Supporting Information

Photocatalyst-free hypervalent iodine reagent catalyzed decarboxylative acylarylation of acrylamides with α-oxocarboxylic acids driven by visible-light irradiation

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1. General considerations

All ^{1}H NMR and ^{13}C NMR spectra were recorded on a 400 MHz Bruker FT-NMR spectrometers (400 MHz or 100 MHz, respectively). All chemical shifts are given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, J, are reported in Hertz (Hz). High resolution mass spectroscopy data of the product were collected on a Waters Micromass GCT instrument. High resolution mass spectroscopy data of the product were collected on an Agilent Technologies 6540 UHD Accurate-Mass Q-TOF LC/MS (ESI).

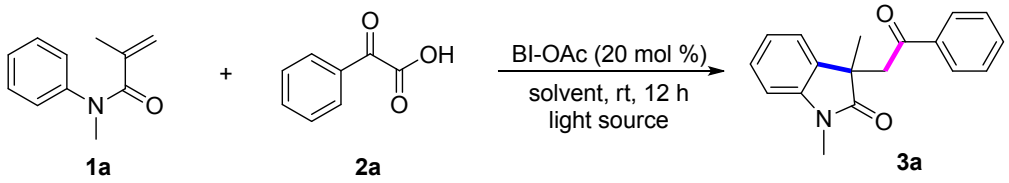
α-Oxocarboxylic acids and acrylamides are prepared according to reported methods (J. Du, X. Zhang, L. Wang, *Chem. Commun.* 2015, **51**, 4372–4375; A. J.-L. Ayitou, J. Sivaguru, *Chem. Commun.* 2011, **47**, 2568–2570), and which must be recrystallized from ethanol/ethyl acetate before use. The chemicals and solvents were purchased from commercial suppliers either Aldrich (USA), or Shanghai Chemical Company (P. R. China). All the solvents were dried and freshly distilled in N_2 prior to use. Products were purified by flash chromatography on 200–300 mesh silica gels, SiO_2.

2. Typical procedure for the decarboxylative acylarylation of acrylamides with α-oxocarboxylic acids

A 5 mL oven-dried reaction vessel equipped with a magnetic stirrer bar was charged with *N*-methyl-*N*-phenylmethacrylamide (**1a**, 35.0 mg, 0.20 mmol), 2-oxo-2-phenylacetic acid (**2a**, 45.0 mg, 0.30 mmol), BI-OAc (12.3 mg, 0.04 mmol), and PhCl (1.0 mL). The reaction vessel was exposed to blue LED (450–455 nm, 1.5 W) irradiation at room temperature in air with stirring for 12 h. 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 = 3:1 to 15:1) to give the desired product **3a** (43.5 mg, 78% yield).

3. Optimization of light source and solvent

Table S1. Screen of light source and solvent[a]



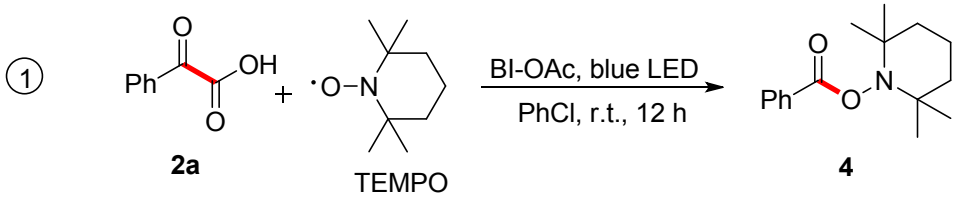
1a + **2a** → **3a**

Entry	Light source	Solvent	Yield[b] (%)
1	blue LED (450–455 nm, 1.5 W)	PhCl	78
2	sunlight	PhCl	69
3	green LED (530–535 nm, 1.5 W)	PhCl	18
4	purple LED (400–405 nm, 1.5 W)	PhCl	31
5	red LED (695–700 nm, 1.5 W)	PhCl	Trace
6	white LED (8 W)	PhCl	10
7	UV (226 nm)	PhCl	34
8	blue LED (450–455 nm, 1.5 W)	PhBr	51
9	blue LED (450–455 nm, 1.5 W)	benzene	47
10	blue LED (450–455 nm, 1.5 W)	DCE	50
11	blue LED (450–455 nm, 1.5 W)	DCM	62
12	blue LED (450–455 nm, 1.5 W)	toluene	26
13	blue LED (450–455 nm, 1.5 W)	DMSO	0
14	blue LED (450–455 nm, 1.5 W)	CH_3CN	0

[a]Reaction conditions: *N*-methyl-*N*-phenyl-methacrylamide (**1a**, 0.20 mmol), 2-oxo-2-phenylacetic acid (**2a**, 0.30 mmol), HIR (20 mol%), solvent (1.0 mL) at room temperature under light irradiation in air for 12 h. [b]Isolated yield.

4. Preliminary mechanistic study

(1) Free radical-trapping experiments



A 5 mL oven-dried reaction vessel equipped with a magnetic stirrer bar was charged with 2-oxo-2-phenylacetic acid (**2a**, 45.0 mg, 0.30 mmol), BI-OAc (12.3 mg, 0.04 mmol), TEMPO

(93.75 mg, 0.60 mmol) and PhCl (1.0 mL). The reaction vessel was exposed to blue LED (450–455 nm, 1.5 W) irradiation in air at room temperature with stirring for 12 h. After that, the mixture was concentrated to yield the crude product, which was further purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 3:1 to 15:1) to give the trapping product **4** in 70% yield (54.8 mg). The following figure is the HRMS analysis of reaction mixture (Figure S1).

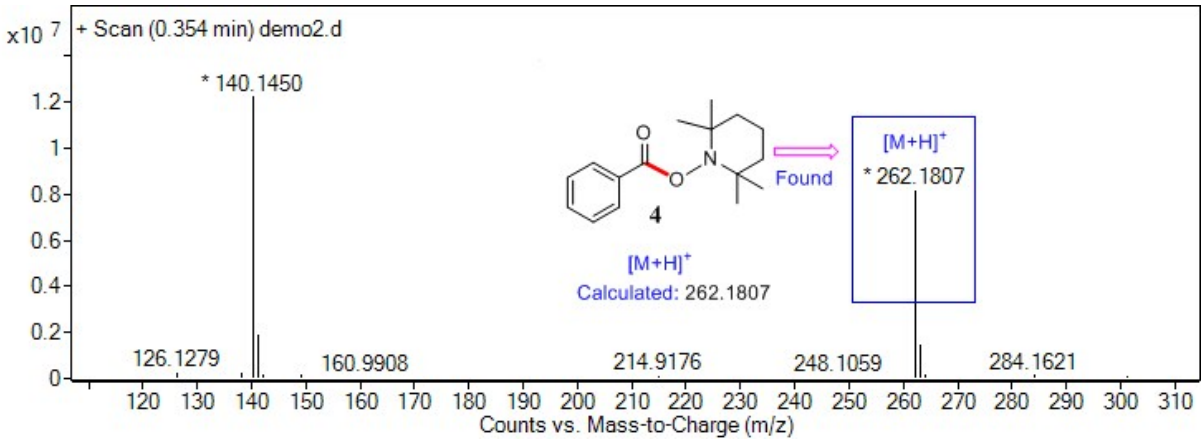
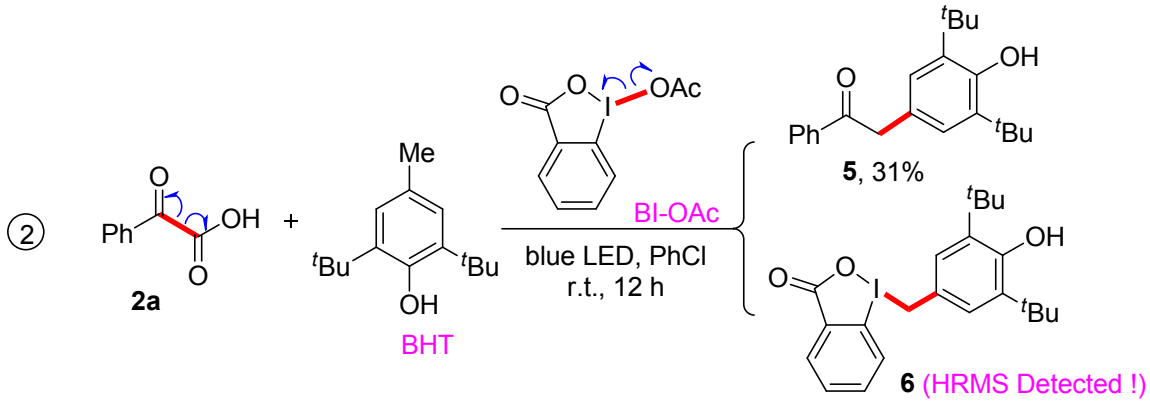


Figure S1. Analysis of reaction mixture by HRMS



A 5 mL oven-dried reaction vessel equipped with a magnetic stirrer bar was charged with 2-oxo-2-phenylacetic acid (**2a**, 225 mg, 1.5 mmol), BHT (2,6-di-*tert*-butyl-4-methylphenol, 661.1 mg, 3.0 mmol), BI-OAc (61.2 mg, 0.20 mmol) and PhCl (4.0 mL). The reaction vessel was exposed to blue LED (450–455 nm) irradiation in air at room temperature with stirring for 12 h. After that, the mixture was concentrated to yield the crude product, which was further

purified by flash chromatography (silica gel, petroleum ether/ethyl acetate = 15:1 to 30:1) to give the trapping product **5** (K. Sun, X. Wang, G. Li, Z. Zhu, *Y. Jiang, B. Xiao, Chem. Commun.,* 2014, **50**, 12880–12883) in 31% isolated yield (150.7 mg) and **6** (A. M. Nicholas, P. Bozo, *J. Am. Chem. Soc.,* 1968, **90**, 4450−4453; M. Ochiai, A. Nakanishi, T. Ito, *J. Org. Chem.,* 1997, **62**, 4253–4259), which was identified by the HRMS analysis of reaction mixture (Figure S2).

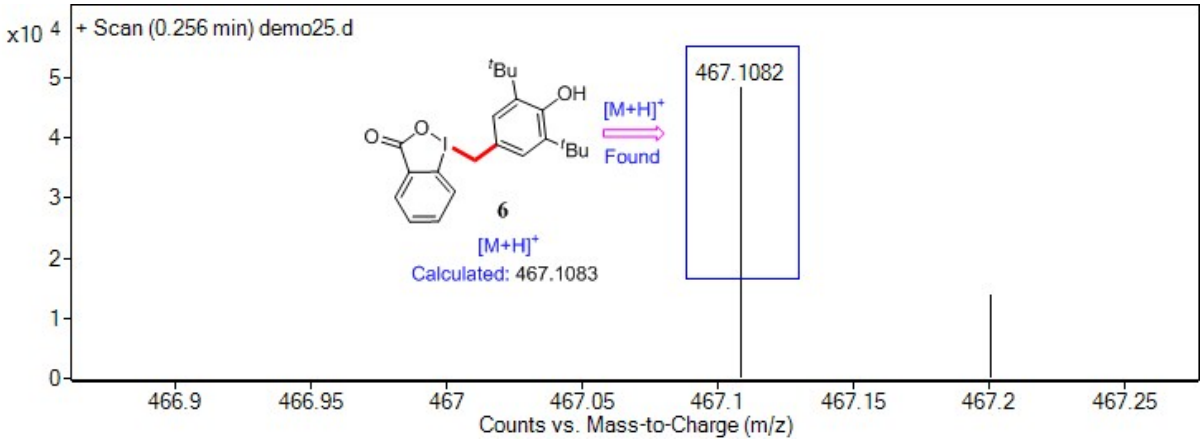
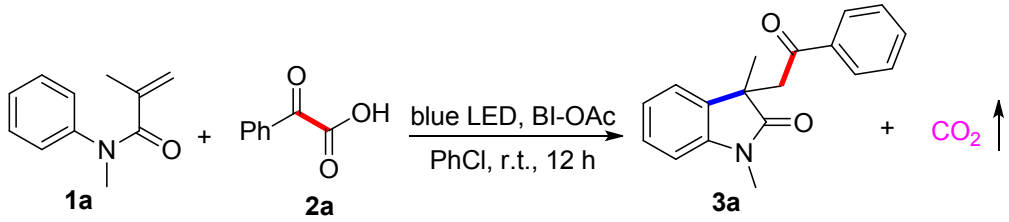


Figure S2. HRMS analysis of reaction mixture

(2) Determination of the resulting CO_2 gas by FT-IR



A 5 mL oven-dried reaction vessel equipped with a magnetic stirrer bar was charged with *N*-methyl-*N*-phenyl-methacrylamide (**1a**, 35.0 mg, 0.20 mmol), 2-oxo-2-phenylacetic acid (**2a**, 45.0 mg, 0.30 mmol), BI-OAc (12.3 mg, 0.04 mmol), and PhCl (1.0 mL) under nitrogen atmosphere. The reaction vessel was exposed to blue LED (450–455 nm) irradiation at room temperature with stirring for 12 h. After completion of the reaction, the resulting gas from the

reaction mixture was directly determined by FT-IR analysis (Figure S3), and the concentration of CO_2 was found to be 1380.27 ppm.

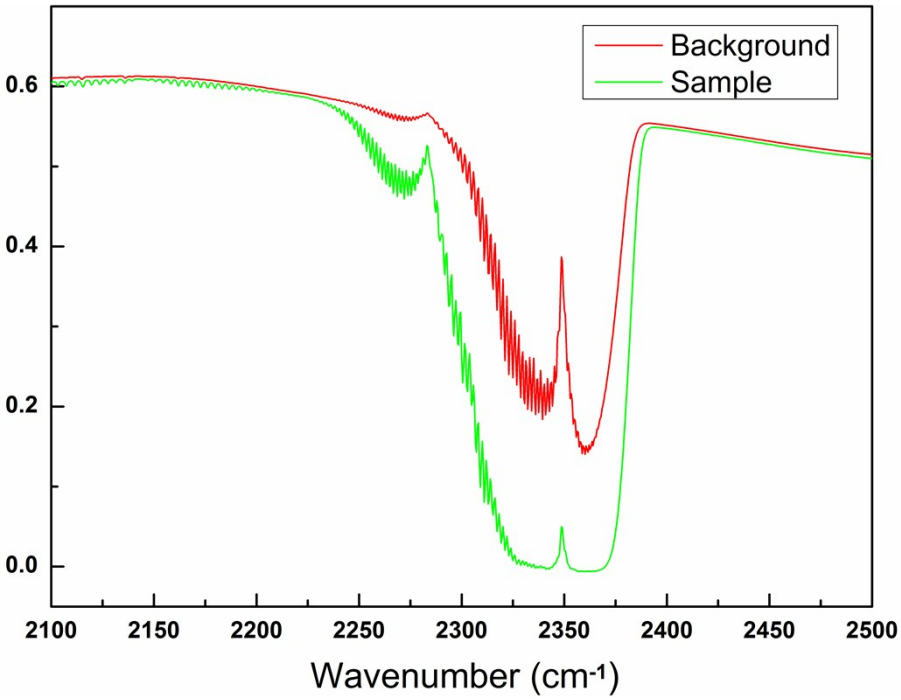
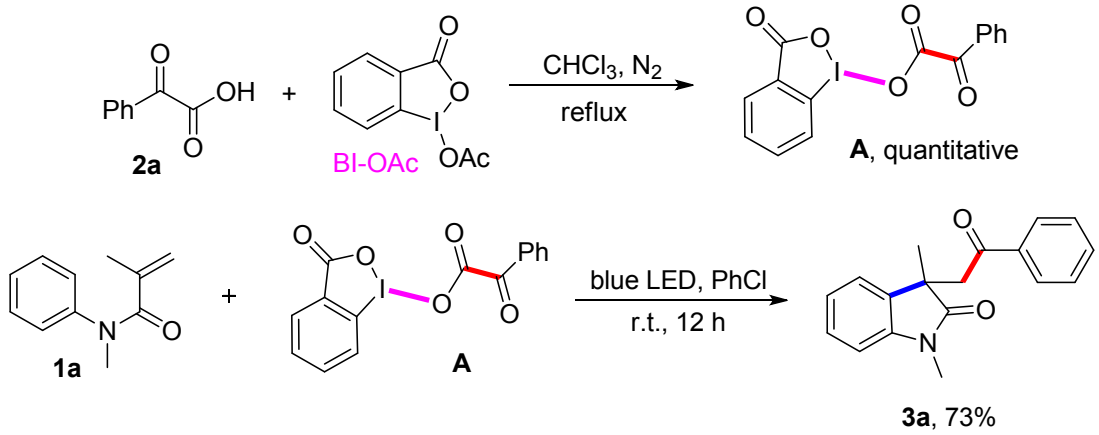


Figure S3. FT-IR analysis of the resulting gas by a Bruker Tensor 27 FT-IR

(3) Typical procedure for the synthesis of **A** and the reaction of **A** with **1a**

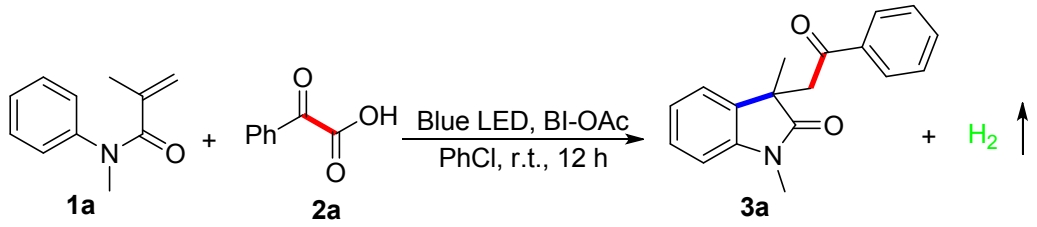


Under nitrogen atmosphere, a 25 mL oven-dried round bottomed flask equipped with a magnetic stirrer bar was charged with dry 2-oxo-2-phenylacetic acid (**2a**, 480.4 mg, 3.2 mmol), BI-OAc (917.8 mg, 3.0 mmol, for the preparation of BI-OAc, see: M. V. Vita, Waser, J. *Org.*

Lett., 2013, **15**, 3246–3249) and anhydrous chloroform (3.0 mL). The mixture was refluxed for 30 min, and next the solvent was removed under vacuum to obtain a viscous fluid. Then the obtained viscous fluid was diluted with dry petroleum (5.0 mL) and cooled in –20 °C until white solid precipitated at the bottom of flask. The formed solid was filtrated and washed with cold, anhydrous CH_3CN, dried under vacuum and kept in dark, affording pure **A** in quantitative yield. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.01 (d, J = 7.6 Hz, 1H), 7.97–7.92 (m, 3H), 7.84 (m, J = 8.4 Hz, 1H), 7.76 (t, J = 7.6 Hz, 1H), 7.69 (t, J = 7.6 Hz, 1H), 7.61 (t, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 189.17, 168.15, 166.55, 135.58, 134.91, 132.35, 132.00, 131.55, 130.82, 129.89, 129.74, 126.74, 120.87.

A 5 mL oven-dried reaction vessel equipped with a magnetic stirrer bar was charged with **1a** (35.0 mg, 0.20 mmol), **A** (79.2 mg, 0.20 mmol) and PhCl (1.0 mL). The reaction vessel was exposed to blue LED (450–455 nm, 1.5 W) irradiation in air at room temperature with stirring for 12 h. 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 = 3:1 to 15:1) to give the desired product **3a** in 73% yield (40.8 mg).

(4) Determination of the resulting H_2 gas by GC



A 10 mL oven-dried reaction vessel equipped with a magnetic stirrer bar was charged with *N*-methyl-*N*-phenyl-methacrylamide (**1a**, 175.1 mg, 1.0 mmol), 2-oxo-2-phenylacetic acid (**2a**, 225.2 mg, 1.5 mmol), BI-OAc (61.2 mg, 0.20 mmol), and PhCl (3.0 mL) under vacuum. The reaction vessel was exposed to blue LED (450–455 nm, 1.5 W) irradiation at room temperature with stirring for 12 h.

During the reaction, the resulting gas from the reaction mixture was directly determined

by GC analysis (Figure S4).

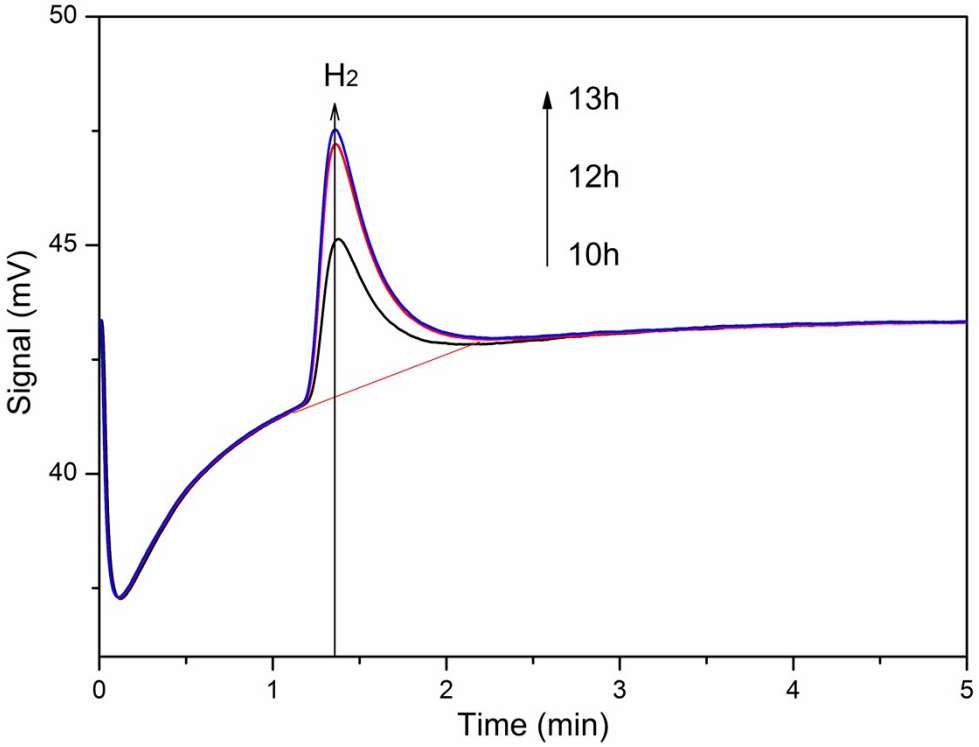
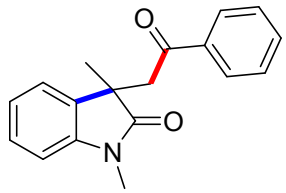
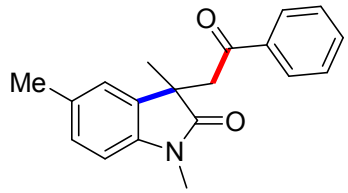


Figure S4. Determination of the resulting H_2 gas by GC

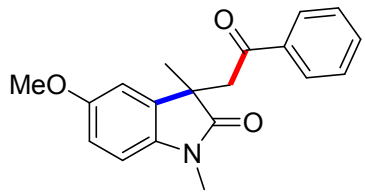
5. Characterization data for the products



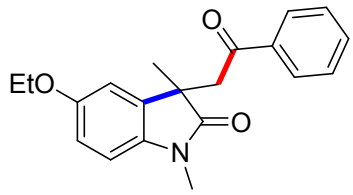
3a:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.84 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H) 7.40 (t, *J* = 7.6 Hz, 2H), 7.28–7.25 (m, 1H), 7.15 (d, *J* = 7.2 Hz, 1H)，6.99 (t, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 7.6 Hz, 1H), 3.75–3.64 (m, 2H), 3.32 (s, 3H), 1.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.01, 180.46, 143.75, 136.31, 133.62, 133.02, 128.36, 127.84, 127.73, 122.04, 121.67, 108.03, 45.92, 45.20, 26.33, 24.79.



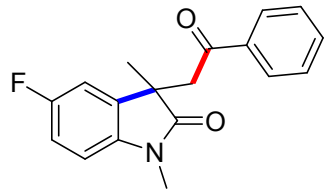
3b:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86 (d, *J* = 7.6 Hz, 2H), 7.53 (t, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.96 (s, 1H), 6.80 (d, *J* = 8.0 Hz, 1H), 3.74–3.64 (m, 2H), 3.30 (s, 3H), 2.28 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.10, 180.52, 141.47, 136.40, 133.77, 133.13, 131.57, 128.47, 128.07, 128.00, 122.71, 107.86, 46.03, 45.32, 26.48, 25.02, 21.13.



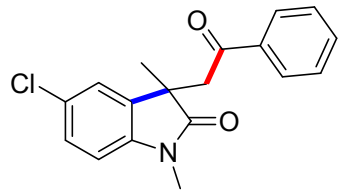
3c:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.84 (d, *J* = 7.6 Hz, 2H), 7.51 (t, *J* = 7.2 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 6.82–6.76 (m, 3H), 3.73 (s, 3H), 3.68–3.67 (m, 2H), 3.29 (s, 3H), 1.44 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.07, 180.22, 155.75, 137.45, 136.36, 135.20, 133.17, 128.49, 127.97, 111.47, 109.95, 108.30, 55.70, 45.99, 45.70, 26.54, 24.97.



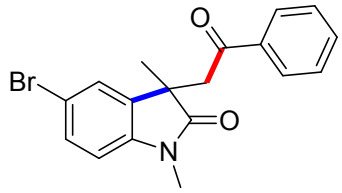
3d: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (d, J = 7.6 Hz, 2H), 7.52 (t, J = 7.2 Hz, 1H), 7.40 (t, J = 7.6 Hz, 2H), 6.81–6.75 (m, 3H), 3.97–3.92 (m, 2H), 3.72–3.62 (m, 2H), 3.29 (s, 3H), 1.43 (s, 3H), 1.36 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.05, 180.25, 155.05, 137.36, 136.34, 135.15, 133.17, 128.48, 127.97, 112.10, 110.55, 108.29, 63.96, 46.00, 45.67, 26.54, 24.98, 14.88. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{22}NO_3$: 324.1600, Found: 324.1604.



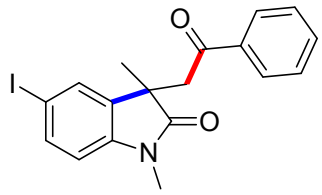
3e:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (d, *J* = 7.6 Hz, 2H), 7.56–7.52 (m, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 6.98–6.90 (m, 2H), 6.84–6.81 (m, 1H), 3.69 (s, 2H), 3.31 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.92, 180.23, 159.13 (d, *J* = 238.5 Hz), 139.81 (d, *J* = 2.0 Hz) , 136.16, 135.46 (d, *J* = 7.8 Hz), 133.34, 128.56, 127.96, 113.84 (d, *J* = 23.2 Hz), 110.16 (d, *J* = 24.7 Hz), 108.51 (d, *J* = 8.2 Hz), 45.97, 45.72 (d, *J* = 1.8 Hz), 26.61, 24.80.



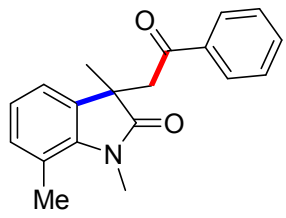
3f:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.6 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 1H), 7.11 (s, 1H), 6.84 (d, *J* = 8.4Hz, 1H), 3.71 (s, 2H), 3.32 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.82, 180.11, 142.52, 136.05, 135.57, 133.39, 128.58, 127.98, 127.72, 127.46, 122.31, 109.06, 46.07, 45.43, 26.59, 24.86.



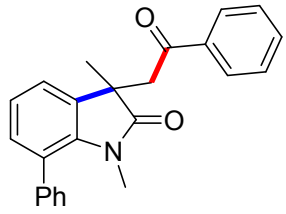
3g:[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.44–7.38 (m, 3H), 7.24 (s, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 3.70 (s, 2H), 3.31 (s, 3H), 1.43 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:195.79, 179.99, 143.03, 136.04, 135.97, 133.39, 130.63, 128.58, 127.98, 125.00, 114.78, 109.60, 46.10, 45.38, 26.57, 24.89.



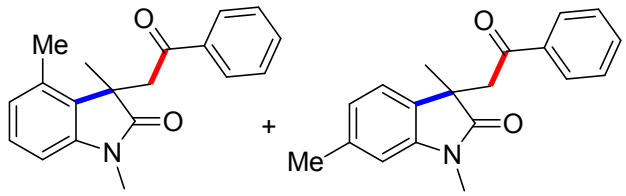
3h:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (d, *J* = 7.6 Hz, 2H), 7.58–7.52 (m, 2H), 7.43–7.40 (m, 3H), 6.70 (d, *J* = 8.0 Hz, 1H), 3.70 (s, 2H), 3.30 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.80, 179.83, 143.72, 136.64, 136.35, 136.02, 133.40, 130.47, 128.58, 128.00, 110.26, 84.68, 46.12, 45.19, 26.53, 24.93.



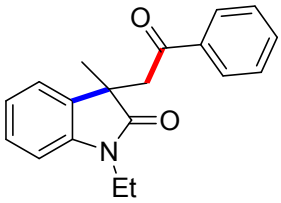
3i:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.84 (d, *J* = 7.6 Hz, 2H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 6.97 (t, *J* = 7.2 Hz, 2H), 6.86 (t, *J* = 7.2 Hz, 1H), 3.74–3.64 (m, 2H), 3.60 (s, 3H), 2.63 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:196.19, 181.38, 141.62, 136.43, 134.39, 133.11, 131.60, 128.46, 127.96, 122.06, 119.73, 119.51, 46.34, 44.65, 29.83, 25.50, 19.13.



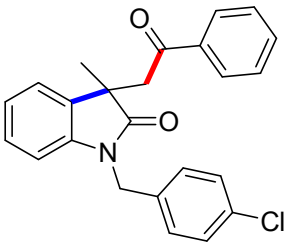
3j: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.87 (d, *J* = 8.0 Hz, 2H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.44–7.41 (m, 7H), 7.15–7.09 (m, 2H), 7.00 (t, *J* = 7.2 Hz, 1H), 3.80–3.69 (m, 2H), 2.85 (s, 3H), 1.51 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.19, 181.73, 140.85, 139.23, 136.45, 134.82, 133.16, 130.85, 128.50, 127.99, 127.51, 125.49, 121.50, 120.71, 46.49, 44.67, 30.55, 25.45. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{24}H_{22}NO_2$: 356.1651, Found: 356.1654.



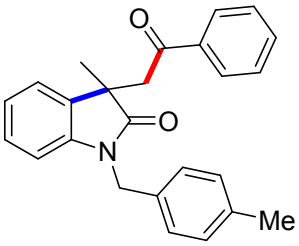
3k+3k' (3 : 2):[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.86–7.81 (m, 3.31H), 7.52 (t, *J* = 7.6 Hz, 1.76H), 7.40 (t, *J* = 7.6 Hz, 3.45H), 7.16 (t, *J* = 7.6 Hz, 1.09H), 7.03 (d, *J* = 7.2 Hz, 0.62H), 6.80 (d, *J* = 7.2 Hz, 0.69H), 6.75 (d, *J* = 7.6 Hz, 2.58H), 4.00–3.95 (m, 1.00H), 3.73–3.62 (m, 2.47H), 3.31 (s, 1.95H), 3.28 (s, 3.10H), 2.38 (s, 2.01H), 2.31 (s, 3.04H), 1.50 (s, 3.00H), 1.44 (s, 2.02H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.33, 196,24, 180,91, 180,47, 144.09, 143.89, 137.85, 136.41, 136.21, 133.15, 133.13, 132,77, 130.79, 130.40, 128.49, 128,48, 127.98, 127.94, 127.66, 124.78, 122.65, 121.53, 109.20, 106.00, 46.12. 46.02, 45.09, 45.01, 26.52, 26.43, 25.01, 22.90, 21.83, 18.25.



3l:[3] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.85 (d, *J* = 8.0 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.25 (t, *J* = 7.6 Hz, 1H), 7.15 (d, *J* = 7.6Hz, 1H), 6.99–6.92 (m, 2H), 3.95–3.79 (m, 2H), 3.76–3.65 (m, 2H), 1.45 (s, 3H), 1.36 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.00, 180.16, 142.88, 136.44, 134.01, 133.11, 128.47, 127.98, 127.75, 121.94, 108.33, 45.96, 45.28, 34.76, 25.06, 12.38.

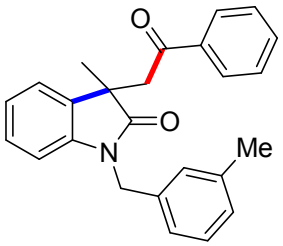


3m: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.45–7.42 (m, 4H), 7.35–7.33 (m, 2H), 7.16–7.13 (m, 2H), 6.97 (t, *J* = 7.2 Hz, 1H), 6.72 (d, *J* = 8.0 Hz, 1H), 5.08–4.95 (m, 2H), 3.84–3.74 (m, 2H), 1.51 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:195.88, 180.62, 142.63, 136.26, 134.81, 133.79, 133.29, 133.23, 128.90, 128.78, 128.55, 128.04, 127.75, 122.39, 121.69, 109.14, 45.90, 45.35, 43.36, 25.67. HRMS (ESI) ($[M+H]^+$) Calcd. For $C_{24}H_{21}ClNO_2$: 390.1261, Found: 390.1259.

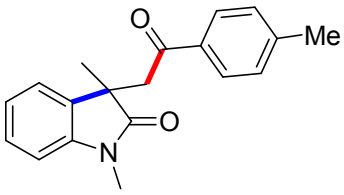


3n: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 7.6 Hz, 2H), 7.18–7.11 (m, 4H), 6.94 (t, *J* = 7.2 Hz, 1H), 6.77 (d,

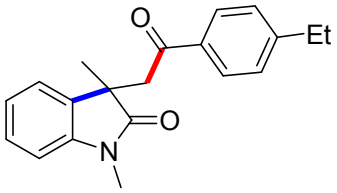
J = 7.6 Hz, 1H), 5.09–4.92 (m, 2H), 3.81–3.71 (m, 2H), 2.34 (s, 3H), 1.51 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:195.93, 180.59, 142.94, 137.02, 136.39, 133.78, 133.22, 133.19, 129.40, 128.51, 128.04, 127.70, 127.29, 122.15, 121.69, 109.33, 45.81, 45.39, 43.74, 25.49, 21.13. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{25}H_{24}NO_2$: 370.1807, Found: 370.1808.



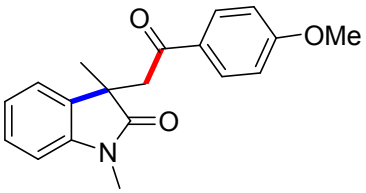
3o: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.91 (d, *J* = 7.6 Hz, 2H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.30–7.25 (m, 3H), 7.17–7.10 (m, 3H), 6.96 (t, *J* = 7.6 Hz, 1H), 6.78 (d, *J* = 7.6 Hz, 1H), 5.09–4.95 (m, 2H), 3.84–3.73 (m, 2H), 2.37 (s, 3H), 1.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.86, 180.64, 143.00, 138.39, 136.40, 136.21, 133.81, 133.20, 128.57, 128.53, 128.18, 128.05, 128.03, 127.73, 124.31, 122.18, 121.67, 109.34, 45.84, 45.40, 43.95, 25.56, 21.48. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{25}H_{24}NO_2$: 370.1807, Found: 370.1809.



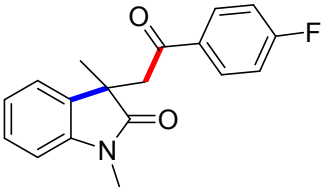
3p:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (d, *J* = 7.6 Hz, 2H), 7.26–7.19 (m, 3H), 7.14 (d, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.6Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 3.74–3.61 (m, 2H), 3.33 (s, 3H) 2.38 (s, 3H) 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.60, 180.58, 143.86, 143.75, 133.81, 133.73, 129.05, 127.98, 127.69, 122.01, 121.63, 108.02, 45.79, 45.19, 26.35, 24.82, 21.51.



3q: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.78 (d, *J* = 8.0 Hz, 2H), 7.27–7.21 (m, 3H), 7.14 (d, *J* = 7.2 Hz, 1H), 6.98 (t, *J* = 7.2 Hz, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 3.75–3.62 (m, 2H), 3.33 (s, 3H), 2.67 (q, *J* = 7.6 Hz, 2H), 1.45 (s, 3H), 1.23 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.63, 180.58, 150.02, 143.75, 134.04, 133.73, 128.09, 127.87, 127.69, 122.01, 121.64, 108.02, 45.82, 45.19, 28.79, 26.35, 24.83, 15.04. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{20}H_{22}NO_2$: 308.1651, Found: 308.1658.

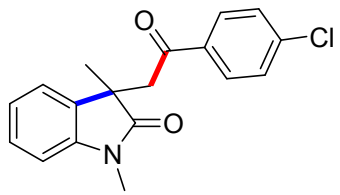


3r:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.83 (d, *J* = 8.4 Hz, 2H), 7.27–7.24 (m, 1H), 7.14 (d, *J* =7.2 Hz, 1H), 6.98 (t, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 8.0 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 2H), 3.84 (s, 3H), 3.71–3.59 (m, 2H), 3.32 (s, 3H), 1,44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.47, 180.62, 163.39, 143.73, 133.80, 130.15, 129.42, 127.66, 121.99, 121.64, 113.50, 108.01, 55.33, 45.56, 45.23, 26.34, 24.84.

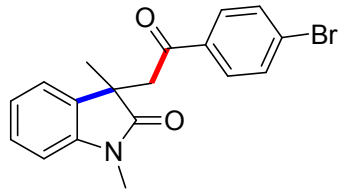


3s:[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89–7.85 (m, 2H), 7.27 (t, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 7.2 Hz, 1H), 7.07 (t, *J* = 8.4 Hz, 2H), 6.99 (t, *J* = 7.2 Hz, 1H), 6.91 (d, *J* = 7.6 Hz, 1H), 3.71–3.61 (m, 2H), 3.32 (s, 3H), 1.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.53, 180.48, 165.75 (d, *J* = 253.5 Hz), 143.83, 133.62, 132.81 (d, *J* = 3.0 Hz), 130.63 (d, *J* = 9.3 Hz),

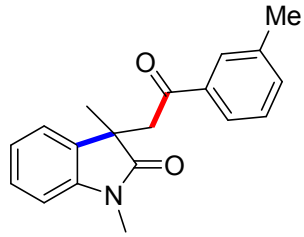
127.91, 122.19, 121.73, 115.60 (d, J = 21.7 Hz), 108.19, 45.92, 45.29, 26.46, 24.94.



3t:[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.77 (d, J = 8.4 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H)　7.28–7.24 (m, 1H), 7.14 (d, J = 7.2 Hz, 1H)，6.99 (t, J = 7.6 Hz, 1H), 6.91 (d, J = 8.0 Hz, 1H), 3.70–3.60 (m, 2H), 3.31 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 194.82, 180.31, 143.71, 139.55, 134.54, 133.42, 129.28, 128.71, 127.84, 122.11, 121.63, 108.10, 45.85, 45.16, 26.35, 24.80.

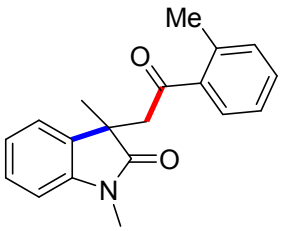


3u:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.70 (d, J = 8.4 Hz, 2H), 7.55 (d, J = 8.4 Hz, 2H), 7.27 (t, J = 7.2 Hz, 1H), 7.14 (d, J = 7.2 Hz, 1H)，6.99 (t, J = 7.6 Hz, 1H), 6.91 (d, J = 7.6 Hz, 1H), 3.69–3.59 (m, 2H), 3.31 (s, 3H), 1.45 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.02, 180.28, 143.70, 134.93, 133.39, 131.70, 129.37, 128.28, 127.84, 122.11, 121.62, 108.10, 45.82, 45.14, 26.35, 24.80.

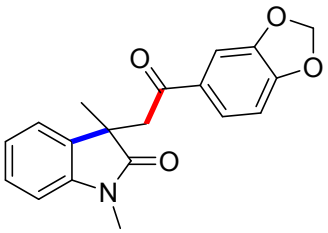


3v:[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (d, J = 8.0 Hz, 2H), 7.27–7.24 (m, 1H), 7.20 (d, J = 8.0 Hz, 2H)，7.14 (d, J = 7.2 Hz, 1H)，6.98 (t, J = 7.2 Hz, 1H), 6.91 (d, J = 7.6 Hz, 1H), 3.74–3.61 (m, 2H), 3.32 (s, 3H), 2.38 (s, 3H), 1.44 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:

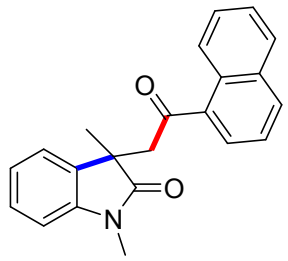
195.60, 180.57, 143.85, 143.75, 133.82, 133.73, 129.05, 127.98, 127.69, 122.01, 121.64, 108.02, 45.80, 45.19, 26.35, 24.82, 21.50.



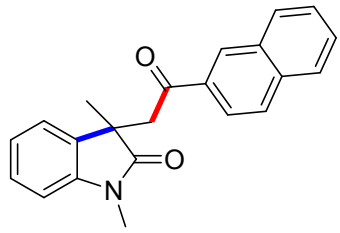
3w:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51 (d, *J* = 7.6 Hz, 1H), 7.33–7.16 (m, 4H), 7.12 (d, *J* = 7.2 Hz, 1H), 7.01 (t, *J* = 7.2 Hz, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 3.65–3.51 (m, 2H), 3.22 (s, 3H), 2.11 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 200.54, 180.22, 143.70, 137.67, 137.47, 133.26, 131.47, 131.01, 128.08, 127.81, 125.29, 122.09, 121.94, 108.06, 49.01, 45.67, 26.26, 24.77, 20.40.



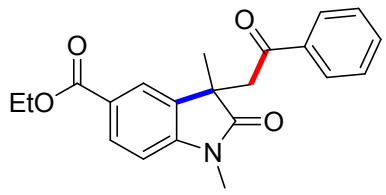
3x: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.47 (dd, *J* = 8.4 Hz, 1.2 Hz, 1H), 7.27–7.23 (m, 2H), 7.13 (d, *J* = 7.2 Hz, 1H), 6.97 (t, *J* = 7.2 Hz, 1H), 6.90 (d, *J* = 8.0 Hz, 1H), 6.78 (d, *J* = 8.0 Hz, 1H), 5.97 (s, 2H), 3.67–3.55 (m, 2H), 3.31(s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:194.09, 180.64, 151.79, 148.07, 143.85, 133.84, 131.31, 127.79, 124.25, 122.11, 121.72, 108.12, 107.73, 107.71, 101.80, 45.72, 45.36, 26.43, 24.92. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{19}H_{18}NO_4$: 324.1236, Found: 324.1236.



3y:[2] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.96–7.91 (m, 2H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.76 (d, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.41–7.38 (m, 1H), 7.26–7.22 (m, 2H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.78 (d, *J* = 8.0 Hz, 1H), 3.83–3.63 (m, 2H), 3.15(s, 3H), 1.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 200.81, 180.25, 143.77, 135.61, 133.68, 133.22, 132.37, 129.72, 128.11, 127.97, 127.60, 127.16, 126.32, 125.48, 124.14, 122.30, 122.16, 108.24, 49.68, 46.01, 26.28, 24.81.

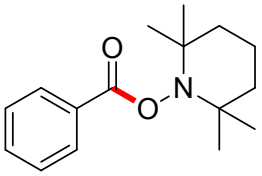


3z:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.41 (s, 1H), 7.93 (d, *J* = 8.0 Hz, 1H), 7.88–7.81 (m, 3H), 7.61–7.52 (m, 2H), 7.29–7.25 (m, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 3.91–3.78 (m, 2H), 3.34 (s, 3H), 1.51 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 196.09, 180.65, 143.89, 135.57, 133.78, 133.76, 132.39, 129.70, 129.53, 128.50, 128.34, 127.87, 127.74, 126.78, 123.66, 122.20, 121.87, 108.17, 46.12, 45.44, 26.47, 24.93.

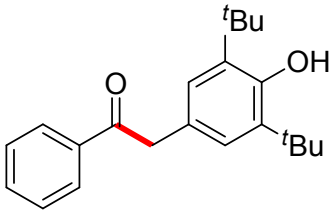


3aa:[1] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.02 (d, *J* = 8.0 Hz, 1H), 7.83 (d, *J* = 7.6 Hz, 2H), 7.78

(s, 1H), 7.52 (t, J = 7.6 Hz, 1H), 7.40 (t, J = 7.6 Hz, 2H), 6.94 (t, J = 8.0 Hz, 1H), 4.32 (q, J = 7.2 Hz, 2H), 3.83–3.71 (m, 2H), 3.36 (s, 3H), 1.45 (s, 3H), 1.35 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 195.84, 180.90, 166.50, 148.16, 136.04, 133.84, 133.31, 130.62, 128.52, 127.96, 124.37, 122.65, 107.60, 60.73, 46.27, 44.95, 26.65, 24.96, 14.36.



4:[4] ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07 (d, J = 7.6 Hz, 2H), 7.56 (t, J = 7.2 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 1.81–1.44 (m, 6H), 1.27 (s, 6H), 1.12 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.34, 132.85, 129.73, 129.54, 128.45, 60.38, 39.07, 31.97, 20.85, 17.01.



5: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.2 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 7.07 (s, 2H), 5.14 (s, 1H), 4.22 (s, 2H), 1.43 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 198.32, 152.71, 136.94, 135.99, 132.97, 128.60, 128.56, 126.18, 124.97, 45.23, 34.30, 30.27. HRMS (ESI) ($[M+H]^+$) Calcd. for $C_{22}H_{29}O_2$: 325.2168, Found: 325.2170.

6. References

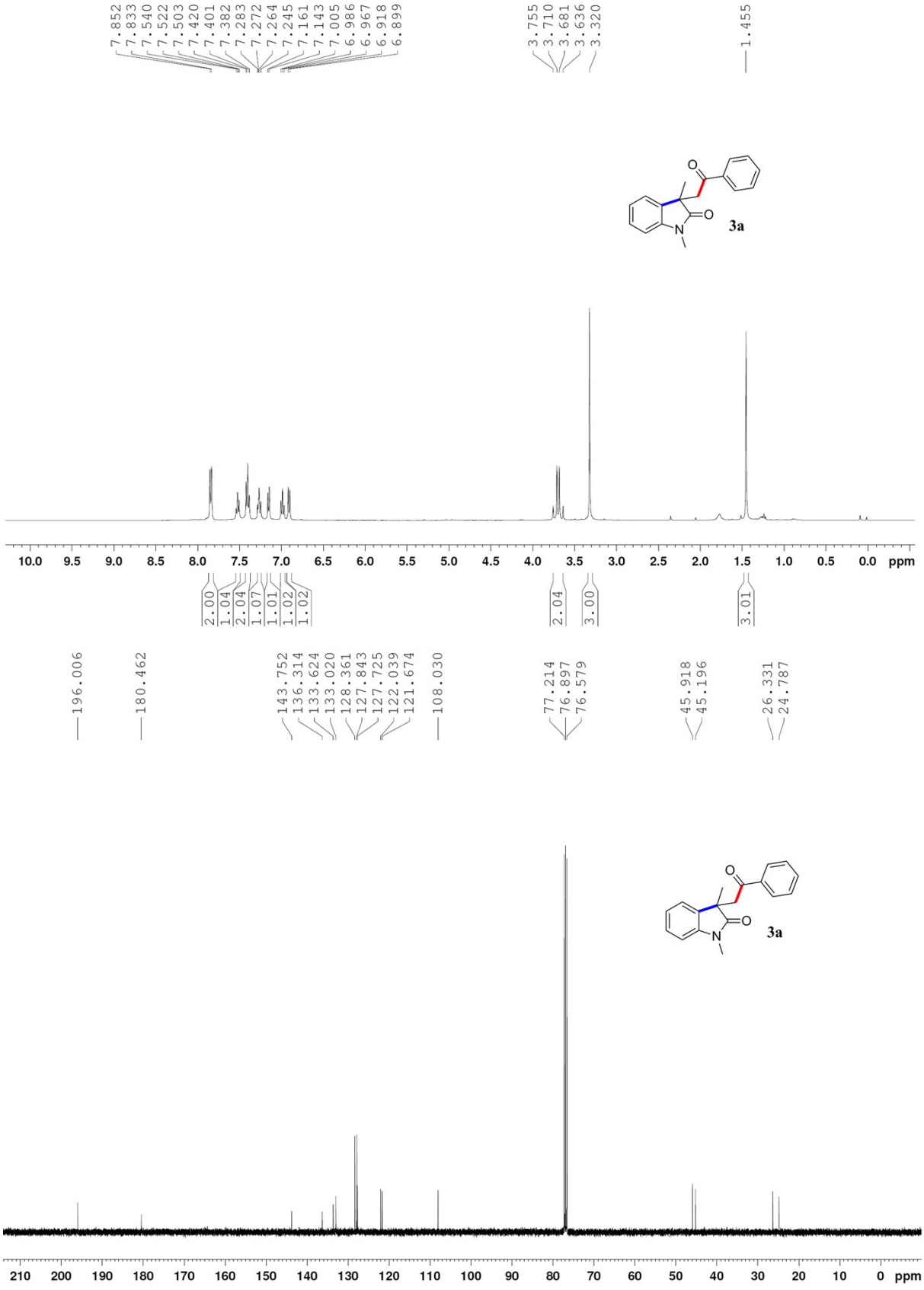
[1] H. Wang, L. Guo and X. Duan, *Adv. Synth. Catal.,* 2013, **355**, 2222–2226.

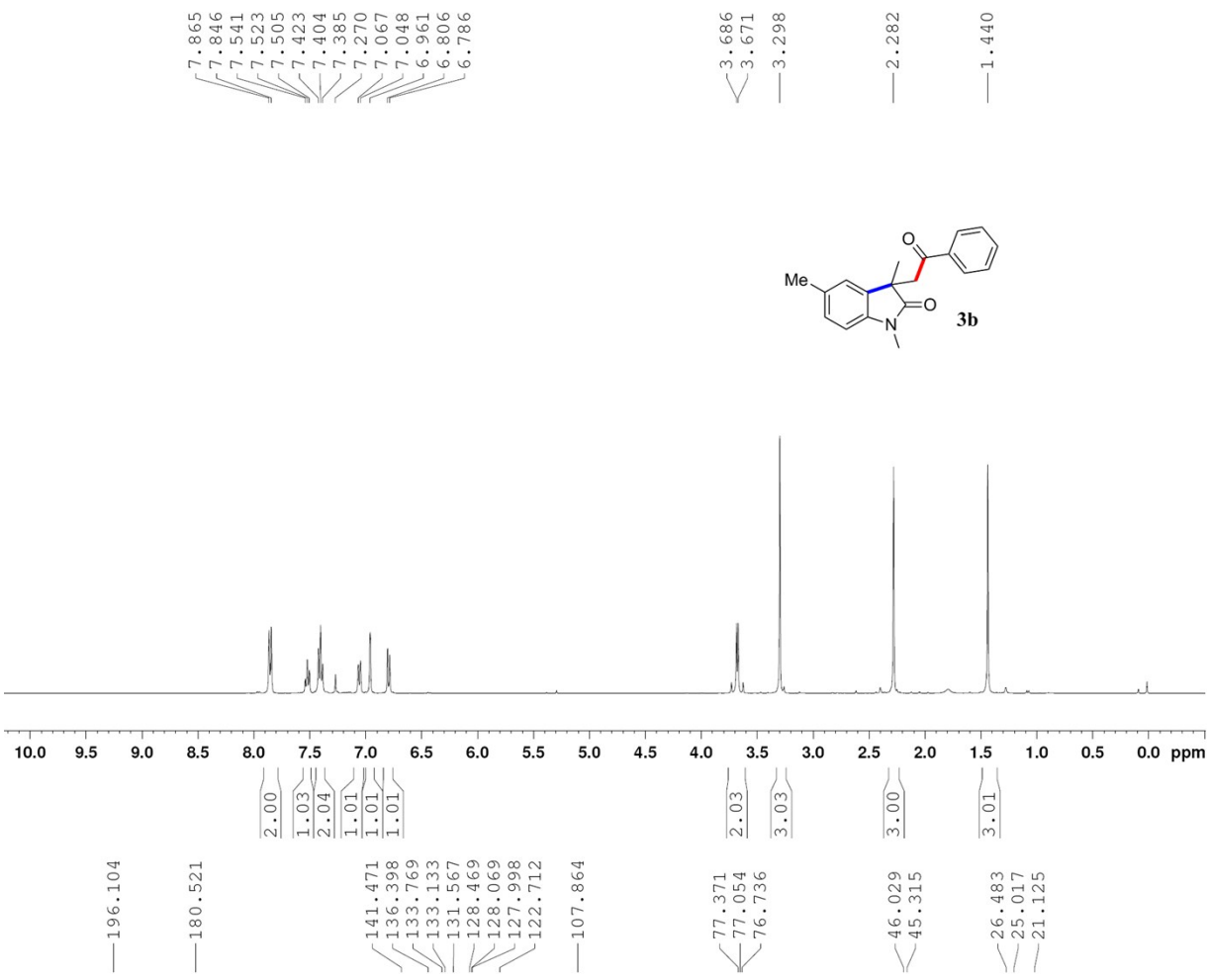
[2] L. Zheng, H. Huang, C. Yang and W. Xia, *Org. Lett.,* 2015, **17**, 1034−1037.

[3] W. Gong, L. Xu, T. Ji, P. Xie, X. Qi, C. U. Pittman Jr and A. Zhou, *RSC Adv.,* 2014, **4**, 6854−6857.

[4] J. Liu, Q. Liu, H. Yi, C. Qin, R. Bai, X. Qi, Y. Lan and A. Lei, *Angew. Chem., Int. Ed.,* 2014, **53**, 502–506.

7. ^{1}H and ^{13}C NMR spectra of the products

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7.385
7.270
7.067
7.048
6.961
6.806
6.786
3.686
3.671
3.298
2.282
1.440
Me
O
O
N
3b
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 ppm
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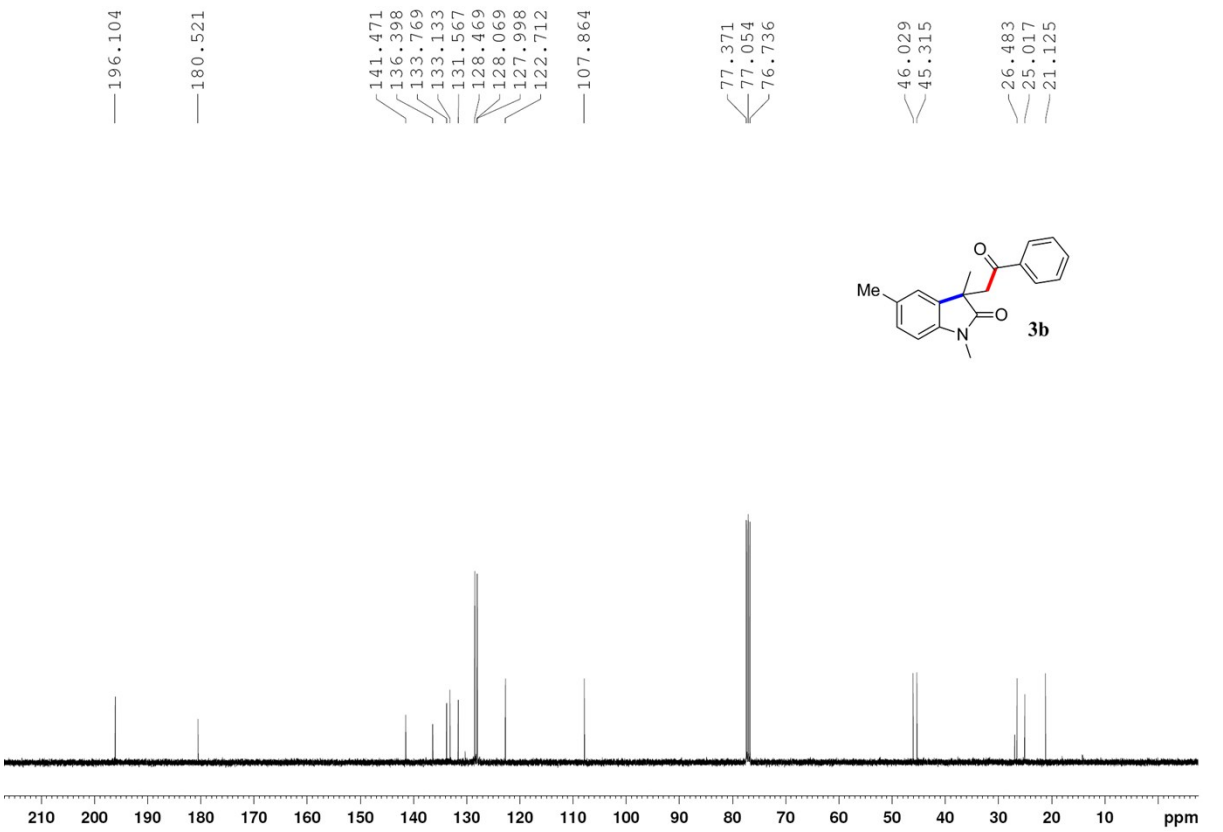
1.03
2.04
1.01
1.01
1.01
2.03
3.03
3.00
3.01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180.521
141.471
136.398
133.769
133.133
131.567
128.469
128.069
127.998
122.712
107.864
77.371
77.054
76.736
46.029
45.315
26.483
25.017
21.125
Me
O
O
N
3b
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


7.853
7.834
7.529
7.511
7.493
7.410
7.391
7.372
7.271
6.819
6.797
6.778
6.760
3.729
3.679
3.671
3.290
1.436
MeO
O
O
N
3c
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 ppm
2.00
1.01
2.01
3.04
3.00
2.00
3.03
3.05
196.072
180.221
155.751
137.447
136.358
135.199
133.174
128.487
127.968
111.468
109.947
108.304
77.424
77.106
76.787
55.702
45.988
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MeO
O
O
N
3c
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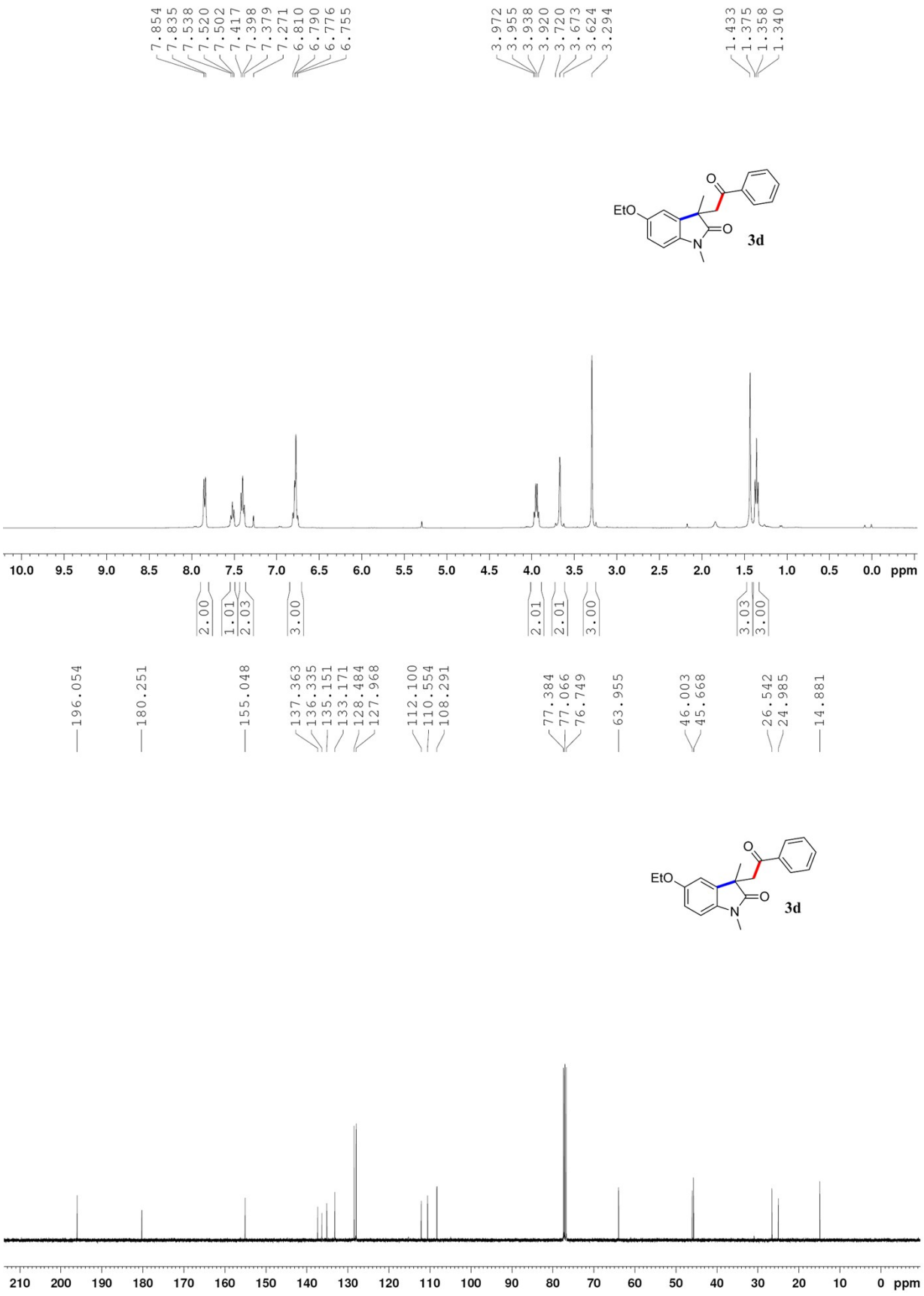
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7.835
7.538
7.520
7.502
7.417
7.398
7.379
7.271
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6.790
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6.755
3.972
3.955
3.938
3.920
3.720
3.673
3.624
3.294
1.433
1.375
1.358
1.340
EtO
O
O
N
3d
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 ppm
2.00
1.01
2.03
3.00
2.01
2.01
3.00
3.03
3.00
196.054
180.251
155.048
137.363
136.335
135.151
133.171
128.484
127.968
112.100
110.554
108.291
77.384
77.066
76.749
63.955
46.003
45.668
26.542
24.985
14.881
EtO
O
O
N
3d
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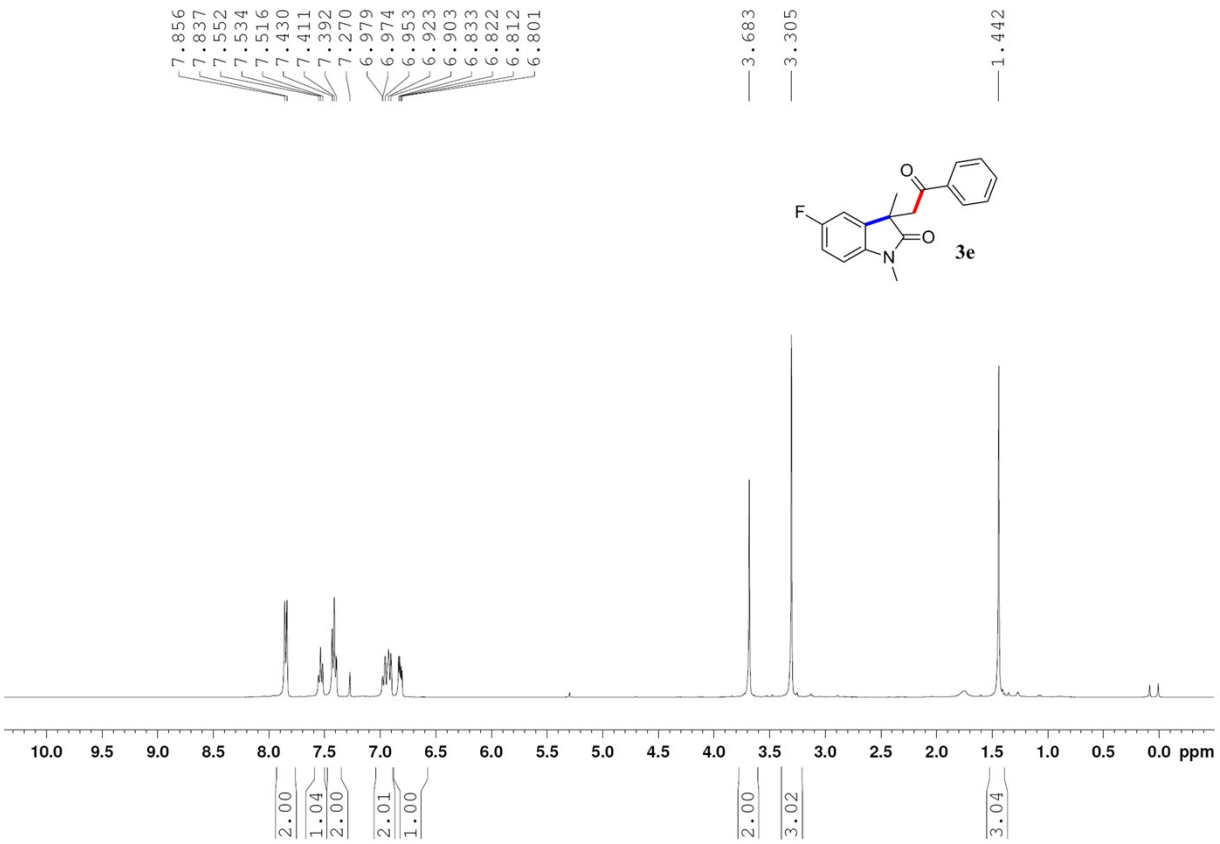
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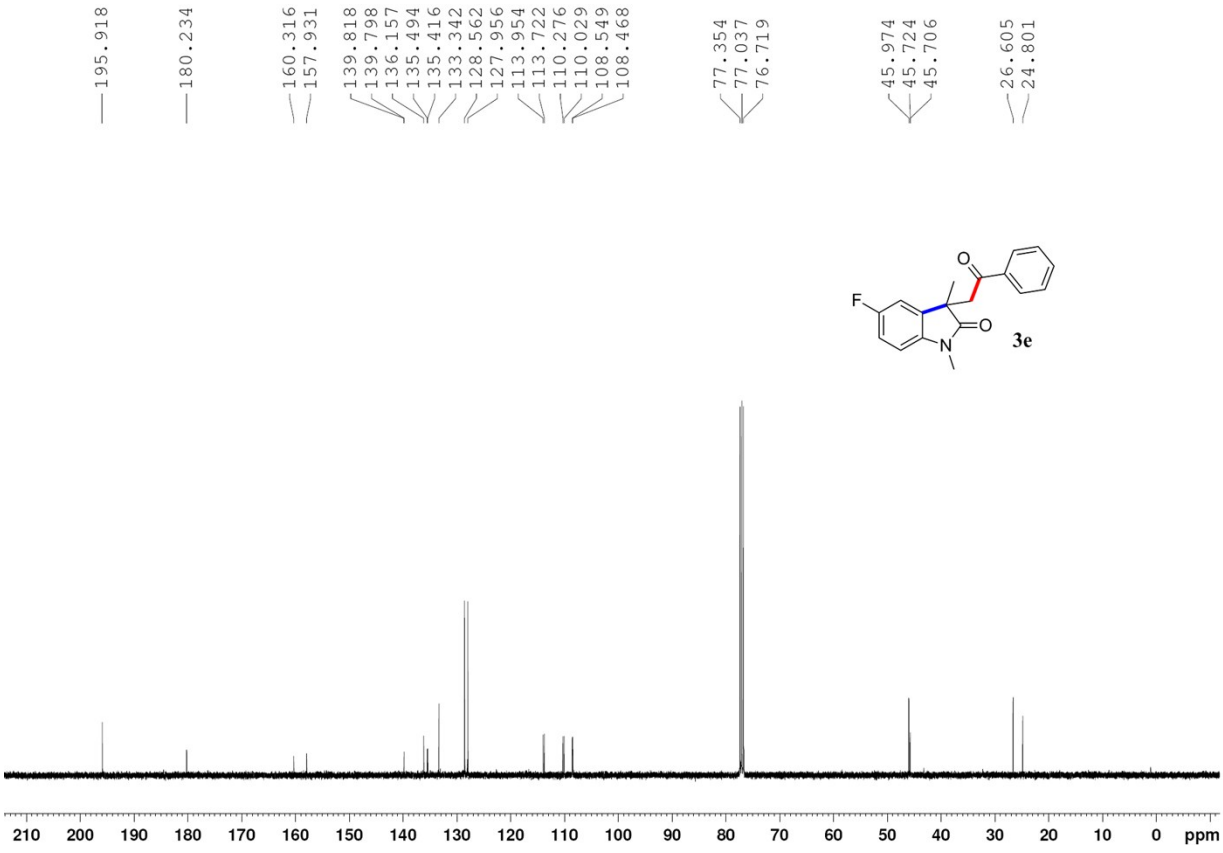
7.837
7.552
7.534
7.516
7.430
7.411
7.392
7.270
6.979
6.974
6.953
6.923
6.903
6.833
6.822
6.812
6.801
3.683
3.305
1.442
F
O
O
N
3e
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.04
2.00
2.01
1.00
2.00
3.02
3.04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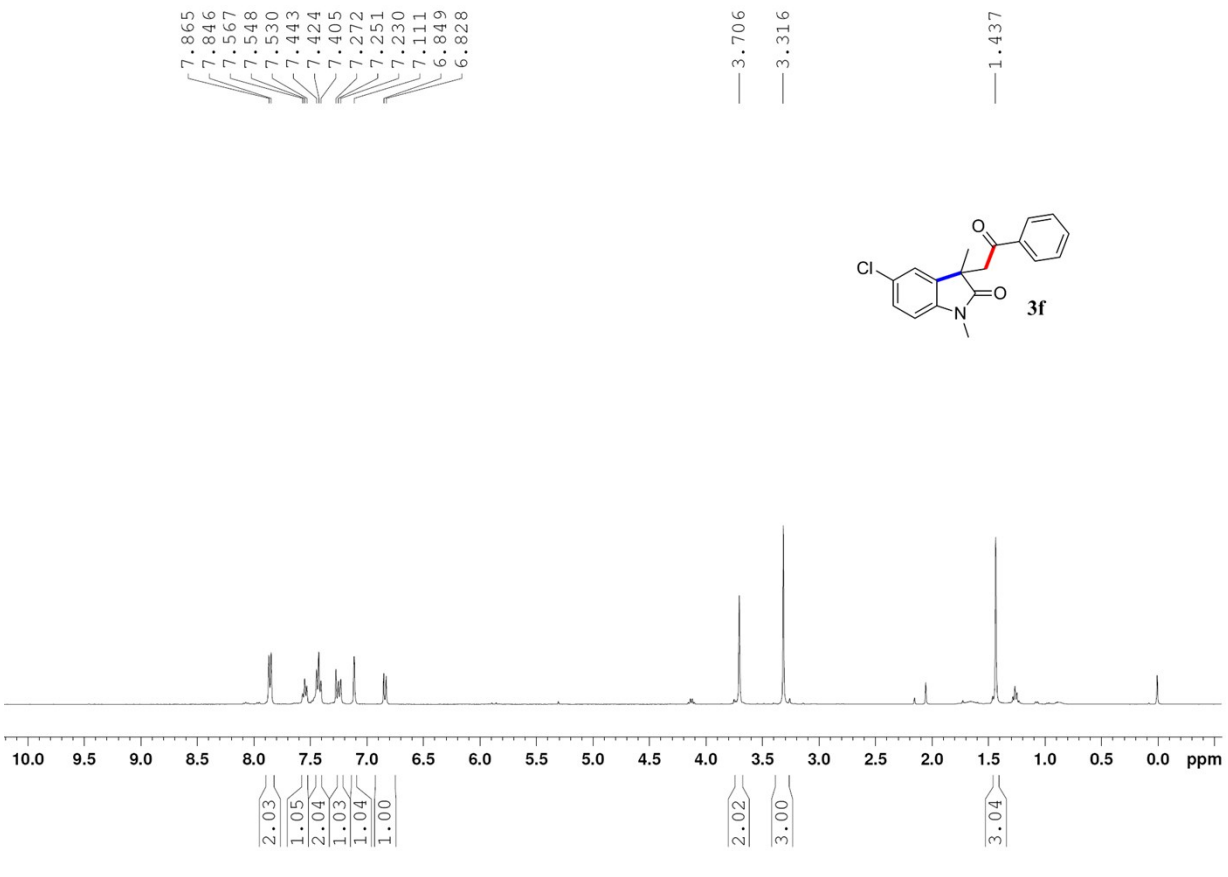
160.316
157.931
139.818
139.798
136.157
135.494
135.416
133.342
128.562
127.956
113.954
113.722
110.276
110.029
108.549
108.468
77.354
77.037
76.719
45.974
45.724
45.706
26.605
24.801
F
O
O
N
3e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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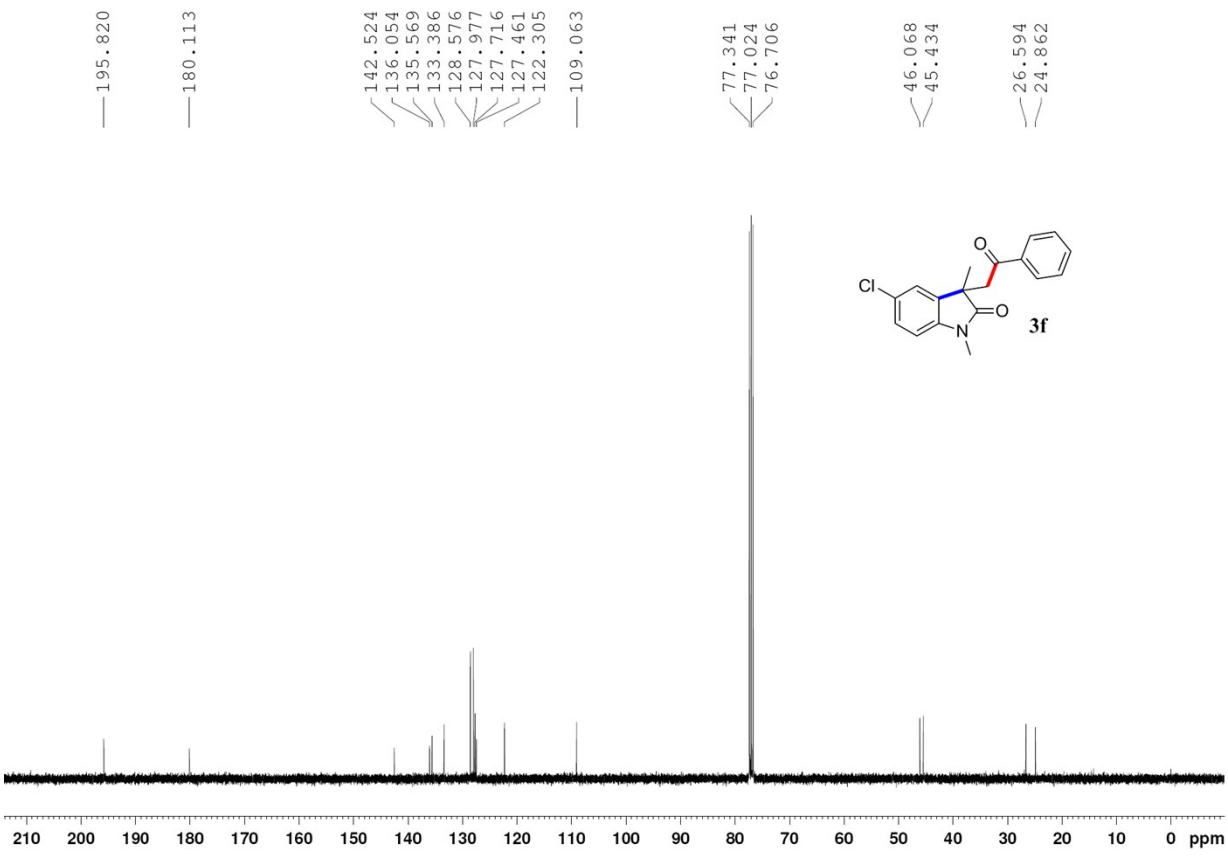
7.846
7.567
7.548
7.530
7.443
7.424
7.405
7.272
7.251
7.230
7.111
6.849
6.828
3.706
3.316
1.437
Cl
O
N
3f
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.03
1.05
2.04
1.03
1.04
1.00
2.02
3.00
3.04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180.113
142.524
136.054
135.569
133.386
128.576
127.977
127.716
127.461
122.305
109.063
77.341
77.024
76.706
46.068
45.434
26.594
24.862
Cl
O
N
3f
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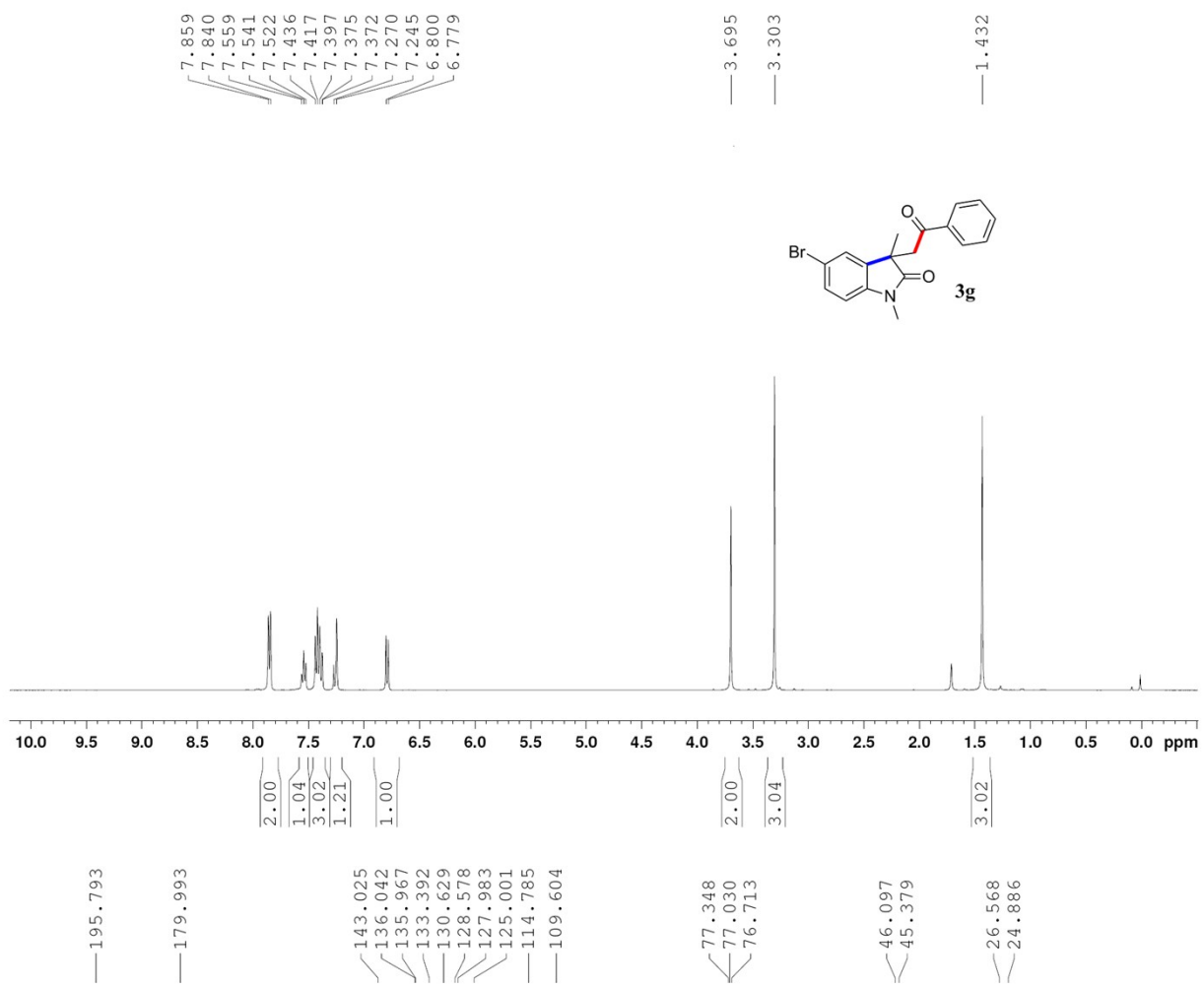
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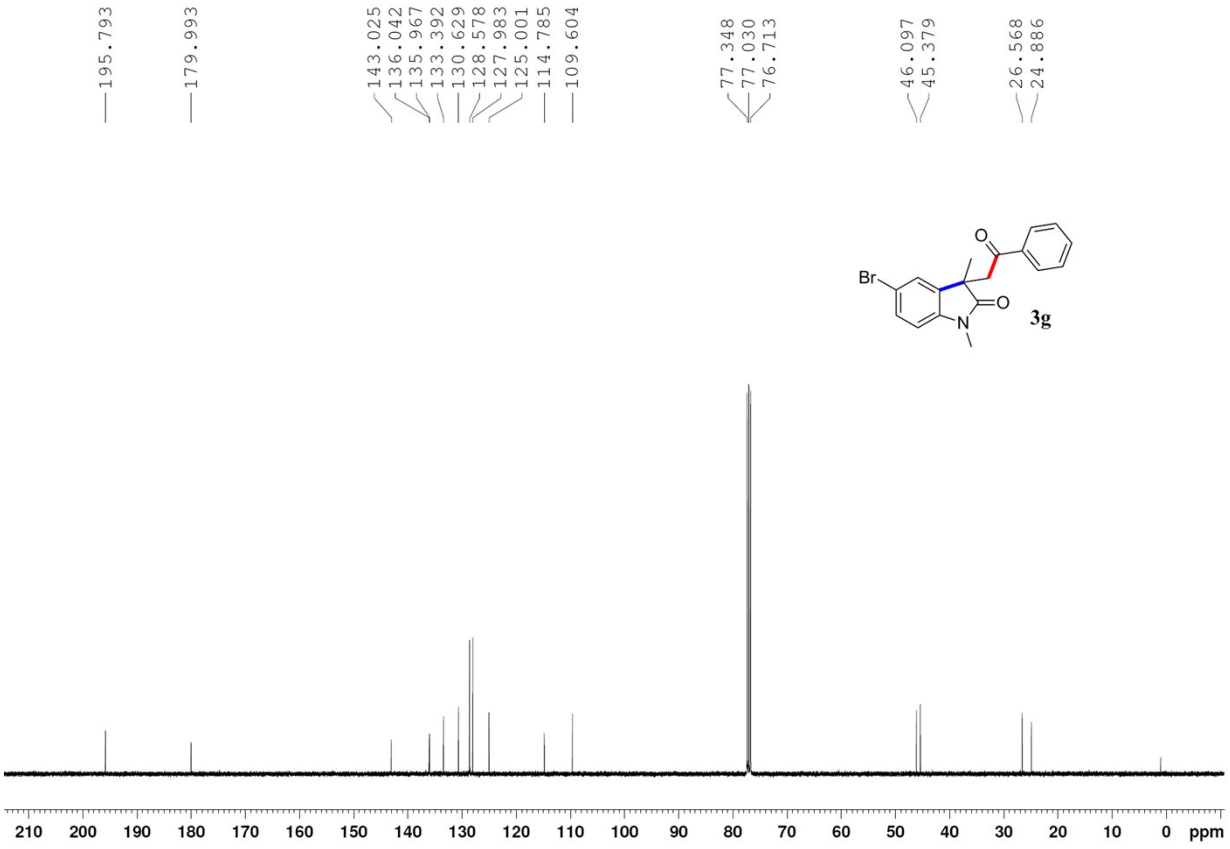
7.541
7.522
7.436
7.417
7.397
7.375
7.372
7.270
7.245
6.800
6.779
3.695
3.303
1.432
Br
O
O
N
3g
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.04
3.02
1.21
1.00
2.00
3.04
3.02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125.001
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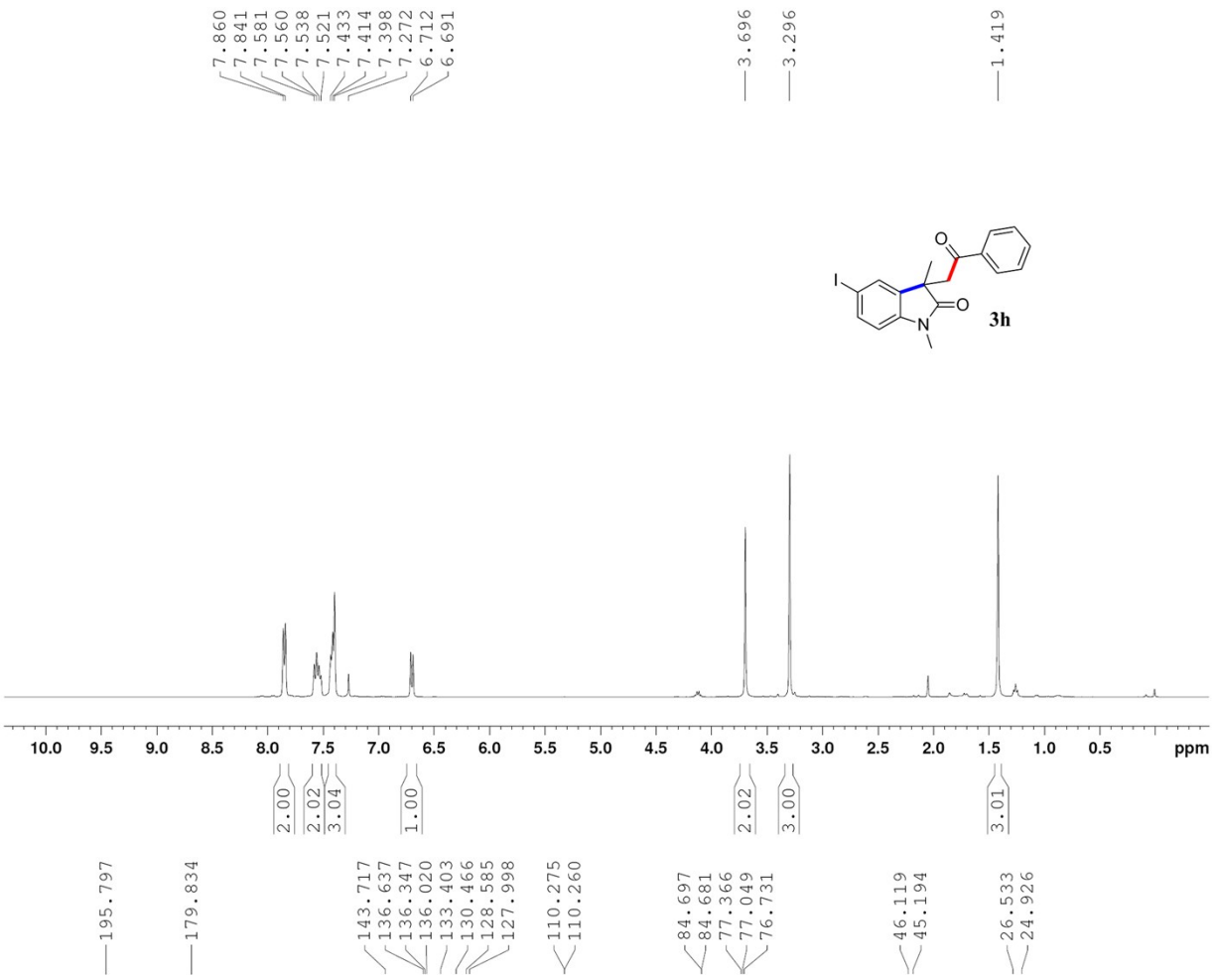
77.348
77.030
76.713
46.097
45.379
26.568
24.886
Br
O
O
N
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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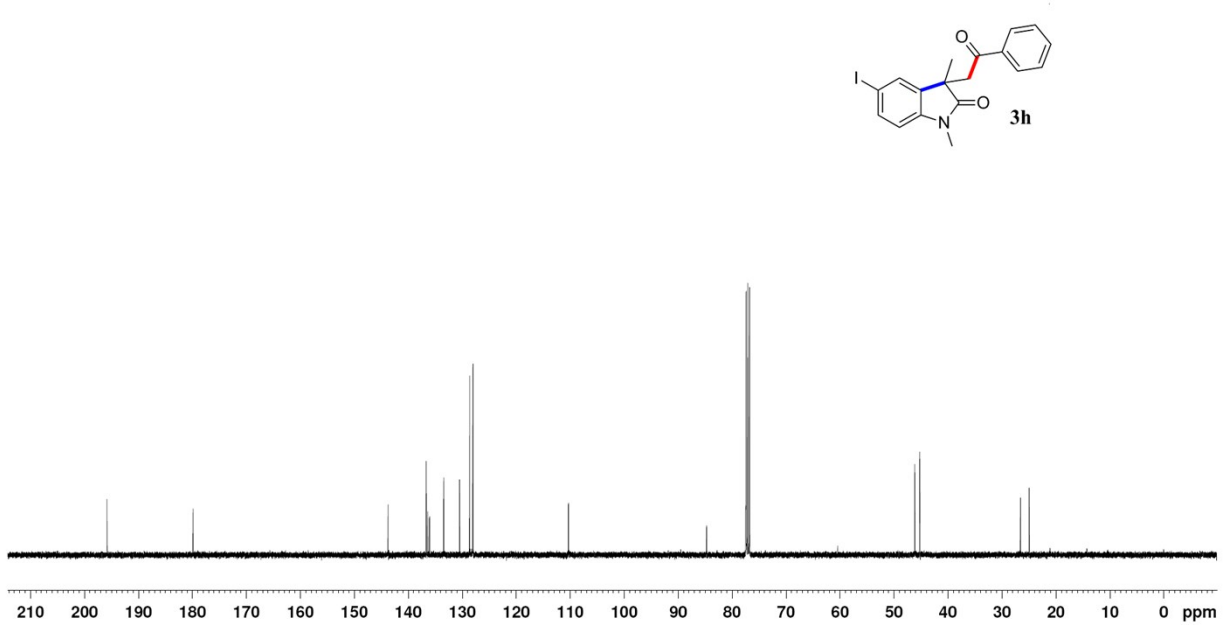
7.433
7.414
7.398
7.272
6.712
6.691
3.696
3.296
1.419
3h
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.02
3.04
1.00
2.02
3.00
3.01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136.347
136.020
133.403
130.466
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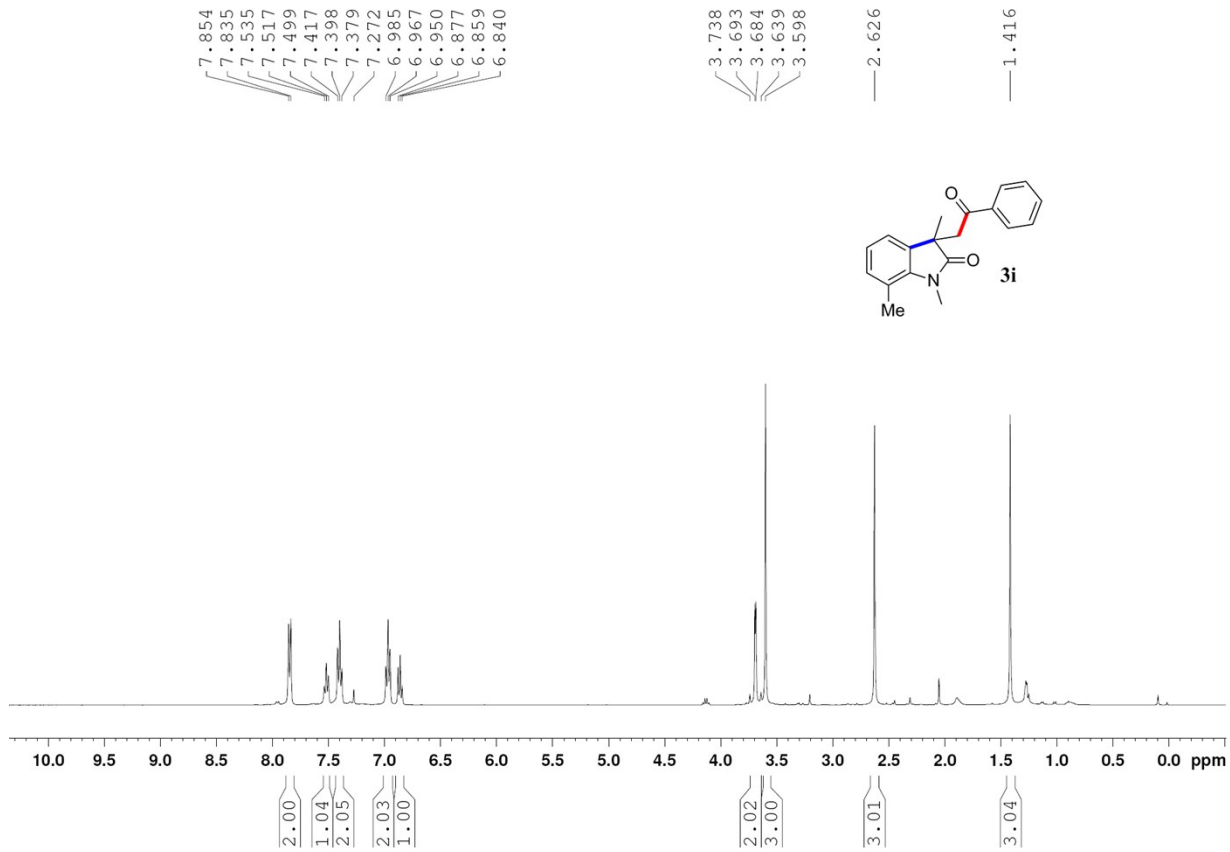
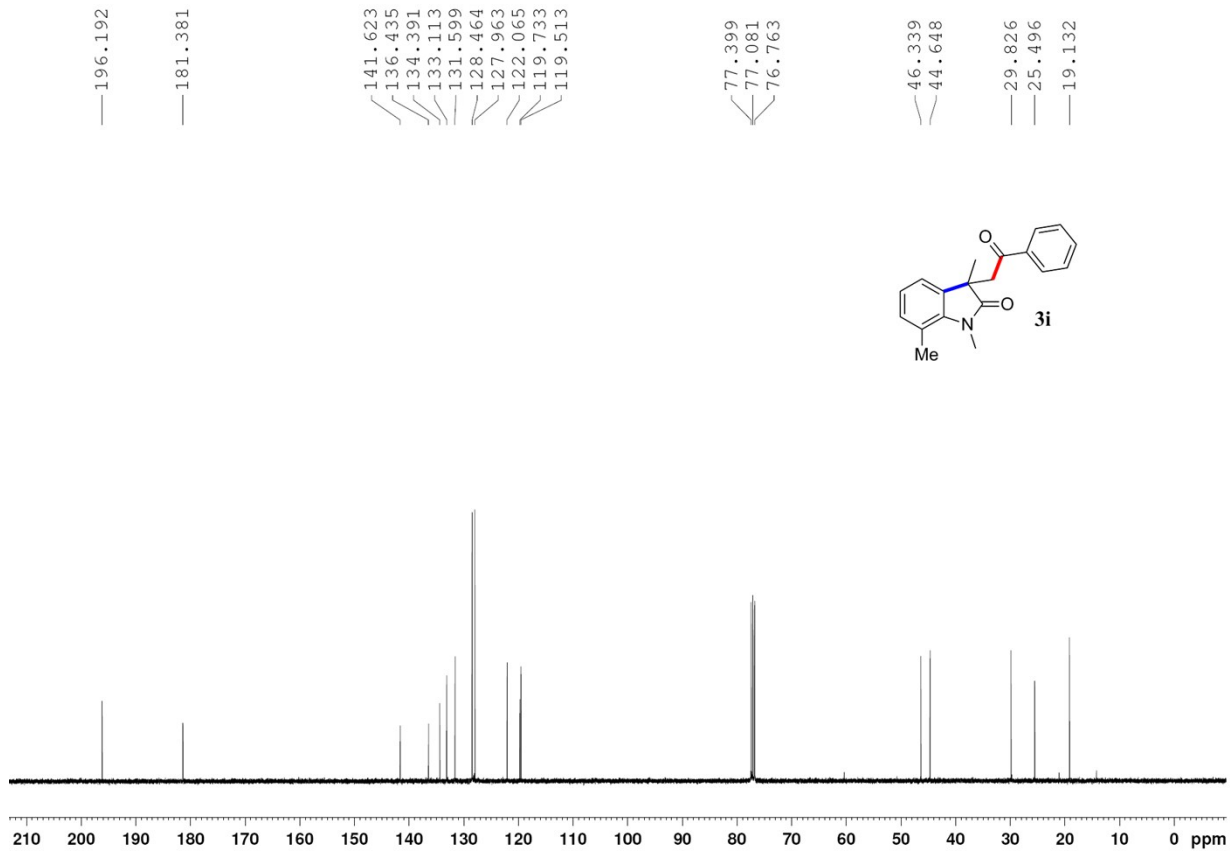
127.998
110.275
110.260
84.697
84.681
77.366
77.049
76.731
46.119
45.194
26.533
24.926
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


7.854
7.835
7.535
7.517
7.499
7.417
7.398
7.379
7.272
6.985
6.967
6.950
6.877
6.859
6.840
3.738
3.693
3.684
3.639
3.598
2.626
1.416
O
O
N
Me
3i
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 ppm
2.00
1.04
2.05
2.03
1.00
2.02
3.00
3.01
3.04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181.381
141.623
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134.391
133.113
131.599
128.464
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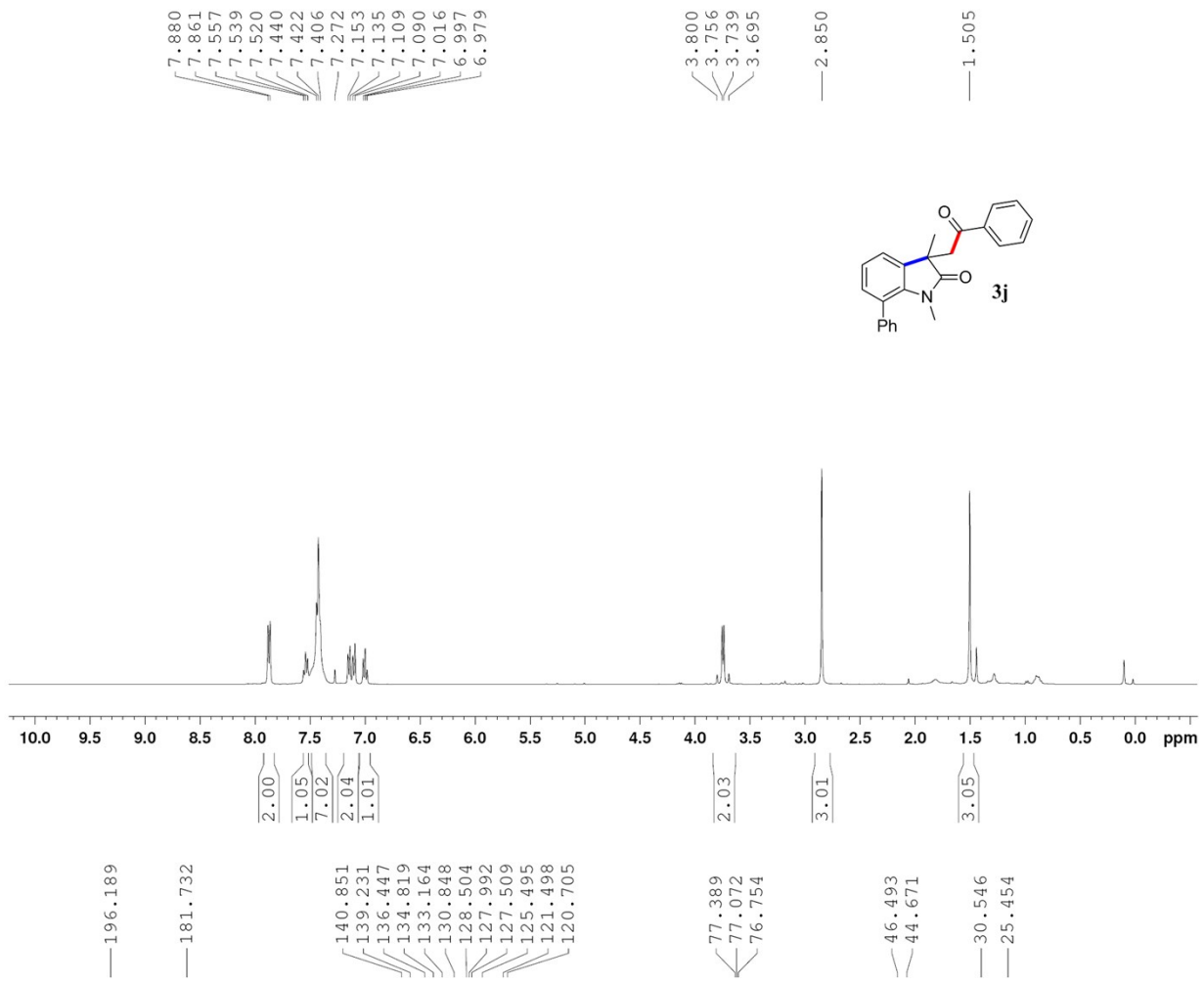
122.065
119.733
119.513
77.399
77.081
76.763
46.339
44.648
29.826
25.496
19.132
O
O
N
Me
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.406
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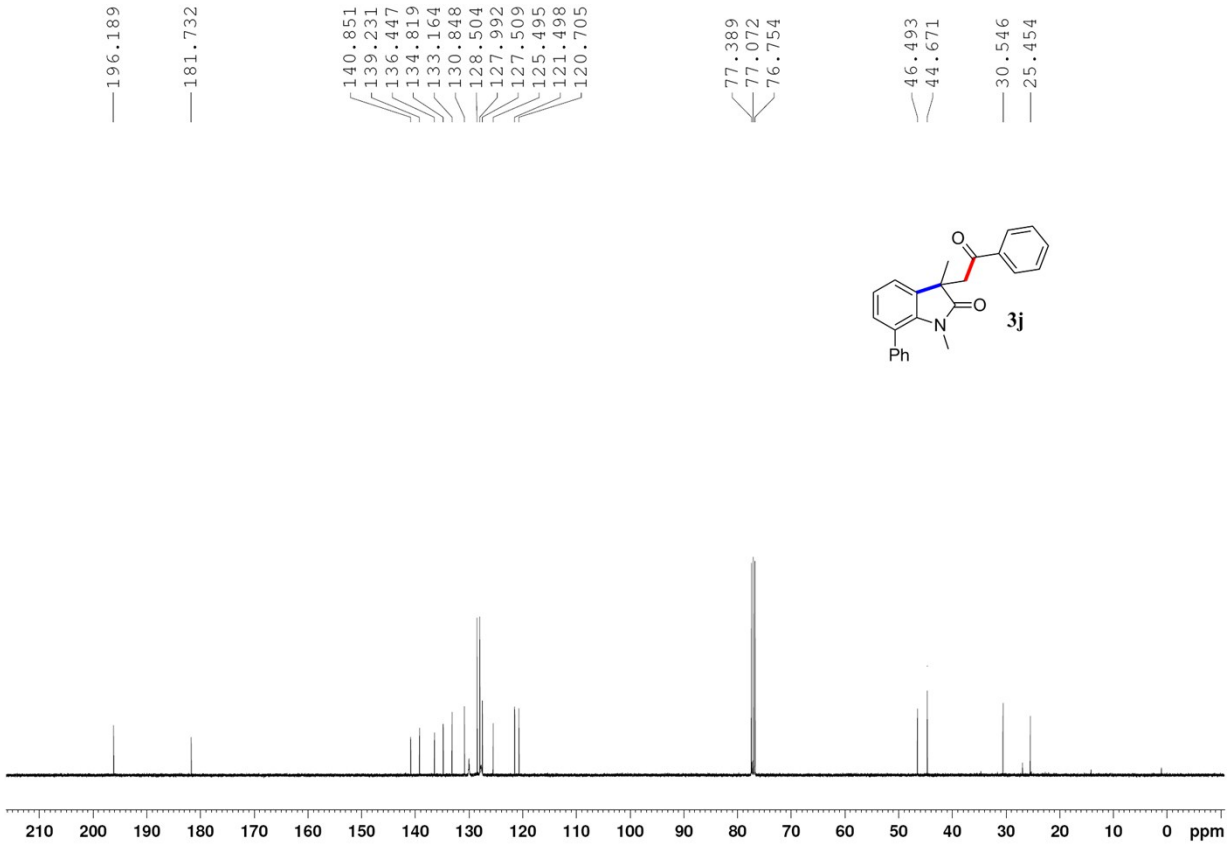
7.109
7.090
7.016
6.997
6.979
3.800
3.756
3.739
3.695
2.850
1.505
3j
Ph
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.05
7.02
2.04
1.01
2.03
3.01
3.05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133.164
130.848
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127.992
127.509
125.495
121.498
120.705
77.389
77.072
76.754
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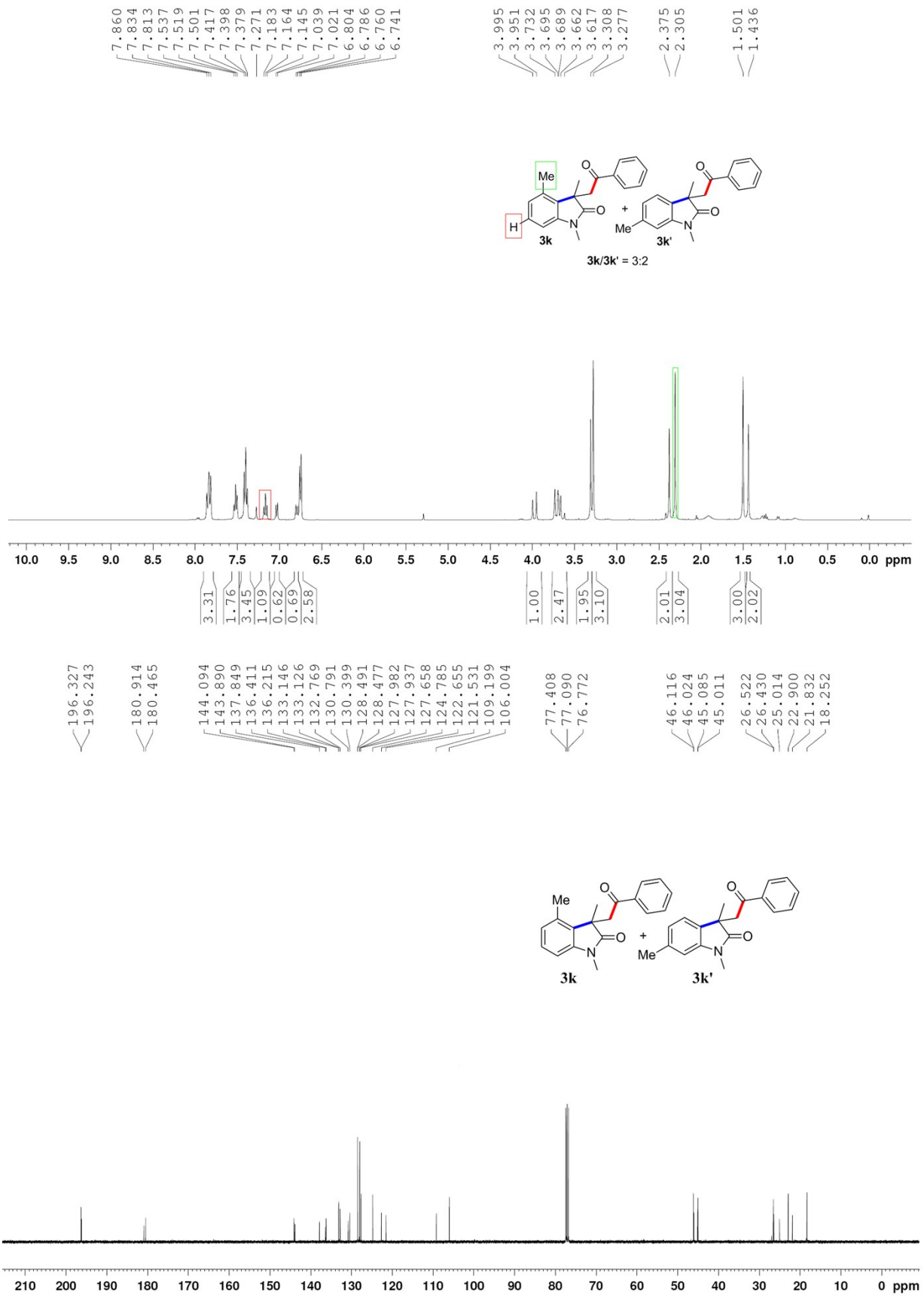
44.671
30.546
25.454
3j
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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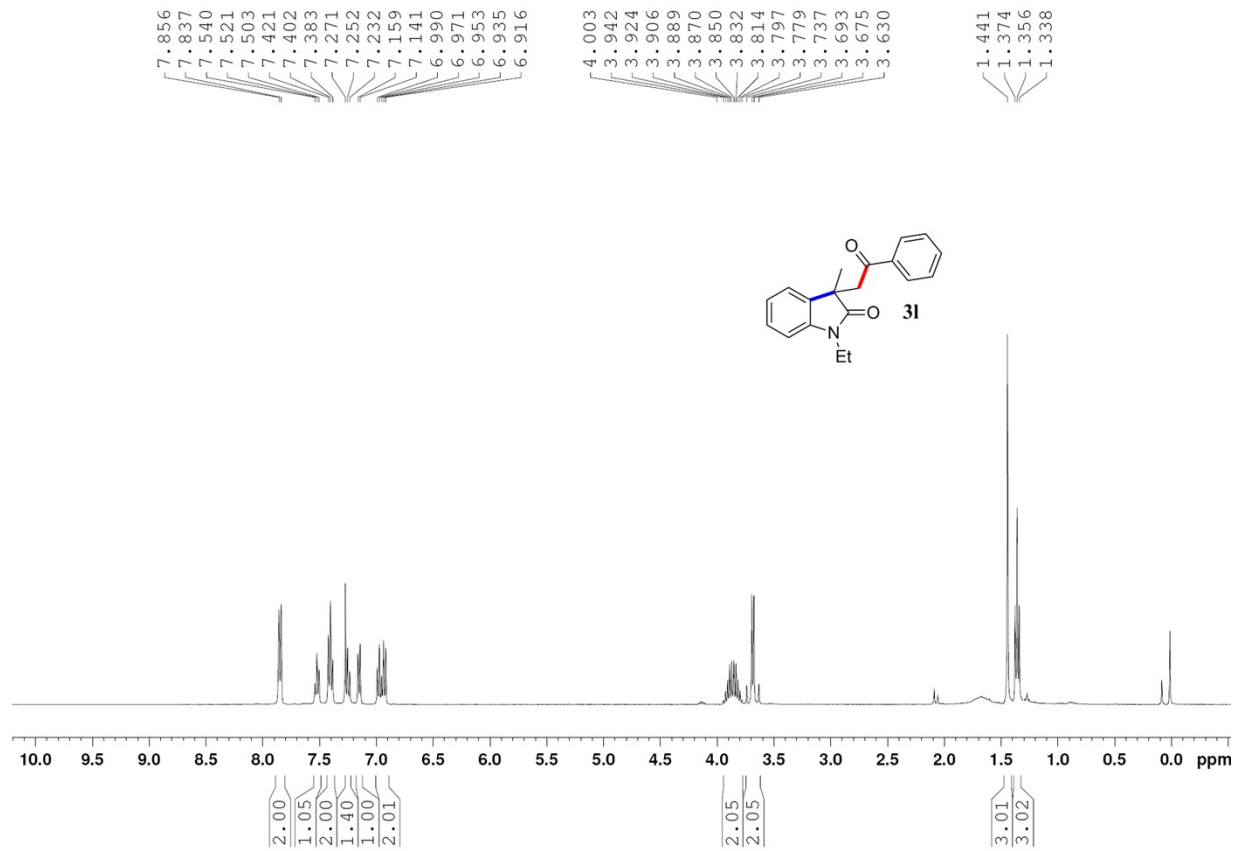
7.834
7.813
7.537
7.519
7.501
7.417
7.398
7.379
7.271
7.183
7.164
7.145
7.039
7.021
6.804
6.786
6.760
6.741
3.995
3.951
3.732
3.695
3.689
3.662
3.617
3.308
3.277
2.375
2.305
1.501
1.436
Me
H
3k
Me
3k'
3k/3k' = 3:2
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
3.31
1.76
3.45
1.09
0.62
0.69
2.58
1.00
2.47
1.95
3.10
2.01
3.04
3.00
2.02
196.327
196.243
180.914
180.465
144.094
143.890
137.849
136.411
136.215
133.146
133.126
132.769
130.791
130.399
128.491
128.477
127.982
127.937
127.658
124.785
122.655
121.531
109.199
106.004
77.408
77.090
76.772
46.116
46.024
45.085
45.011
26.522
26.430
25.014
22.900
21.832
18.252
Me
3k
Me
3k'
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.521
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7.402
7.383
7.271
7.252
7.232
7.159
7.141
6.990
6.971
6.953
6.935
6.916
4.003
3.942
3.924
3.906
3.889
3.870
3.850
3.832
3.814
3.797
3.779
3.737
3.693
3.675
3.630
1.441
1.374
1.356
1.338
O
O
N
Et
3l
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 ppm
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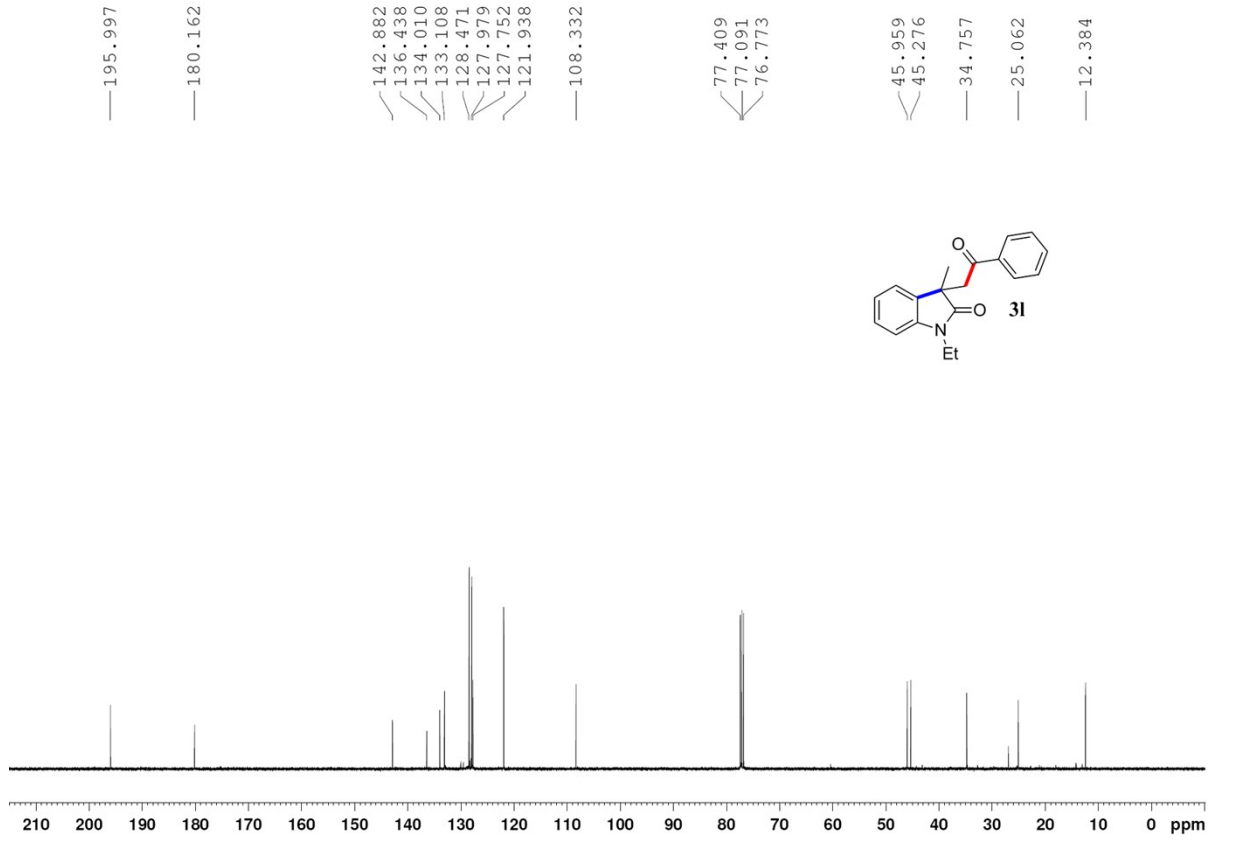
1.05
2.00
1.40
1.00
2.01
2.05
2.05
3.01
3.02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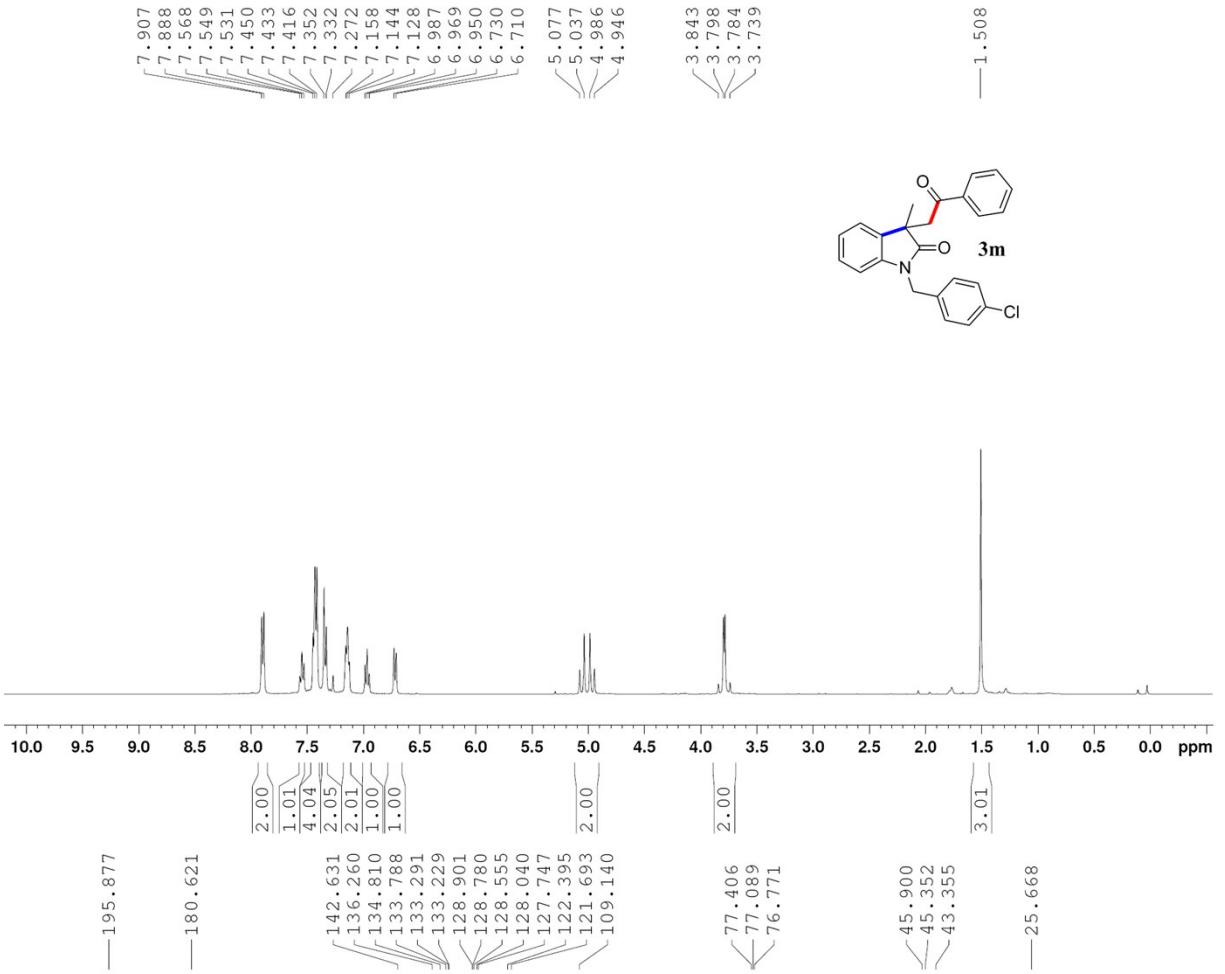
180.162
142.882
136.438
134.010
133.108
128.471
127.979
127.752
121.938
108.332
77.409
77.091
76.773
45.959
45.276
34.757
25.062
12.384
O
O
N
Et
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.531
7.450
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7.416
7.352
7.332
7.272
7.158
7.144
7.128
6.987
6.969
6.950
6.730
6.710
5.077
5.037
4.986
4.946
3.843
3.798
3.784
3.739
1.508
3m
Cl
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 ppm
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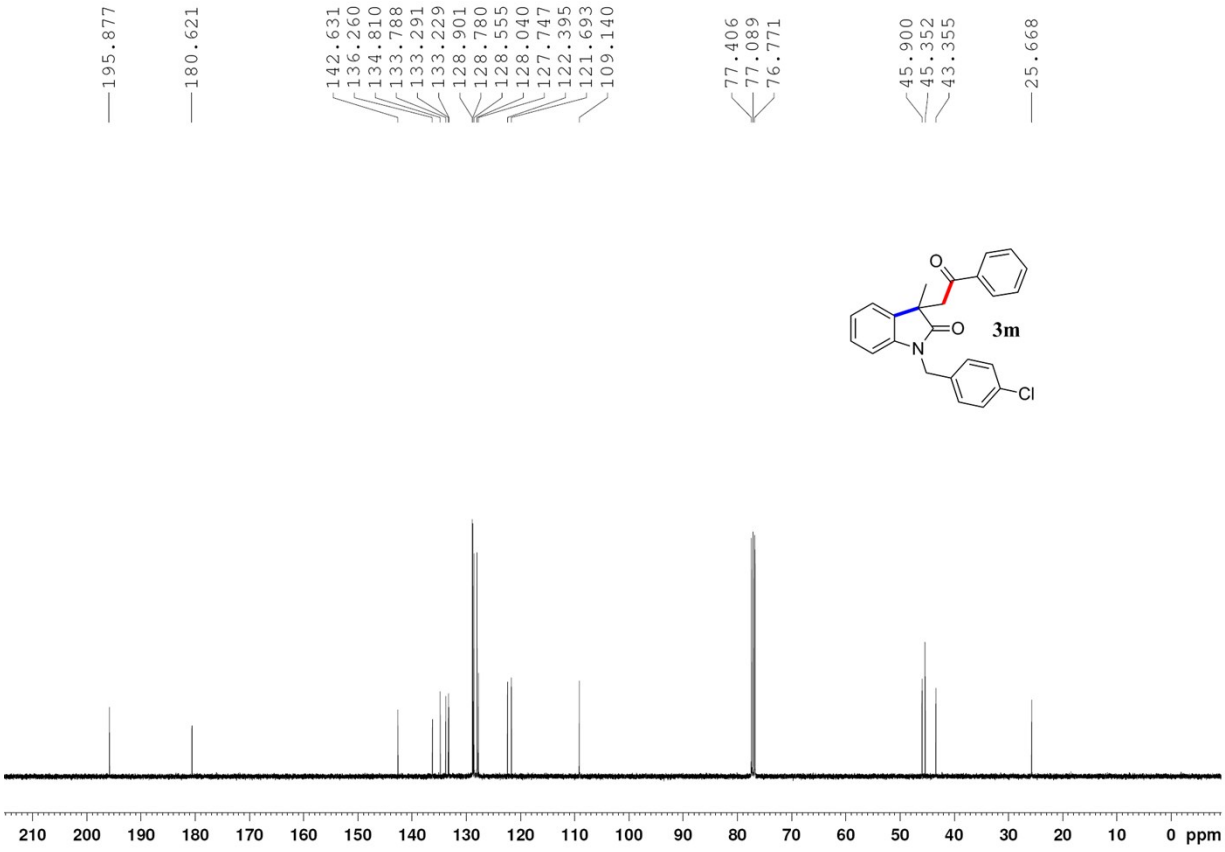
1.01
4.04
2.05
2.01
1.00
1.00
2.00
2.00
3.01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180.621
142.631
136.260
134.810
133.788
133.291
133.229
128.901
128.780
128.555
128.040
127.747
122.395
121.693
109.140
77.406
77.089
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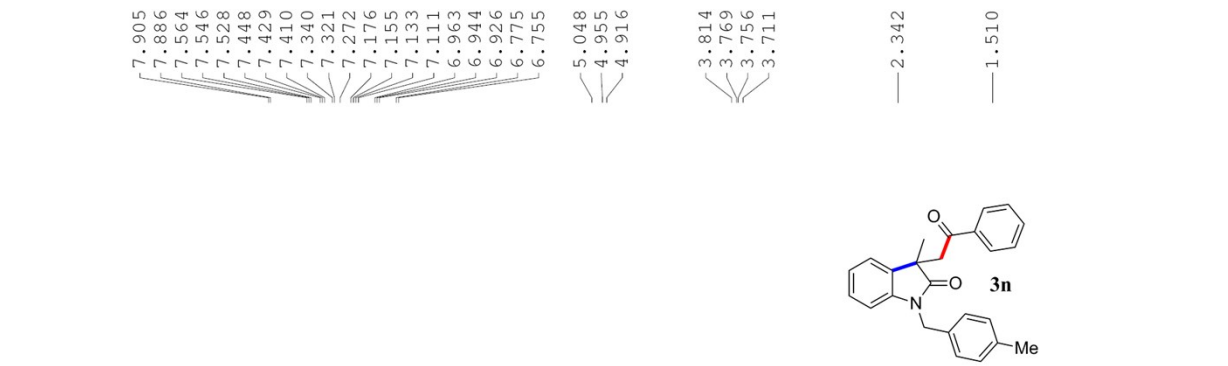
45.900
45.352
43.355
25.668
3m
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.546
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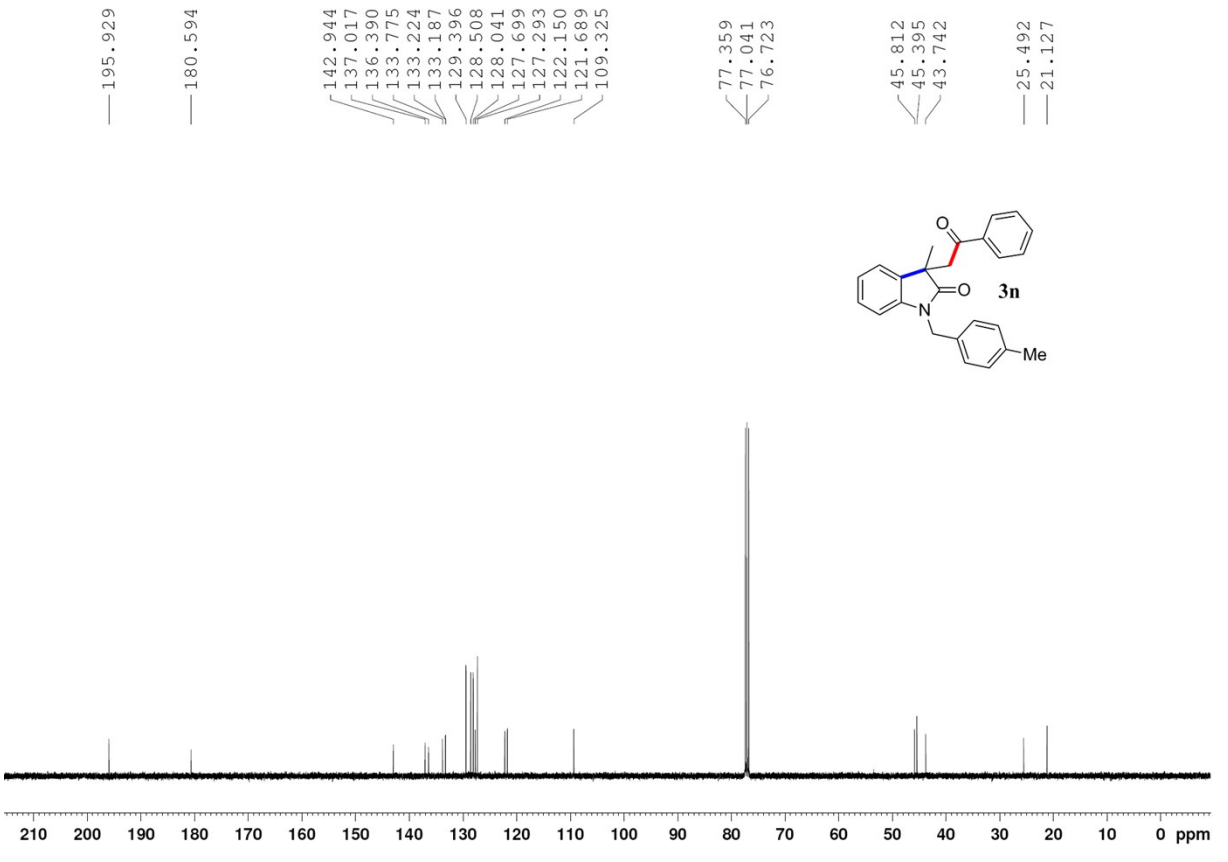
7.429
7.410
7.340
7.321
7.272
7.176
7.155
7.133
7.111
6.963
6.944
6.926
6.775
6.755
5.048
4.955
4.916
3.814
3.769
3.756
3.711
2.342
1.510
O
O
N
3n
Me
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.04
2.04
2.03
4.04
1.04
1.00
2.00
2.00
3.00
3.02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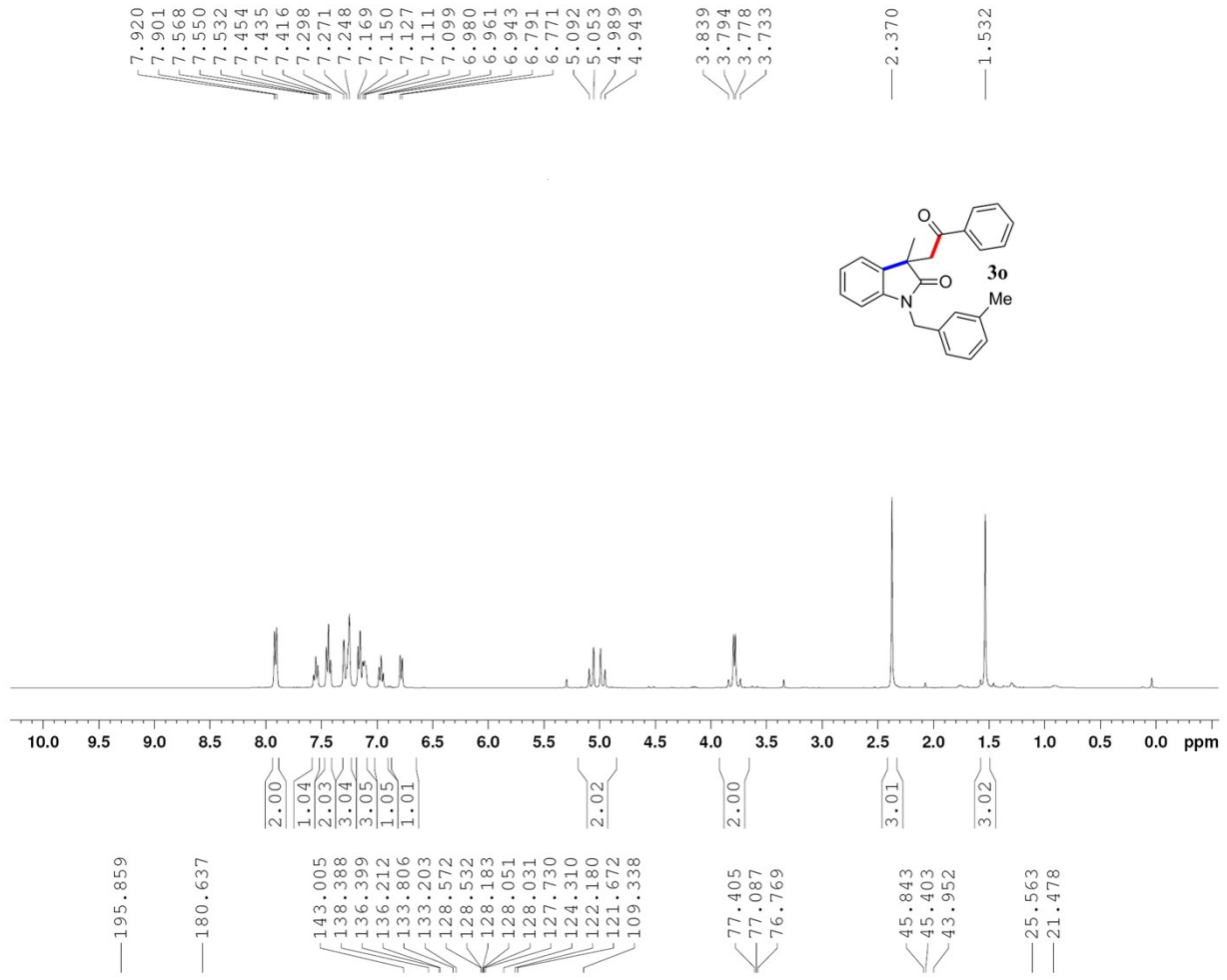
133.187
129.396
128.508
128.041
127.699
127.293
122.150
121.689
109.325
77.359
77.041
76.723
45.812
45.395
43.742
25.492
21.127
O
O
N
3n
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.901
7.568
7.550
7.532
7.454
7.435
7.416
7.298
7.271
7.248
7.169
7.150
7.127
7.111
7.099
6.980
6.961
6.943
6.791
6.771
5.092
5.053
4.989
4.949
3.839
3.794
3.778
3.733
2.370
1.532
O
O
N
3o
Me
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 ppm
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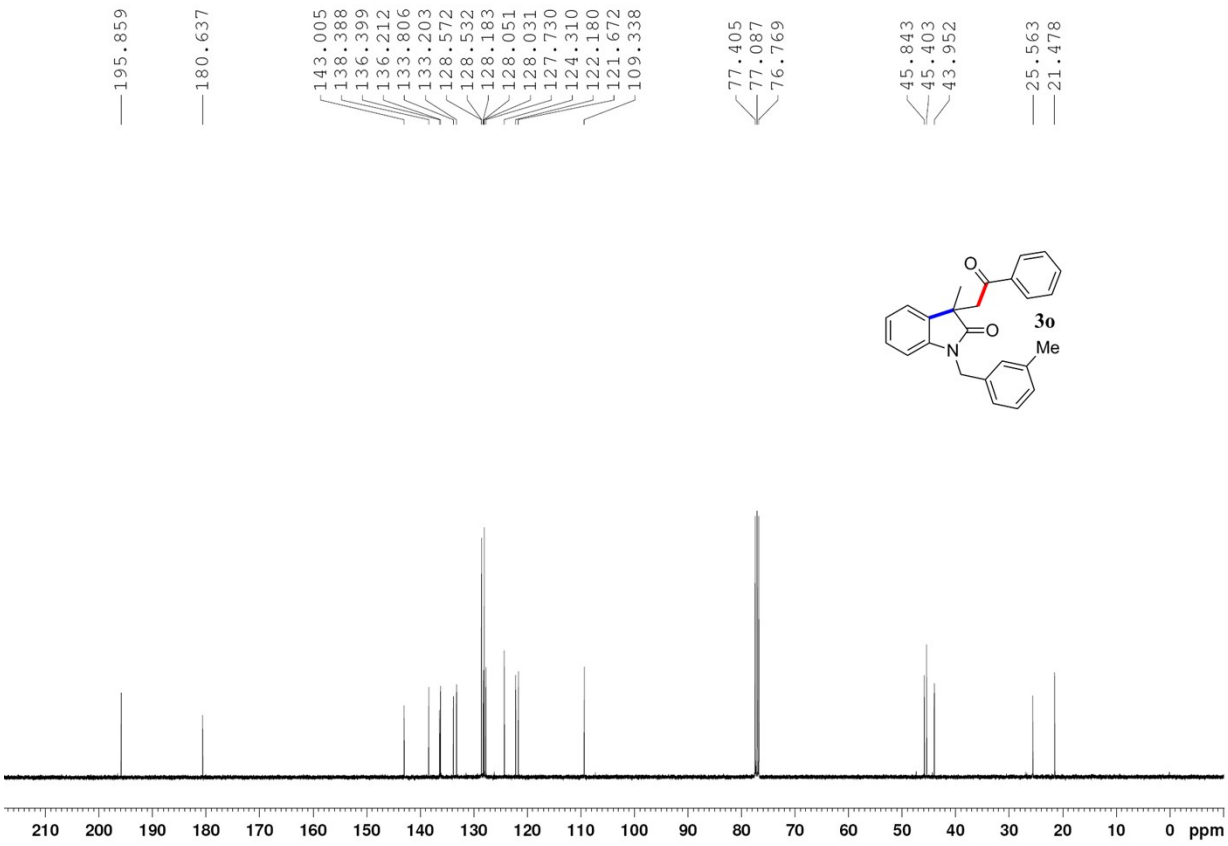
1.04
2.03
3.04
3.05
1.05
1.01
2.02
2.00
3.01
3.02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143.005
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133.203
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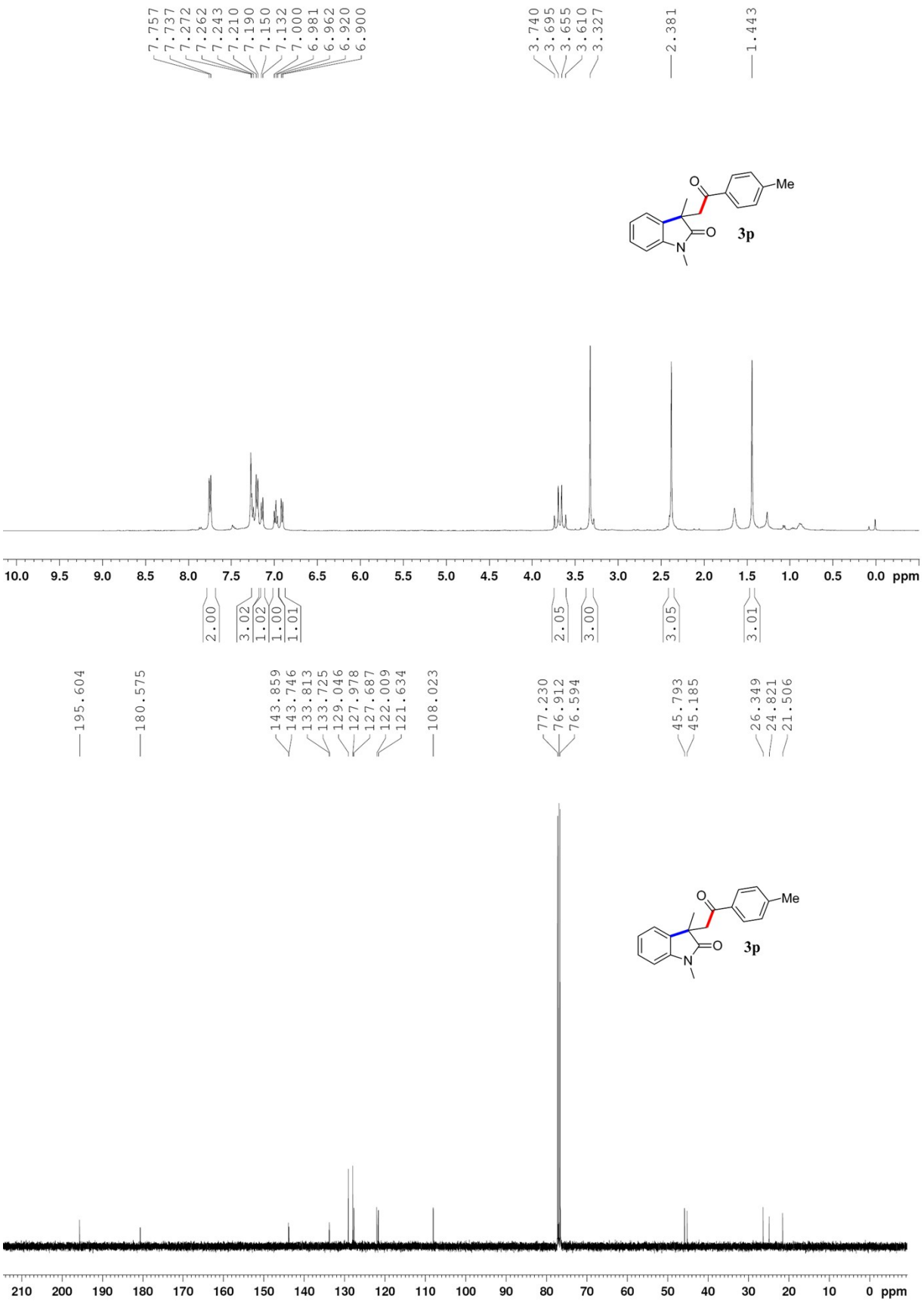
128.532
128.183
128.051
128.031
127.730
124.310
122.180
121.672
109.338
77.405
77.087
76.769
45.843
45.403
43.952
25.563
21.478
O
O
N
3o
Me
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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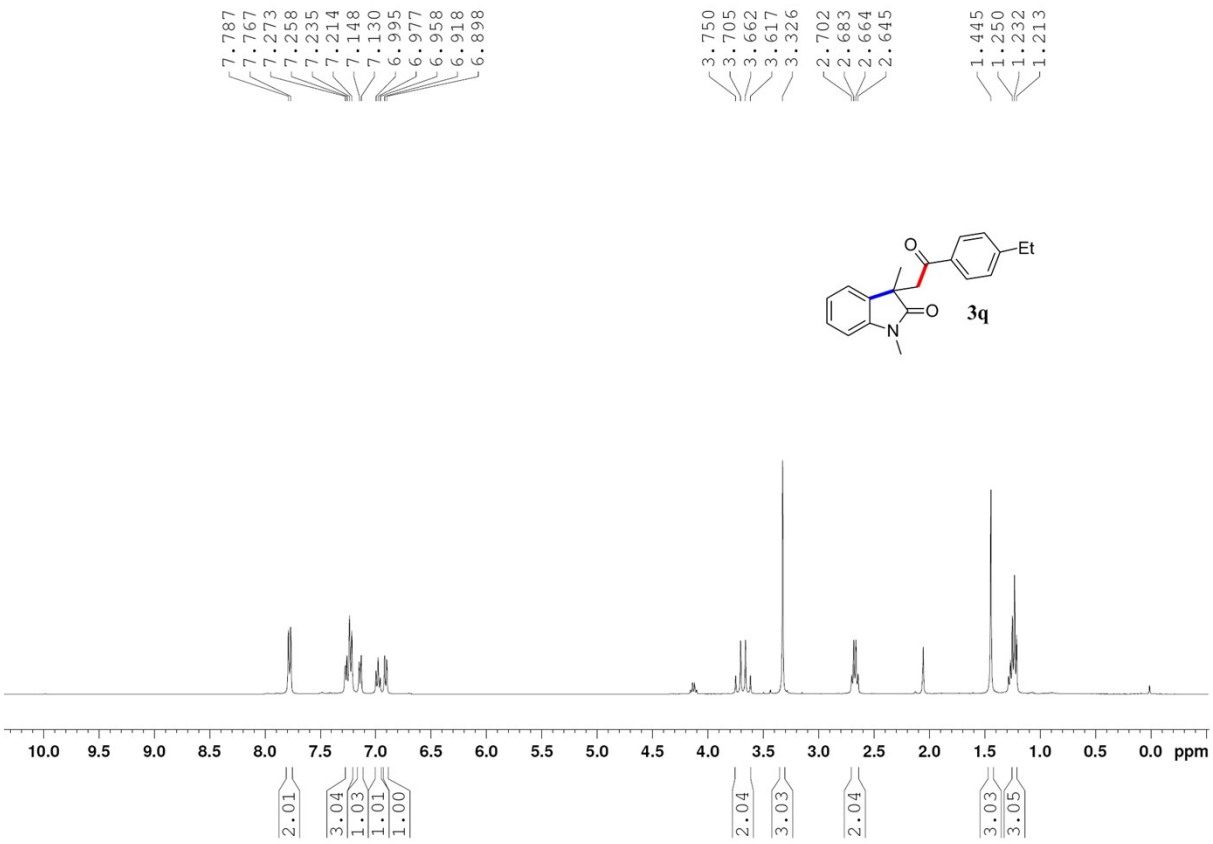
7.737
7.272
7.262
7.243
7.210
7.190
7.150
7.132
7.000
6.981
6.962
6.920
6.900
3.740
3.695
3.655
3.610
3.327
2.381
1.443
Me
O
O
N
3p
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
3.02
1.02
1.00
1.01
2.05
3.00
3.05
3.01
195.604
180.575
143.859
143.746
133.813
133.725
129.046
127.978
127.687
122.009
121.634
108.023
77.230
76.912
76.594
45.793
45.185
26.349
24.821
21.506
Me
O
O
N
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.767
7.273
7.258
7.235
7.214
7.148
7.130
6.995
6.977
6.958
6.918
6.898
3.750
3.705
3.662
3.617
3.326
2.702
2.683
2.664
2.645
1.445
1.250
1.232
1.213
Et
O
O
N
3q
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 ppm
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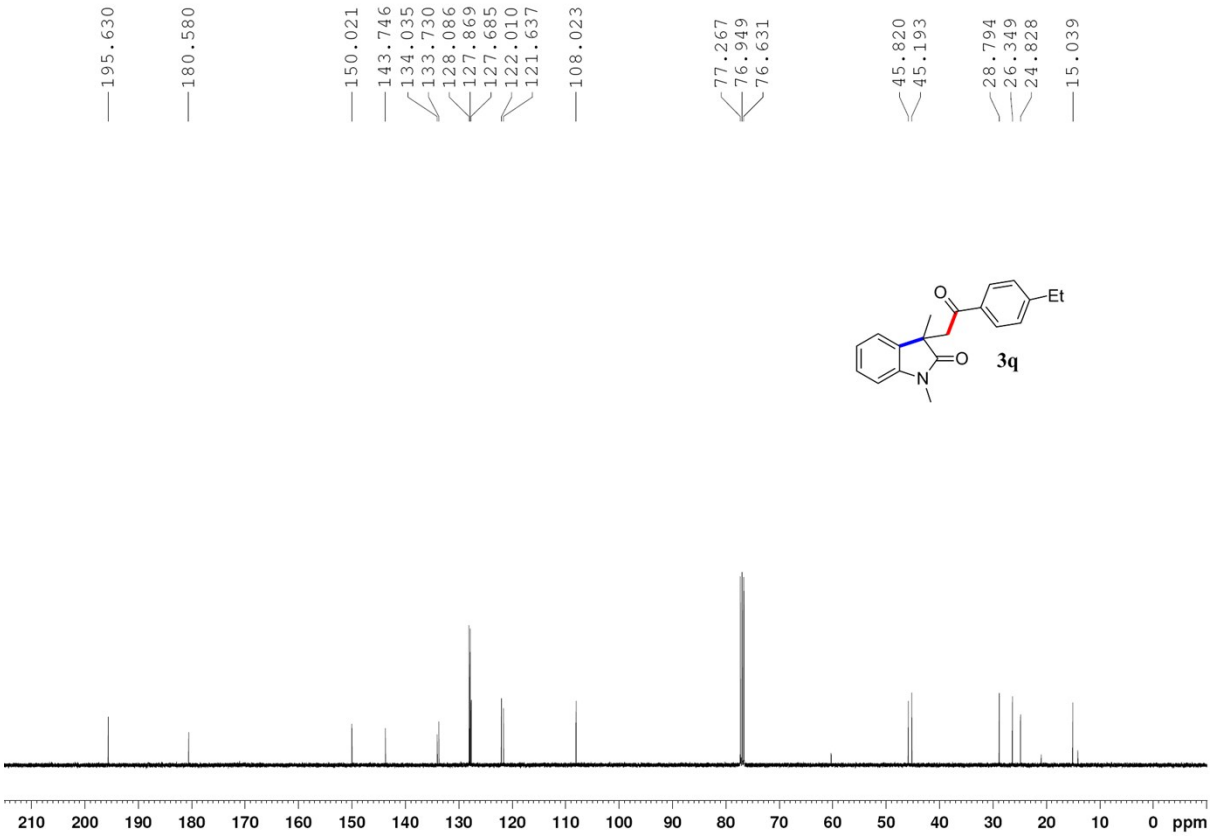
3.04
1.03
1.01
1.00
2.04
3.03
2.04
3.03
3.05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150.021
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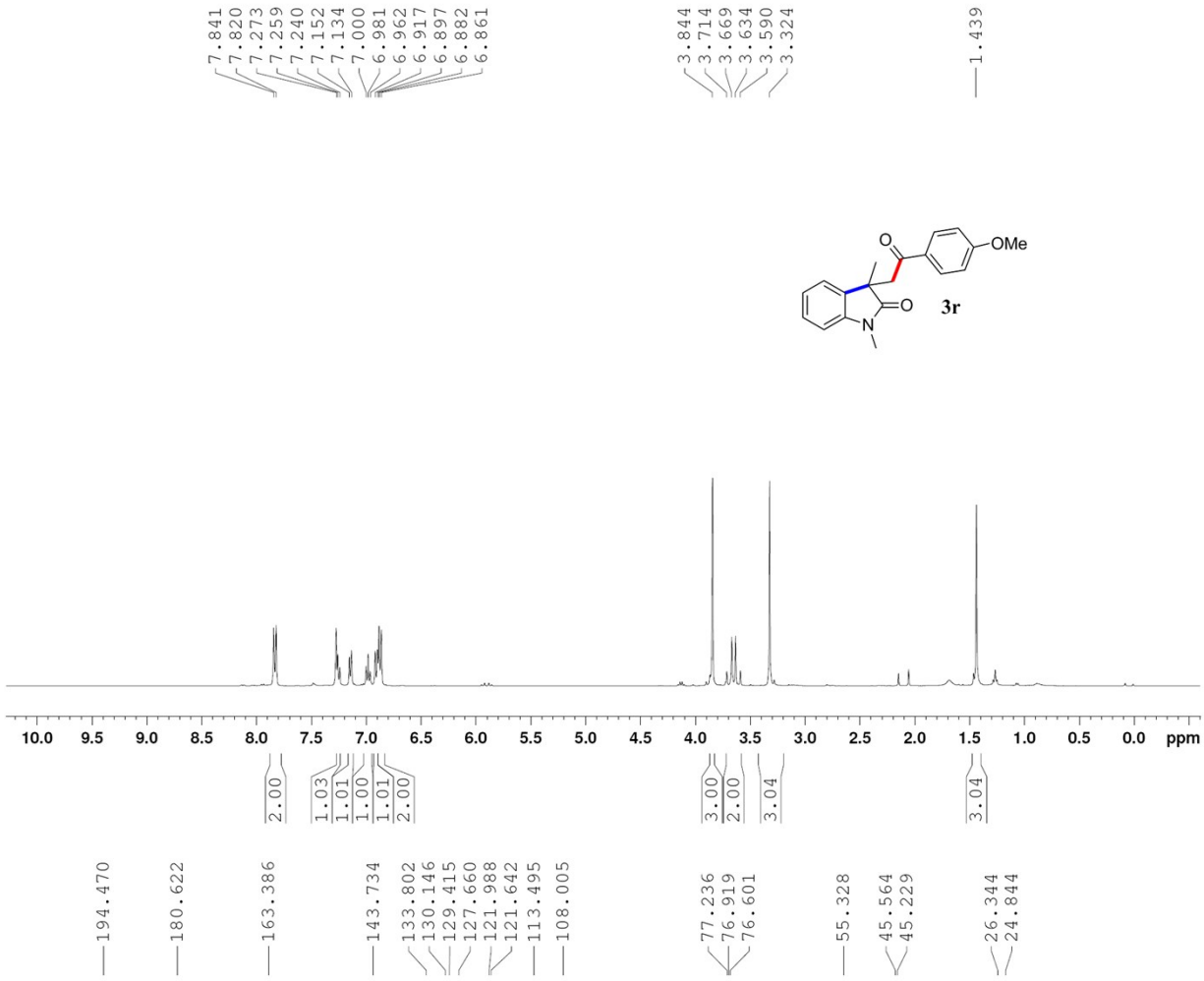
134.035
133.730
128.086
127.869
127.685
122.010
121.637
108.023
77.267
76.949
76.631
45.820
45.193
28.794
26.349
24.828
15.039
Et
O
O
N
3q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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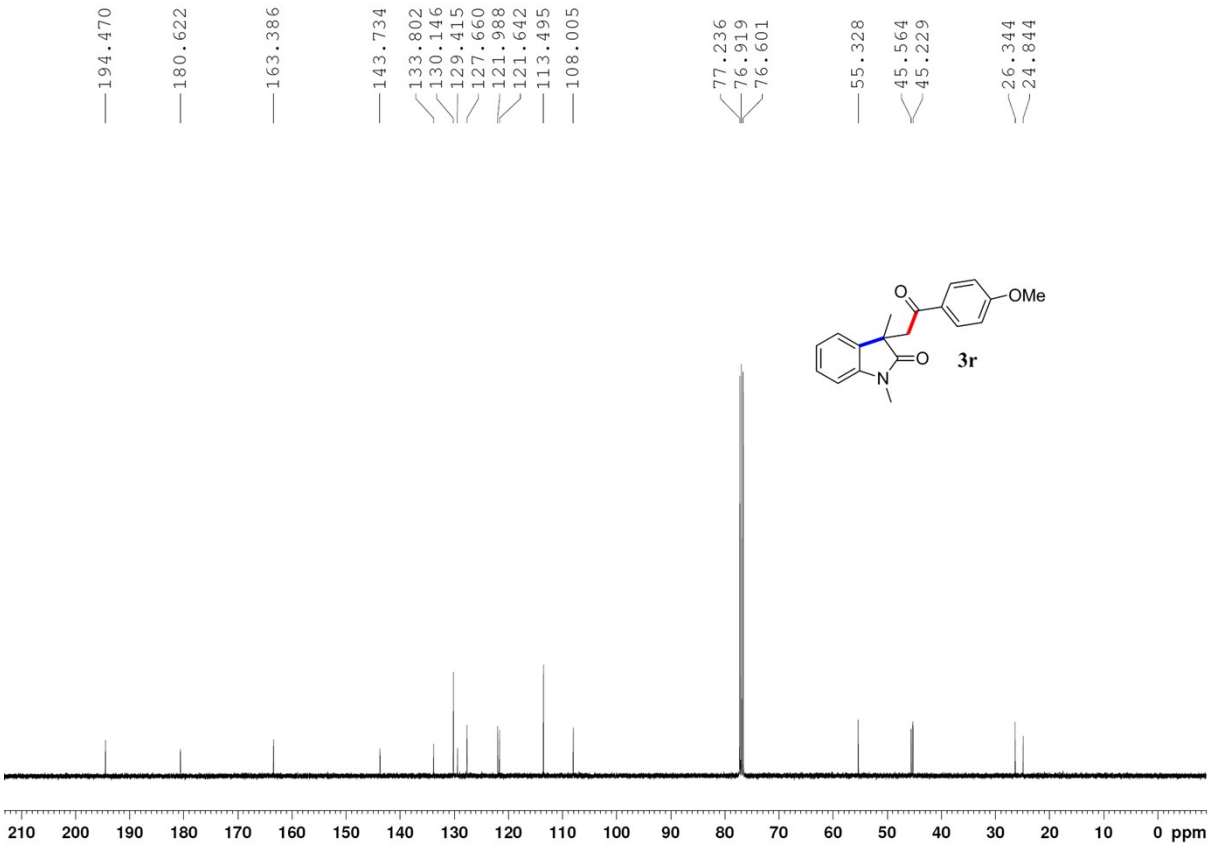
7.820
7.273
7.259
7.240
7.152
7.134
7.000
6.981
6.962
6.917
6.897
6.882
6.861
3.844
3.714
3.669
3.634
3.590
3.324
1.439
OMe
O
O
N
3r
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.03
1.01
1.00
1.01
2.00
3.00
2.00
3.04
3.04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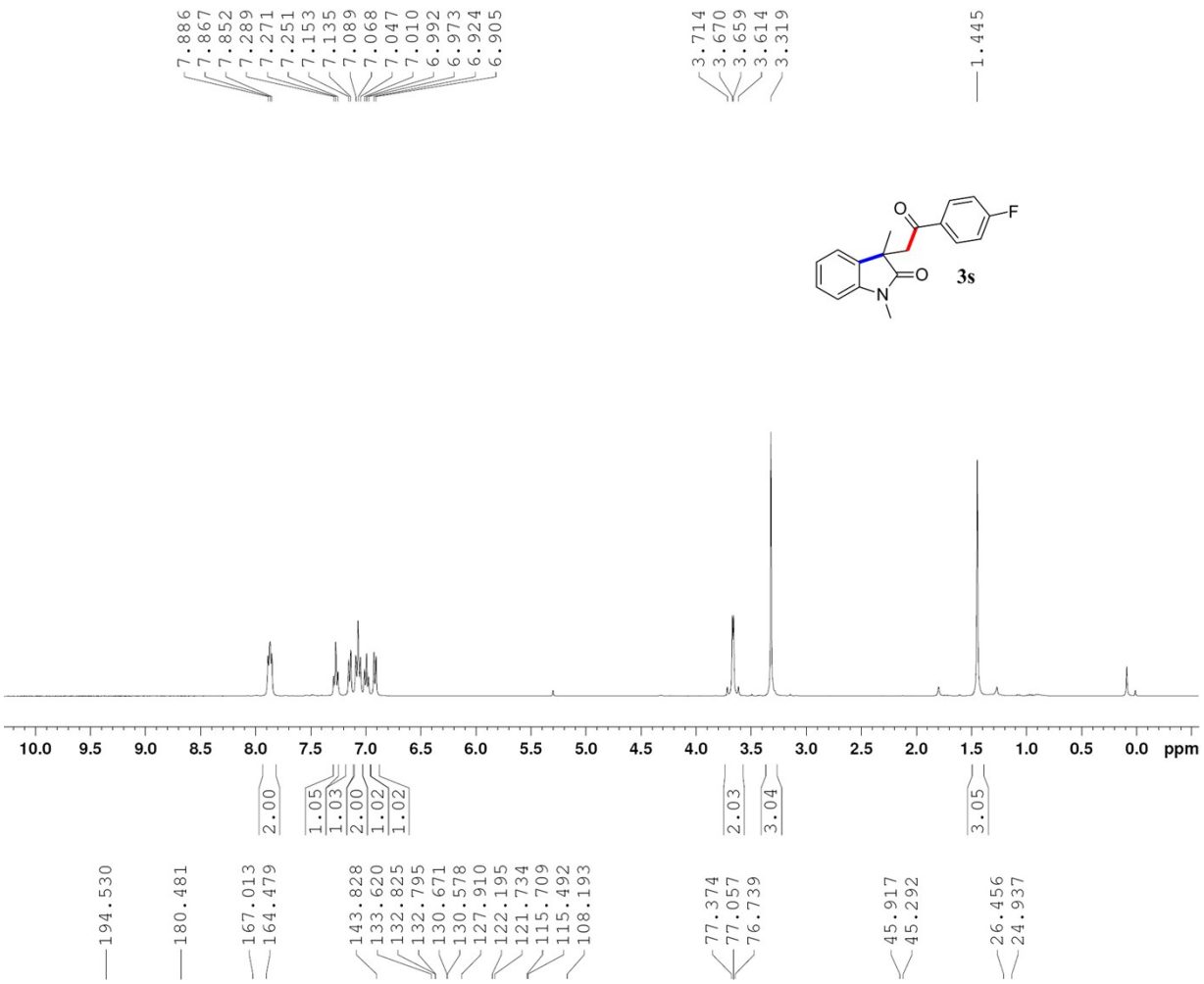
180.622
163.386
143.734
133.802
130.146
129.415
127.660
121.988
121.642
113.495
108.005
77.236
76.919
76.601
55.328
45.564
45.229
26.344
24.844
OMe
O
O
N
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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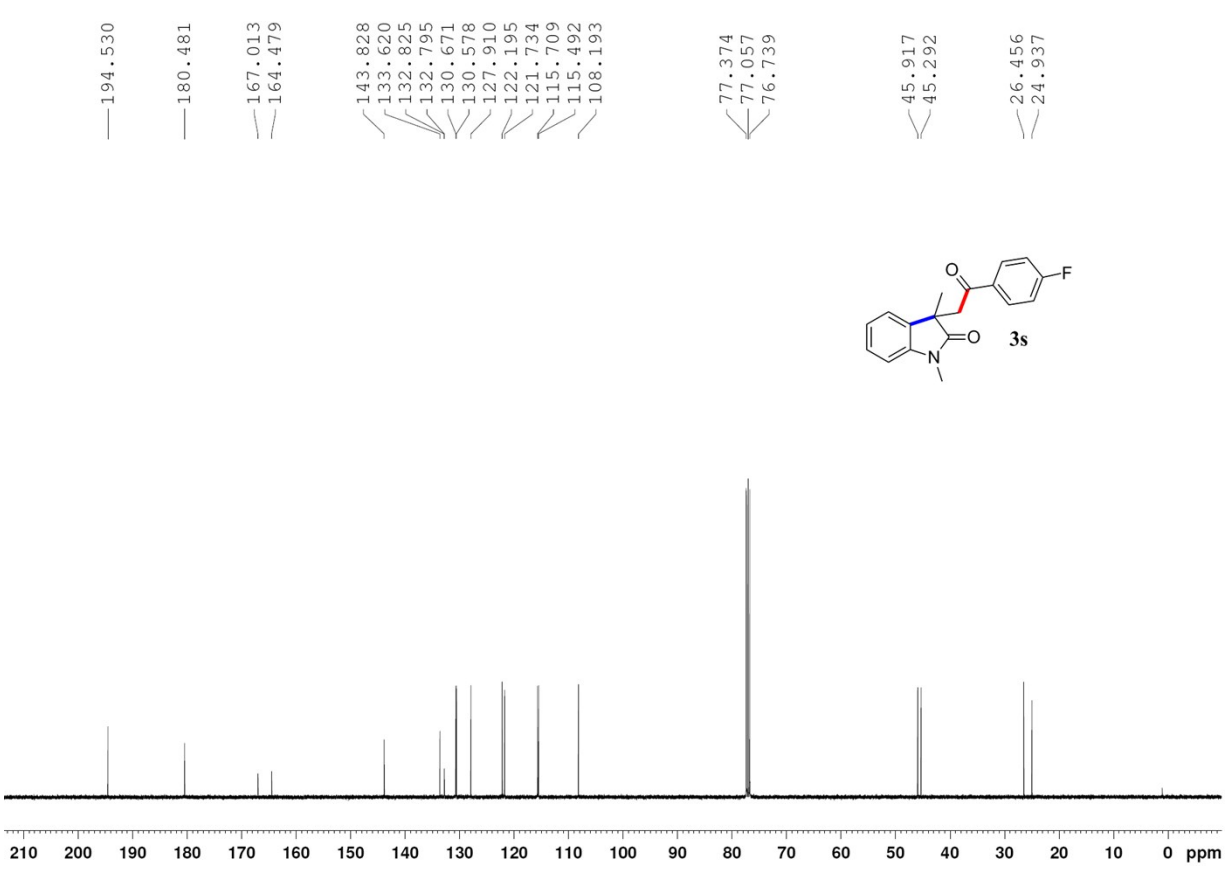
7.867
7.852
7.289
7.271
7.251
7.153
7.135
7.089
7.068
7.047
7.010
6.992
6.973
6.924
6.905
3.714
3.670
3.659
3.614
3.319
1.445
3s
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.05
1.03
2.00
1.02
1.02
2.03
3.04
3.05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167.013
164.479
143.828
133.620
132.825
132.795
130.671
130.578
127.910
122.195
121.734
115.709
115.492
108.193
77.374
77.057
76.739
45.917
45.292
26.456
24.937
3s
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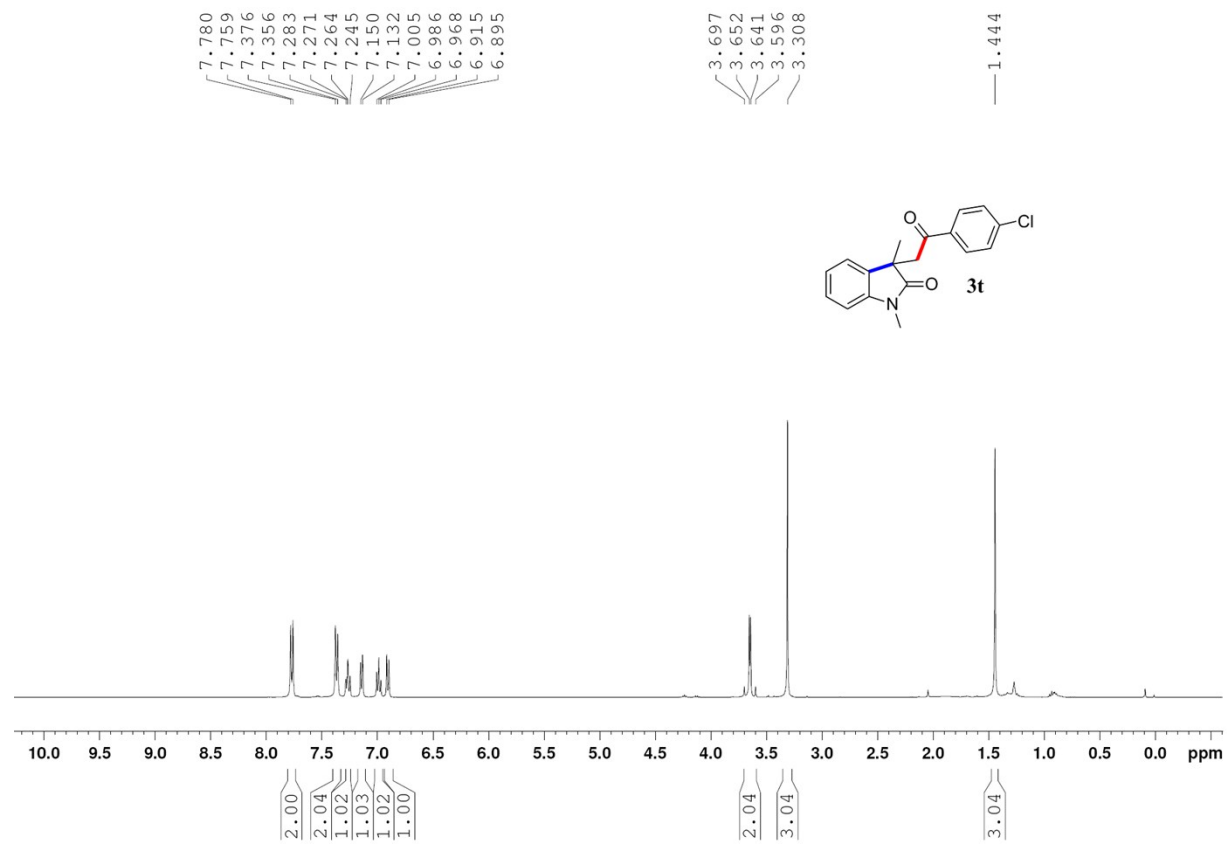
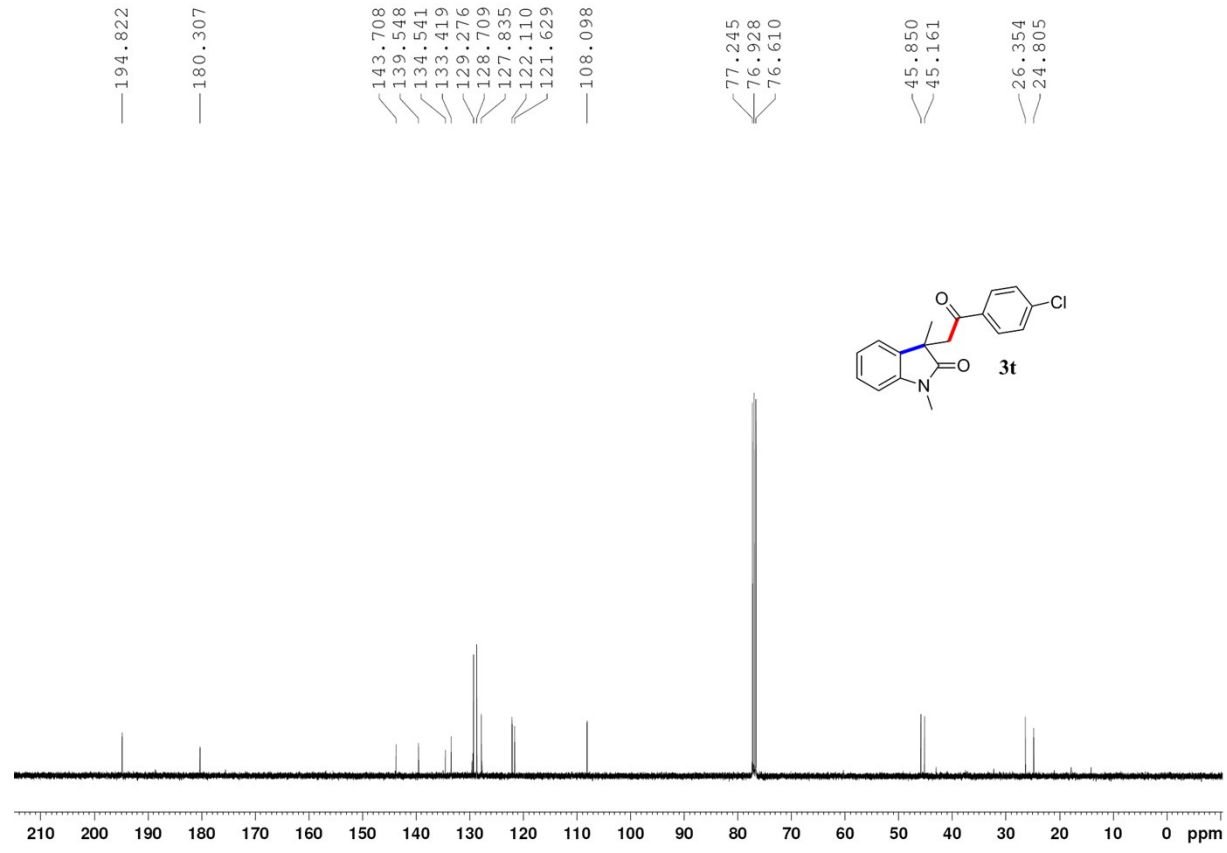

7.780
7.759
7.376
7.356
7.283
7.271
7.264
7.245
7.150
7.132
7.005
6.986
6.968
6.915
6.895
3.697
3.652
3.641
3.596
3.308
1.444
O
Cl
O
N
3t
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
2.04
1.02
1.03
1.02
1.00
2.04
3.04
3.04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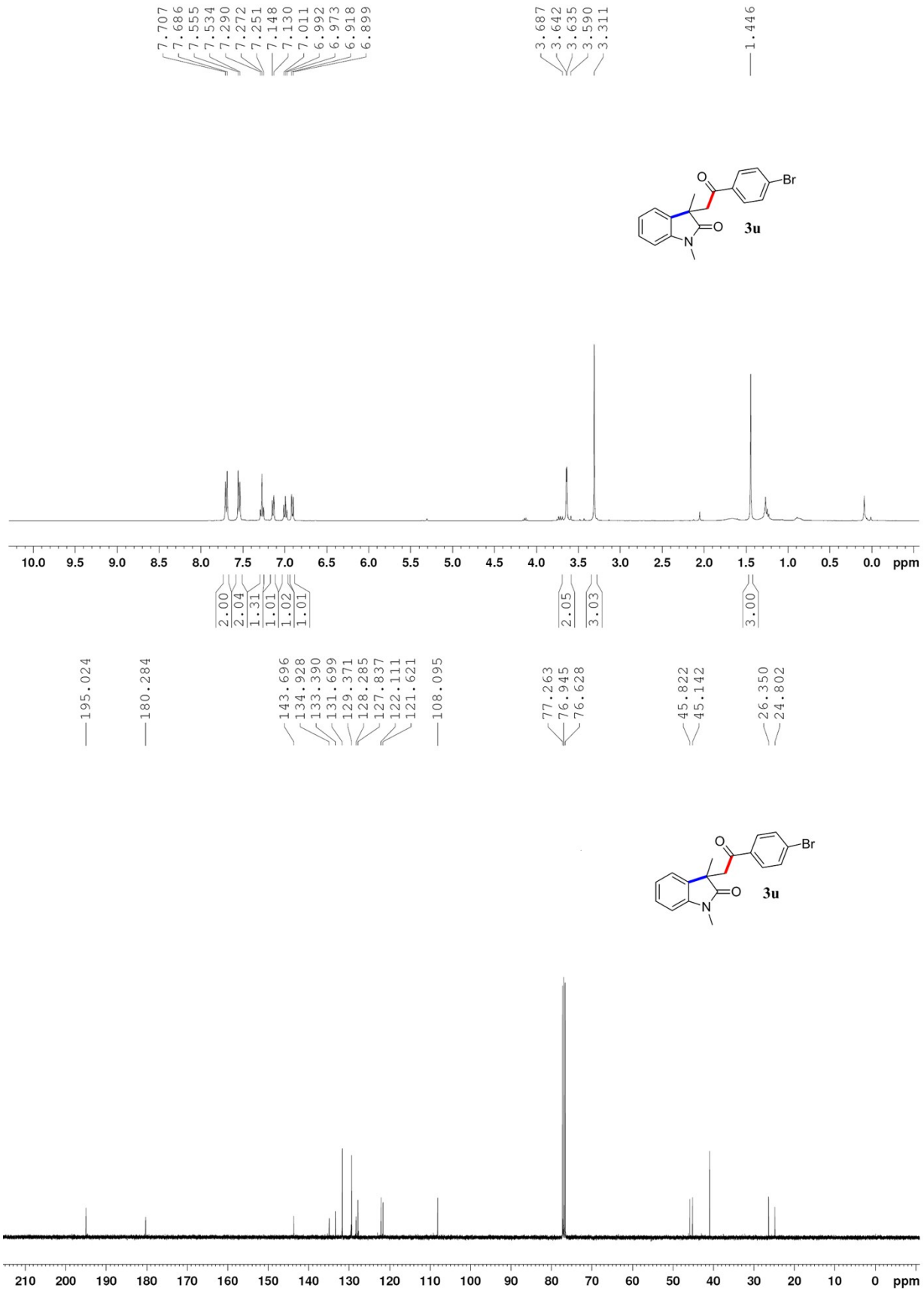
133.419
129.276
128.709
127.835
122.110
121.629
108.098
77.245
76.928
76.610
45.850
45.161
26.354
24.805
O
Cl
O
N
3t
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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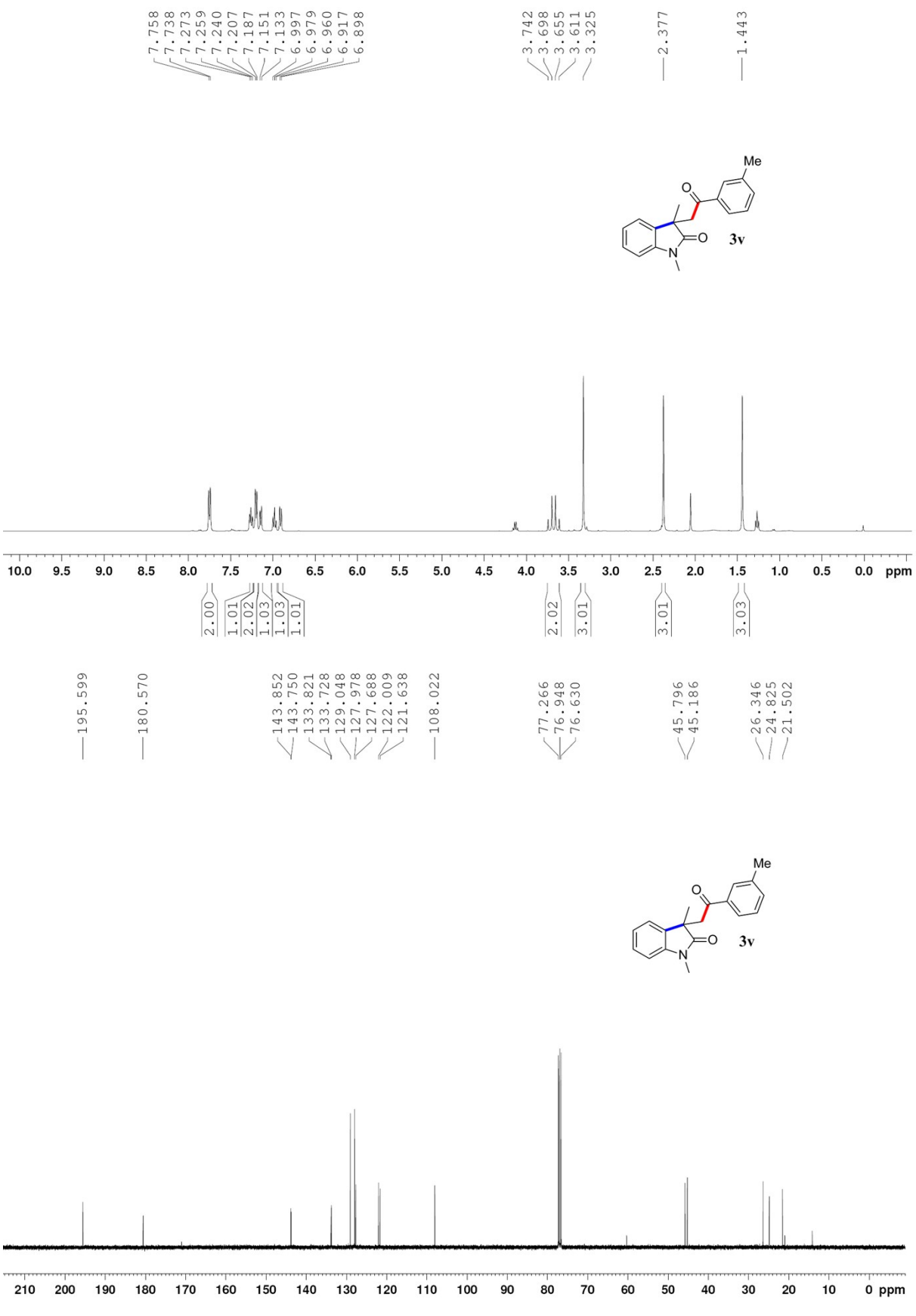
7.686
7.555
7.534
7.290
7.272
7.251
7.148
7.130
7.011
6.992
6.973
6.918
6.899
3.687
3.642
3.635
3.590
3.311
1.446
Br
O
O
N
3u
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
2.04
1.31
1.01
1.02
1.01
2.05
3.03
3.00
195.024
180.284
143.696
134.928
133.390
131.699
129.371
128.285
127.837
122.111
121.621
108.095
77.263
76.945
76.628
45.822
45.142
26.350
24.802
Br
O
O
N
3u
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.259
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7.187
7.151
7.133
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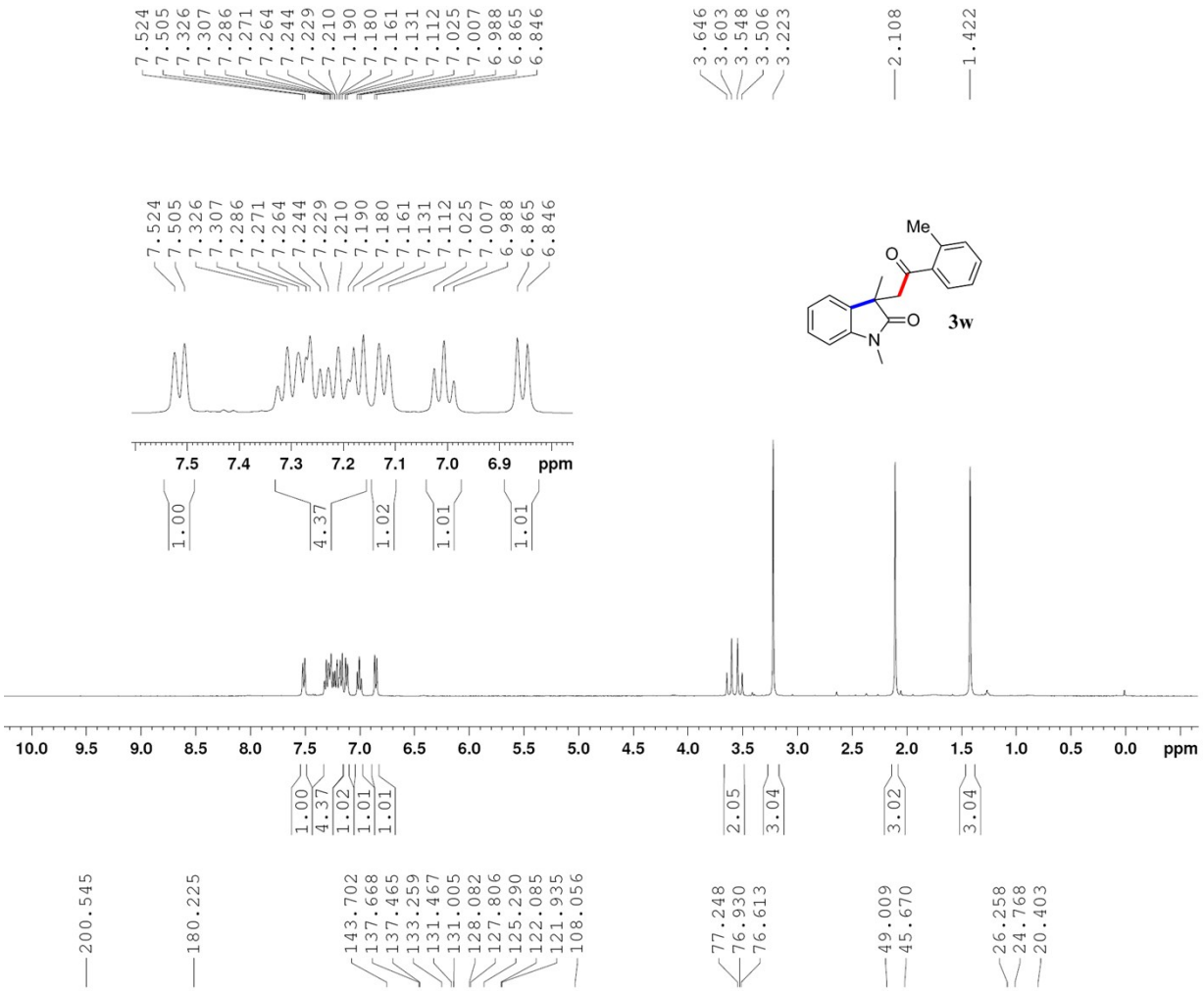
6.979
6.960
6.917
6.898
3.742
3.698
3.655
3.611
3.325
2.377
1.443
Me
O
O
N
3v
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.00
1.01
2.02
1.03
1.03
1.01
2.02
3.01
3.01
3.03
195.599
180.570
143.852
143.750
133.821
133.728
129.048
127.978
127.688
122.009
121.638
108.022
77.266
76.948
76.630
45.796
45.186
26.346
24.825
21.502
Me
O
O
N
3v
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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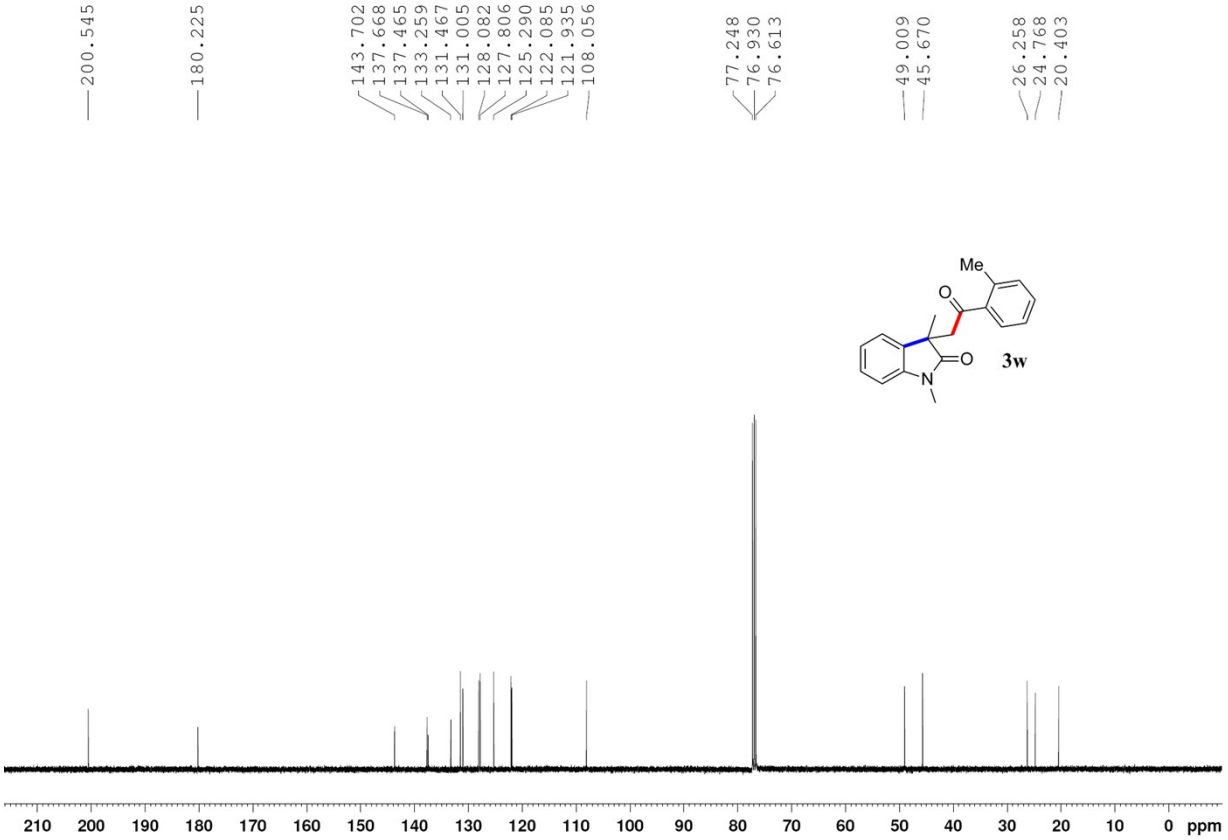
7.244
7.229
7.210
7.190
7.180
7.161
7.131
7.112
7.025
7.007
6.988
6.865
6.846
3.646
3.603
3.548
3.506
3.223
2.108
1.422
Me
O
O
N
3w
7.5
7.4
7.3
7.2
7.1
7.0
6.9
ppm
1.00
4.37
1.02
1.01
1.01
2.05
3.04
3.02
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm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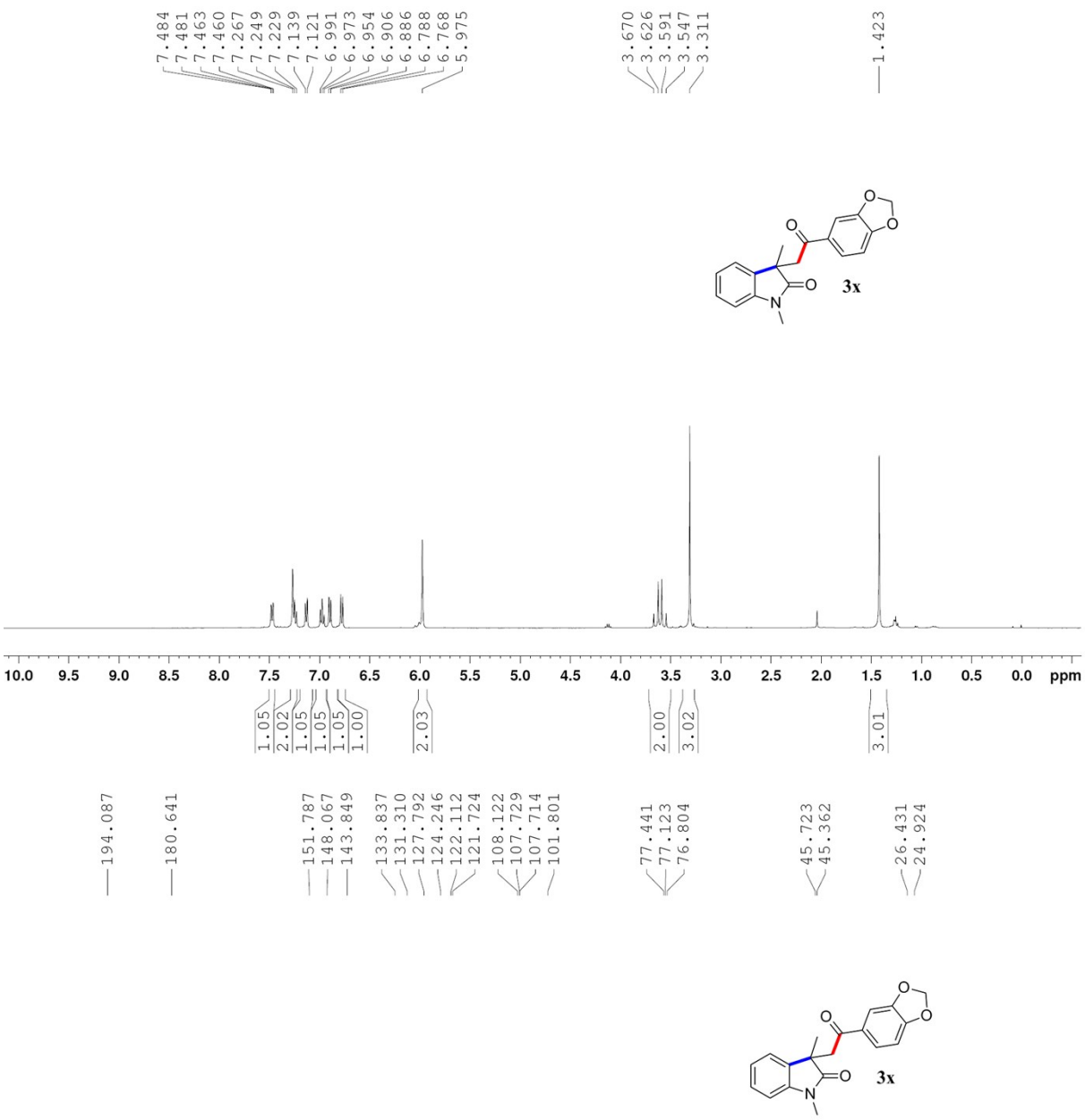
143.702
137.668
137.465
133.259
131.467
131.005
128.082
127.806
125.290
122.085
121.935
108.056
77.248
76.930
76.613
49.009
45.670
26.258
24.768
20.403
Me
O
O
N
3w
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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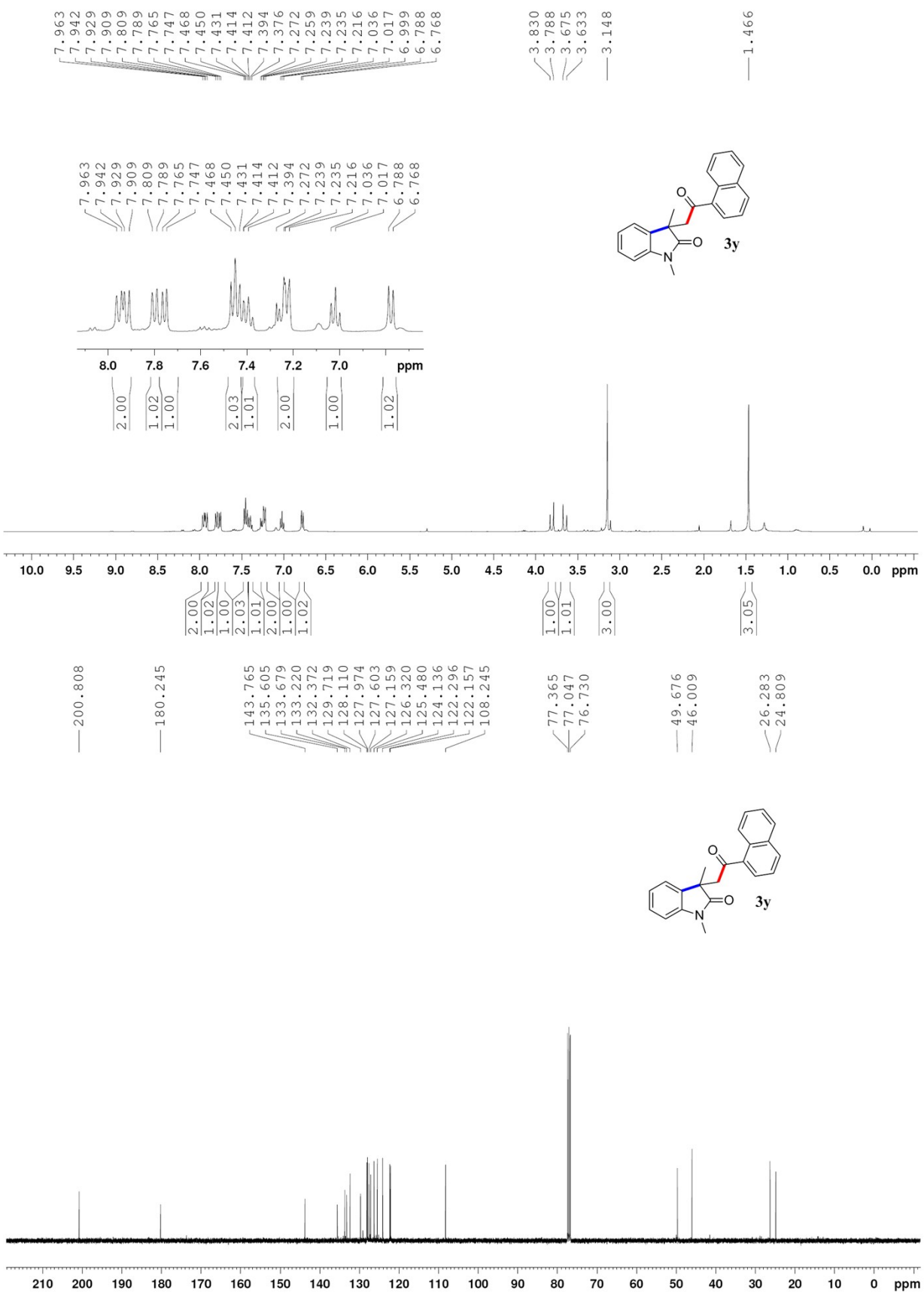
7.229
7.139
7.121
6.991
6.973
6.954
6.906
6.886
6.788
6.768
5.975
3.670
3.626
3.591
3.547
3.311
1.423
3x
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.05
2.02
1.05
1.05
1.05
1.00
2.03
2.00
3.02
3.01
194.087
180.641
151.787
148.067
143.849
133.837
131.310
127.792
124.246
122.112
121.724
108.122
107.729
107.714
101.801
77.441
77.123
76.804
45.723
45.362
26.431
24.924
3x
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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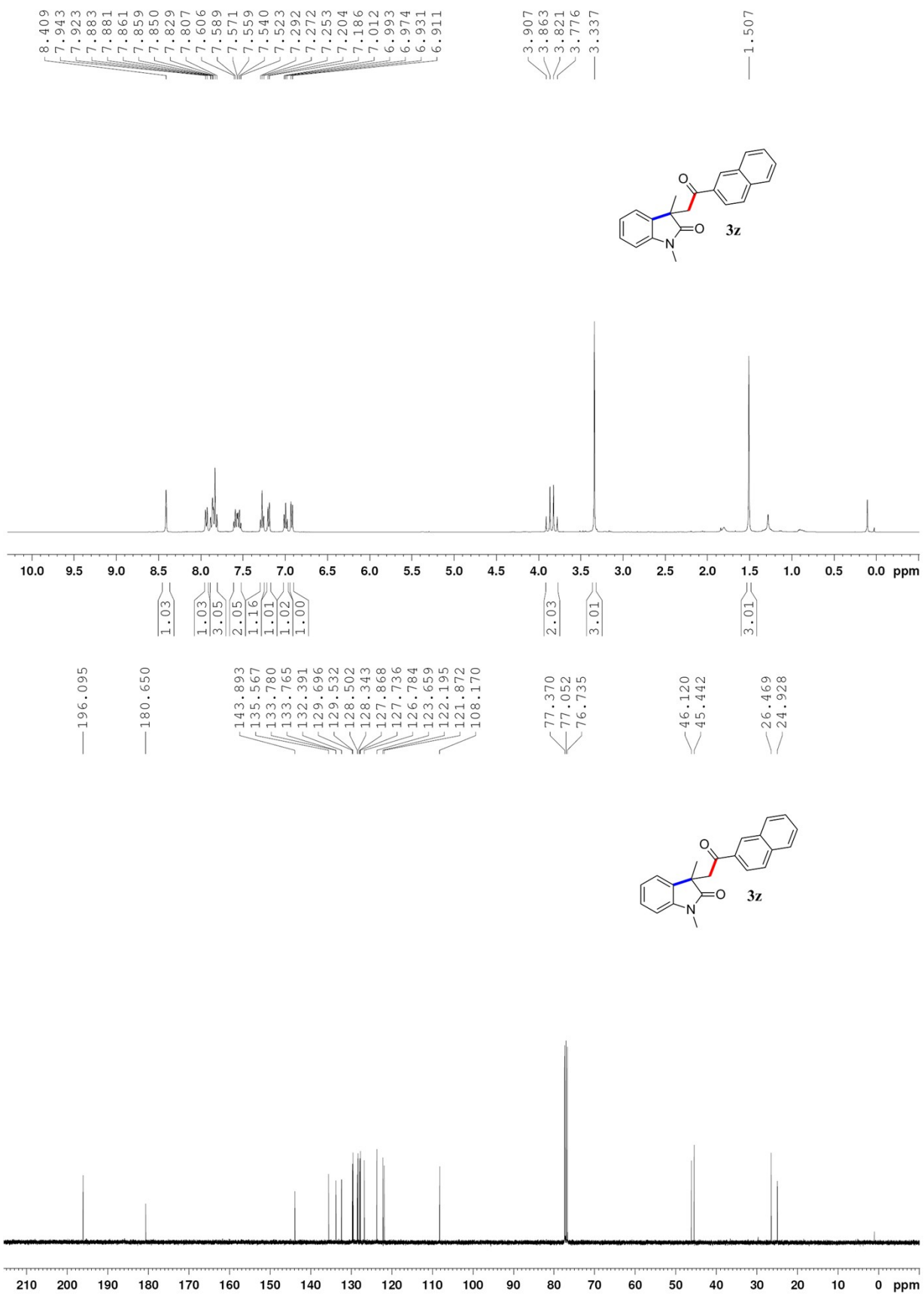
7.765
7.747
7.468
7.450
7.431
7.414
7.412
7.394
7.376
7.272
7.259
7.239
7.235
7.216
7.036
7.017
6.999
6.788
6.768
3.830
3.788
3.675
3.633
3.148
1.466
3y
8.0
7.8
7.6
7.4
7.2
7.0
ppm
2.00
1.02
1.00
2.03
1.01
2.00
1.00
1.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.00
1.01
3.00
3.05
200.808
180.245
143.765
135.605
133.679
133.220
132.372
129.719
128.110
127.974
127.603
127.159
126.320
125.480
124.136
122.296
122.157
108.245
77.365
77.047
76.730
49.676
46.009
26.283
24.809
3y
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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7.850
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7.807
7.606
7.589
7.571
7.559
7.540
7.523
7.292
7.272
7.253
7.204
7.186
7.012
6.993
6.974
6.931
6.911
3.907
3.863
3.821
3.776
3.337
1.507
3z
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 ppm
1.03
1.03
3.05
2.05
1.16
1.01
1.02
1.00
2.03
3.01
3.01
196.095
180.650
143.893
135.567
133.780
133.765
132.391
129.696
129.532
128.502
128.343
127.868
127.736
126.784
123.659
122.195
121.872
108.170
77.370
77.052
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45.442
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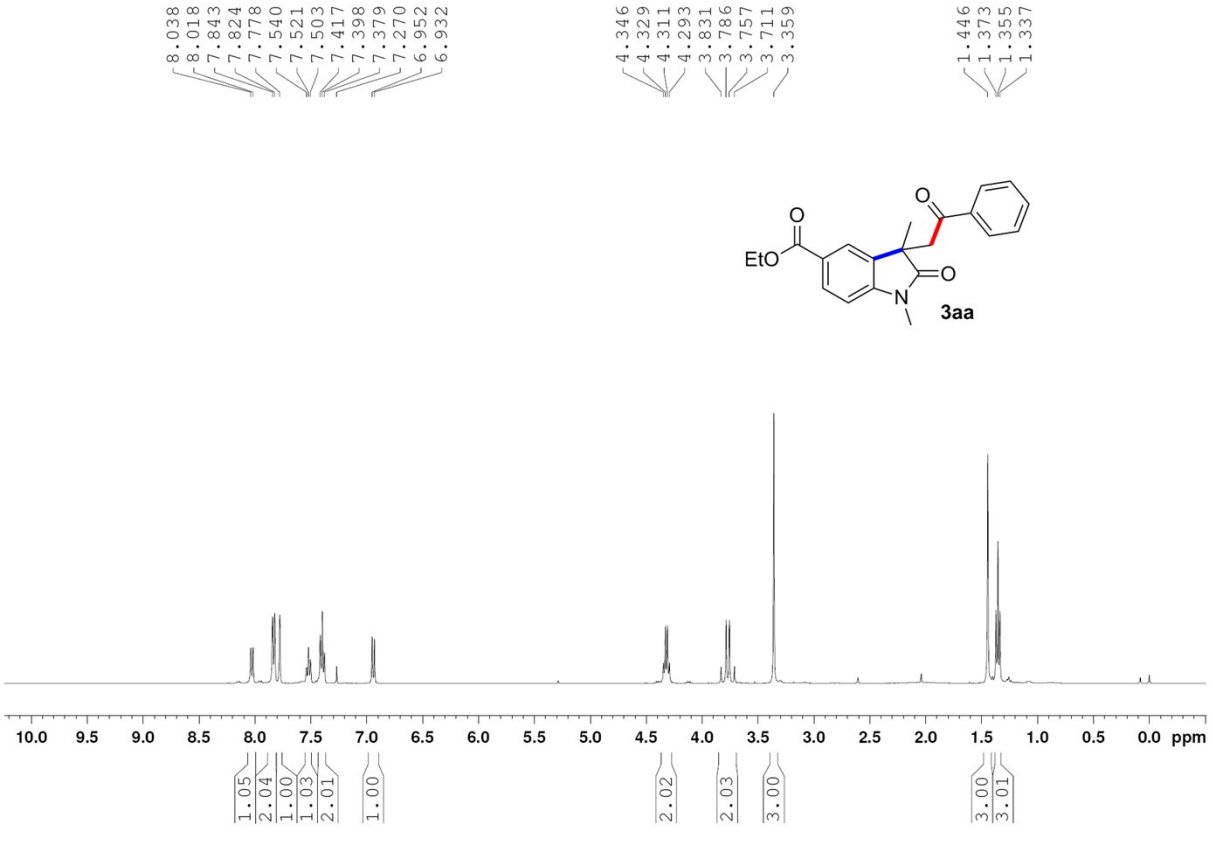
3z
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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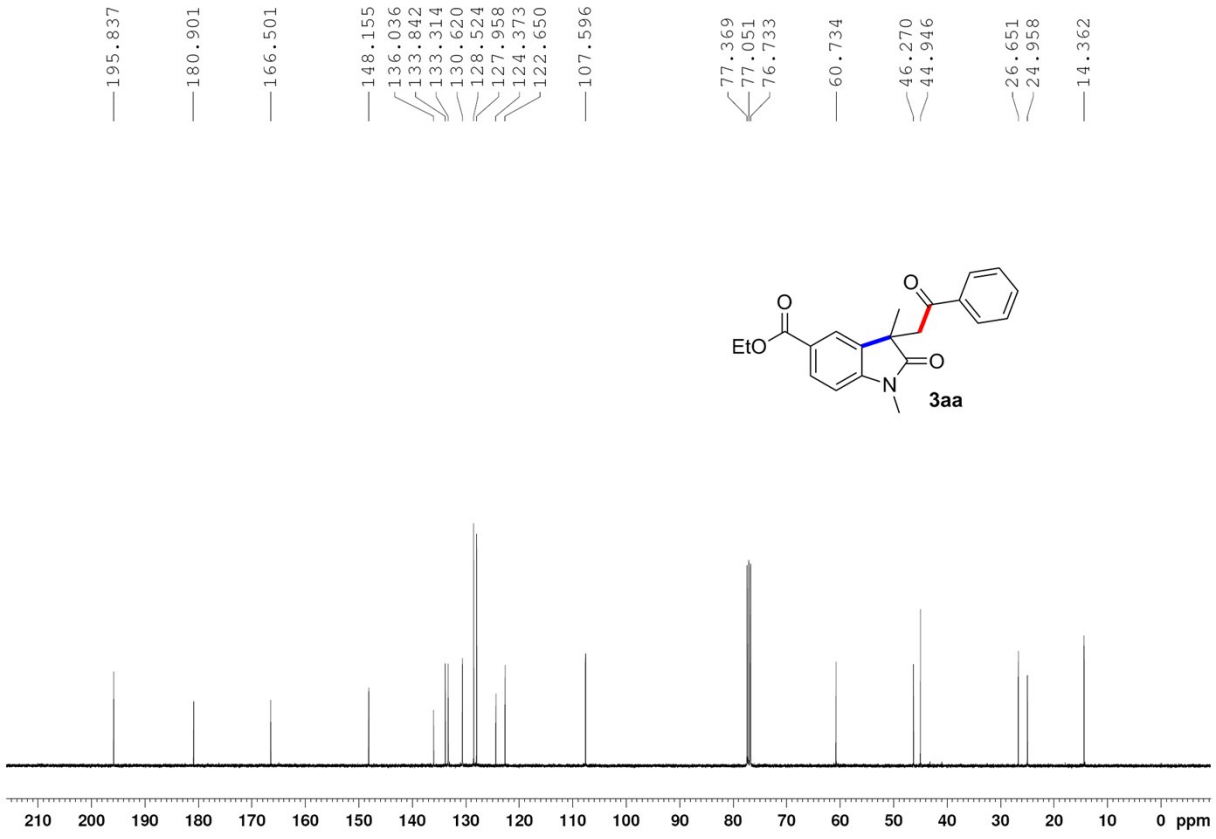
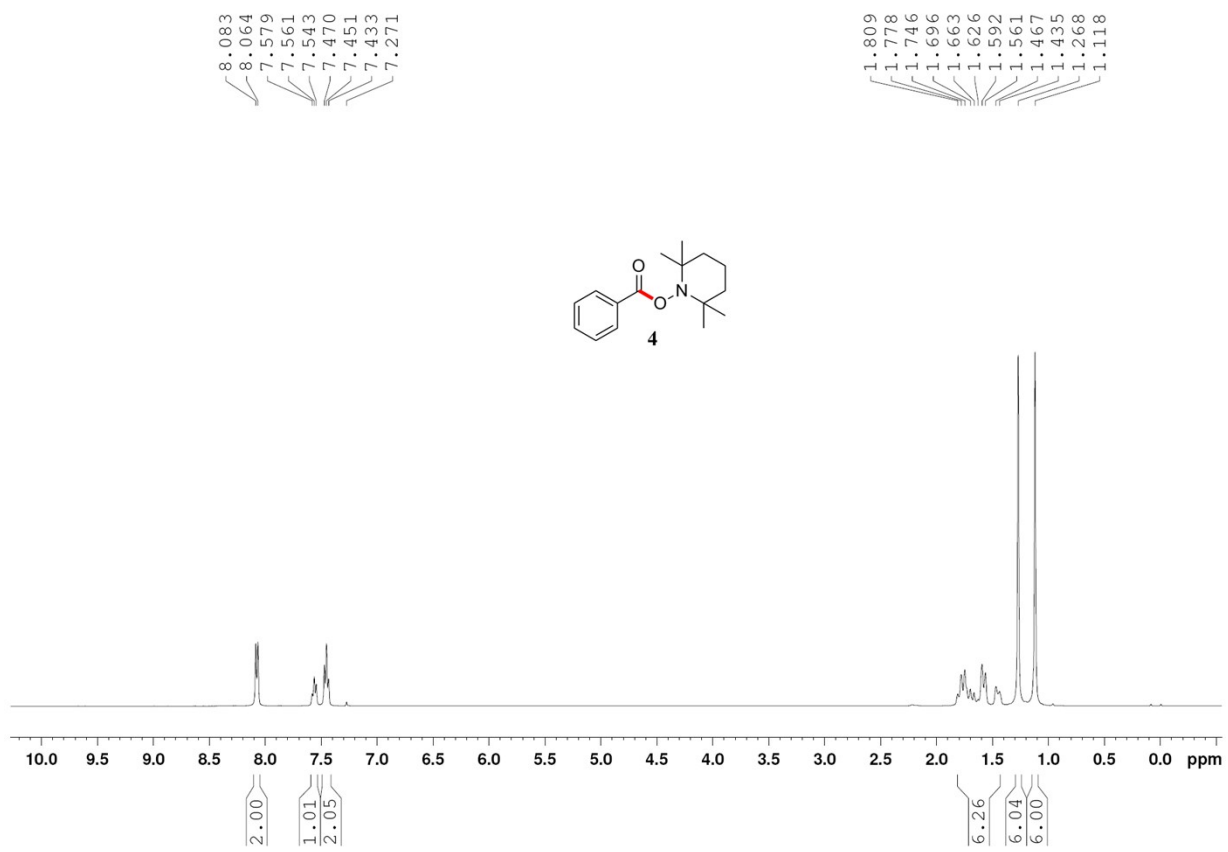
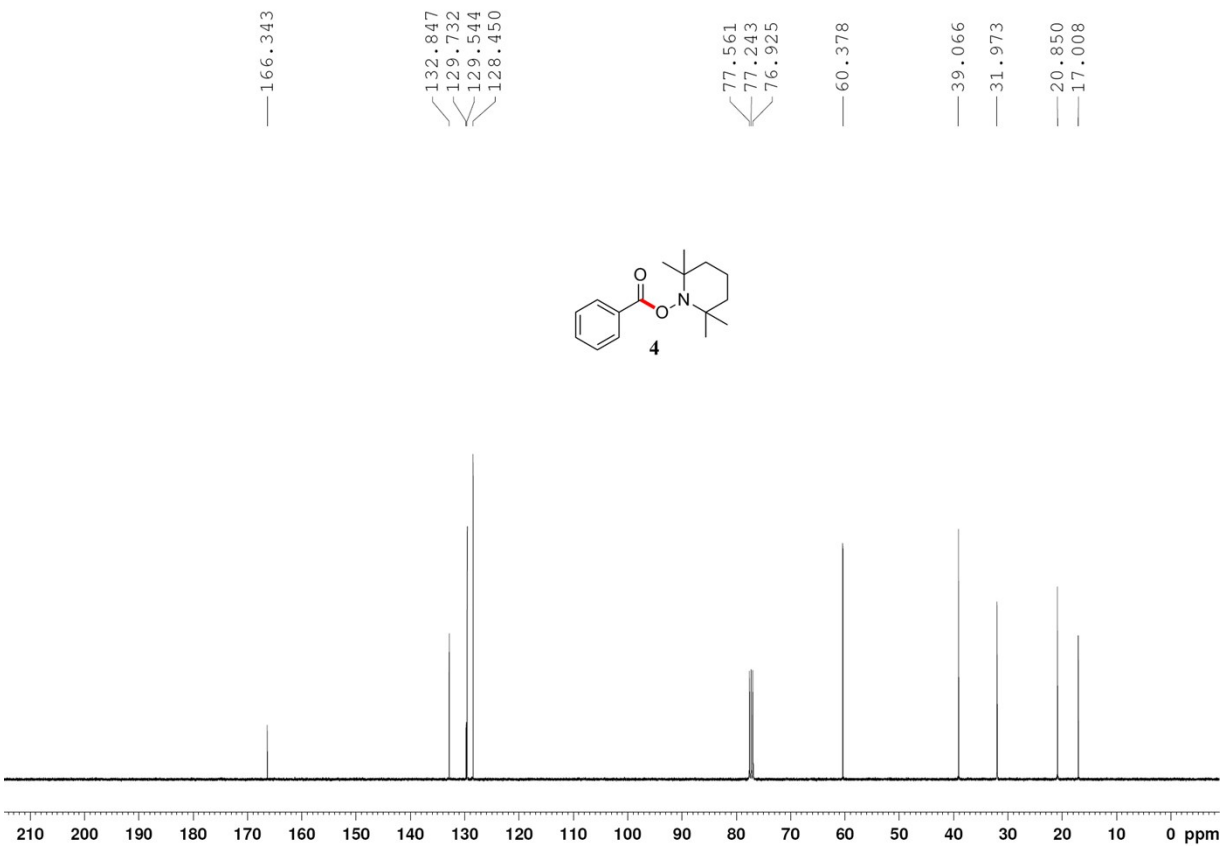
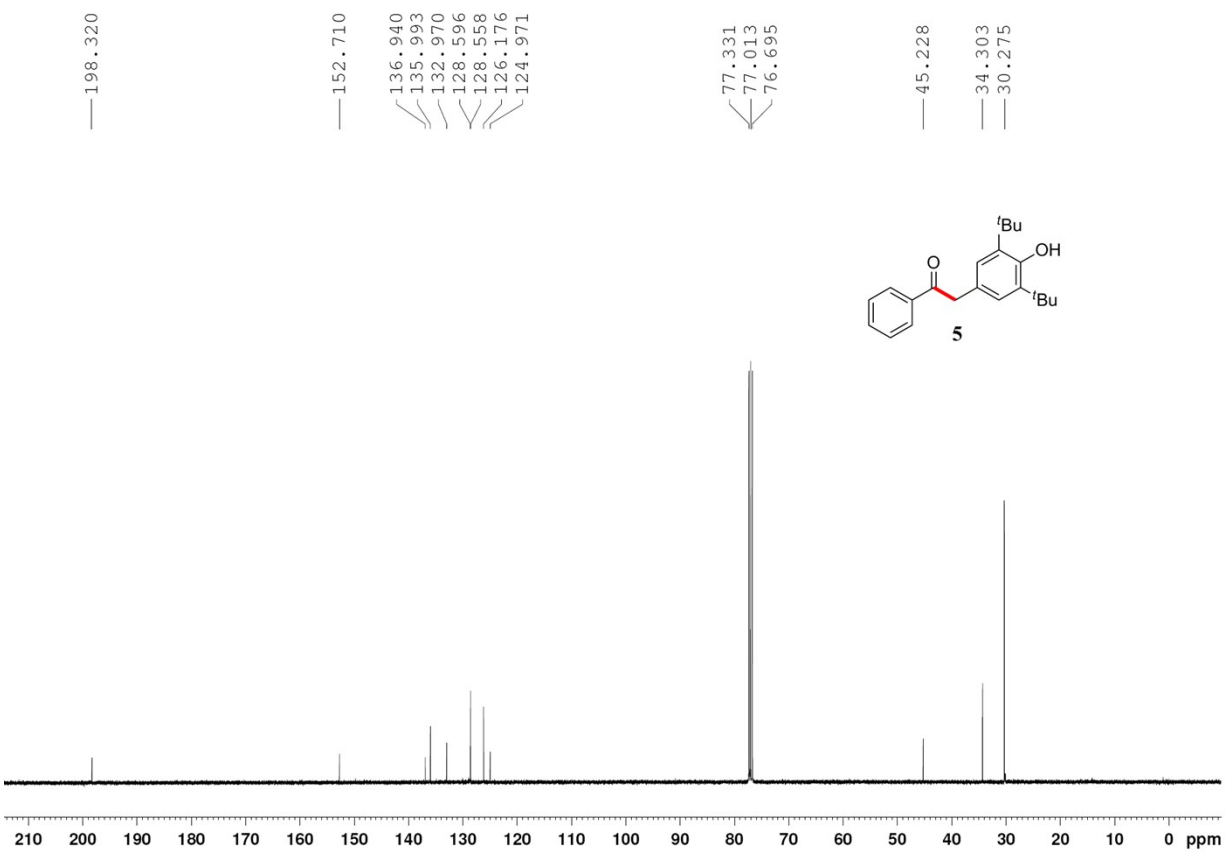
8.018
7.843
7.824
7.778
7.540
7.521
7.503
7.417
7.398
7.379
7.270
6.952
6.932
4.346
4.329
4.311
4.293
3.831
3.786
3.757
3.711
3.359
1.446
1.373
1.355
1.337
EtO
O
N
3aa
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.05
2.04
1.00
1.03
2.01
1.00
2.02
2.03
3.00
3.00
3.01

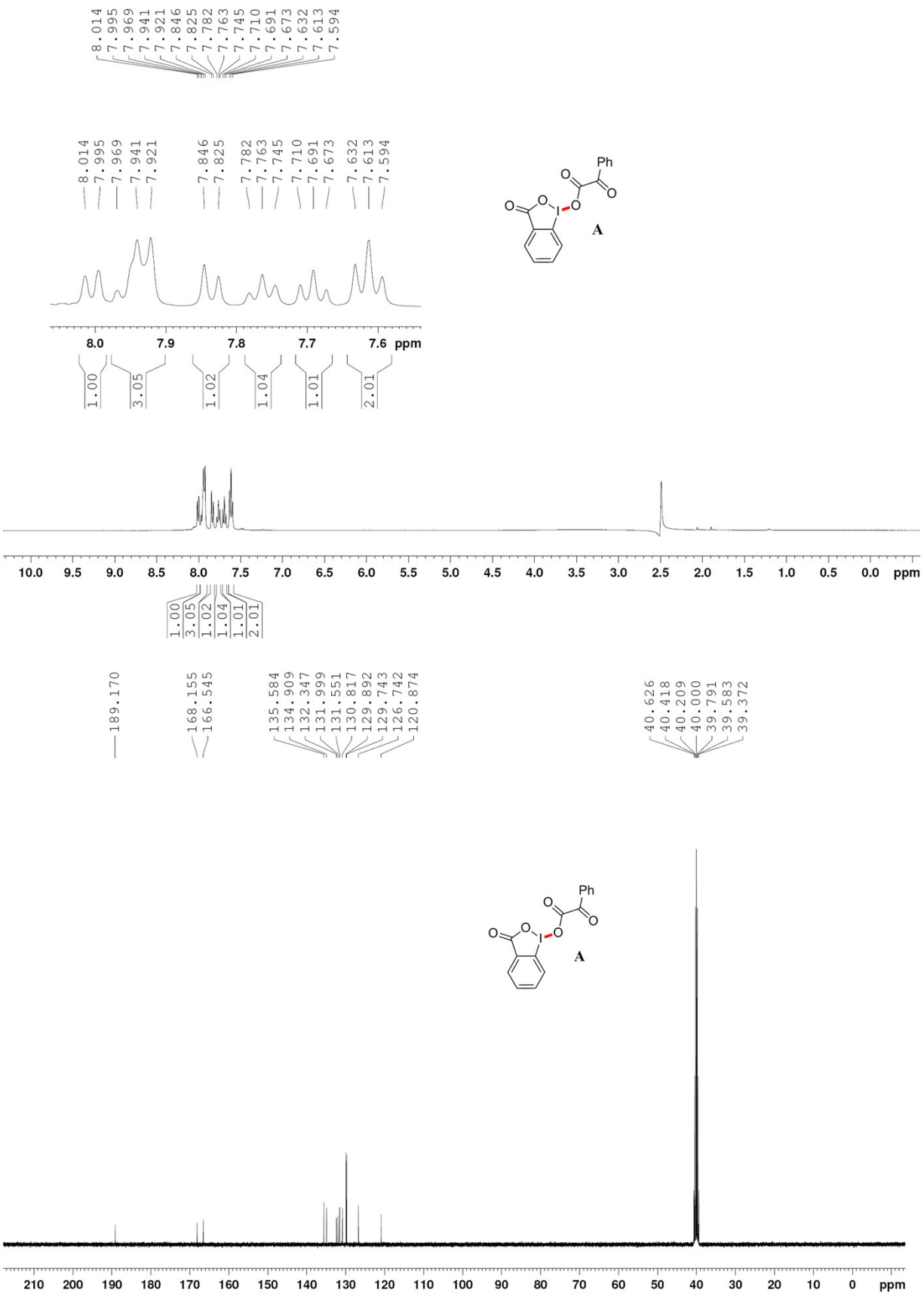

195.837
180.901
166.501
148.155
136.036
133.842
133.314
130.620
128.524
127.958
124.373
122.650
107.596
77.369
77.051
76.733
60.734
46.270
44.946
26.651
24.958
14.362
EtO
O
N
3aa
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.083
8.064
7.579
7.561
7.543
7.470
7.451
7.433
7.271
1.809
1.778
1.746
1.696
1.663
1.626
1.592
1.561
1.467
1.435
1.268
1.118
4
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 ppm
2.00
1.01
2.05
6.26
6.04
6.00


166.343
132.847
129.732
129.544
128.450
77.561
77.243
76.925
60.378
39.066
31.973
20.850
17.008
4
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.060
8.041
7.593
7.575
7.556
7.501
7.482
7.463
7.273
7.066
5.140
4.219
1.431
tBu
OH
tBu
O
5
8.0
7.8
7.6
7.4
7.2
ppm
2.03
1.01
2.02
2.00
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
ppm
2.03
1.01
2.02
2.00
1.04
2.01
18.04


198.320
152.710
136.940
135.993
132.970
128.596
128.558
126.176
124.971
77.331
77.013
76.695
45.228
34.303
30.275
tBu
OH
tBu
O
5
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.014
7.995
7.969
7.941
7.921
7.846
7.825
7.782
7.763
7.745
7.710
7.691
7.673
7.632
7.613
7.594
Ph
A
8.0
7.9
7.8
7.7
7.6 ppm
1.00
3.05
1.02
1.04
1.01
2.01
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 ppm
189.170
168.155
166.545
135.584
134.909
132.347
131.999
131.551
130.817
129.892
129.743
126.742
120.874
40.626
40.418
40.209
40.000
39.791
39.583
39.372
Ph
A
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm