Electronic Supplementary Information

Metal-free cascade oxidative decarbonylative alkylarylation of acrylamides with aliphatic aldehydes: a convenient approach to oxindoles via dual C(sp2)-H bonds functionalization

Luo Yang*,[a] Wen Lu,[a] Wang Zhou[a] and Feng Zhang*,[b]

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I. General information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Dry solvents (toluene, benzene, chlorobenzene *o*-dichlorobenzene, 1,2-dichloroethane) were used as commercially available;

Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm) or Sorbent Silica Gel 60 F254 plates. The developed chromatography was analyzed by UV lamp (254 nm). High-resolution mass spectra (HRMS) were obtained from a JEOL JMS-700 instrument (ESI). Melting points are uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Chemical shifts for ^{1}H NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent resonance as the internal standard (chloroform: δ 7.26 ppm). Chemical shifts for ^{13}C NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: δ 77.16 ppm). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet, br = broad signal), coupling constant (Hz), and integration.

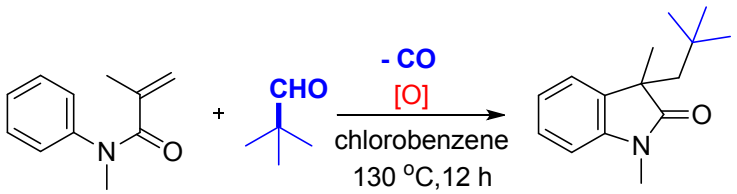
II. General experimental procedures

A general experimental procedure is described as following:

To a solution of N-methyl-N-phenylmethacrylamide (**1a**, 0.2 mmol, 1 equiv.) and pivalaldehyde (**2a**, 0.6 mmol, 3 equiv.) in chlorobenzene (1.5 mL) at ambient temperature, DTBP (0.4mmol, 2.0 equiv.) was added with vigorous stirring. The reaction mixture was stirred at 140°C (oil bath temperature) for 12 h. Afterwards The resulting mixture was cooled to room temperature, transferred to silica gel column directly and purified by column chromatography on silica gel with a mixture of EtOAc in petroleum ether as eluent to afford the pure product **3a**.

III. Condition optimization

Table S1. Optimization of the oxidants[a]

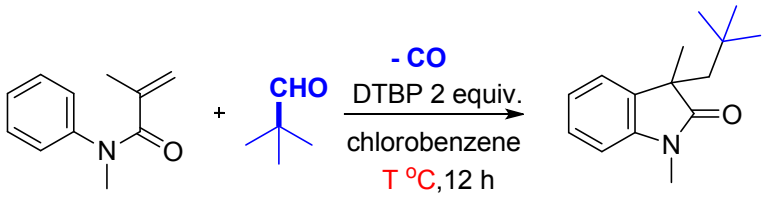


1a **2a** **3a**

Entry	Oxidant (equiv)	Yield (%)[b]
1	**DTBP**	**65**
2	BPO	26
3	DCP	45
4	H_2O_2	34
5	$PhI(OAc)_2$	41
6	TBHP	56

[a]To a solution of **1a** (0.2 mmol, 1 equiv.) and **2a** (0.6 mmol, 3 equiv.) in chlorobenzene (1.5 mL) at ambient temperature, oxidant (0.4 mmol, 2.0 equiv.) was added with vigorous stirring. The reaction mixture was stirring at 130°C for 12 h under air. [b] Isolated yields.

Table S2. Optimization of the temperature[a]

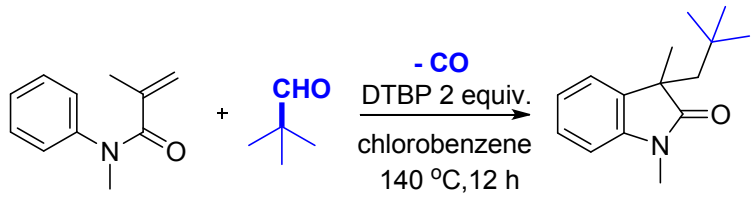


1a **2a** **3a**

Entry	Temperature (°C)	Yield (%)[b]
1	**140**	**76**
2	130	65
3	150	57

[a]To a solution of **1a** (0.2 mmol, 1 equiv.) and **2a** (0.6 mmol, 3 equiv.) in chlorobenzene (1.5 mL) at ambient temperature, DTBP (0.4 mmol, 2.0 equiv.) was added with vigorous stirring. The reaction mixture was stirring at given temperature for 12 h under air. [b] Isolated yields.

Table S3. Optimization of the reactants ratio[a]

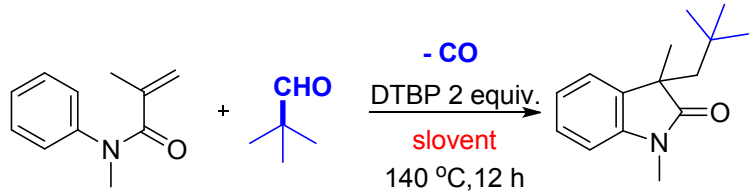


1a **2a** **3a**

Entry	2a (X equiv)	DTBP (Y equiv)	Yield (%)[b]
1	3	1.5	59
2	3	2.0	65
3	3	2.5	65
4	1.5	2.0	66
5	**3.0**	**2.0**	**76**
6	5.0	2.0	71

[a]To a solution of **1a** (0.2 mmol, 1 equiv.) and **2a** (X equiv.) in chlorobenzene (1.5 mL) at ambient temperature, DTBP (Y equiv.) was added with vigorous stirring. The reaction mixture was stirring at 140°C for 12 h under air. [b] Isolated yields.

Table S4. Optimization of the solvents[a]

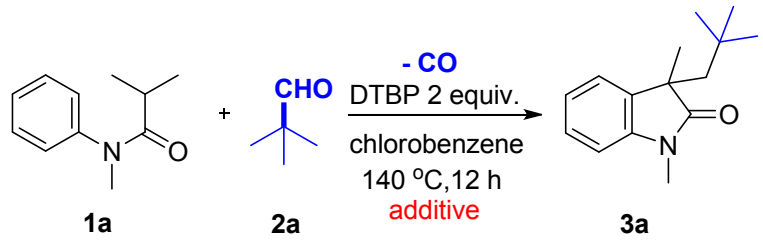


1a **2a** **3a**

Entry	Solvent (mL)	Yield (%)[b]
1	Benzene (1.5)	53
2	*o*-Dichlorobenzene (1.5)	74
3	Toluene (1.5)	33
4	Fluorobenzene (1.5)	51
5	**chorobenzene (1.5)**	**76**
6	Fluorobenzene (1.5)	36
9	1,2-Dichloroethane (1.5)	43

[a]To a solution of **1a** (0.2 mmol, 1 equiv.) and **2a** (0.6 mmol, 3 equiv.) in solvent (1.5 mL) at ambient temperature, DTBP (0.4 mmol, 2.0 equiv.) was added with vigorous stirring. The reaction mixture was stirring at 140°C for 12 h under air. [b] Isolated yields.

Table S5. Optimization of the additives[a]



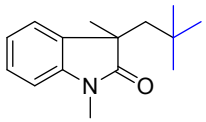
1a 2a 3a

Entry	Additive (mL)	Yield (%)[b]
1	1.5 equiv. Na_2CO_3	53
2	1.5 equiv. Pivalic acid	74

[a]To a solution of **1a** (0.2 mmol, 1 equiv.), **2a** (0.6 mmol, 3 equiv.) and additive in chlorobenzene (1.5 mL) at ambient temperature, DTBP (0.4 mmol, 2.0 equiv.) was added with vigorous stirring. The reaction mixture was stirring at 140°C for 12 h under air. [b] Isolated yields.

V. Spectra data of products 3a-3h, 4b-4x, 5, 6, 6'

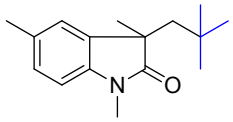
(3a) 1,3-dimethyl-3-neopentylindolin-2-one [1]



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow solid (35.1 mg, 76%).

M.p. 104-105°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, J = 7.2 Hz, 1H), 7.20 (d, J = 6.8 Hz, 1H), 7.03 (t, J = 7.2 Hz, 1H), 6.85 (d, J = 7.6Hz, 1H), 3.22 (s, 4H), 2.16 (d, J = 14.4 Hz, 1H), 1.86 (d, J = 14.4 Hz, 1H), 1.29 (s, 3H), 0.61 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.12, 142.96, 134.30, 127.64, 123.96, 122.08, 108.12, 50.89, 47.49, 31.87, 30.92, 28.38, 26.33.

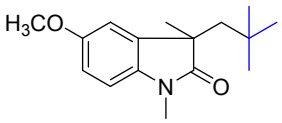
(3b) N-methyl-N-(p-tolyl)methacrylamide [2]



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-(p-tolyl)methacrylamide (**1b**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (42.14mg, 86%).

M.p. 120-121 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, J = 8.0 Hz, 1H), 7.01 (s, 1H), 6.73 (d, J = 8.0 Hz, 1H), 3.20 (s, 3H), 2.34 (s, 3H), 2.14 (d, J = 14.4 Hz, 1H), 1.83 (d, J = 14.4 Hz, 1H), 1.28 (s, 3H), 0.61 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.08, 140.63, 134.37, 131.48, 127.83, 124.80, 107.80, 50.88, 47.53, 31.86, 30.92, 28.41, 26.34, 21.23.

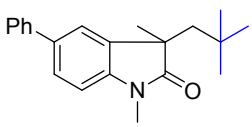
(3c) 5-methoxy-1,3-dimethyl-3-neopentylindolin-2-one [2]



The title compound was prepared according to the general procedure described above by the reaction between N-(4-methoxyphenyl)-N-methylmethacrylamide (**1c**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (39.67 mg, 76%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.85 – 6.63 (m, 3H), 3.79 (s, 3H), 3.19 (s, 3H), 2.15 (d, J = 14.4 Hz, 1H), 1.82 (d, J = 14.4 Hz, 3H), 1.28 (s, 1H), 0.63 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.80, 155.83, 136.69, 135.82, 111.88, 111.68, 108.32, 55.98, 50.92, 47.95, 31.90, 30.96, 28.47, 26.42.

(3d) N-([1,1'-biphenyl]-4-yl)-N-methylmethacrylamide



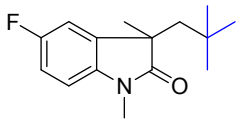
The title compound was prepared according to the general procedure described above by the

[1] J. Xie, P. Xu, H.-M Li, Q.-C Xue, H.-M Jin, Y.-X Cheng and C.- J Zhu, *Chem.Commun*. **2013**, *49*, 5672.
[2] T. Wu, H. Zhang, G.-S Liu, *Tetrahedron Lett*. **2012**, *68*, 5229.

reaction between N-([1,1'-biphenyl]-4-yl)-N-methylmethacrylamide (**1d**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (46.66 mg, 76%).

M.p. 125-126 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.2 Hz, 2H), 7.49 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.44 (t, *J* = 8.0Hz, 3H), 7.33 (t, *J* = 7.2 Hz, 1H), 6.92 (d, *J* = 8.0 Hz, 1H), 3.26 (s, 3H), 2.20 (d, *J* = 14.8 Hz, 1H), 1.92 (d, *J* = 14.4 Hz, 1H), 1.34 (s, 3H), 0.65 (s, 9H).^{13}C NMR (100 MHz, $CDCl_3$) δ 181.20, 142.38, 141.28, 135.52, 134.83, 128.90, 127.00, 126.93, 126.59, 122.95, 108.35, 50.92, 47.66, 31.93, 30.99, 28.44, 26.47. IR(cm^{-1}): 2969, 2355, 1706, 1617, 1489, 1473, 1349, 758, 688; HRMS: calcd. for $[M+H]^+$ $C_{21}H_{26}NO$: 308.20146, found: 308.20089.

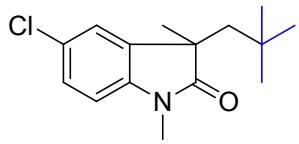
(3e) 5-fluoro-1,3-dimethyl-3-neopentylindolin-2-one [2]



The title compound was prepared according to the general procedure described above by the reaction between N-(4-fluorophenyl)-N-methylmethacrylamide (**1e**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (38.34 mg, 77%).

M.p. 121-123 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98-6.93 (m,2H), 6.76 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.21 (s, 3H), 2.16 (d, *J* = 14.4 Hz, 1H), 1.82 (d, *J* = 14.4 Hz, 1H), 1.29 (s, 3H), 0.63 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.69, 160.41, 158.03, 138.93, 136.22, 136.14, 113.95, 113.72, 112.10, 111.85, 108.54, 108.46, 50.91, 47.99, 47.97, 31.87, 30.92, 28.30, 26.45.

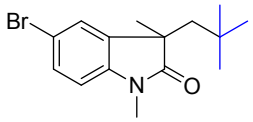
(3f) 5-chloro-1,3-dimethyl-3-neopentylindolin-2-one [2]



The title compound was prepared according to the general procedure described above by the reaction between N-(4-chlorophenyl)-N-methylmethacrylamide (**1f**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (33.86 mg, 81%).

M.p. 132-134 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.21 (m, 1H), 7.17 (s, 1H), 6.77 (d, *J* = 8.0 Hz, 1H), 3.21 (s, 3H), 2.16 (d, *J* = 14.4 Hz, 1H), 1.83 (d, *J* = 14.4 Hz, 1H), 1.29 (s, 3H), 0.63 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.56, 141.55, 136.25, 127.60, 124.41, 109.05, 50.93, 47.90, 31.93, 30.97, 28.36, 26.49.

(3g) 5-bromo-1,3-dimethyl-3-neopentylindolin-2-one [2]

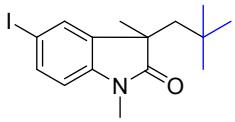


M.p. 147-149 °C;The title compound was prepared according to the general procedure described above by the reaction between N-(4-bromophenyl)-N-methylmethacrylamide (**1g**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (36.46 mg, 59%).

^{1}H NMR (400 MHz, DMSO) δ 7.39 (d, *J* = 8.0 Hz, 1H), 7.31 (s, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 3.20 (s, 3H), 2.15 (d, *J* = 14.4 Hz, 1H), 1.83 (d, *J* = 14.4 Hz, 1H), 1.29 (s, 3H), 0.62 (s, 9H).^{13}C NMR (100

MHz, $CDCl_3$) δ 180.47, 142.08, 136.64, 130.53, 127.19, 114.94, 109.60, 50.97, 47.80, 31.94, 31.00, 28.34, 26.47.

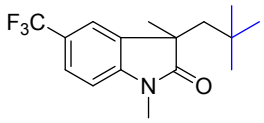
(3h)5-iodo-1,3-dimethyl-3-neopentylindolin-2-one [2]



The title compound was prepared according to the general procedure described above by the reaction between N-(4-iodophenyl)-N-methylmethacrylamide (**1h**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (59.26 mg, 83%).

M.p.110-111 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.0 Hz, 1H), 7.48 (s, 1H), 6.63 (d, *J* = 8.4 Hz, 1H), 3.19 (s, 3H), 2.13 (d, *J* = 14.4 Hz, 1H), 1.82 (d, *J* = 14.4 Hz, 1H), 1.28 (s, 3H), 0.62 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.31, 142.70, 136.94, 136.48, 132.76, 110.24, 84.71, 50.90, 47.60, 31.94, 30.99, 28.29, 26.42.

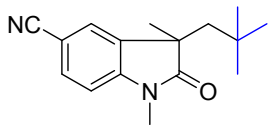
(3i) N-methyl-N-(4-(trifluoromethyl)phenyl)methacrylamide [2]



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-(4-(trifluoromethyl)phenyl)methacrylamide (**1i**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (47.24 mg, 79%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 (d, *J* = 8.0 Hz, 1H), 7.42 (s, 1H), 6.91 (d, *J* = 8.0 Hz, 1H), 3.25 (s, 3H), 2.19 (d, *J* = 14.4 Hz, 1H), 1.89 (d, *J* = 14.4 Hz, 1H), 1.32 (s, 3H), 0.60 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.04 , 181.04 , 145.97 , 135.02 , 131.36 – 131.16 (m), 126.01 , 125.49 (q, *J* = 3.9 Hz), 124.67 , 124.35 , 123.31 , 121.02 (q, *J* = 3.6 Hz), 107.87 , 50.98 , 47.53 , 31.92 , 30.93 , 28.26 , 26.58 .

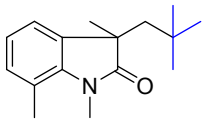
(3j) 1,3-dimethyl-3-neopentyl-2-oxoindoline-5-carbonitrile



The title compound was prepared according to the general procedure described above by the reaction between N-(4-cyanophenyl)-N-methylmethacrylamide (**1j**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light white solid(38.40 mg, 75%).

M.p.174-175°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61(s, 1H), 7.44(s, 1H), 6.92(s, 1H), 3.25(s,3H), 2.18 (d, *J* = 14.4 Hz, 1H), 1.87(d, *J* = 14.4 Hz, 1H), 1.31(s, 3H), 0.61(s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.63, 146.80, 135.42, 133.09, 127.14, 119.47, 108.59, 105.21, 50.85, 47.29, 31.87, 30.93, 28.17, 26.56. IR(cm^{-1}): 2941, 2216, 1717, 1607, 1499, 1346, 813. MS (EI) m/z(%): 256(68)$[M]^+$, 201(31), 186(100), 155(13).

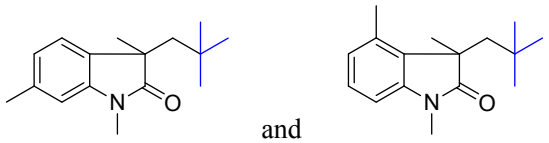
(3k) 1,3,7-trimethyl-3-neopentylindolin-2-one [2]



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-(o-tolyl)methacrylamide (**1j**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (23.03 mg, 47%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (d, J = 7.2 Hz, 1H), 6.98 (d, J = 7.2 Hz, 1H), 6.91 (t, J = 7.4 Hz, 1H), 3.50 (s, 3H), 2.59 (s, 3H), 2.13 (d, J = 14.4 Hz, 1H), 1.82 (d, J = 14.4 Hz, 1H), 1.27 (s,3H), 0.61 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.91, 140.75, 134.91, 131.31, 121.97, 121.95, 119.70, 51.11, 46.82, 31.89, 30.95, 29.70, 28.78, 19.22.

(3l) 1,3,6-trimethyl-3-neopentylindolin-2-one and 1,3,4-trimethyl-3-neopentylindolin-2-one

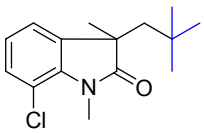


[A mixture of regio-isomers (1,3,5-trimethyl-3-neopentylindolin-2-one and 1,3,4- trimethyl-3-neopentylindolin-2-one) in a ratio of 66:34]

The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-(m-tolyl)methacrylamide (**1k**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (38.22 mg, 78%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (t, J = 7.7 Hz, 0.38×1H), 7.07 (d, J = 7.4 Hz, 0.69×1H), 6.84 (d, J = 7.3 Hz, 0.67×1H), 6.80 (d, J = 7.7 Hz, 0.36×1H), 6.69 (d, J = 13.1 Hz, 1H), 3.20 (s, 3H), 2.39 (s, 3H), 2.15-2.09 (m, 0.76×2H), 1.83 (d, J = 14.4 Hz, 0.36×2H), 1.36 (s, 0.34×3H), 1.27 (s, 0.70×3H), 0.62 (d, J = 7.4 Hz, 9H).^{13}C NMR (100 MHz, $CDCl_3$) δ 181.47, 181.14, 143.18, 142.97, 137.63, 134.97, 131.26, 127.55, 125.00, 123.69, 122.60, 109.06, 105.86, 50.83, 49.18, 48.27, 47.27, 31.82, 30.91, 30.09, 28.46, 26.37, 26.27, 25.34, 21.87, 18.83. HRMS: calcd. for $[M+H]^+C_{16}H_{24}NO$: 246.18538; found: 246.18524.

(3m) 7-chloro-1,3-dimethyl-3-neopentylindolin-2-one

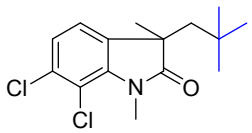


The title compound was prepared according to the general procedure described above by the reaction between N-(2-chlorophenyl)-N-methylmethacrylamide (**1l**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (31.27 mg, 59%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (d, J = 7.6Hz, 1H), 7.07 (d, J = 6.8 Hz, 1H), 6.93 (t, J = 7.6 Hz, 1H), 3.59 (s, 3H), 2.15 (d, J = 14.4 Hz, 1H), 1.83 (d, J = 14.3 Hz, 1H), 1.28 (s, 3H), 0.62 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 181.31, 138.88, 137.17, 129.97, 122.87, 122.48, 115.58, 51.17, 47.31, 31.93, 30.98, 29.71, 28.70. IR(cm^{-1}): 2957, 1718, 1605, 1583, 1470, 1364, 739. IR(cm^{-1}):2 957, 1718, 1605, 1617, 1583, 1473, 1470, 1364, 739; HRMS: calcd. for $[M+H]^+$ $C_{15}H_{21}NOCl$: 266.13112; found:266.13062.

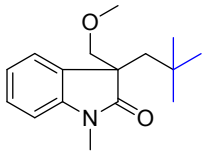
(3n) 6,7-dichloro-1,3-dimethyl-3-neopentylindolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N-(2,3-dichlorophenyl)-N-methylmethacrylamide (**1m**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (38.27 mg, 64%).

M.p. 75-76 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (d, *J* = 8.0 Hz, 1H), 7.01 (d, *J* = 7.6Hz, 1H), 3.62 (s, 3H), 2.16 (d, *J* = 14.4 Hz, 1H), 1.82 (d, *J* = 14.4 Hz, 1H), 1.27 (s, 3H), 0.63 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.31, 140.68, 135.22, 132.96, 123.63, 122.66, 114.7 7,51.10, 47.14, 31.95, 31.02, 30.01,28.67. IR(cm^{-1}): 2960, 2363, 1714, 1602, 1493, 1462, 1362, 807. HRMS: calcd.for$[M+H]^+$ $C_{15}H_{20}NOCl_2$: 300.09222; found: 266.09165.

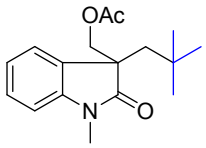
(3o)3-(methoxymethyl)-1-methyl-3-neopentylindolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N-ethyl-2-(methoxymethyl)-N-phenylacrylamide (**1n**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow solid (45.41 mg,87%).

M.p. 73-74 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.27 (m, 2H), 7.03 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 1H), 3.54 (d, *J* = 8.4 Hz, 1H), 3.44 (d, *J* = 8.8 Hz, 1H), 3.20 (d, *J* = 16.4 Hz, 6H), 2.02 (d, *J* = 14.0 Hz, 1H), 1.92 (d, *J* = 14.4 Hz, 1H), 0.62 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.83, 144.02, 131.24, 128.03, 124.89, 121.98, 108.04, 79.48, 59.69, 53.02, 45.66, 31.76, 31.12, 26.40. IR(cm^{-1}): 2953, 2365, 1702, 1612, 1468, 1380, 1344, 748. HRMS: calcd. for $[M+Na]^+C_{16}H_{23}NO_2Na$: 284.16220; found: 284.16210.

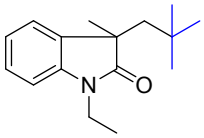
(3p) (1-methyl-3-neopentyl-2-oxoindolin-3-yl)methyl acetate



The title compound was prepared according to the general procedure described above by the reaction between 2-(ethyl(phenyl)carbamoyl)allyl acetate (**1o**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow oil (52.60 mg,91%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (t, *J* = 7.6 Hz, 1H), 7.23 (d, *J* = 7.2 Hz, 1H), 7.03 (t, *J* = 7.6Hz, 1H), 6.85 (d, *J* = 7.6 Hz, 1H), 4.41 (d, *J* = 10.4 Hz, 1H), 4.01 (d, *J* = 10.4 Hz, 1H), 3.23 (s, 3H), 2.07 (d, *J* = 14.4 Hz, 1H), 1.89 (d, *J* = 14.0 Hz, 1H), 1.81 (s, 3H), 0.64 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 178.05, 170.43, 144.03, 129.66, 128.47, 125.00, 122.18, 108.14, 69.55, 51.66, 45.46, 31.78, 31.07, 26.44, 20.63. IR(cm^{-1}): 3060, 2949, 2364, 1719, 1618, 1498, 1467, 1372, 1215, 750. HRMS: calcd. for $[M+Na]^+C_{17}H_{23}NO_3Na$: 312.15710; found: 312.15701.

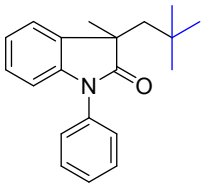
(3q) 1-ethyl-3-methyl-3-neopentylindolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N-ethyl-N-phenylmethacrylamide (**1p**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow solid (31.85 mg, 65%).

M.p. 60-61 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (dd, *J* = 13.6, 8.0 Hz, 2H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 7.6 Hz, 1H), 3.915-3.826(m, 1H), 3.730-3.641 (m,1H),2.16 (d, *J* = 14.4 Hz, 1H), 1.86 (d, *J* = 14.4 Hz, 1H), 1.36 – 1.20 (m, 3H), 0.63 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.70, 142.08, 134.59, 127.55, 124.22, 121.83, 108.29, 50.70, 47.53, 34.64, 31.98, 31.02, 28.76, 12.37. IR(cm^{-1}): 2955, 2363, 1703, 1610, 1487, 1466, 1374, 753. HRMS: calcd. for $[M+H]^+C_{16}H_{24}NO$: 246.18547; found: 246.18524.

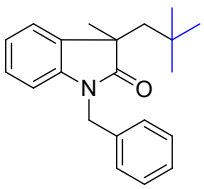
(3r) 3-methyl-3-neopentyl-1-phenylindolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N,N-diphenylmethacrylamide (**1q**) with pivalaldehyde (**2a**), and purified by flash column chromatography as white solid (42.19 mg, 72%).

M.p. 103-104 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (t, *J* = 7.6 Hz, 2H), 7.40 (dd, *J* = 13.6, 7.6 Hz, 3H), 7.27 (d, *J* = 7.6 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 7.07 (t, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 2.25 (d, *J* = 14.4 Hz, 1H), 1.95 (d, *J* = 14.4 Hz, 1H), 1.42 (s, 3H), 0.73 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.43, 142.83, 135.00, 134.12, 129.67, 127.91, 127.55, 126.42, 124.39, 122.56, 109.57, 51.13, 47.73, 32.10, 31.15, 29.03. IR(cm^{-1}): 2949, 2363, 1713, 1608, 1496, 1470, 1375, 754, 692.

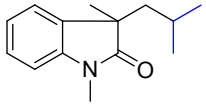
(3s) 1-benzyl-3-methyl-3-neopentylindolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N-benzyl-N-phenylmethacrylamide (**1r**) with pivalaldehyde (**2a**), and purified by flash column chromatography as light yellow solid (38.07 mg, 62%).

M.p. 107-108 °C;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.27 (m, 5H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 7.2 Hz, 1H), 6.77 (d, *J* = 7.6 Hz, 1H), 5.06 (d, *J* = 15.6 Hz, 1H), 4.78 (d, *J* = 15.6 Hz, 1H), 2.21 (d, *J* = 14.4 Hz, 1H), 1.90 (d, *J* = 14.4 Hz, 1H), 1.35 (s, 4H), 0.64 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.22, 142.20, 136.27, 134.39, 128.82, 127.77, 127.67, 127.54, 124.12, 122.10, 109.26, 50.67, 47.64, 44.08, 32.00, 31.08,29.19. IR(cm^{-1}): 2949, 2356, 1705, 1607, 1493, 1466, 1357, 758, 693. HRMS: calcd. for $[M+H]^+$ $C_{21}H_{26}NO$: 308.20102, found: 308.20089.

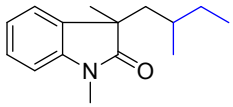
(4b) 3-isobutyl-1,3-dimethylindolin-2-one [1]



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with isobutylaldehyde (**2b**), and purified by flash column chromatography as light yellow oil (27.78 mg, 64%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 7.06 (t, *J* = 7.6 Hz,1H), 6.84 (d, *J* = 7.6Hz, 1H), 3.22 (s, 3H), 1.94 (dd, *J* =14.0, 7.6 Hz, 1H), 1.76 (dd, *J* = 14, 5.6 Hz, 1H), 1.32 (s, 3H), 1.24 (m, 1H), 0.65 (d, *J* = 6.8 Hz, 3H), 0.61 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.19, 143.29, 134.31, 127.66, 122.91, 122.43, 108.05, 48.18, 46.84, 26.28, 26.24, 25.63, 24.22,22.93.

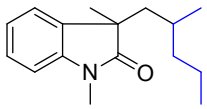
(4c)1,3-dimethyl-3-(2-methylbutyl)indolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with 2-methylbutyraldehyde (**2c**), and purified by flash column chromatography as light yellow oil (34.19 mg, 74%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.24 (m, 1H), 7.18-7.14 (m, 1H), 7.08-7.03 (m, 1H), 6.86-6.83 (m, 1H), 3.21 (s, 3H), 2.05-2.00 (m, 0.57×1H), 1.86 (d, *J* = 5.7 Hz, 1H), 1.68-1.64 (m, 0.60×1H), 1.33 (s, 3H), 1.16-0.92 (m, 3H), 0.74-0.69 (m, 3H), 0.60 (d, *J* = 6.2 Hz, 0.59×3H), 0.49 (d, *J* = 6.4 Hz, 0.52×3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.40, 143.30, 134.65, 134.13, 127.64, 122.95, 122.88, 122.42, 122.37, 110.09, 108.02, 108.00, 48.26, 48.00, 45.12, 44.51, 31.74, 31.67, 30.82, 30.15, 26.29, 26.23, 25.88, 20.41, 19.37, 11.16, 11.03. IR(cm^{-1}): 3059, 2933, 1705, 1607, 1496, 1467, 1374, 756. HRMS: calcd. for $[M+H]^+$ $C_{15}H_{22}NO$: 232.16995, found: 232.16959.

(4d) 1,3-dimethyl-3-(2-methylpentyl)indolin-2-one [3]

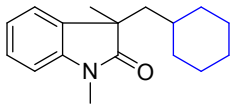


The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with methyl valeraldehyde (**2d**), and purified by flash column chromatography as light yellow oil (36.75 mg, 75%).

1H NMR (400 MHz, $CDCl_3$) δ 7.28-7.24 (m, 1H), 7.18-7.14 (m, 1H), 7.08-7.03 (m, 1H), 6.85-6.82 (m, 1H), 3.21 (s, 3H), 2.04-1.99 (m, 0.51×1H), 1.86 (d, *J* = 6.0 Hz, 1H), 1.68-1.63 (m, 0.53×1H), 1.33 (s, 3H), 1.26-0.89 (m, 5H), 0.77-0.69 (m, 3H), 0.60 (d, *J* = 6.4 Hz, 0.53×3H), 0.50 (d, *J* = *6.5* Hz, 0.63×3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.97, 142.06, 136.45, 129.64, 127.63, 127.01, 126.92, 124.88, 118.93, 37.85, 29.72, 20.27, 12.49.

(4e) 3-(cyclohexylmethyl)-1,3-dimethylindolin-2-one [3]

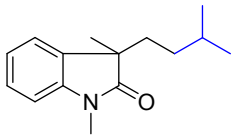
[3] Z.-J Li, Y. Zhang, L.-Z Zhang, and Z.-Q Liu, *Org. Lett*. **2014**, *16*, 382.



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with cyclohexanecarbaldehyde (**2e**), and purified by flash column chromatography as light yellow oil (38.55 mg, 75%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (s, 1H), 7.15 (d, *J* = 6.4 Hz, 1H), 7.06 (d, *J* = 6.4 Hz, 1H), 6.84 (d, *J* = 6.8 Hz, 1H), 3.22 (s, 3H), 1.92 (dd, *J* = 13.6, 6.0 Hz, 1H), 1.72 (d, *J* = 13.6 Hz, 1H), 1.46 (d, *J* = 9.2 Hz, 3H), 1.31 (s, 3H), 1.20 (d, *J* = 12.4 Hz, 1H), 1.09 – 0.65 (m, 7H).^{13}C NMR (100 MHz, $CDCl_3$) δ181.27, 143.15, 134.45, 127.61, 122.80, 122.44, 108.06, 47.95, 45.48, 34.82, 34.53, 33.58, 26.31, 26.17, 26.11.

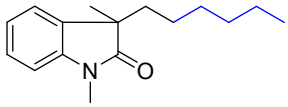
(4f) 3-isopentyl-1,3-dimethylindolin-2-one



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with 3-methylbutanal (**2f**), and purified by flash column chromatography as light yellow oil (15.25 mg, 33%).

^{1}H NMR (400 MHz, $CDCl_3$) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, *J* = 14.4 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 7.06 (t, *J* = 7.6 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 1.92-1.84 (m, 1H), 1.76-1.69 (m, 1H), 1.35 (s, 3H), 0.96 – 0.82 (m, 1H), 0.77 (t, *J* = 6.8Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 181.03, 143.46, 134.44, 127.68, 122.56, 107.99, 48.49, 36.48, 33.30, 28.28, 26.24, 24.00, 22.61, 22.40. IR(cm-1): 3057, 2962, 1703, 1610, 1495, 1472, 1387, 753. HRMS: calcd. for $[M+H]^+$ $C_{15}H_{22}NO$:232.16998found: 232.16959.

(4g) 3-hexyl-1,3-dimethylindolin-2-one [3]

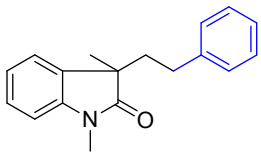


The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with hexanal (**2g**), and purified by flash column chromatography as light yellow oil (21.07 mg, 43%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, *J* = 8.0 Hz, 1H), 7.17 (d, *J* = 6.4 Hz, 1H), 7.07 (t, *J* = 7.2 Hz, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 3.21 (s, 3H), 1.88 (td, *J* = 12.8, 4.4 Hz, 1H), 1.72 (td, *J* = 12.8, 4.4 Hz, 1H), 1.35 (s, 3H), 1.27 – 0.89 (m, 8H), 0.80 (t, *J* = 6.8 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 181.03, 143.44, 134.45, 127.68, 122.58, 122.53, 107.97, 48.59, 38.66, 31.64, 29.52, 26.23, 24.52, 23.92, 22.67, 14.14.

(4h)1,3-dimethyl-3-phenethylindolin-2-one [4]

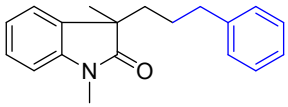
[4] M.-Bo Zhou, C.-Y Wang, R.-J Song, Y.- L, W.-T Wei, and J.-H Li, *Chem.Commun*. **2013**, *49*, 10817.



The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with 2-phenylacetaldehyde (**2h**), and purified by flash column chromatography as light yellow oil (30.74mg, 58%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 15.8,7.6 Hz, 1H), 7.21 (dd, *J* = 13.6,6.8 Hz, 3H), 7.12 (dd, *J* = 14.8, 7.2 Hz, 2H), 7.02 (d, *J* = 7.2 Hz, 2H), 6.87 (d, *J* = 7.6Hz, 1H), 3.21 (s, 3H), 2.43 – 2.20 (m, 2H), 2.19 – 2.08 (m, 1H), 2.07 – 1.91 (m, 1H), 1.40 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.46, 143.53, 141.50, 133.86, 128.39, 128.34, 127.95, 125.95, 122.71, 122.59, 108.13, 48.49, 40.35, 31.08, 26.24, 24.07.

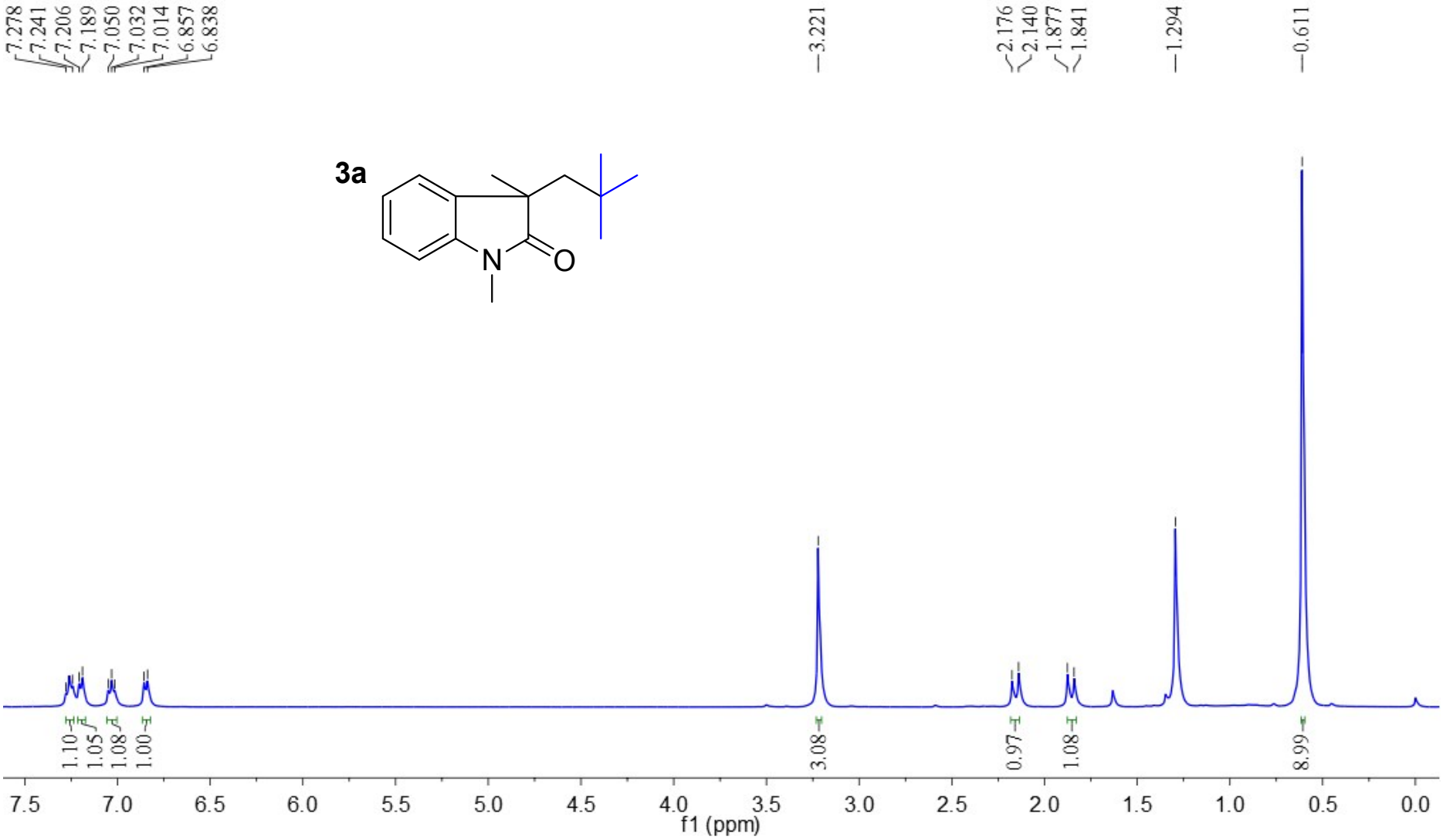
(4i)1,3-dimethyl-3-(3-phenylpropyl)indolin-2-one [1]

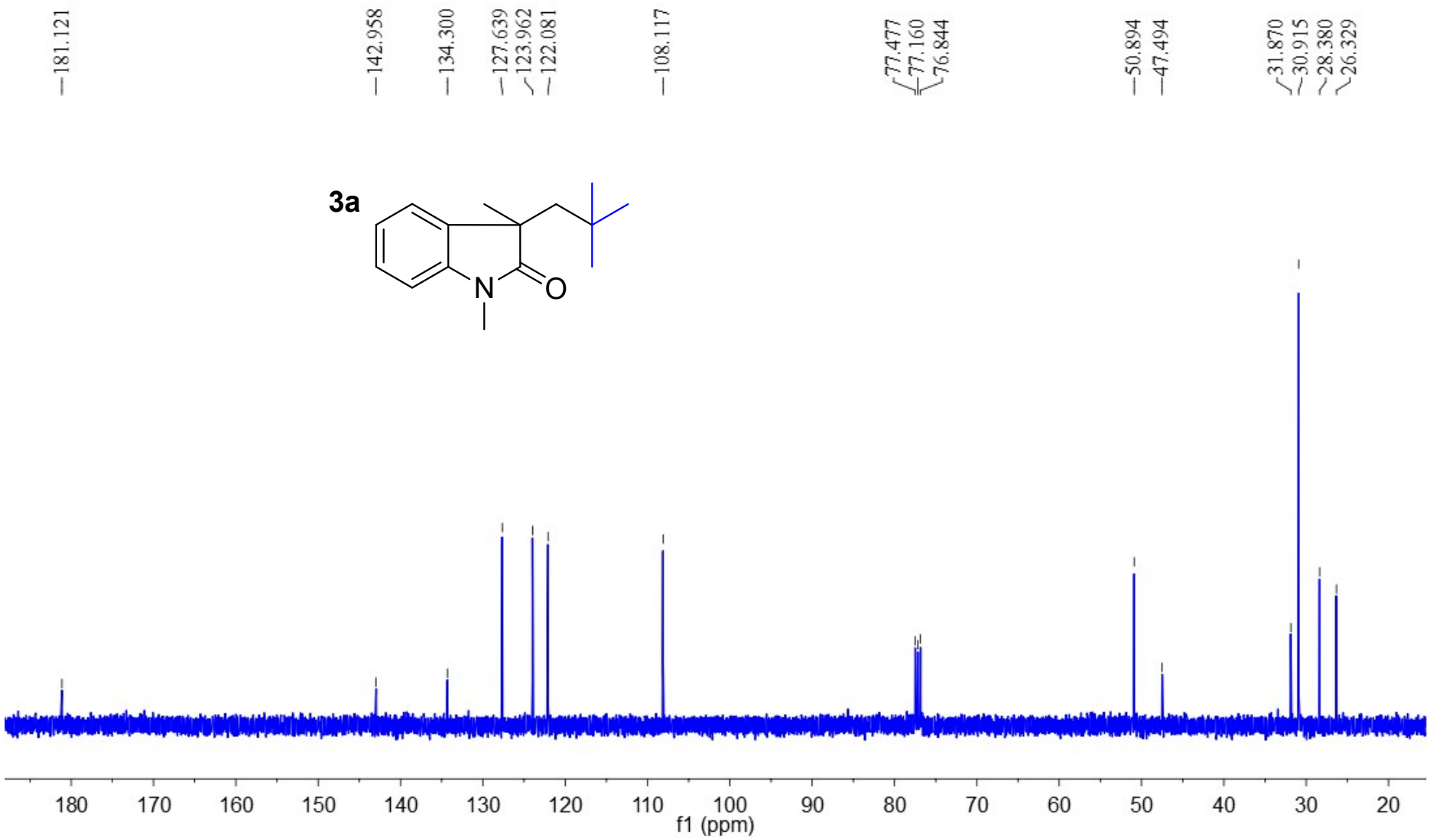


The title compound was prepared according to the general procedure described above by the reaction between N-methyl-N-phenylmethacrylamide (**1a**) with 3-phenylpropanal (**2i**), and purified by flash column chromatography as light yellow oil (25.11 mg, 45%).

^{1}H NMR (400 MHz, D_2O) δ 7.22 (dd, *J* = 13.6, 7.2 Hz, 3H), 7.13 (t, *J* = 7.2 Hz, 2H), 7.05 (t, *J* = 7.2 Hz, 3H), 6.82 (d, *J* = 7.6 Hz, 1H), 3.20 (s, 3H), 2.80 – 2.28 (m, 2H), 1.96 (td, *J* = 13.2, 4.4 Hz, 1H), 1.78 (td, *J* = 12.8, 4.0 Hz, 1H), 1.34 (s, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.85, 143.42, 142.04, 134.16, 128.48, 128.37, 127.80, 125.87, 122.60, 108.07, 48.49, 38.27, 36.09, 26.51, 26.27, 24.01.

VI. Copies of 1H and 13C NMR spectra of products




181.121
142.958
134.300
127.639
123.962
122.081
108.117
77.477
77.160
76.844
50.894
47.494
31.870
30.915
28.380
26.329
3a
N
O
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150
140
130
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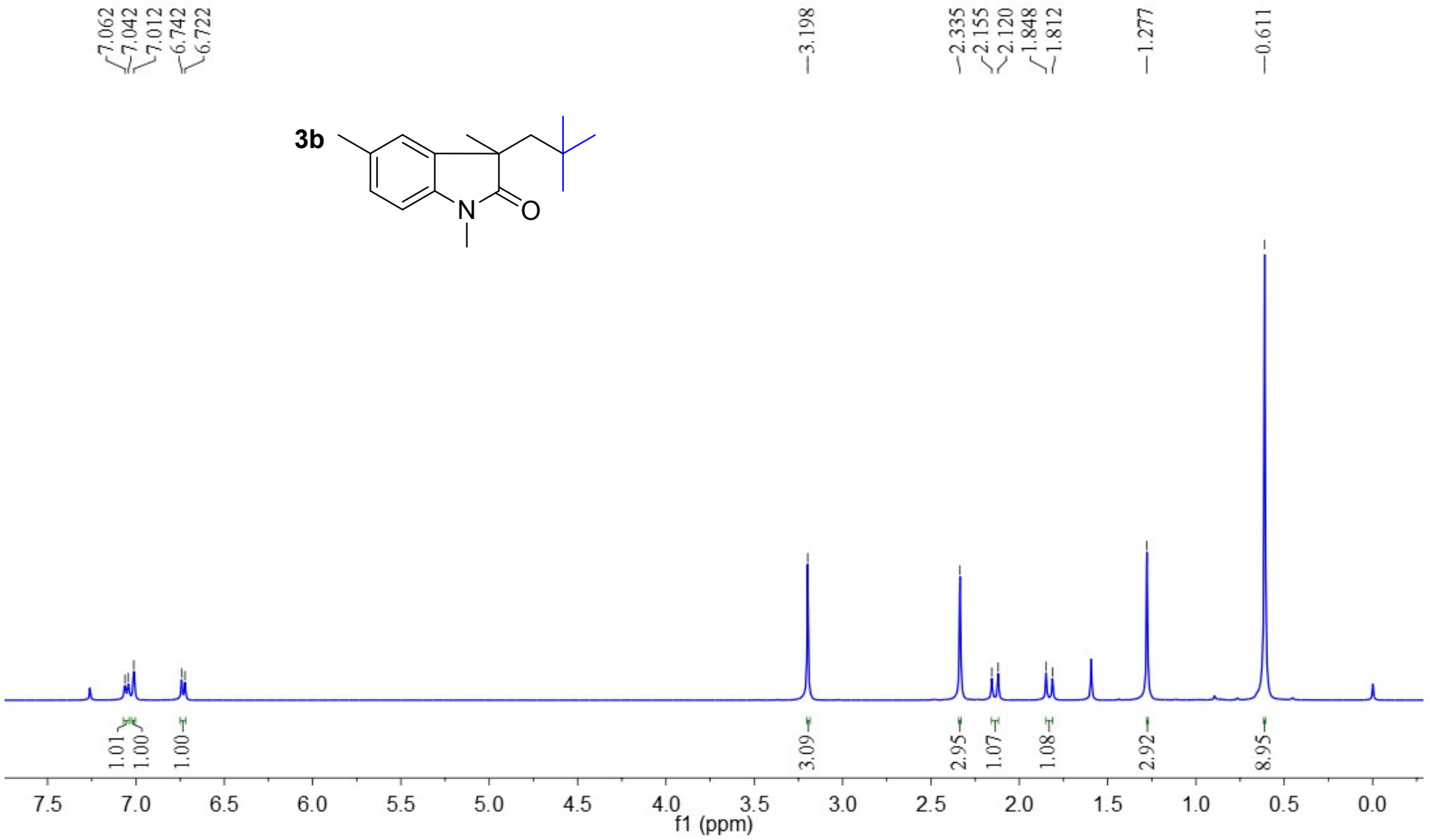
30
20
f1 (ppm)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7.012
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2.335
2.155
2.120
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1.812
1.277
0.611
3b
N
O
1.01
1.00
1.00
3.09
2.95
1.07
1.08
2.92
8.95
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
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2.5
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1.0
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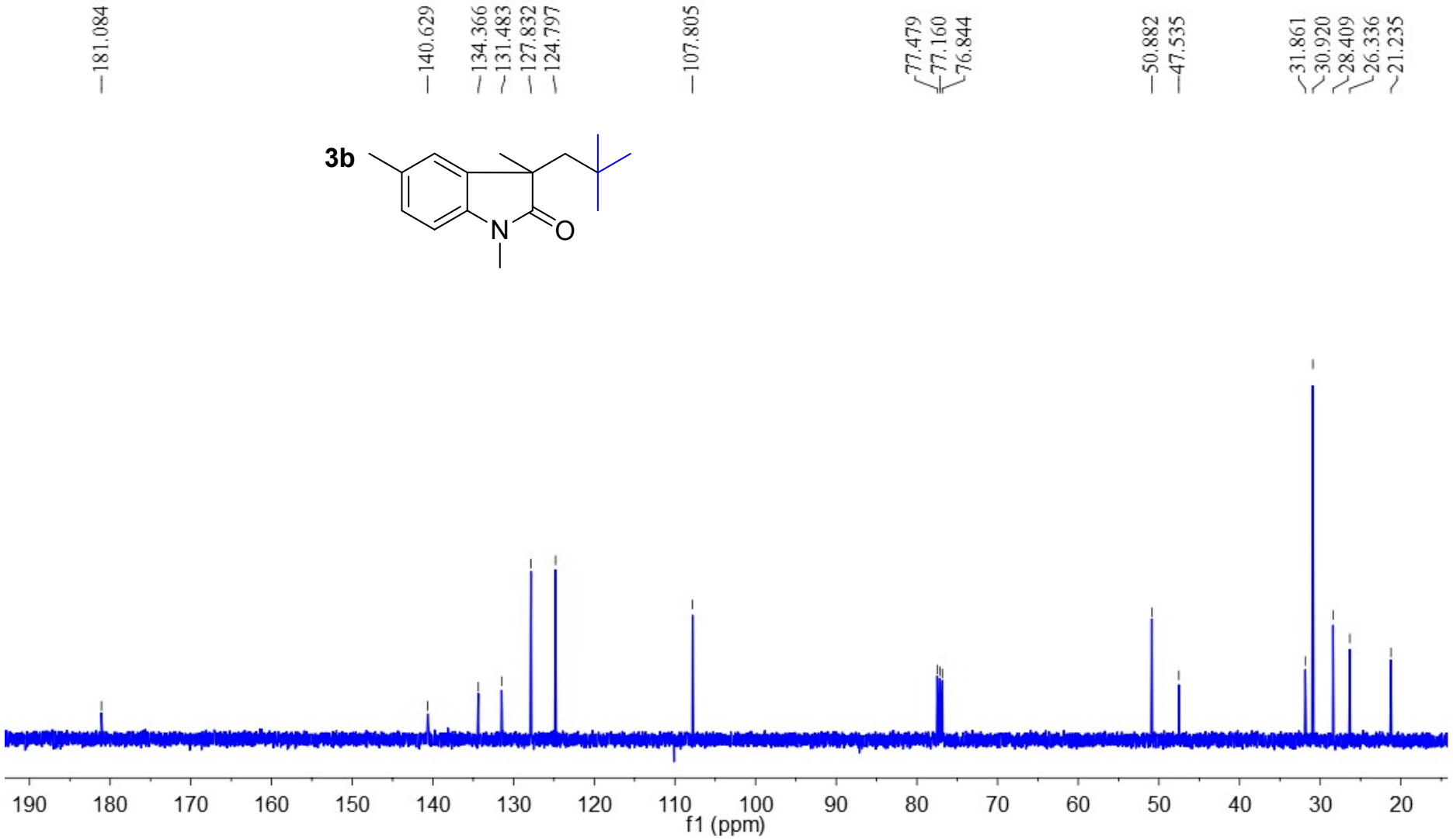
f1 (ppm)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107.805
77.479
77.160
76.844
50.882
47.535
31.861
30.920
28.409
26.336
21.235
3b
N
O
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170
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150
140
130
120
110
100
90
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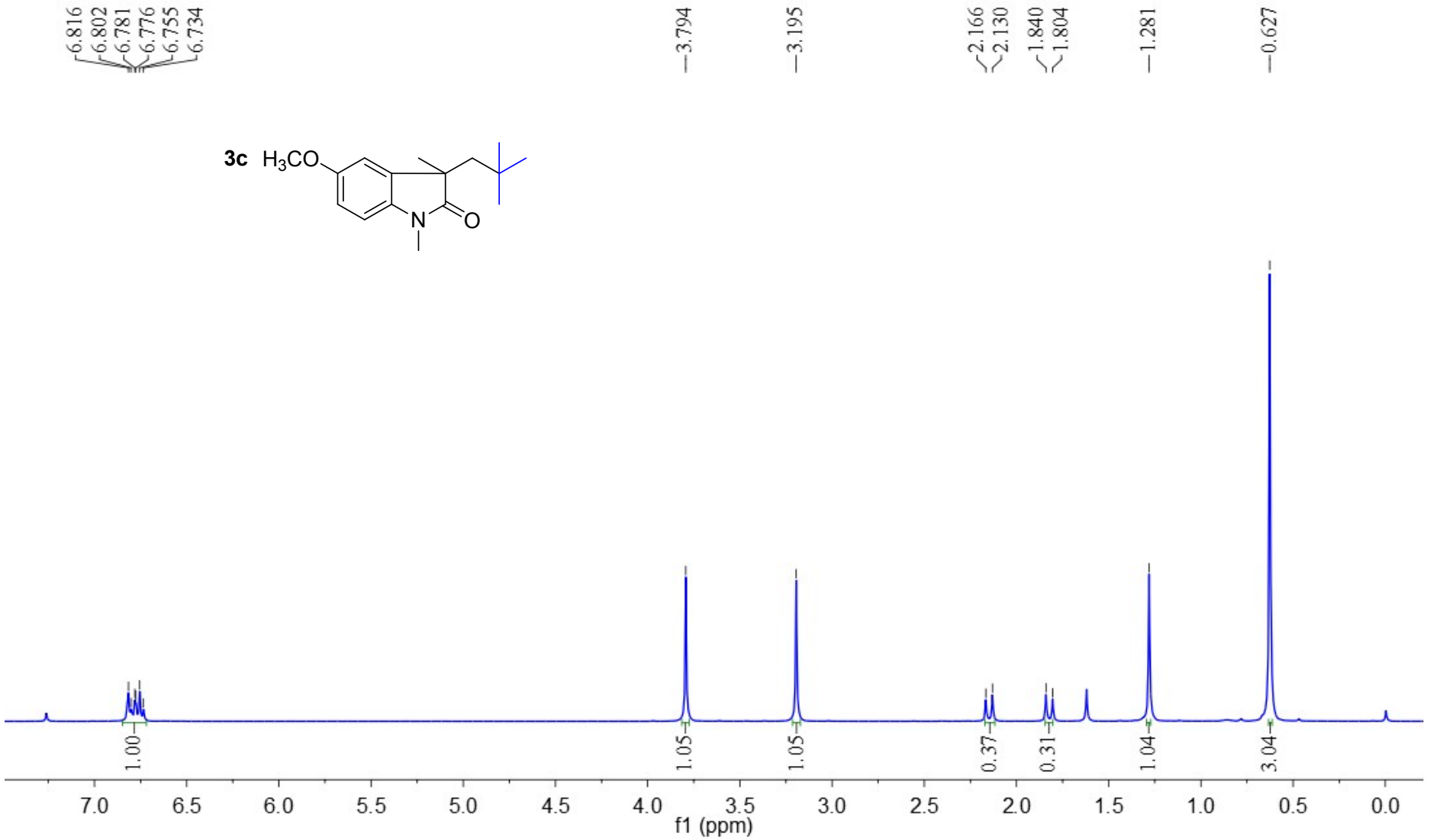
70
60
50
40
30
20
f1 (ppm)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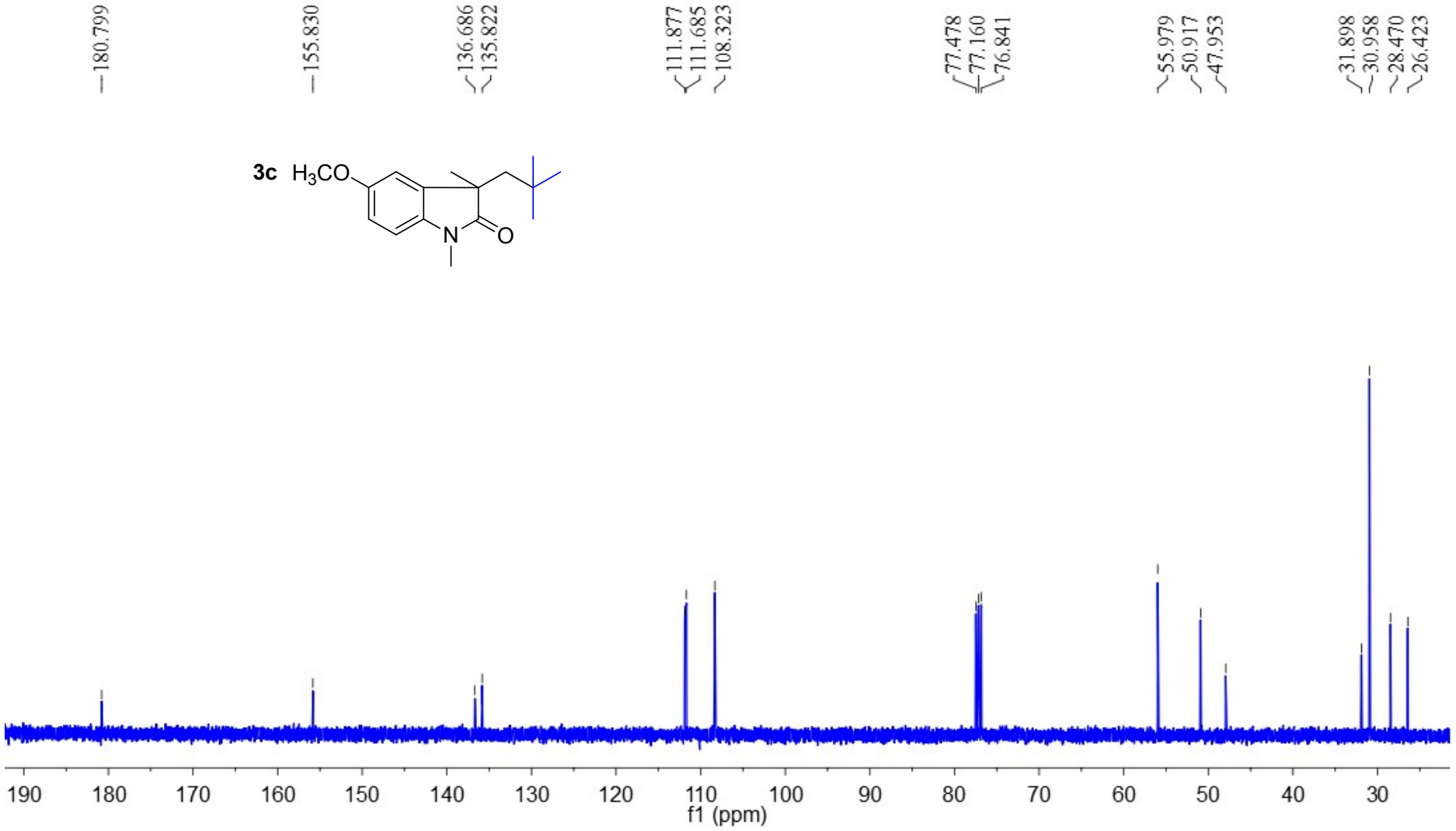
6.802
6.781
6.776
6.755
6.734
3.794
3.195
2.166
2.130
1.840
1.804
1.281
0.627
3c
H3CO
N
O
1.00
1.05
1.05
0.37
0.31
1.04
3.04
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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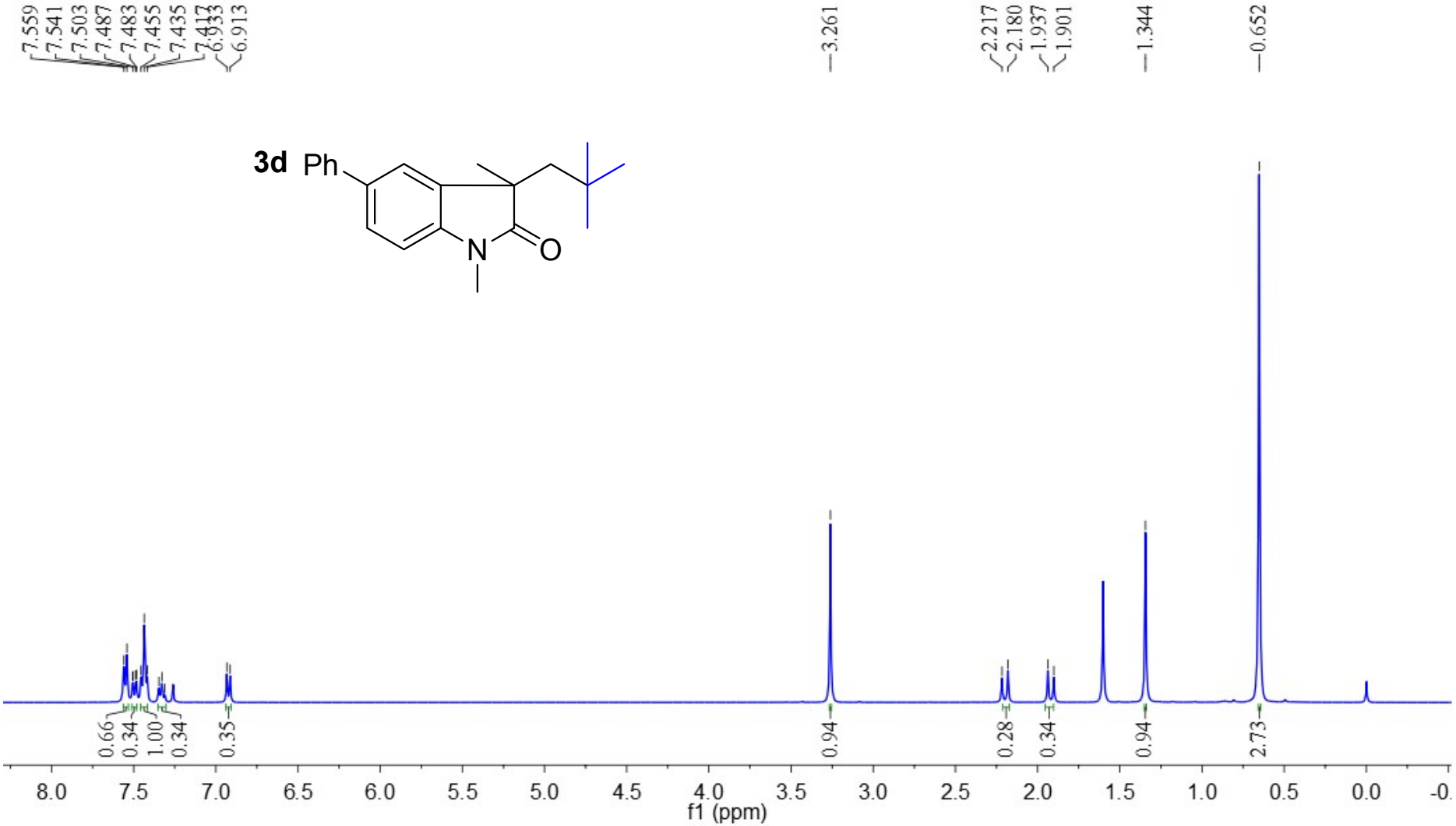
136.686
135.822
111.877
111.685
108.323
77.478
77.160
76.841
55.979
50.917
47.953
31.898
30.958
28.470
26.423
3c
H3CO
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)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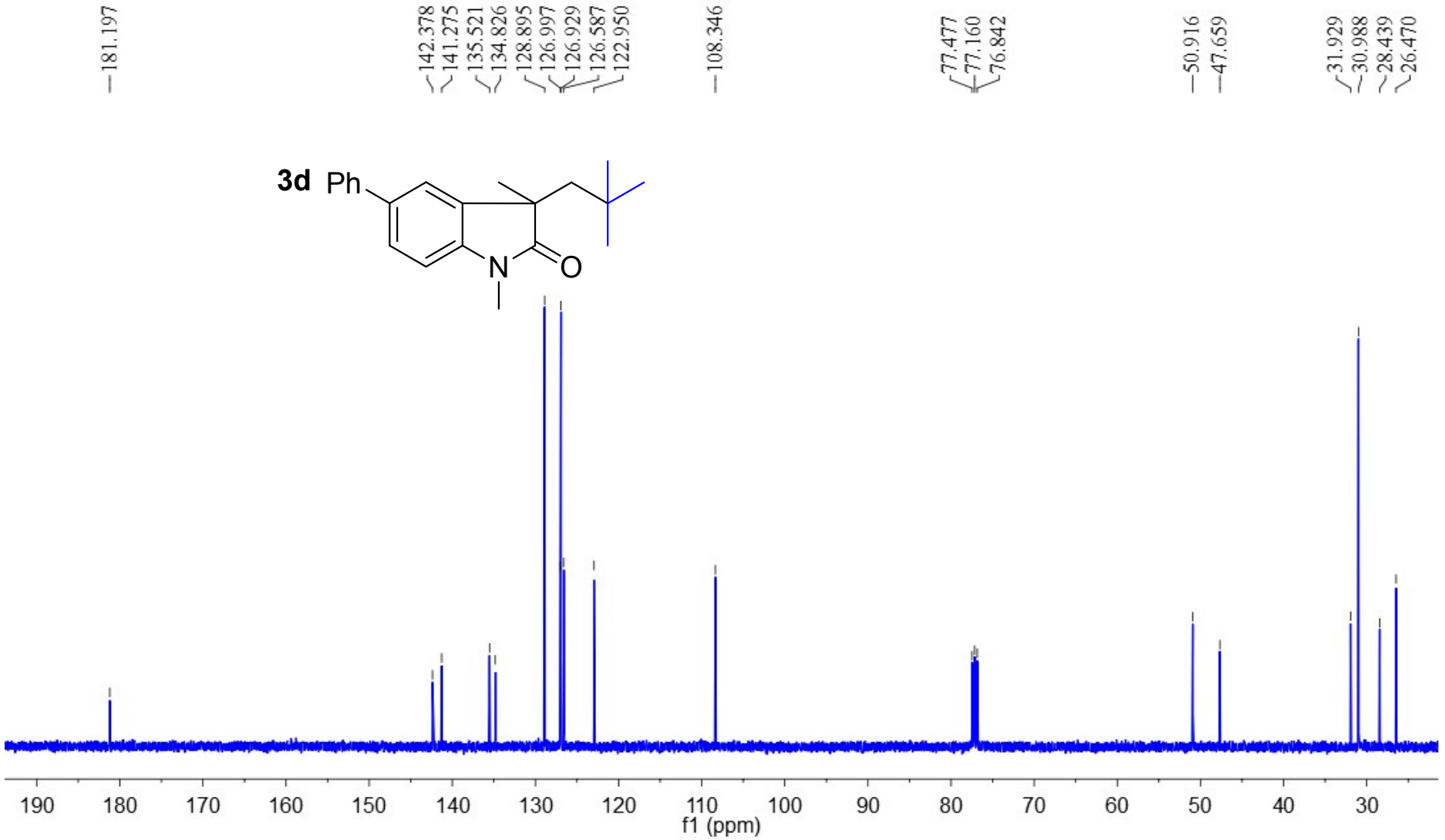
7.541
7.503
7.487
7.483
7.455
7.435
7.417
6.933
6.913
3.261
2.217
2.180
1.937
1.901
1.344
0.652
3d
Ph
N
O
0.66
0.34
1.00
0.34
0.35
0.94
0.28
0.34
0.94
2.73
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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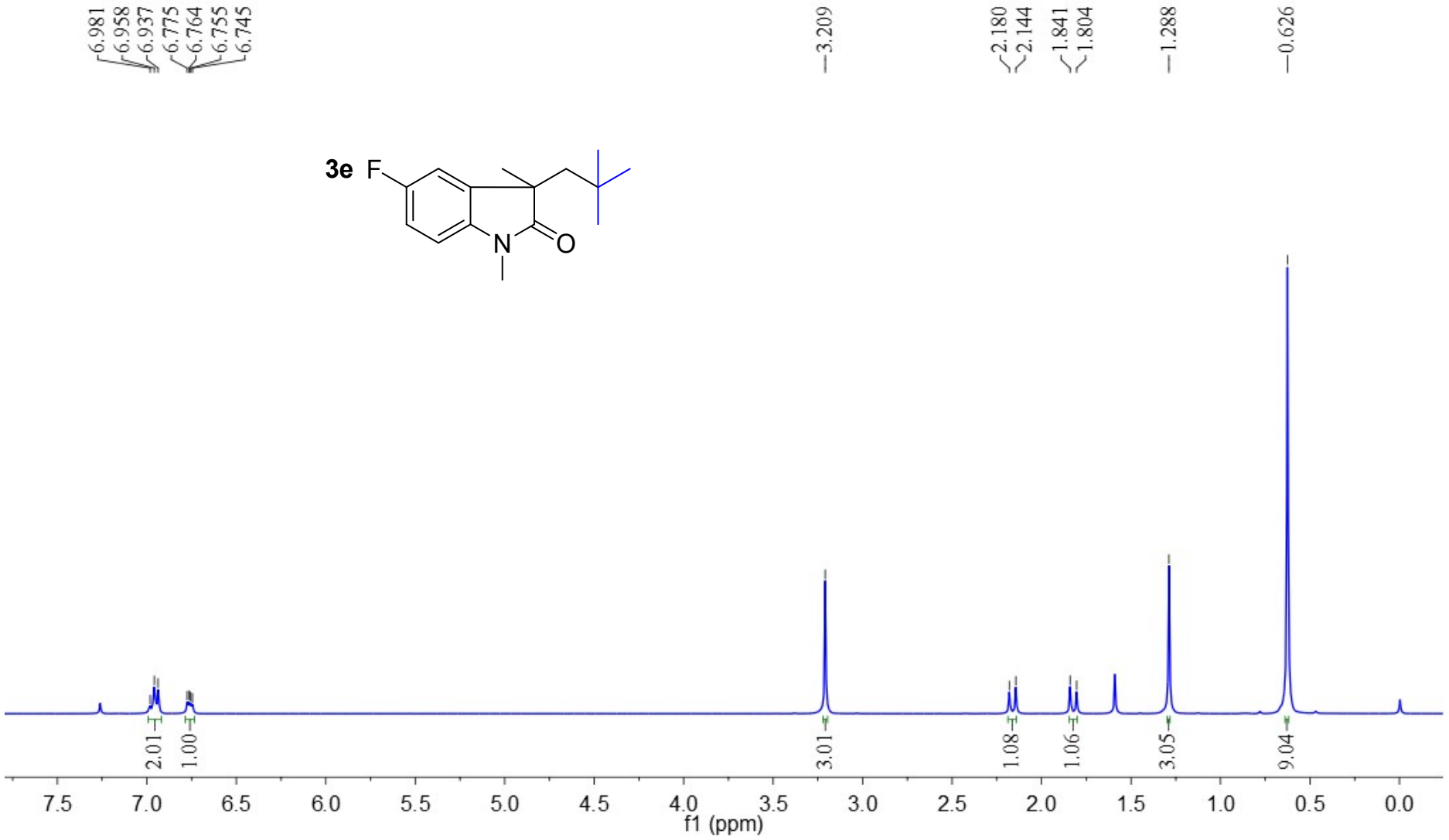
142.378
141.275
135.521
134.826
128.895
126.997
126.929
126.587
122.950
108.346
77.477
77.160
76.842
50.916
47.659
31.929
30.988
28.439
26.470
3d
Ph
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)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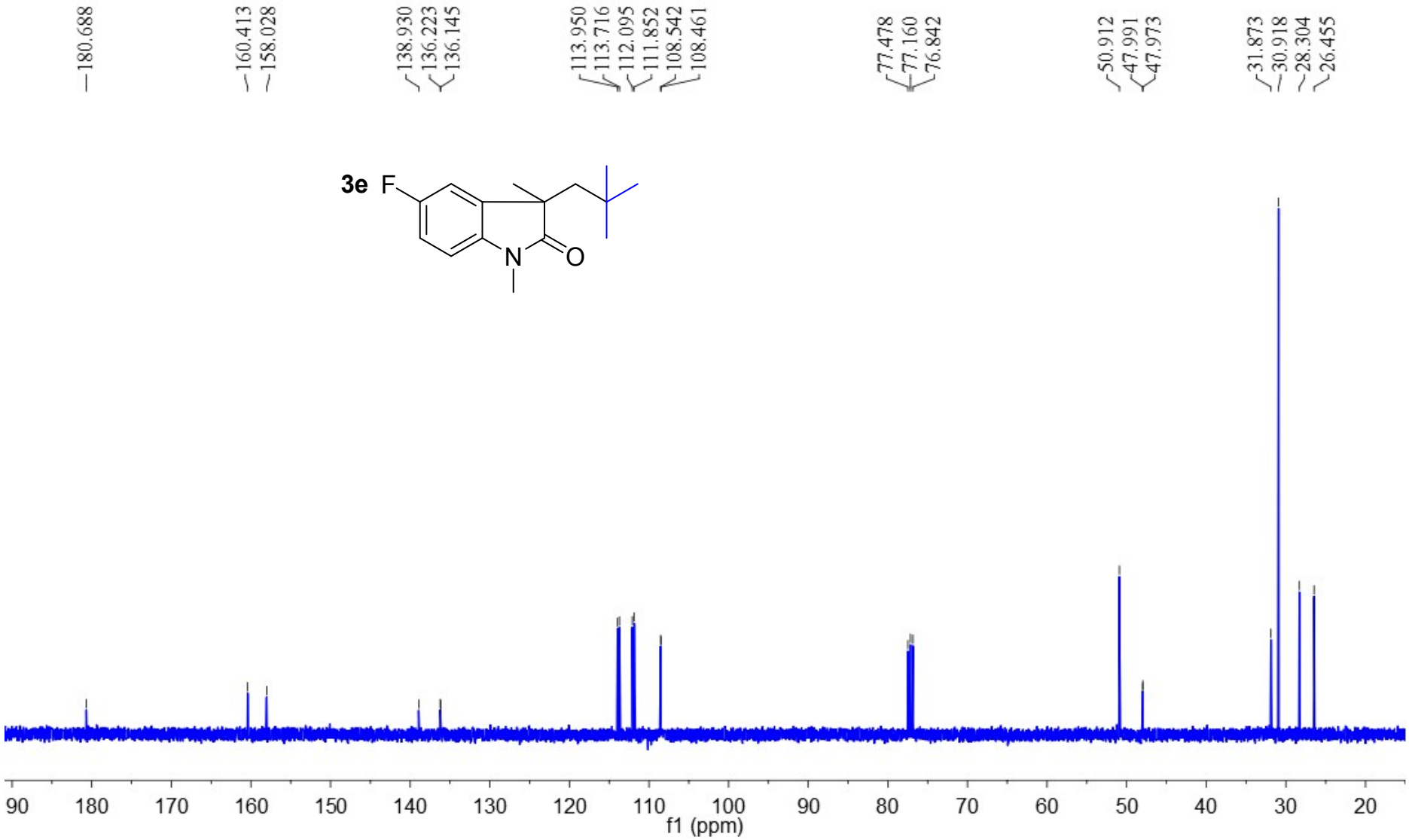
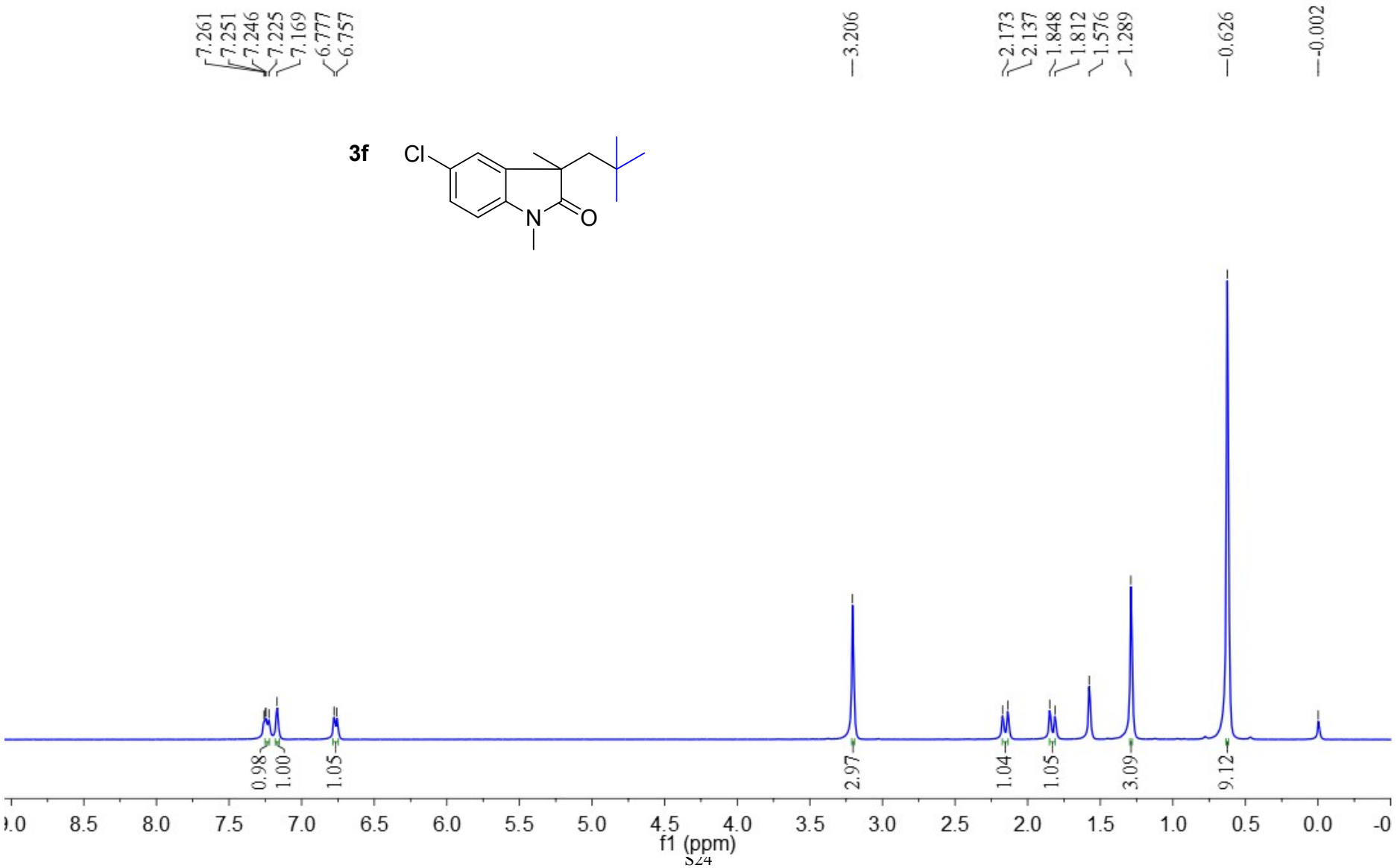
6.958
6.937
6.775
6.764
6.755
6.745
3.209
2.180
2.144
1.841
1.804
1.288
0.626
3e
F
N
O
2.01
1.00
3.01
1.08
1.06
3.05
9.04
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


180.688
160.413
158.028
138.930
136.223
136.145
113.950
113.716
112.095
111.852
108.542
108.461
77.478
77.160
76.842
50.912
47.991
47.973
31.873
30.918
28.304
26.455
3e
F
N
O
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)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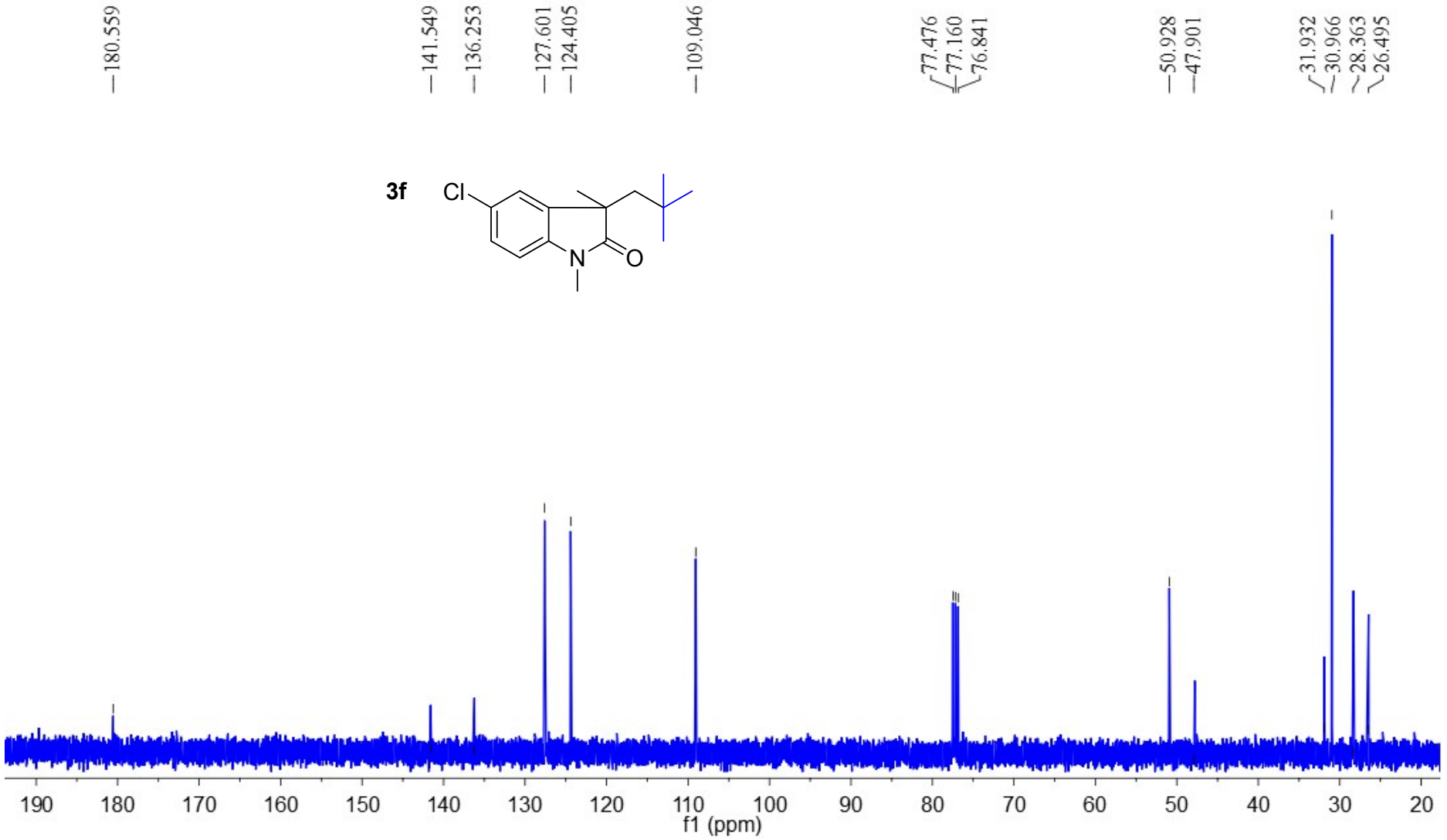
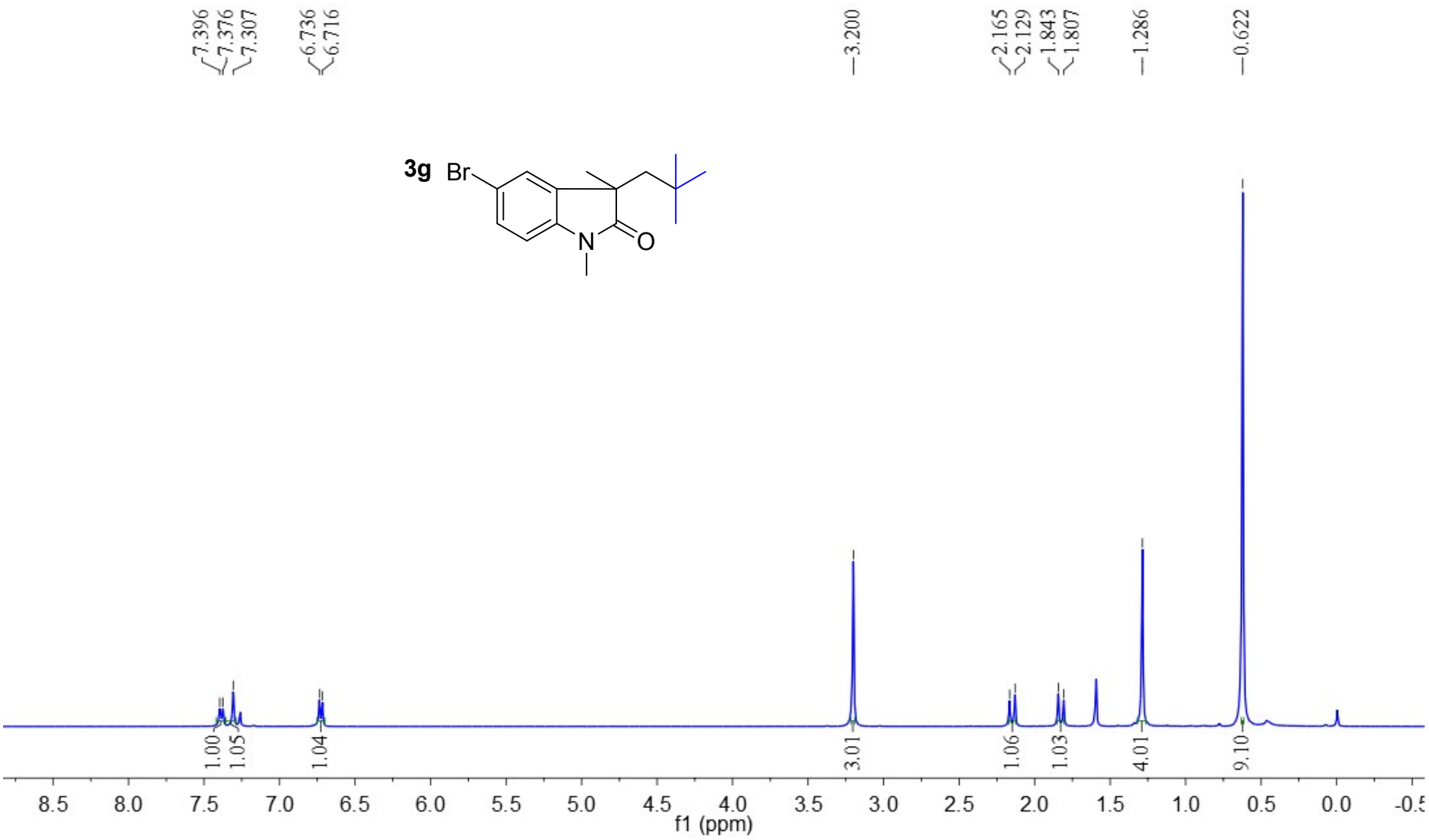
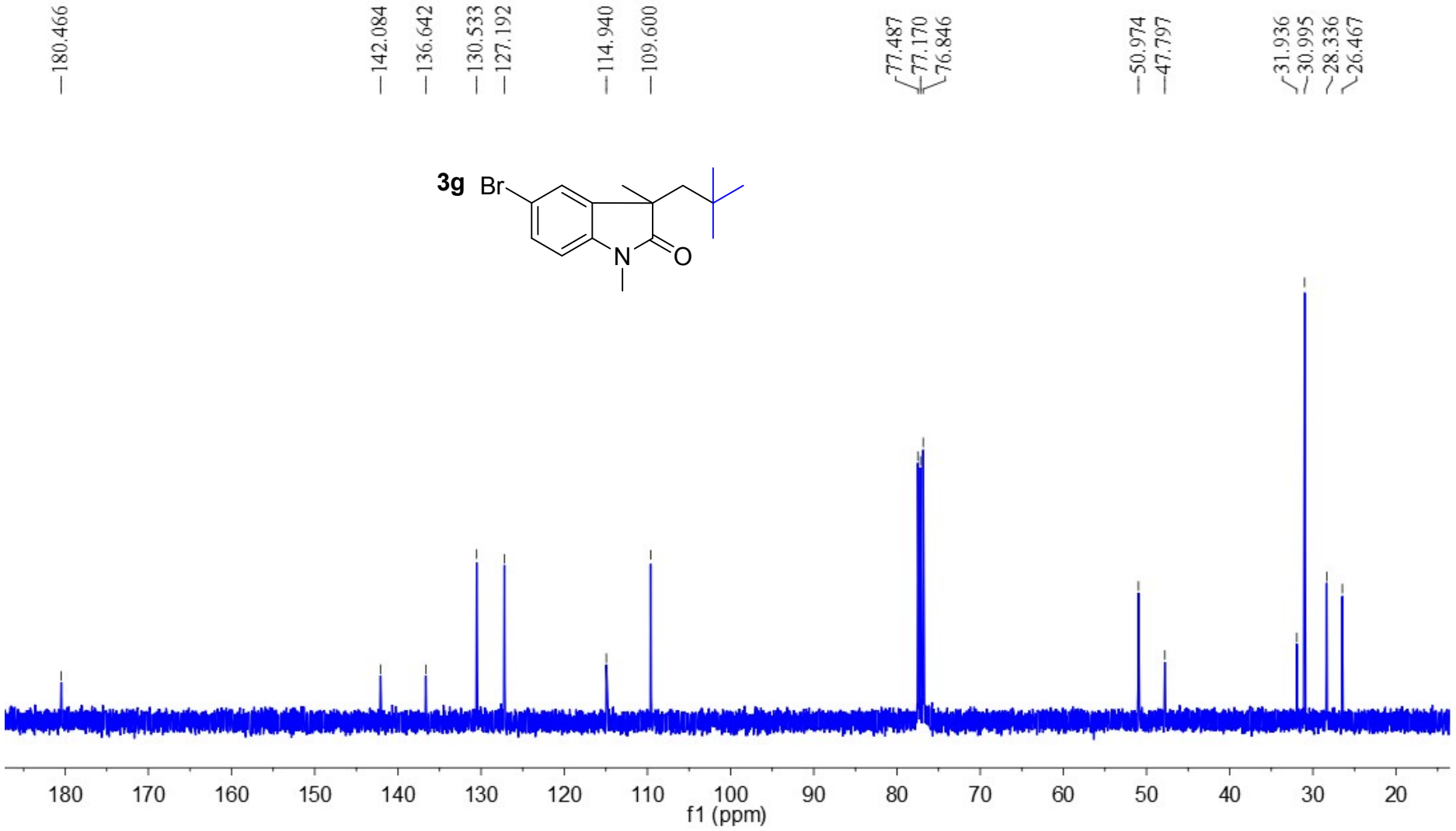
7.251
7.246
7.225
7.169
6.777
6.757
3.206
2.173
2.137
1.848
1.812
1.576
1.289
0.626
-0.002
3f
Cl
N
O
0.98
1.00
1.05
2.97
1.04
1.05
3.09
9.12
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


180.559
141.549
136.253
127.601
124.405
109.046
77.476
77.160
76.841
50.928
47.901
31.932
30.966
28.363
26.495
3f
Cl
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.396
7.376
7.307
6.736
6.716
3.200
2.165
2.129
1.843
1.807
1.286
0.622
3g
Br
N
O
1.00
1.05
1.04
3.01
1.06
1.03
4.01
9.10
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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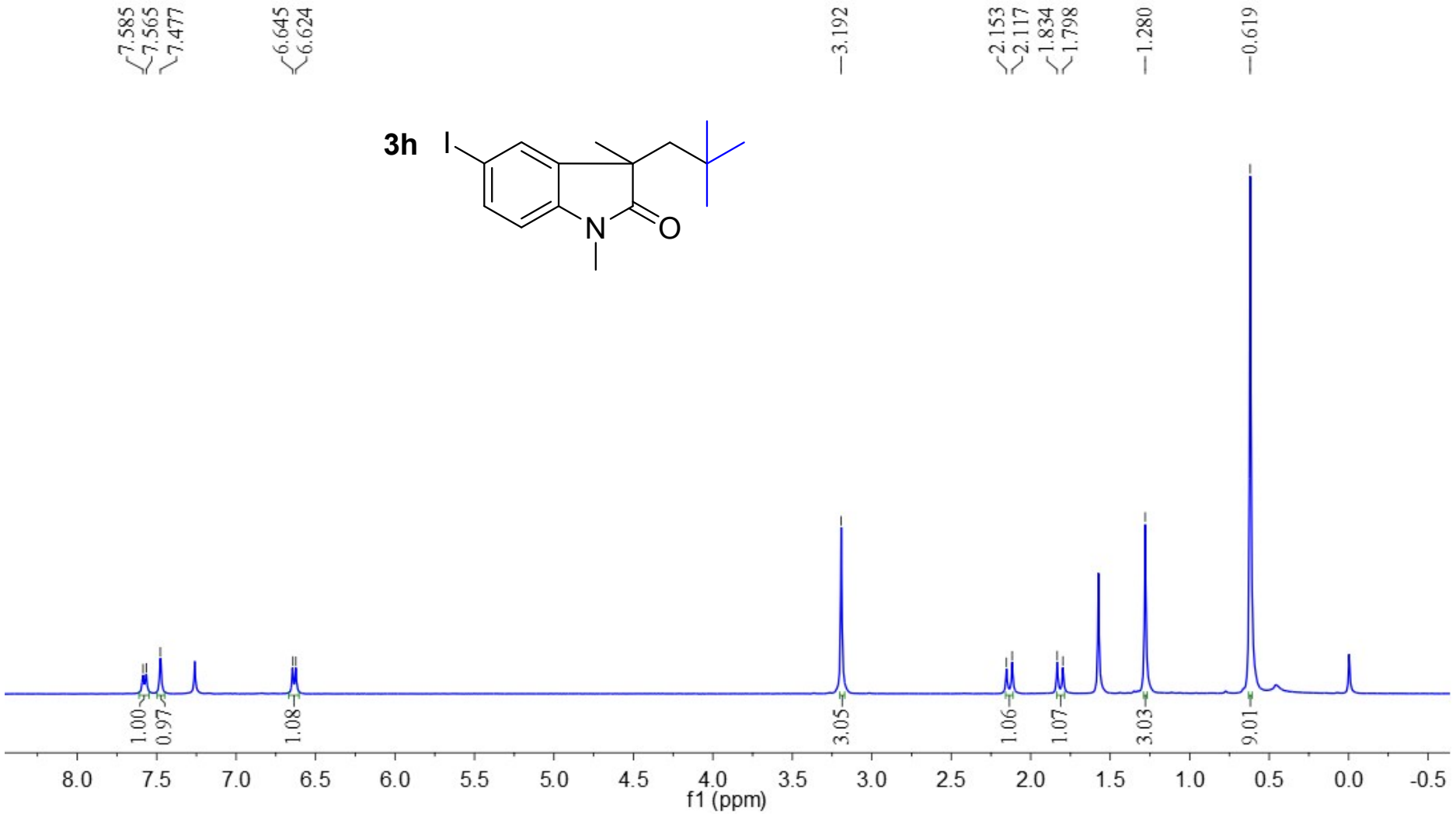
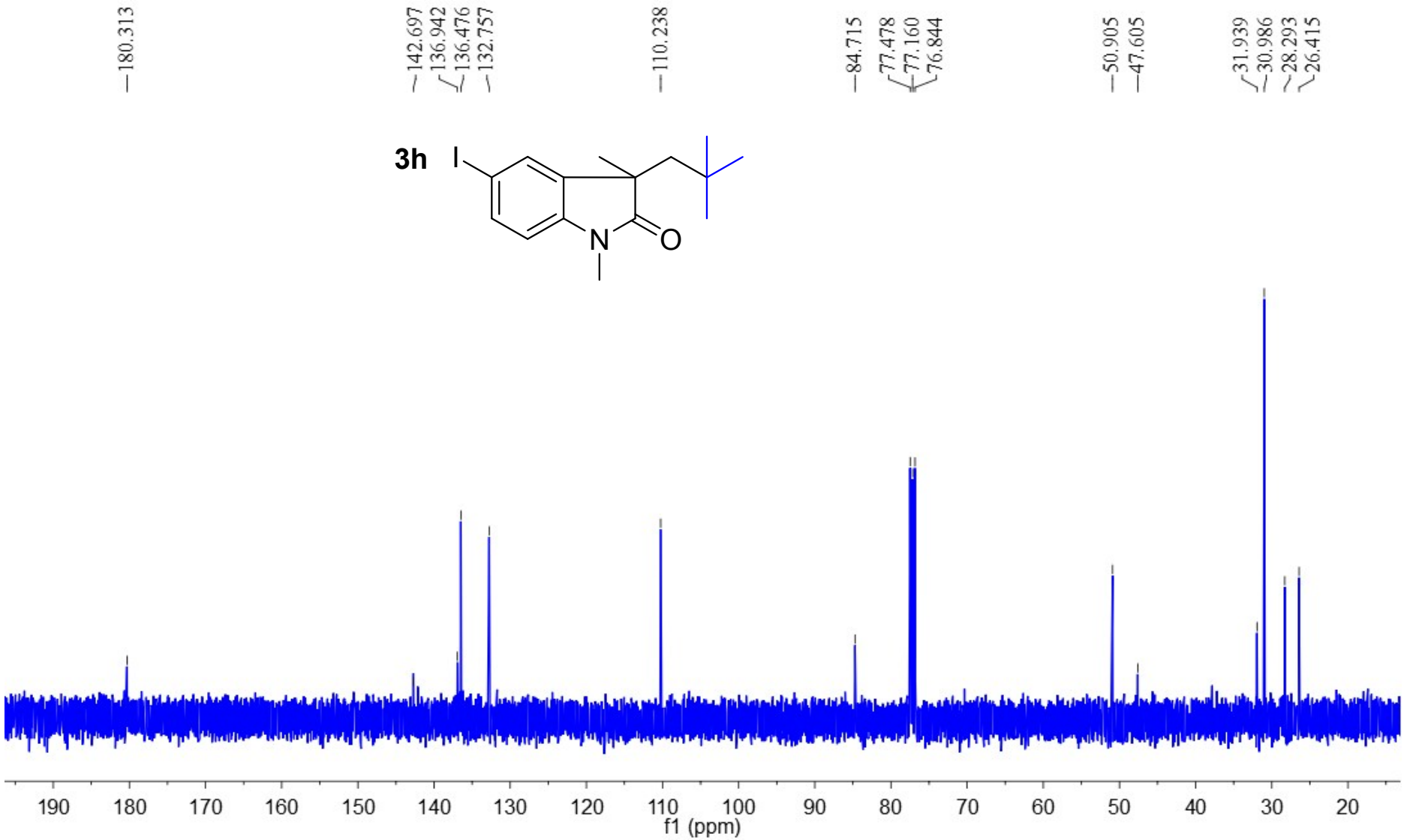
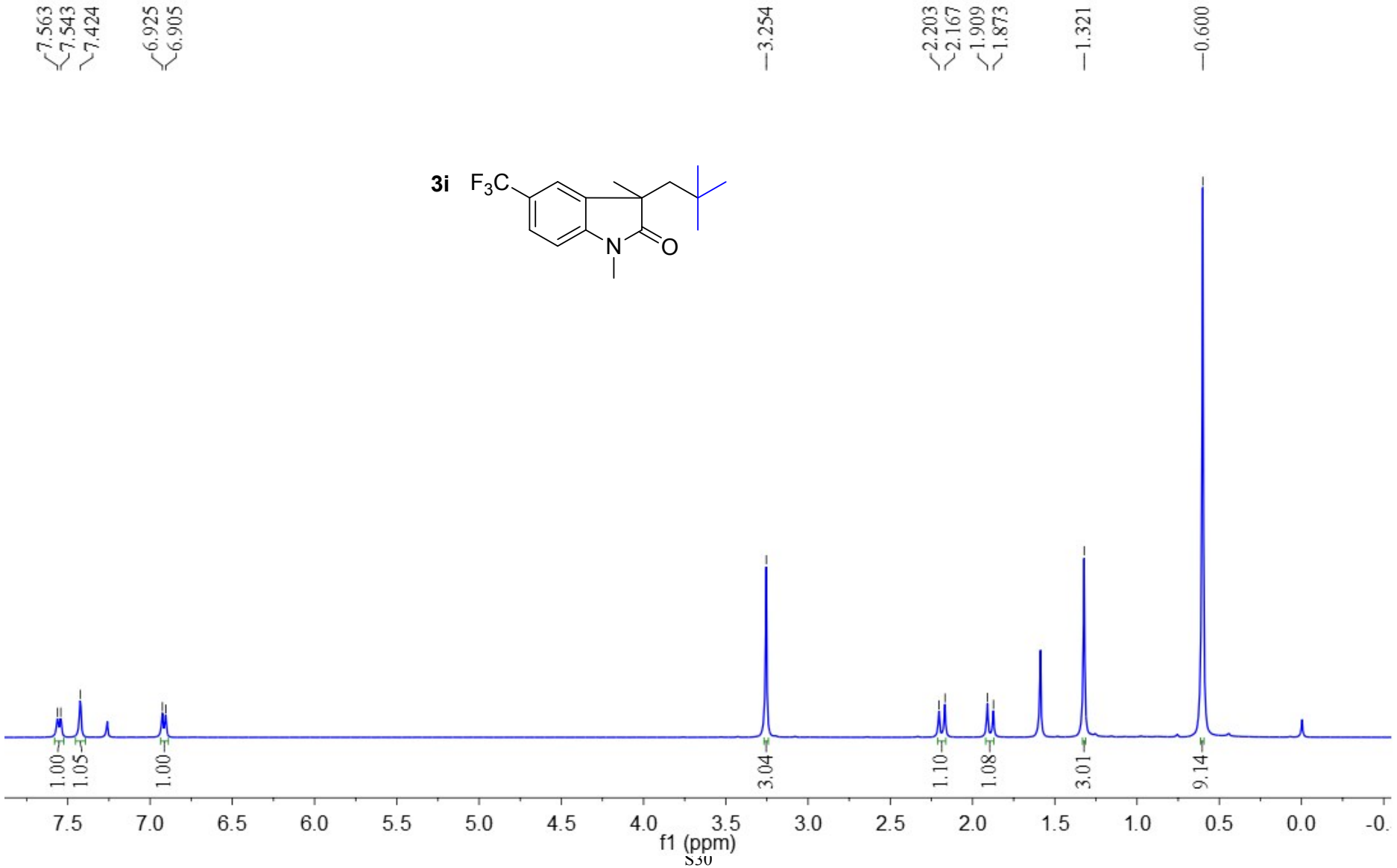
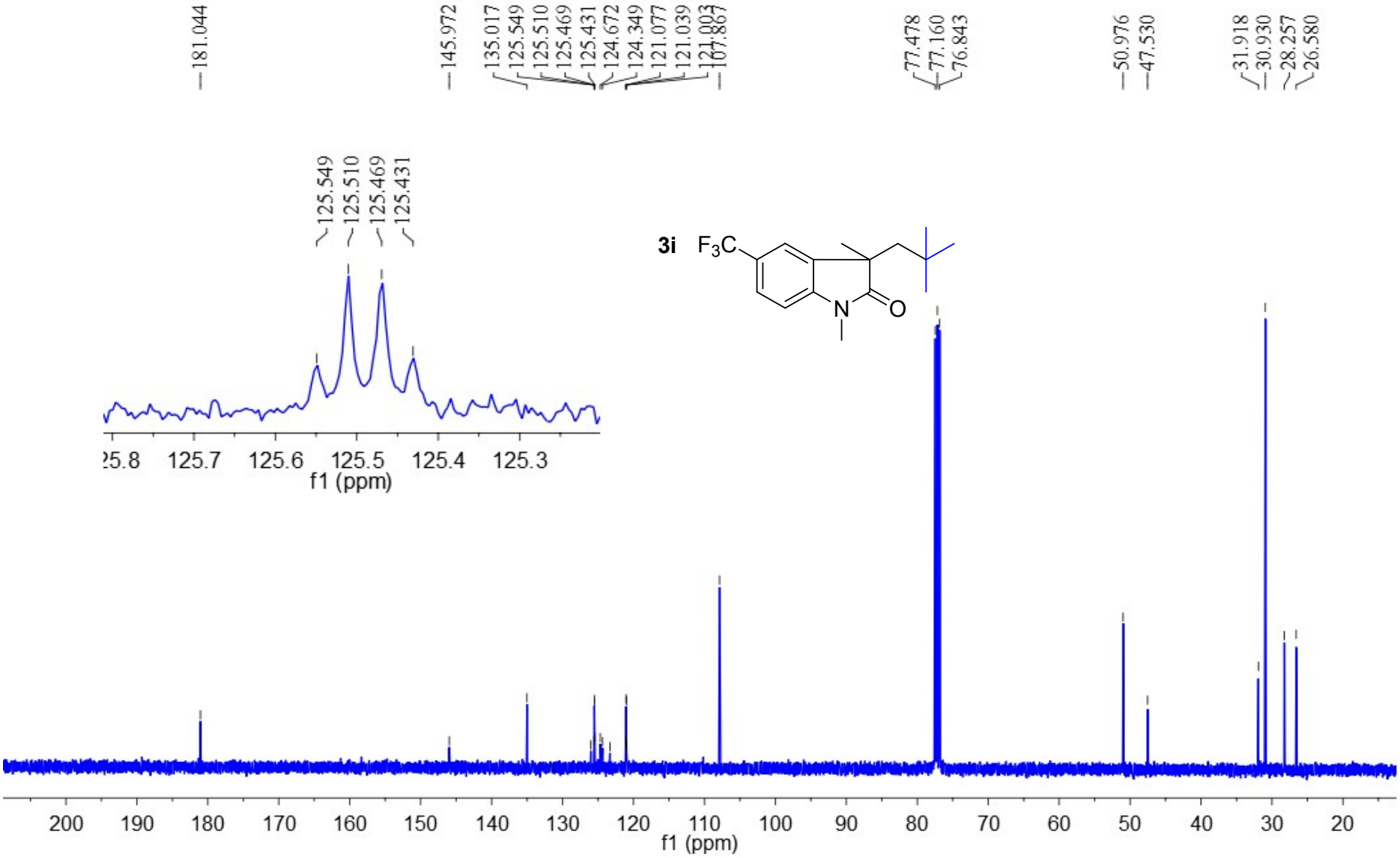
136.642
130.533
127.192
114.940
109.600
77.487
77.170
76.846
50.974
47.797
31.936
30.995
28.336
26.467
3g
Br
N
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.585
7.565
7.477
6.645
6.624
3.192
2.153
2.117
1.834
1.798
1.280
0.619
3h
I
N
O
1.00
0.97
1.08
3.05
1.06
1.07
3.03
9.01
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
-0.5
f1 (ppm)


180.313
142.697
136.942
136.476
132.757
110.238
84.715
77.478
77.160
76.844
50.905
47.605
31.939
30.986
28.293
26.415
3h
I
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.563
7.543
7.424
6.925
6.905
3.254
2.203
2.167
1.909
1.873
1.321
0.600
3i
F3C
N
O
1.00
1.05
1.00
3.04
1.10
1.08
3.01
9.14
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
-0.
f1 (ppm)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125.549
125.510
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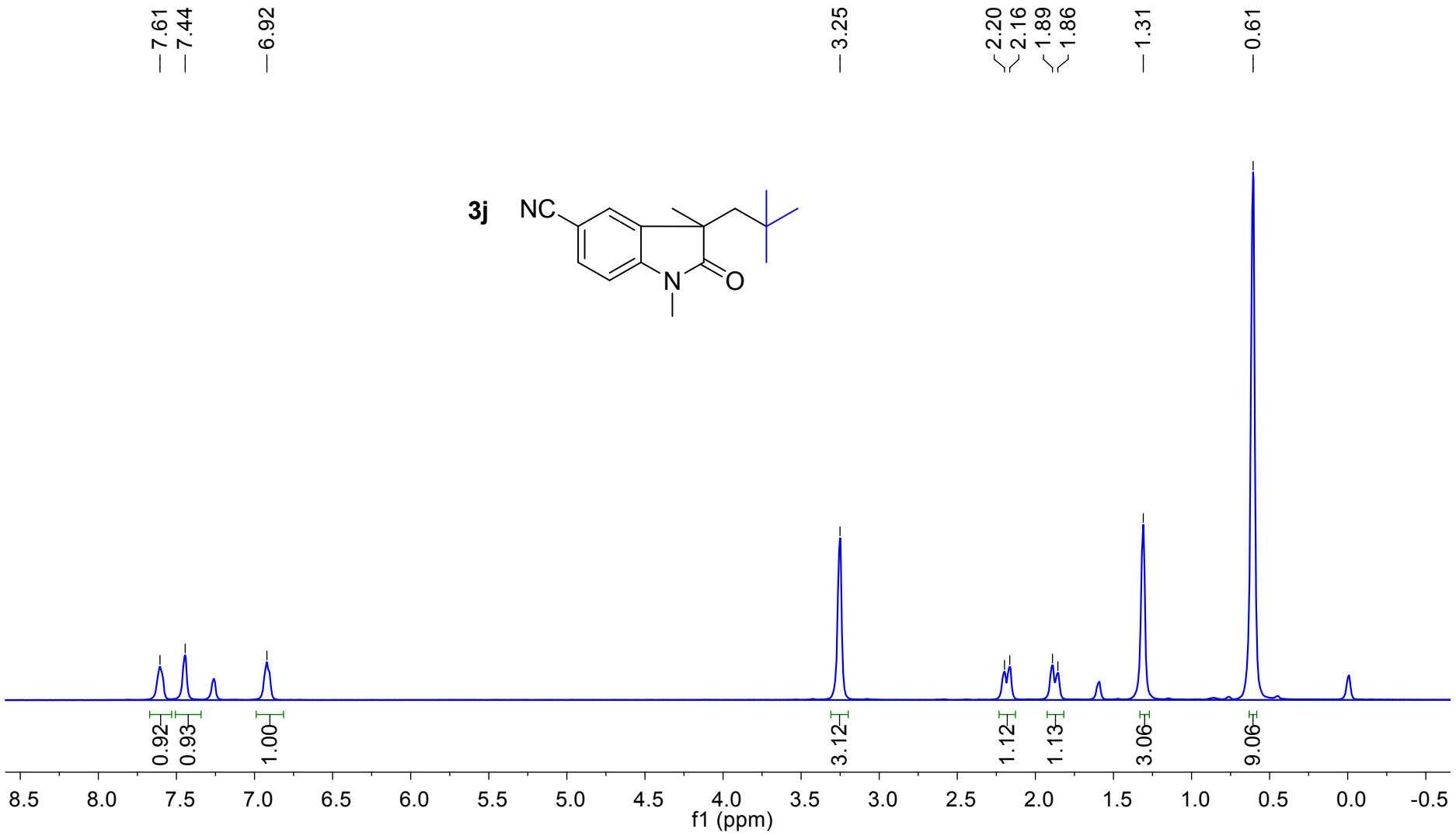
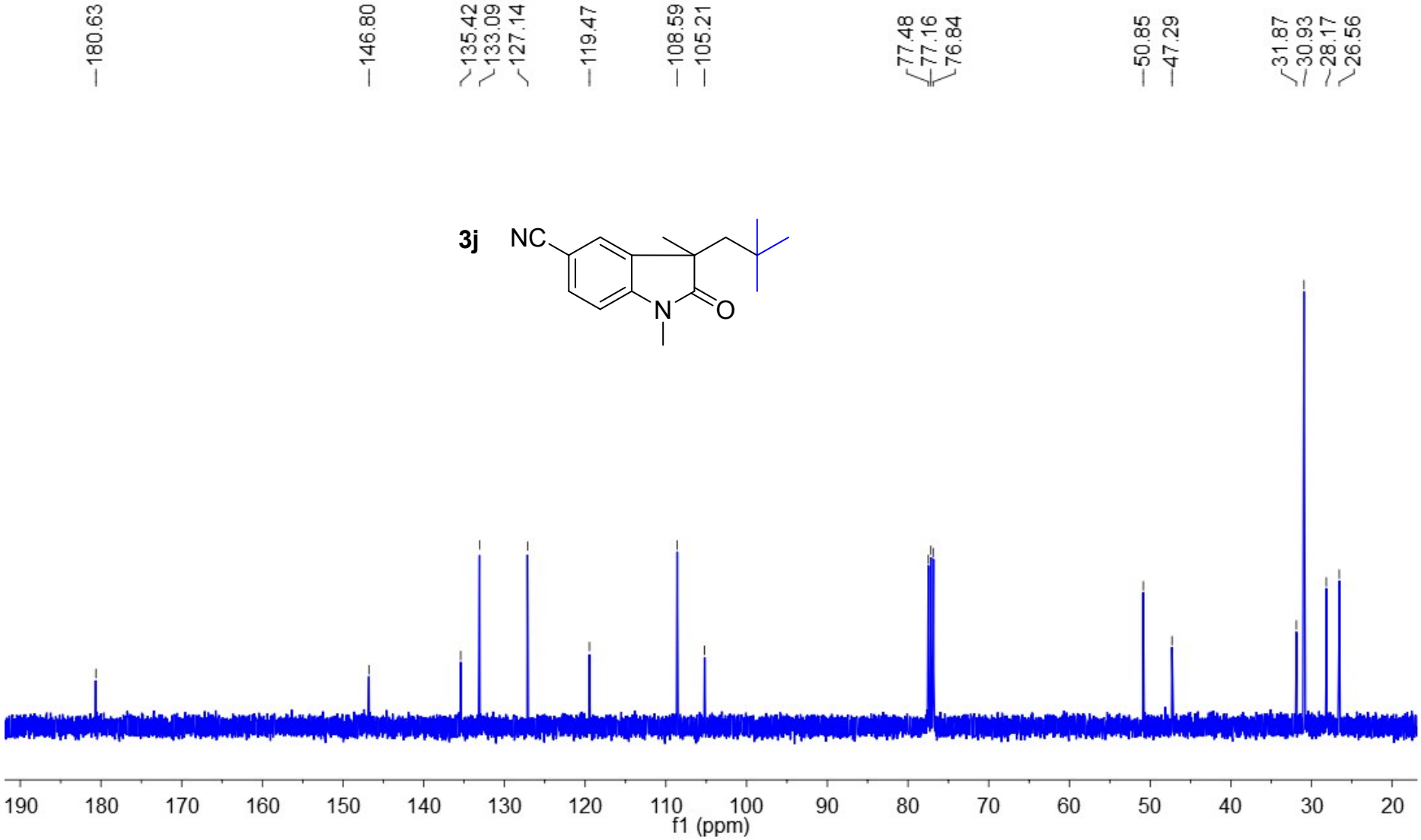
125.431
124.672
124.349
121.077
121.039
121.003
107.867
77.478
77.160
76.843
50.976
47.530
31.918
30.930
28.257
26.580
125.549
125.510
125.469
125.431
3i
F3C
N
O
25.8
125.7
125.6
125.5
125.4
125.3
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.61
7.44
6.92
3.25
2.20
2.16
1.89
1.86
1.31
0.61
3j
NC
N
O
0.92
0.93
1.00
3.12
1.12
1.13
3.06
9.06
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
-0.5
f1 (ppm)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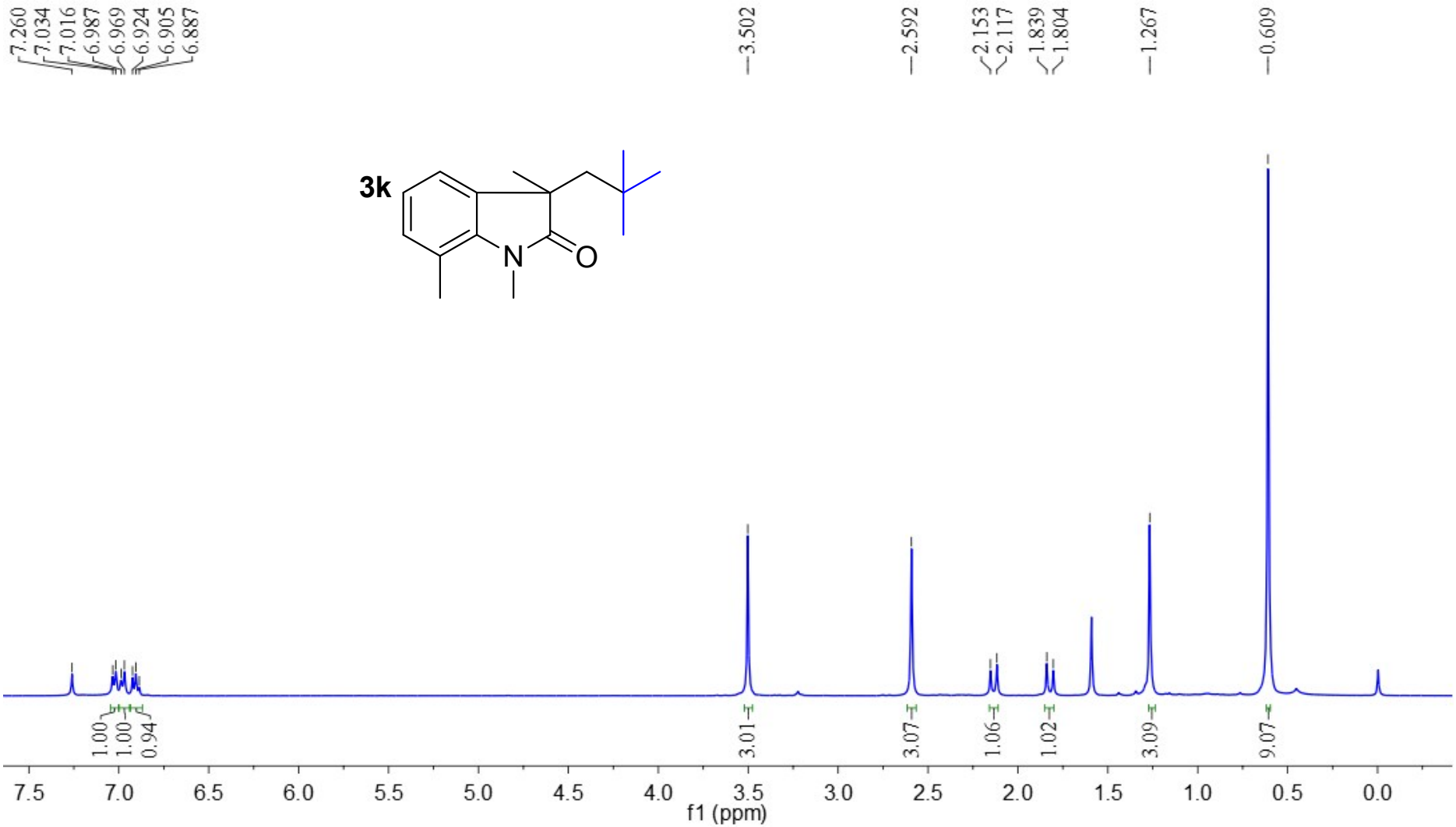
119.47
108.59
105.21
77.48
77.16
76.84
50.85
47.29
31.87
30.93
28.17
26.56
3j
NC
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)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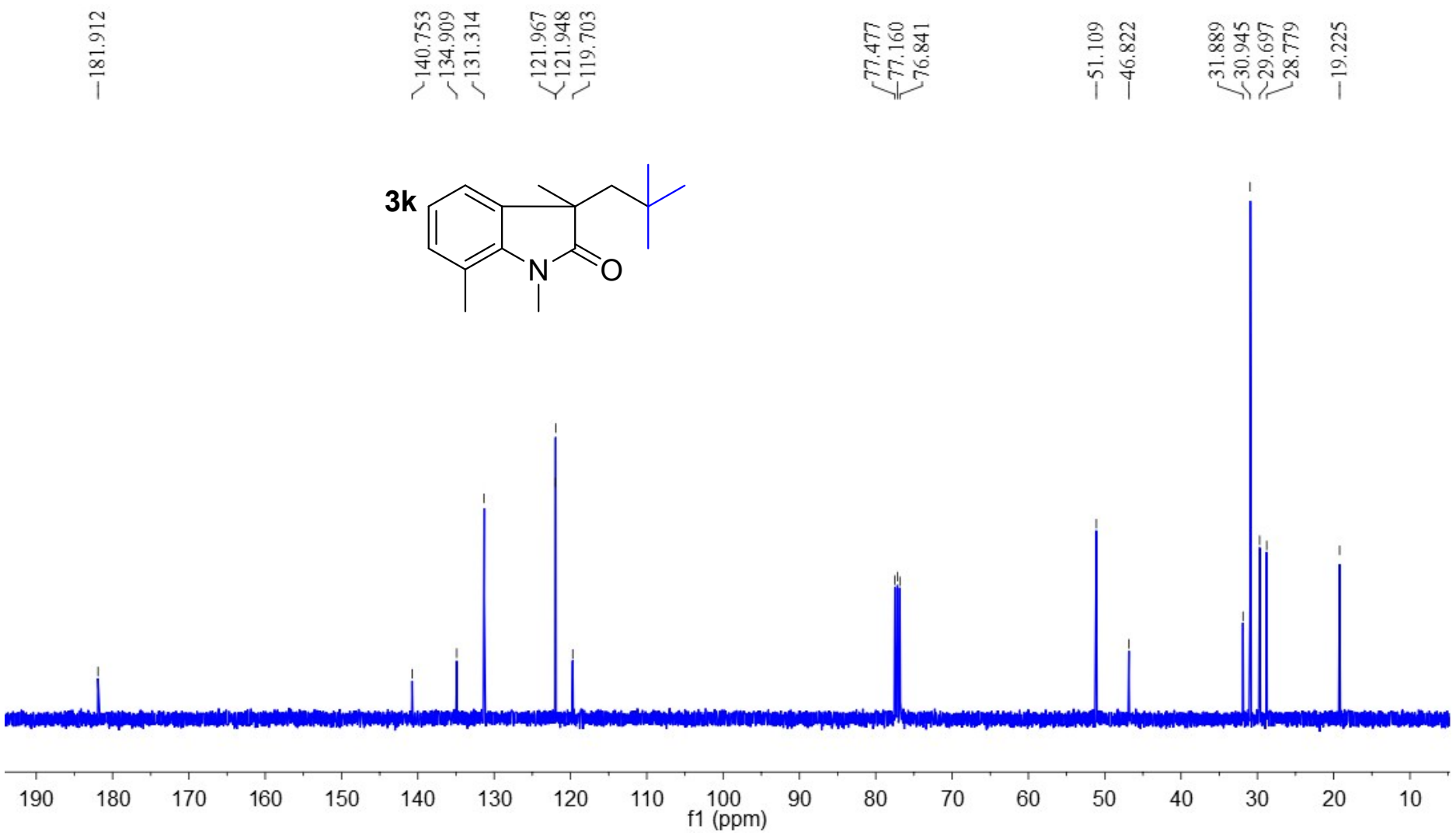
7.034
7.016
6.987
6.969
6.924
6.905
6.887
3.502
2.592
2.153
2.117
1.839
1.804
1.267
0.609
3k
N
O
1.00
1.00
0.94
3.01
3.07
1.06
1.02
3.09
9.07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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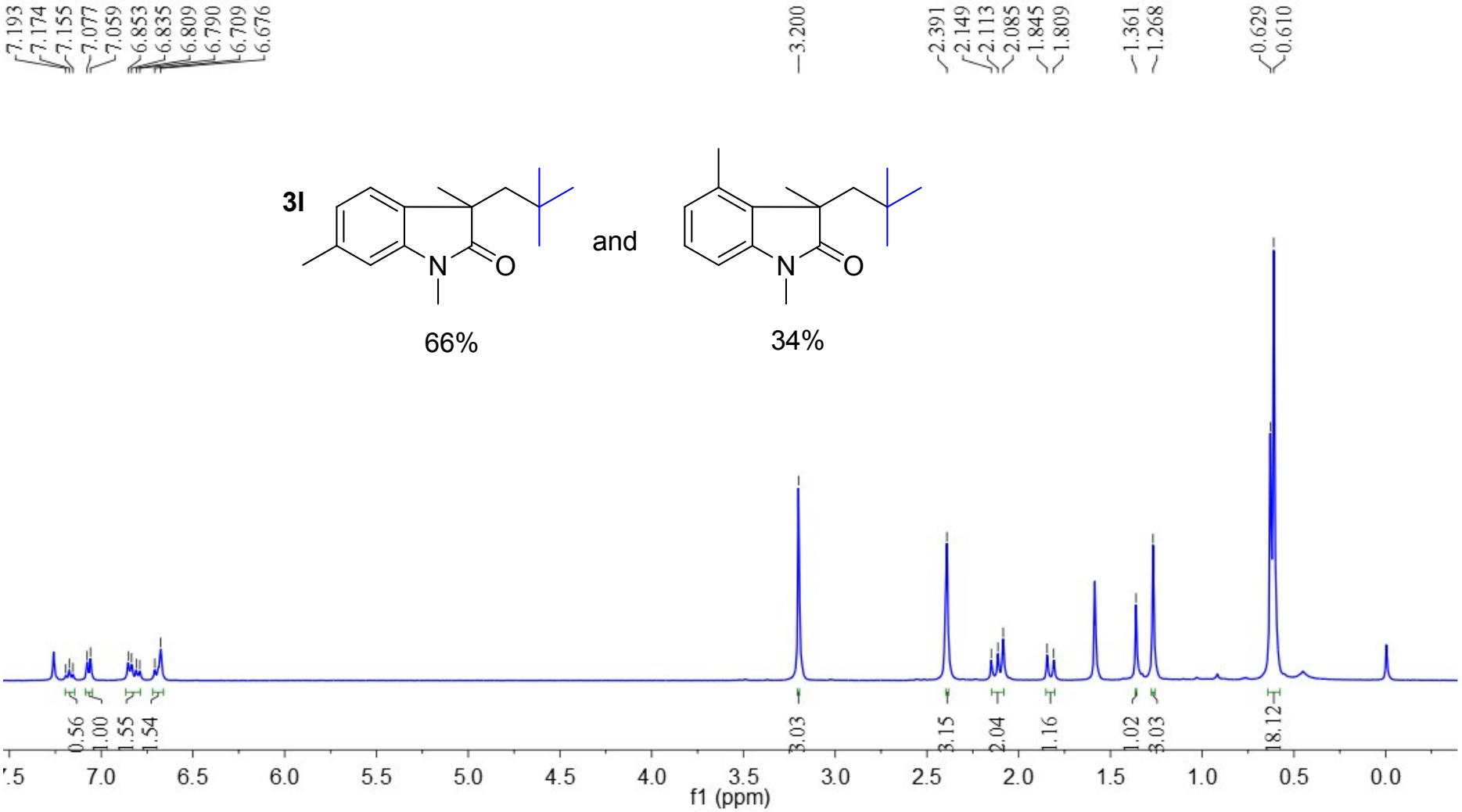
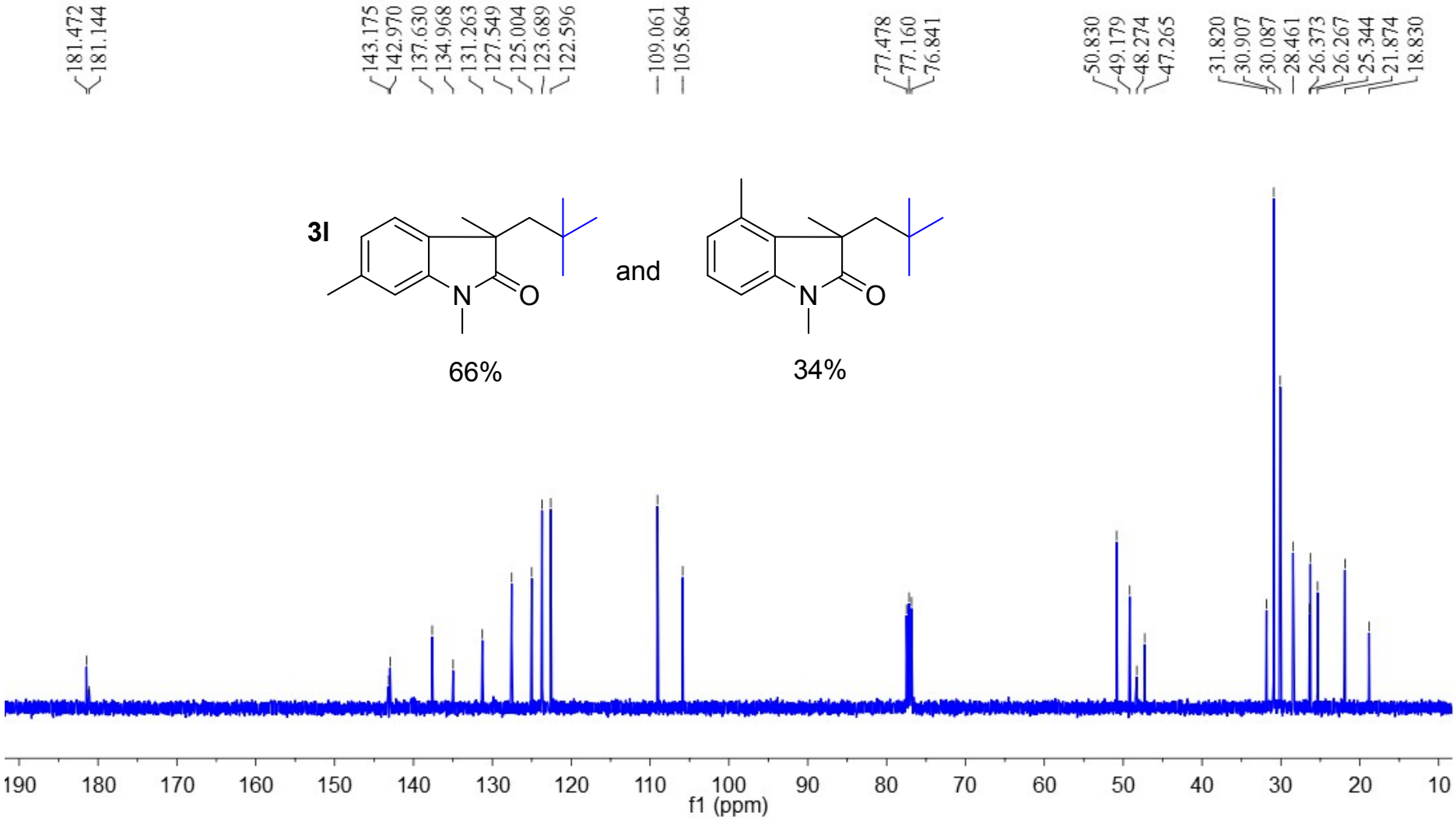
131.314
121.967
121.948
119.703
77.477
77.160
76.841
51.109
46.822
31.889
30.945
29.697
28.779
19.225
3k
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.193
7.174
7.155
7.077
7.059
6.853
6.835
6.809
6.790
6.709
6.676
3.200
2.391
2.149
2.113
2.085
1.845
1.809
1.361
1.268
0.629
0.610
3I
and
N
O
66%
34%
0.56
1.00
1.55
1.54
3.03
3.15
2.04
1.16
1.02
3.03
18.12
f1 (ppm)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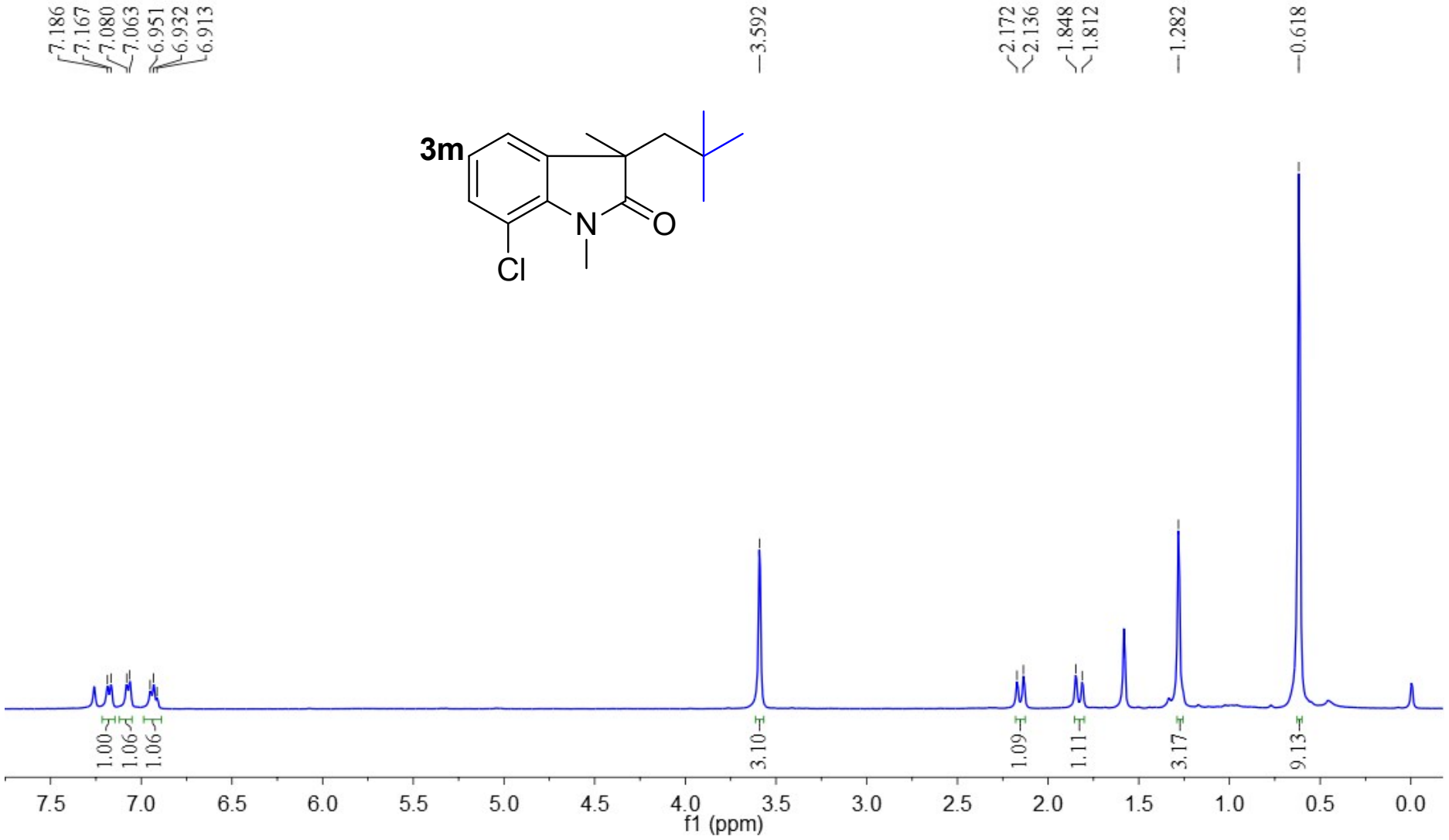
181.144
143.175
142.970
137.630
134.968
131.263
127.549
125.004
123.689
122.596
109.061
105.864
77.478
77.160
76.841
50.830
49.179
48.274
47.265
31.820
30.907
30.087
28.461
26.373
26.267
25.344
21.874
18.830
3l
N
O
and
N
O
66%
34%
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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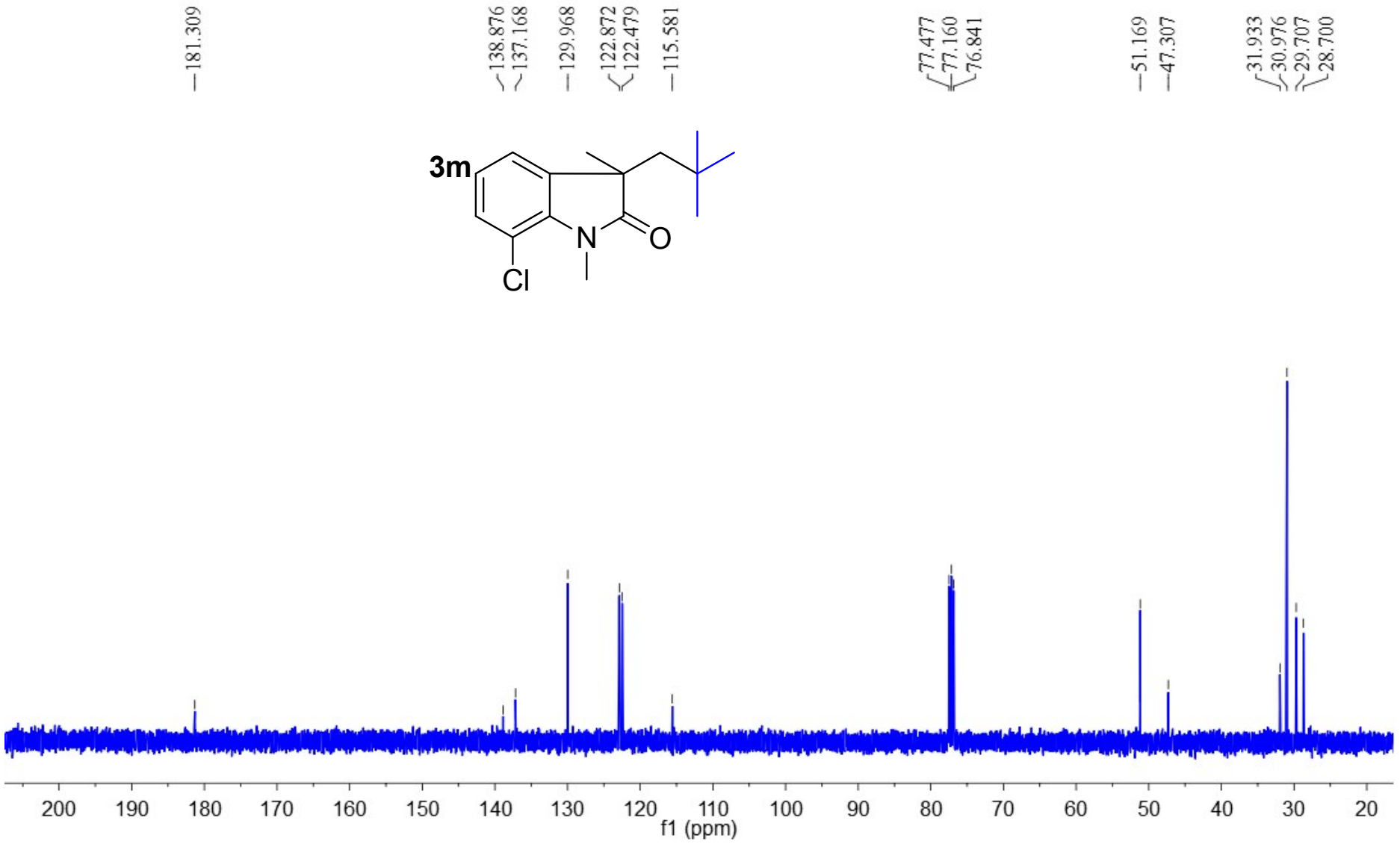
6.951
6.932
6.913
3.592
2.172
2.136
1.848
1.812
1.282
0.618
3m
N
O
Cl
1.00
1.06
1.06
3.10
1.09
1.11
3.17
9.13
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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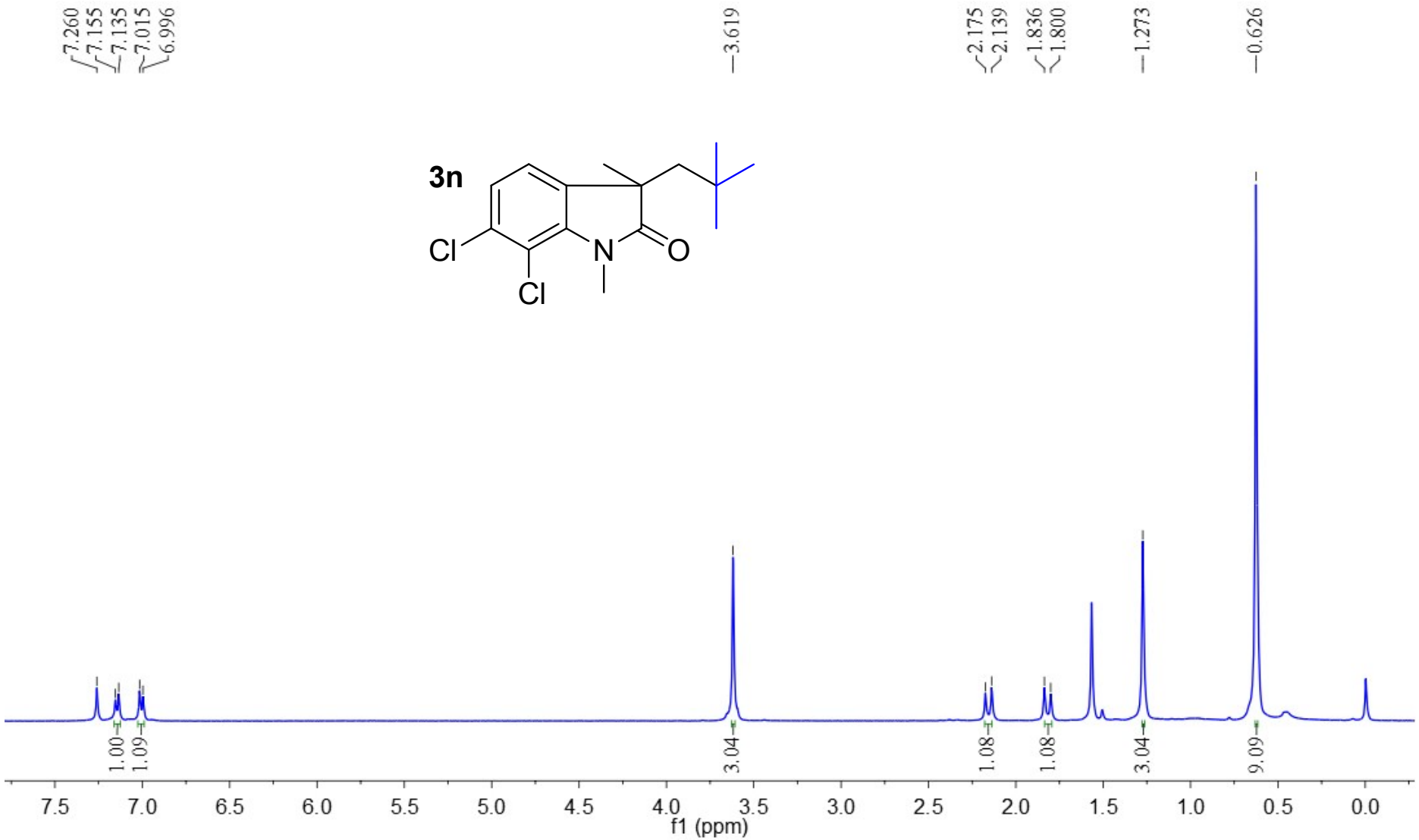
129.968
122.872
122.479
115.581
77.477
77.160
76.841
51.169
47.307
31.933
30.976
29.707
28.700
3m
N
O
Cl
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.260
7.155
7.135
7.015
6.996
3.619
2.175
2.139
1.836
1.800
1.273
0.626
3n
Cl
Cl
N
O
1.00
1.09
3.04
1.08
1.08
3.04
9.09
7.5
7.0
6.5
6.0
5.5
5.0
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3.5
3.0
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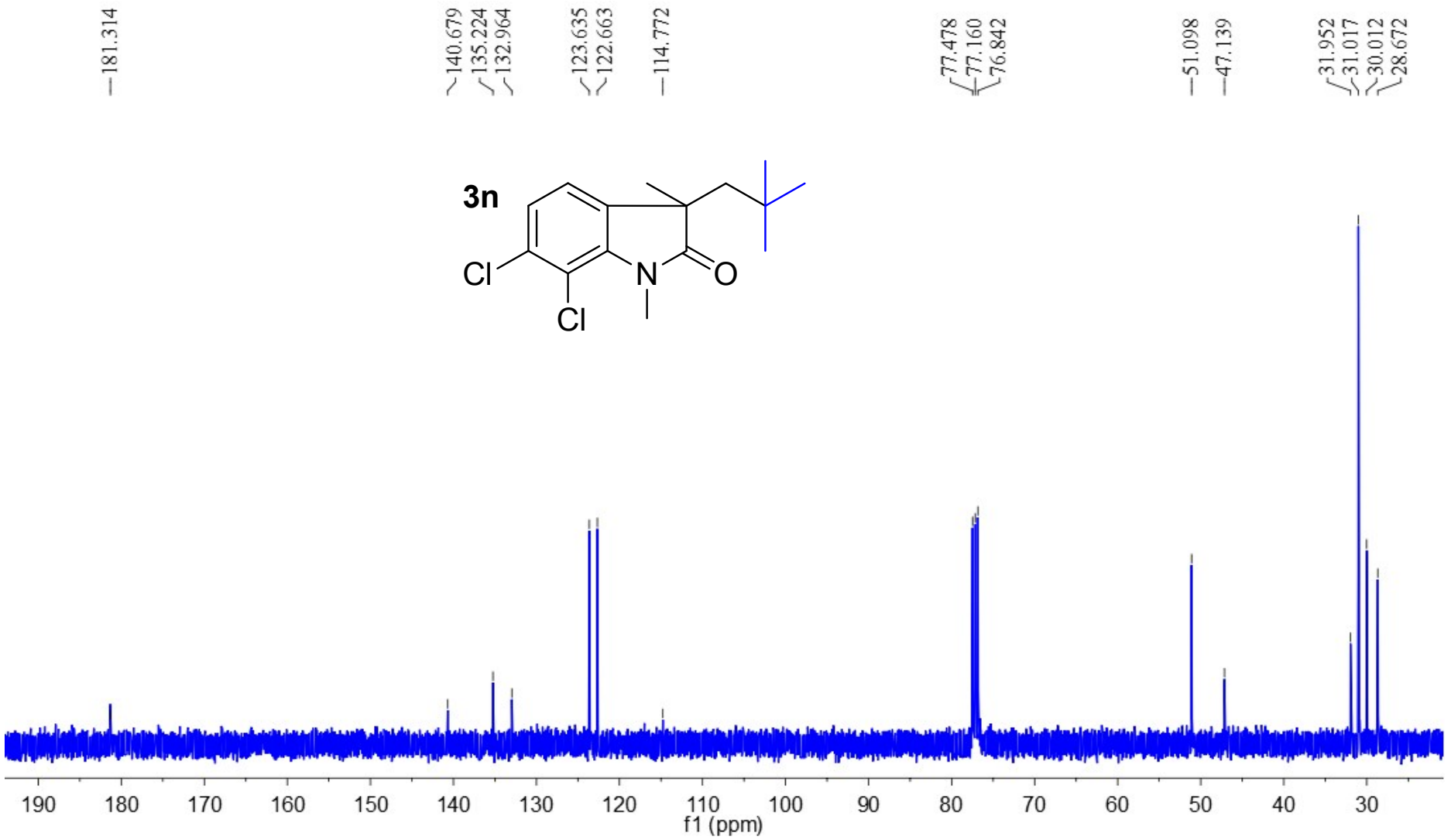
0.0
f1 (ppm)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122.663
114.772
77.478
77.160
76.842
51.098
47.139
31.952
31.017
30.012
28.672
3n
Cl
Cl
N
O
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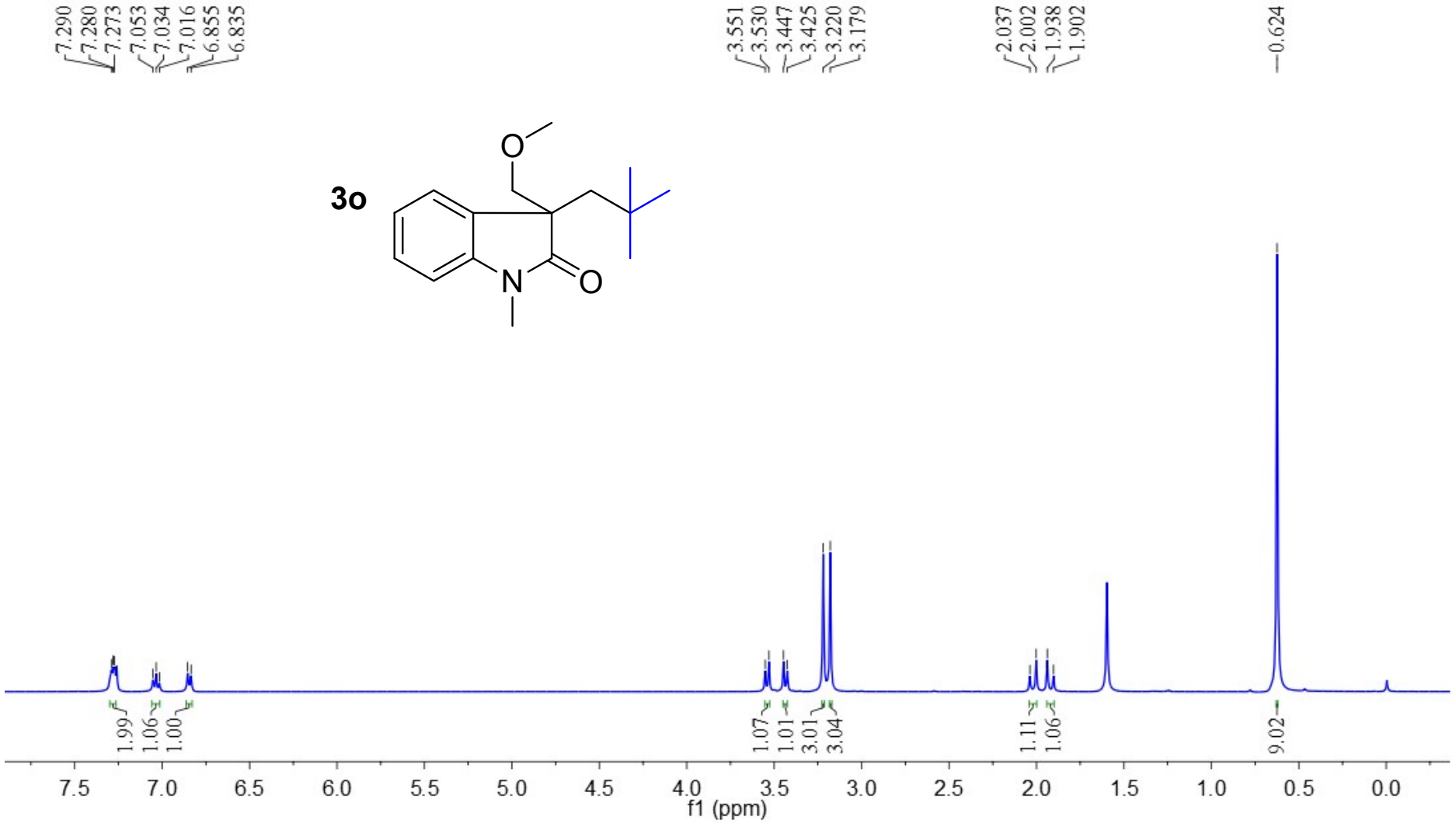
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)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7.016
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6.835
3.551
3.530
3.447
3.425
3.220
3.179
2.037
2.002
1.938
1.902
0.624
3o
O
N
O
1.99
1.06
1.00
1.07
1.01
3.01
3.04
1.11
1.06
9.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
3.0
2.5
2.0
1.5
1.0
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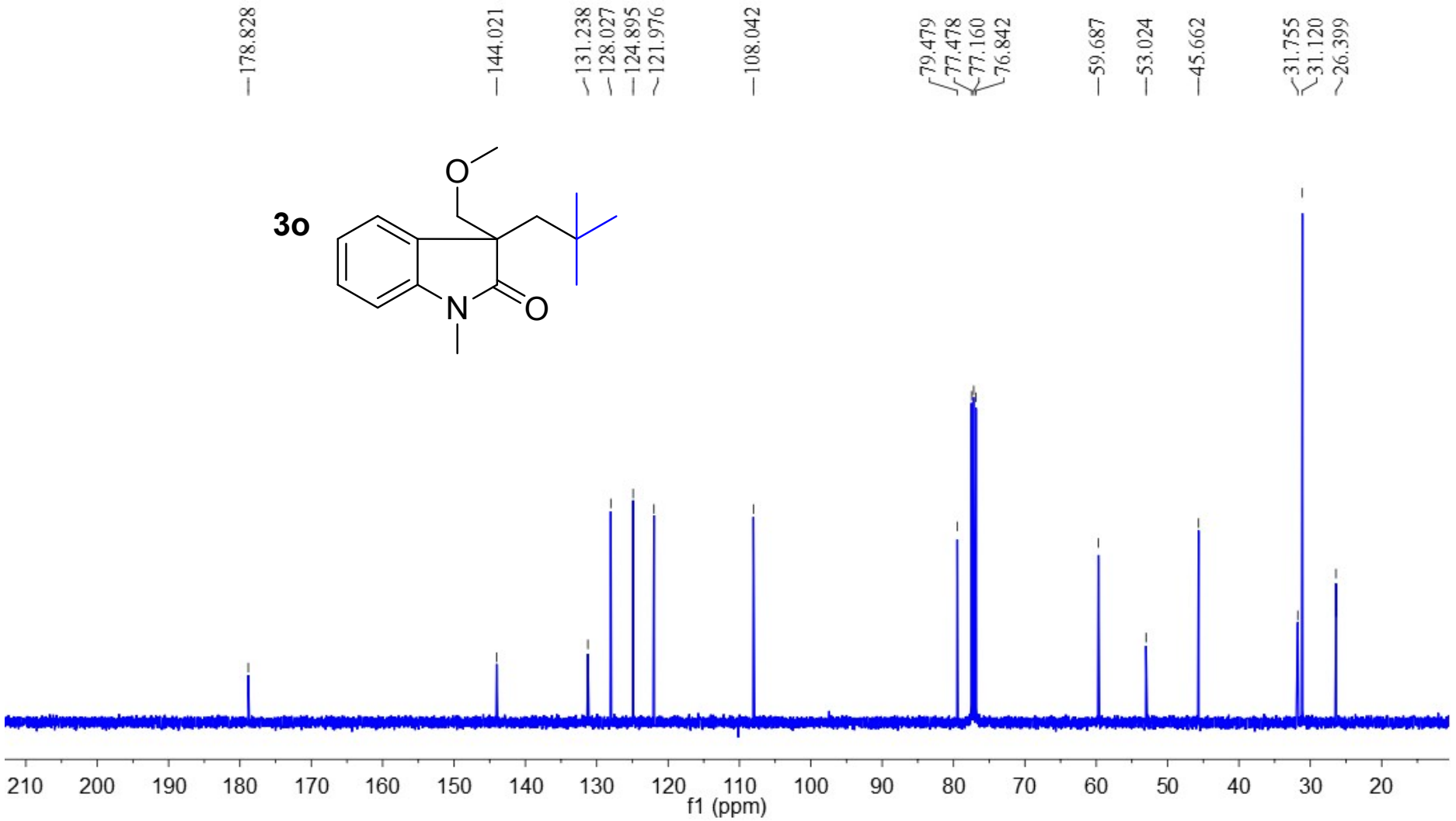
0.0
f1 (ppm)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108.042
79.479
77.478
77.160
76.842
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53.024
45.662
31.755
31.120
26.399
3o
O
N
O
210
200
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150
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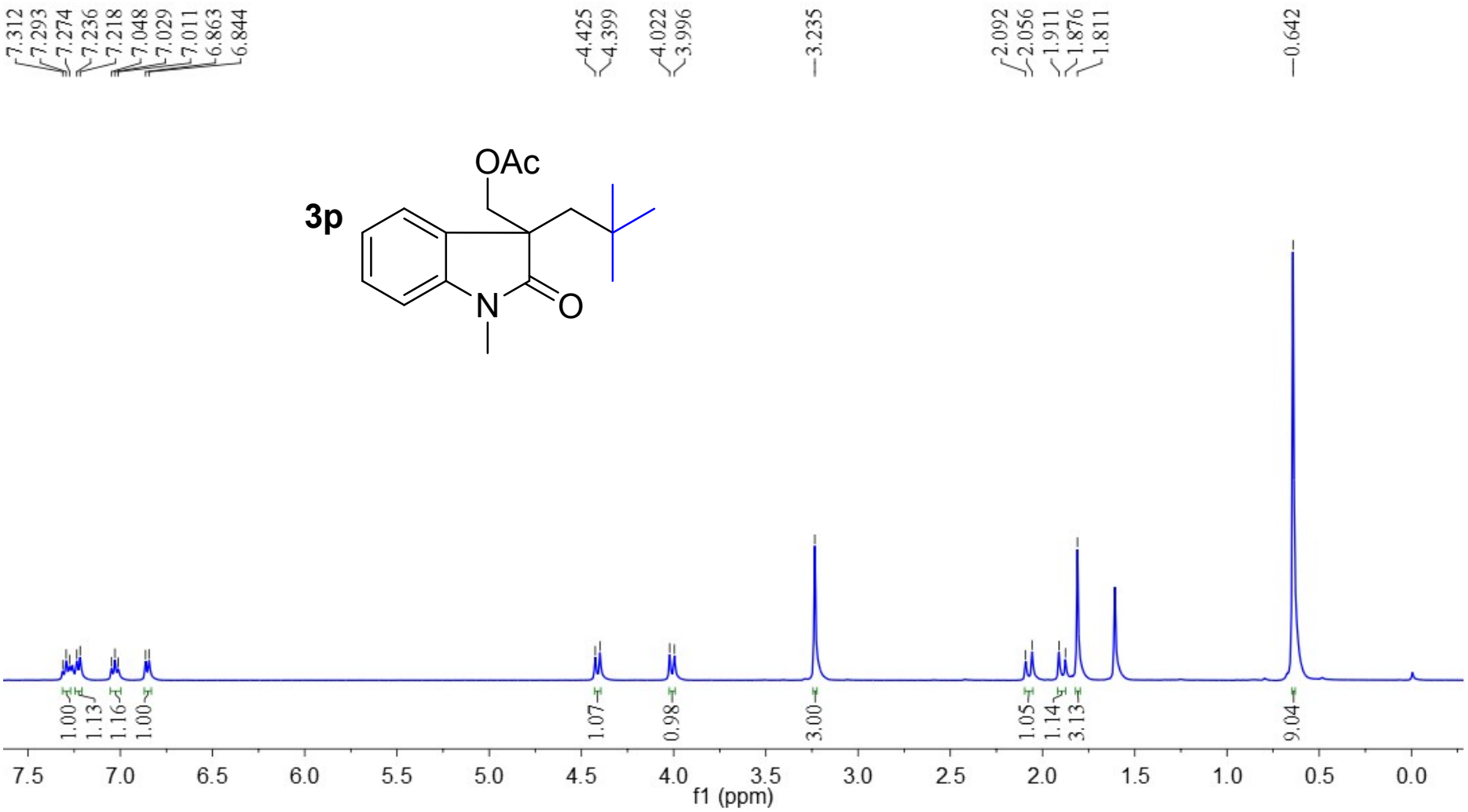
f1 (ppm)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7.236
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7.029
7.011
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6.844
4.425
4.399
4.022
3.996
3.235
2.092
2.056
1.911
1.876
1.811
0.642
3p
OAc
N
O
1.00
1.13
1.16
1.00
1.07
0.98
3.00
1.05
1.14
3.13
9.04
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
3.5
3.0
2.5
2.0
1.5
1.0
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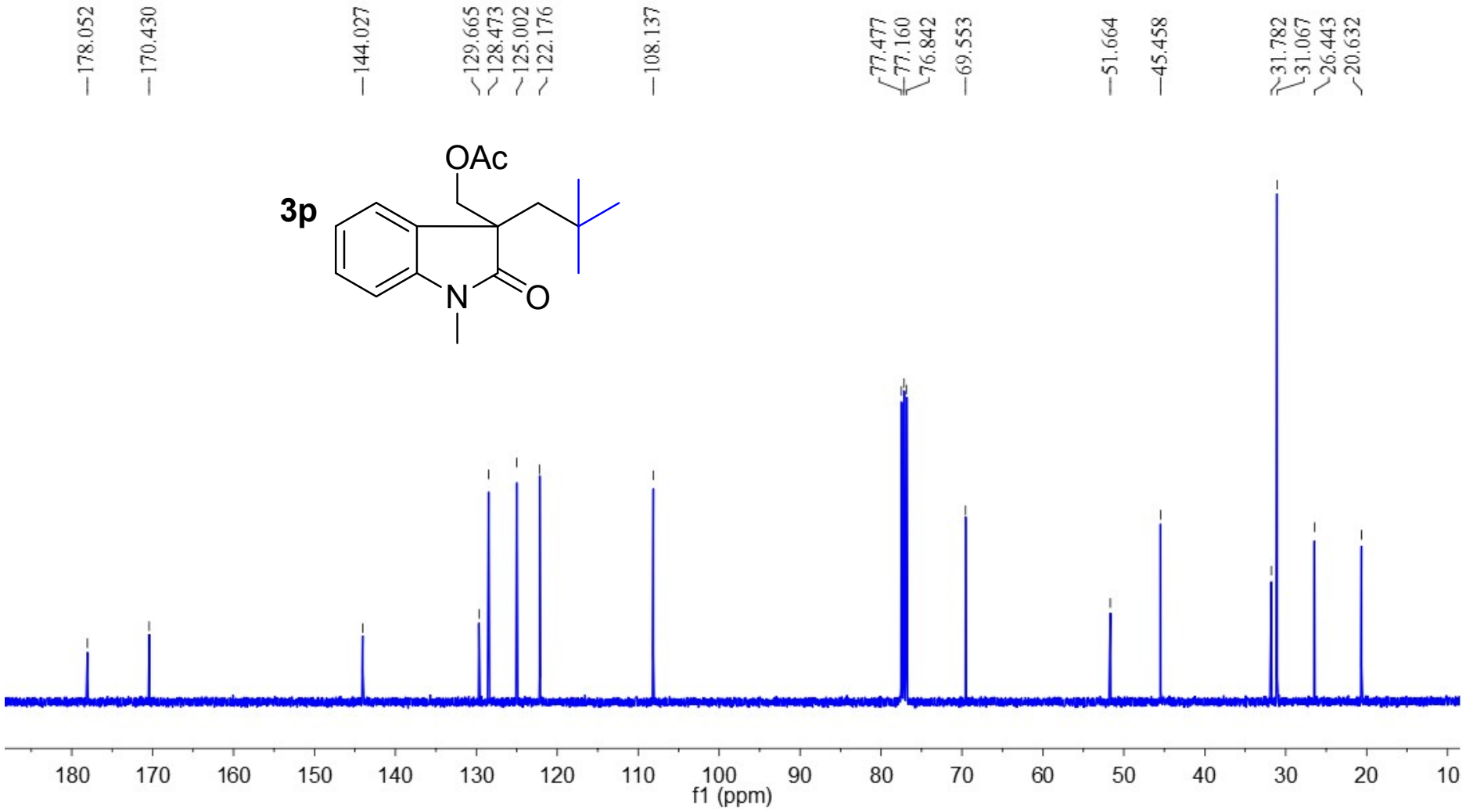
0.0
f1 (ppm)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108.137
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77.160
76.842
69.553
51.664
45.458
31.782
31.067
26.443
20.632
3p
OAc
N
O
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150
140
130
120
110
100
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80
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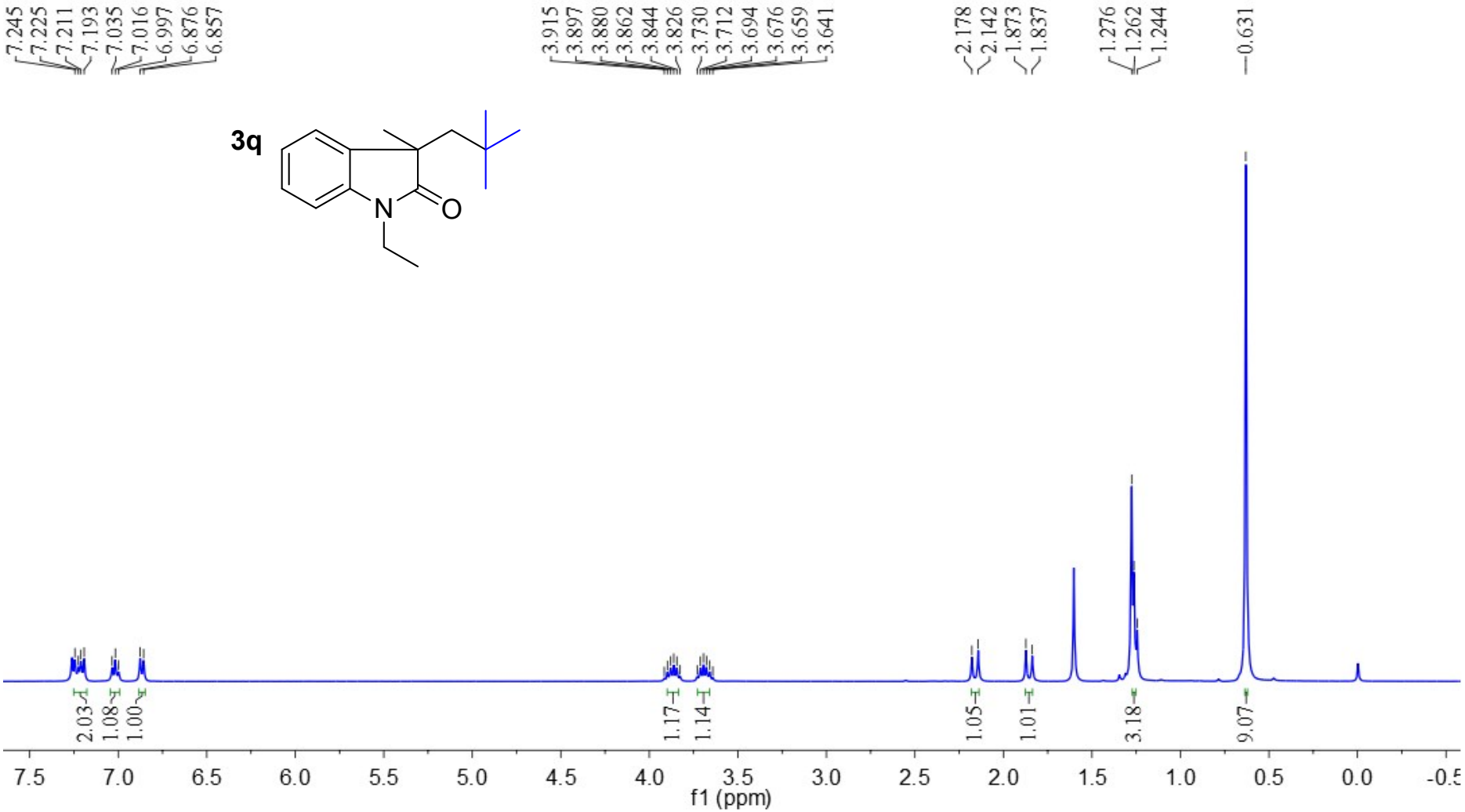
60
50
40
30
20
10
f1 (ppm)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7.193
7.035
7.016
6.997
6.876
6.857
3.915
3.897
3.880
3.862
3.844
3.826
3.730
3.712
3.694
3.676
3.659
3.641
2.178
2.142
1.873
1.837
1.276
1.262
1.244
0.631
3q
N
O
2.03
1.08
1.00
1.17
1.14
1.05
1.01
3.18
9.07
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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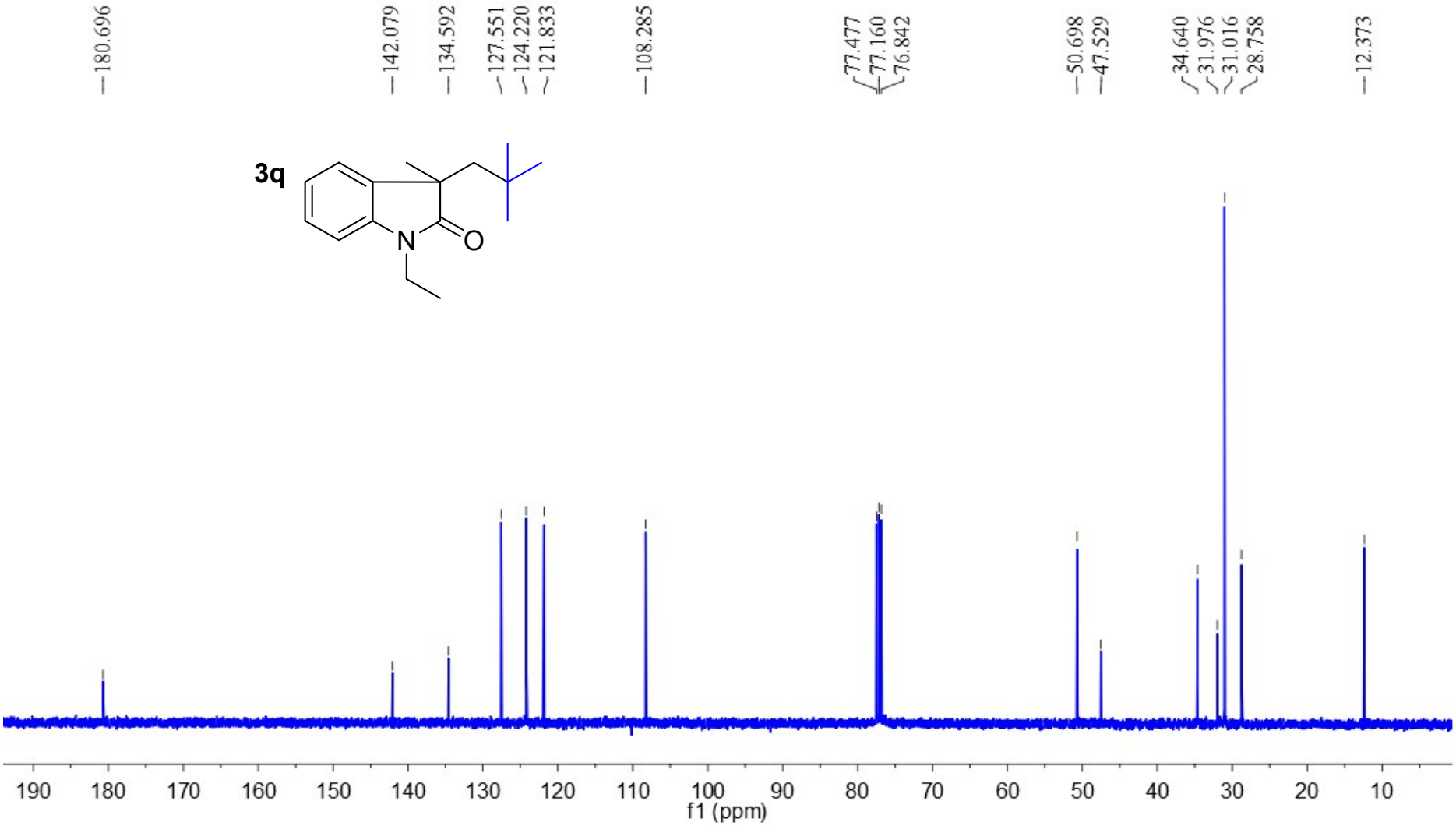
0.0
f1 (ppm)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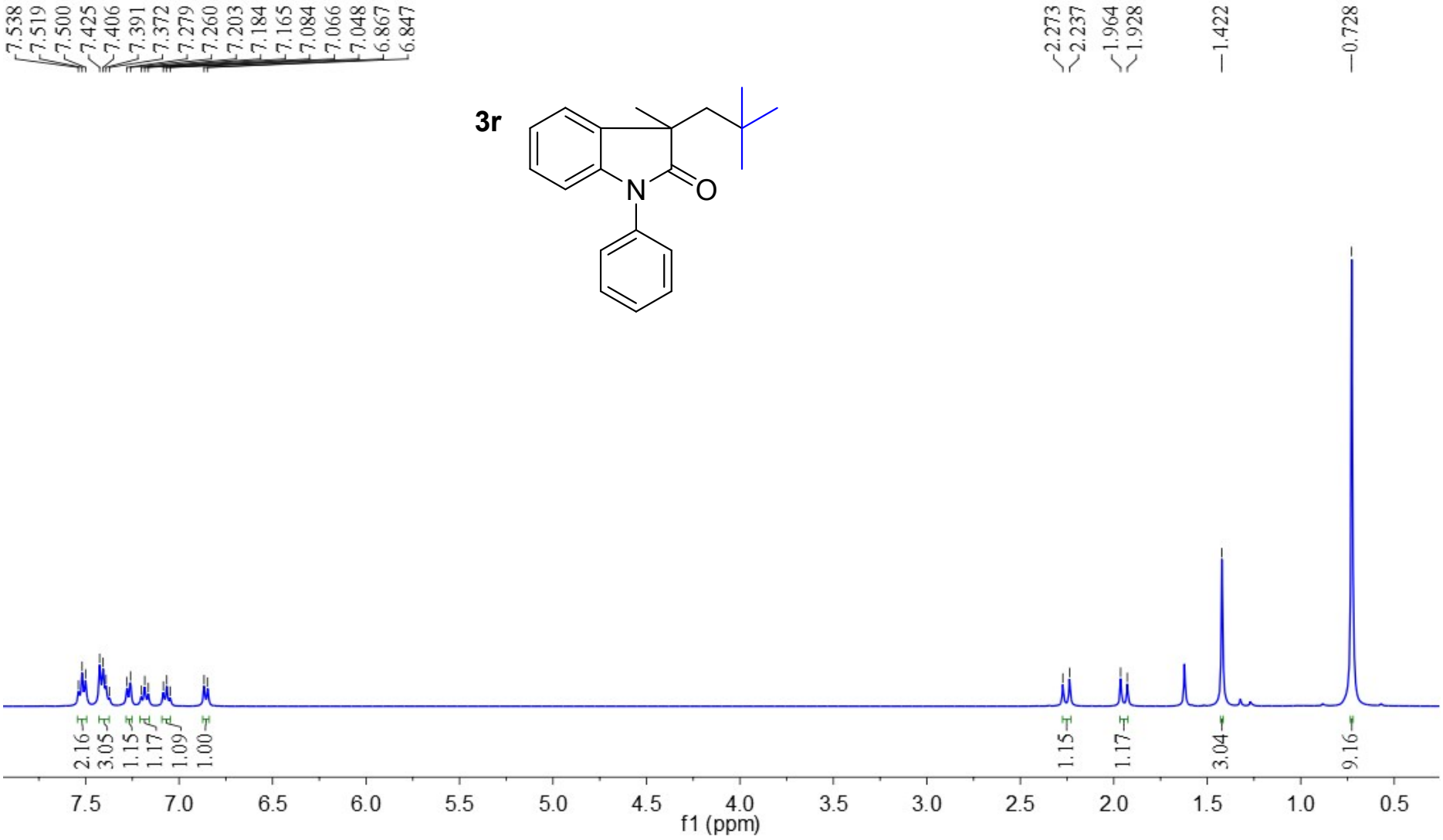
134.592
127.551
124.220
121.833
108.285
77.477
77.160
76.842
50.698
47.529
34.640
31.976
31.016
28.758
12.373
3q
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.500
7.425
7.406
7.391
7.372
7.279
7.260
7.203
7.184
7.165
7.084
7.066
7.048
6.867
6.847
2.273
2.237
1.964
1.928
1.422
0.728
3r
N
O
2.16
3.05
1.15
1.17
1.09
1.00
1.15
1.17
3.04
9.16
7.5
7.0
6.5
6.0
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5.0
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4.0
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3.0
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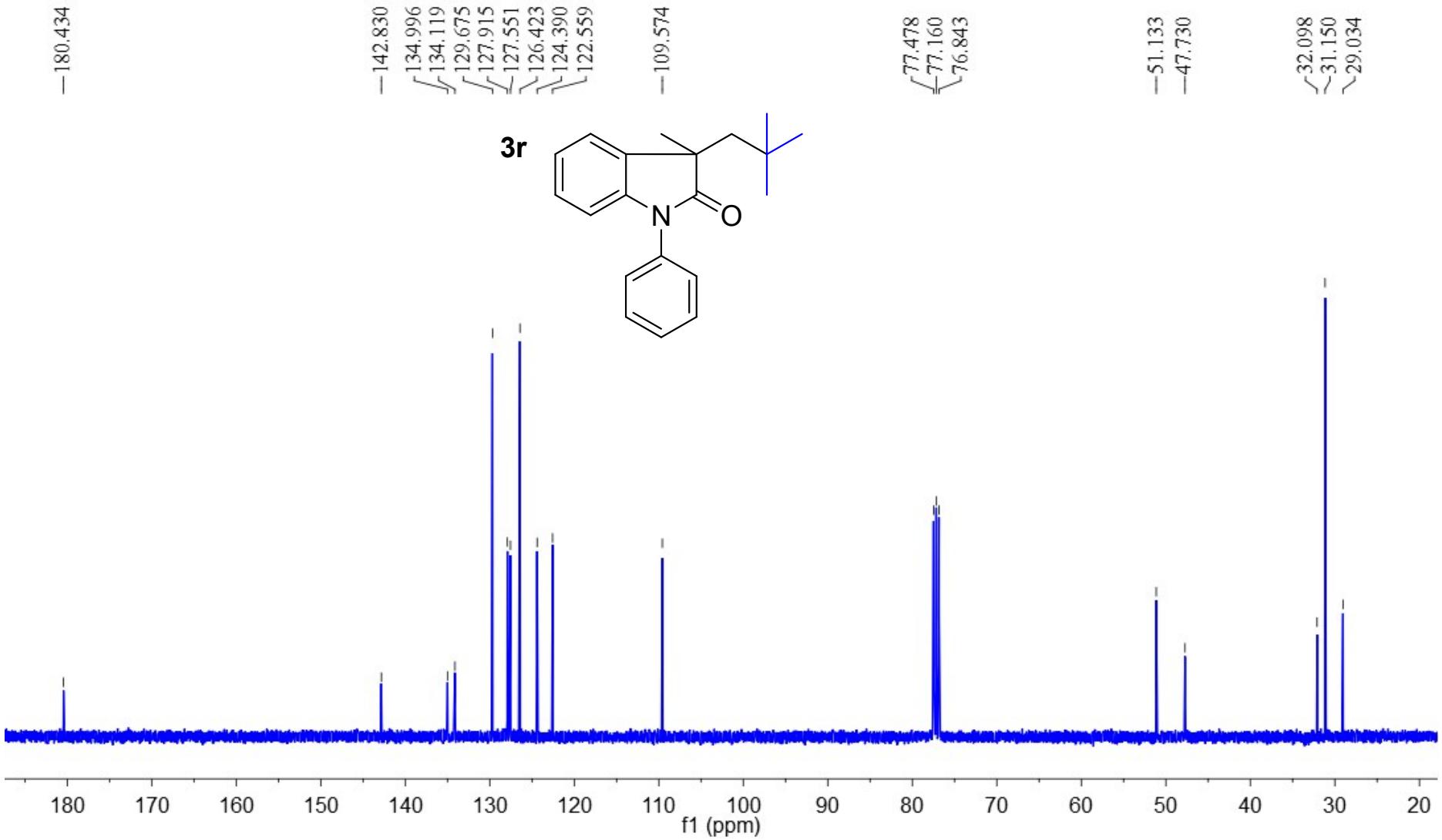
2.0
1.5
1.0
0.5
f1 (ppm)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129.675
127.915
127.551
126.423
124.390
122.559
109.574
77.478
77.160
76.843
51.133
47.730
32.098
31.150
29.034
3r
N
O
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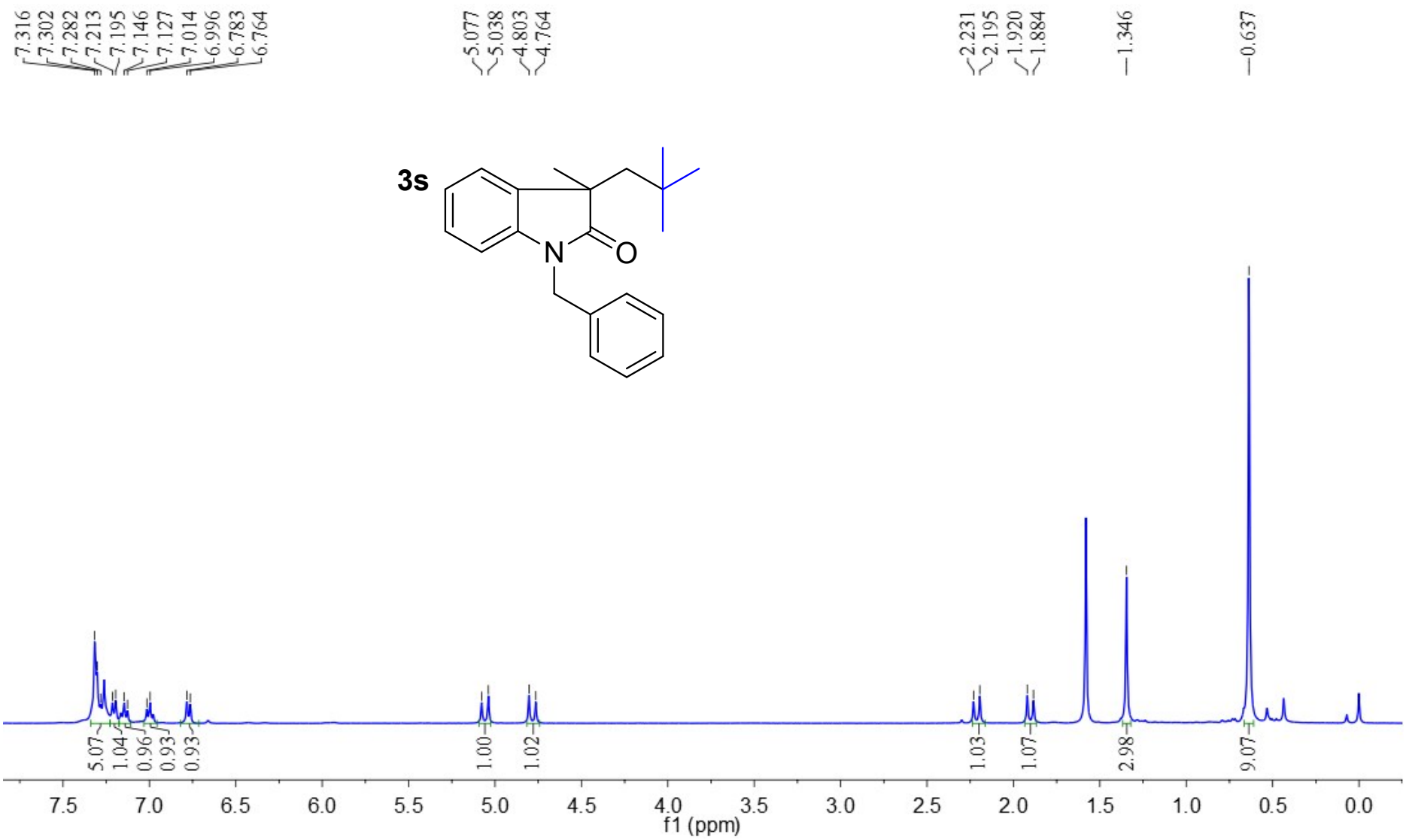
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)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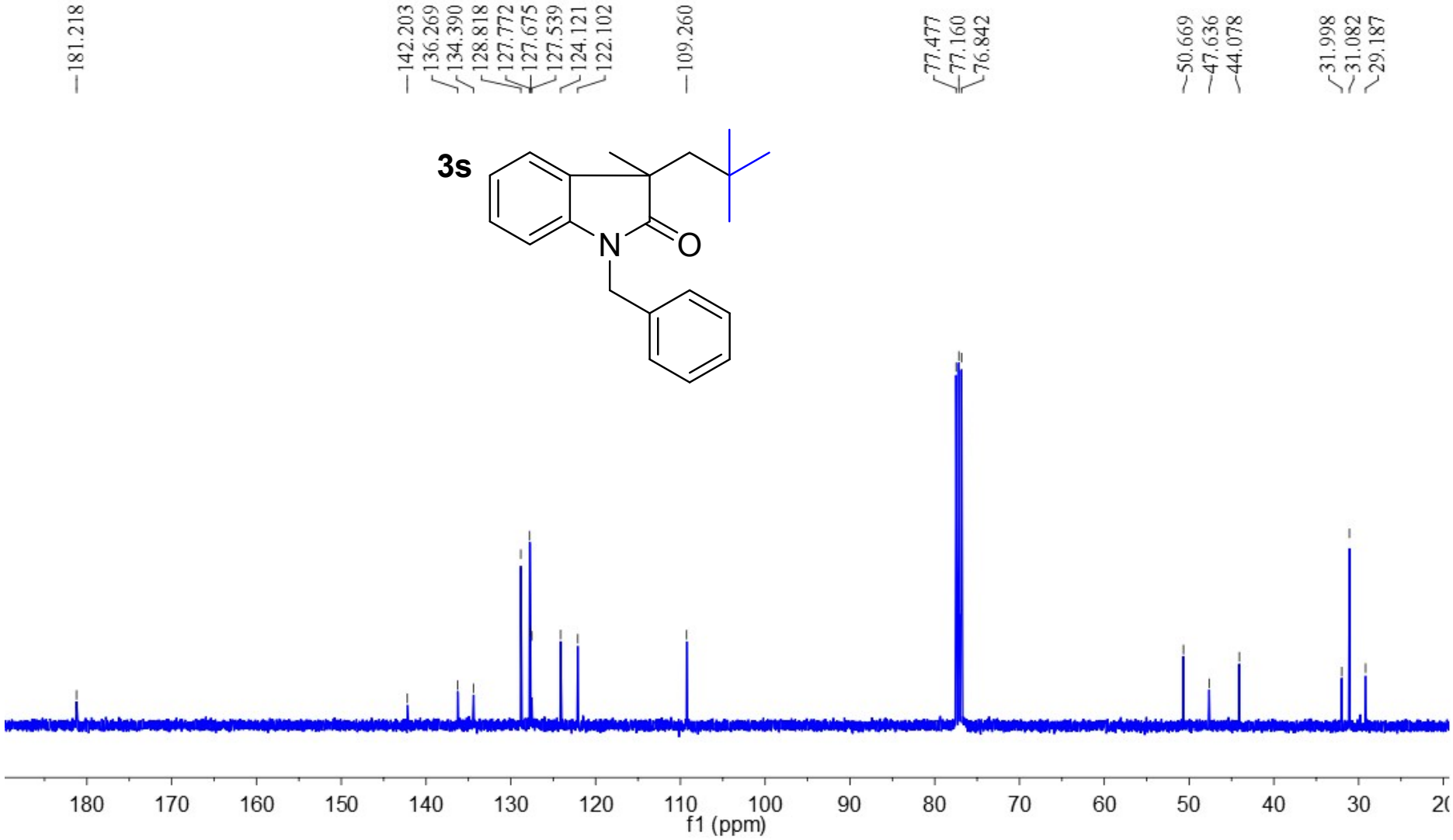
7.213
7.195
7.146
7.127
7.014
6.996
6.783
6.764
5.077
5.038
4.803
4.764
2.231
2.195
1.920
1.884
1.346
0.637
3s
N
O
5.07
1.04
0.96
0.93
0.93
1.00
1.02
1.03
1.07
2.98
9.07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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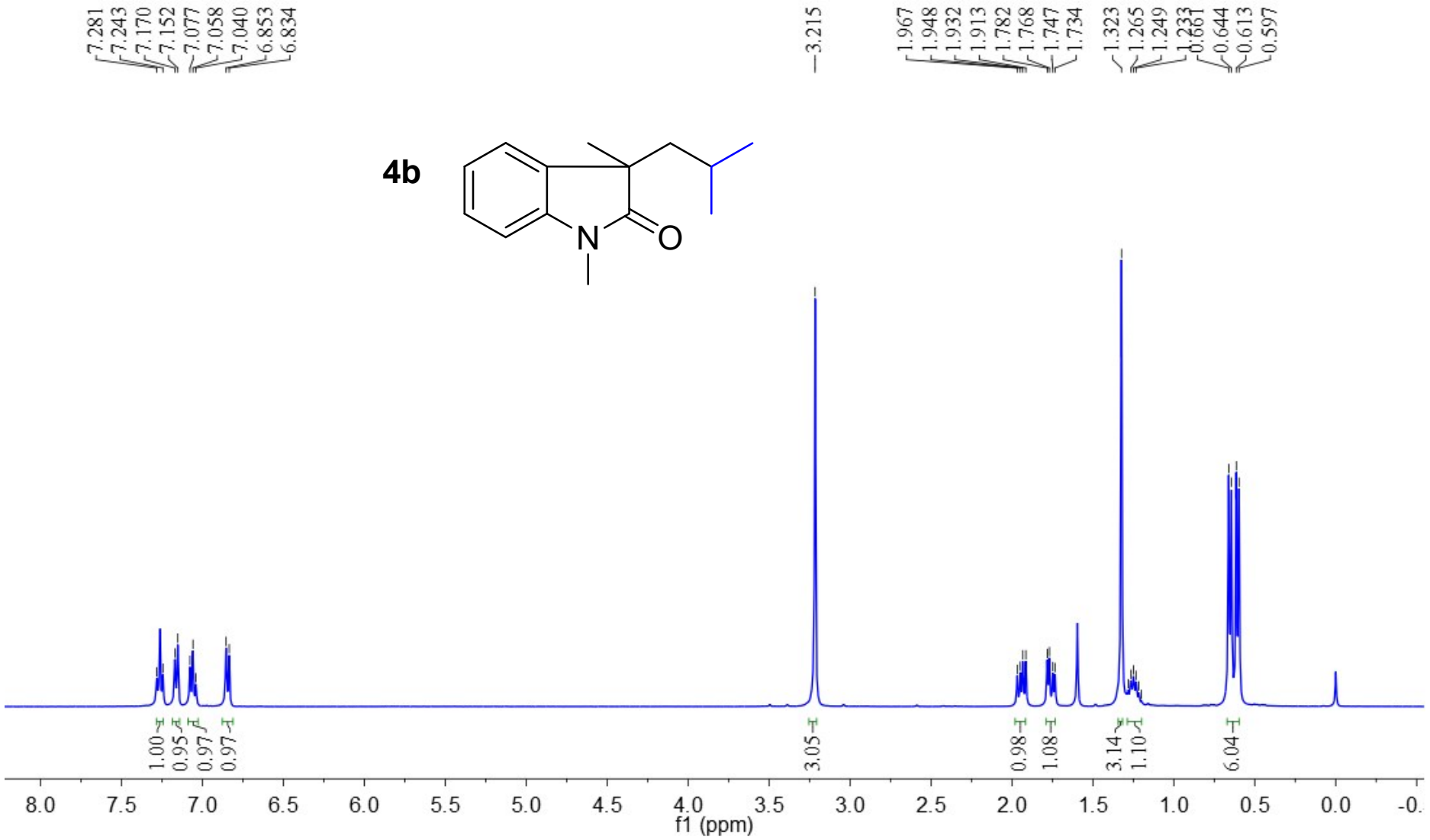
127.772
127.675
127.539
124.121
122.102
109.260
77.477
77.160
76.842
50.669
47.636
44.078
31.998
31.082
29.187
3s
N
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)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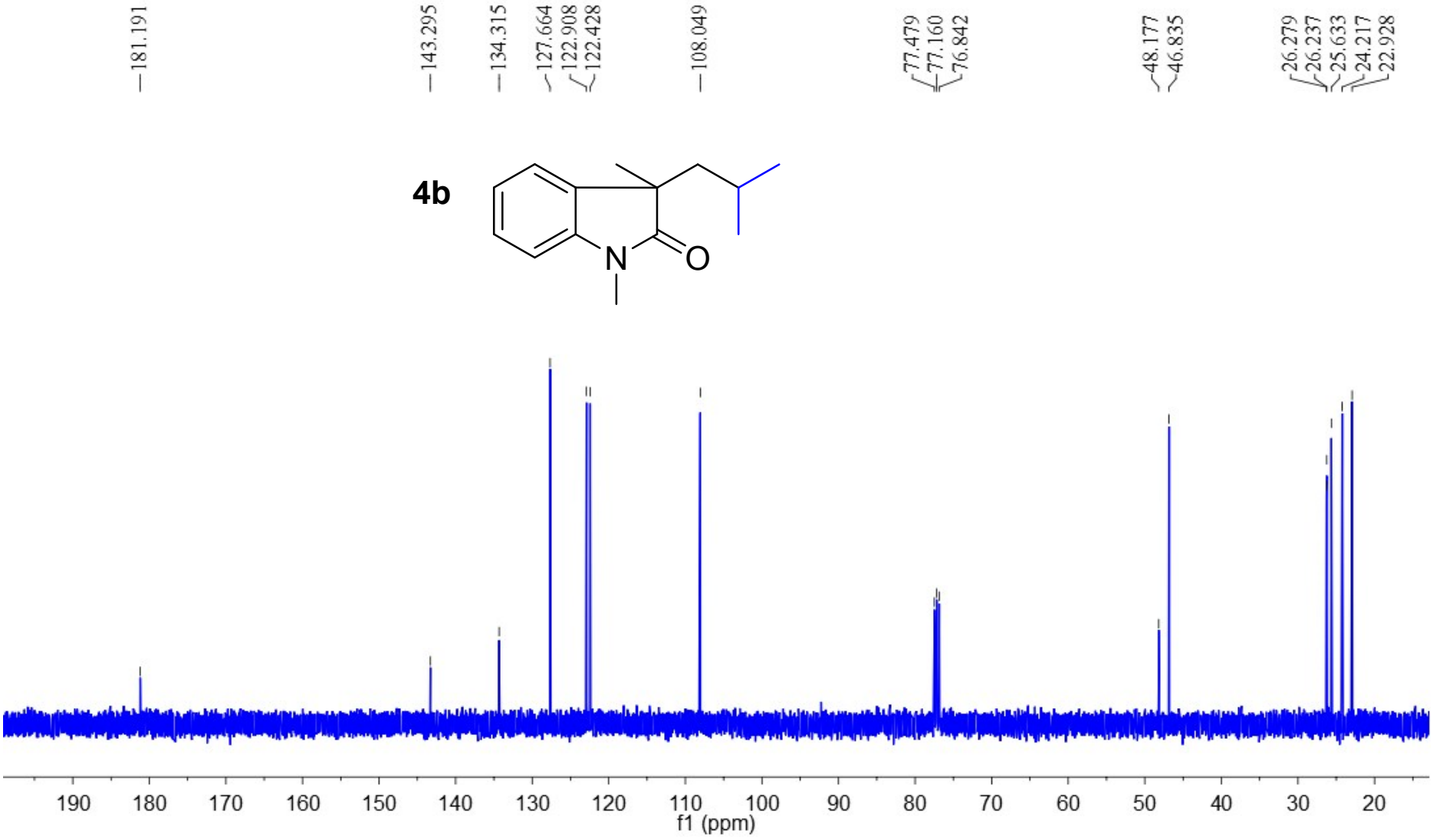
7.152
7.077
7.058
7.040
6.853
6.834
3.215
1.967
1.948
1.932
1.913
1.782
1.768
1.747
1.734
1.323
1.265
1.249
1.233
0.681
0.644
0.613
0.597
4b
N
O
1.00
0.95
0.97
0.97
3.05
0.98
1.08
3.14
1.10
6.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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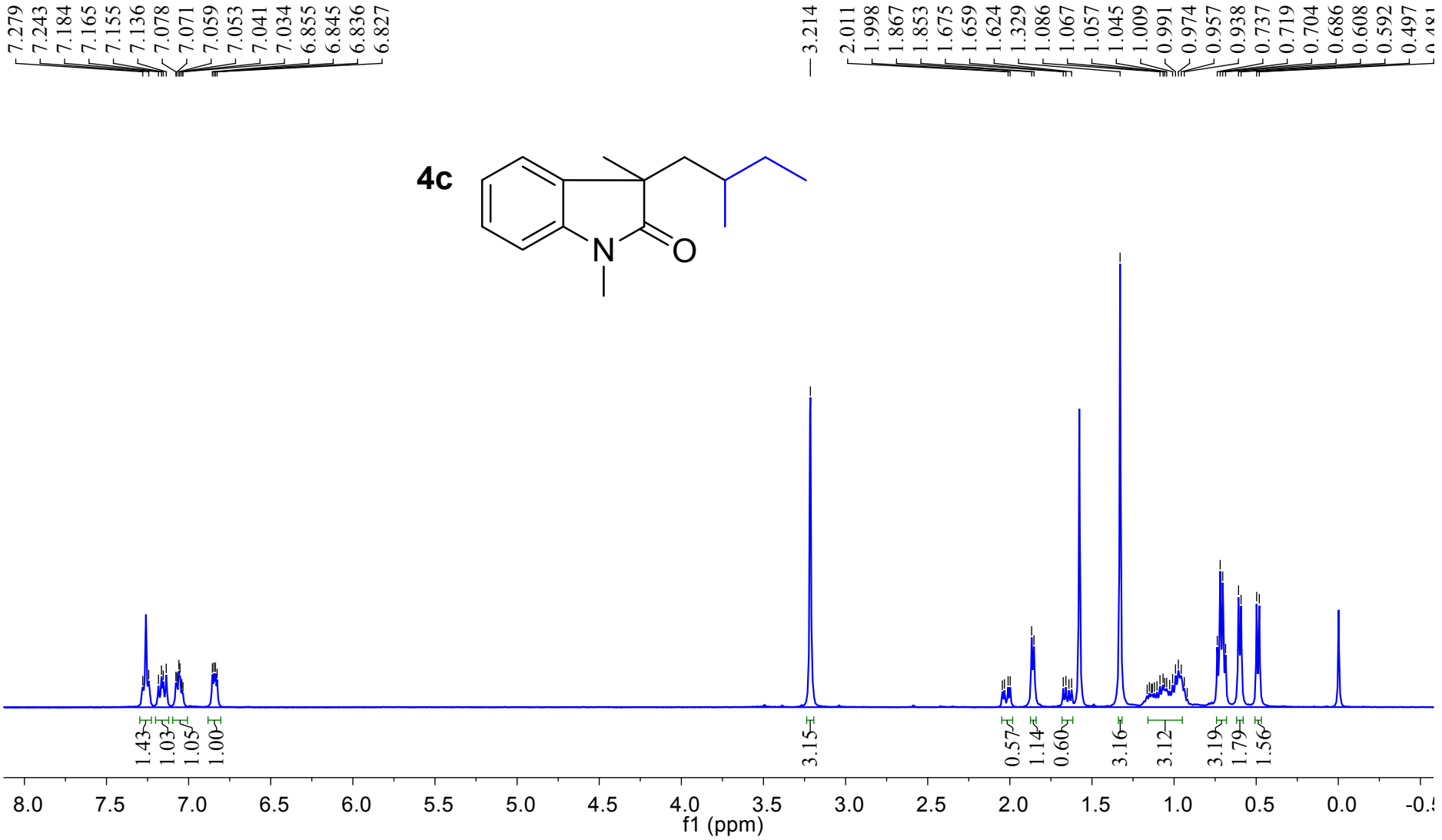
77.160
76.842
48.177
46.835
26.279
26.237
25.633
24.217
22.928
4b
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)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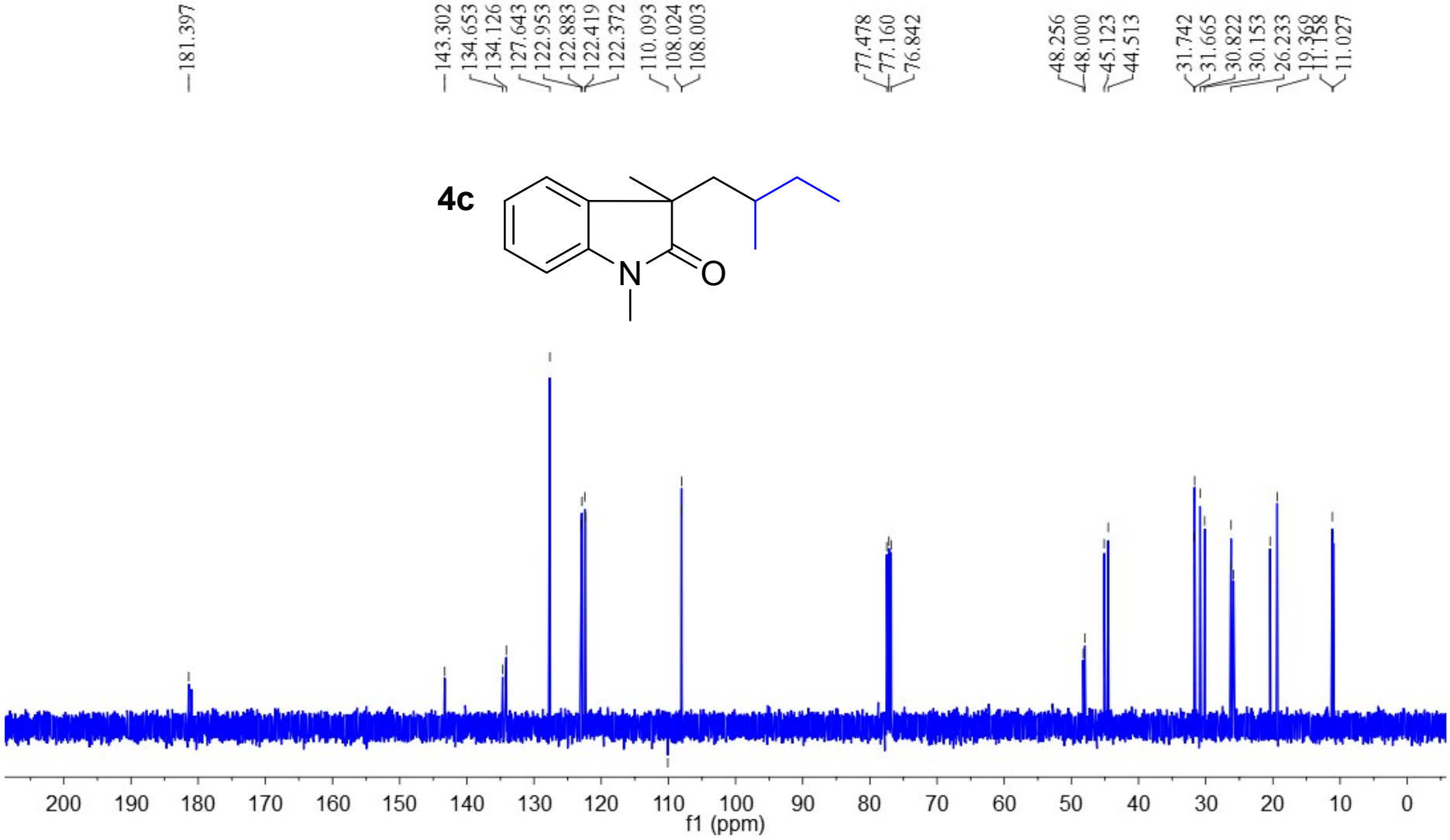
7.155
7.136
7.078
7.071
7.059
7.053
7.041
7.034
6.855
6.845
6.836
6.827
3.214
2.011
1.998
1.867
1.853
1.675
1.659
1.624
1.329
1.086
1.067
1.057
1.045
1.009
0.991
0.974
0.957
0.938
0.737
0.719
0.704
0.686
0.608
0.592
0.497
4c
N
O
1.43
1.03
1.05
1.00
3.15
0.57
1.14
0.60
3.16
3.12
3.19
1.79
1.56
8.0
7.5
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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77.160
76.842
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48.000
45.123
44.513
31.742
31.665
30.822
30.153
26.233
19.369
11.158
11.027
4c
N
O
200
190
180
170
160
150
140
130
120
110
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90
80
70
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50
40
30
20
10
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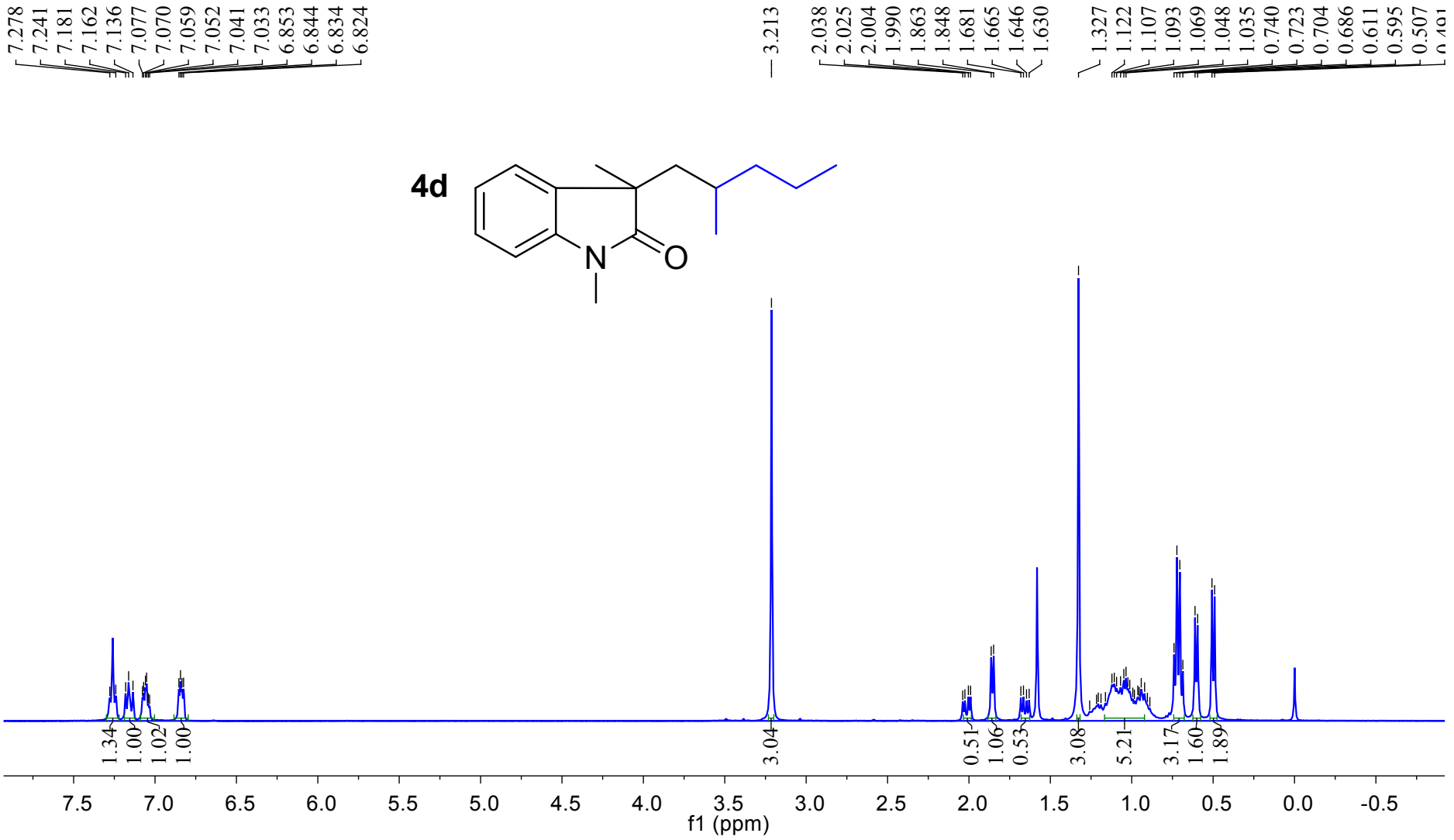
f1 (ppm)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7.059
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7.041
7.033
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6.844
6.834
6.824
3.213
2.038
2.025
2.004
1.990
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1.848
1.681
1.665
1.646
1.630
1.327
1.122
1.107
1.093
1.069
1.048
1.035
0.740
0.723
0.704
0.686
0.611
0.595
0.507
4d
N
O
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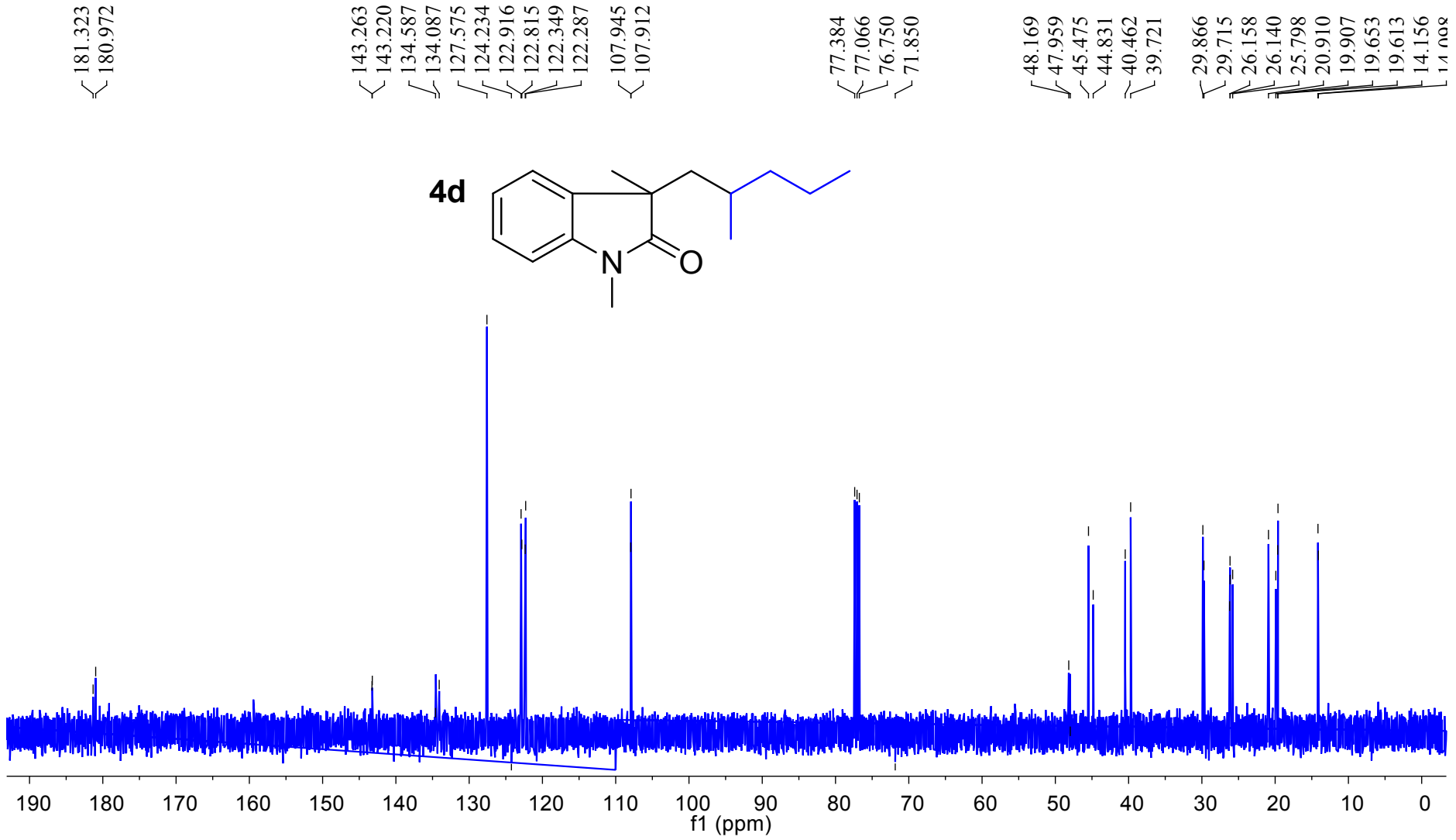
1.00
1.02
1.00
3.04
0.51
1.06
0.53
3.08
5.21
3.17
1.60
1.89
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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124.234
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107.945
107.912
77.384
77.066
76.750
71.850
48.169
47.959
45.475
44.831
40.462
39.721
29.866
29.715
26.158
26.140
25.798
20.910
19.907
19.653
19.613
14.156
4d
N
O
190
180
170
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150
140
130
120
110
100
90
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70
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50
40
30
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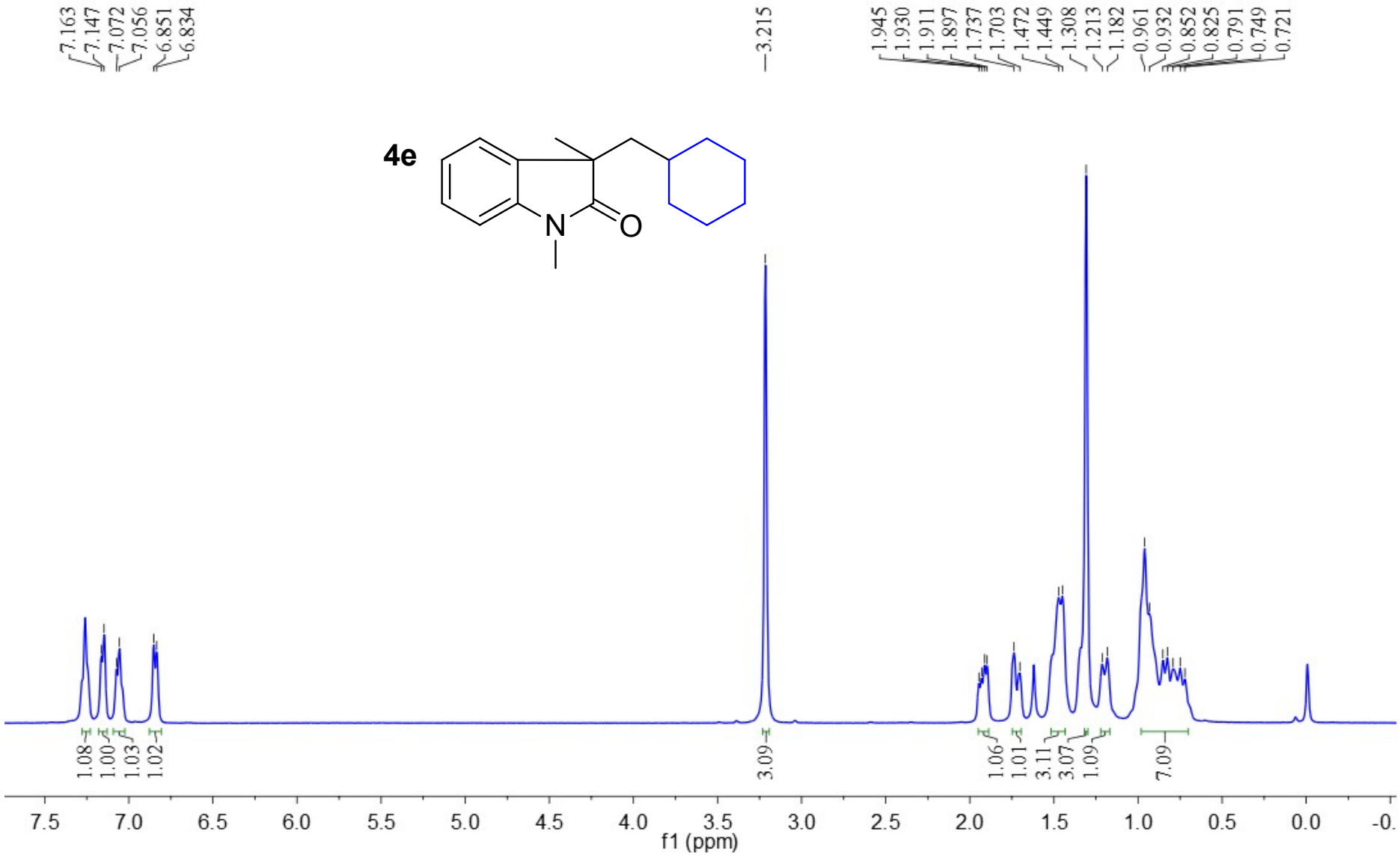
f1 (ppm)


4e
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6.834
3.215
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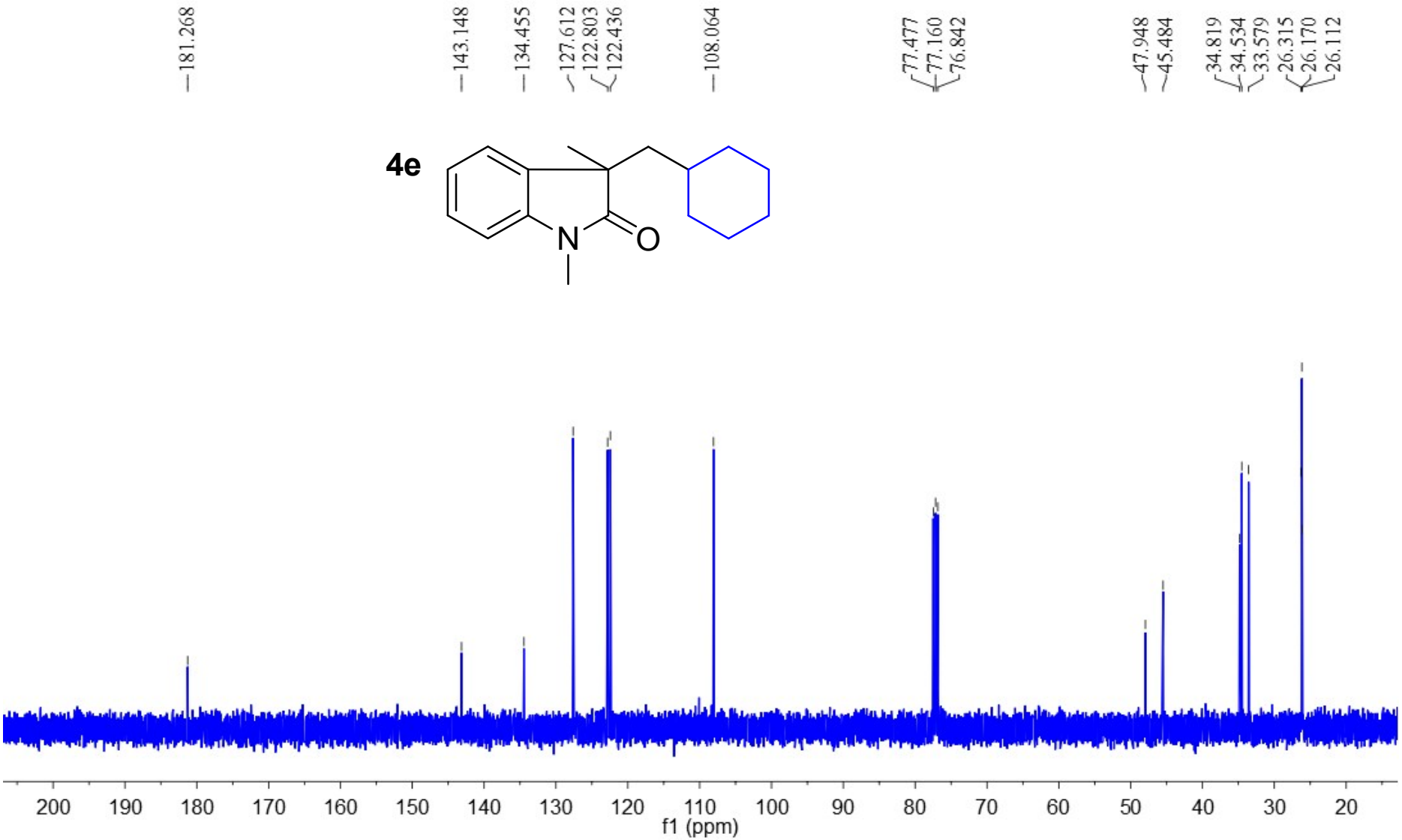
1.930
1.911
1.897
1.737
1.703
1.472
1.449
1.308
1.213
1.182
0.961
0.932
0.852
0.825
0.791
0.749
0.721
1.08
1.00
1.03
1.02
3.09
1.06
1.01
3.11
3.07
1.09
7.09
f1 (ppm)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77.160
76.842
47.948
45.484
34.819
34.534
33.579
26.315
26.170
26.112
4e
N
O
200
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150
140
130
120
110
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70
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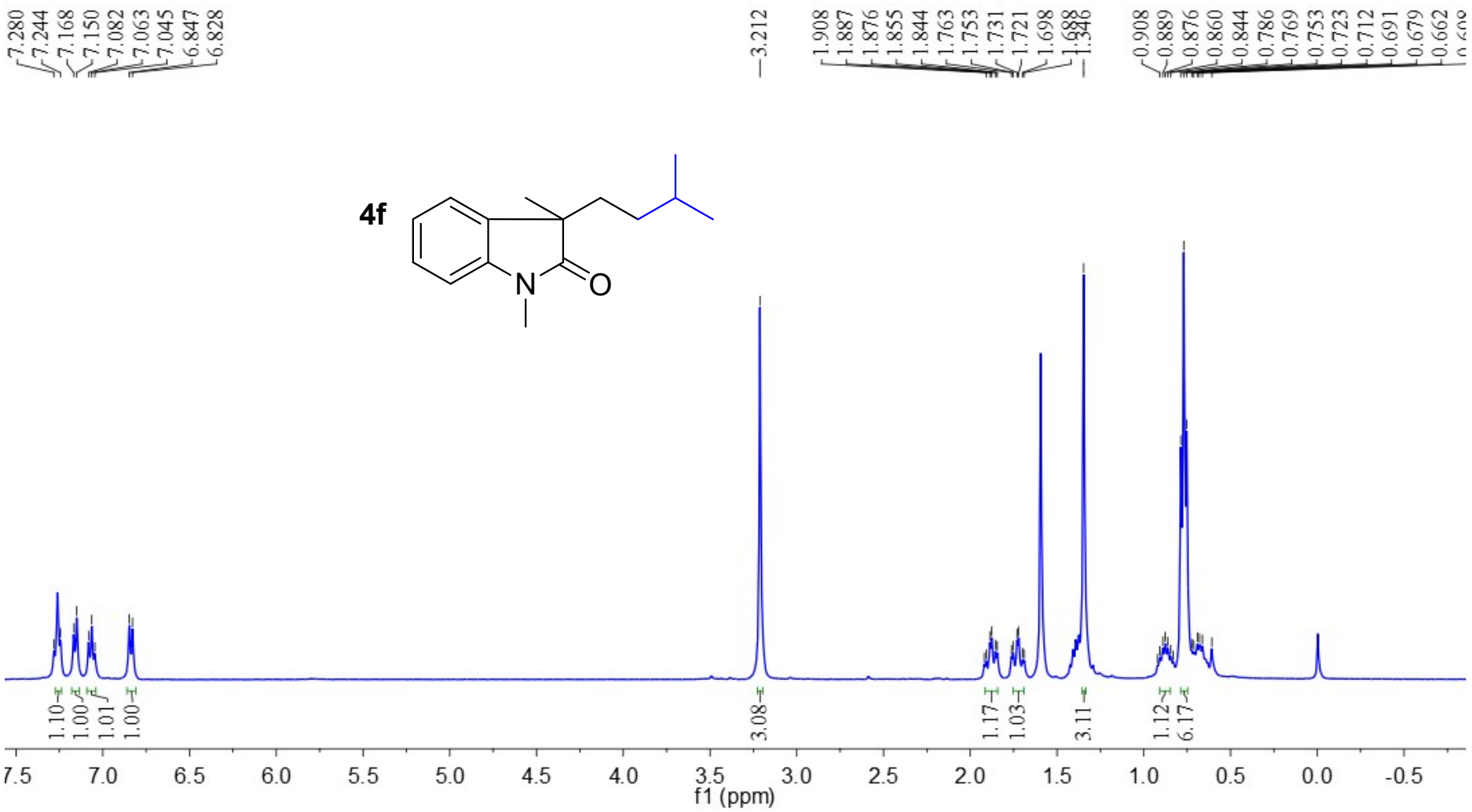
30
20
f1 (ppm)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6.828
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1.887
1.876
1.855
1.844
1.763
1.753
1.731
1.721
1.698
1.688
1.346
0.908
0.889
0.876
0.860
0.844
0.786
0.769
0.753
0.723
0.712
0.691
0.679
0.662
4f
N
O
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1.00
1.01
1.00
3.08
1.17
1.03
3.11
1.12
6.17
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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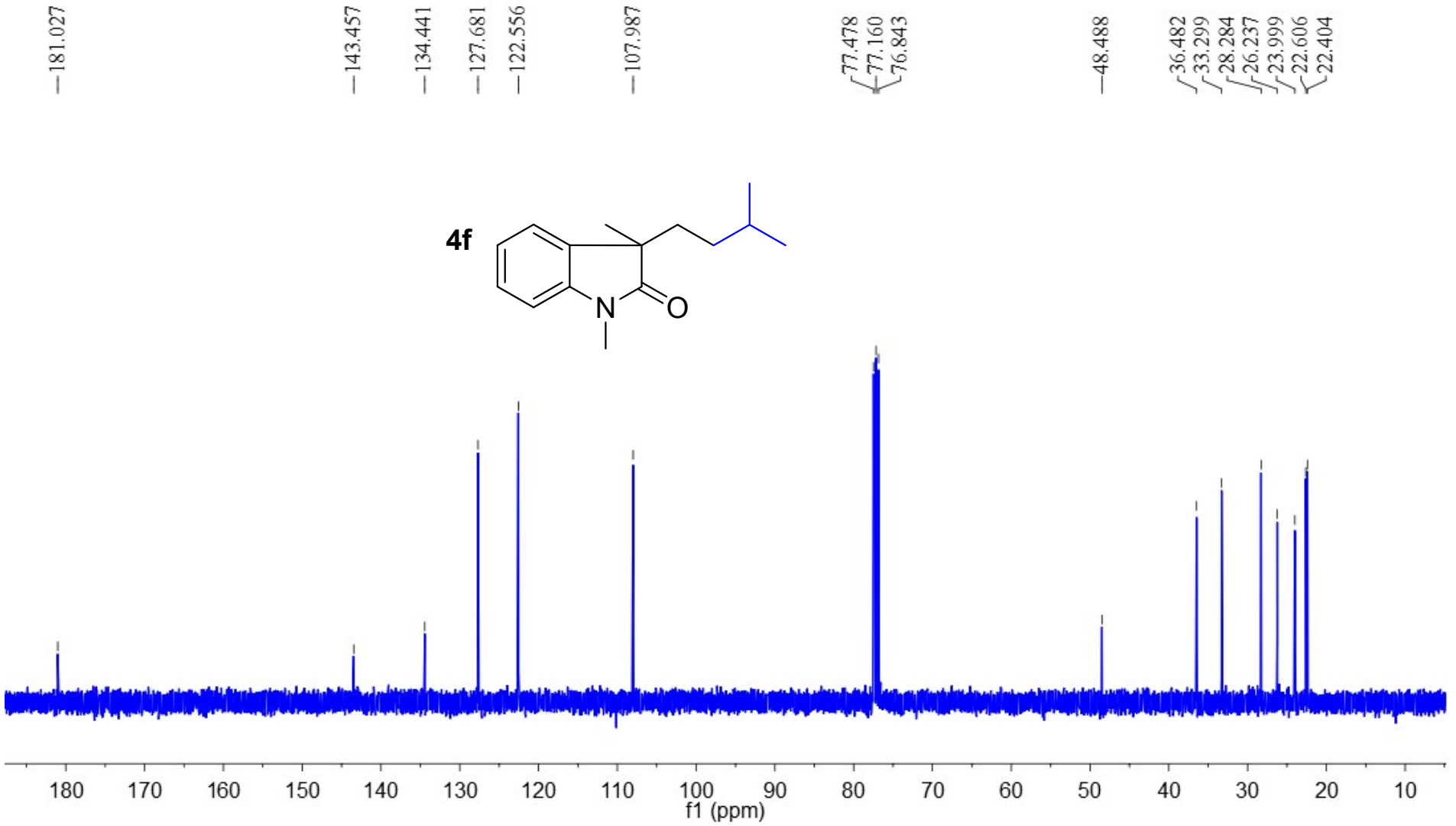
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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77.160
76.843
48.488
36.482
33.299
28.284
26.237
23.999
22.606
22.404
4f
N
O
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120
110
100
90
80
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60
50
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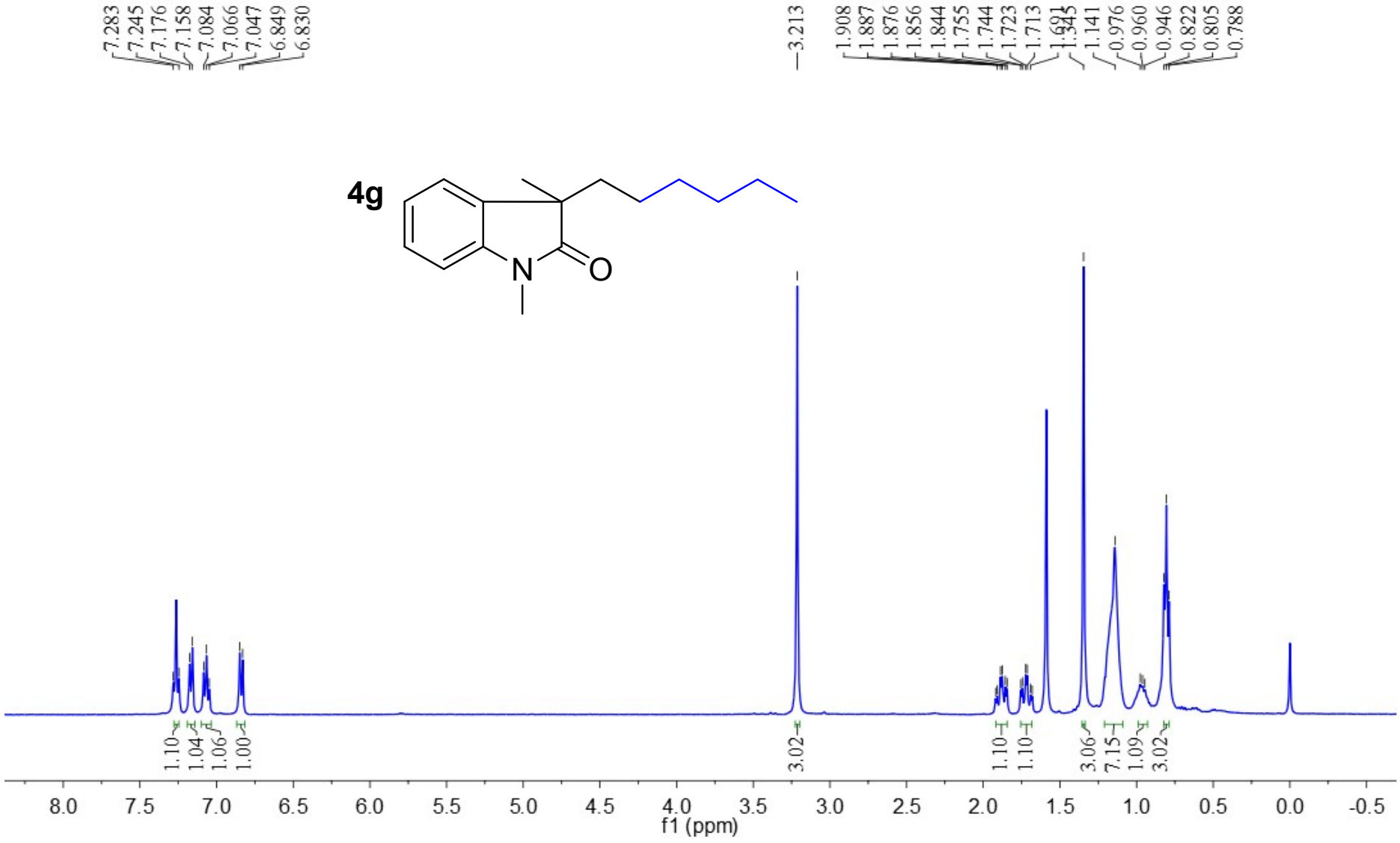
30
20
10
f1 (ppm)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7.084
7.066
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3.213
1.908
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1.876
1.856
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1.345
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0.822
0.805
0.788
4g
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O
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1.06
1.00
3.02
1.10
1.10
3.06
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1.09
3.02
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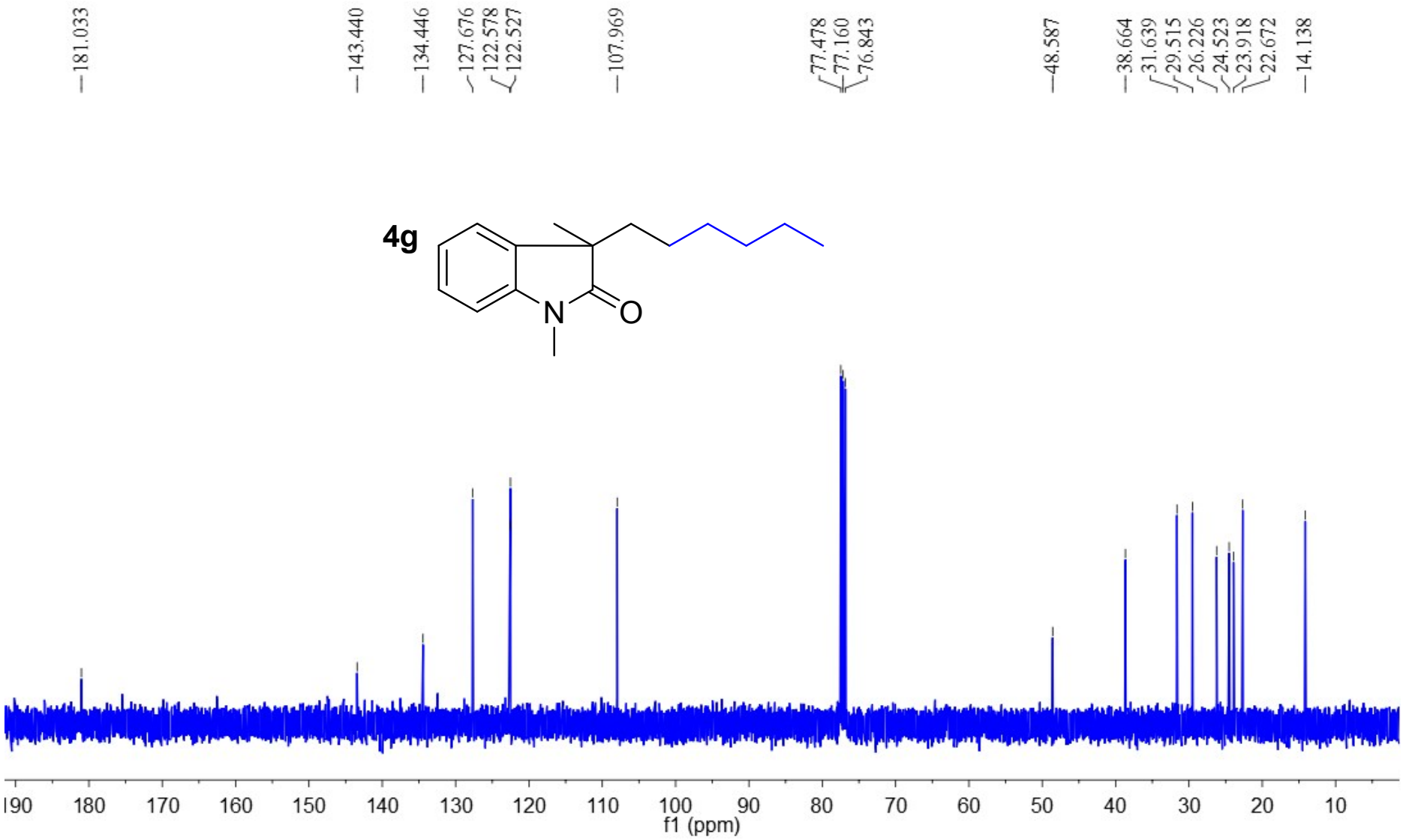
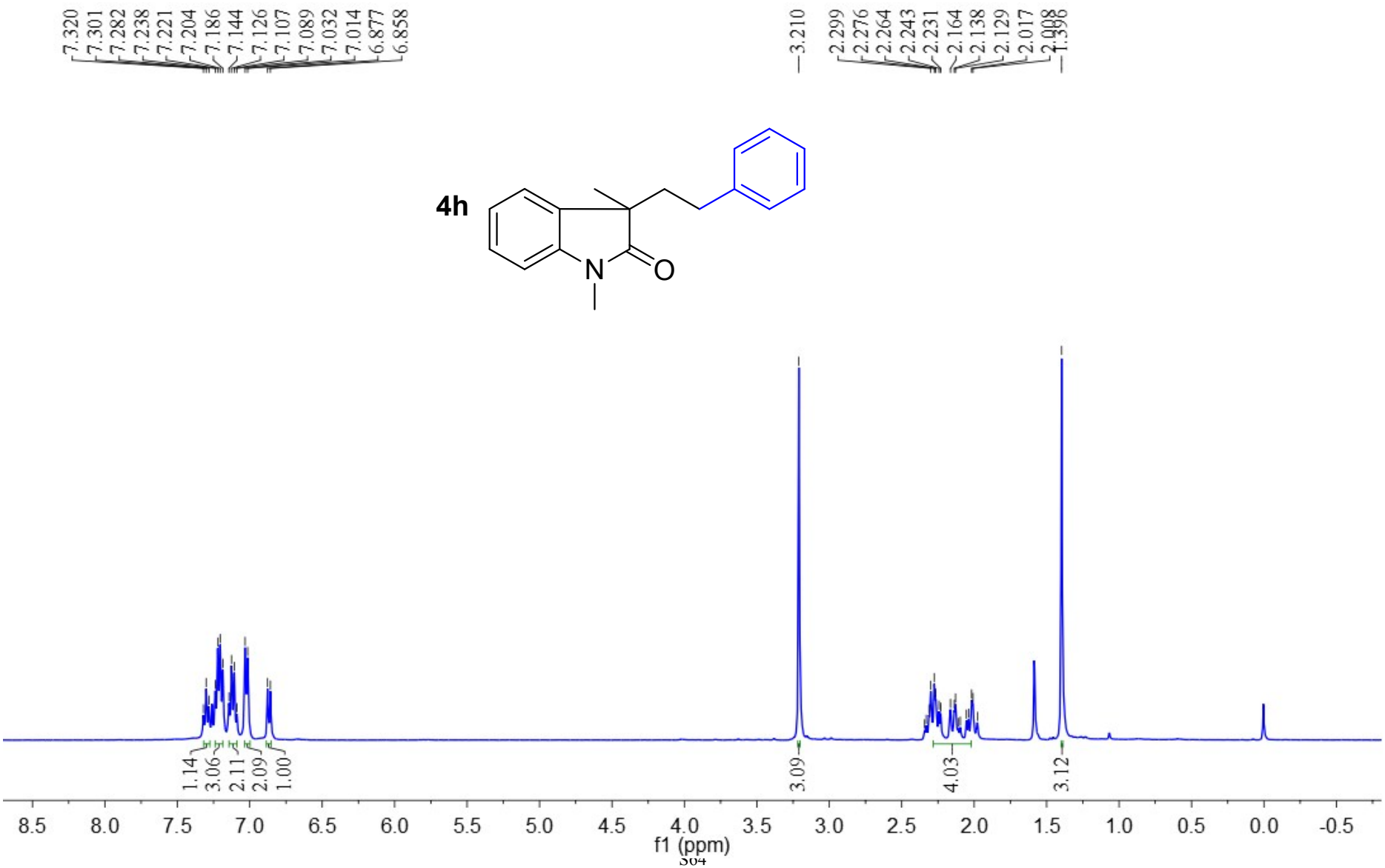
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


181.033
143.440
134.446
127.676
122.578
122.527
107.969
77.478
77.160
76.843
48.587
38.664
31.639
29.515
26.226
24.523
23.918
22.672
14.138
4g
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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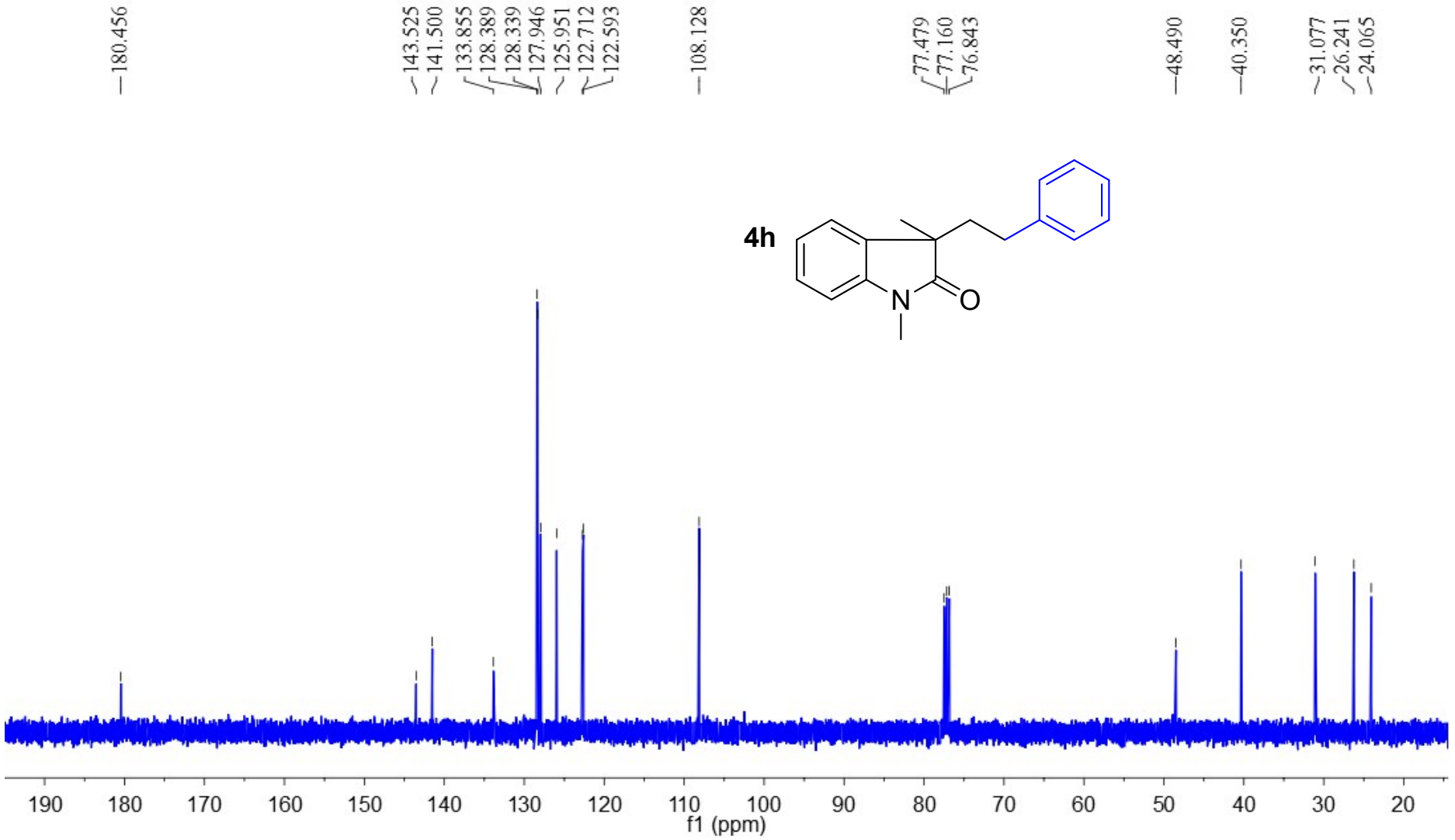
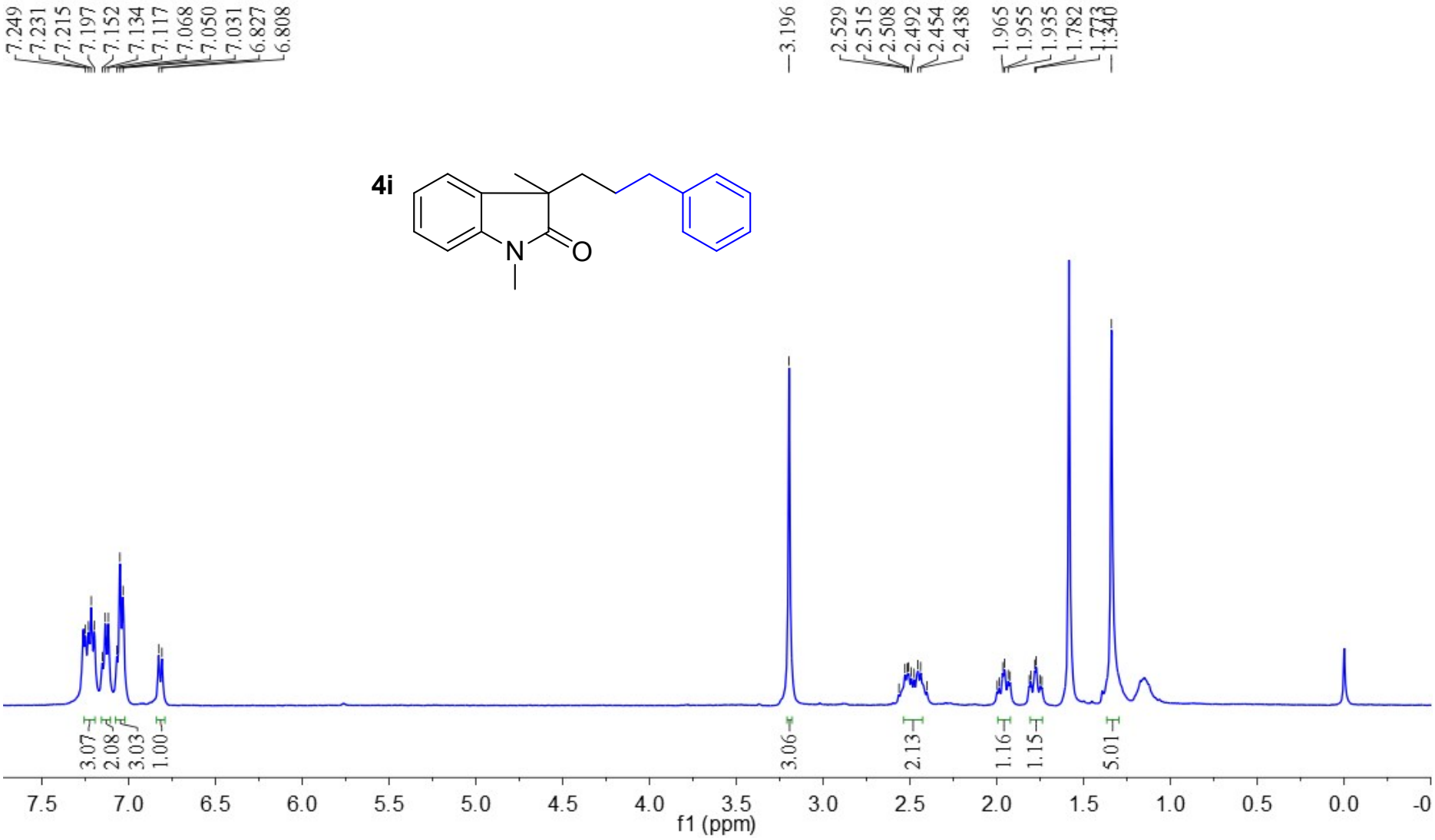
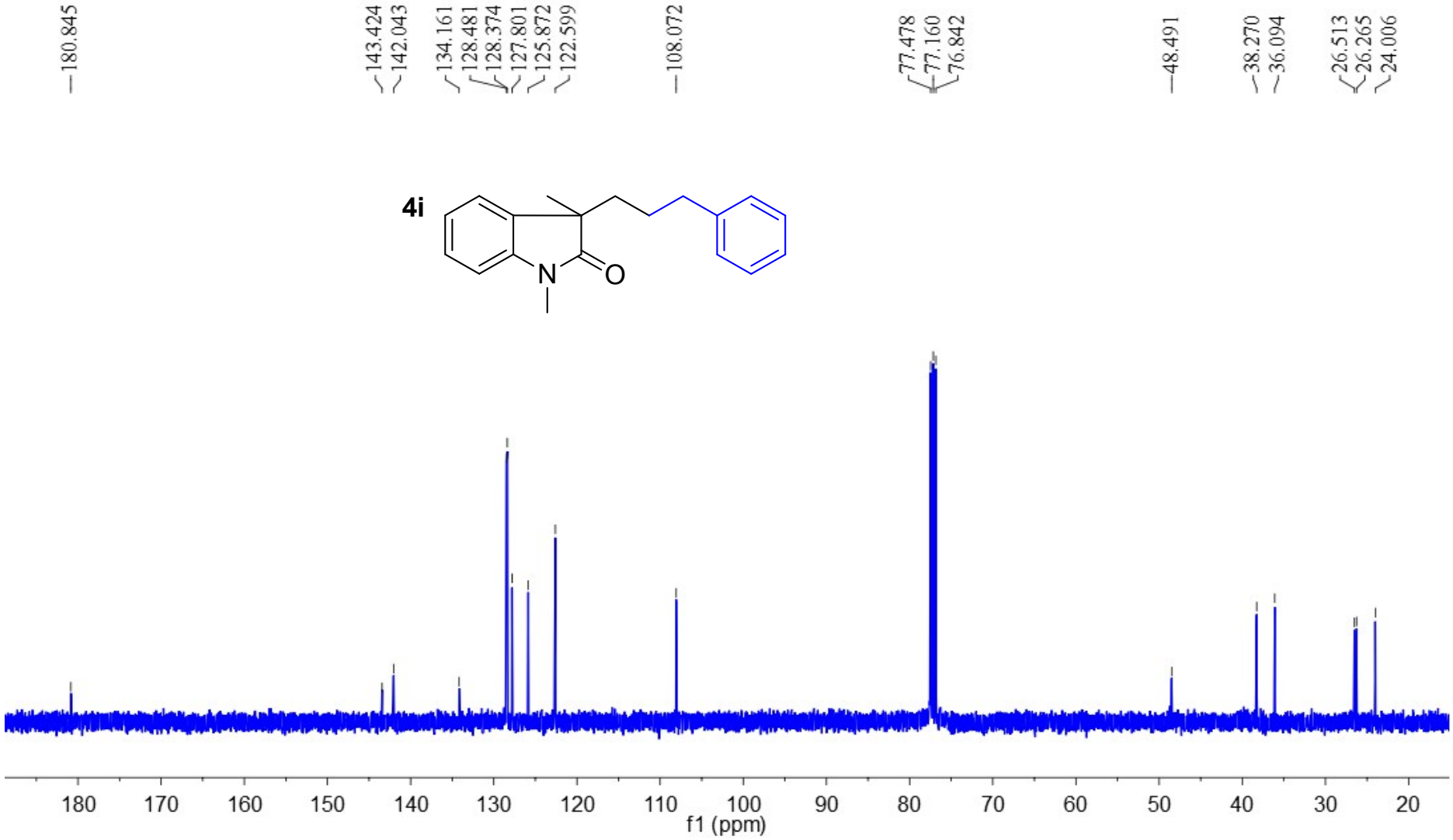
7.301
7.282
7.238
7.221
7.204
7.186
7.144
7.126
7.107
7.089
7.032
7.014
6.877
6.858
3.210
2.299
2.276
2.264
2.243
2.231
2.164
2.138
2.129
2.017
2.008
1.398
4h
N
O
1.14
3.06
2.11
2.09
1.00
3.09
4.03
3.12
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


180.456
143.525
141.500
133.855
128.389
128.339
127.946
125.951
122.712
122.593
108.128
77.479
77.160
76.843
48.490
40.350
31.077
26.241
24.065
4h
N
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.249
7.231
7.215
7.197
7.152
7.134
7.117
7.068
7.050
7.031
6.827
6.808
3.196
2.529
2.515
2.508
2.492
2.454
2.438
1.965
1.955
1.935
1.782
1.373
1.340
4i
N
O
3.07
2.08
3.03
1.00
3.06
2.13
1.16
1.15
5.01
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
-0
f1 (ppm)


180.845
143.424
142.043
134.161
128.481
128.374
127.801
125.872
122.599
108.072
77.478
77.160
76.842
48.491
38.270
36.094
26.513
26.265
24.006
4i
N
O
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)