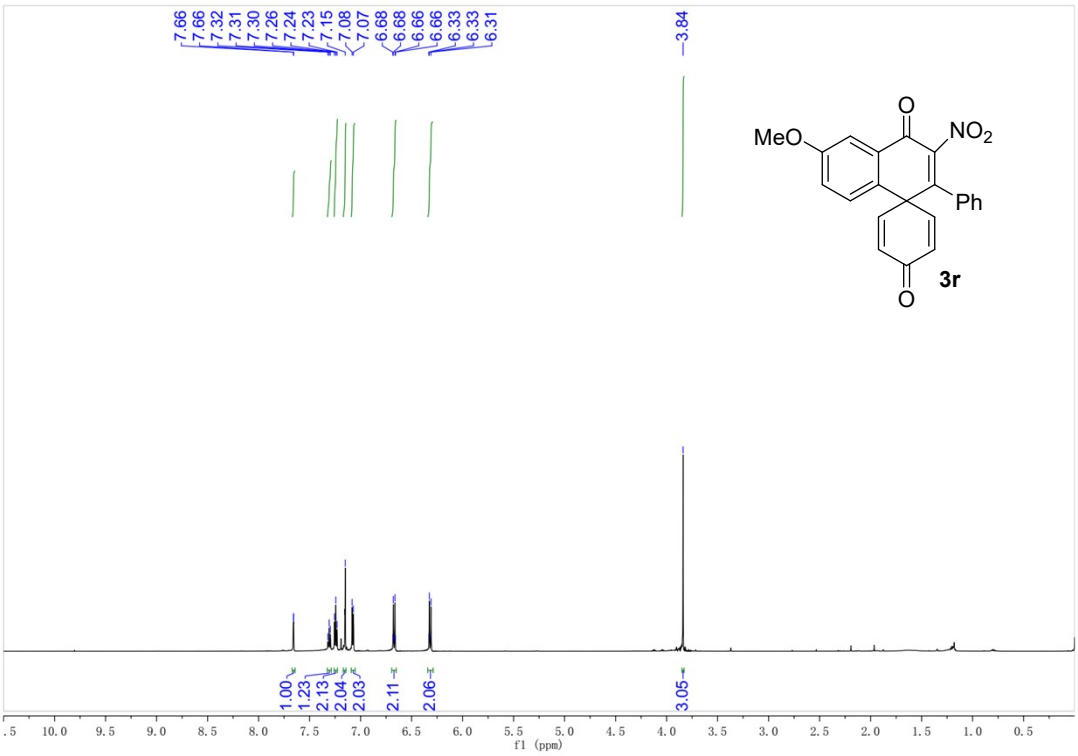
76.01
75.80
54.86
49.38
O
NO2
MeO
Ph
O
3q
f1 (ppm)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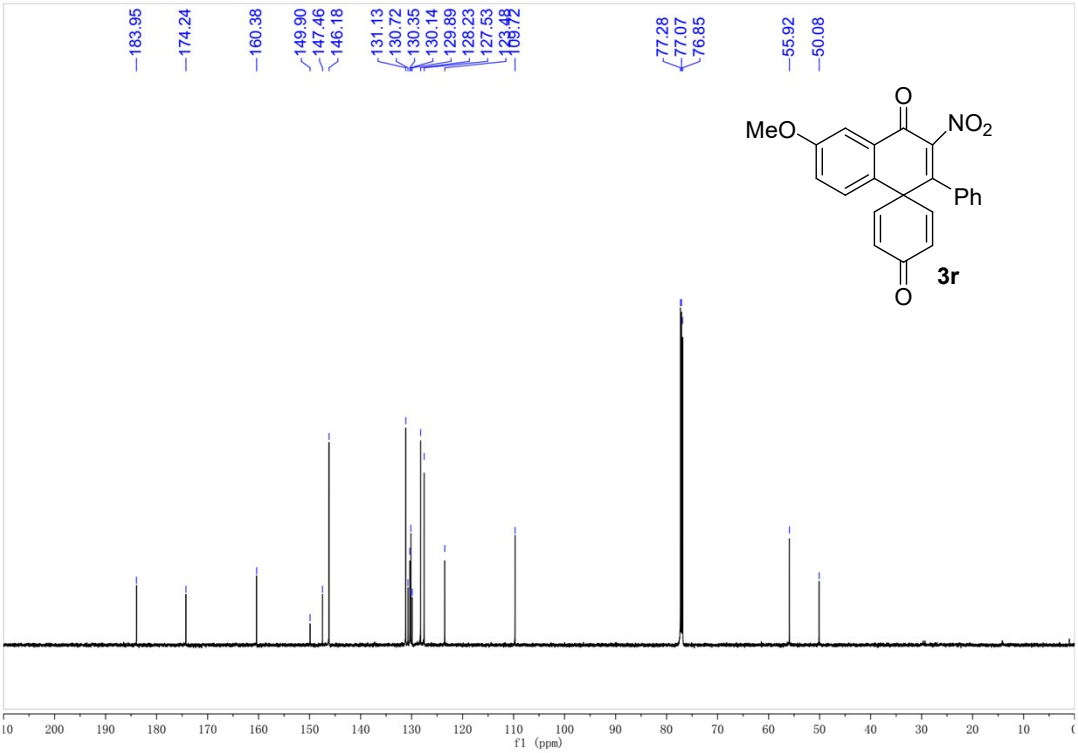
7.24
7.23
7.15
7.08
7.07
6.68
6.68
6.66
6.66
6.33
6.33
6.31
3.84
MeO
NO2
Ph
3r
1.00
1.23
2.13
2.04
2.03
2.11
2.06
3.05
f1 (ppm)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160.38
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147.46
146.18
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130.72
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130.14
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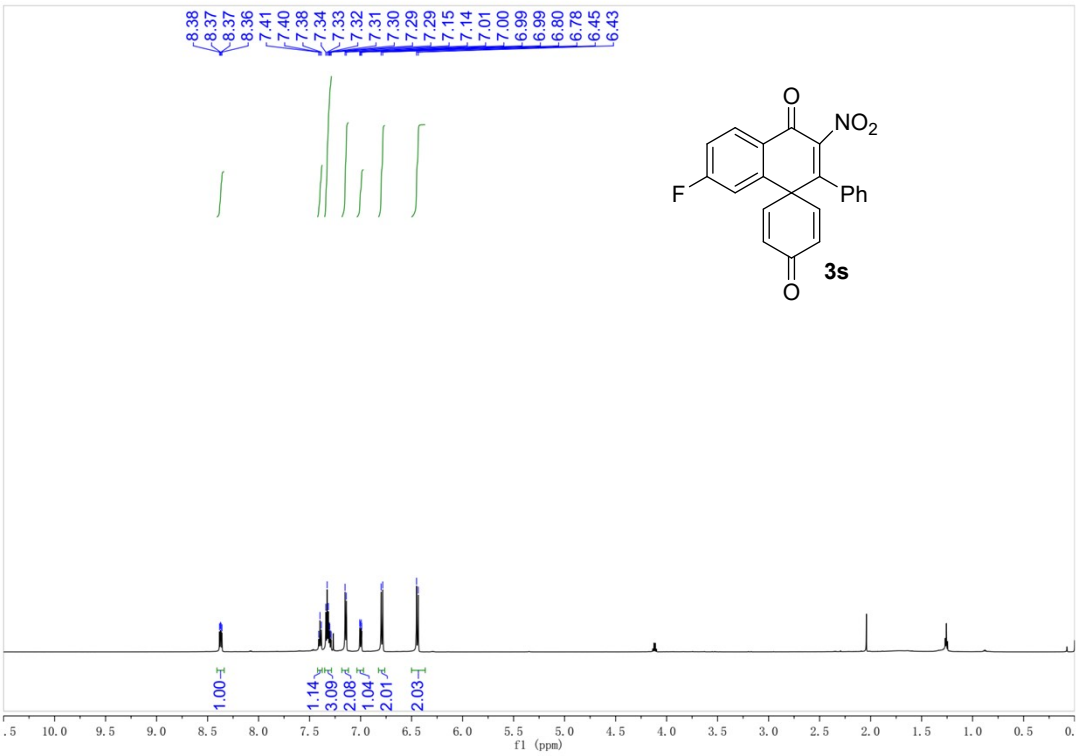
127.53
77.28
77.07
76.85
55.92
50.08
MeO
NO2
Ph
3r
f1 (ppm)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7.32
7.31
7.30
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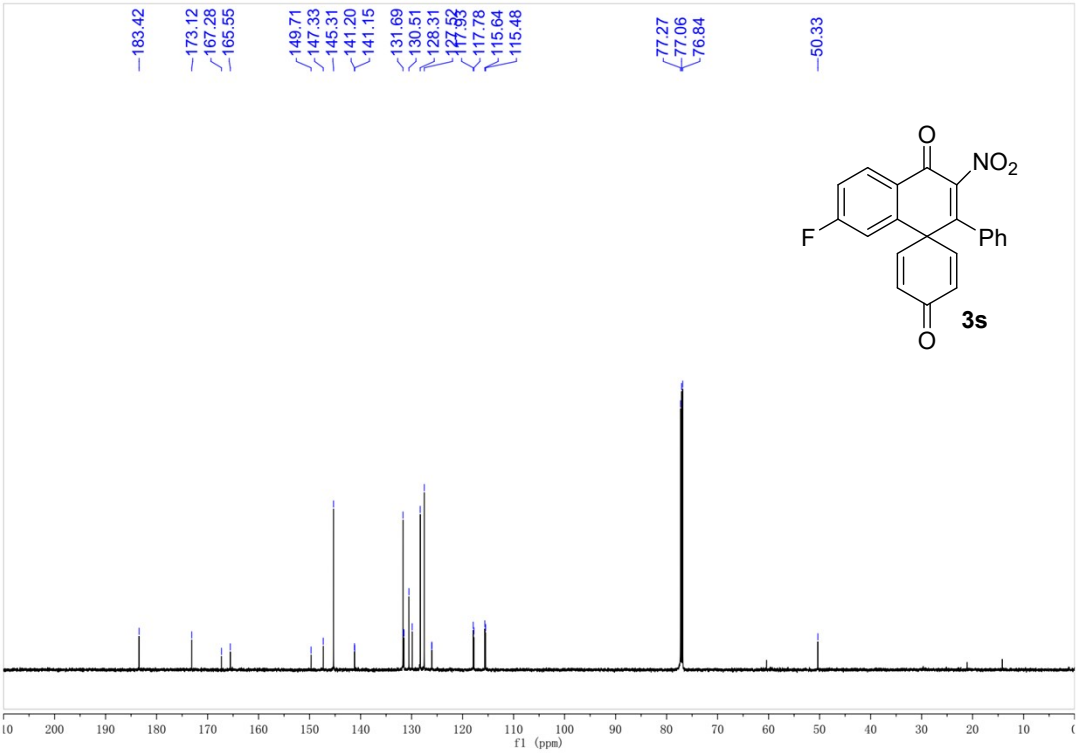
7.29
7.15
7.14
7.01
7.00
6.99
6.99
6.80
6.78
6.45
6.43
O
NO2
F
Ph
O
3s
1.00
1.14
3.09
2.08
1.04
2.01
2.03
f1 (ppm)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145.31
141.20
141.15
131.69
130.51
128.31
117.78
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77.27
77.06
76.84
50.33
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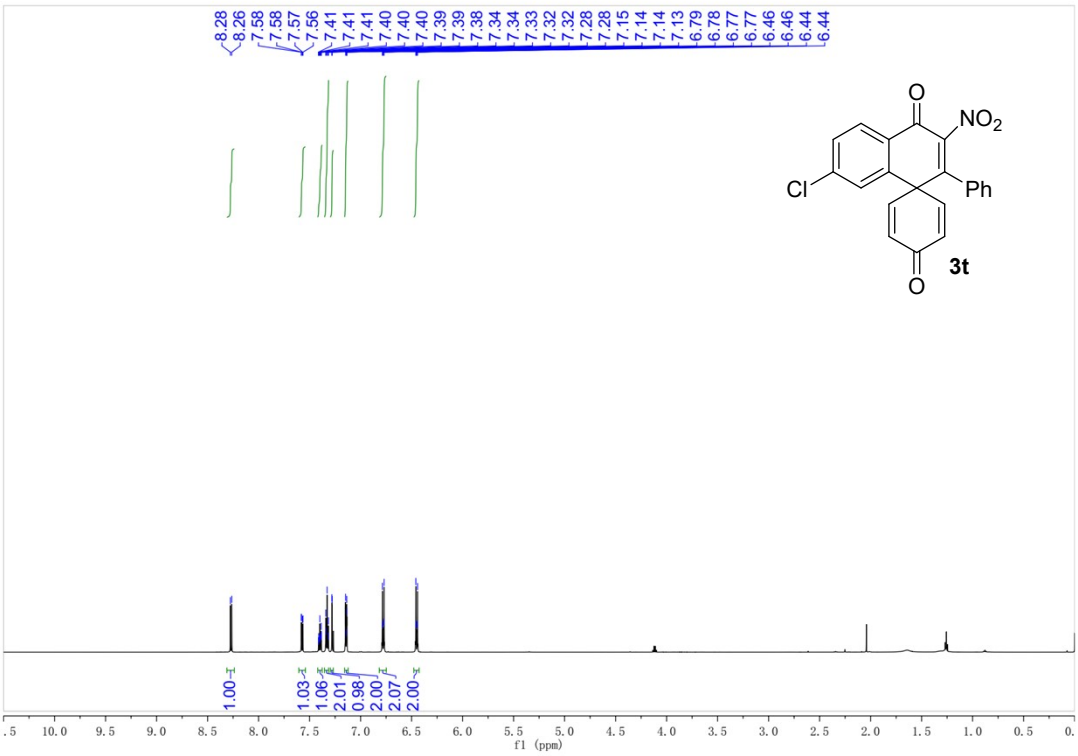
NO2
F
Ph
3s
O
f1 (ppm)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7.41
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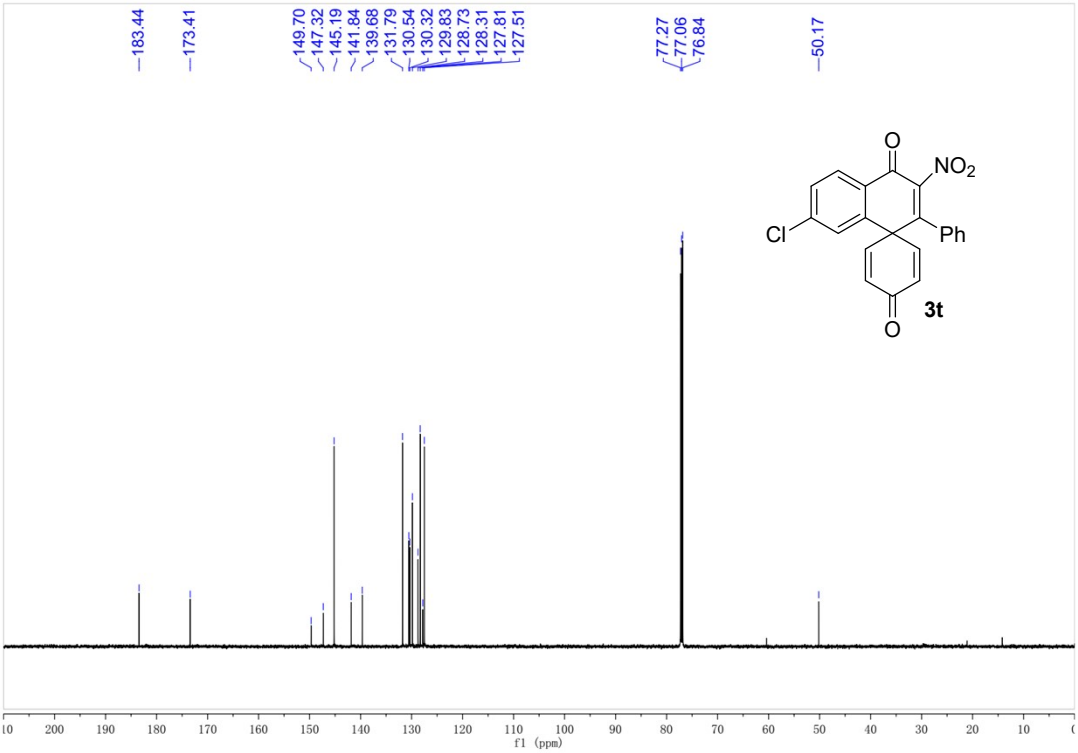
7.39
7.39
7.38
7.34
7.34
7.33
7.32
7.32
7.28
7.28
7.15
7.14
7.14
7.13
6.79
6.78
6.77
6.77
6.46
6.44
6.44
O
NO2
Cl
Ph
O
3t
1.00
1.03
1.06
2.01
0.98
2.00
2.07
2.00
f1 (ppm)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141.84
139.68
131.79
130.54
130.32
129.83
128.73
128.31
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127.51
77.27
77.06
76.84
50.17
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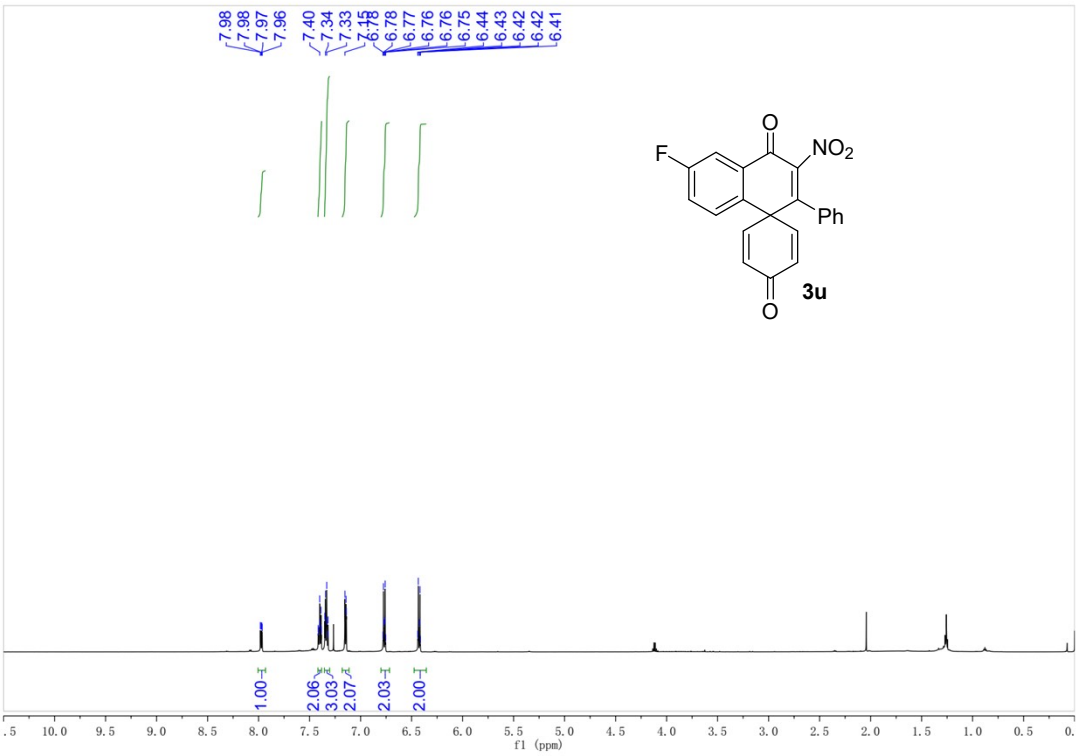
NO2
Cl
Ph
O
3t
f1 (ppm)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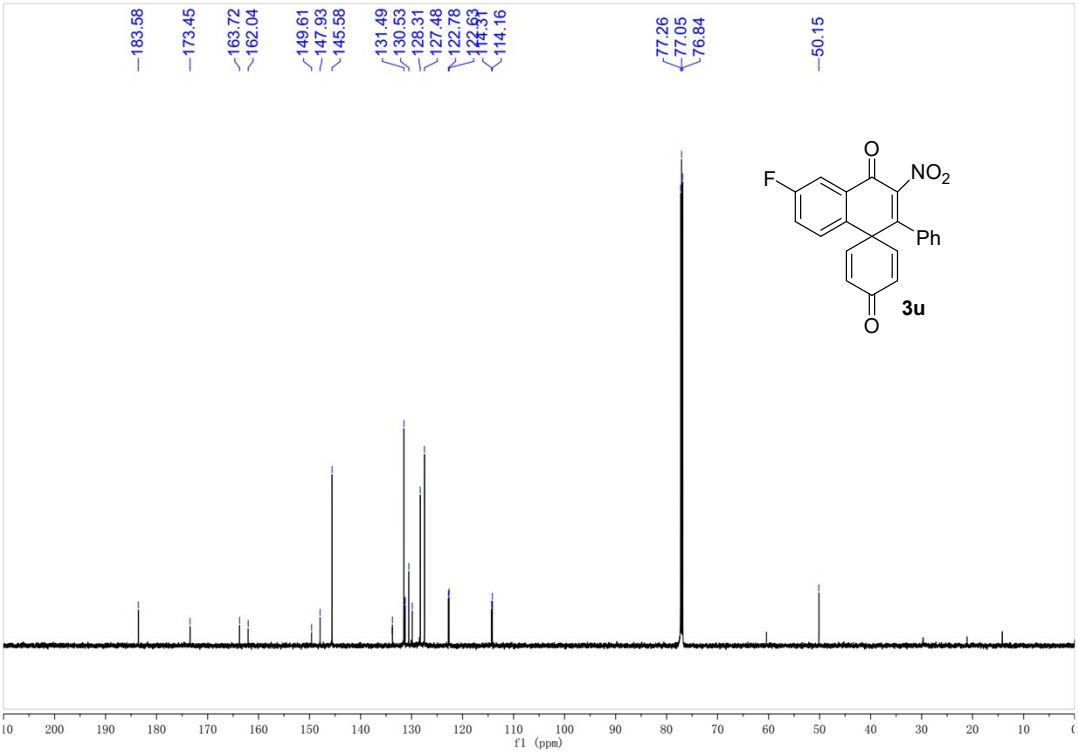
7.98
7.97
7.96
7.40
7.34
7.33
6.78
6.77
6.76
6.76
6.75
6.44
6.43
6.42
6.42
6.41
O
NO2
F
Ph
O
3u
1.00
2.06
3.03
2.07
2.03
2.00
f1 (ppm)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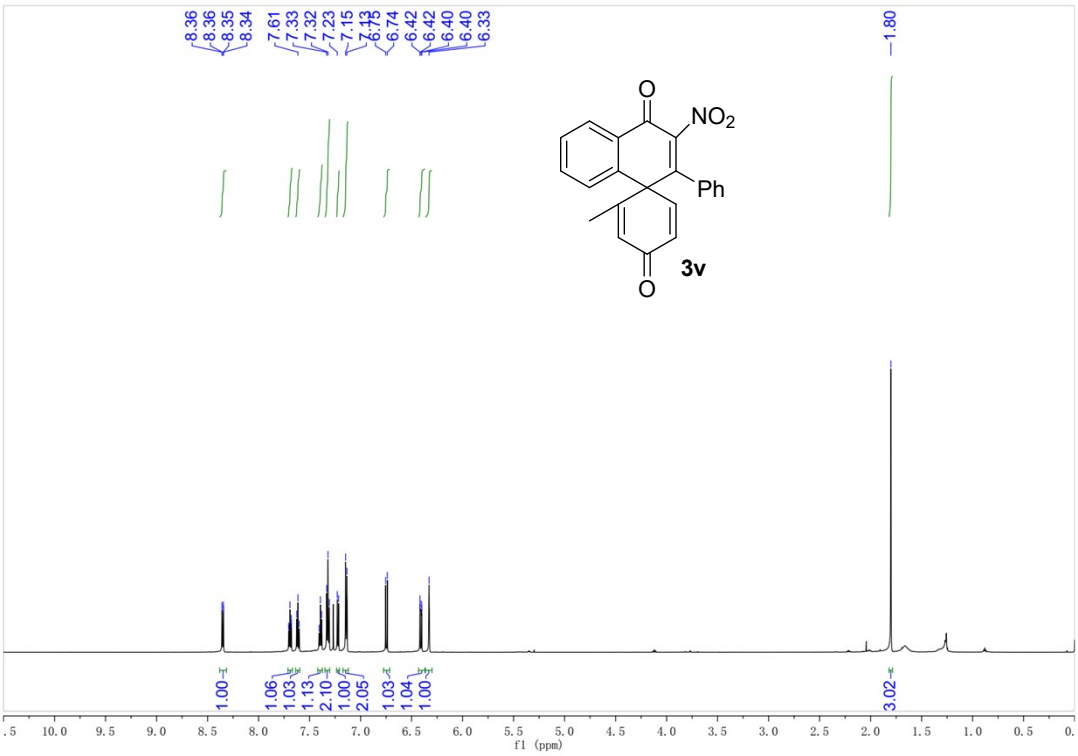
163.72
162.04
149.61
147.93
145.58
131.49
130.53
128.31
127.48
122.78
77.26
77.05
76.84
50.15
O
NO2
F
Ph
O
3u
f1 (ppm)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7.33
7.32
7.23
7.15
6.74
6.42
6.42
6.40
6.40
6.33
1.80
O
NO2
Ph
3v
O
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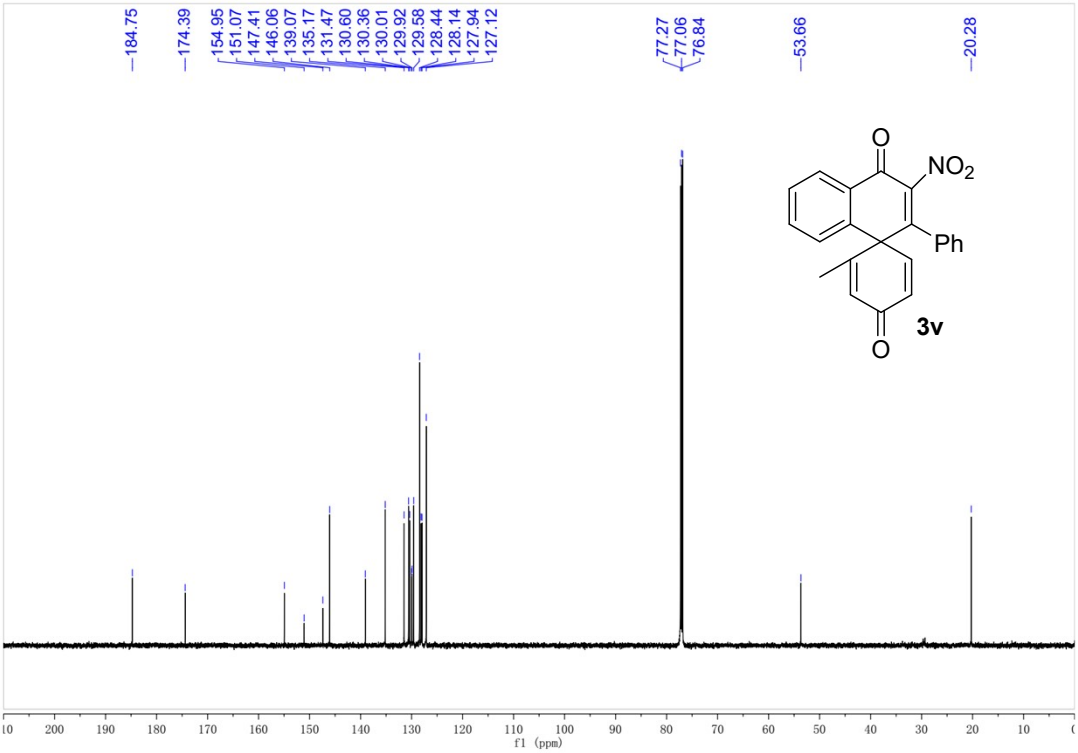
1.06
1.03
1.13
2.10
1.00
2.05
1.03
1.04
1.00
3.02
f1 (ppm)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146.06
139.07
135.17
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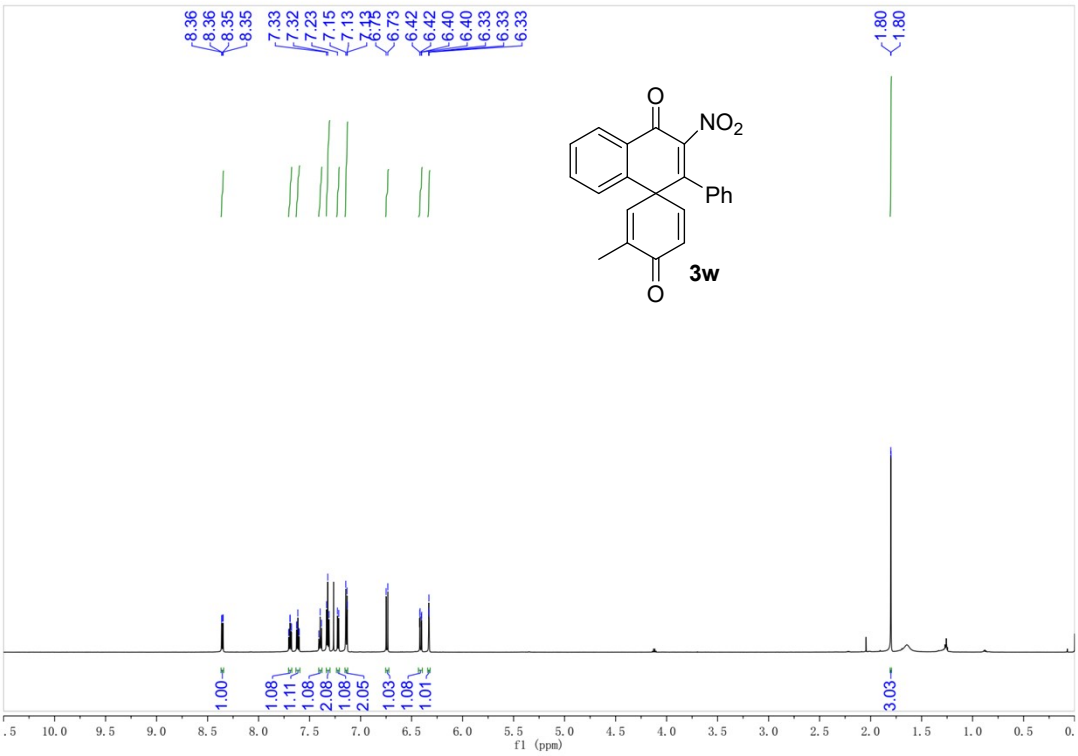
130.60
130.36
130.01
129.92
129.58
128.44
128.14
127.94
127.12
77.27
77.06
76.84
53.66
20.28
O
NO2
Ph
3v
O
f1 (ppm)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7.32
7.23
7.15
7.13
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6.42
6.42
6.40
6.40
6.33
6.33
1.80
1.80
O
NO2
Ph
O
3w
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1.08
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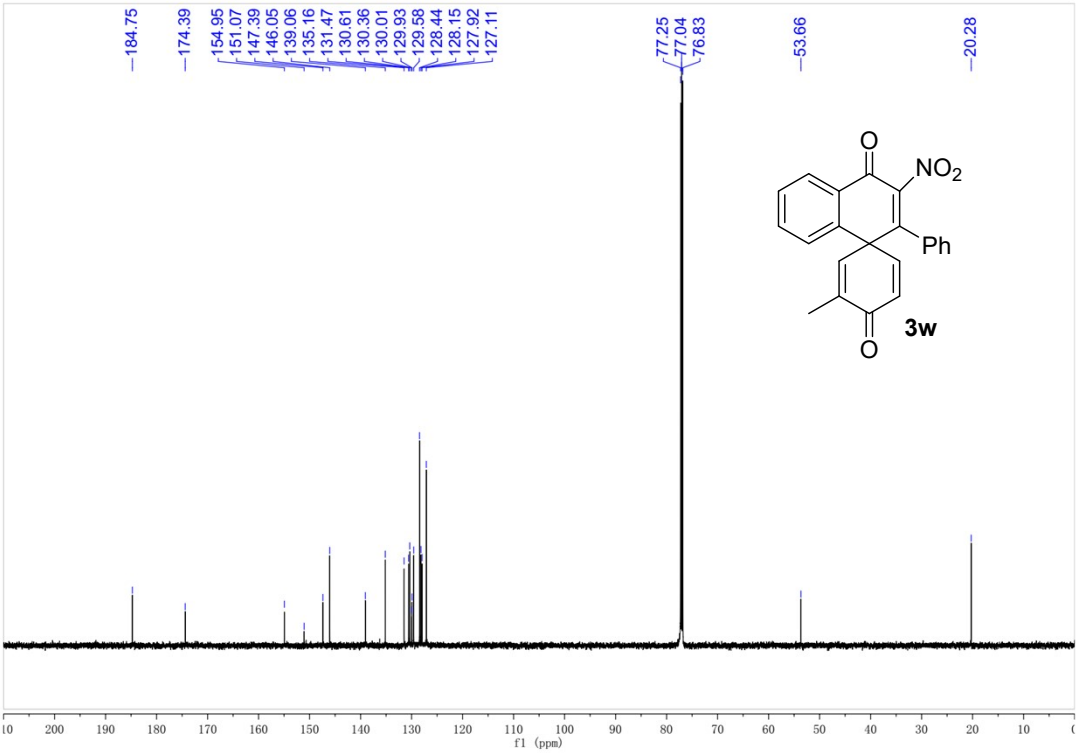
1.08
2.08
1.08
2.05
1.03
1.08
1.01
3.03
f1 (ppm)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151.07
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130.36
130.01
129.93
129.58
128.44
128.15
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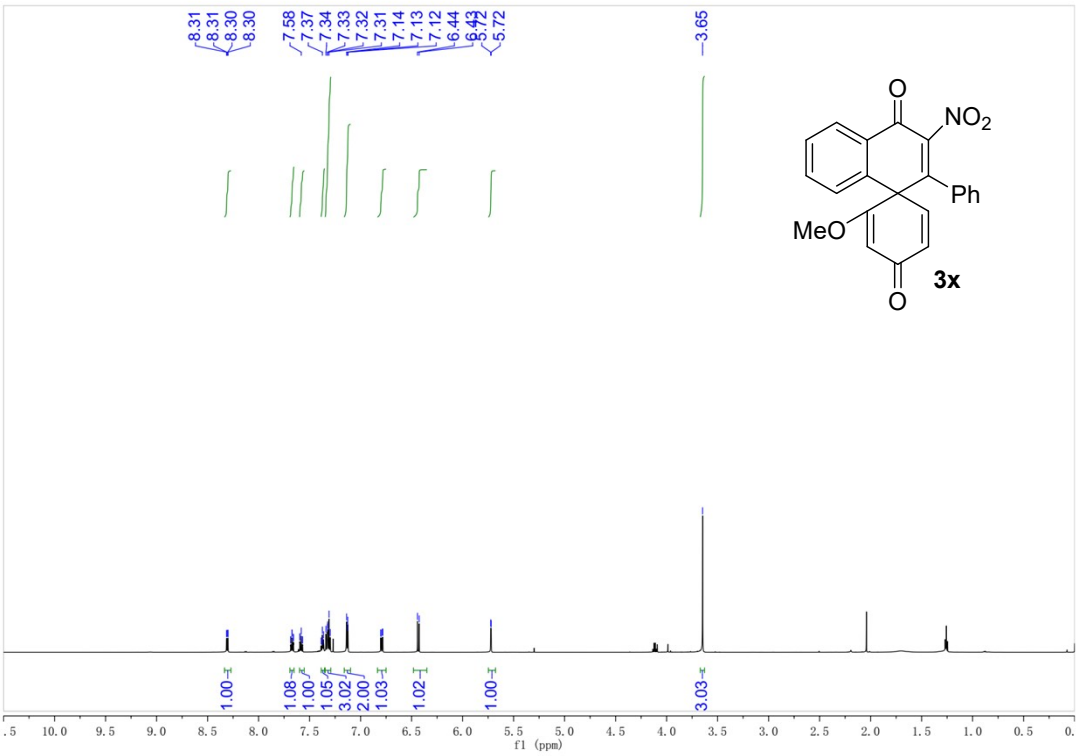
77.25
77.04
76.83
53.66
20.28
O
NO2
Ph
O
3w
f1 (ppm)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7.37
7.34
7.33
7.32
7.31
7.14
7.13
7.12
6.44
5.72
3.65
O
NO2
Ph
MeO
O
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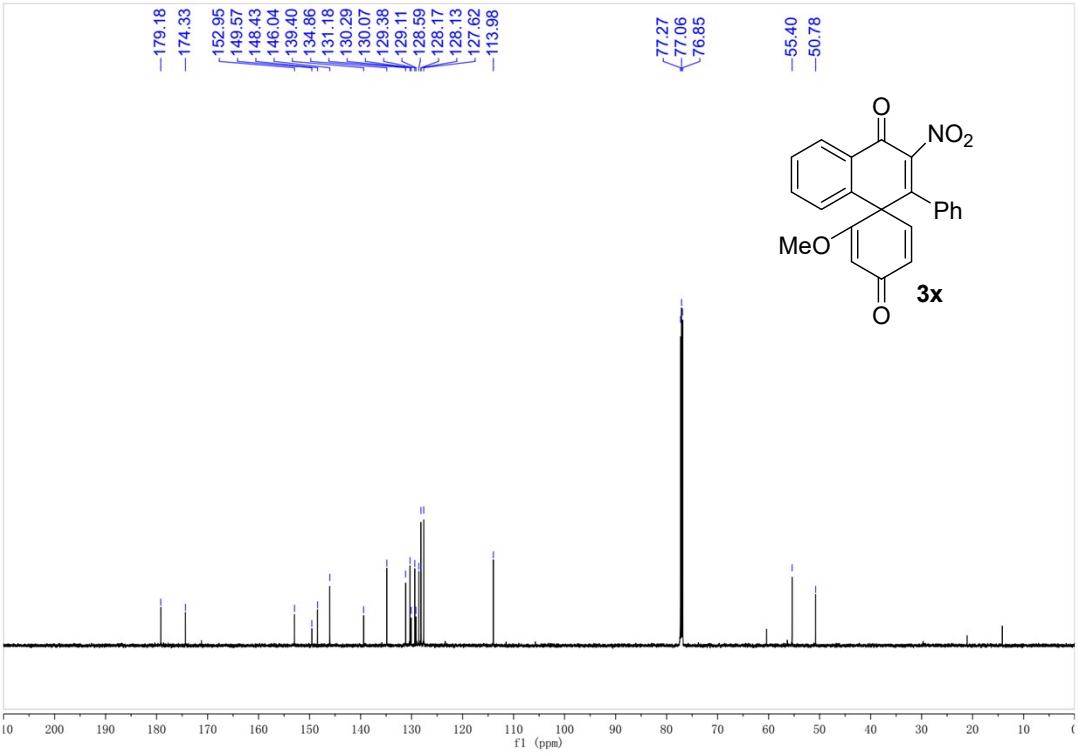
1.00
1.08
1.00
1.05
3.02
2.00
1.03
1.02
1.00
3.03
f1 (ppm)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129.11
128.59
128.17
128.13
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77.06
76.85
55.40
50.78
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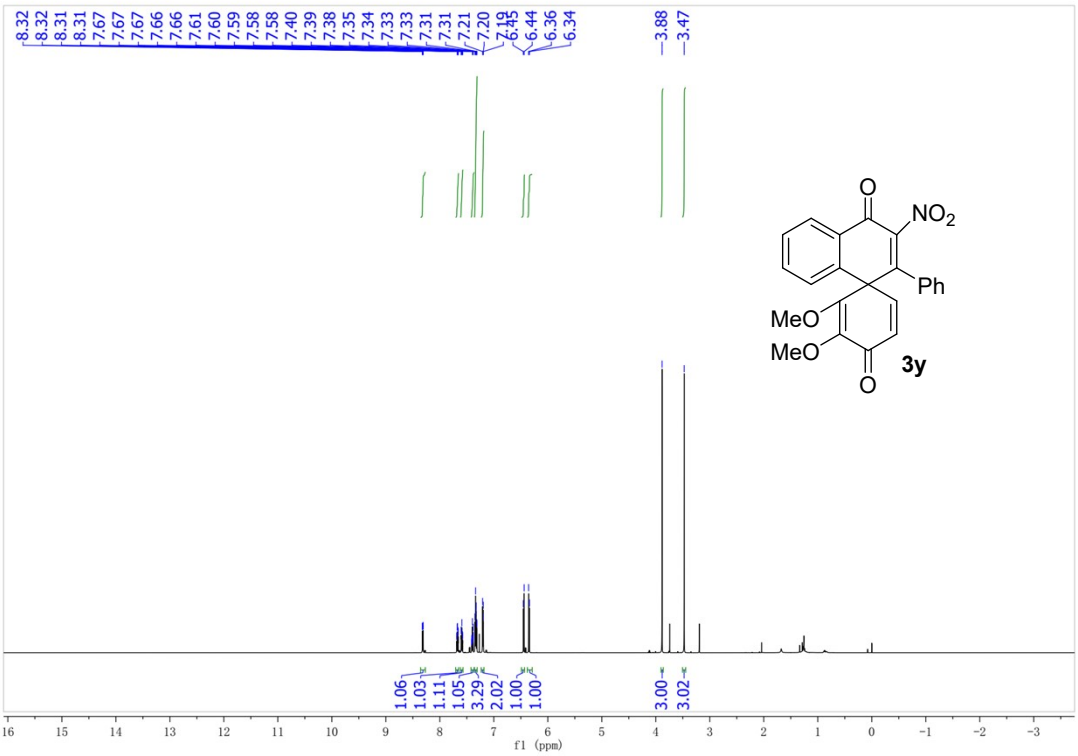
NO2
Ph
MeO
O
3x
f1 (ppm)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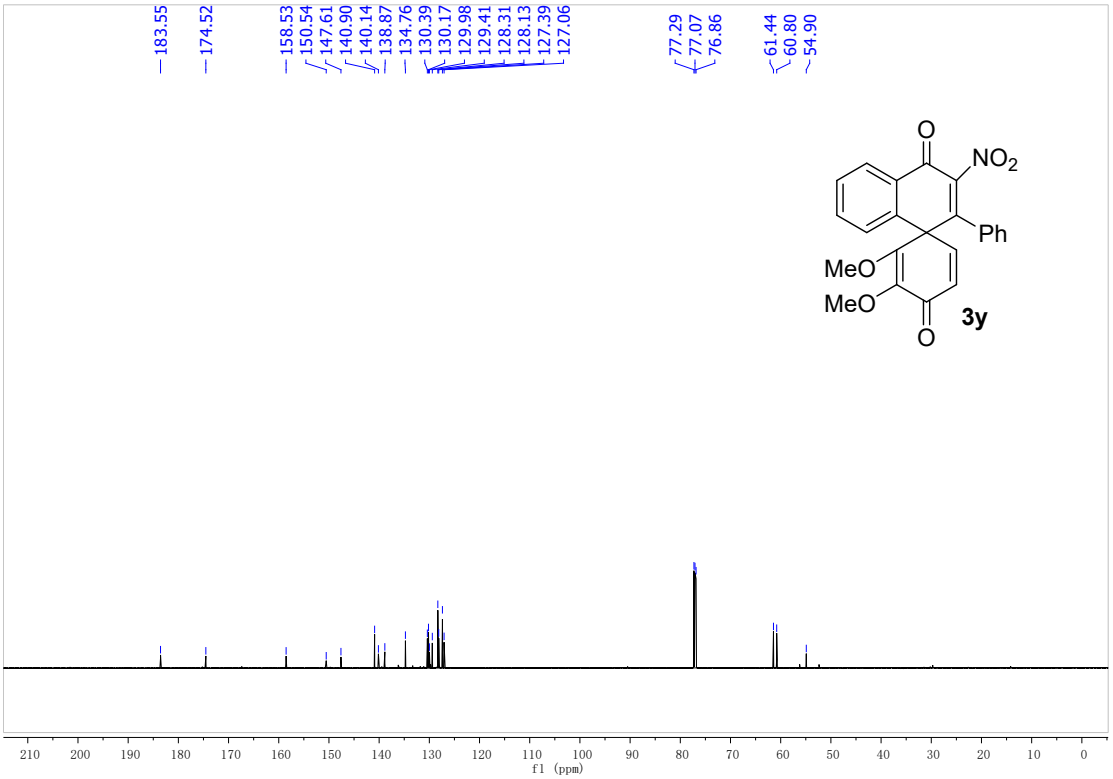
7.58
7.58
7.40
7.39
7.38
7.35
7.34
7.33
7.33
7.31
7.31
7.21
7.20
7.19
6.45
6.44
6.36
6.34
3.88
3.47
1.06
1.03
1.11
1.05
3.29
2.02
1.00
1.00
3.00
3.02
f1 (ppm)
O
NO2
Ph
MeO
MeO
O
3y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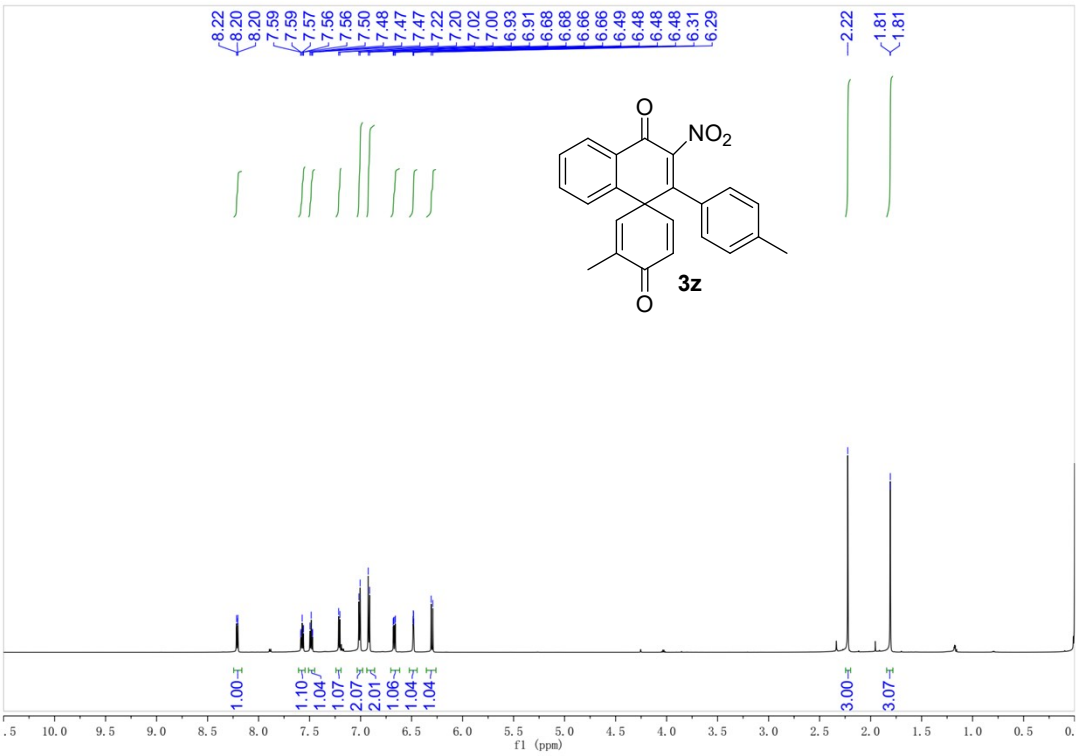
129.41
128.31
128.13
127.39
127.06
77.29
77.07
76.86
61.44
60.80
54.90
f1 (ppm)
O
NO2
Ph
MeO
MeO
O
3y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7.22
7.20
7.02
7.00
6.93
6.91
6.68
6.68
6.66
6.66
6.49
6.48
6.48
6.48
6.31
6.29
2.22
1.81
1.81
O
NO2
O
3z
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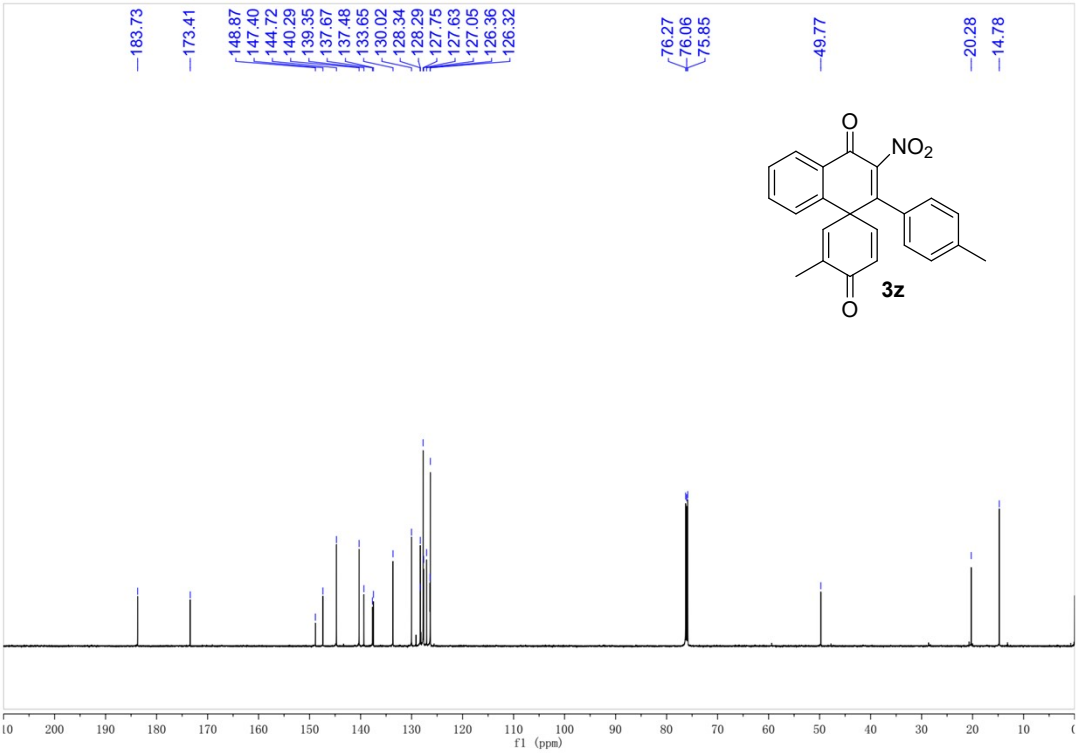
1.07
2.07
2.01
1.06
1.04
1.04
3.00
3.07
f1 (ppm)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137.67
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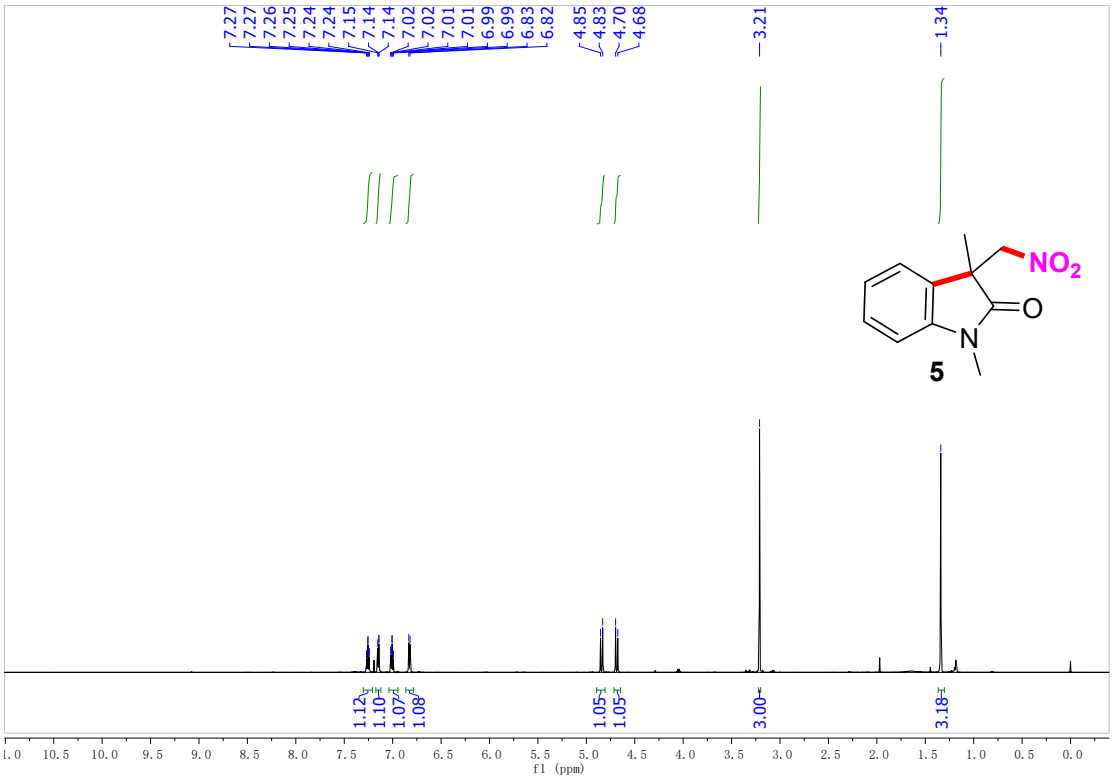
133.65
130.02
128.34
128.29
127.75
127.63
127.05
126.36
126.32
76.27
76.06
75.85
49.77
20.28
14.78
O
NO2
O
3z
f1 (ppm)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7.02
7.02
7.01
7.01
6.99
6.99
6.83
6.82
4.85
4.83
4.70
4.68
3.21
1.34
NO2
O
N
5
1.12
1.10
1.07
1.08
1.05
1.05
3.00
3.18
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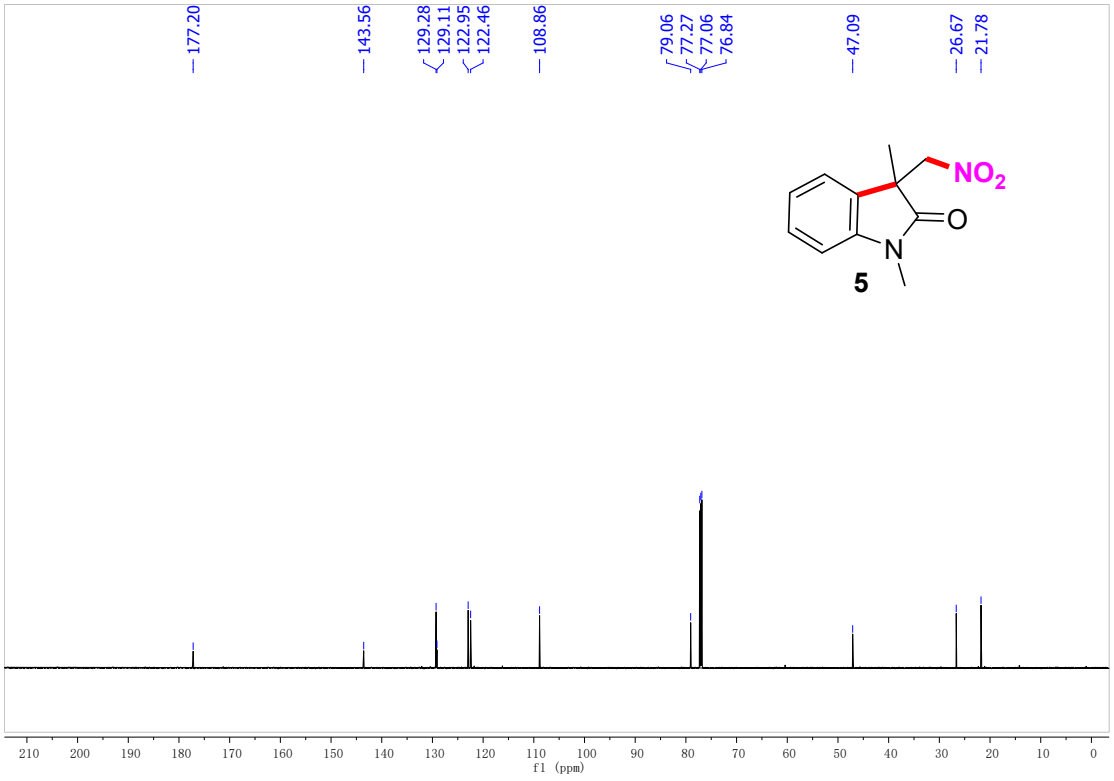
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129.11
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79.06
77.27
77.06
76.84
47.09
26.67
21.78
NO2
O
N
5
f1 (ppm)