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# Supporting Information for

# An Intermolecular Functionalization Method for the Synthesis of 3-Hydroxy-2-oxindoles

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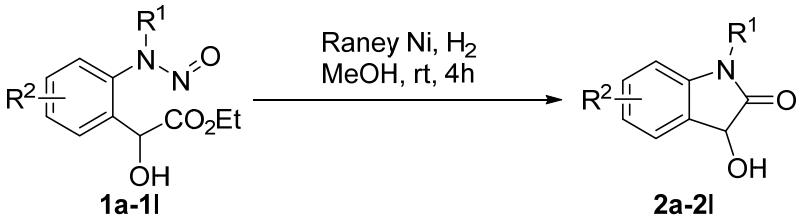
## 1. General Methods

### 1) Materials

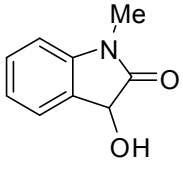
All reagents and solvents were purchased from commercial sources and used without further purification unless otherwise noted. MeOH was freshly distilled over Na before use. Raney Ni was purchased from TCI. $^{1}H$ and $^{13}C$ NMR spectra were recorded in $CDCl_3$, or DMSO-$d_6$ on a Bruker AVANCE 400 MHz or 500 MHz spectrometer. The following notations were used: s – singlet, d – doublet, t – triplet, q – quartet, m – multiplet, dd – doublet of doublet, dt – doublet of triplet, td – triplet of doublet, ddd – doublet of doublet of doublet. High-resolution MS (HRMS) spectra were obtained on a Waters Micromass GCT Premier or a Thermo Fisher Scientific LTQ FT Ultra facility. The method for the synthesis of C-H functionalization products (**1a-1w)** as substrate was described in our previous manuscript. [S1]

## 2. Synthesis and Characterization of 3-Hydroxy-2-oxindoles

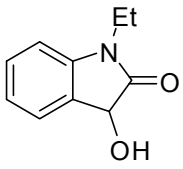



**General procedure for the synthesis of 3-hydroxy-2-oxindoles under Raney Ni/$H_2$ condition:**

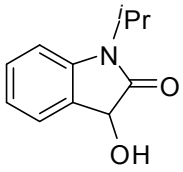
To a 25 mL Schlenk flask were charged with the C-H functionalization product (for example, **1a**, 47.6 mg, 0.2 mmol) and a slurry of Raney nickel (240 mg, 4 mmol). The Schlenk flask was sealed with a rubber septum and filled with hydrogen by 5 evacuation/backfill cycles, and dry MeOH (2 mL) was then added. The reaction mixture was stirred vigorously. To the flask was then attached a hydrogen balloon as the hydrogen source. The reaction was allowed to proceed at room temperature (rt) for 4 h. The solution was then filtered by cannula under Ar. The solvent was removed in vacuo to directly yield the product unless otherwise noted (recrystallization from EtOAc).



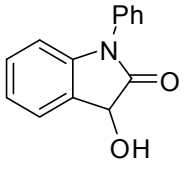

**3-Hydroxy-1-methylindolin-2-one (2a):** The title compound was obtained as a white powder in 95% yield. **$^{1}H$ NMR (400 MHz, $CDCl_3$)** δ 7.47 (d, *J* = 7.3 Hz, 1H), 7.34 (t, *J* = 7.7 Hz, 1H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 5.10 (s, 1H), 3.19 (s, 3H). **$^{13}C$ NMR (101 MHz, $CDCl_3$)** δ 177.38, 144.00, 129.98, 127.19, 125.32, 123.47, 108.68, 70.03, 26.46. **HRMS (EI)** Calcd. for $C_9H_9NO_2$: $[M]^+$, 163.0633. Found: *m/z* 163.0636.



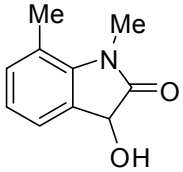
**1-Ethyl-3-hydroxyindolin-2-one (2b):** The title compound was obtained as a white powder in 95% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.47 (d, *J* = 7.3 Hz, 1H), 7.33 (t, *J* = 7.7 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.9 Hz, 1H), 5.07 (s, 1H), 3.83 – 3.71 (m, 2H), 1.28 (t, *J* = 7.2 Hz, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.17, 143.05, 129.85, 127.56, 125.53, 123.25, 108.79, 70.04, 35.02, 12.72. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_2$: $[M]^+$, 177.0790. Found: *m/z* 177.0794.



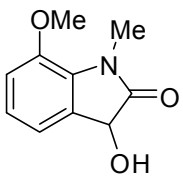
**3-Hydroxy-1-isopropylindolin-2-one (2c):** The title compound was obtained as a white powder in 65% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.48 (d, *J* = 7.3 Hz, 1H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.99 (d, *J* = 7.9 Hz, 1H), 5.04 (s, 1H), 4.57 (dt, *J* = 14.1, 7.0 Hz, 1H), 4.17 (s, 1H), 1.48 (dd, *J* = 7.0, 2.8 Hz, 6H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.05, 142.67, 129.66, 127.69, 125.59, 122.87, 110.36, 69.99, 44.26, 19.58, 19.33. **HRMS (EI)** Calcd. for $C_{11}H_{13}NO_2$: $[M]^+$, 191.0946. Found: *m/z* 191.0948.



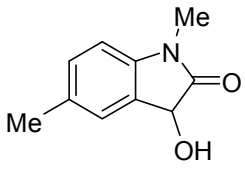
**3-Hydroxy-1-phenylindolin-2-one (2d):** The title compound was obtained as a white powder in 78% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.53 (t, *J* = 7.8 Hz, 3H), 7.42 (td, *J* = 7.5, 1.2 Hz, 3H), 7.29 – 7.21 (m, 1H), 7.13 (t, *J* = 7.5 Hz, 1H), 6.79 (d, *J* = 7.9 Hz, 1H), 5.29 (s, 1H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 176.89, 144.07, 134.09, 129.92, 129.85, 128.56, 126.97, 126.61, 125.69, 123.89, 109.97, 69.98. **HRMS (EI)** Calcd. for $C_{14}H_{11}NO_2$: $[M]^+$, 225.0790. Found: *m/z* 225.0792.



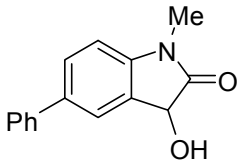
**3-Hydroxy-1,7-dimethylindolin-2-one (2e):** The title compound was obtained as a slightly grayish white powder in 98% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.30 (d, *J* = 7.2 Hz, 1H), 7.07 (d, *J* = 7.7 Hz, 1H), 6.99 (t, *J* = 7.5 Hz, 1H), 3.47 (s, 3H), 3.33 (s, 1H), 2.55 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 176.61, 141.21, 132.54, 129.18, 122.39, 122.15, 119.61, 68.29, 28.81, 18.39. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_2$: $[M]^+$, 177.0790. Found: *m/z* 177.0792.



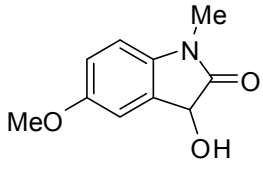
**3-Hydroxy-7-methoxy-1-methylindolin-2-one (2f):** The title compound was obtained as a slightly grayish white powder in 87% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.09 (d, *J* = 7.2 Hz, 1H), 7.05 (t, *J* = 7.7 Hz, 1H), 6.91 (d, *J* = 7.8 Hz, 1H), 5.02 (s, 1H), 3.86 (s, 3H), 3.45 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 175.25, 144.74, 130.93, 130.13, 123.00, 117.34, 113.74, 68.75, 56.34, 28.94. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_3$: $[M]^+$, 193.0739. Found: *m/z* 193.0738.



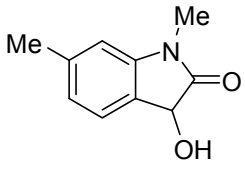
**3-Hydroxy-1,5-dimethylindolin-2-one (2g):** The title compound was obtained as a white powder in 98% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.29 (m, 1H), 7.14 (d, *J* = 7.7 Hz, 1H), 6.72 (d, *J* = 7.8 Hz, 1H), 5.05 (s, 1H), 3.18 (s, 3H), 2.35 (s, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.69, 141.47, 133.04, 129.93, 127.44, 126.12, 108.33, 70.02, 26.42, 21.19. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_2$: $[M]^+$, 177.0790. Found: *m/z* 177.0794.



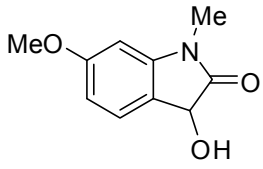
**3-Hydroxy-1-methyl-5-phenylindolin-2-one (2h):** The title compound was obtained as a white powder in 77% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.71 (s, 1H), 7.60 – 7.54 (m, 3H), 7.44 (dd, *J* = 10.4, 4.8 Hz, 2H), 7.34 (t, *J* = 7.3 Hz, 1H), 6.90 (d, *J* = 8.1 Hz, 1H), 5.14 (s, 1H), 3.59 (s, 1H), 3.23 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 176.09, 143.24, 140.07, 134.47, 129.35, 128.95, 127.48, 126.92, 126.23, 122.81, 108.81, 68.87, 25.90. **HRMS (EI)** Calcd. for $C_{15}H_{13}NO_2$: $[M]^+$, 239.0946. Found: *m/z* 239.0948.



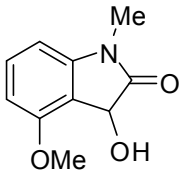
**3-Hydroxy-5-methoxy-1-methylindolin-2-one (2i):** The title compound was obtained as a white powder in 88% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.10 (d, *J* = 2.0 Hz, 1H), 6.86 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.74 (d, *J* = 8.5 Hz, 1H), 5.07 (s, 1H), 3.81 (s, 3H), 3.17 (s, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.07, 156.69, 137.32, 128.47, 114.68, 112.25, 109.15, 70.34, 56.06, 26.53. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_3$: $[M]^+$, 193.0739. Found: *m/z* 193.0741.



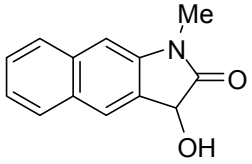
**3-Hydroxy-1,6-dimethylindolin-2-one (2ja):** The title compound was obtained as a pale yellow powder in 97% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.33 (d, *J* = 7.5 Hz, 1H), 6.91 (d, *J* = 7.4 Hz, 1H), 6.65 (s, 1H), 5.03 (s, 1H), 3.17 (s, 3H), 2.39 (s, 3H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.76, 144.10, 140.25, 125.07, 124.35, 123.86, 109.58, 69.86, 26.39, 22.10. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_2$: $[M]^+$, 177.0790. Found: *m/z* 177.0793.



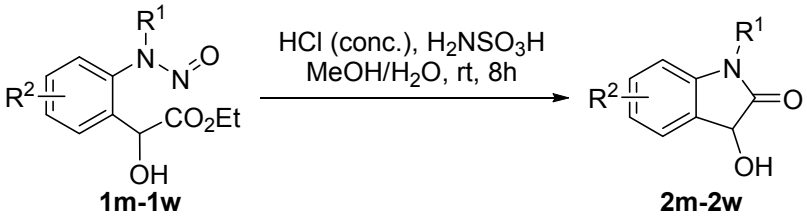
**3-Hydroxy-6-methoxy-1-methylindolin-2-one (2ka):** The title compound was obtained as a pale yellow powder in 79% yield. **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 8.0 Hz, 1H), 6.59 (d, *J* = 8.1 Hz, 1H), 6.41 (s, 1H), 5.00 (s, 1H), 3.84 (s, 3H), 3.17 (s, 3H), 2.99 (s, 1H). **$^{13}$C NMR (126 MHz, DMSO)** δ 176.52, 160.57, 145.10, 125.23, 120.36, 106.35, 96.04, 68.27, 55.39, 25.76. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_3$: $[M]^+$, 193.0739. Found: *m/z* 193.0743.



**3-Hydroxy-4-methoxy-1-methylindolin-2-one (2kb):** The title compound was obtained as a pale yellow powder in 76% yield. **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.31 (t, *J* = 8.1 Hz, 1H), 6.66 (d, *J* = 6.7 Hz, 1H), 6.49 (d, *J* = 5.8 Hz, 1H), 5.15 (s, 1H), 3.91 (s, 3H), 3.17 (s, 3H), 2.89 (s, 1H). **$^{13}$C NMR (126 MHz, DMSO)** δ 175.78, 156.44, 145.34, 130.72, 113.68, 106.73, 101.65, 67.45, 55.31, 25.90. **HRMS (EI)** Calcd. for $C_{10}H_{11}NO_3$: $[M]^+$, 193.0739. Found: *m/z* 193.0740.

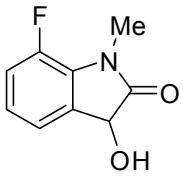


**3-Hydroxy-1-methyl-1*H*-benzo[f]indol-2(3*H*)-one (2l):** The title compound was obtained as a white powder in 89% yield. **$^1$H NMR (400 MHz, DMSO)** δ 7.90 (d, *J* = 8.0 Hz, 1H), 7.85 (d, *J* = 10.2 Hz, 2H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.31 (s, 1H), 6.39 (d, *J* = 7.6 Hz, 1H), 5.07 (d, *J* = 6.8 Hz, 1H), 3.19 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 175.52, 141.87, 133.99, 129.88, 129.72, 128.24, 126.96, 126.63, 123.02, 123.94, 103.50, 68.25, 26.02. **HRMS (EI)** Calcd. for $C_{13}H_{11}NO_2$: $[M]^+$, 213.0790. Found: *m/z* 213.0791.

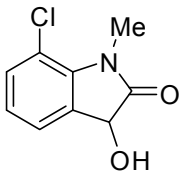



**General procedure for the synthesis of 3-hydroxy-2-oxindoles under $HCl/H_2NSO_3H$ condition:**

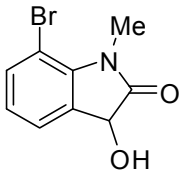
To a 25 mL Schlenk flask were charged with the C-H functionalization product (for example, **1a**, 47.6 mg, 0.2 mmol, 1 equiv) and $H_2NSO_3H$ (97 mg, 1 mmol, 5 equiv). Then $H_2O$ (1 mL), MeOH (1 mL), and conc. HCl (1 mL) were added to the flask. The reaction was allowed to proceed at rt for 8 h. To the resulting solution was added 10 mL $CH_2Cl_2$. The two phases were separated with a separation funnel, and the aqueous phase was extracted twice with $CH_2Cl_2$ (10 mL). The organic phase are combined and washed with saturated brine. The solvent was removed in vacuo to directly yield the product unless otherwise noted (recrystallization from EtOAc).



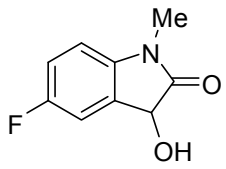
**7-Fluoro-3-hydroxy-1-methylindolin-2-one (2m):** The title compound was obtained as a slightly grayish white powder in 68% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.24 (m, 1H), 7.11 – 7.00 (m, 2H), 5.07 (s, 1H), 3.41 (d, *J* = 2.7 Hz, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 175.79, 146.85 (d, *J* = 241.5 Hz), 131.77 (d, *J* = 2.8 Hz), 129.87 (d, *J* = 8.1 Hz), 123.16 (d, *J* = 6.3 Hz), 120.79 (d, *J* = 3.0 Hz), 116.93 (d, *J* = 19.1 Hz), 68.72 (d, *J* = 2.6 Hz), 28.15 (d, *J* = 5.4 Hz). **HRMS (EI)** Calcd. for $C_9H_8FNO_2$: $[M]^+$, 181.0539. Found: *m/z* 181.0542.



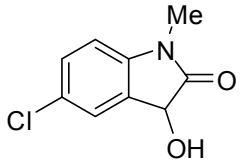

**7-Chloro-3-hydroxy-1-methylindolin-2-one (2n):** The title compound was obtained through recrystallization as a pale yellow powder in 97% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.35 (d, *J* = 7.3 Hz, 1H), 7.29 (m, 1H), 7.08 – 6.98 (m, 1H), 5.04 (s, 1H), 3.56 (s, 3H), 3.41 (s, 1H). **$^{13}$C NMR (126 MHz, DMSO)** δ 176.31, 174.48, 139.37, 139.15, 132.08, 131.89, 130.86, 129.38, 124.13, 123.52, 123.22, 114.05, 77.51, 68.23, 28.91. **HRMS (EI)** Calcd. for $C_9H_8ClNO_2$: $[M]^+$, 197.0244. Found: *m/z* 197.0240.



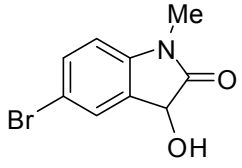

**7-Bromo-3-hydroxy-1-methylindolin-2-one (2o):** The title compound was obtained as a pale yellow powder in 67% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.45 (d, *J* = 8.2 Hz, 1H), 7.39 (dt, *J* = 7.3, 1.1 Hz, 1H), 6.96 (dd, *J* = 8.1, 7.4 Hz, 1H), 5.03 (s, 1H), 3.58 (s, 3H). **$^{13}$C NMR (126 MHz, DMSO)** δ 176.50, 140.61, 134.18, 132.15, 123.92, 101.36, 68.22, 29.13. **HRMS (EI)** Calcd. for $C_9H_8BrNO_2$: $[M]^+$, 240.9738. Found: *m/z* 240.9736.



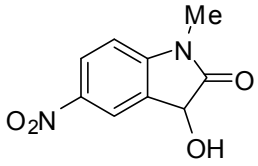

**5-Fluoro-3-hydroxy-1-methylindolin-2-one (2p):** The title compound was obtained as a white powder in 95% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.22 (ddd, *J* = 7.6, 2.6, 0.8 Hz, 1H), 7.05 (td, *J* = 8.8, 2.6 Hz, 1H), 6.76 (dd, *J* = 8.5, 4.0 Hz, 1H), 5.07 (s, 1H), 3.49 (s, 1H), 3.19 (s, 3H). **$^{13}$C NMR (126 MHz, DMSO)** δ 175.77, 158.47 (d, *J* = 237.8 Hz), 139.83, 130.41 (d, *J* = 7.6 Hz), 115.02 (d, *J* = 23.3 Hz), 112.32 (d, *J* = 24.6 Hz), 109.20 (d, *J* = 7.7 Hz), 68.86, 25.89. **HRMS (EI)** Calcd. for $C_9H_8FNO_2$: $[M]^+$, 181.0539. Found: *m/z* 181.0541.



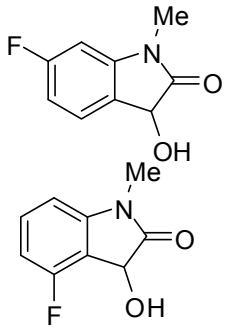

**5-Chloro-3-hydroxy-1-methylindolin-2-one (2q):** The title compound was obtained as a white powder in 82% yield. **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.44 (s, 1H), 7.32 (d, *J* = 8.3 Hz, 1H), 6.75 (d, *J* = 8.3 Hz, 1H), 5.05 (s, 1H), 3.18 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 175.66, 142.59, 130.68, 128.79, 126.29, 124.54, 109.94, 68.66, 25.94. **HRMS (EI)** Calcd. for $C_9H_8ClNO_2$: $[M]^+$, 197.0244. Found: *m/z* 197.0241.



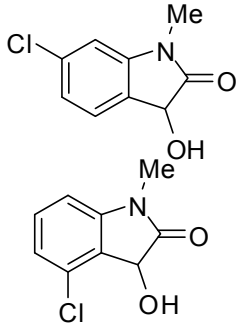

**5-Bromo-3-hydroxy-1-methylindolin-2-one (2r):** The title compound was obtained as a white powder in 96% yield. **$^1$H NMR (500 MHz, $CDCl_3$)** δ 7.58 (s, 1H), 7.47 (d, *J* = 7.4 Hz, 1H), 6.71 (d, *J* = 8.3 Hz, 1H), 5.06 (s, 1H), 3.18 (s, 3H). **$^{13}$C NMR (126 MHz, DMSO)** δ 175.47, 142.97, 131.60, 131.03, 127.20, 113.93, 110.41, 68.56, 25.86. **HRMS (EI)** Calcd. for $C_9H_8BrNO_2$: $[M]^+$, 240.9738. Found: *m/z* 240.9741.



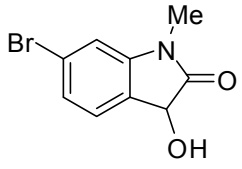
**3-Hydroxy-1-methyl-5-nitroindolin-2-one (2s):** The title compound was obtained through recrystallization as a pale yellow powder in 56% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.41 – 8.24 (m, 2H), 6.93 (d, *J* = 8.6 Hz, 1H), 5.14 (s, 1H), 3.27 (s, 3H), 3.12 (s, 1H). **$^{13}$C NMR (101 MHz, DMSO)** δ 176.48, 149.82, 142.51, 129.69, 126.37, 119.67, 108.77, 68.20, 26.31. **HRMS (EI)** Calcd. for $C_9H_8N_2O_4$: $[M]^+$, 208.0484. Found: *m/z* 208.0487.



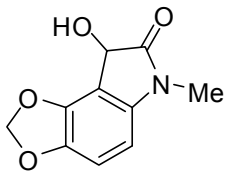
**6-Fluoro-3-hydroxy-1-methylindolin-2-one (2ta), 4-fluoro-3-hydroxy-1-methylindolin-2-one (2tb):** The title compounds were obtained as a white solid of inseparable mixture (1:0.2 by $^1$H NMR) in 76% yield. The $^{13}$C NMR data listed here represent peak information only for **2ta**. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.41 (ddd, *J* = 8.1, 5.4, 0.9 Hz, 1H × 0.2), 7.33 (ddd, *J* = 13.4, 6.7, 3.0 Hz, 1H), 6.84 – 6.75 (m, 1H + 1H × 0.2), 6.65 (d, *J* = 7.8 Hz, 1H), 6.58 (dd, *J* = 8.7, 2.3 Hz, 1H × 0.2), 5.25 (s, 1H), 5.05 (s, 1H × 0.2), 3.20 (s, 3H), 3.18 (s, 3H × 0.2). **$^{13}$C NMR (101 MHz, DMSO)** δ 175.34, 158.85 (d, *J* = 247.5 Hz), 146.09 (d, *J* = 9.3 Hz), 131.41 (d, *J* = 8.9 Hz), 113.92 (d, *J* = 21.0 Hz), 109.78 (d, *J* = 20.8 Hz), 105.01 (d, *J* = 2.7 Hz), 67.07, 26.23. **HRMS (EI)** Calcd. for $C_9H_8FNO_2$: $[M]^+$, 181.0539. Found: *m/z* 181.0540.



**6-Chloro-3-hydroxy-1-methylindolin-2-one (2ua) , 4-chloro-3-hydroxy-1-methylindolin-2-one (2ub):** The title compounds were obtained as a white solid of inseparable mixture (1:0.1 by $^1$H NMR) in 85% yield. The $^{13}$C NMR data listed here represent peak information only for **2ua**. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.37 (d, *J* = 8.2 Hz, 1H), 7.29 (s, 1H × 0.1), 7.09 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 1H × 0.1), 6.84 (d, *J* = 1.7 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H × 0.1), 5.15 (s, 1H × 0.1), 5.03 (s, 1H), 3.20 (s, 3H × 0.1), 3.18 (s, 3H). **$^{13}$C NMR (126 MHz, DMSO)** δ 176.03, 145.24, 133.49, 127.39, 125.69, 121.65, 108.88, 68.27, 25.93. **HRMS (EI)** Calcd. for $C_9H_8ClNO_2$: $[M]^+$, 197.0244. Found: *m/z* 197.0239.



**6-Bromo-3-hydroxy-1-methylindolin-2-one (2va):** The title compound was obtained as a pale yellow powder in 81% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.32 (d, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 1.6 Hz, 1H), 6.99 (d, *J* = 1.5 Hz, 1H), 5.01 (s, 1H), 3.18 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 175.98, 145.37, 127.88, 126.11, 124.67, 121.83, 111.63, 68.36, 25.98. **HRMS (EI)** Calcd. for $C_9H_8BrNO_2$: $[M]^+$, 240.9738. Found: *m/z* 240.9739.




**8-Hydroxy-6-methyl-6*H*-[1,3]dioxolo[4,5-e]indol-7(8*H*)-one (2w):** The title compound was obtained through recrystallization as a pale yellow powder in 45% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 6.75 (d, *J* = 8.0 Hz, 1H), 6.24 (d, *J* = 8.0 Hz, 1H), 6.03 (d, *J* = 4.9 Hz, 2H), 5.17 (s, 1H), 3.16 (s, 3H). **$^{13}$C NMR (101 MHz, DMSO)** δ 174.86, 144.12, 143.86, 138.91, 109.10, 107.40, 101.54, 99.96, 67.05, 26.19. **HRMS (EI)** Calcd. for $C_{10}H_9NO_4$: $[M]^+$, 207.0532. Found: *m/z* 207.0533.

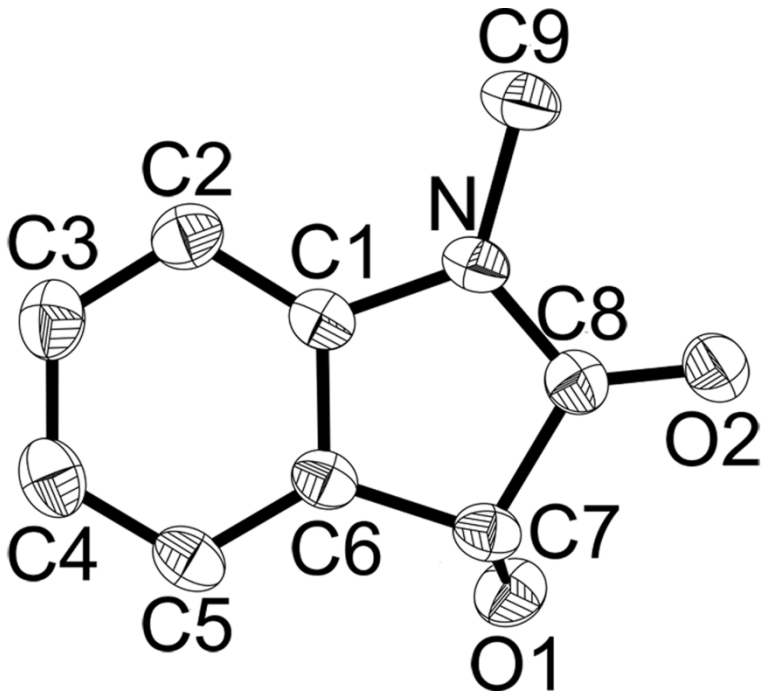
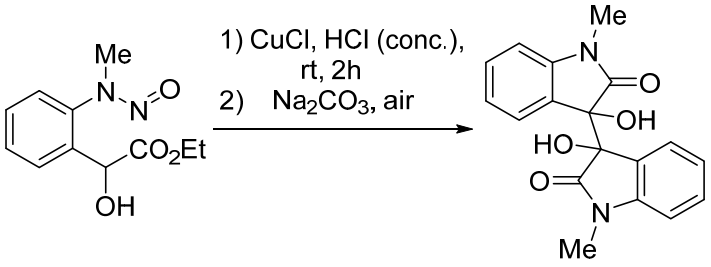



**Figure S1.** ORTEP drawing of **2a** showing 30% probability thermal ellipsoids. Hydrogen atoms are omitted for clarity.

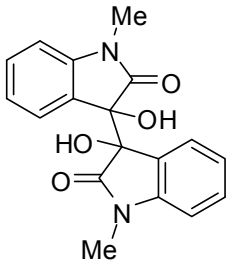
### 3. Synthesis of Dimeric Product of 3-Hydroxy-2-oxindoles

**Procedure for the synthesis of dimeric product:**

To a 5 mL round-bottom flask equipped with magnetic stir bar was charged with **1a** (47.6 mg, 0.2 mmol, 1 equiv). An excess cold conc. HCl solution (2 mL) of CuCl (350 mg, 3.5 mmol, 17.5 equiv) was further added. The mixture was stirred at rt for 2 h and monitored by TLC. The reaction solution was diluted four times by ice water. The mixture was neutralized with saturated $Na_2CO_3$ solution until no bubble could be observed, and then extracted with $CH_2Cl_2$ for at least three times. The organic phase was combined, washed with saturated brine solution, dried over $MgSO_4$, filtered and the solvent was removed in vacuo. The residue was purified by flash column chromatography on silica gel with petroleum ether/EtOAc as the eluent. An orange oil was obtained.




**1a** **2a-dim** 85%

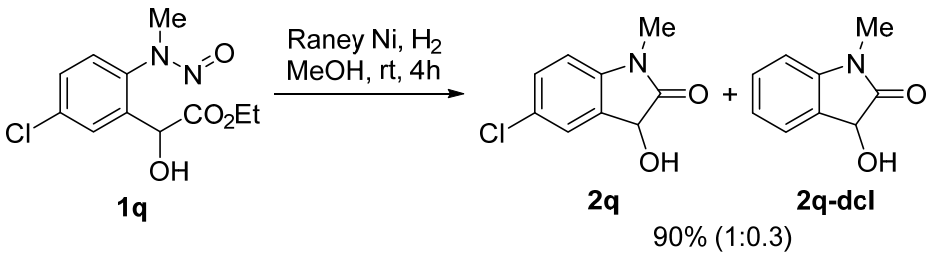



**3,3'-Dihydroxy-1,1'-dimethyl-3,3'-biindoline-2,2'-dione (2a-dim):** The title compound was obtained as an orange oil in 85% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.69 – 7.56 (m, 4H), 7.17 – 7.11 (m, 2H), 6.90 (d, *J* = 8.1 Hz, 2H), 3.26 (s, 6H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 183.53, 158.38, 151.60, 138.62, 125.40, 124.01, 117.55, 110.13, 26.38. **HRMS (MALDI)** Calcd. for $C_{18}H_{16}N_2NaO_4$: $[M + Na]^+$, 347.1002. Found: *m/z* 347.1002.

## 4. Halogen Removal from 3-Hydroxy-2-oxindoles under Raney Ni/$H_2$ Condition

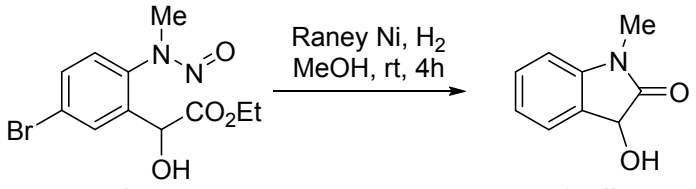
**Procedure for the synthesis of halogen-removed 3-hydroxy-2-oxindoles:**

The procedure has been described in the "General procedure for Synthesis and Characterization of 3-Hydroxy-2-oxindoles under Raney Ni/$H_2$ condition" section. Products **2q** and **2q-dcl** (identical to **2a**) were obtained in a combined yield of 90% (with a ratio of 1:0.3) starting from **2q**. Product **2r-dbr** (identical to **2a**) was obtained in 88% yield.




**1q** **2q** **2q-dcl**

90% (1:0.3)

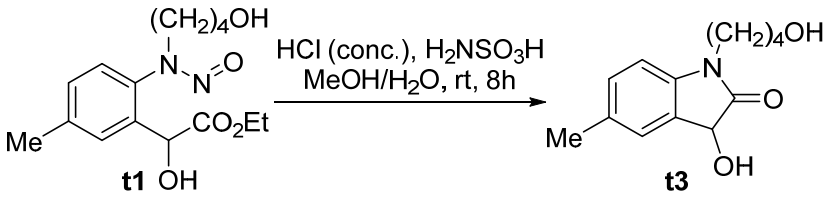


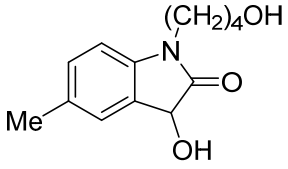

**1r** **2r-dbr**

88%

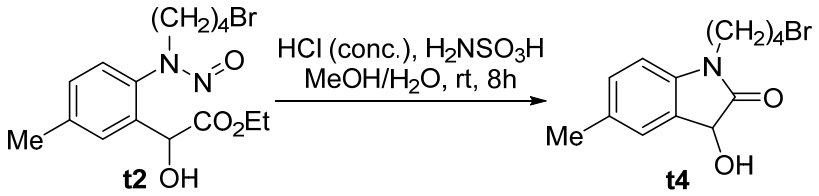
## 5. Synthesis and Characterization of t3 and t4

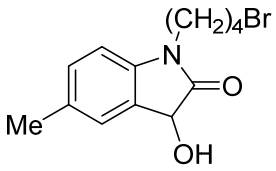
The procedure has been described in the "General procedure for Synthesis and Characterization of 3-Hydroxy-2-oxindoles under $HCl/H_2NSO_3H$ condition" section.




**3-hydroxy-1-(4-hydroxybutyl)-5-methylindolin-2-one (t3):** the title compound was obtained as a yellow oil in 51% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.25 (s, 1H), 7.07 (d, *J* = 7.9 Hz, 1H), 6.71 (d, *J* = 7.9 Hz, 1H), 5.00 (s, 1H), 3.66 (dd, *J* = 12.9, 6.9 Hz, 2H), 3.61 (t, *J* = 6.3 Hz, 2H), 2.30 (s, 3H), 1.73 (m, 2H), 1.56 (m, 2H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.34, 140.44, 132.69, 129.72, 127.60, 126.03, 108.56, 69.80, 61.81, 39.76, 29.51, 23.68, 20.97. **HRMS (DART)** Calcd. for $C_{13}H_{18}NO_3$: $[M + H]^+$, 236.1281. Found: *m/z* 236.1280.



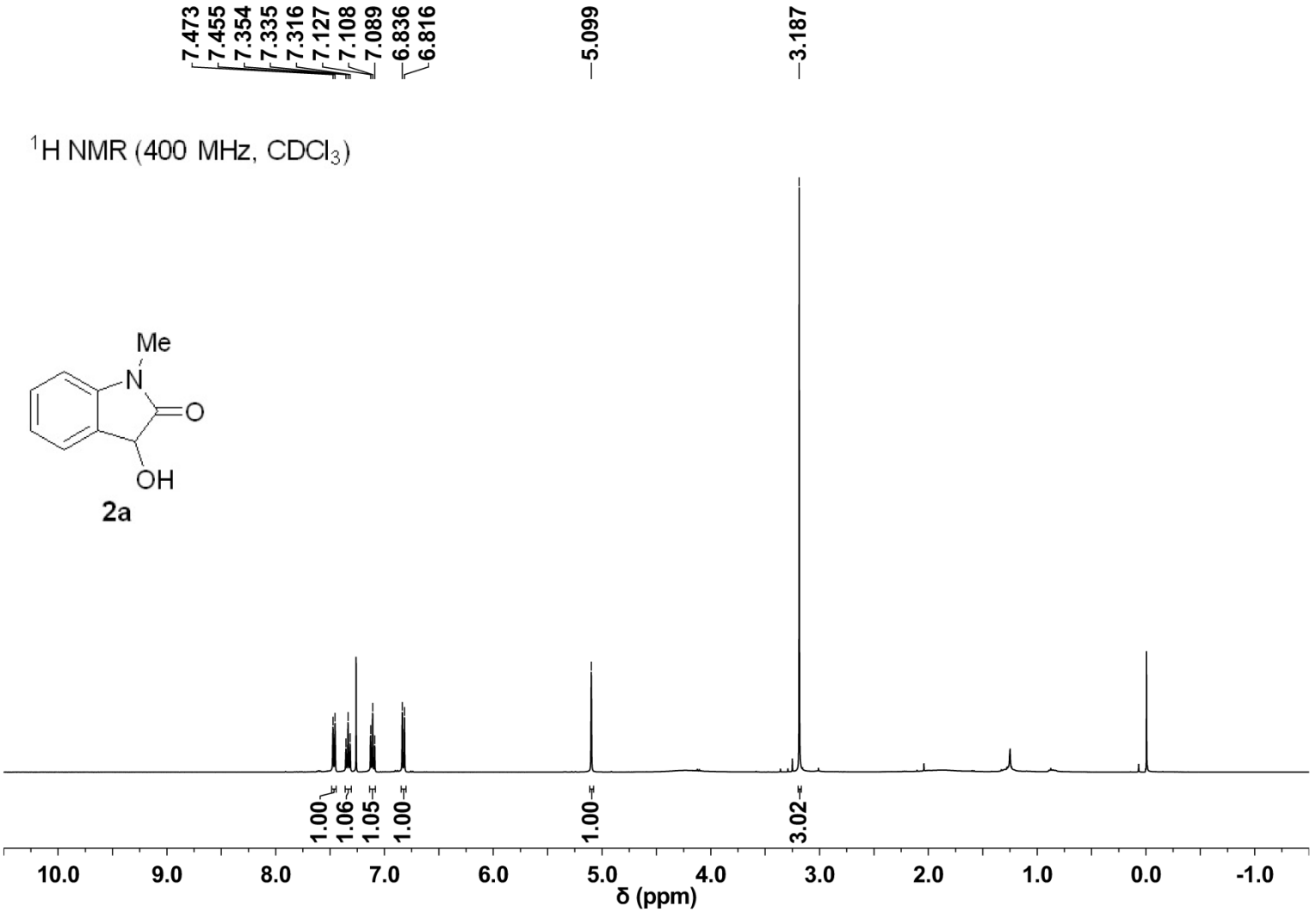



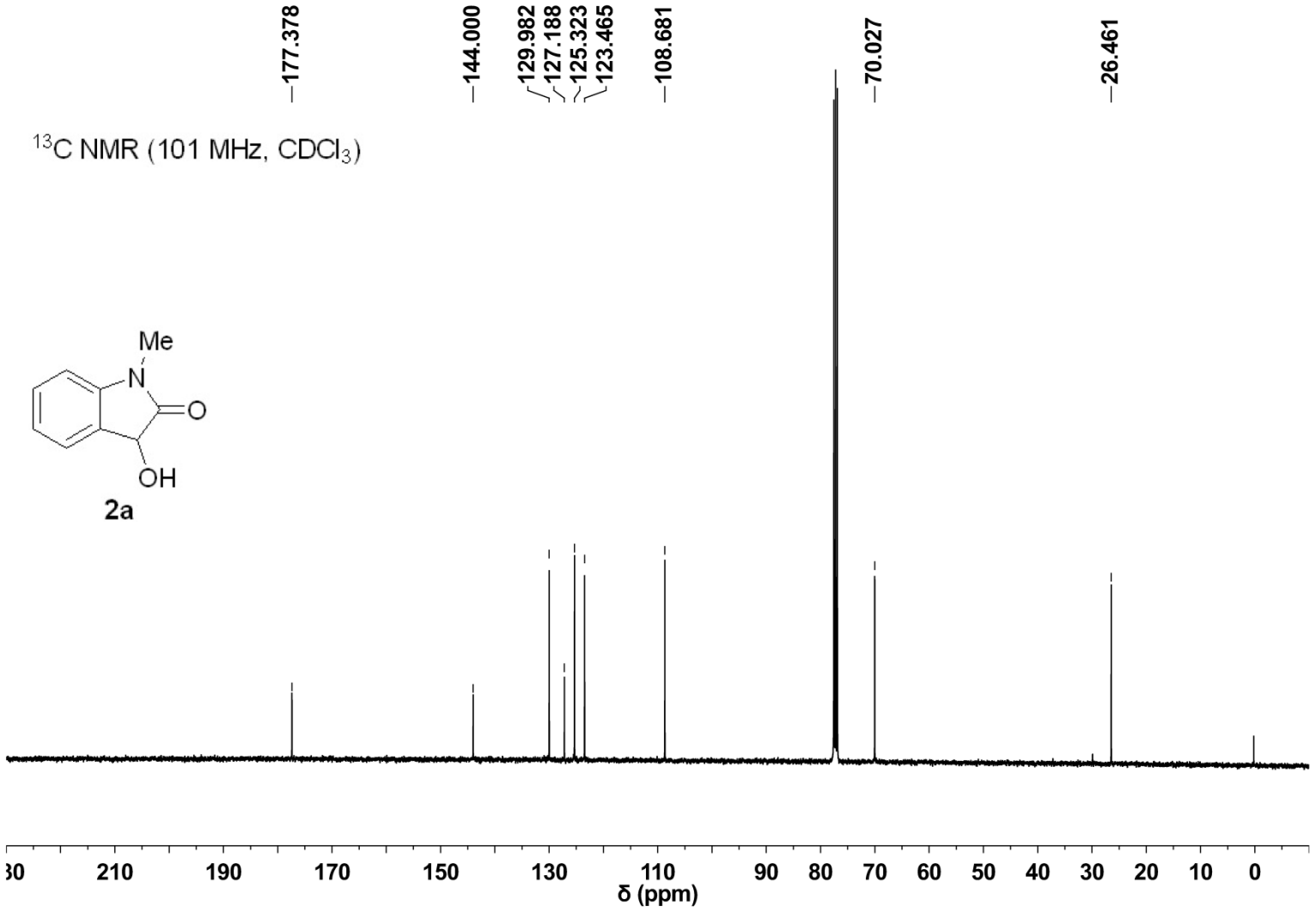

**1-(4-bromobutyl)-3-hydroxy-5-methylindolin-2-one (t4):** the title compound was obtained as an orange solid in 64% yield. **$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.30 (s, 1H), 7.12 (d, *J* = 7.9 Hz, 1H), 6.74 (d, *J* = 8.0 Hz, 1H), 5.47 – 4.81 (m, 1H), 4.31 (s, 1H), 3.80 – 3.58 (m, 2H), 3.55 – 3.39 (m, 2H), 2.32 (d, *J* = 12.0 Hz, 3H), 1.96 – 1.68 (m, 4H). **$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 177.15, 140.47, 132.91, 129.93, 127.15, 126.23, 108.43, 69.82, 39.06, 33.00, 29.64, 25.80, 21.01. **HRMS (DART)** Calcd. for $C_{13}H_{17}NO_2Br$: $[M + H]^+$, 298.0437. Found: *m/z* 298.0435.

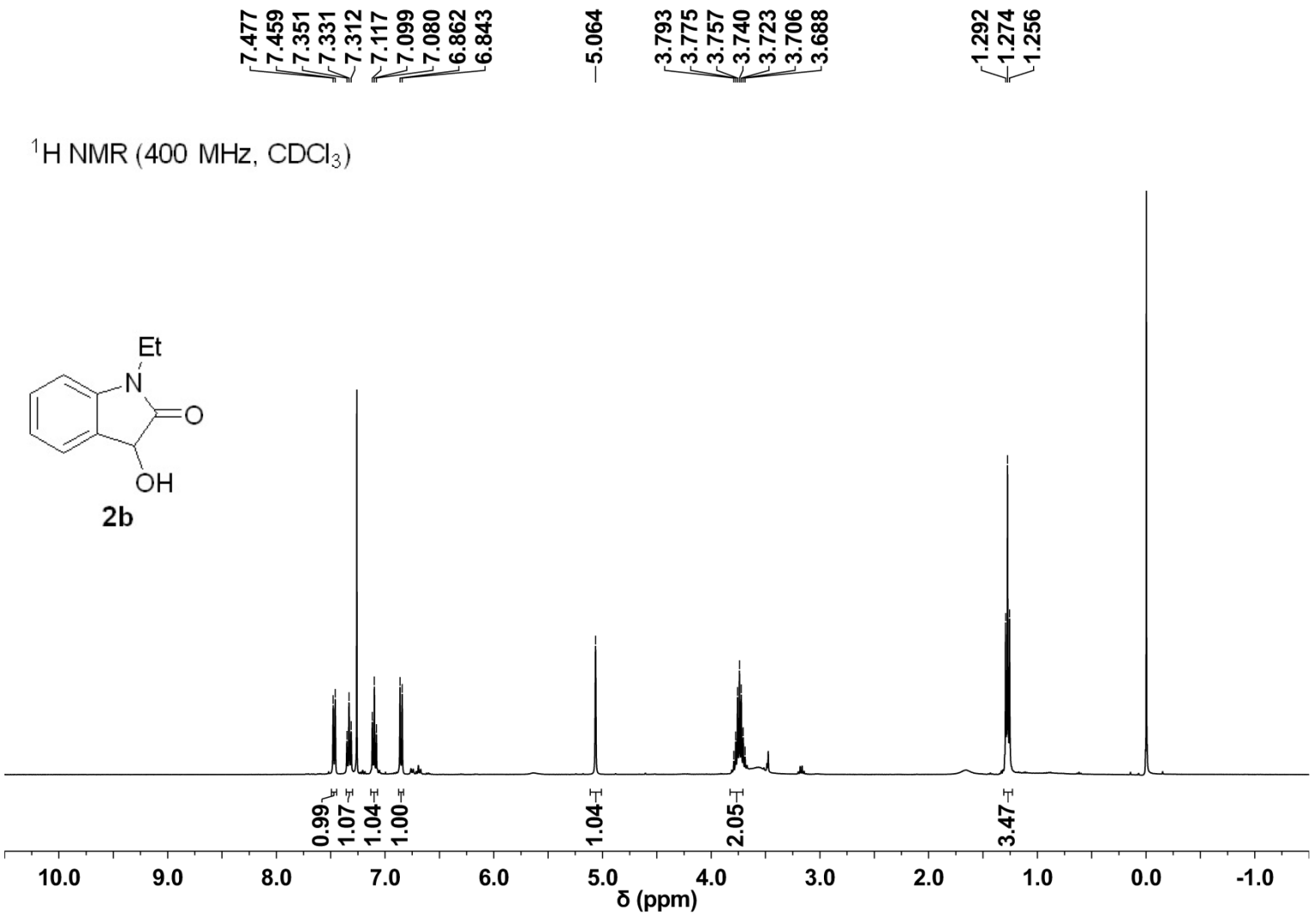
## 6. Reference

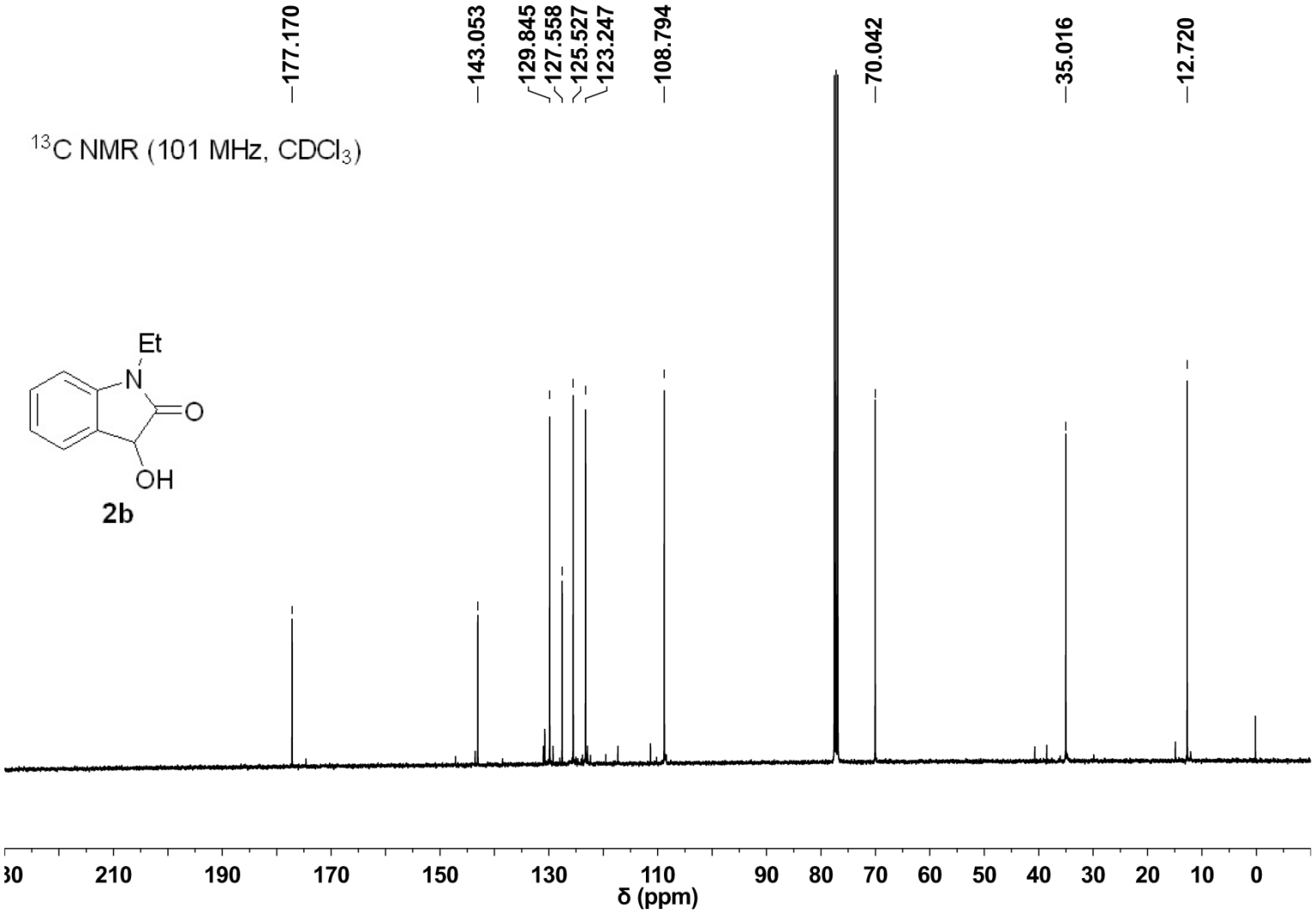
S1 J. Chen, P. Chen, C. Song and J. Zhu, *Chem. - Eur. J.*, 2014, In Press.

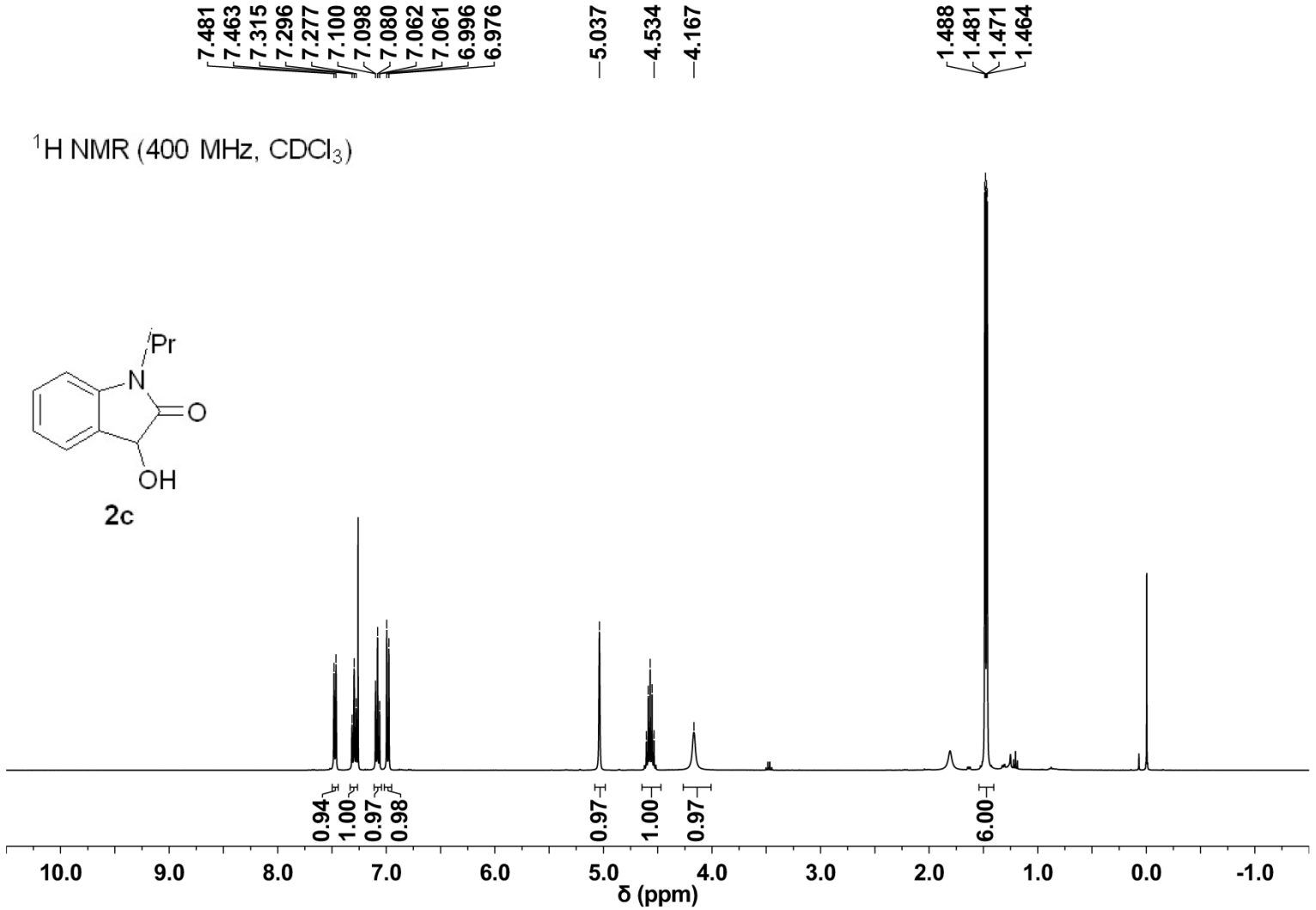
## 7. $^{1}$H and $^{13}$C NMR Spectra for the Compounds

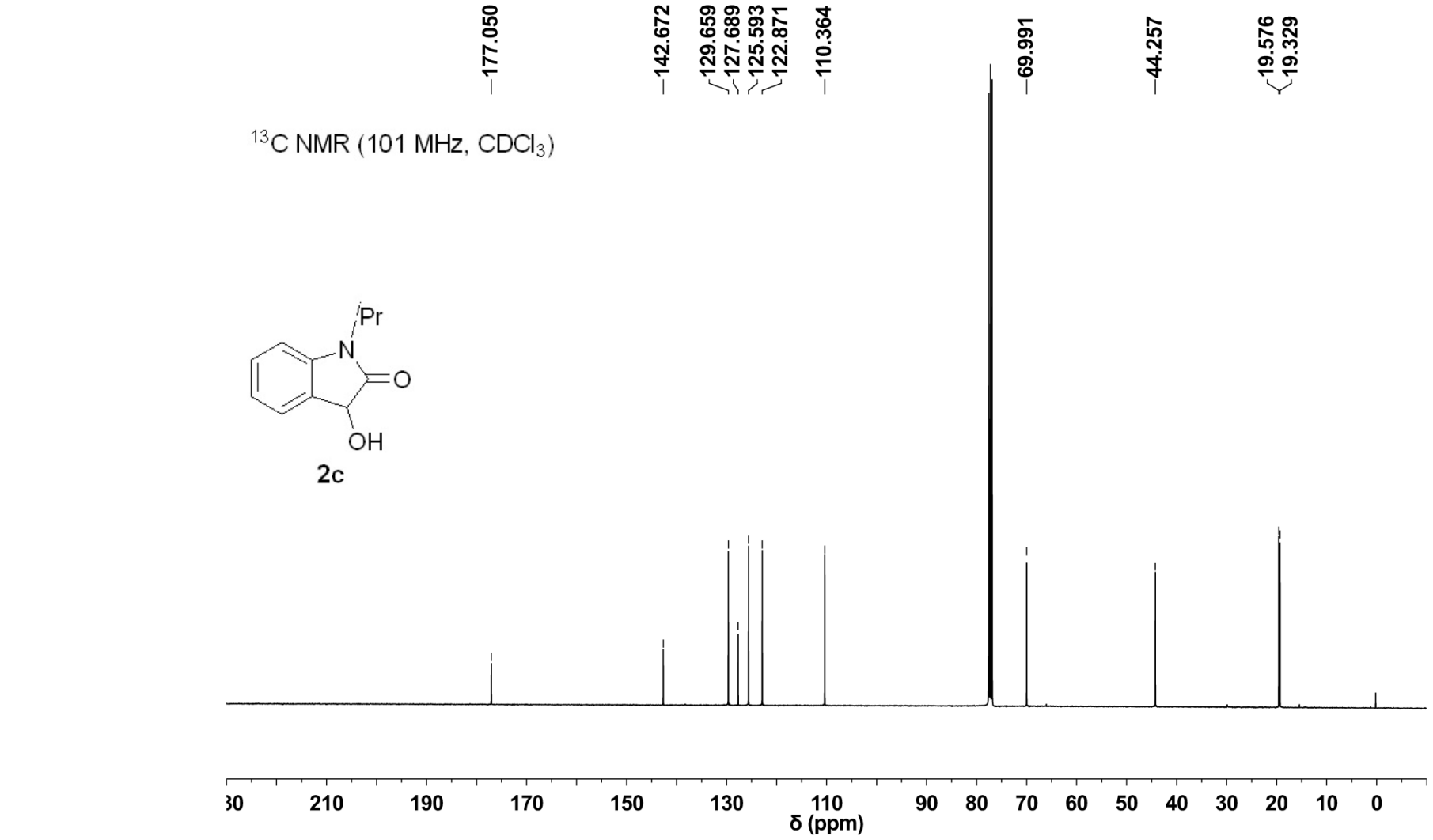

7.473
7.455
7.354
7.335
7.316
7.127
7.108
7.089
6.836
6.816
5.099
3.187
$^1$H NMR (400 MHz, $CDCl_3$)
Me
N
O
OH
2a
1.00
1.06
1.05
1.00
1.00
3.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

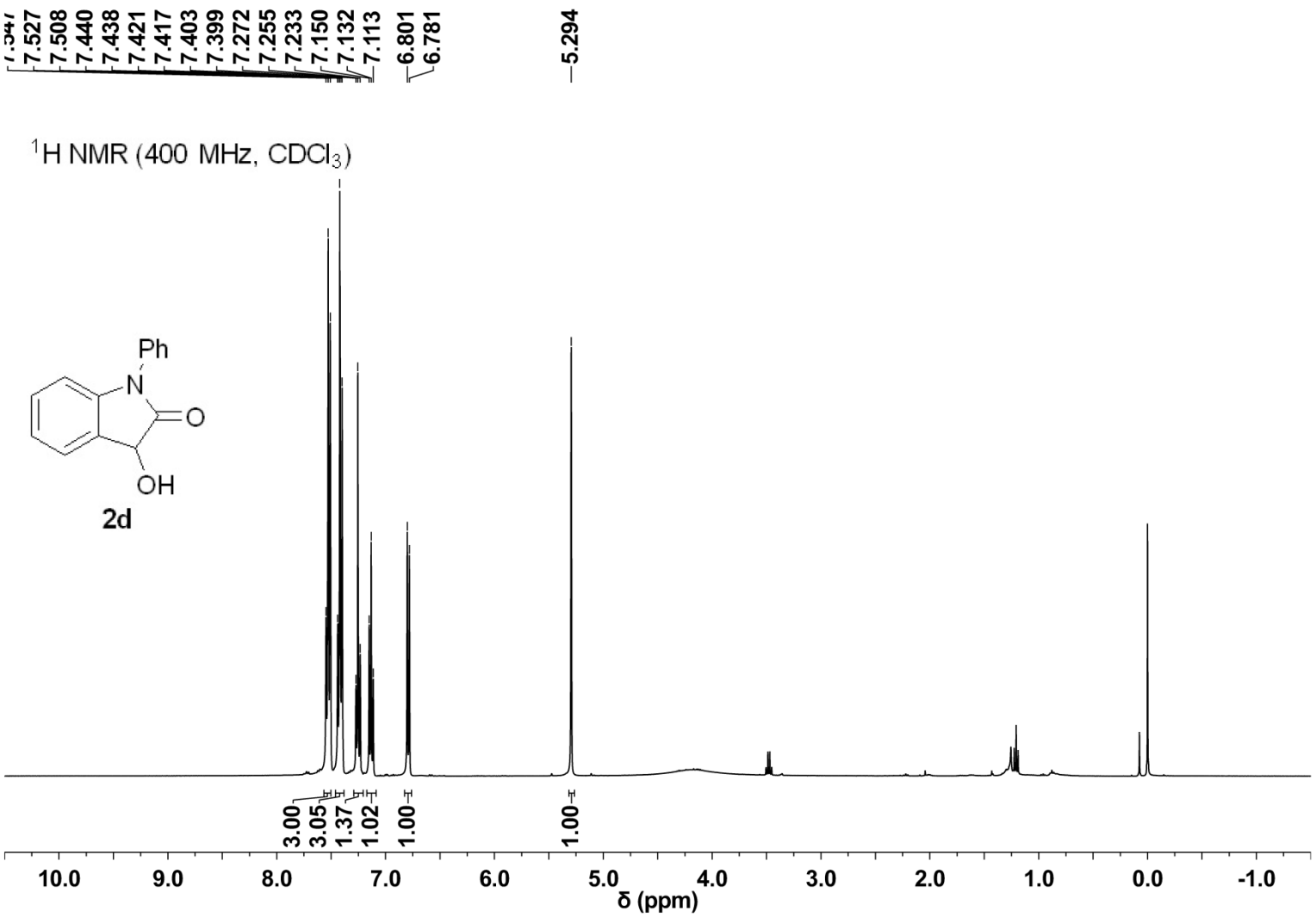

177.378
144.000
129.982
127.188
125.323
123.465
108.681
70.027
26.461
$^{13}$C NMR (101 MHz, $CDCl_3$)
Me
N
O
OH
2a
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

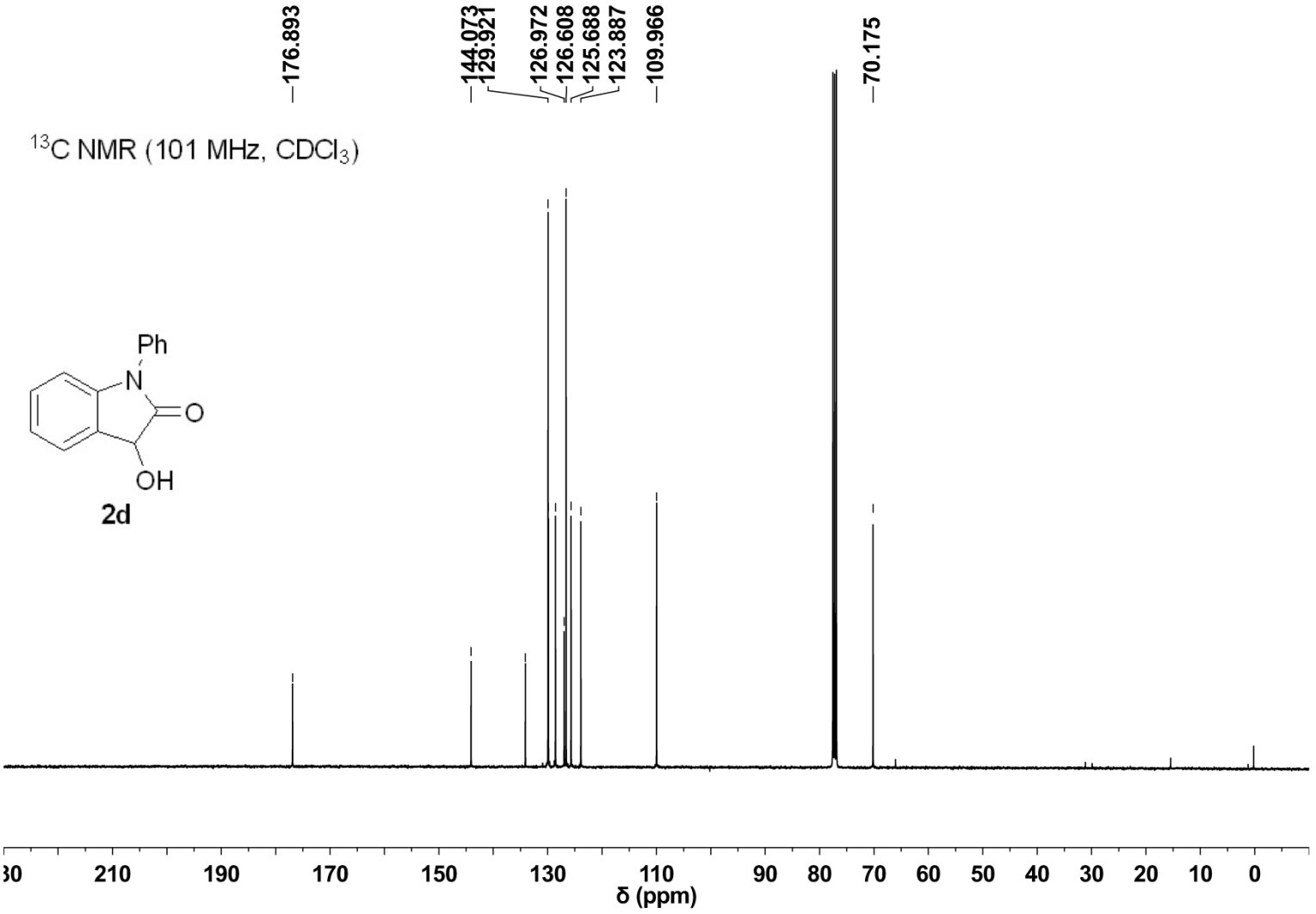

7.477
7.459
7.351
7.331
7.312
7.117
7.099
7.080
6.862
6.843
5.064
3.793
3.775
3.757
3.740
3.723
3.706
3.688
1.292
1.274
1.256
$^1$H NMR (400 MHz, CDCl$_3$)
Et
N
O
OH
2b
0.99
1.07
1.04
1.00
1.04
2.05
3.47
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

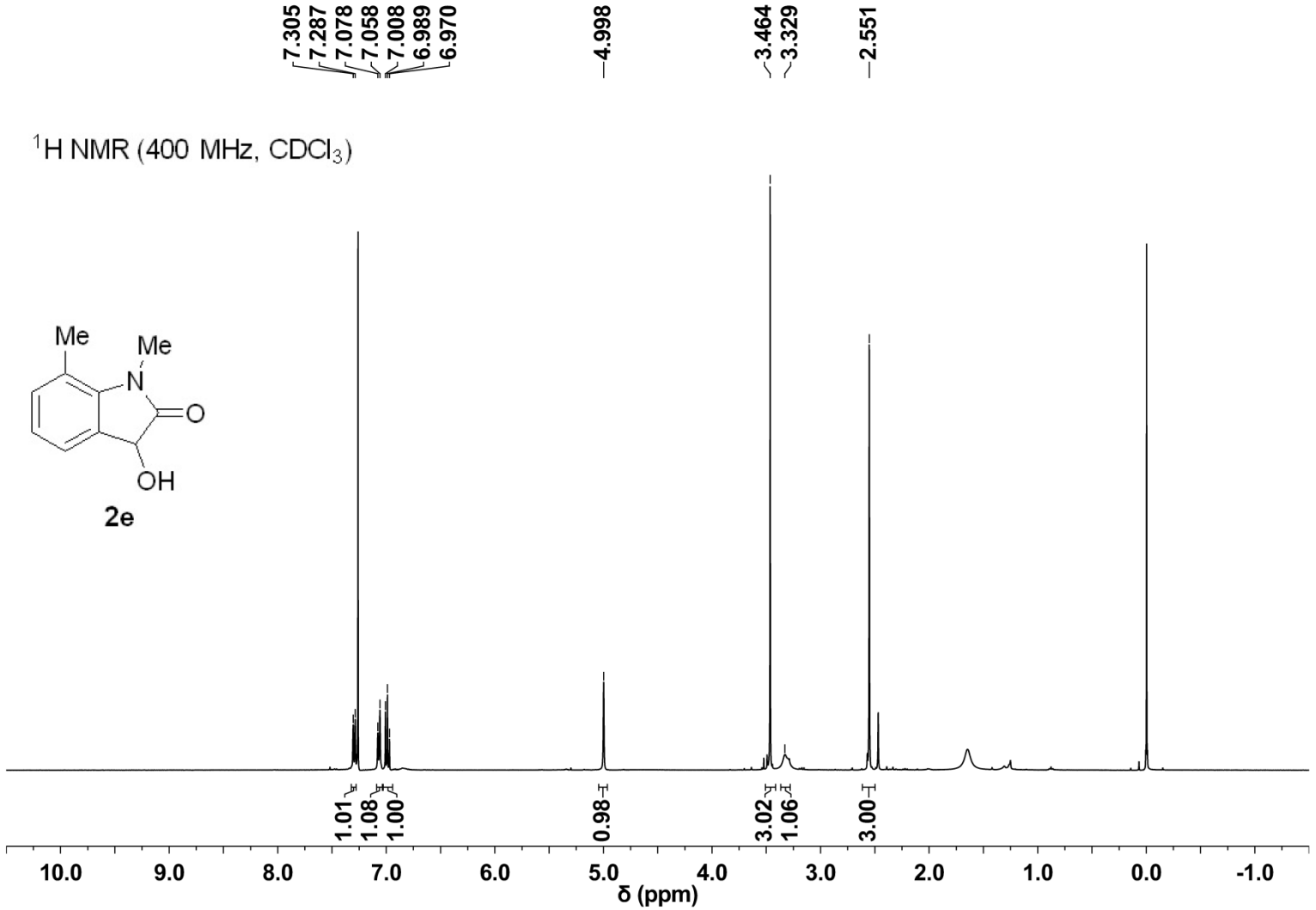

177.170
143.053
129.845
127.558
125.527
123.247
108.794
70.042
35.016
12.720
$^{13}$C NMR (101 MHz, $CDCl_3$)
Et
N
O
OH
2b
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

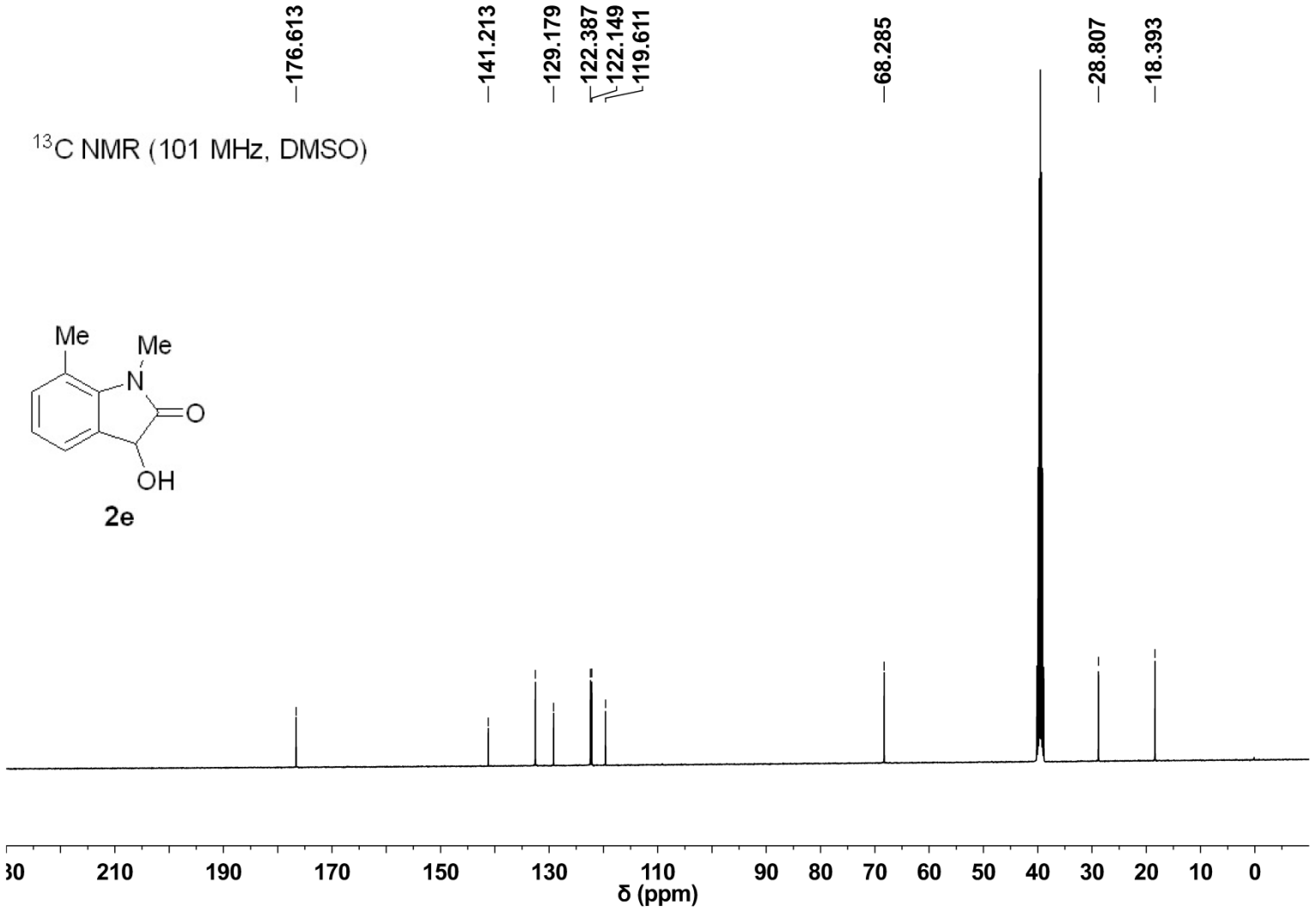

7.481
7.463
7.315
7.296
7.277
7.100
7.098
7.080
7.062
7.061
6.996
6.976
5.037
4.534
4.167
1.488
1.481
1.471
1.464
$^1$H NMR (400 MHz, CDCl$_3$)
Pr
N
O
OH
2c
0.94
1.00
0.97
0.98
0.97
1.00
0.97
6.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

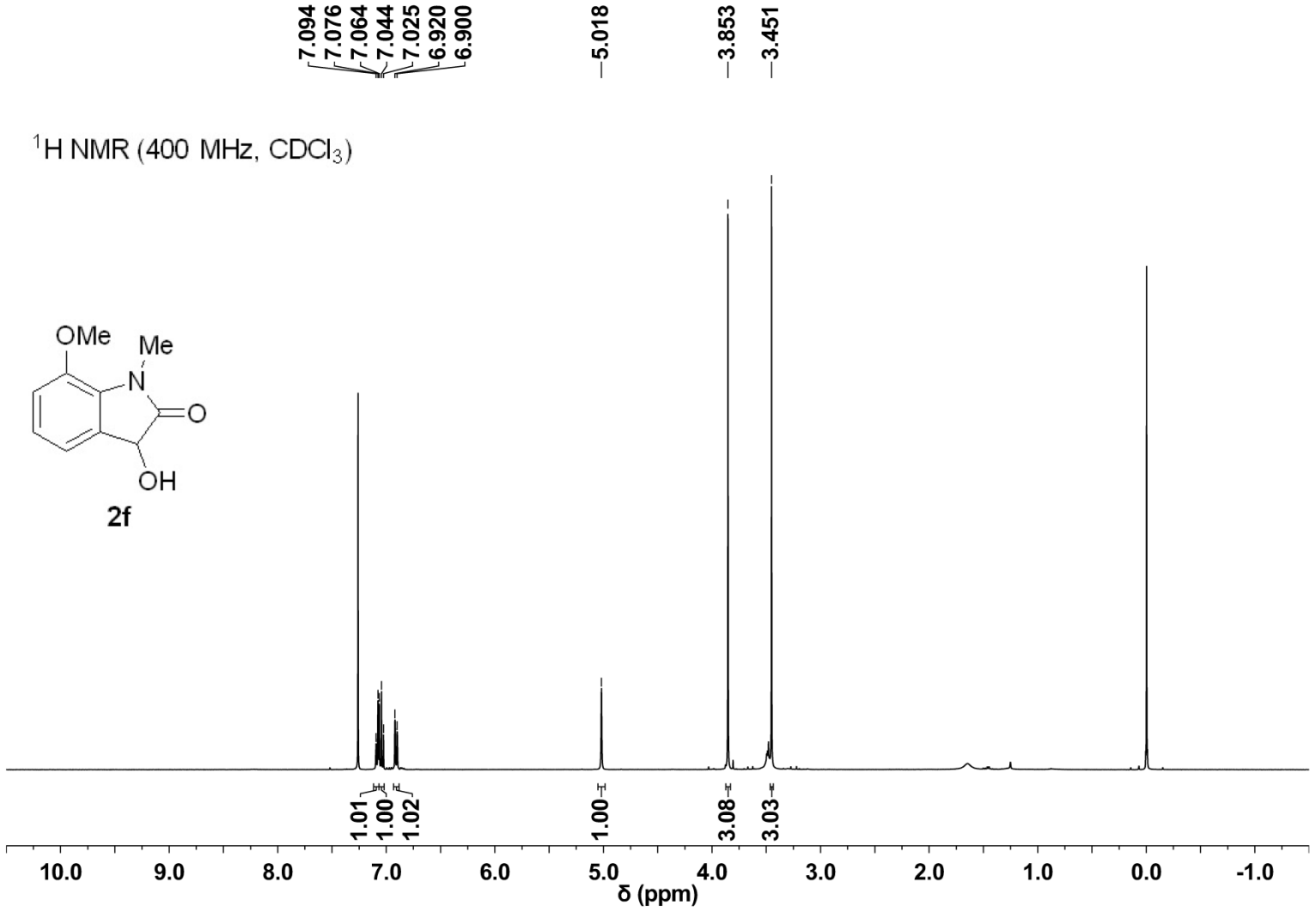

177.050
142.672
129.659
127.689
125.593
122.871
110.364
69.991
44.257
19.576
19.329
13C NMR (101 MHz, CDCl3)
Pr
N
O
OH
2c
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

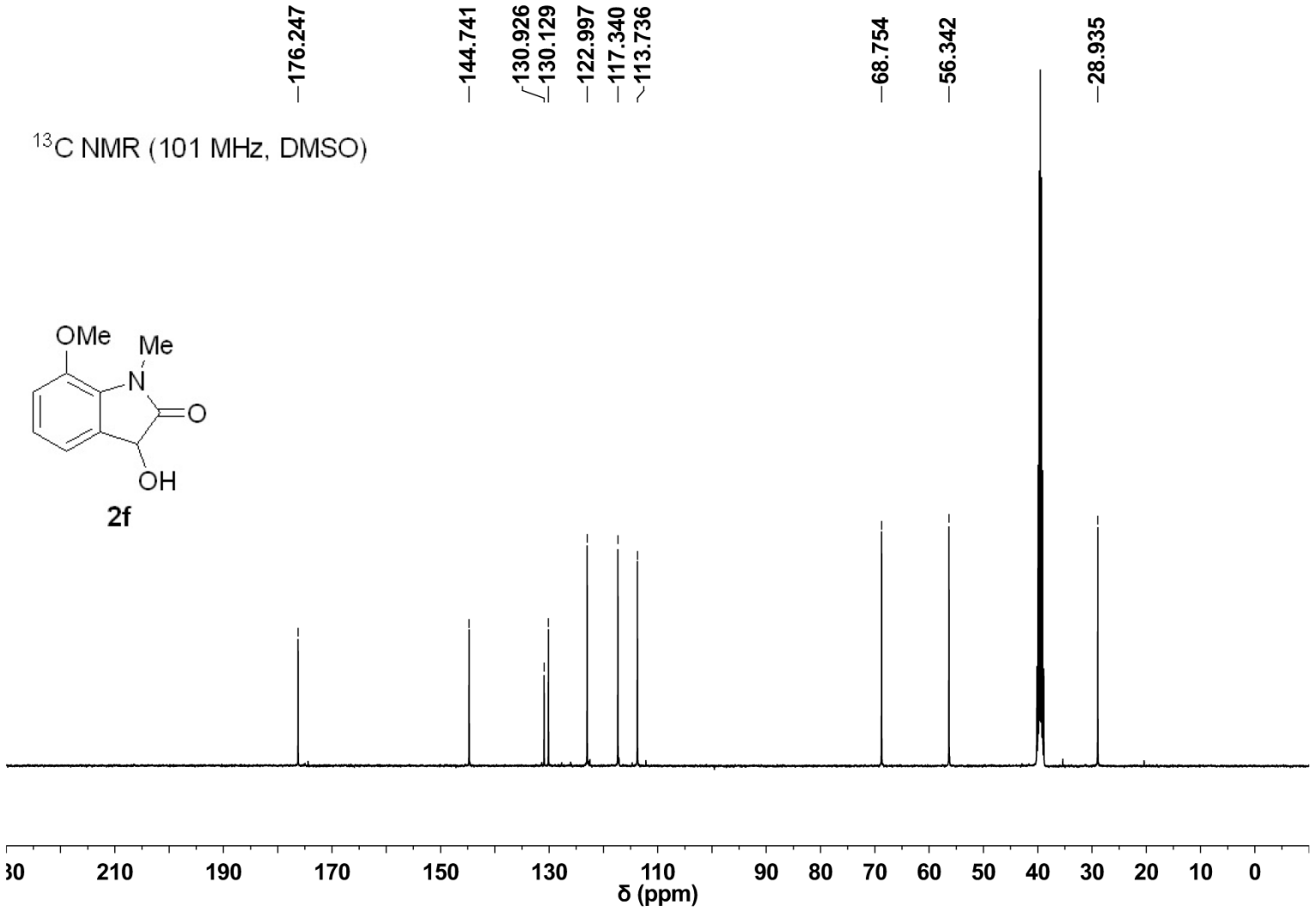

7.527
7.508
7.440
7.438
7.421
7.417
7.403
7.399
7.272
7.255
7.233
7.150
7.132
7.113
6.801
6.781
5.294
$^1$H NMR (400 MHz, CDCl$_3$)
Ph
N
O
OH
2d
3.00
3.05
1.37
1.02
1.00
1.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

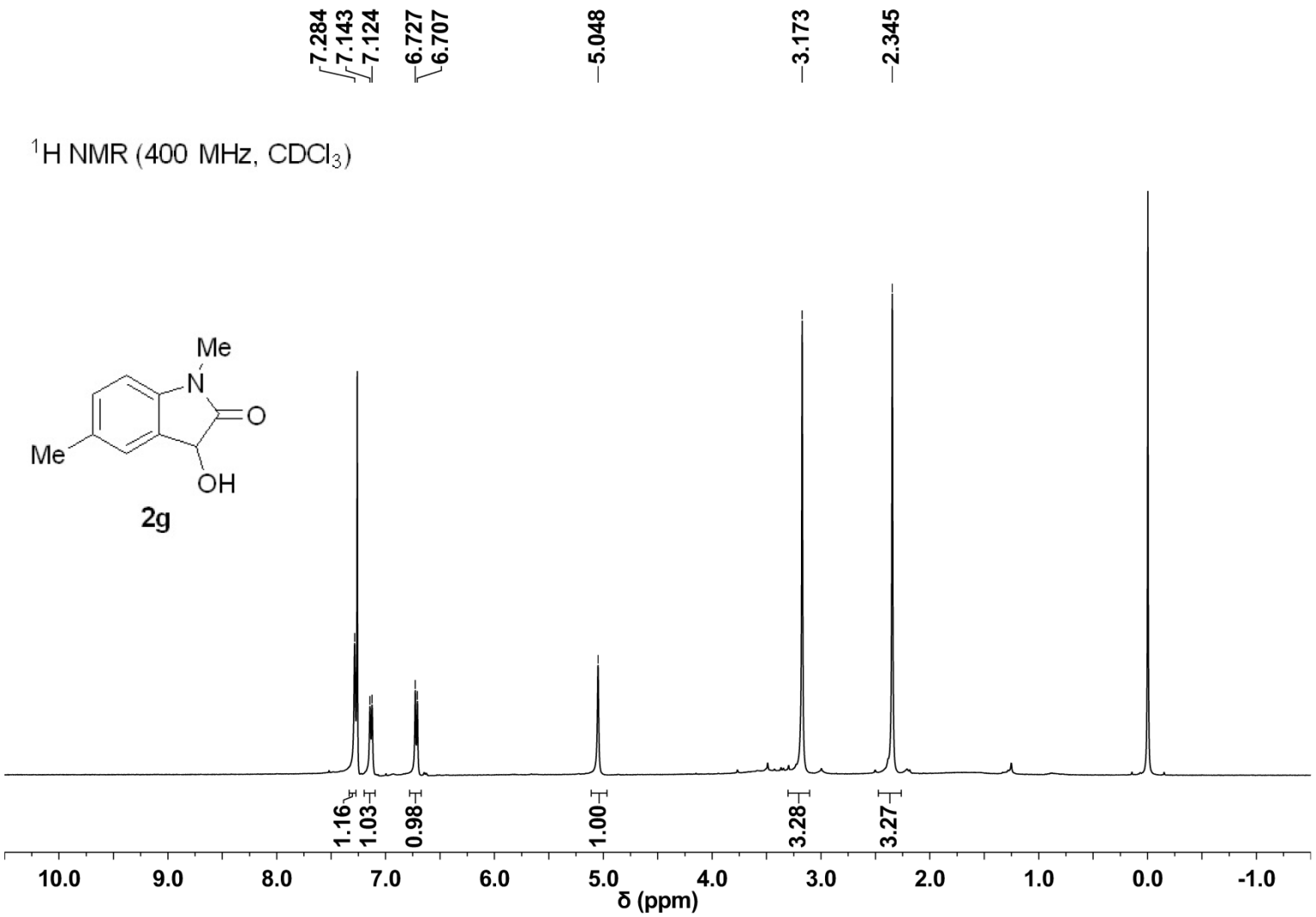

176.893
144.073
139.921
126.972
126.608
125.688
123.887
109.966
70.175
$^{13}$C NMR (101 MHz, $CDCl_3$)
Ph
N
O
OH
2d
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

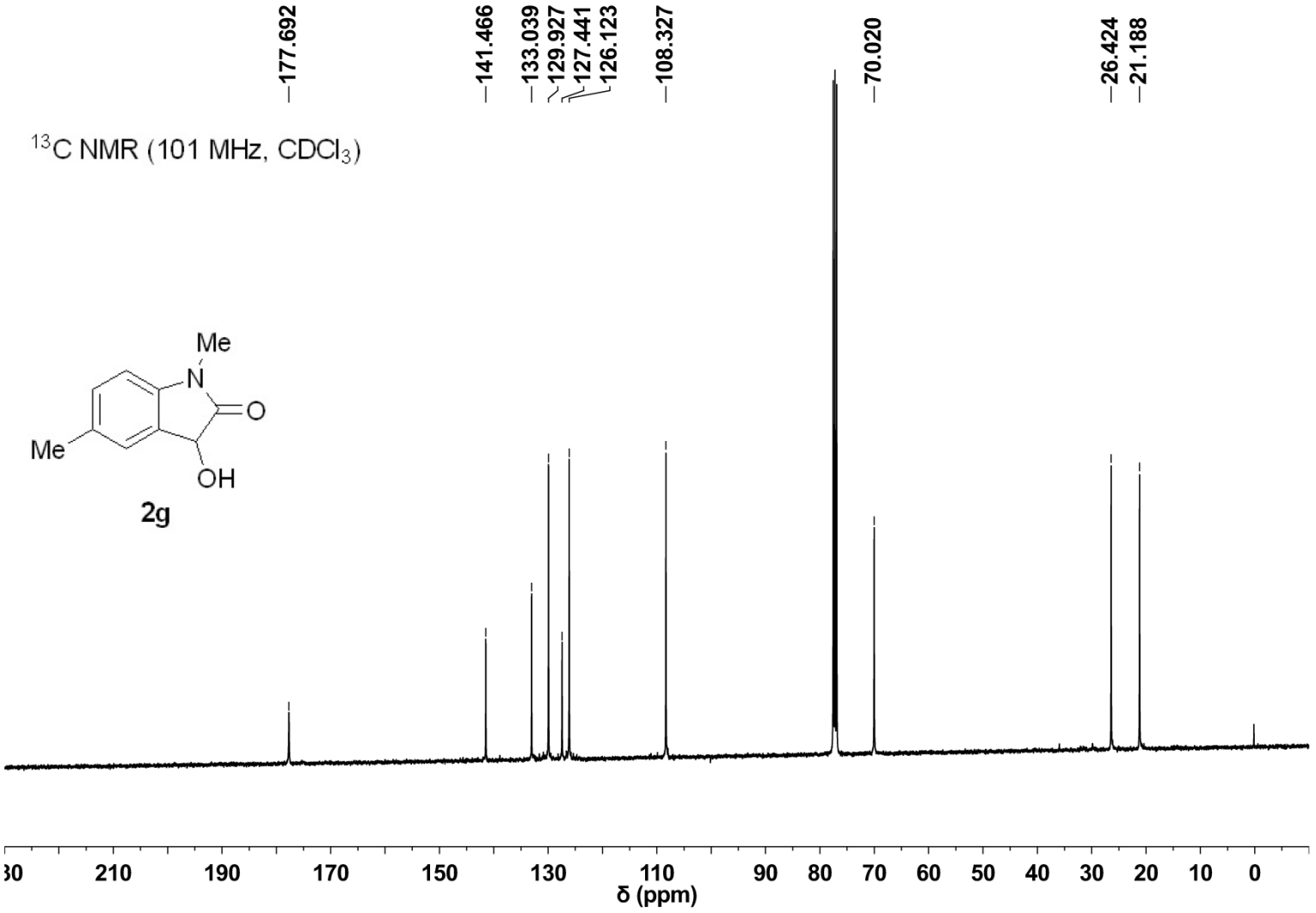

7.305
7.287
7.078
7.058
7.008
6.989
6.970
4.998
3.464
3.329
2.551
$^1$H NMR (400 MHz, $CDCl_3$)
Me
Me
N
O
OH
2e
1.01
1.08
1.00
0.98
3.02
1.06
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

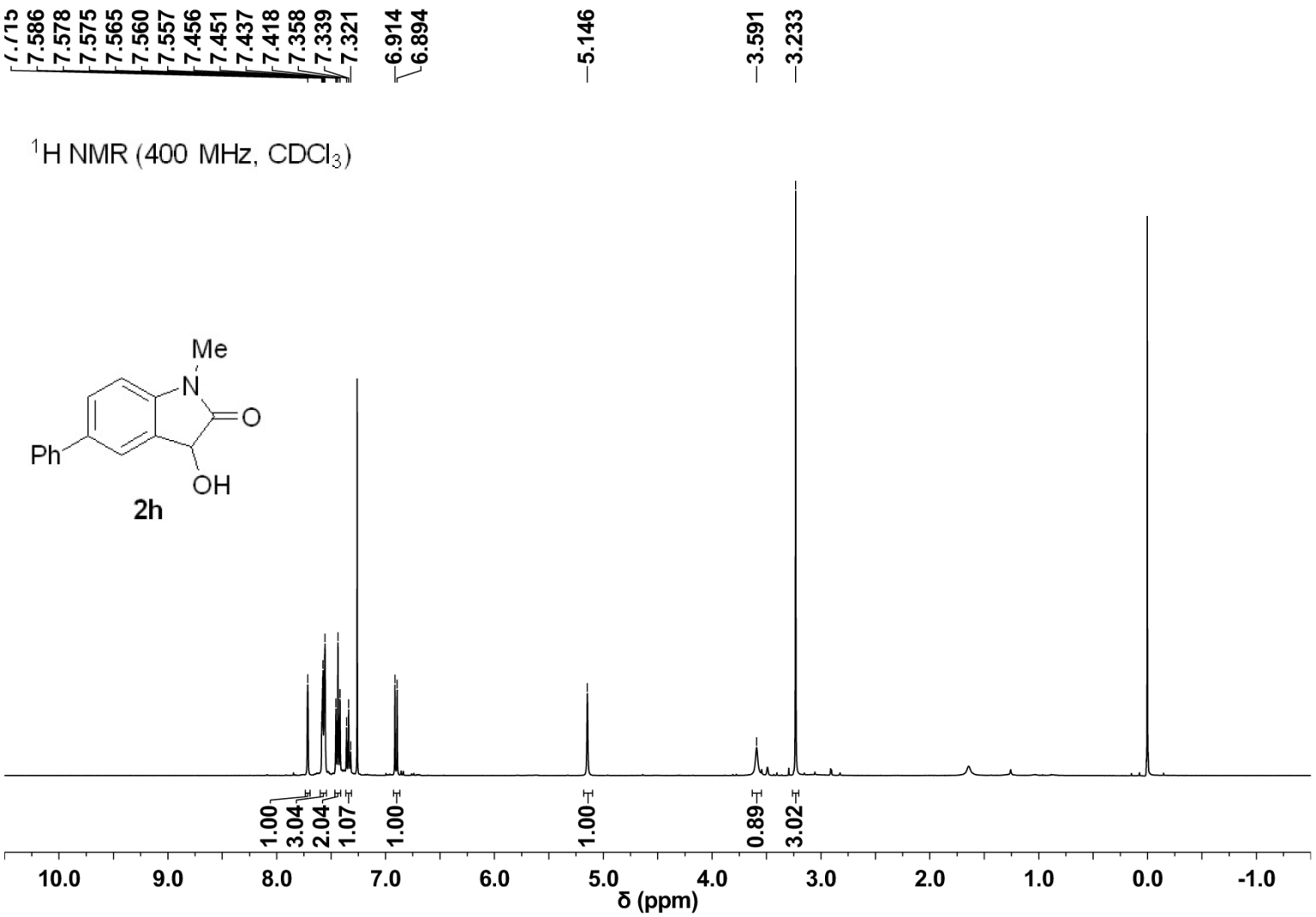

176.613
141.213
129.179
122.387
122.149
119.611
68.285
28.807
18.393
$^{13}$C NMR (101 MHz, DMSO)
Me
Me
N
O
OH
2e
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

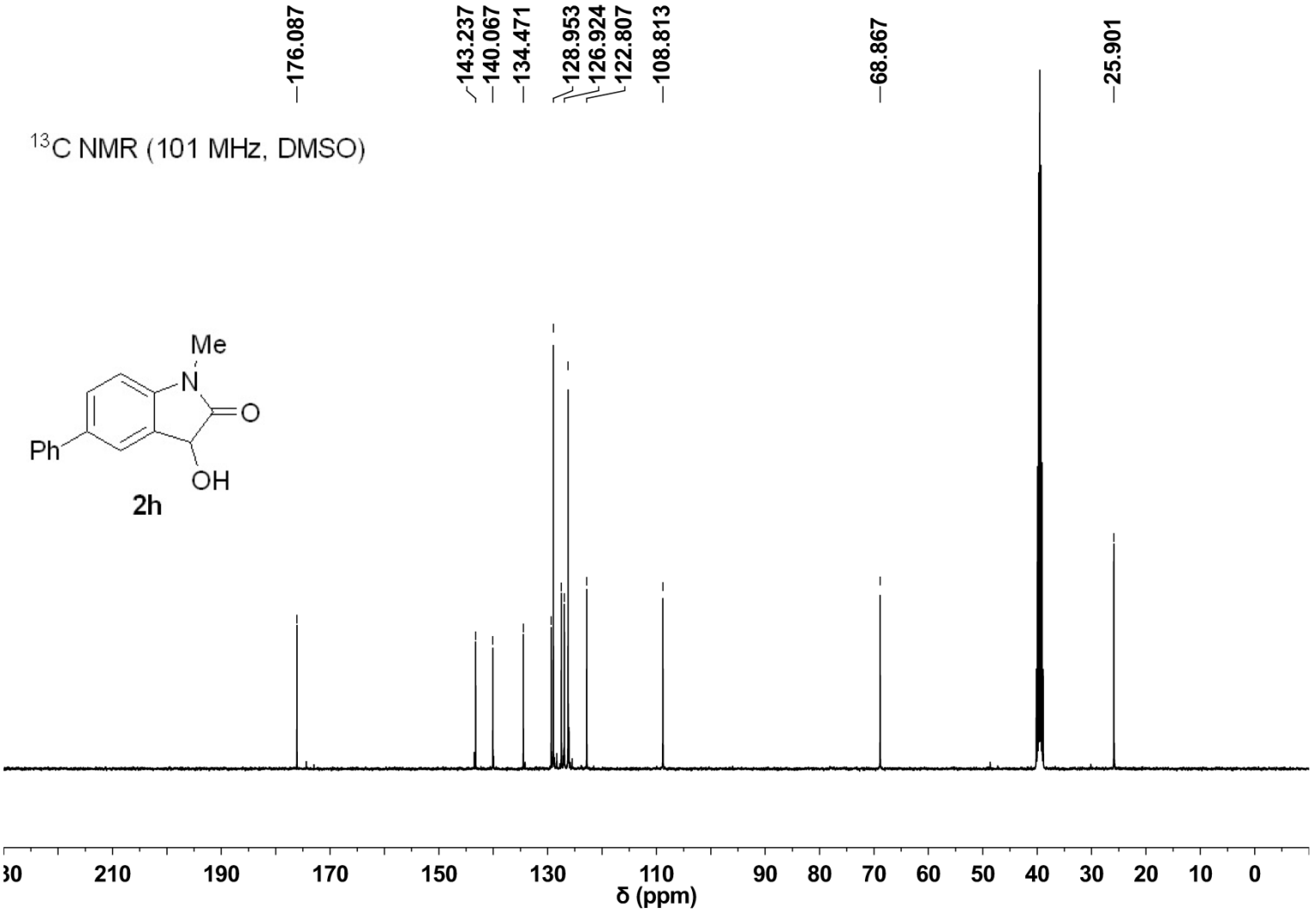

7.094
7.076
7.064
7.044
7.025
6.920
6.900
5.018
3.853
3.451
$^1$H NMR (400 MHz, $CDCl_3$)
OMe
Me
N
O
OH
2f
1.01
1.00
1.02
1.00
3.08
3.03
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

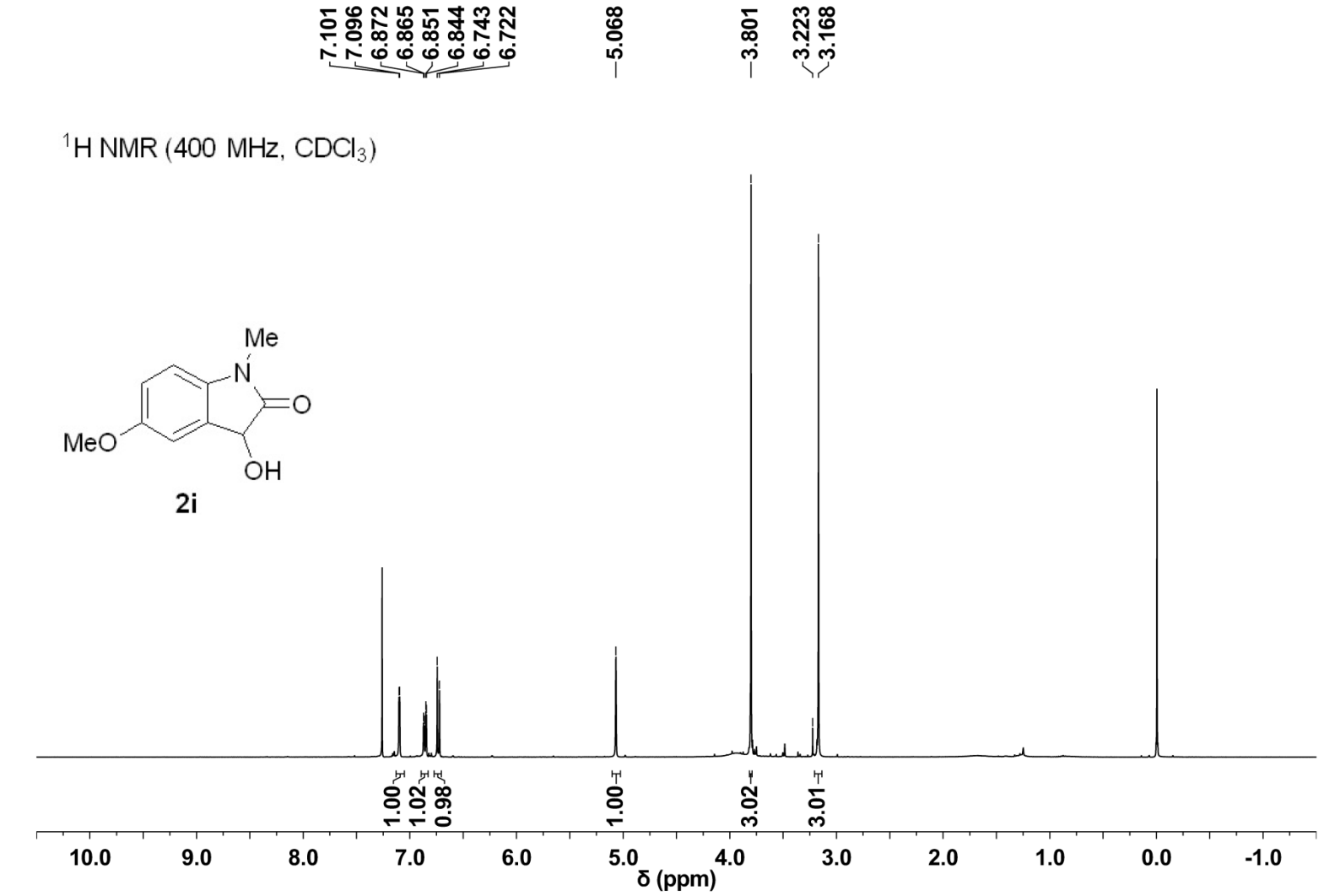

176.247
144.741
130.926
130.129
122.997
117.340
113.736
68.754
56.342
28.935
$^{13}$C NMR (101 MHz, DMSO)
OMe
Me
N
O
OH
2f
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)


7.284
7.143
7.124
6.727
6.707
5.048
3.173
2.345
$^1$H NMR (400 MHz, $CDCl_3$)
Me
N
O
Me
OH
2g
1.16
1.03
0.98
1.00
3.28
3.27
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

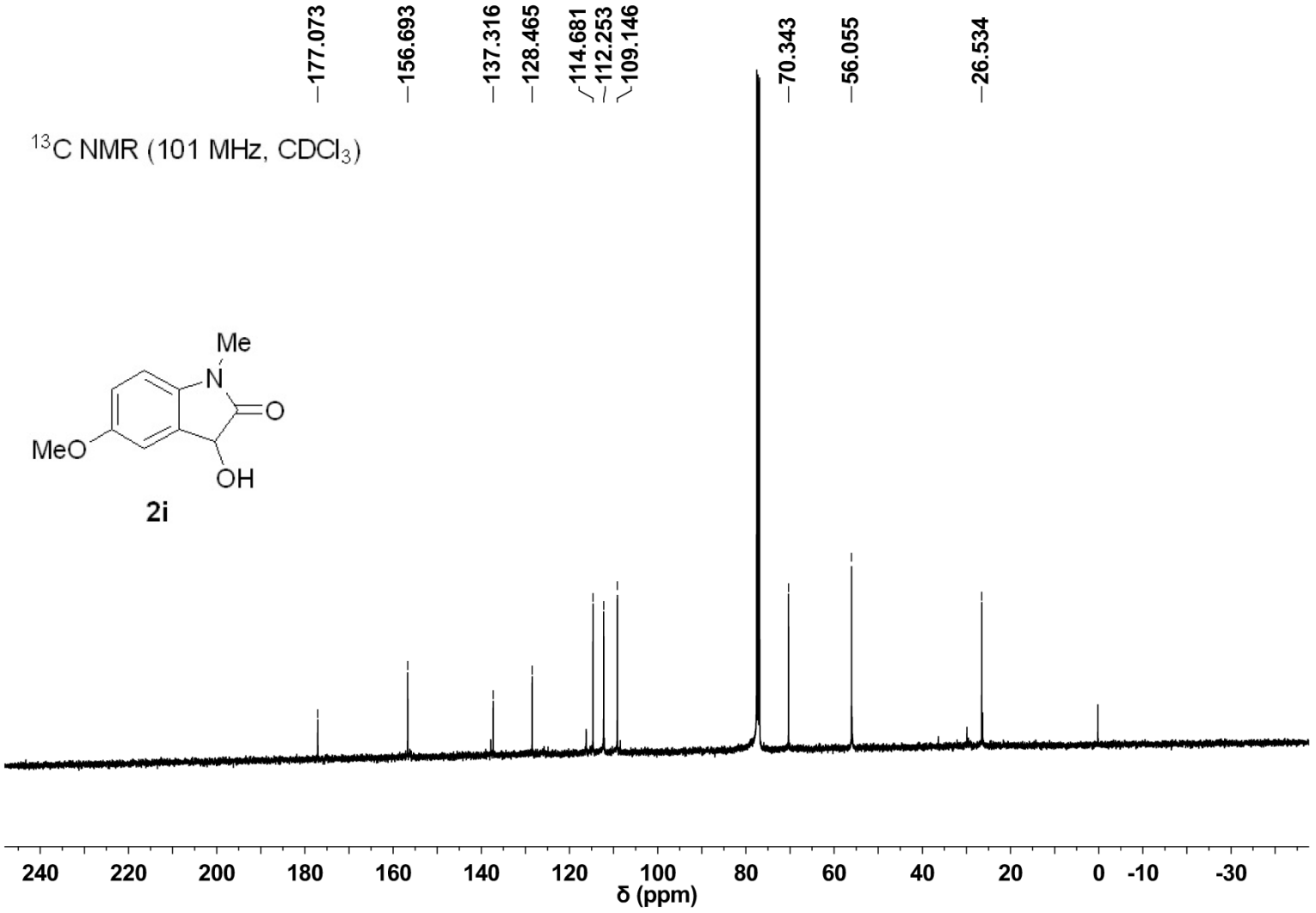

177.692
141.466
133.039
129.927
127.441
126.123
108.327
70.020
26.424
21.188
13C NMR (101 MHz, CDCl3)
Me
N
O
Me
OH
2g
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

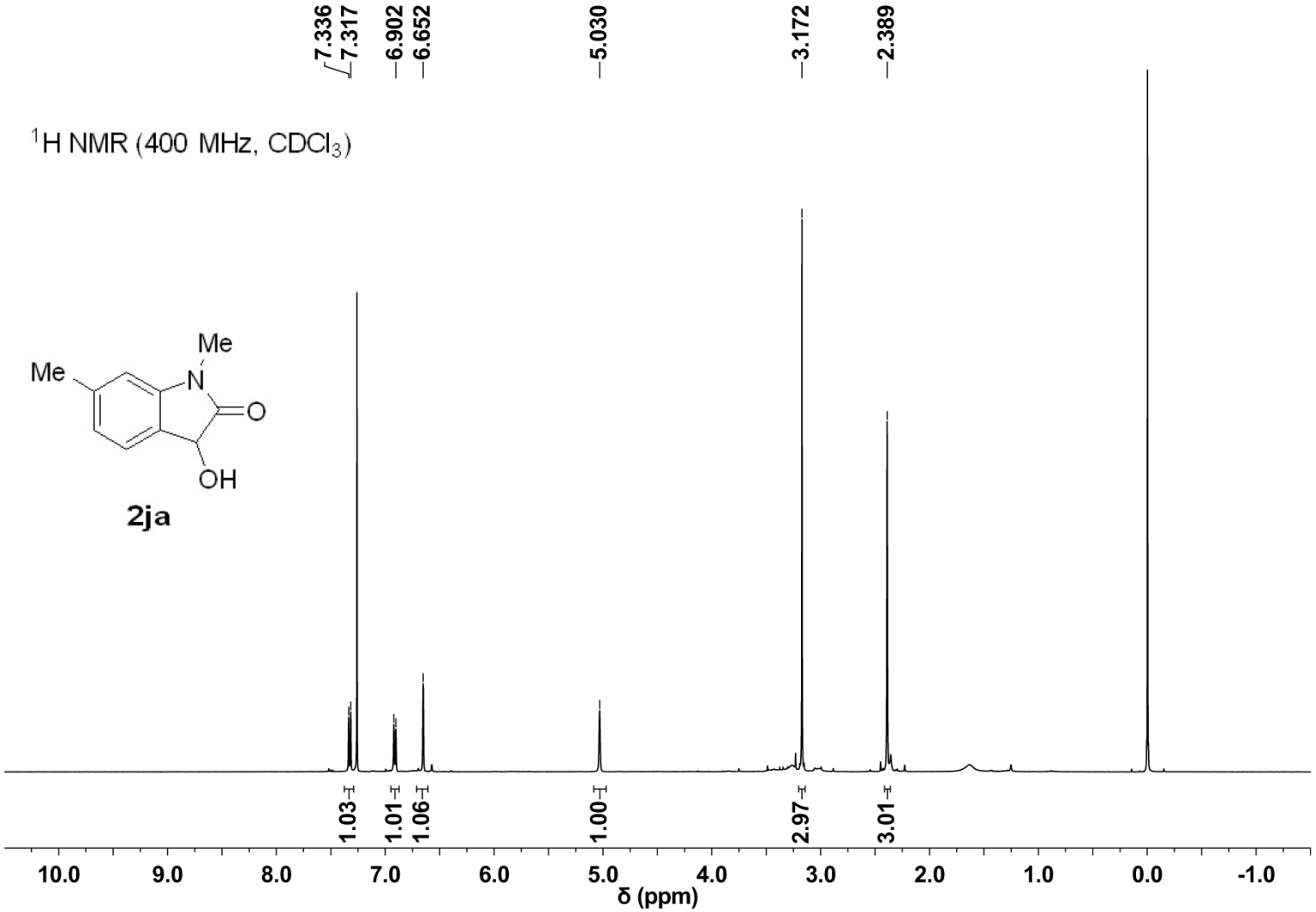

7.586
7.578
7.575
7.565
7.560
7.557
7.456
7.451
7.437
7.418
7.358
7.339
7.321
6.914
6.894
5.146
3.591
3.233
1H NMR (400 MHz, CDCl3)
Me
N
O
Ph
OH
2h
1.00
3.04
2.04
1.07
1.00
1.00
0.89
3.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

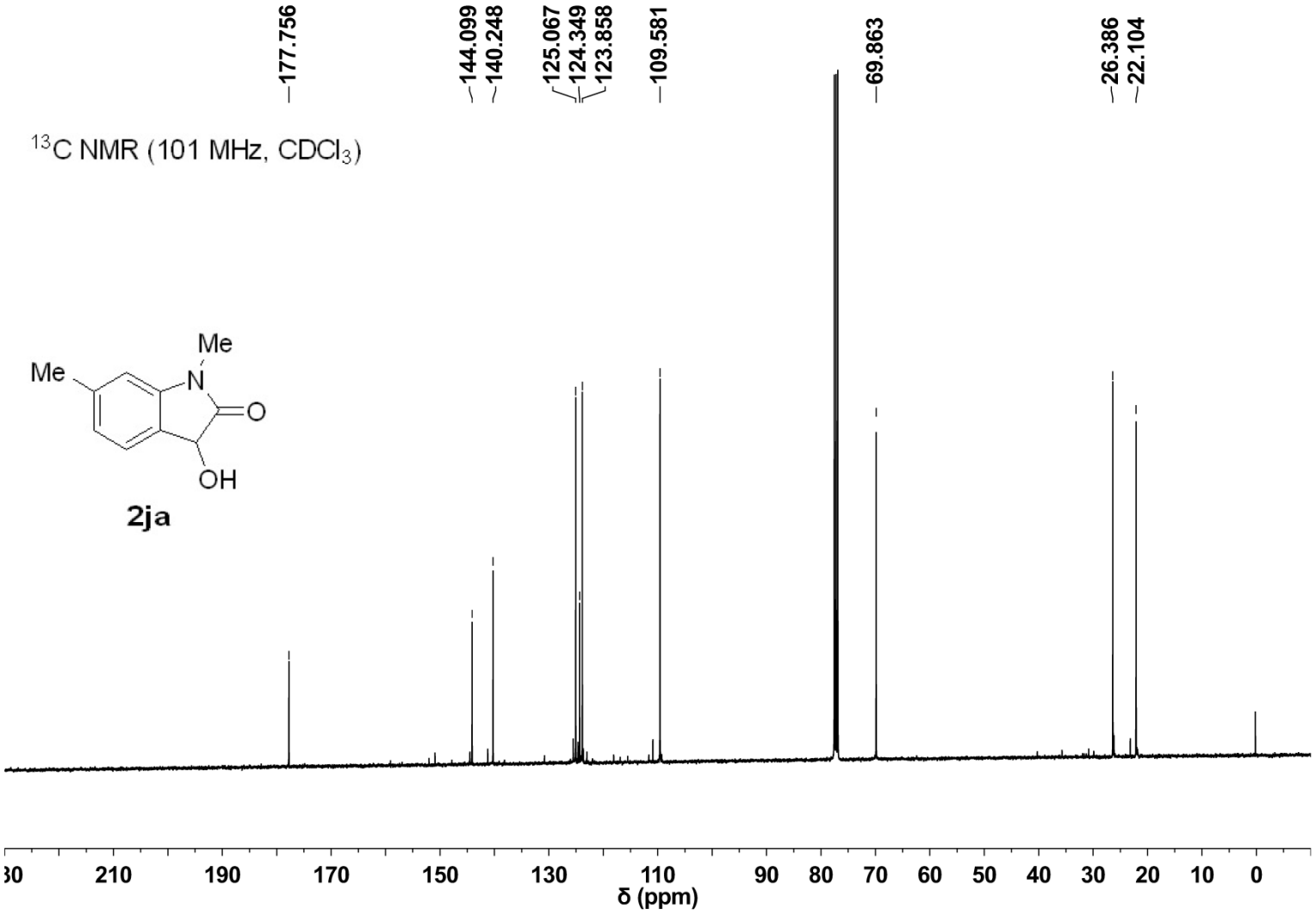

176.087
143.237
140.067
134.471
128.953
126.924
122.807
108.813
68.867
25.901
13C NMR (101 MHz, DMSO)
Me
N
O
Ph
OH
2h
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

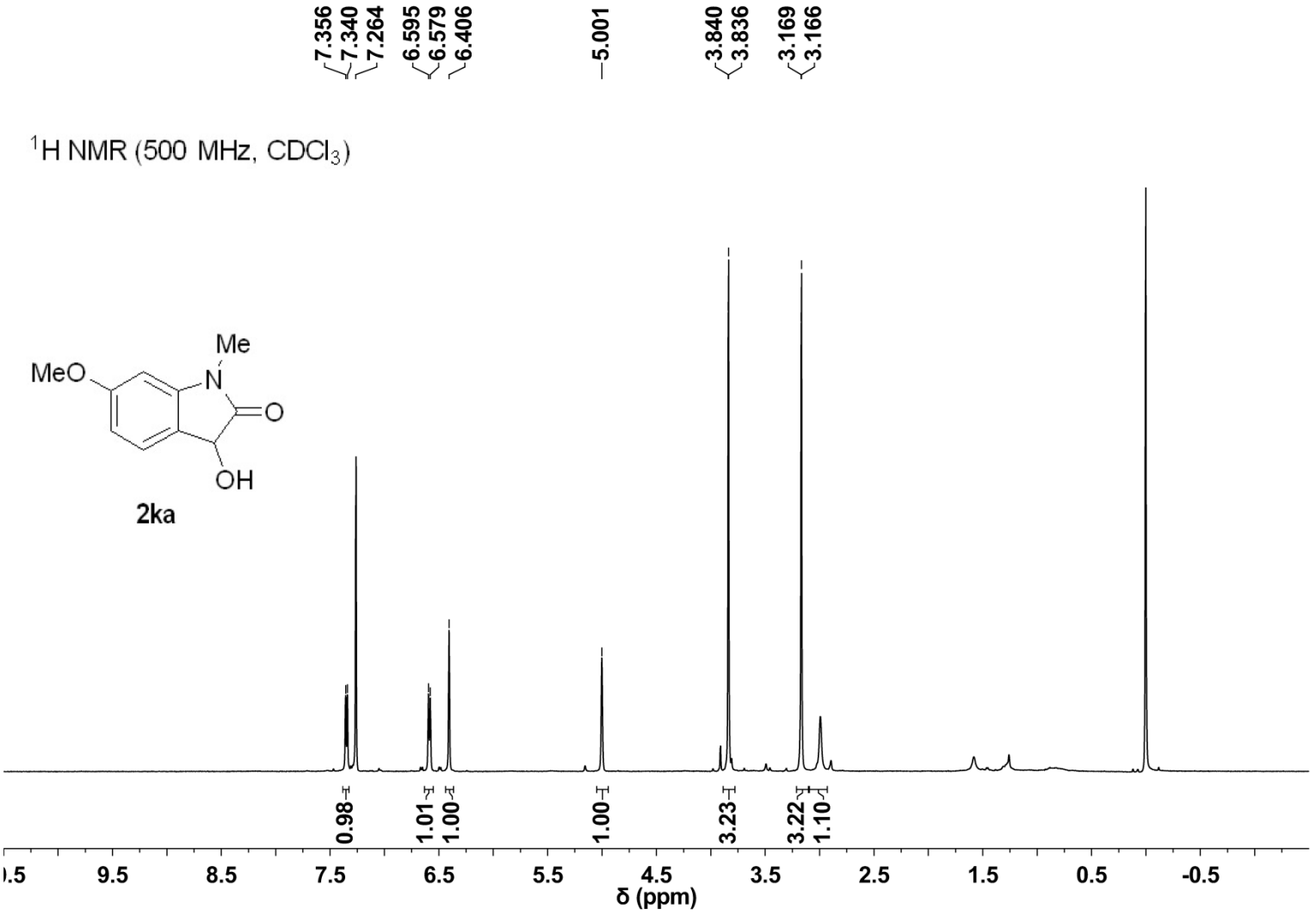

7.101
7.096
6.872
6.865
6.851
6.844
6.743
6.722
5.068
3.801
3.223
3.168
$^1$H NMR (400 MHz, $CDCl_3$)
Me
N
O
MeO
OH
2i
1.00
1.02
0.98
1.00
3.02
3.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

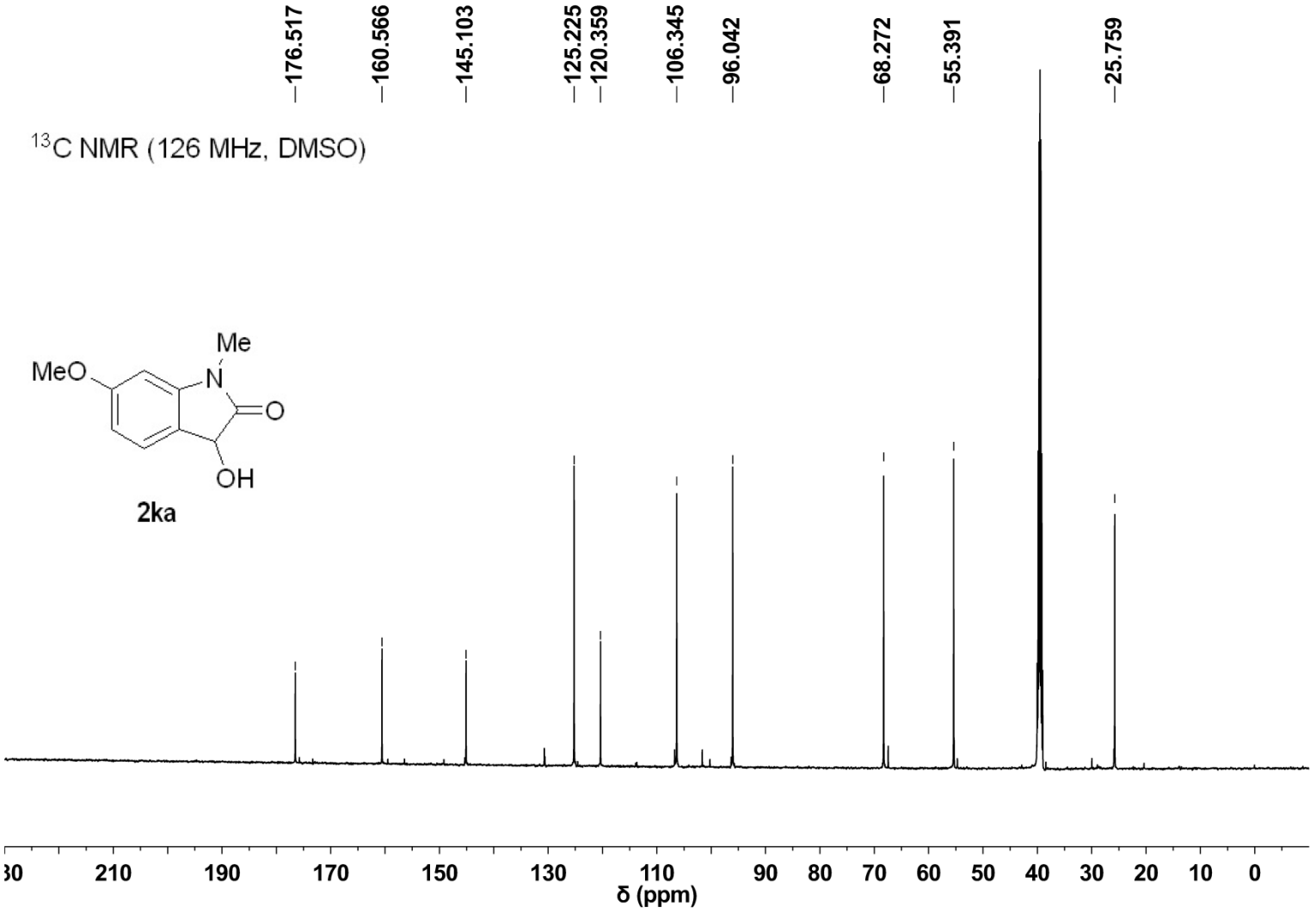
c

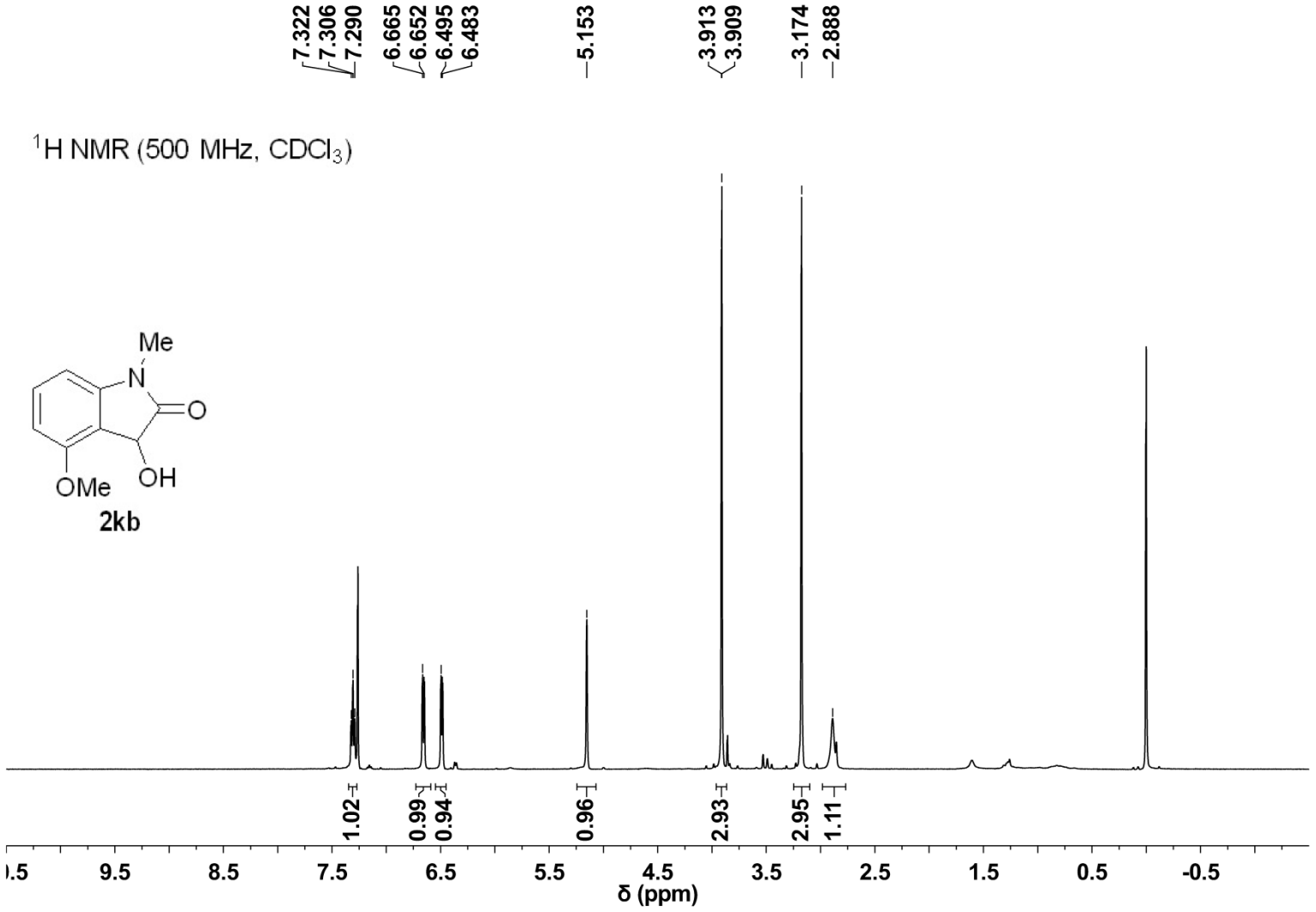

177.073
156.693
137.316
128.465
114.681
112.253
109.146
70.343
56.055
26.534
13C NMR (101 MHz, CDCl3)
Me
N
O
MeO
OH
2i
240
220
200
180
160
140
120
100
80
60
40
20
0
-10
-30
δ (ppm)

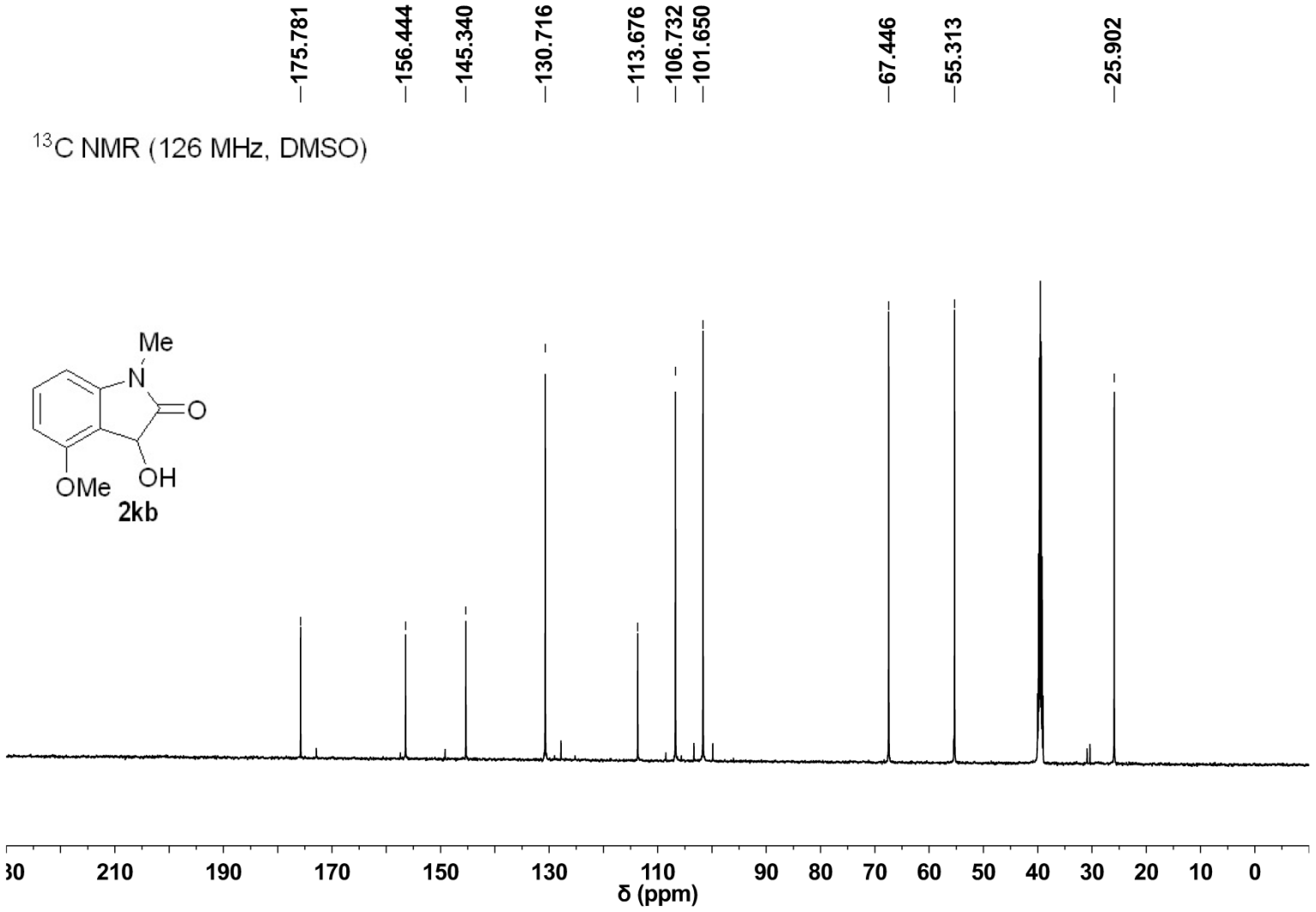

7.336
7.317
6.902
6.652
5.030
3.172
2.389
$^1$H NMR (400 MHz, $CDCl_3$)
Me
Me
N
O
OH
2ja
1.03
1.01
1.06
1.00
2.97
3.01
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

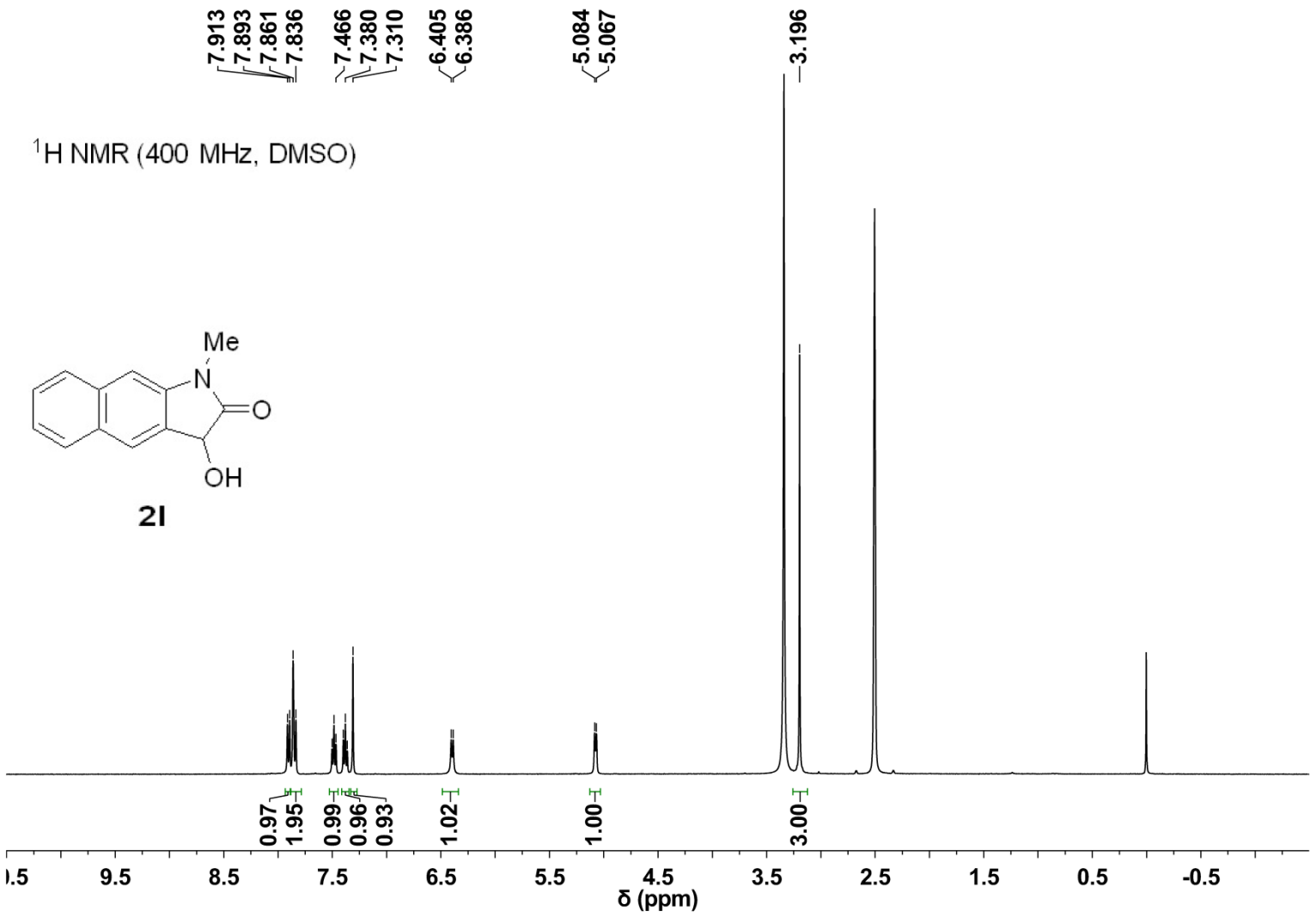

177.756
144.099
140.248
125.067
124.349
123.858
109.581
69.863
26.386
22.104
$^{13}$C NMR (101 MHz, $CDCl_3$)
Me
Me
N
O
OH
2ja
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

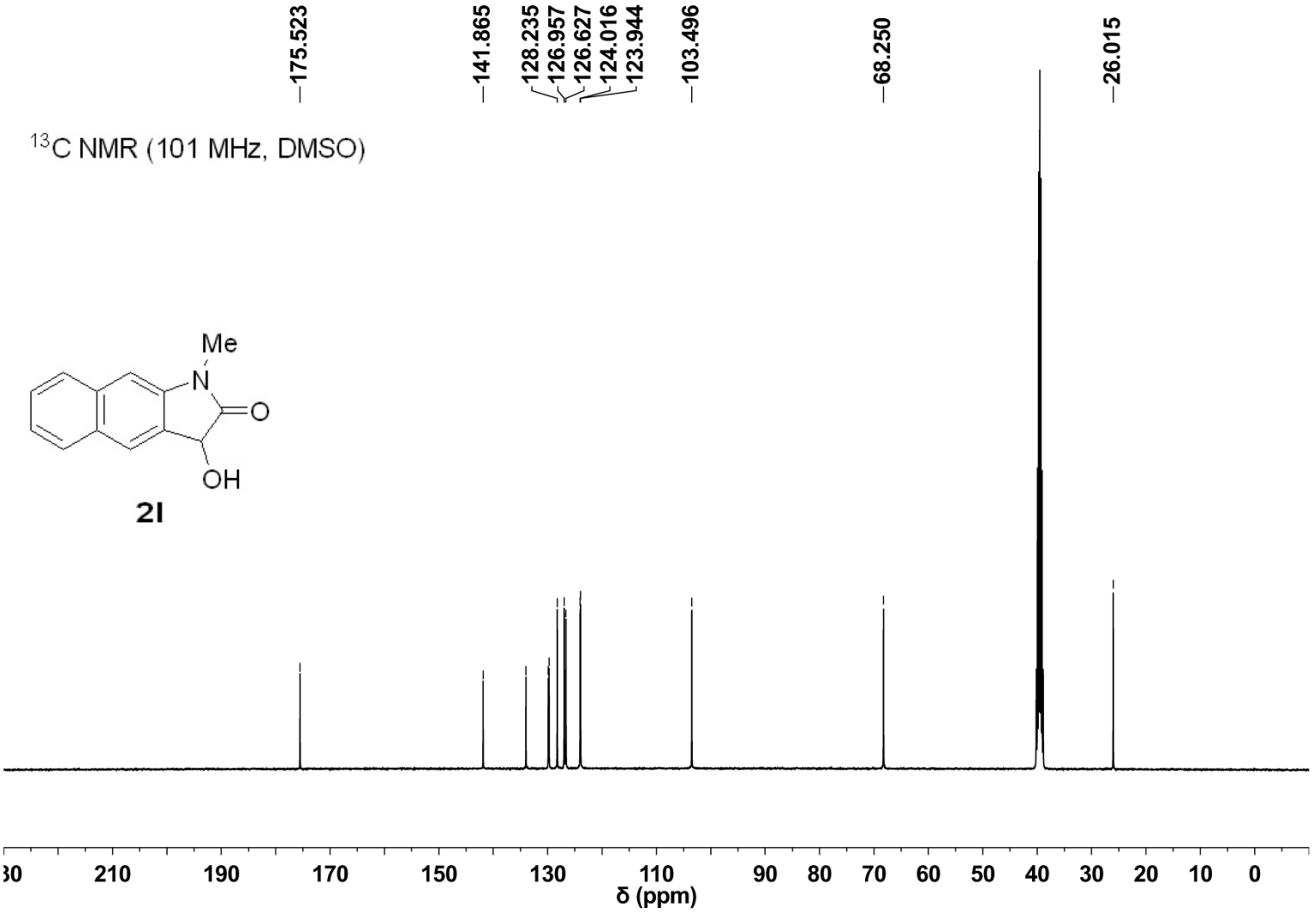

7.356
7.340
7.264
6.595
6.579
6.406
5.001
3.840
3.836
3.169
3.166
$^1$H NMR (500 MHz, $CDCl_3$)
Me
MeO
N
O
OH
2ka
0.98
1.01
1.00
1.00
3.23
3.22
1.10
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)

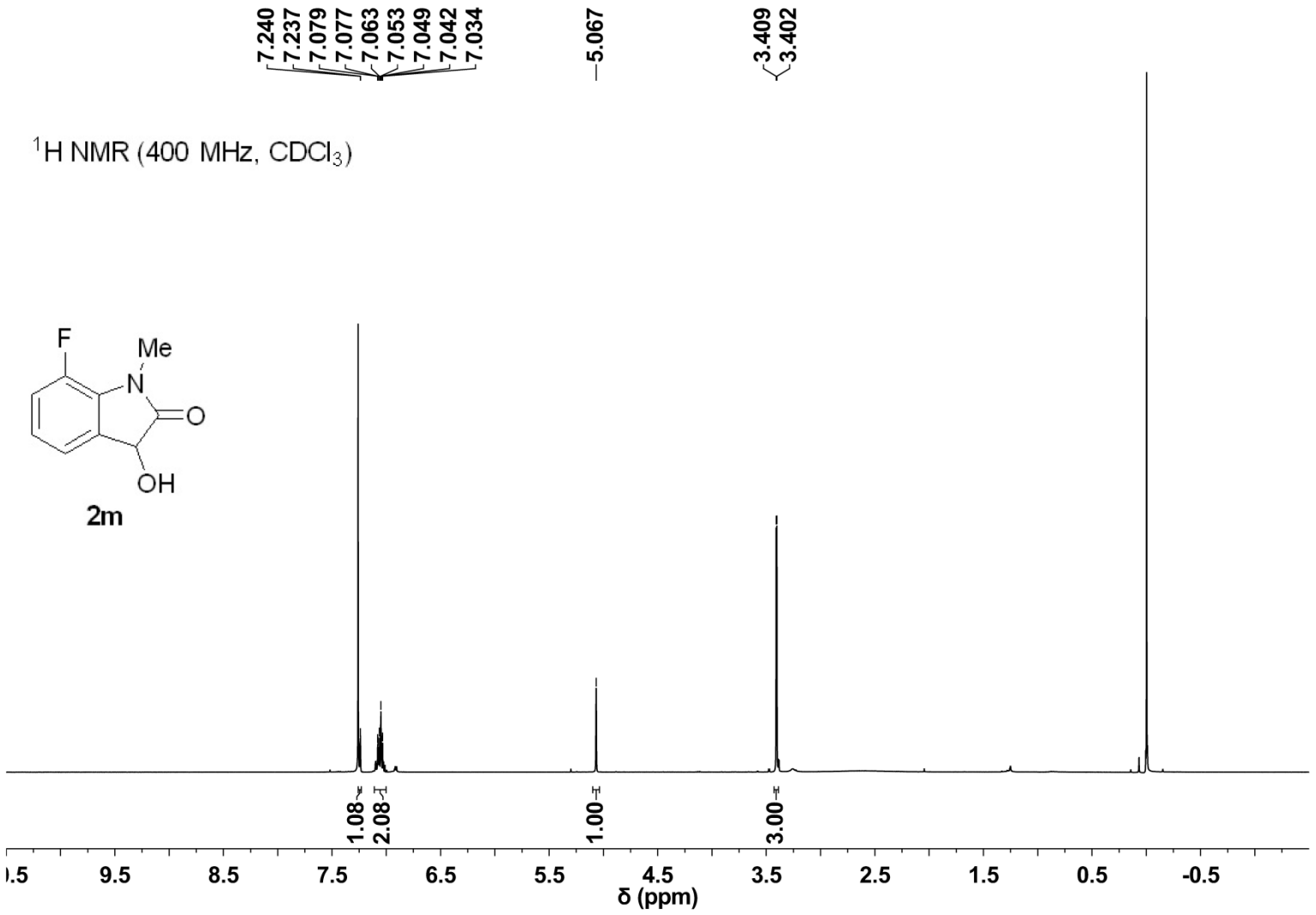

176.517
160.566
145.103
125.225
120.359
106.345
96.042
68.272
55.391
25.759
13C NMR (126 MHz, DMSO)
Me
MeO
N
O
OH
2ka
30
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

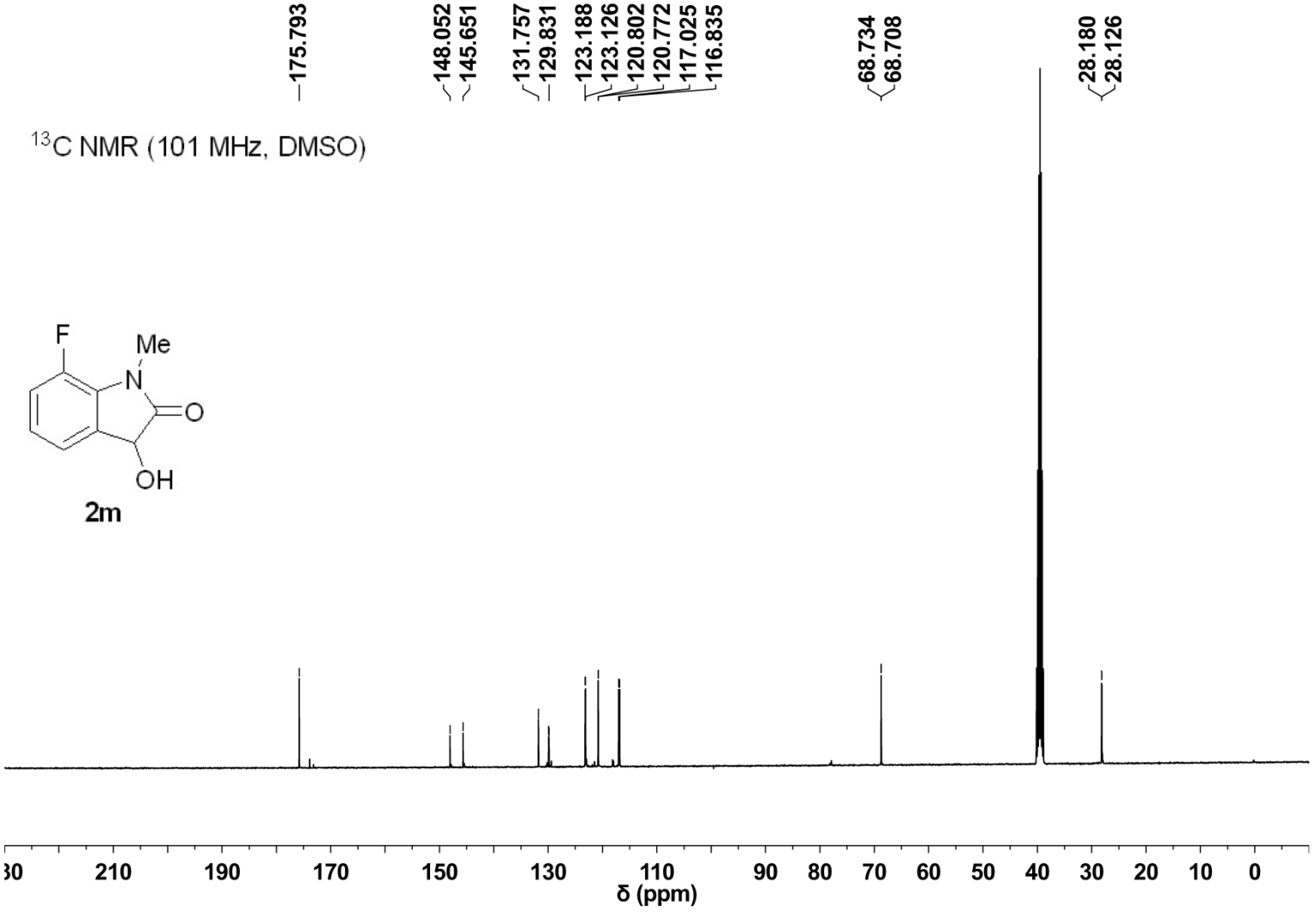

7.322
7.306
7.290
6.665
6.652
6.495
6.483
5.153
3.913
3.909
3.174
2.888
$^1$H NMR (500 MHz, $CDCl_3$)
Me
N
O
OMe
OH
2kb
1.02
0.99
0.94
0.96
2.93
2.95
1.11
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)

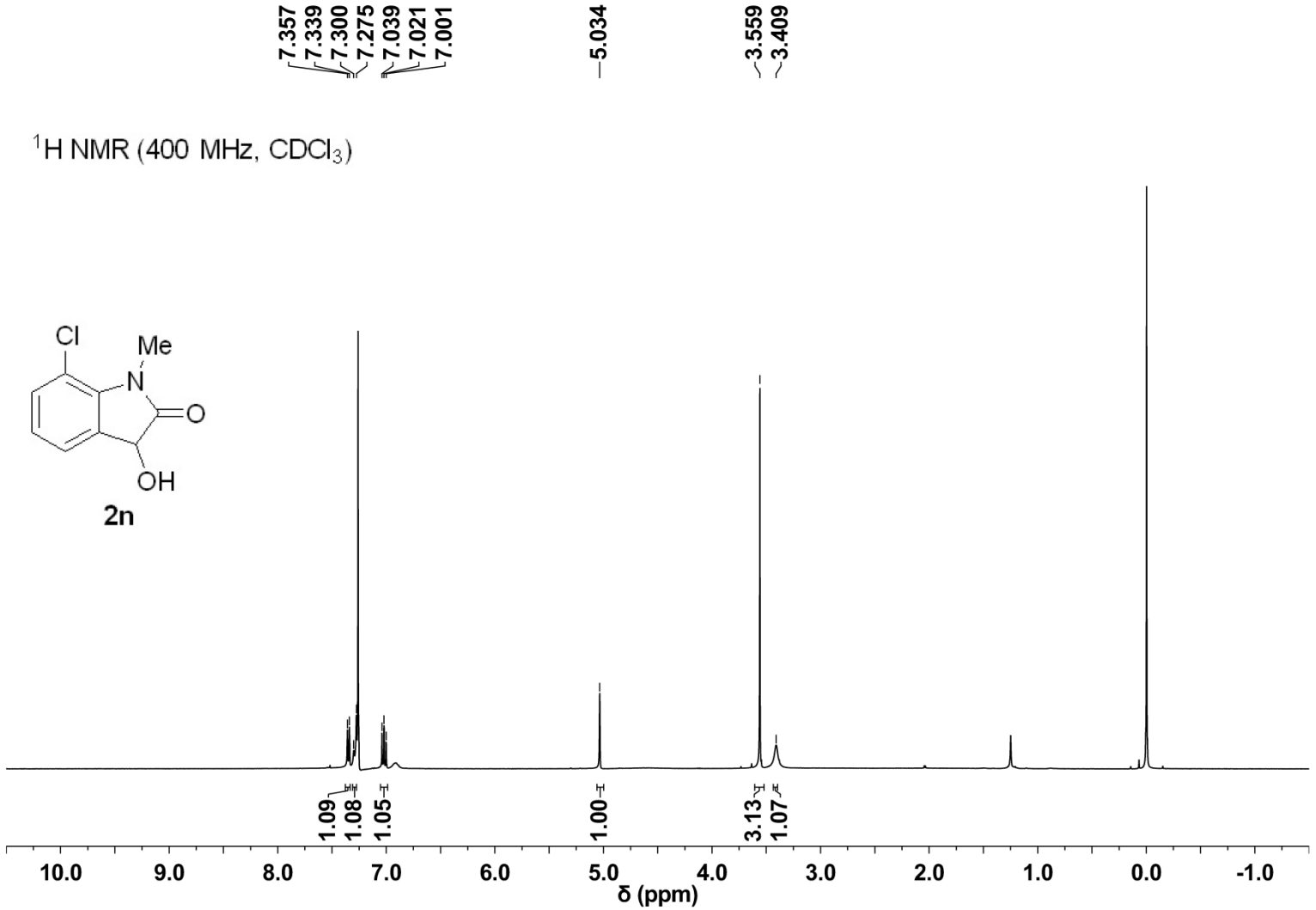

175.781
156.444
145.340
130.716
113.676
106.732
101.650
67.446
55.313
25.902
13C NMR (126 MHz, DMSO)
Me
N
O
OMe
OH
2kb
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

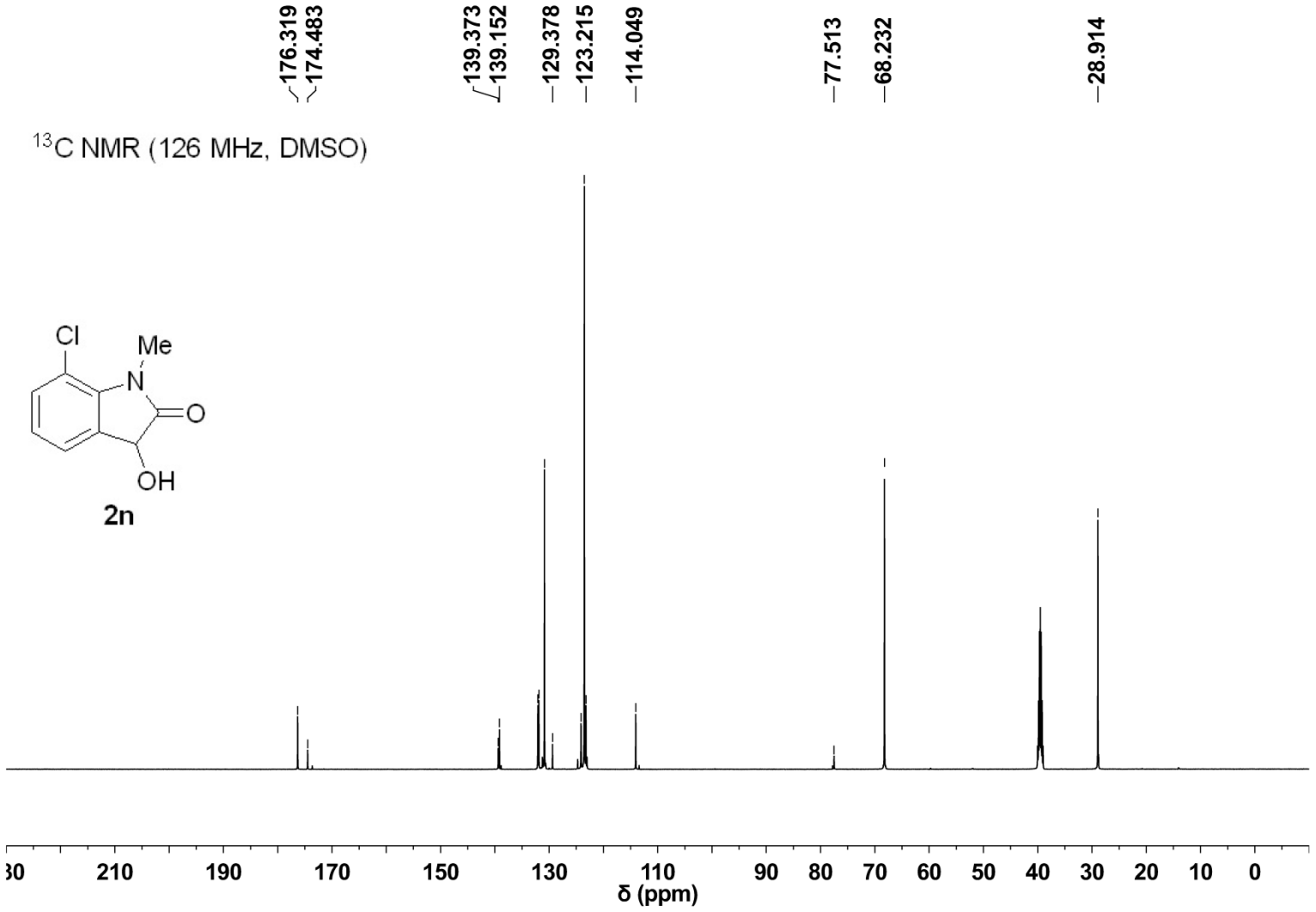

7.913
7.893
7.861
7.836
7.466
7.380
7.310
6.405
6.386
5.084
5.067
3.196
$^1$H NMR (400 MHz, DMSO)
Me
N
O
OH
2l
0.97
1.95
0.99
0.96
0.93
1.02
1.00
3.00
δ (ppm)

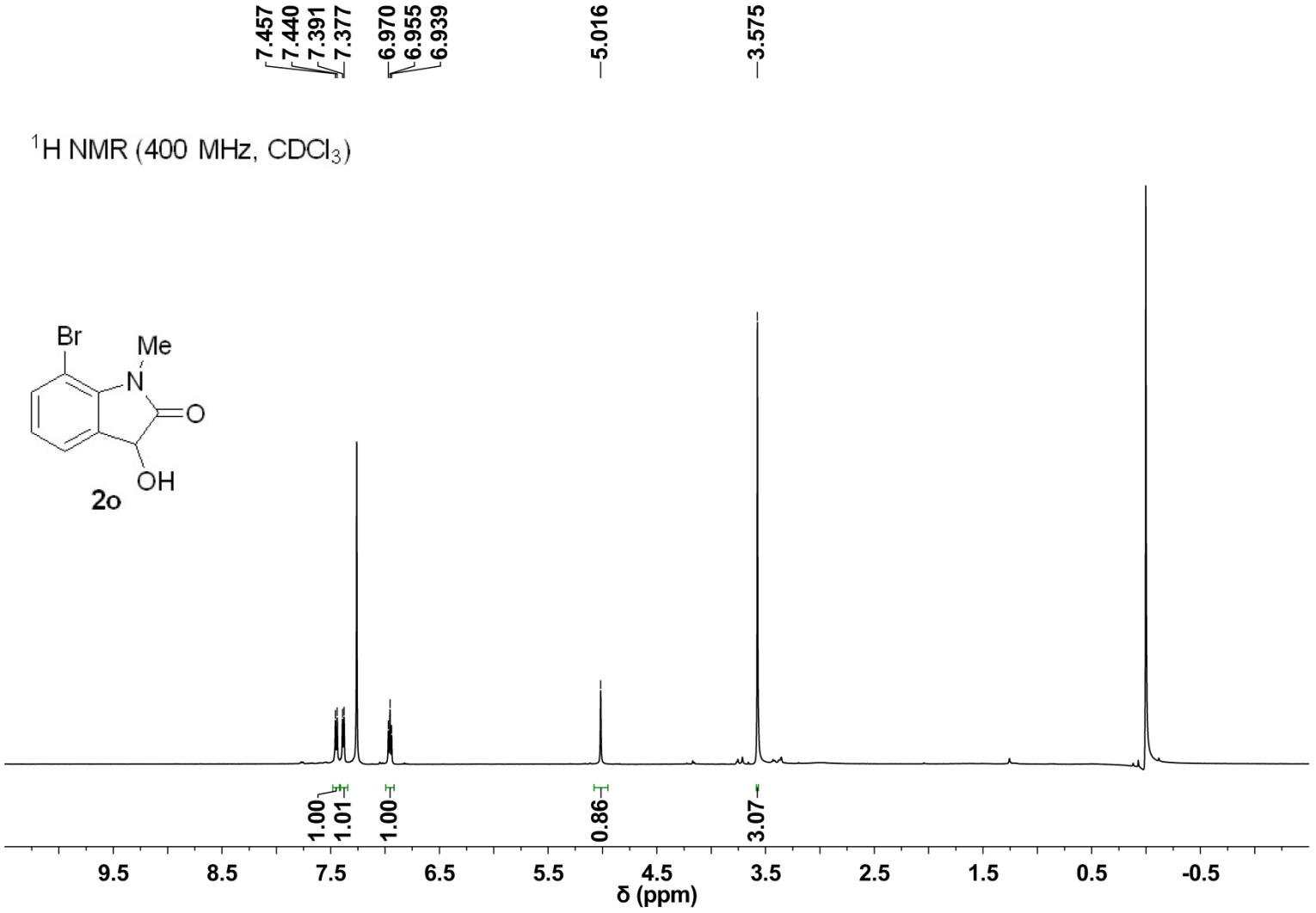

175.523
141.865
128.235
126.957
126.627
124.016
123.944
103.496
68.250
26.015
13C NMR (101 MHz, DMSO)
Me
N
O
OH
2l
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

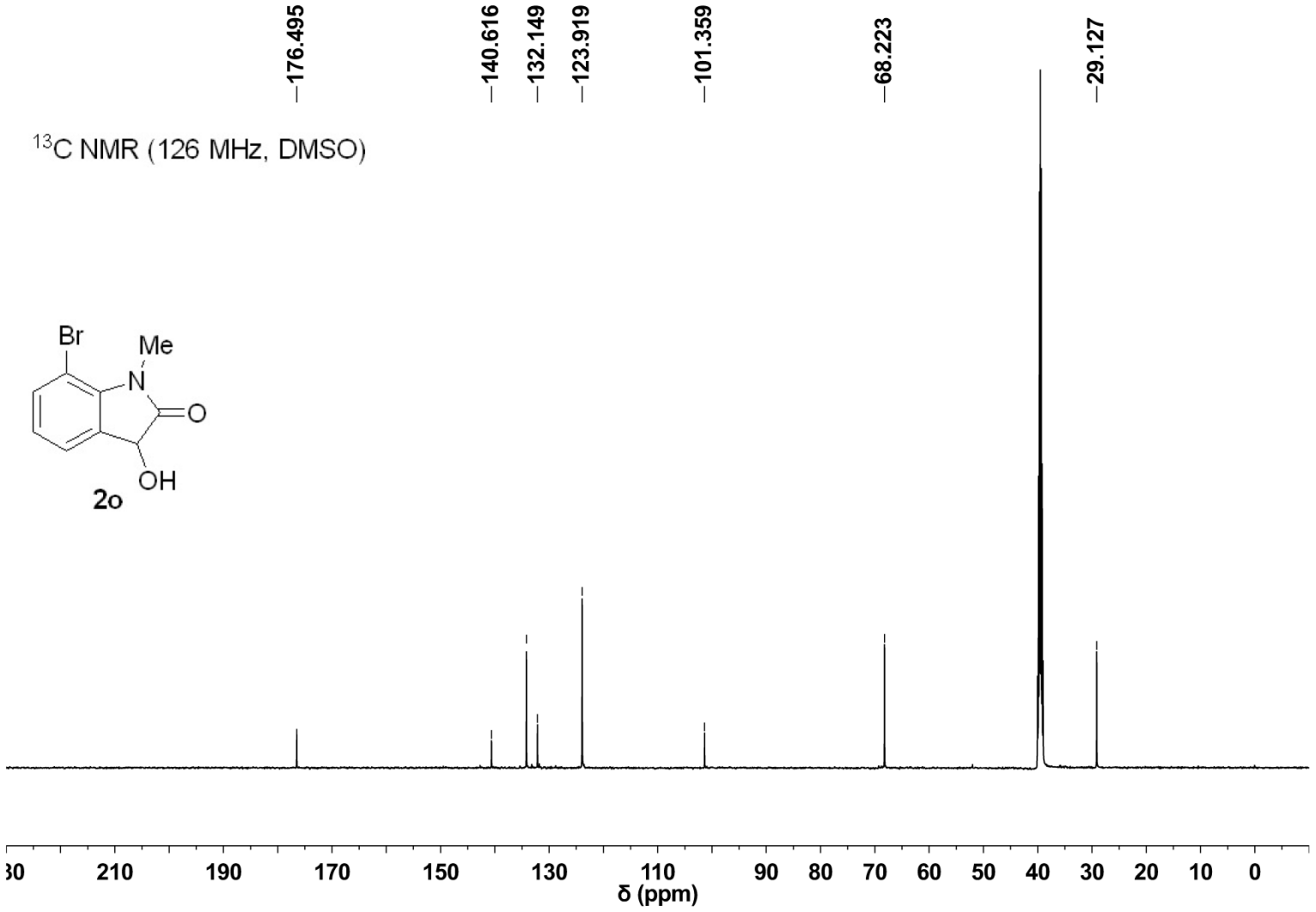

7.240
7.237
7.079
7.077
7.063
7.053
7.049
7.042
7.034
5.067
3.409
3.402
$^1$H NMR (400 MHz, $CDCl_3$)
F
Me
N
O
OH
2m
1.08
2.08
1.00
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)

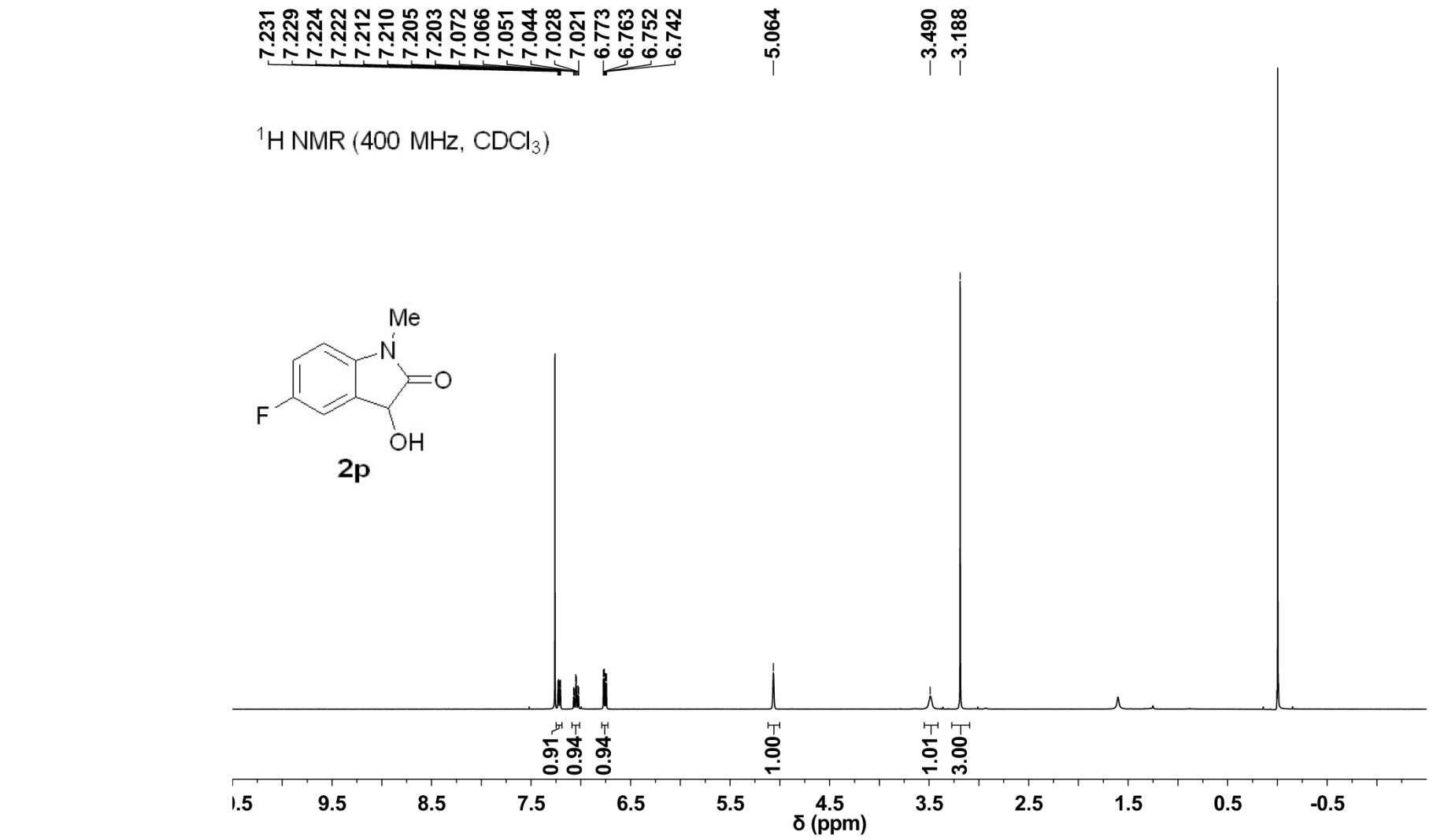

175.793
148.052
145.651
131.757
129.831
123.188
123.126
120.802
120.772
117.025
116.835
68.734
68.708
28.180
28.126
13C NMR (101 MHz, DMSO)
F
Me
N
O
OH
2m
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

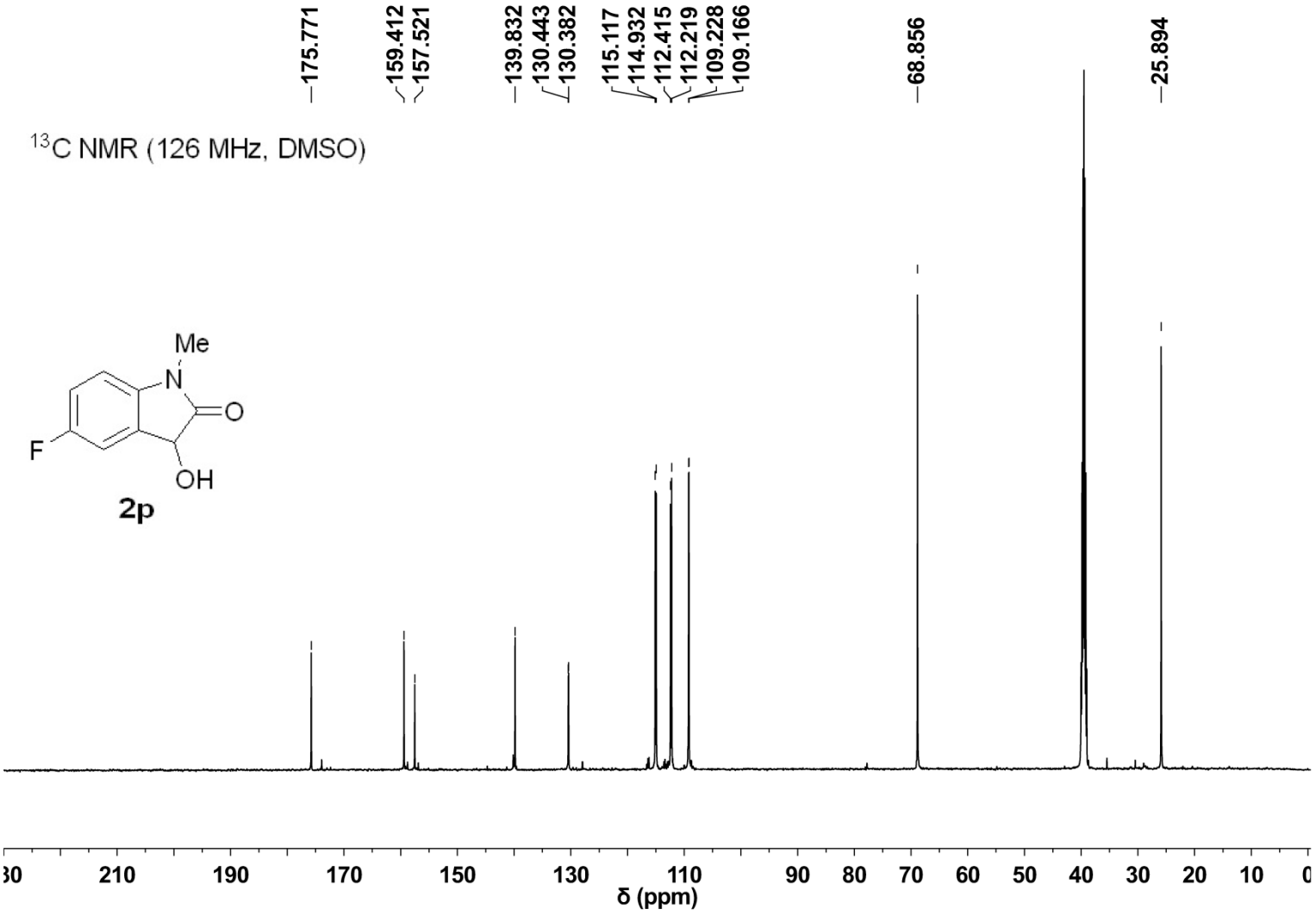

7.357
7.339
7.300
7.275
7.039
7.021
7.001
5.034
3.559
3.409
$^1$H NMR (400 MHz, $CDCl_3$)
Cl
Me
N
O
OH
2n
1.09
1.08
1.05
1.00
3.13
1.07
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

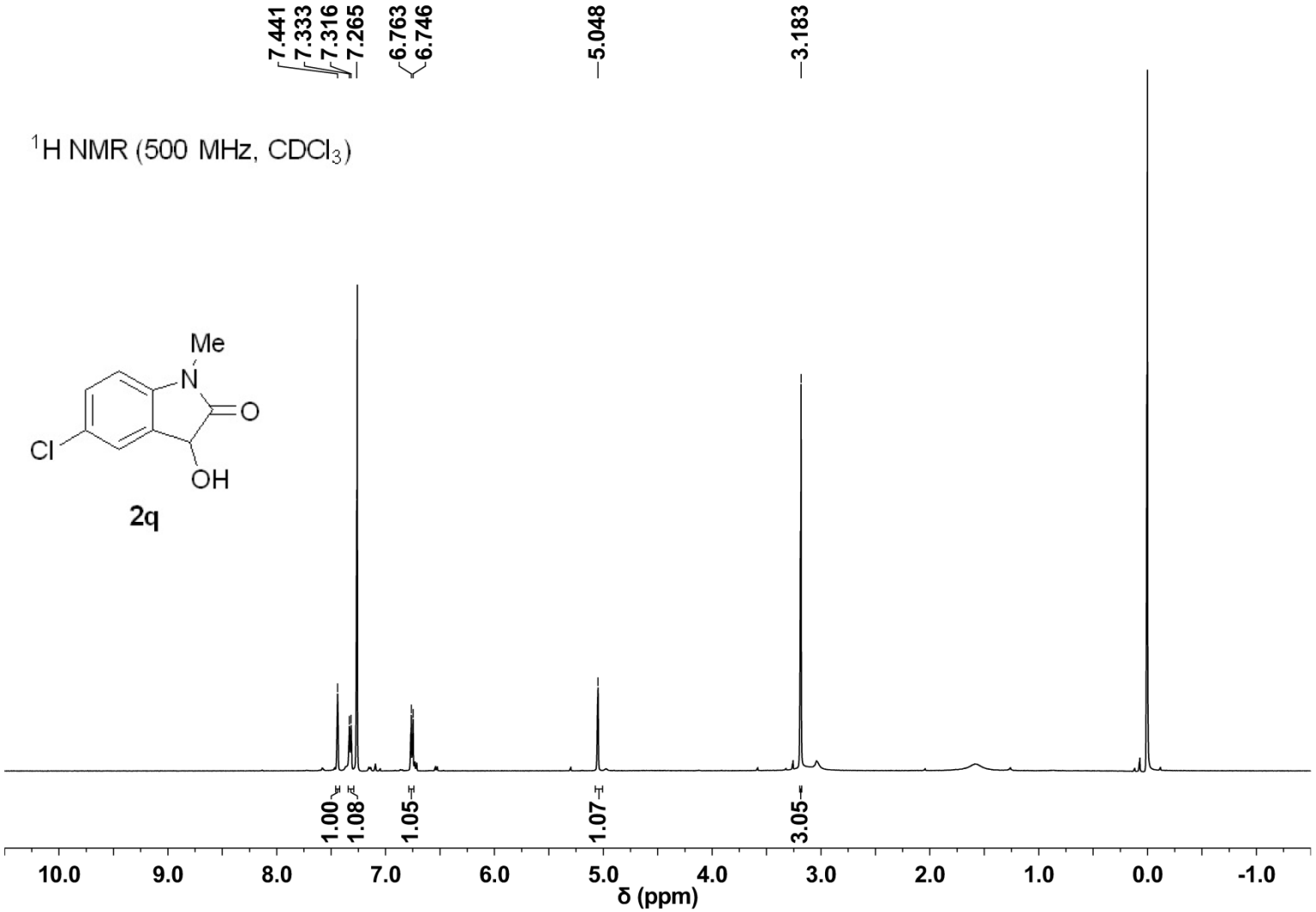

176.319
174.483
139.373
139.152
129.378
123.215
114.049
77.513
68.232
28.914
13C NMR (126 MHz, DMSO)
Cl
Me
N
O
OH
2n
30
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

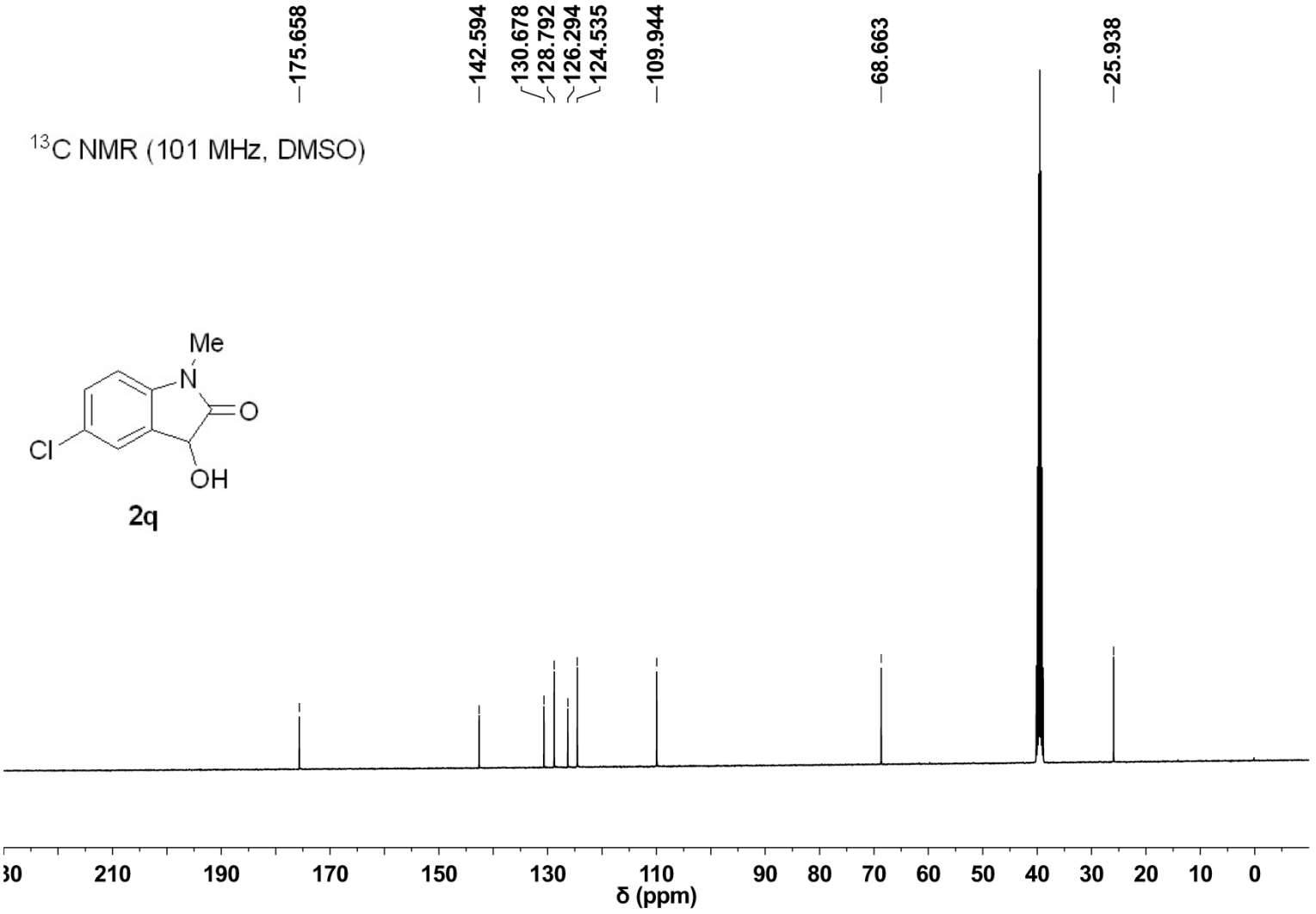

7.457
7.440
7.391
7.377
6.970
6.955
6.939
5.016
3.575
1H NMR (400 MHz, CDCl3)
Br
Me
N
O
OH
2o
1.00
1.01
1.00
0.86
3.07
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)

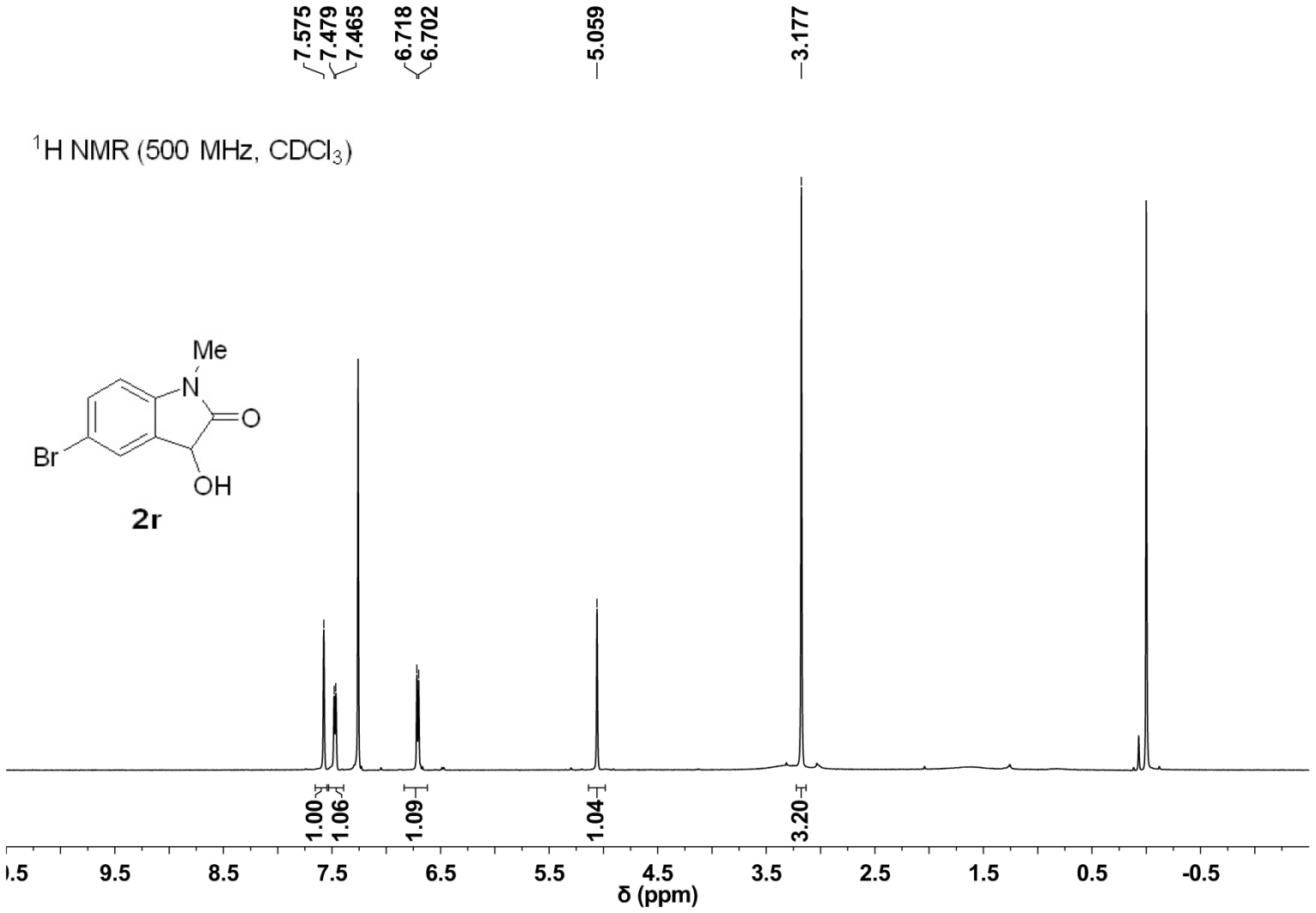

176.495
140.616
132.149
123.919
101.359
68.223
29.127
13C NMR (126 MHz, DMSO)
Br
Me
N
O
OH
2o
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

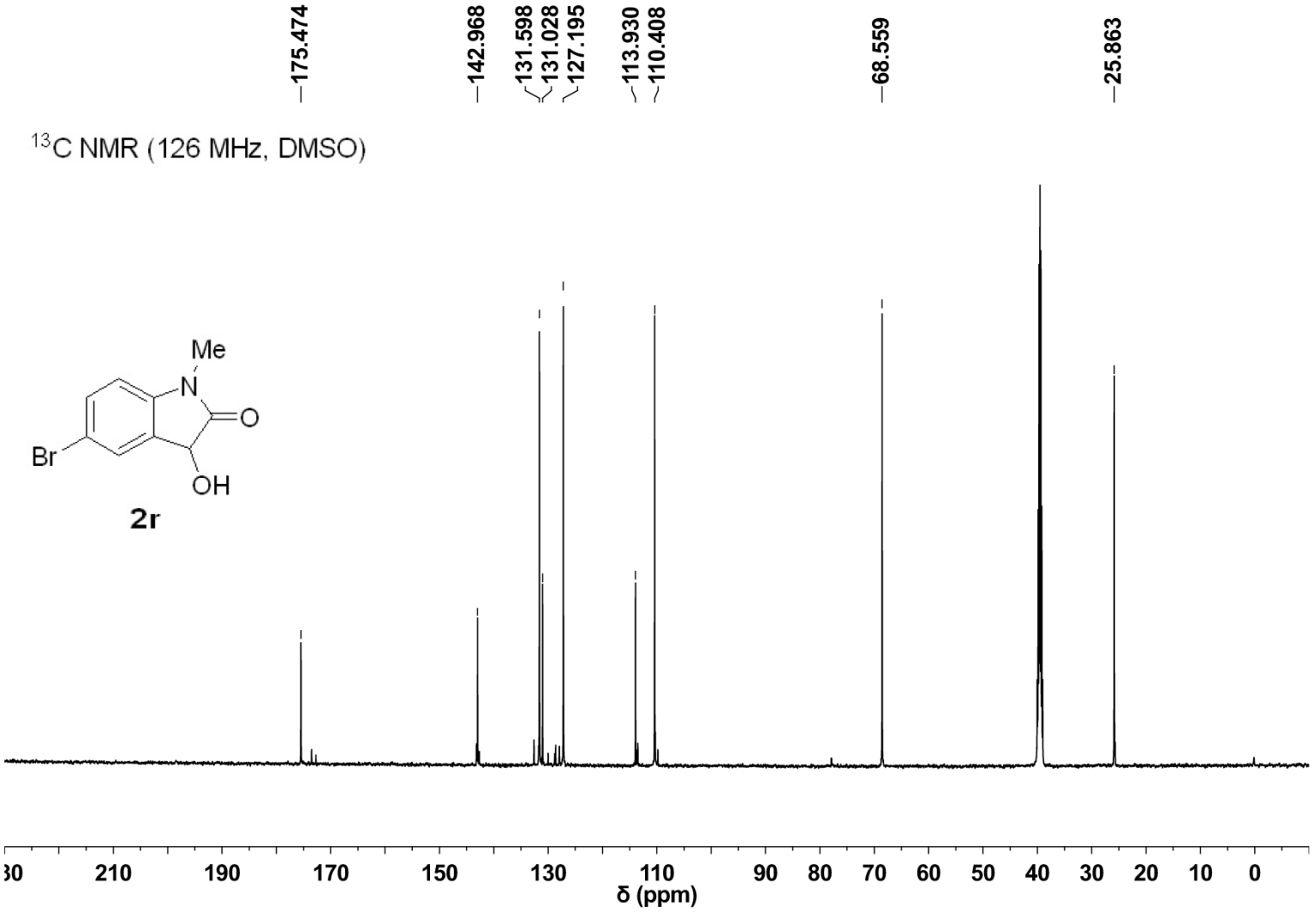

7.231
7.229
7.224
7.222
7.212
7.210
7.205
7.203
7.072
7.066
7.051
7.044
7.028
7.021
6.773
6.763
6.752
6.742
5.064
3.490
3.188
1H NMR (400 MHz, CDCl3)
Me
N
O
F
OH
2p
0.91
0.94
0.94
1.00
1.01
3.00
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)

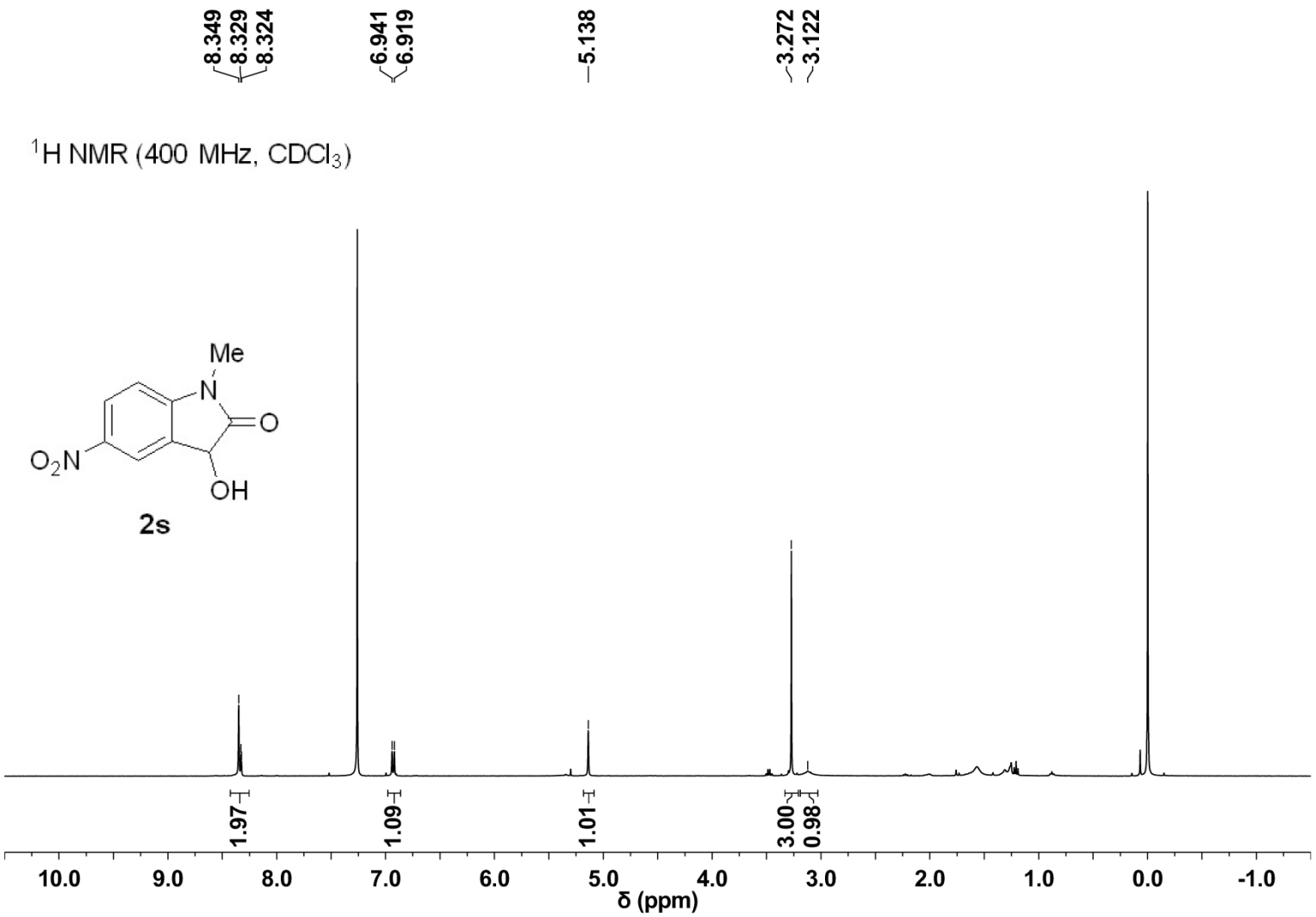

175.771
159.412
157.521
139.832
130.443
130.382
115.117
114.932
112.415
112.219
109.228
109.166
68.856
25.894
13C NMR (126 MHz, DMSO)
Me
N
O
F
OH
2p
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
δ (ppm)

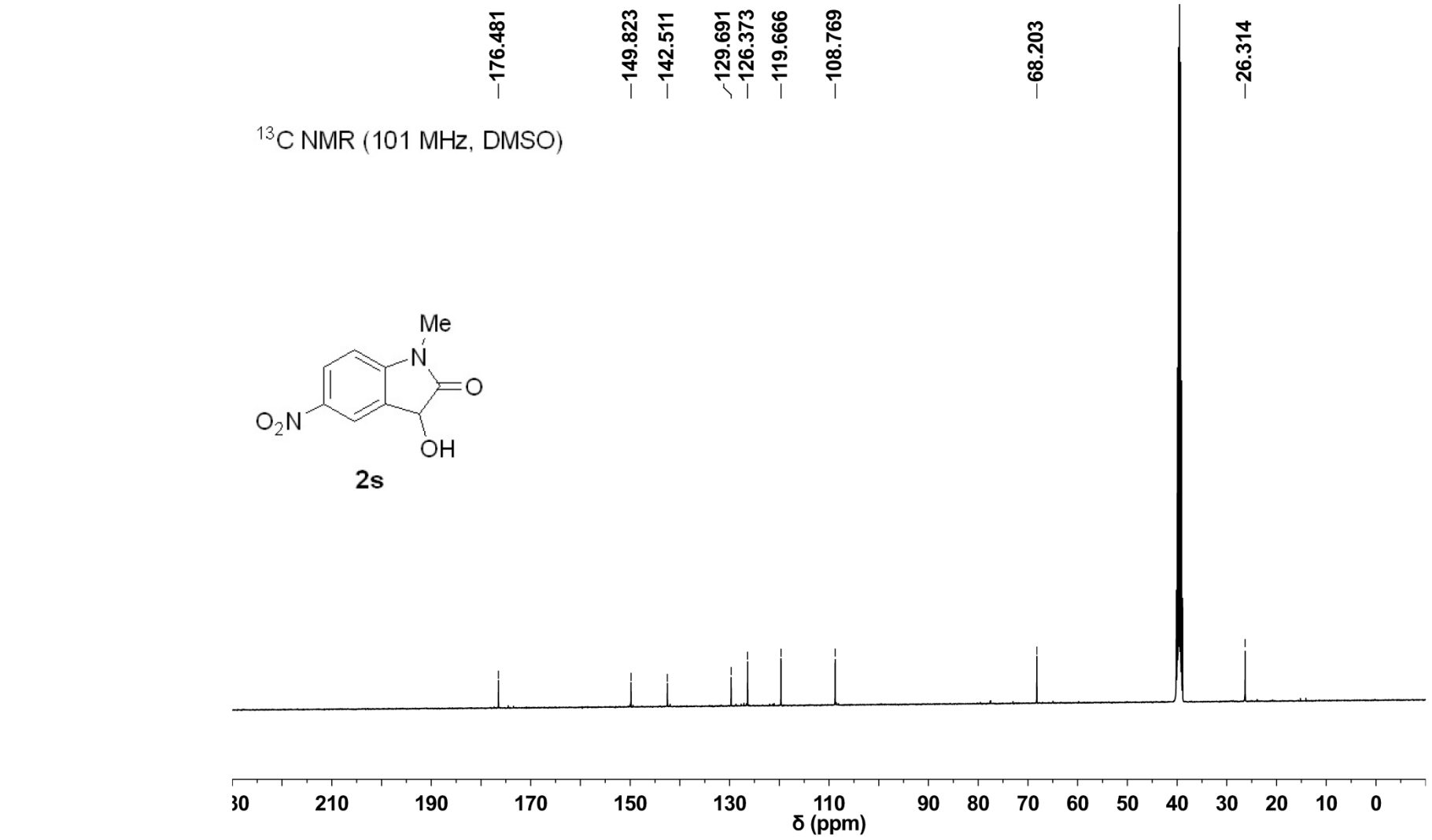

7.441
7.333
7.316
7.265
6.763
6.746
5.048
3.183
$^1$H NMR (500 MHz, $CDCl_3$)
Me
N
O
Cl
OH
2q
1.00
1.08
1.05
1.07
3.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

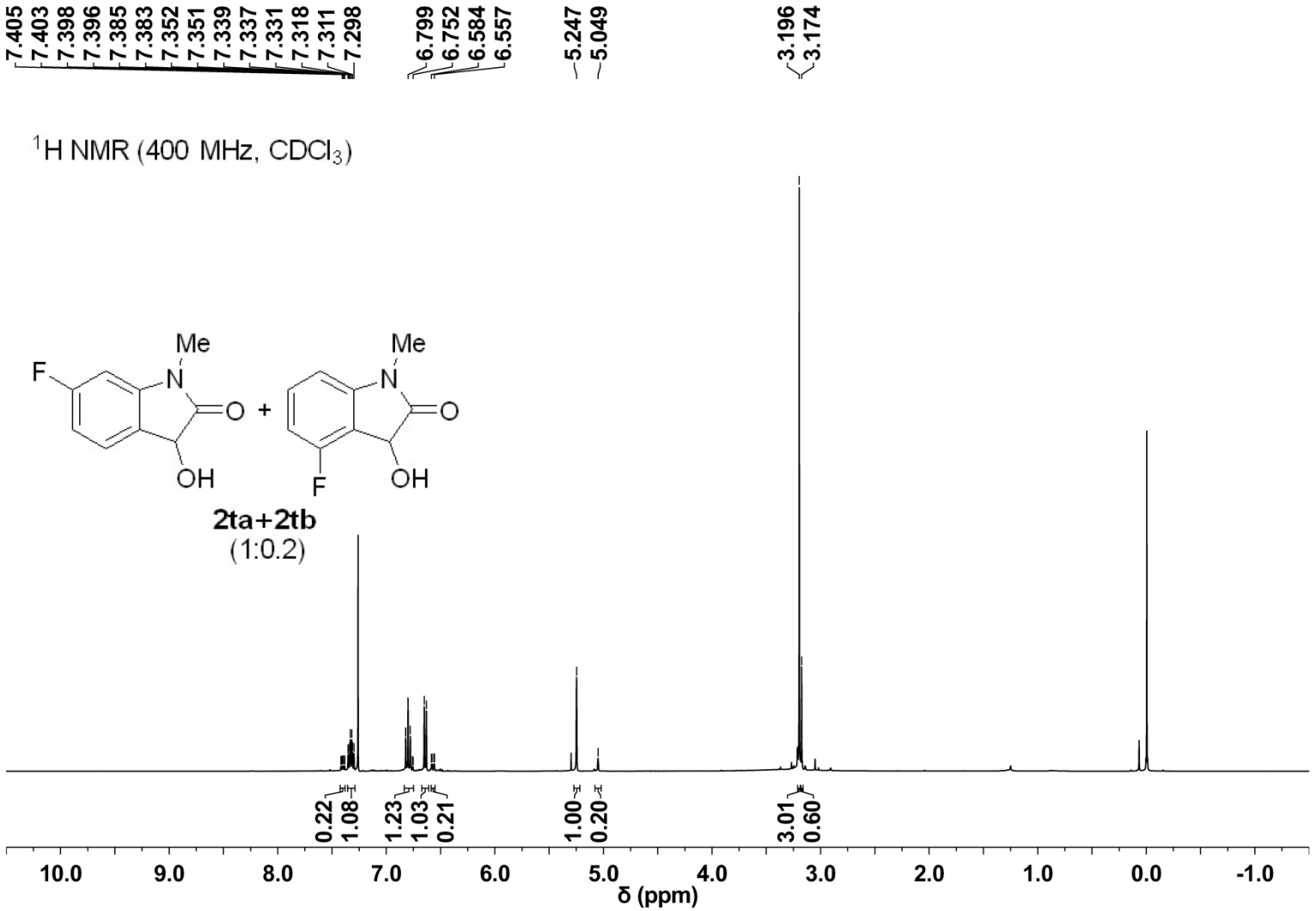

175.658
142.594
130.678
128.792
126.294
124.535
109.944
68.663
25.938
13C NMR (101 MHz, DMSO)
Me
N
O
Cl
OH
2q
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

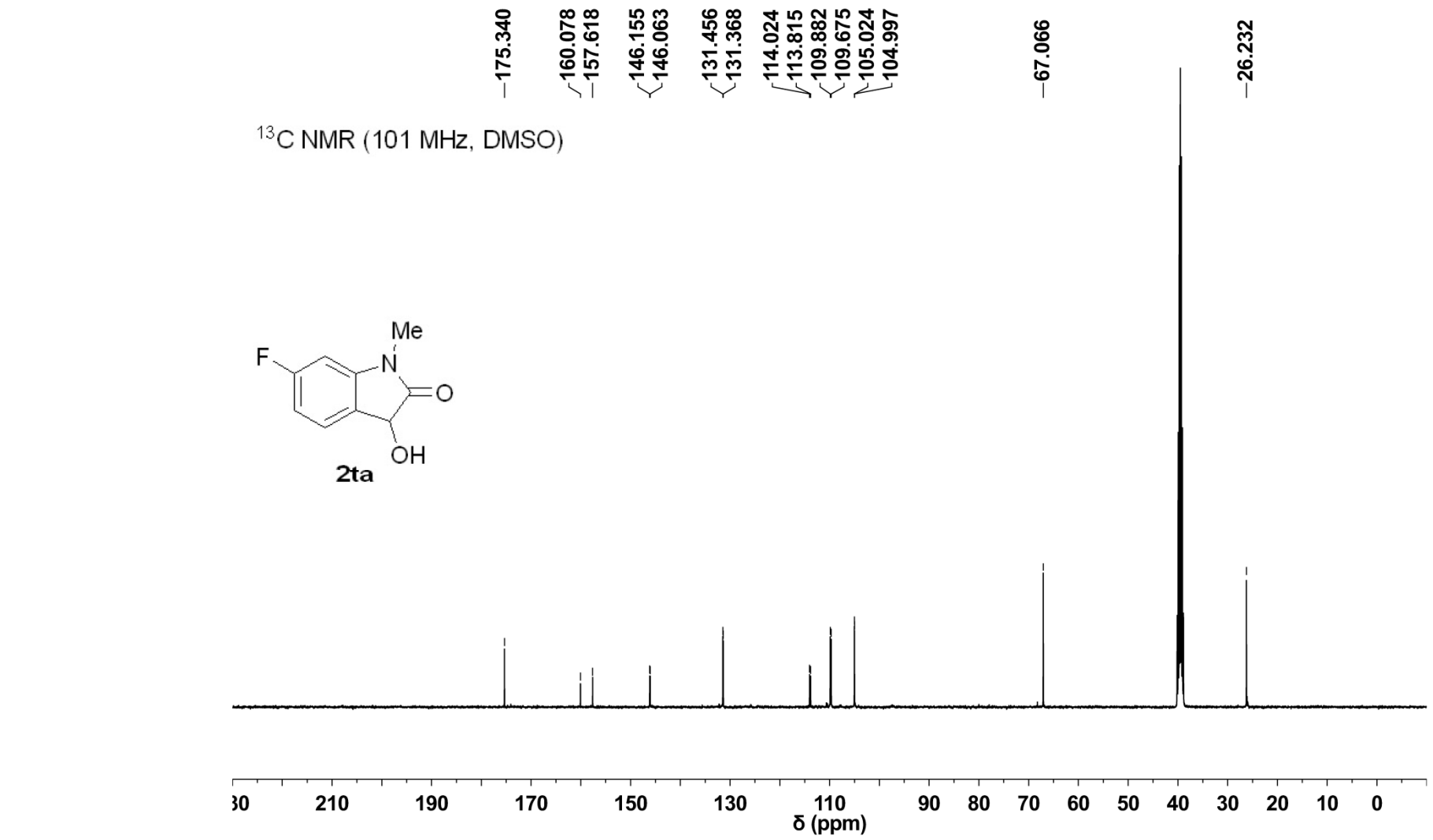

7.575
7.479
7.465
6.718
6.702
5.059
3.177
$^1$H NMR (500 MHz, $CDCl_3$)
Me
N
O
Br
OH
2r
1.00
1.06
1.09
1.04
3.20
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)

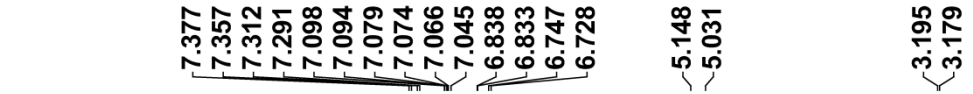

175.474
142.968
131.598
131.028
127.195
113.930
110.408
68.559
25.863
13C NMR (126 MHz, DMSO)
Me
N
O
Br
OH
2r
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)


8.349
8.329
8.324
6.941
6.919
5.138
3.272
3.122
$^1$H NMR (400 MHz, $CDCl_3$)
Me
N
O
$O_2N$
OH
2s
1.97
1.09
1.01
3.00
0.98
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

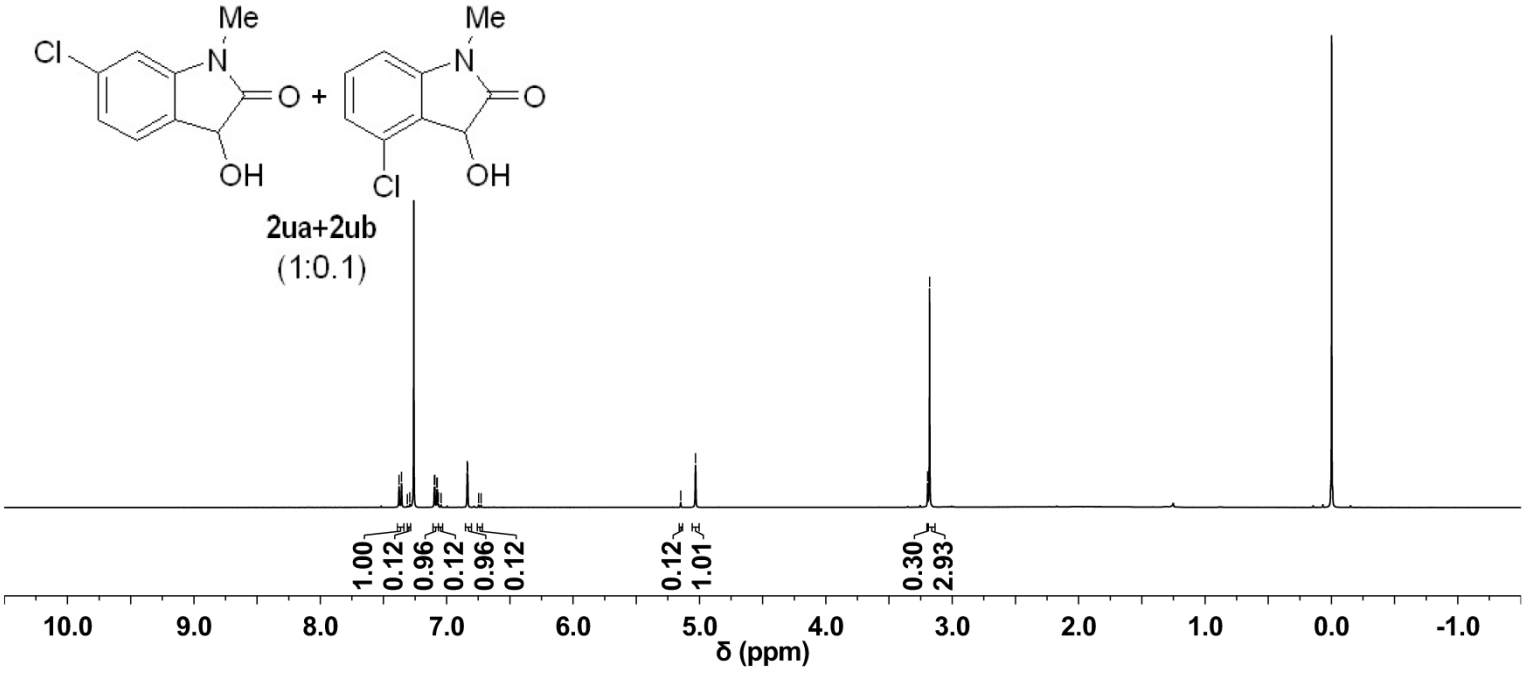

176.481
149.823
142.511
129.691
126.373
119.666
108.769
68.203
26.314
13C NMR (101 MHz, DMSO)
Me
N
O
O2N
OH
2s
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

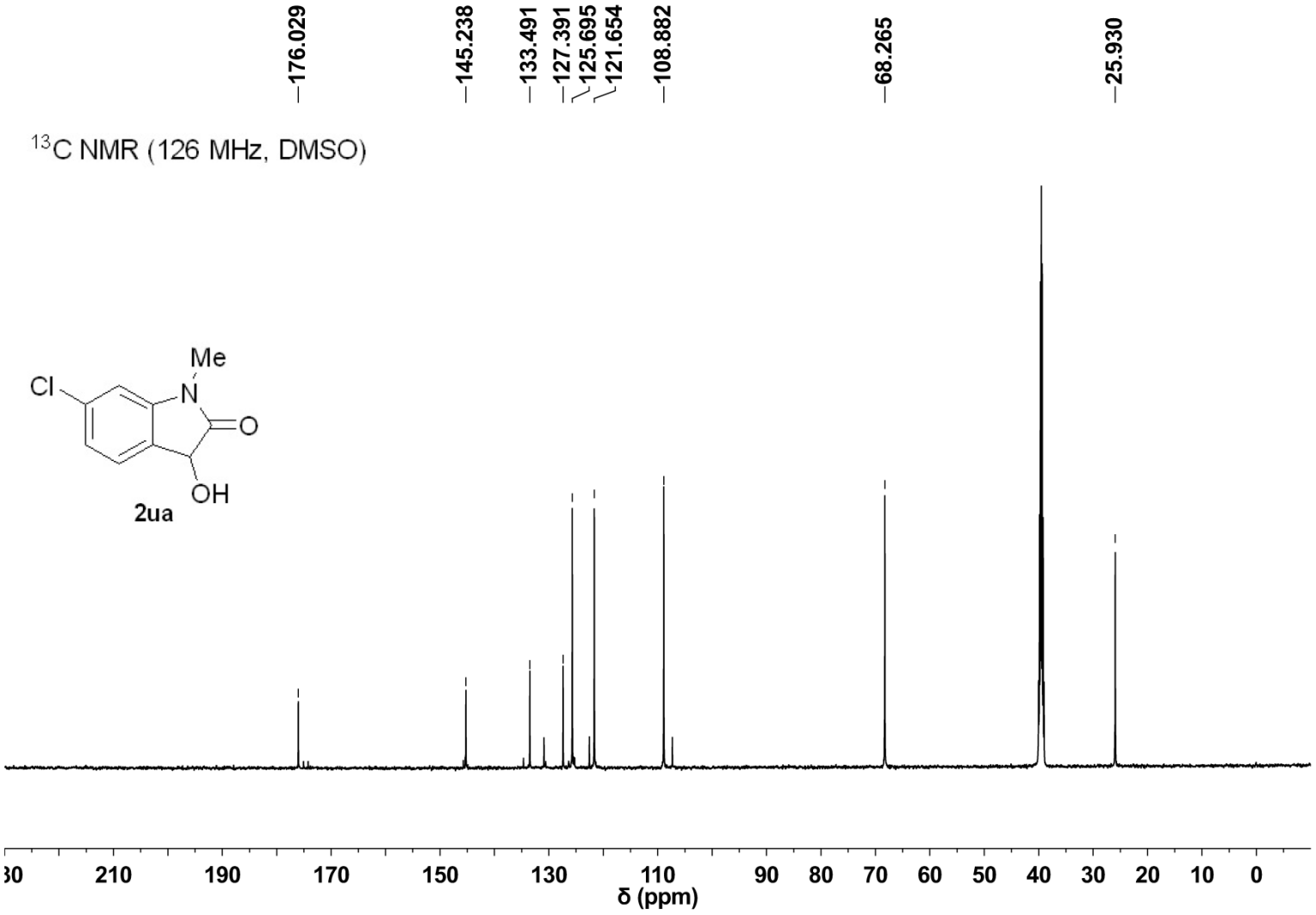

7.405
7.403
7.398
7.396
7.385
7.383
7.352
7.351
7.339
7.337
7.331
7.318
7.311
7.298
6.799
6.752
6.584
6.557
5.247
5.049
3.196
3.174
1H NMR (400 MHz, CDCl3)
Me
N
O
F
OH
+
2ta+2tb
(1:0.2)
0.22
1.08
1.23
1.03
0.21
1.00
0.20
3.01
0.60
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

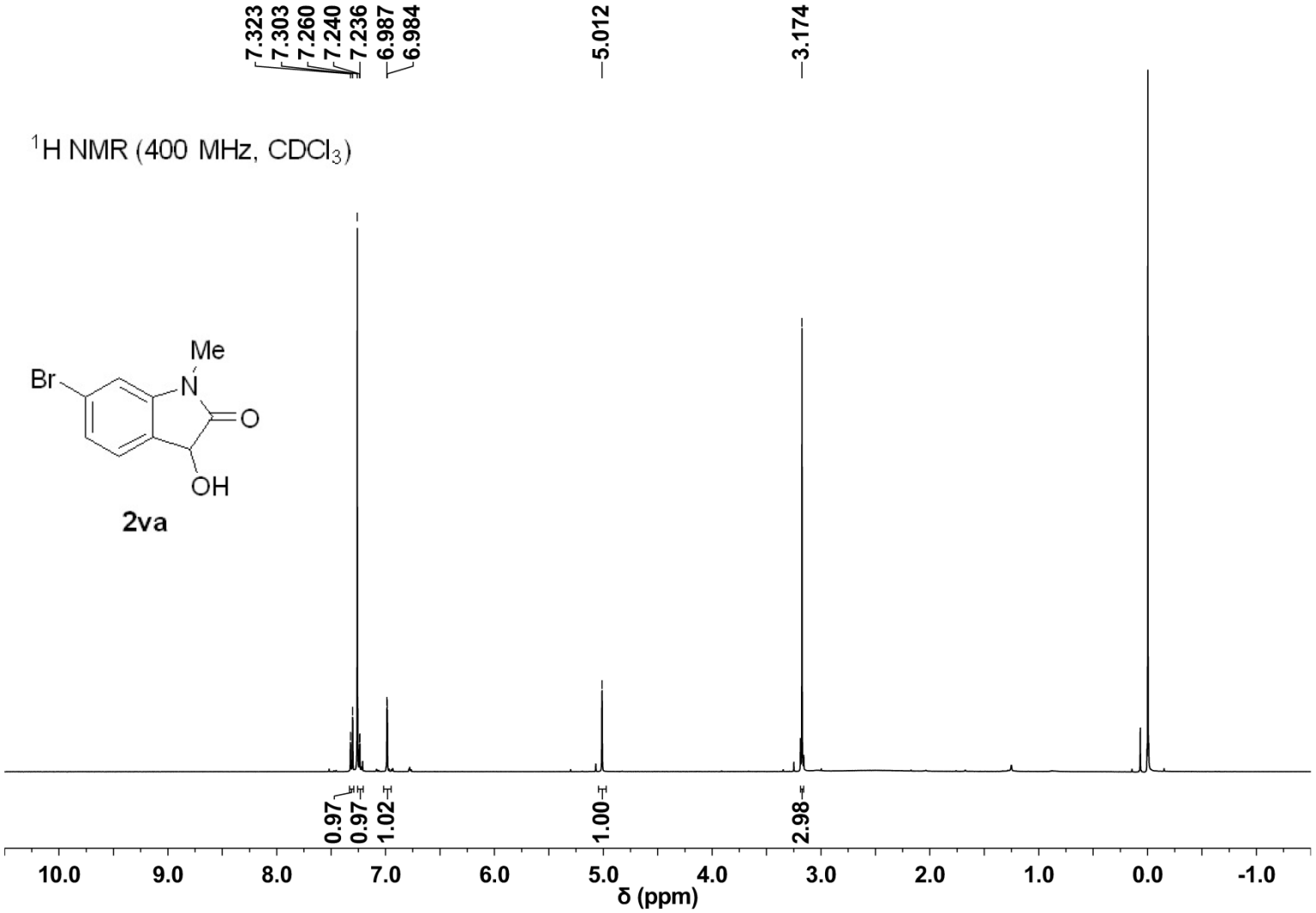

175.340
160.078
157.618
146.155
146.063
131.456
131.368
114.024
113.815
109.882
109.675
105.024
104.997
67.066
26.232
13C NMR (101 MHz, DMSO)
Me
F
N
O
OH
2ta
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

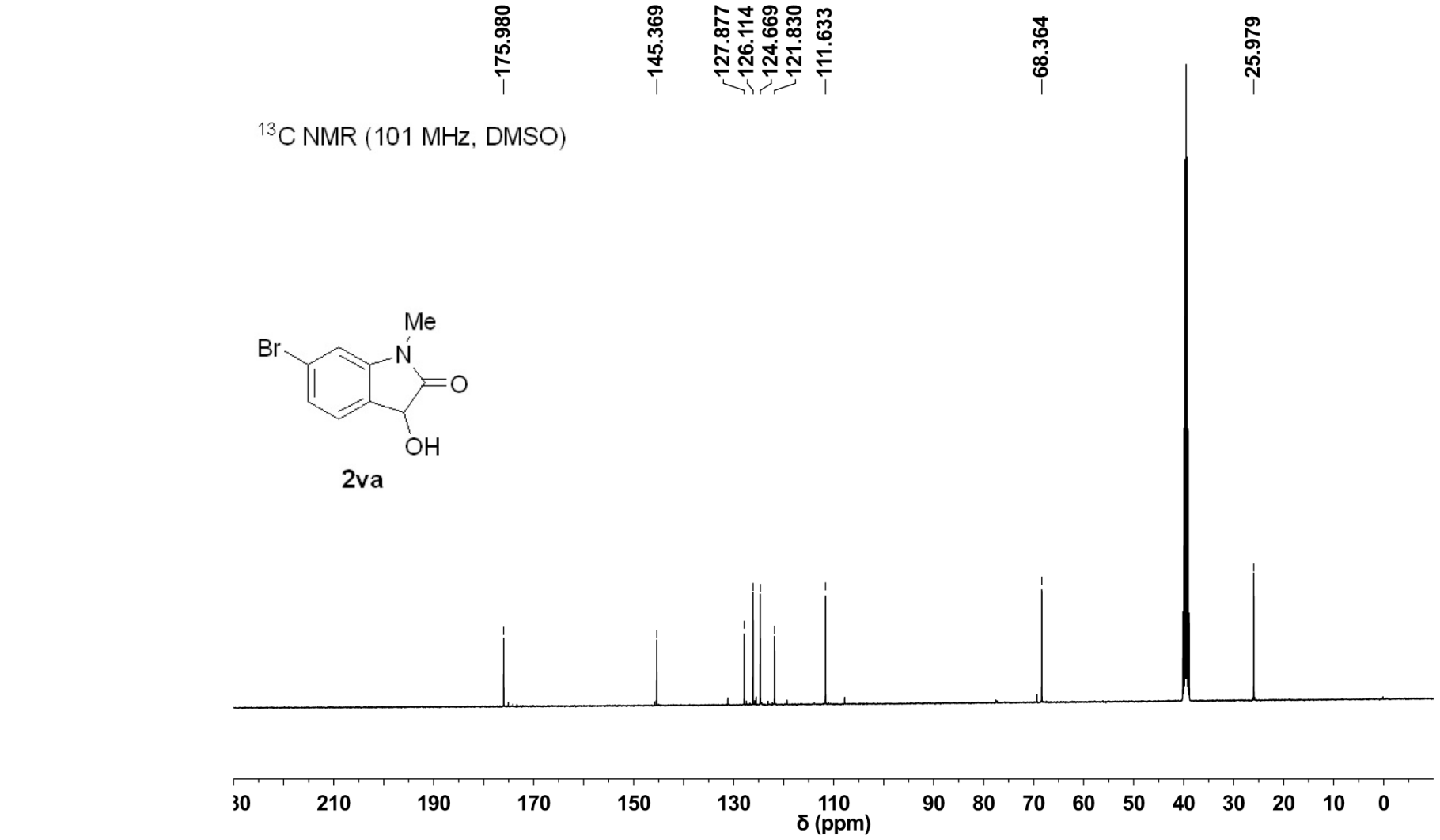


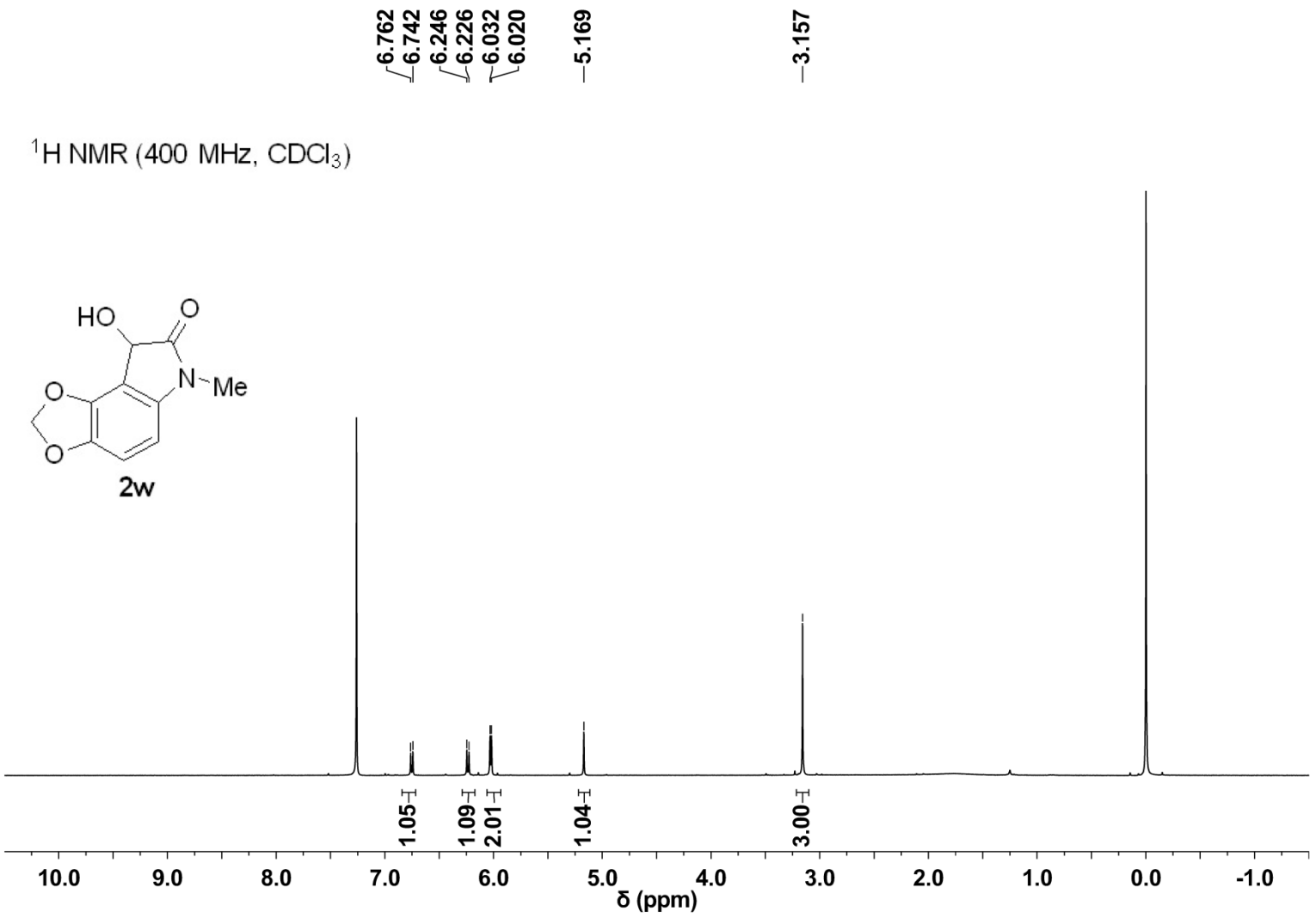

$^1$H NMR (400 MHz, $CDCl_3$)

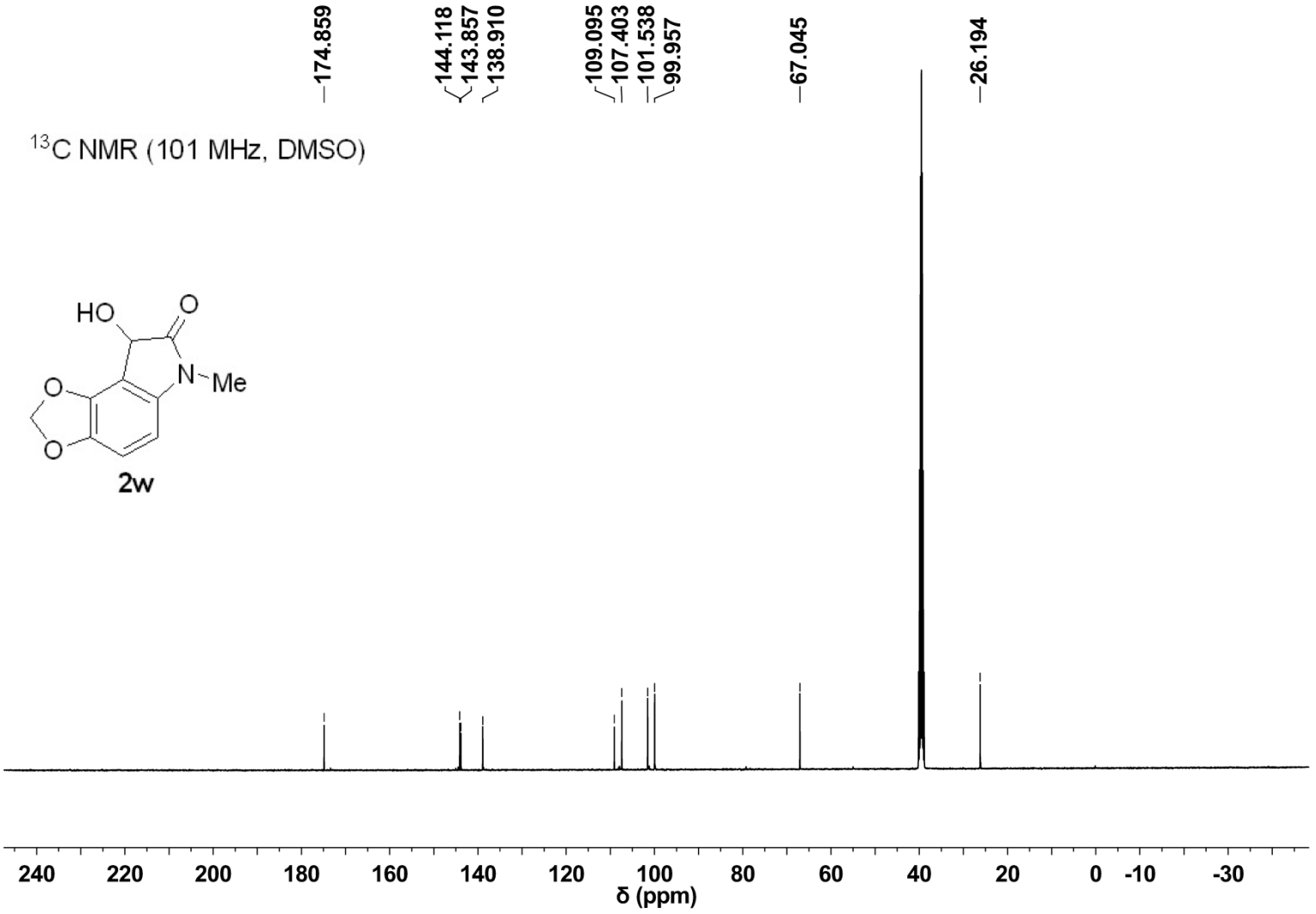


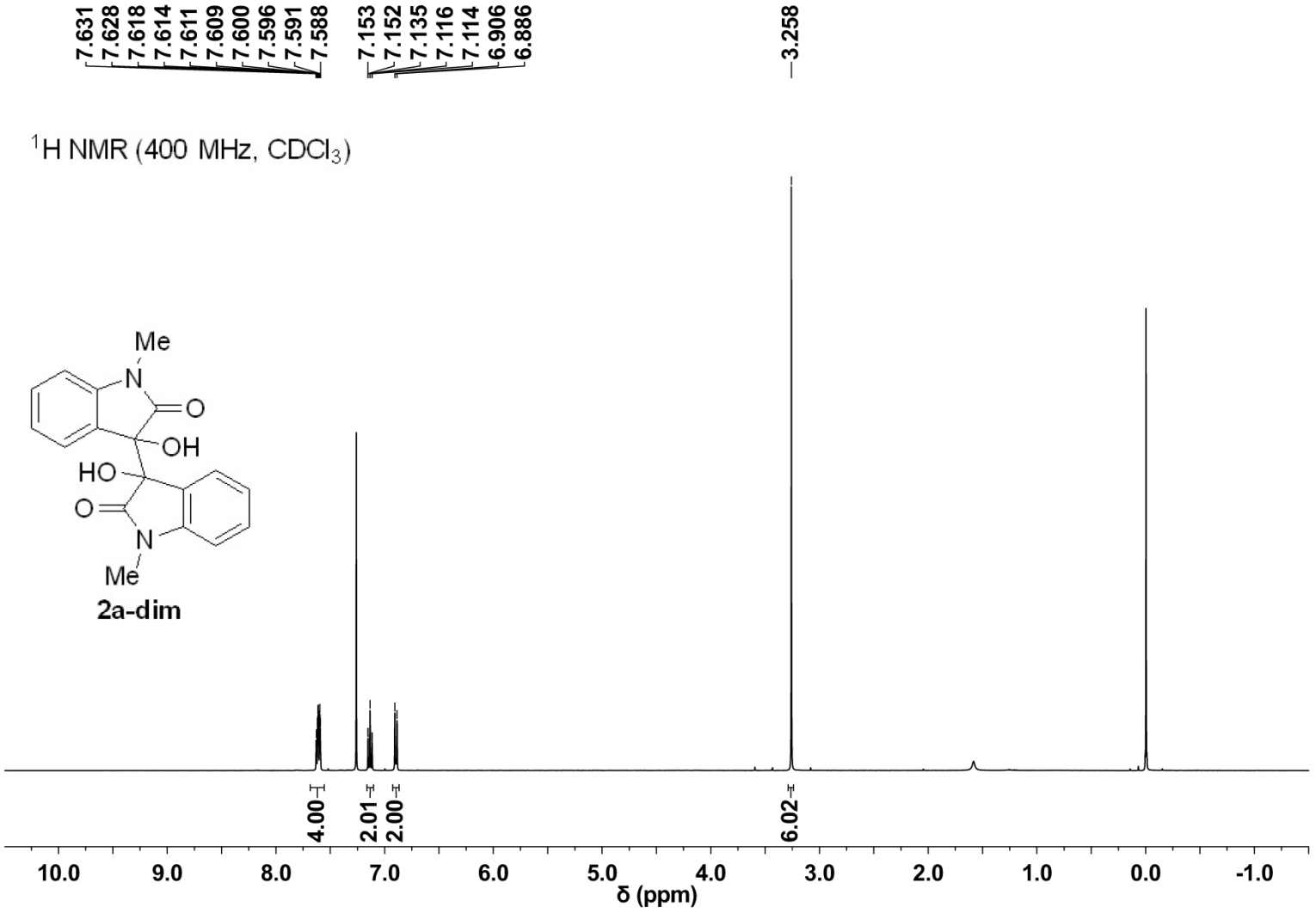

176.029
145.238
133.491
127.391
125.695
121.654
108.882
68.265
25.930
13C NMR (126 MHz, DMSO)
Me
Cl
N
O
OH
2ua
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

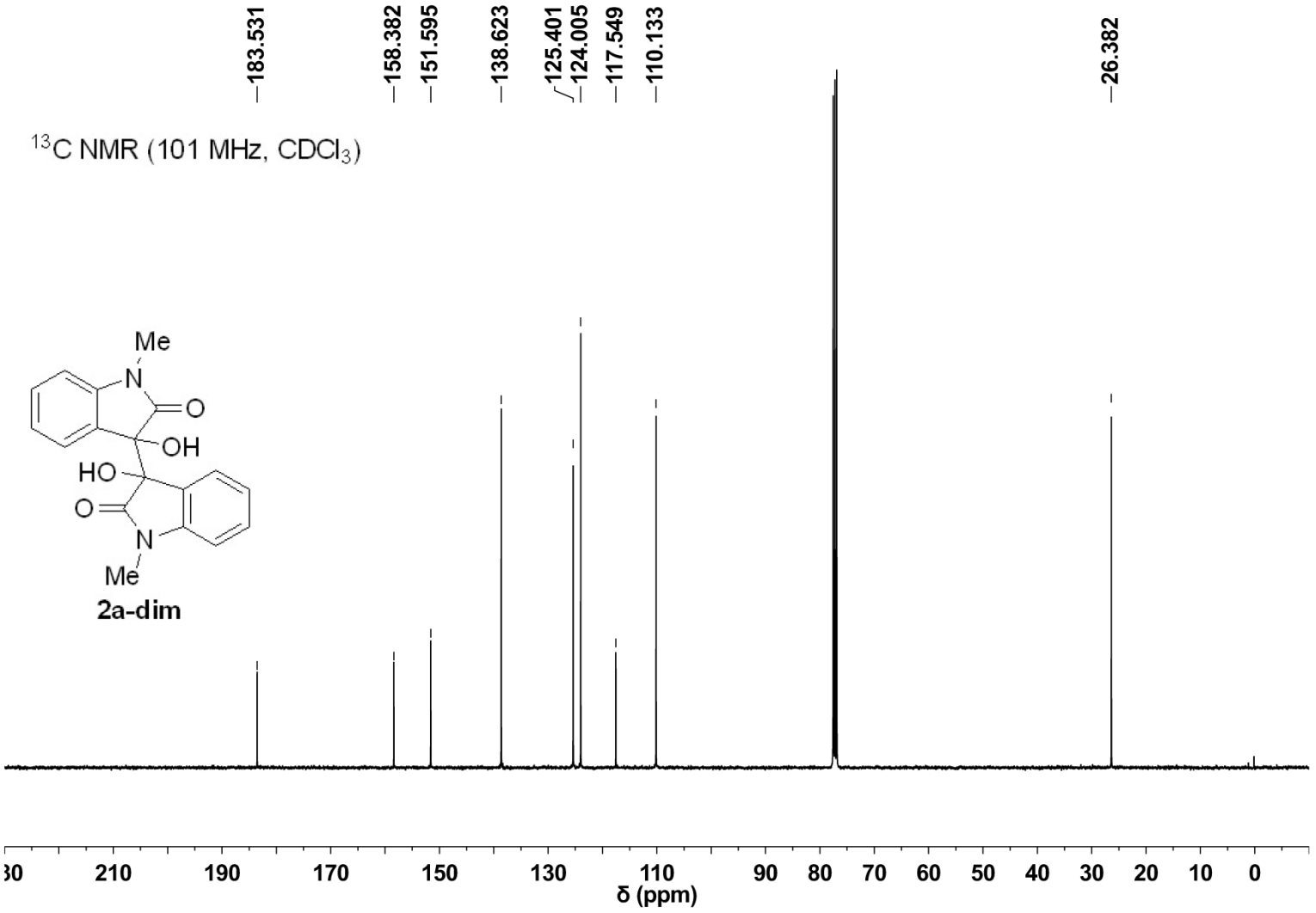

7.323
7.303
7.260
7.240
7.236
6.987
6.984
5.012
3.174
$^1$H NMR (400 MHz, $CDCl_3$)
Me
N
Br
O
OH
2va
0.97
0.97
1.02
1.00
2.98
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

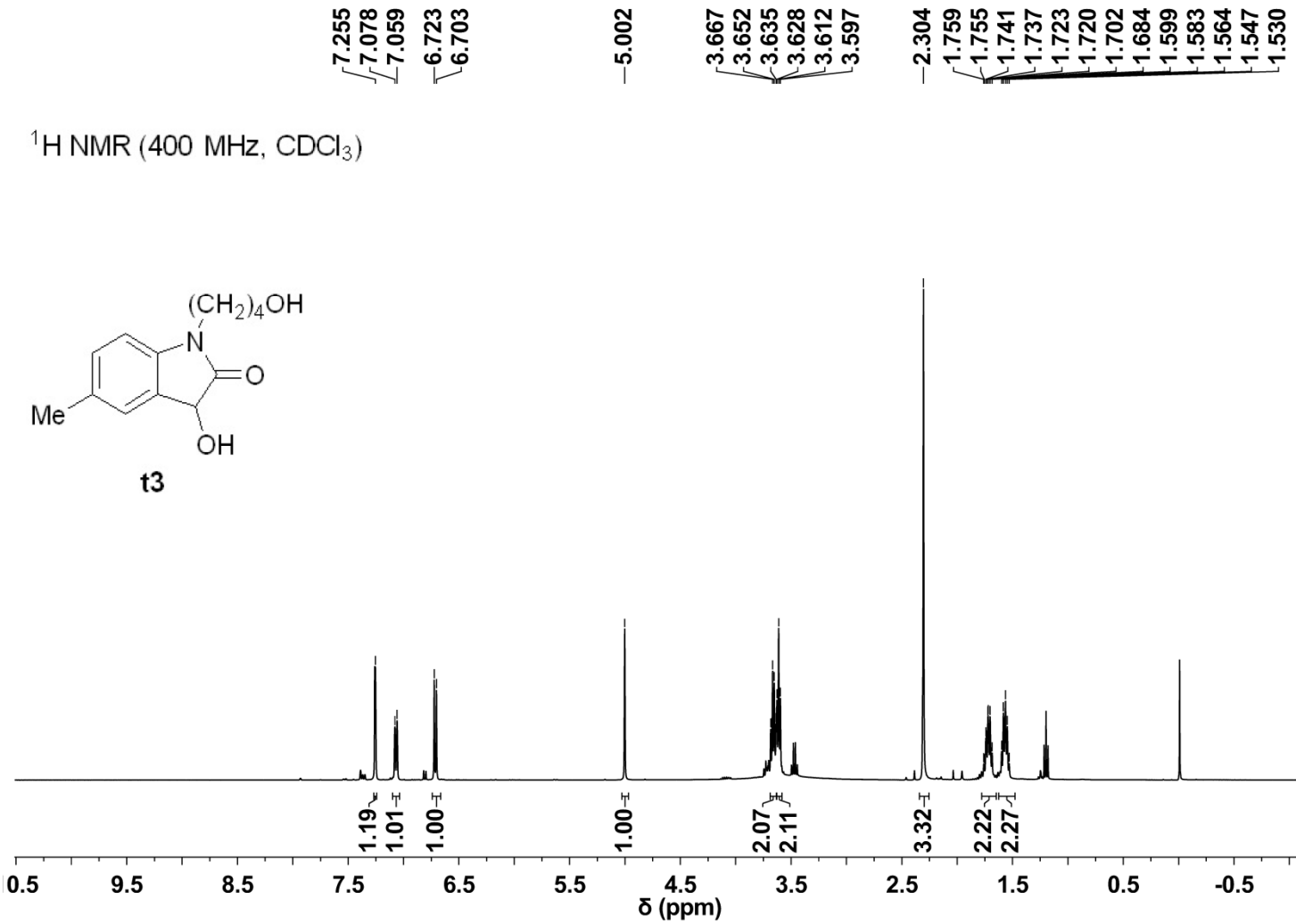

175.980
145.369
127.877
126.114
124.669
121.830
111.633
68.364
25.979
13C NMR (101 MHz, DMSO)
Me
Br
N
O
OH
2va
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)

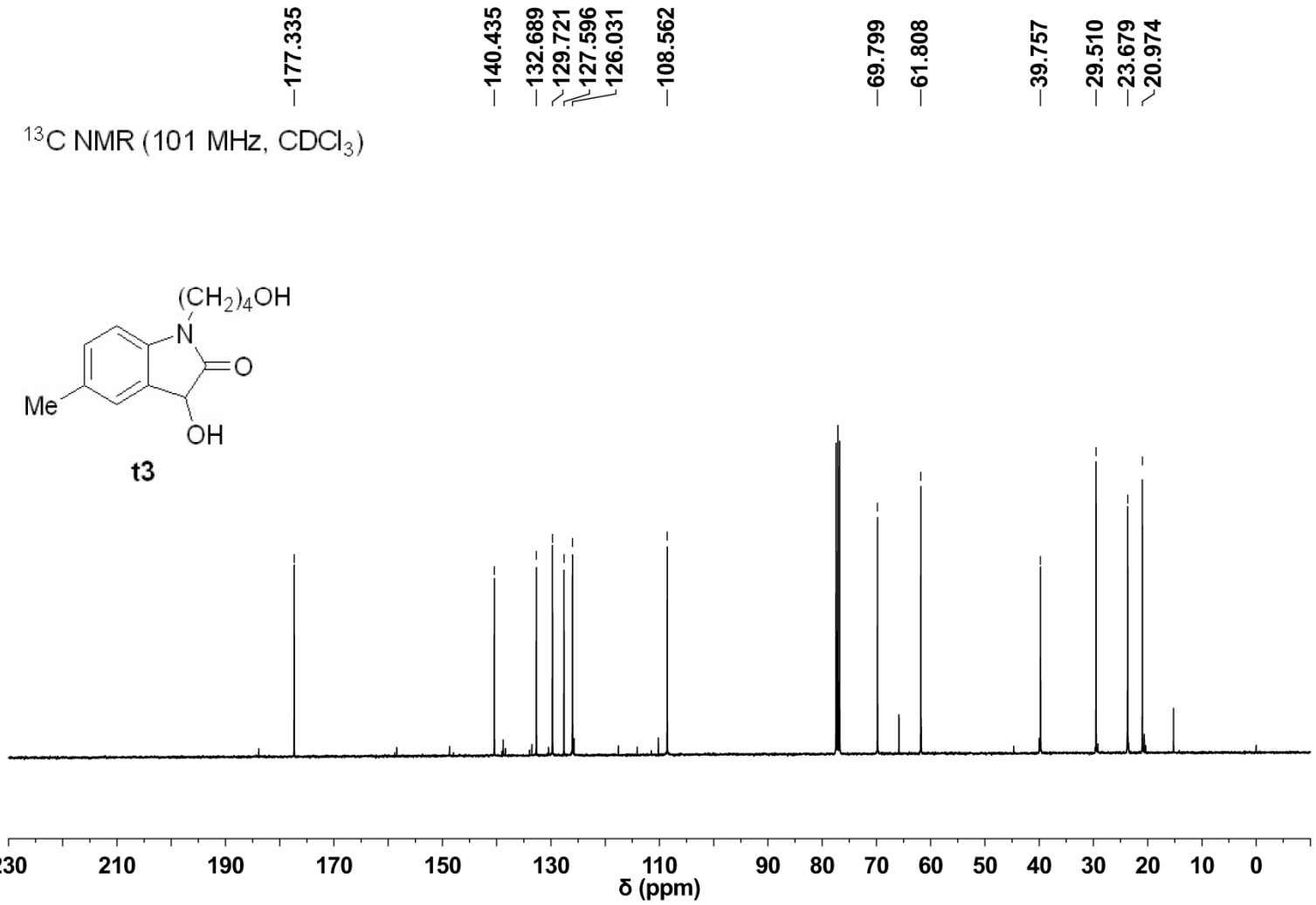

6.762
6.742
6.246
6.226
6.032
6.020
5.169
3.157
1H NMR (400 MHz, CDCl3)
HO
O
N
Me
O
O
2w
1.05
1.09
2.01
1.04
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)

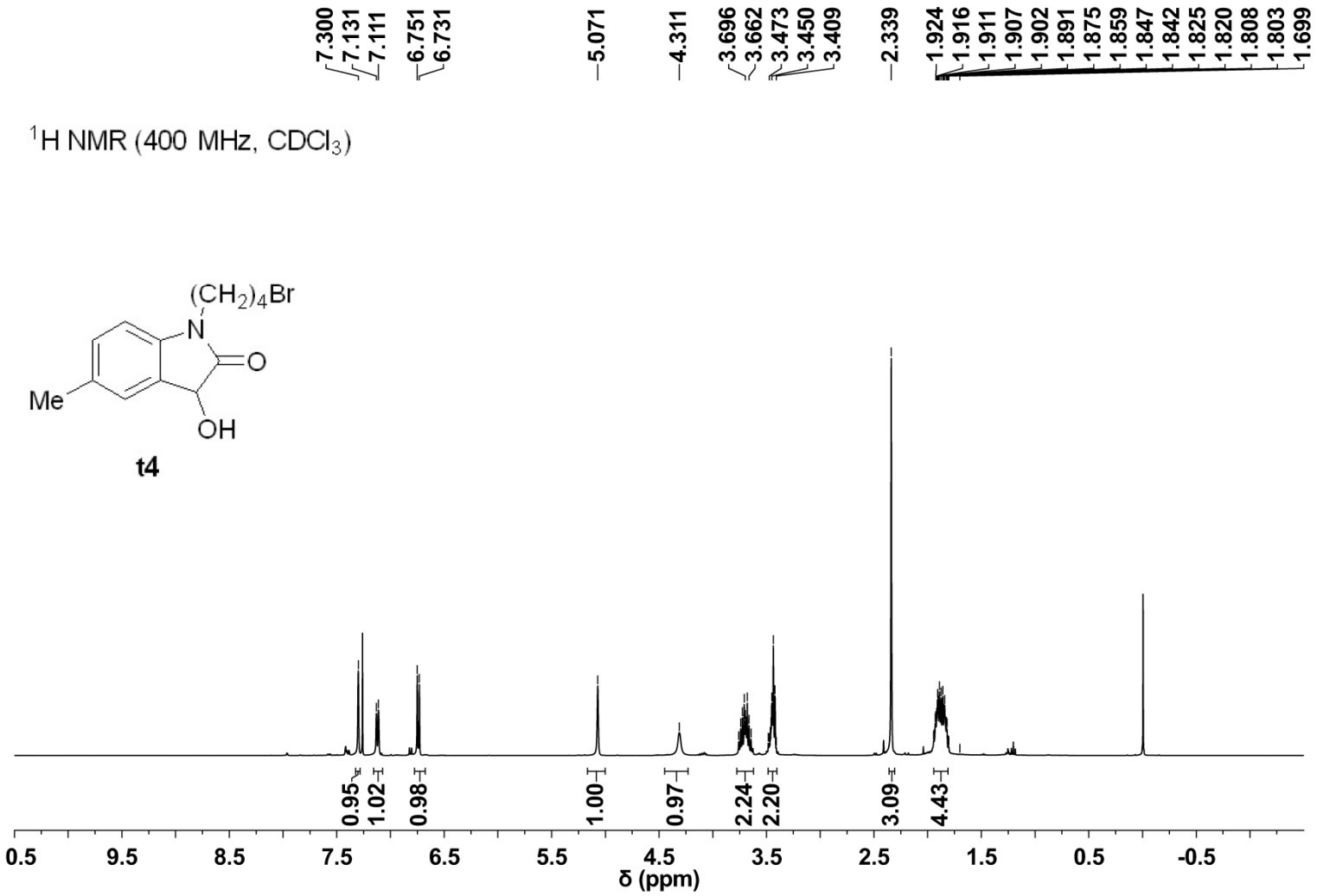

174.859
144.118
143.857
138.910
109.095
107.403
101.538
99.957
67.045
26.194
13C NMR (101 MHz, DMSO)
HO
O
N
Me
O
O
2w
240
220
200
180
160
140
120
100
80
60
40
20
0
-10
-30
δ (ppm)

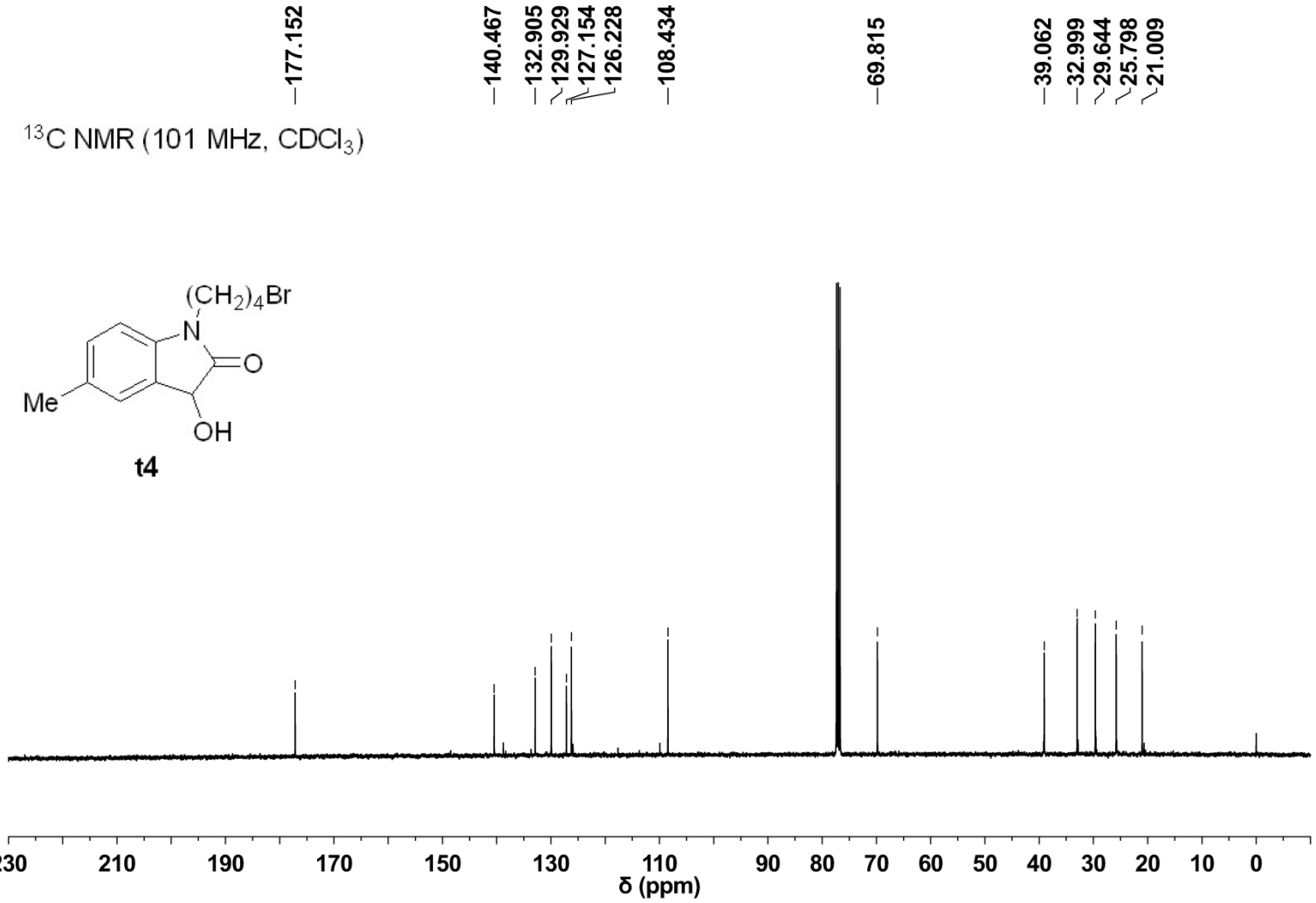

7.631
7.628
7.618
7.614
7.611
7.609
7.600
7.596
7.591
7.588
7.153
7.152
7.135
7.116
7.114
6.906
6.886
3.258
1H NMR (400 MHz, CDCl3)
Me
N
O
OH
HO
O
N
Me
2a-dim
4.00
2.01
2.00
6.02
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
δ (ppm)


183.531
158.382
151.595
138.623
125.401
124.005
117.549
110.133
26.382
13C NMR (101 MHz, CDCl3)
Me
N
O
OH
HO
O
N
Me
2a-dim
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)


7.255
7.078
7.059
6.723
6.703
5.002
3.667
3.652
3.635
3.628
3.612
3.597
2.304
1.759
1.755
1.741
1.737
1.723
1.720
1.702
1.684
1.599
1.583
1.564
1.547
1.530
$^1$H NMR (400 MHz, $CDCl_3$)
$(CH_2)_4OH$
N
O
Me
OH
t3
1.19
1.01
1.00
1.00
2.07
2.11
3.32
2.22
2.27
0.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)


177.335
140.435
132.689
129.721
127.596
126.031
108.562
69.799
61.808
39.757
29.510
23.679
20.974
13C NMR (101 MHz, CDCl3)
(CH2)4OH
N
O
Me
OH
t3
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)


7.300
7.131
7.111
6.751
6.731
5.071
4.311
3.696
3.662
3.473
3.450
3.409
2.339
1.924
1.916
1.911
1.907
1.902
1.891
1.875
1.859
1.847
1.842
1.825
1.820
1.808
1.803
1.699
$^1$H NMR (400 MHz, $CDCl_3$)
$(CH_2)_4Br$
N
O
Me
OH
t4
0.95
1.02
0.98
1.00
0.97
2.24
2.20
3.09
4.43
0.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
δ (ppm)


177.152
140.467
132.905
129.929
127.154
126.228
108.434
69.815
39.062
32.999
29.644
25.798
21.009
13C NMR (101 MHz, CDCl3)
(CH2)4Br
N
O
Me
OH
t4
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
δ (ppm)