Supplementary Information

Controllable Synthesis of 3-Choloro- and 3,3-Dicholoro-2-oxindoles via Hypervalent Iodine Mediate Chlorooxidation

Xinpeng Jiang, Liechao Yang, Wenlong Yang, Yu Zhu, Liyun Fang, Chuanming Yu *

** College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou, P.R. China Fax: (+86)571-88320867, E-mail: ycm@zjut.edu.cn*

Table of Contents

1. General information

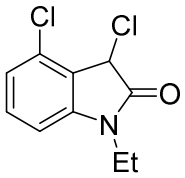
Unless otherwise stated, all reagents and solvents were obtained from commercial sources and without further purification. NMR spectra were recorded using Varian Mercury Plus 400 MHz or Bruker Avance III 600 MHz spectrometers. Chemical shifts of ^{1}H-NMR were reported relative to the solvent signal ($CDCl_3$: δ = 7.26 ppm; DMSO-d_6: δ =2.50 ppm). Chemical shifts of ^{13}C NMR were reported relative to the solvent signal ($CDCl_3$: δ = 77.00 ppm; DMSO-d_6: δ = 39.50 ppm). HRMS spectra were recorded on an electrospray ionization quadrupole time-of-flight (ESI-Q-TOF) mass spectrometer. The reactions were monitored by TLC visualized by UV (254 nm). Column chromatography was performed on silica gel (300-400 mesh).

2. Preparation of the starting materials

The 1-chloro-1,2-benziodoxol-3-(1*H*)-one **2a** were prepared according to literature procedures.[1] The (dichloroiodo)benzene **2b** were prepared according to literature procedures.[2] Substrates **1a-1k**, **1m**, **1n**, **1p**, **1q** and **1r** are known compounds and were prepared according to the literature reports.[3] Substrates **1o** was prepared according to the literature reports.[4]

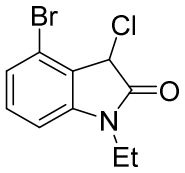
3. General preparation methods and characterization of 3 and 4.

1-Eethyl-4-chloro-3-chloroindoln-2-one (3a)



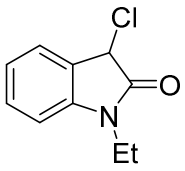
To the solution of DMF (1.5 mL) and TFA (1.0 mL) was added H_2O (0.2 mL). After the mixture cooled to room temperature, 4-chloro-1-ethyl-1*H*-indole (90 mg, 0.5mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (282 mg, 1.0mmol) were added successively, and then stirred at room temperature for 10 min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash column chromatography (hexane/ethyl acetate = 20:1 to 10:1) on silica gel to afford **3a** as a white solid (102 mg, 89%). M.p. = 124-125°C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.44 (t, J = 8.1 Hz, 1H), 7.17 – 7.13 (m, 2H), 5.68 (s, 1H), 3.76 – 3.69 (m, 2H), 1.16 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 144.6, 132.8, 131.7, 123.7, 123.2, 107.2, 50.7, 35.5, 12.4; HRMS (ESI) *m/z*: calcd for $C_{10}H_9Cl_2NNaO$ $[M+Na]^+$ 251.9953, found: 251.9944.

1-Eethyl-4-bromo-3-chloroindoln-2-one (3b)



Following the procedure for the preparation of **3a**, the reaction of 4-bromo-1-ethyl-1*H*-indole (102 mg, 0.455 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (257 mg, 0.91 mmol) afforded the product **3b** as a white solid (114 mg, 91%). M.p. = 118-119°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.26 – 7.21 (m, 2H), 6.82 (dd, J = 6.7, 1.9 Hz, 1H), 5.05 (s, 1H), 3.76 (q, J = 7.3 Hz, 2H), 1.28 (t, J = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.6, 144.7, 131.9, 126.6, 125.2, 121.2, 107.7, 52.1, 35.4, 12.4; HRMS (ESI) *m/z*: calcd for $C_{10}H_9BrClNNaO$ $[M+Na]^+$ 295.9448, found: 295.9451.

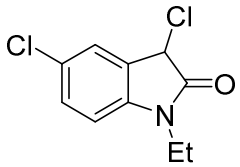
1-Eethyl-3-chloroindoln-2-one (3c)



Following the procedure for the preparation of **3a**, the reaction of 1-ethyl-1*H*-indole (73 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (282mg, 1.0 mmol) afforded the product **3c** as a

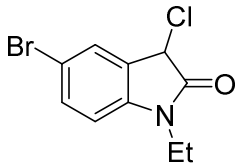
white solid (80 mg, 82%). M.p. =100-102°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.4 Hz, 1H), 7.36 (t, *J* = 7.8 Hz, 1H), 7.11 (t, *J* = 7.6 Hz, 1H), 6.87 (d, *J* = 7.9 Hz, 1H), 5.12 (s, 1H), 3.82 – 3.72 (m, 2H), 1.29 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.6, 142.9, 130.4, 125.9, 125.8, 123.1, 108.8, 51.5, 35.2, 12.4; HRMS (ESI) *m/z*: calcd for $C_{10}H_{11}ClNO$ $[M+H]^+$ 196.0524, found: 196.0530.

1-Eethyl-5-chloro-3-chloroindoln-2-one (3d)



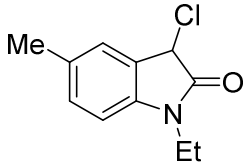
Following the procedure for the preparation of **3a**, the reaction of 5-chloro-1-ethyl-1*H*-indole (90 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3d** as a white solid (94 mg, 82%). M.p. = 119-120°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.34 – 7.31 (m, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 5.10 (s, 1H), 3.82 – 3.71 (m, 2H), 1.28 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.1, 141.4, 130.3, 128.5, 127.4, 126.2, 109.7, 50.9, 35.4, 12.3; HRMS (ESI) *m/z*: calcd for $C_{10}H_9Cl_2NNaO$ $[M+Na]^+$ 251.9953, found: 251.9951.

1-Eethyl-5-bromo-3-chloroindoln-2-one (3e)



Following the procedure for the preparation of **3a**, the reaction of 5-bromo-1-ethyl-1*H*-indole (112 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (282 mg, 1.0 mmol) afforded the product **3e** as a white solid (94 mg, 68%). M.p. = 136-137°C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.60 (s, 1H), 7.59 – 7.56 (m, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 5.67 (s, 1H), 3.70 (q, *J* = 7.2 Hz, 2H), 1.14 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 170.7, 142.1, 133.0, 128.3, 128.2, 114.3, 111.3, 51.0, 34.7, 12.2; HRMS (ESI) *m/z*: calcd for $C_{10}H_9BrClNNaO$ $[M+Na]^+$ 295.9448, found: 295.9449.

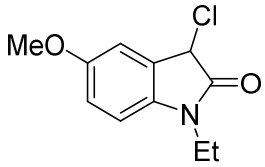
1-Eethyl-5-methyl-3-chloroindoln-2-one (3f)



Following the procedure for the preparation of **3a**, the reaction of 1-ethyl-5-methyl-1*H*-indole (80 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3f** as a white solid (81 mg, 77%). M.p. = 114-115°C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.24 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 6.99 (d, *J* = 8.0 Hz, 1H), 5.61 (s, 1H), 3.68 (q, *J* = 7.2 Hz, 2H), 2.29 (s, 3H), 1.14 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 171.0, 140.3, 132.0,

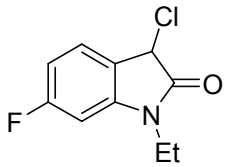
130.5, 126.1, 125.8, 109.0, 51.8, 34.5, 20.4, 12.3; HRMS (ESI) *m/z*: calcd for $C_{11}H_{12}ClNNaO$ $[M+Na]^+$ 232.0500; found：232.0505.

1-Eethyl-5-methoxy-3-chloroindoln-2-one (3g)



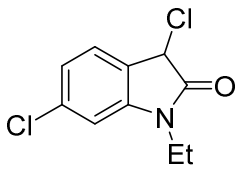
Following the procedure for the preparation of **3a**, the reaction of 1-ethyl-5-methoxy-1*H*-indole (88 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3g** as a yellow solid (76mg, 67%). M.p. = 94-95°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.03 (s, 1H), 6.88 (dd, *J* = 8.5, 2.2 Hz, 1H), 6.77 (d, *J* = 8.5 Hz, 1H), 5.09 (s, 1H), 3.80 (s, 3H), 3.77 – 3.69 (m, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.1, 156.1, 136.0, 126.8, 115.0, 112.5, 109.2, 55.7, 51.7, 35.1, 12.3; HRMS (ESI) *m/z*: calcd for $C_{11}H_{12}ClNNaO_2$ $[M+Na]^+$ 248.0449, found： 248.0448.

1-Eethyl-6-fluoro-3-chloroindoln-2-one (3h)



Following the procedure for the preparation of **3a**, the reaction of 1-ethyl-6-fluoro-1*H*-indole (82 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (297 mg, 1.05 mmol) afforded the product **3h** as a white solid (65 mg, 61%). M.p. = 99-100°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 – 7.35 (m, 1H), 6.78 (td, *J* = 9.4, 2.2 Hz, 1H), 6.61 (dd, *J* = 8.7, 2.1 Hz, 1H), 5.09 (s, 1H), 3.80 – 3.69 (m, 2H), 1.29 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.9, 164.2 (d, $J_{C\text{-}F}$ = 247.2 Hz), 144.7 (d, $J_{C\text{-}F}$ = 11.7 Hz), 127.2 (d, $J_{C\text{-}F}$ = 10.1 Hz), 121.2 (d, $J_{C\text{-}F}$ = 3.0 Hz), 109.4 (d, $J_{C\text{-}F}$ = 22.7 Hz), 97.6 (d, $J_{C\text{-}F}$ = 27.8 Hz), 50.9, 35.4, 12.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ -108.1; HRMS (ESI) *m/z*: calcd for $C_{10}H_9ClFNNaO$ $[M+Na]^+$ 236.0249, found: 236.0253.

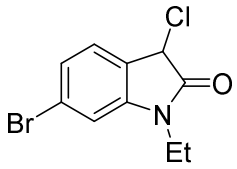
1-Eethyl-6-chloro-3-chloroindoln-2-one (3i)



Following the procedure for the preparation of **3a**, the reaction of 6-chloro-1-ethyl-1*H*-indole (90 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (282 mg, 1.0 mmol) afforded the product **3i** as a white solid (75 mg, 65%). M.p. = 104-105°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.0 Hz, 1H), 7.01 (dd, *J* = 8.0, 1.7 Hz, 1H), 6.79 (d, *J* = 1.7 Hz, 1H), 5.01 (s, 1H), 3.73 – 3.62 (m, 2H), 1.21 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.5, 144.1, 136.3, 126.7, 124.1, 123.0, 109.5, 50.8, 35.4, 12.3; HRMS (ESI) *m/z*: calcd for $C_{10}H_9Cl_2NNaO$ $[M+Na]^+$

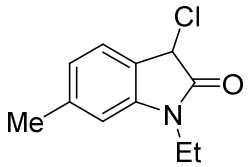
251.9953, found：251.9941.

1-Eethyl-6-bromo-3-chloroindoln-2-one (3j):



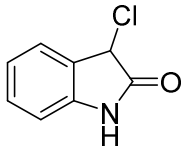
Following the procedure for the preparation of **3a**, the reaction of 6-bromo-1-ethyl-1*H*-indole (112 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3j** as a white solid (98 mg, 71%). M.p. = 119-120°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.9 Hz, 1H), 7.25 - 7.24 (m, 1H), 7.01 (s, 1H), 5.06 (s, 1H), 3.80 - 3.69 (m, 2H), 1.29 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.4, 144.2, 127.0, 126.0, 124.7, 124.2, 112.2, 50.9, 35.4, 12.4; HRMS (ESI) *m/z*: calcd for $C_{10}H_{10}BrClNO$ $[M+H]^+$ 273.9629, found: 273.9638.

1-Ethyl-6-methyl-3-chloroindoln-2-one (3k)



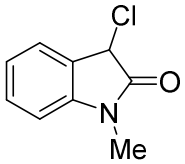
Following the procedure for the preparation of **3a**, the reaction of 1-ethyl-6-methyl-1*H*-indole (103 mg, 0.645 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (365 mg, 1.295 mmol) afforded the product **3k** as a yellow solid (107 mg, 79%). M.p. = 106-107°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.6 Hz, 1H), 6.91 (d, *J* = 7.5 Hz, 1H), 6.68 (s, 1H), 5.08 (s, 1H), 3.80 – 3.69 (m, 2H), 2.40 (s, 3H), 1.28 (td, *J* = 7.2, 1.0 Hz, 3H); ^{13}C NMR (150 MHz, CDCl3) δ 171.9, 142.9, 140.9, 125.4, 123.6, 122.8, 109.6, 51.5, 35.0, 21.9, 12.4; HRMS (ESI) *m/z*: calcd for $C_{11}H_{13}ClNO$ $[M+H]^+$ 210.0680, found: 210.0687.

3-Chloroindoln-2-one (3l):



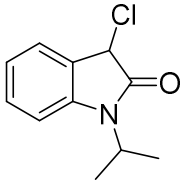
Following the procedure for the preparation of **3a**, the reaction of 1*H*-indole (59 mg, 0.5mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3l** as a brown solid (21 mg, 25%). M.p. = 134-136°C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 10.76 (br, 1H), 7.36 (d, *J* = 7.4 Hz, 1H), 7.29 (t, *J* = 7.7 Hz, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 5.57 (s, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 173.1, 142.4, 130.3, 126.5, 125.6, 122.3, 110.1, 52.2; ESI-MS *m/z*: 167.7 $[M+H]^+$, the spectral data matched with those previously reported.[5]

1-Methyl-3-chloroindoln-2-one (3m):



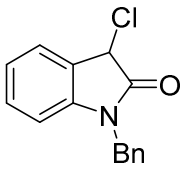
Following the procedure for the preparation of **3a**, the reaction of 1-methyl-1*H*-indole (65 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3m** as a white solid (63 mg, 69%). M.p. = 93-94°C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.35 (d, *J* = 7.4 Hz, 1H), 7.29 (t, *J* = 7.8 Hz, 1H), 7.05 (t, *J* = 7.6 Hz, 1H), 6.77 (d, *J* = 7.8 Hz, 1H), 5.05 (s, 1H), 3.15 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 172.0, 143.8, 130.4, 125.6, 125.5, 123.3, 108.6, 51.4, 26.6; ESI-MS *m/z*: 181.7 $[M+H]^+$, the spectral data matched those previously reported. [6]

1-Isopropyl-3-chloroindoln-2-one (3n)



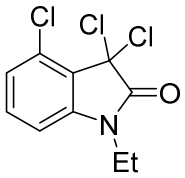
Following the procedure for the preparation of **3a**, the reaction of 1-isopropyl-1*H*-indole (80 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) afforded the product **3n** as a yellow oil (100 mg, 95%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.4 Hz, 1H), 7.33 (t, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 5.08 (s, 1H), 4.63 – 4.55 (m, 1H), 1.49 (d, *J* = 7.1 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.6, 142.5, 130.1, 126.0, 125.8, 122.7, 110.2, 51.6, 44.4, 19.1; HRMS (ESI) *m/z*: calcd for $C_{11}H_{13}ClNO$ $[M+H]^+$ 210.0680, found: 210.0681.

1-Bn-3-chloroindoln-2-one (3o)



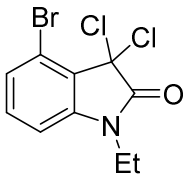
Following the procedure for the preparation of **3a**, the reaction of 1-benzyl-1*H*-indole (116 mg, 0.56 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (316 mg, 1.12 mmol) afforded the product **3o** as a yellow solid (111 mg, 77%). M.p. = 143-145°C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.45 (d, *J* = 7.4 Hz, 1H), 7.37 – 7.25 (m, 6H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 7.9 Hz, 1H), 5.81 (s, 1H), δ 4.92 (d, *J* = 2.9 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 171.8, 142.7, 135.8, 130.3, 128.7, 127.6, 127.2, 125.8, 125.5, 123.1, 109.8, 51.6, 42.9; ESI-MS *m/z*: 280.0 $[M+Na]^+$, the spectral data matched those previously reported. [6]

1-Eethyl-4-chloro-3,3-dichloro-2-oxindole (4a)



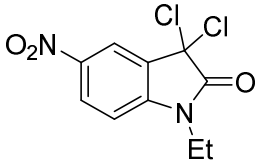
To the solution of 1,4-dioxane (3.0 mL) and H_2O (0.2 mL) was added 4-chloro-1-ethyl-1*H*-indole (92 mg, 0.512 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (434 mg, 1.536 mmol) successively. The reaction mixture was then stirred at 80 °C for 30min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash column chromatography on silica gel (hexane/ethyl acetate = 30:1 to 20:1) to afford **4a** as a yellow solid (128 mg, 95%).M.p. = 116-117 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.55 (t, J = 8.1 Hz, 1H), 7.28 (dd, J = 8.1, 2.5 Hz, 2H), 3.80 (q, J = 7.1 Hz, 2H), 1.18 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, DMSO-d_6) δ 166.8, 141.6, 134.0, 131.1, 124.7, 123.8, 109.4, 73.6, 40.1, 35.7, 12.0; HRMS (ESI) *m/z*: calcd for $C_{10}H_8Cl_3NNaO$ $[M+Na]^+$ 285.9564, found: 285.9564.

1-Eethyl-4-bromo-3,3-dichloro-2-oxindole (4b)



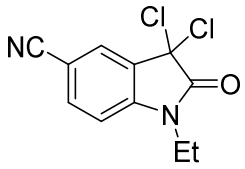
Following the procedure for the preparation of **4a**, the reaction of 4-bromo-1-ethyl-1*H*-indole (112 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (423mg, 1.5 mmol) afforded the product **4b** as a yellow solid (153 mg, 99%). M.p. = 132-133 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.47 – 7.41 (m, 2H), 7.30 (d, J = 6.6 Hz, 1H), 3.79 (q, J = 7.2 Hz, 2H), 1.18 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 166.9, 141.9, 134.0, 127.9, 125.3, 119.8, 109.8, 75.0, 35.6, 12.0; HRMS (ESI) *m/z*: calcd for $C_{10}H_8BrCl_2NNaO$ $[M+Na]^+$ 329.9059, found: 329.9058.

1-Eethyl-5-nitro-3,3-dichloro-2-oxindole (4c)



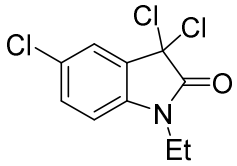
Following the procedure for the preparation of **4a**, the reaction of 1-ethyl-5-nitro-1*H*-indole (95 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afford the product **4c** as a yellow solid (127 mg, 92%). M.p. = 111-112 °C. 1H NMR (600 MHz, DMSO-*d*6) δ 8.55 (d, J = 2.2 Hz, 1H), 8.44 (dd, J = 8.8, 2.3 Hz, 1H), 7.56 (d, J = 8.8 Hz, 1H), 3.89 (q, J = 7.2 Hz, 2H), 1.22 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 167.9, 145.2, 143.8, 129.0, 128.8, 120.3, 111.2, 72.7, 36.1, 12.1; HRMS (ESI) *m/z*: calcd for $C_{10}H_8Cl_2N_2O_3$ $[M+Na]^+$ 296.9804, found: 296.9817.

1-Eethyl-5-cyan-3,3-dichloro-2-oxindole (4d)



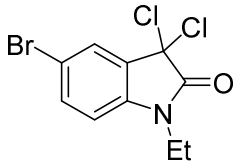
Following the procedure for the preparation of **4a**, the reaction of 1-ethyl-1*H*-indole-5-carbonitrile (76 mg, 0.446 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (378 mg, 1.338 mmol) afford the product **4d** as a yellow solid (109 mg, 96%). M.p. = 148-149 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.91 (s, 1H), 7.74 (dd, *J* = 8.2, 1.4 Hz, 1H), 7.02 (d, *J* = 8.2 Hz, 1H), 3.85 (q, *J* = 7.3 Hz, 2H), 1.35 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.0, 143.5, 136.5, 130.4, 128.6, 117.8, 109.9, 107.5, 72.4, 36.1, 12.2; HRMS (EI) *m/z*: calcd for $C_{11}H_8Cl_2N_2O$ $[M]^+$ 254.0014, found: 254.0020.

1-Eethyl-5-chloro-3,3-dichloro-2-oxindole (4e)



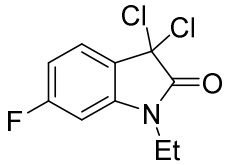
Following the procedure for the preparation of **4a**, the reaction of 5-chloro-1-ethyl-1*H*-indole (99 mg, 0.552 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (468 mg, 1.656 mmol) afforded the product **4e** as a yellow solid (136 mg, 93%). M.p. = 98-99 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.87 (d, *J* = 2.0 Hz, 1H), 7.58 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.31 (d, *J* = 8.5 Hz, 1H), 3.78 (q, *J* = 7.2 Hz, 2H), 1.17 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 167.2, 138.4, 132.4, 129.9, 128.1, 124.8, 112.2, 73.7, 35.5, 12.0; HRMS (ESI) *m/z*: calcd for $C_{10}H_8Cl_3NNaO$ $[M+Na]^+$ 285.9564, found: 285.9566.

1-Eethyl-5- bromo-3,3-dichloro-2-oxindole (4f)



Following the procedure for the preparation of **4a**, the reaction of 5-bromo-1-ethyl-1*H*-indole (112 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the product **4f** as a white solid (143 mg, 93%). M.p. = 120-121 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.96 (d, *J* = 2.0 Hz, 1H), 7.71 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.25 (d, *J* = 8.5 Hz, 1H), 3.78 (q, *J* = 7.2 Hz, 2H), 1.17 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 167.1, 138.8, 135.2, 130.1, 127.5, 115.6, 112.6, 73.6, 35.5, 12.0; HRMS (ESI) *m/z*: calcd for $C_{10}H_8BrCl_2NNaO$ $[M+Na]^+$ 329.9059, found: 329.9038.

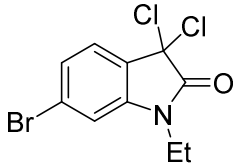
1-Eethyl-6-fluoro-3,3-dichloro-2-oxindole (4g)



Following the procedure for the preparation of **4a**, the reaction of 1-ethyl-6-fluoro-1*H*-indole (82 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the

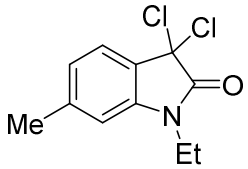
product **4g** as a white solid (96 mg, 77%). M.p. = 78-79 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.79 (dd, J = 8.4, 5.3 Hz, 1H), 7.31 (dd, J = 9.3, 2.2 Hz, 1H), 7.07 – 7.01 (m, 1H), 3.78 (q, J = 7.2 Hz, 2H), 1.18 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 167.8, 164.5 (d, $J_{C\text{-}F}$ = 247.1 Hz)，141.7 (d, $J_{C\text{-}F}$ = 12.8 Hz), 126.9 (d, $J_{C\text{-}F}$ = 10.5 Hz), 124.3 (d, $J_{C\text{-}F}$ = 3.0 Hz), 110.6 (d, $J_{C\text{-}F}$ = 23.4 Hz), 99.3 (d, $J_{C\text{-}F}$ = 28.7 Hz), 73.8, 35.6, 12.1; ^{19}F NMR (376 MHz, DMSO-d_6) δ -105.4; HRMS (EI) *m/z*: calcd for $C_{10}H_8Cl_2FNO$ $[M]^+$ 246.9976, found: 246.9975.

1-Eethyl-6-bromo-3,3-dichloro-2-oxindole (4h)



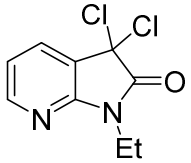
Following the procedure for the preparation of **4a**, the reaction of 6-bromo-1-ethyl-1*H*-indole (112 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the product **4h** as a white solid (151 mg, 98%). M.p. = 98-99 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 7.67 (d, J = 8.1 Hz, 1H), 7.60 (d, J = 1.6 Hz, 1H), 7.43 (dd, J = 8.1, 1.6 Hz, 1H), 3.79 (q, J = 7.2 Hz, 2H), 1.17 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 167.4, 141.0, 127.4, 126.9, 126.4, 125.7, 113.6, 73.8, 35.5, 12.1; HRMS (ESI) *m/z*: calcd for $C_{10}H_{12}BrCl_2N_2O$ $[M+NH_4]^+$ 324.9505, found: 324.9494.

1-Eethyl-6-methyl -3,3-dichloro-2-oxindole (4i)



Following the procedure for the preparation of **4a**, the reaction of 1-ethyl-6-methyl-1*H*-indole (97 mg, 0.61 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (517 mg, 1.83 mmol) afforded the product **4i** as a brown solid (138 mg, 93%). M.p. = 103-104 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.51 (d, J = 7.7 Hz, 1H), 6.98 (d, J = 7.5 Hz, 1H), 6.71 (s, 1H), 3.78 (q, J = 7.2 Hz, 2H), 2.42 (s, 3H), 1.31 (t, J = 7.2 Hz, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.8, 142.7, 139.8, 126.6, 124.7, 124.5, 109.9, 74.5, 35.6, 22.0, 12.3; HRMS (ESI) *m/z*: calcd for $C_{11}H_{12}Cl_2NO$ $[M+H]^+$ 244.0296, found: 244.0298.

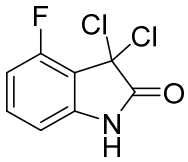
3,3-Dichloro-1-H-pyrrolo[3,2-b]pyridin-2-one (4j)



Following the procedure for the preparation of **4a**, the reaction of 1-ethyl-1*H*-pyrrolo[2,3-b]pyridine (73 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the product **4j** as an colorless oil (106 mg, 91%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 (dd, J = 5.3, 1.5 Hz, 1H), 7.85 (dd, J = 7.5, 1.6 Hz, 1H), 7.10 (dd, J = 7.5, 5.3

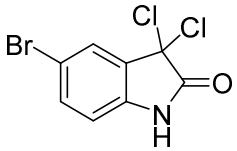
Hz, 1H), 3.92 (q, J = 7.2 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 153.3, 150.5, 132.4, 124.2, 119.5, 73.0, 35.1, 12.7; HRMS (ESI) *m/z*: calcd for $C_9H_9Cl_2N_2O$ $[M+H]^+$ 231.0086, found: 231.0095.

4-Fluoro-3,3-dichloro-2-oxindole (4k)



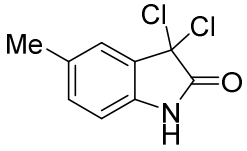
Following the procedure for the preparation of **4a**, the reaction of 4-fluoro-1*H*-indole (68 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the product **4k** as a white solid (99 mg, 90%). M.p. = 168-169 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.63 (br, 1H), 7.51 – 7.47 (m, 1H), 7.00 (t, J = 9.1 Hz, 1H), 6.83 (d, J = 7.8 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 168.5, 158.0 (d, $J_{C\text{-}F}$ = 252.3 Hz), 141.0 (d, $J_{C\text{-}F}$ = 6.0 Hz), 135.0 (d, $J_{C\text{-}F}$ = 9.2 Hz), 115.1 (d, $J_{C\text{-}F}$ = 17.0 Hz), 110.7 (d, $J_{C\text{-}F}$ = 18.9 Hz), 107.7 (d, $J_{C\text{-}F}$ = 3.0 Hz), 72.1; ^{19}F NMR (565 MHz, $CDCl_3$) δ -114.7; HRMS (ESI) *m/z*: calcd for $C_8H_3Cl_2FNO[M\text{-}H]^-$ 217.9581, found: 217.9581.

5-Bromo-3,3-dichloro-2-oxindole (4l)



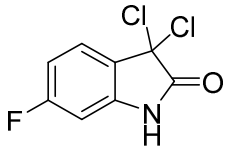
Following the procedure for the preparation of **4a**, the reaction of 5-bromo-1*H*-indole (104 mg, 0.53 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (449 mg, 1.59 mmol) afforded the product **4l** as a yellow solid (148 mg, 99%). M.p. = 209-210 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.49 (br, 1H), 7.88 (dd, J = 4.3, 2.1 Hz, 1H), 7.62 – 7.57 (m, 1H), 6.94 (d, J = 8.4 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 168.7, 138.5, 135.2, 130.8, 127.6, 114.9, 113.4, 74.2; ESI-MS *m/z*: 280.3$[M+H]^+$, the spectral data matched those previously reported. [7]

5-Methyl-3,3-dichloro-2-oxindole (4m)



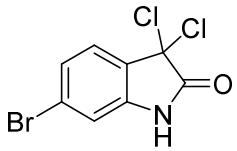
Following the procedure for the preparation of **4a**, the reaction of 5-methyl-1*H*-indole (66 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the product **4m** as a white solid (94 mg, 87%). M.p. = 194-195 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.22 (br, 1H), 7.46 (s, 1H), 7.21 (d, J = 7.8 Hz, 1H), 6.85 (d, J = 8.0 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 169.2, 136.7, 133.1, 132.8, 128.9, 125.2, 111.1, 75.5, 20.5; ESI-MS *m/z*: 253.8 $[M+K]^+$, the spectral data matched with those previously reported. [7]

6- Fluoro-3,3-dichloro-2-oxindole (4n)



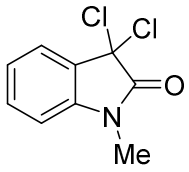
Following the procedure for the preparation of **4a**, the reaction of 6-fluoro-1*H*-indole (83 mg, 0.614 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (520 mg, 1.842 mmol) afforded the product **4n** as a yellow solid (130 mg, 96%). M.p. = 174-175 °C. 1H NMR (600 MHz, DMSO-*d*6) δ 11.55 (br, 1H), 7.74 (dd, *J* = 8.4, 5.3 Hz, 1H), 7.02 – 6.97 (m, 1H), 6.83 (dd, *J* = 8.9, 2.4 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-*d6*) δ 169.3, 164.1 (d, *JC-F* = 247.1 Hz), 141.2 (d, *JC-F* = 12.8 Hz), 127.1 (d, *JC-F* = 10.7 Hz),125.0 (d, *JC-F* =2.9 Hz),110.3 (d, *JC-F* = 23.1 Hz), 99.5 (d,*JC-F* =27.5 Hz),74.3; 19F NMR (565 MHz, $CDCl_3$) δ -114.7; HRMS (ESI) *m/z*: calcd for $C_8H_3Cl_2FNO[M-H]^-$ 217.9581, found: 217.9574.

6-Bromo-3,3-dichloro-2-oxindole (4o)



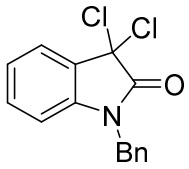
Following the procedure for the preparation of **4a**, the reaction of 6-bromo-1*H*-indole (83 mg, 0.423 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (358 mg, 1.27 mmol) afforded the product **4o** as a yellow solid (114 mg, 95%). M.p. = 184-185 °C. ^{1}H NMR (600 MHz, DMSO-d_6) δ 11.52 (br, 1H), 7.62 (d, *J* = 8.1 Hz, 1H), 7.36 (dd, *J* = 8.1, 1.6 Hz, 1H), 7.14 (d, *J* = 1.6 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 168.9, 140.7, 128.0, 126.6, 126.4, 125.2, 114.2, 74.3; HRMS (ESI) *m/z*: calcd for $C_8H_3BrCl_2NO$ $[M-H]^-$ 277.8781, found: 277.8788.

1-Methyl-3,3-dichloro-2-oxindole (4p)



Following the procedure for the preparation of **4a**, the reaction of 1-methyl-1*H*-indole (66 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5 mmol) afforded the product **4p** as a white solid (102 mg, 94%). M.p. = 144-145 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.5 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 7.9 Hz, 1H), 3.27 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.8, 140.6, 131.9, 129.2, 124.7, 124.2, 109.1, 74.2, 27.0; ESI-MS *m/z*: 216.2 $[M+H]^+$, the spectral data matched with those previously reported. [8]

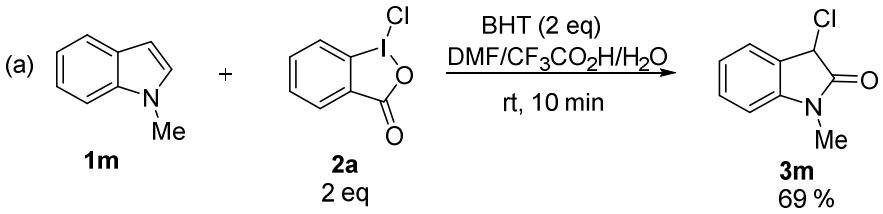
1-Bn-3,3-dichloro-2-oxindole (4q)



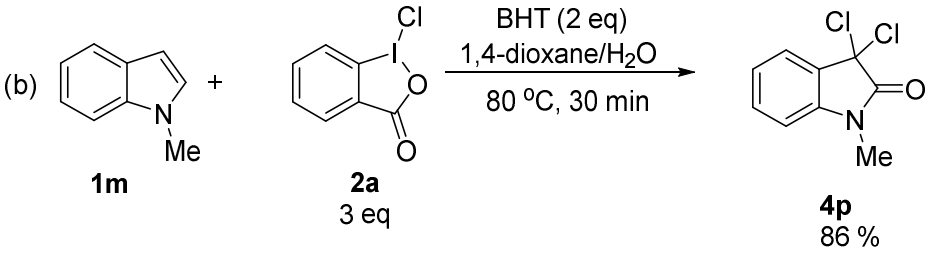
Following the procedure for the preparation of **4a**, the reaction of 1-benzyl-1*H*-indole (136 mg, 0.656 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (555 mg, 1.968 mmol) afforded the product **4q** as a white solid (180 mg, 94%). M.p. = 124-125 °C. ^{1}H NMR (600 MHz, DMSO-d_6)δ 7.74 (d, J = 7.0 Hz, 1H)., 7.44 (td, J = 7.9, 1.1 Hz, 1H), 7.39 – 7.27 (m, 5H), 7.23 (t, J = 7.6 Hz, 1H), 7.13 (d, J = 8.0 Hz, 1H), 5.00 (s, 2H); ^{13}C NMR (150 MHz, DMSO-d_6) δ 168.2, 139.5, 135.2, 132.5, 128.9, 128.2, 127.8, 127.1, 124.8, 124.5, 110.8, 74.5, 43.4; ESI-MS *m/z*: 314.2 $[M+Na]^+$, the spectral data matched with those previously reported.[9]

4. Control experiments

1) Radica inhibiting experiment.

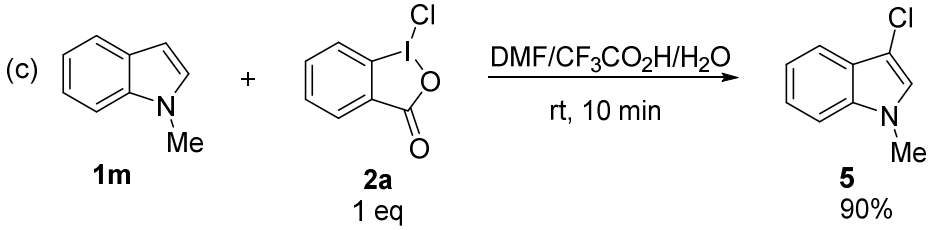


To the solution of DMF (1.5 mL) and TFA (1.0 mL) was added H_2O (0.2 mL). After the mixture cooled to room temperature, 1-methyl-1*H*-indole (65 mg, 0.5 mmol) ,BHT (221mg, 1 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (282 mg, 1.0mmol) were added successively, and then stirred at room temperature for 10 min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash chromatography (hexane/ethyl acetate = 20:1 to 10:1) on silica gel to afford **3m** as a white solid (62 mg, 69%).



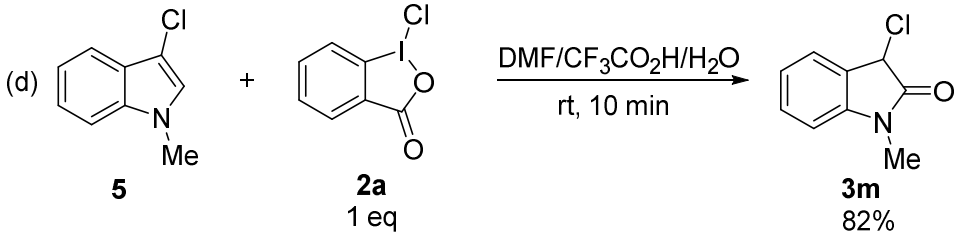
To the solution of 1,4-dioxane (3.0 mL) and H_2O (0.2 mL) was added 1-methyl-1*H*-indole (66 mg, 0.5 mmol) ,BHT (221mg, 1 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (424 mg, 1.5mmol) successively. The reaction mixture was then stirred at 80 °C for 30min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexane/ethyl acetate = 30:1 to 20:1) to afford **4p** as a white solid (93 mg, 86%).

2) Intermediate capture experiment

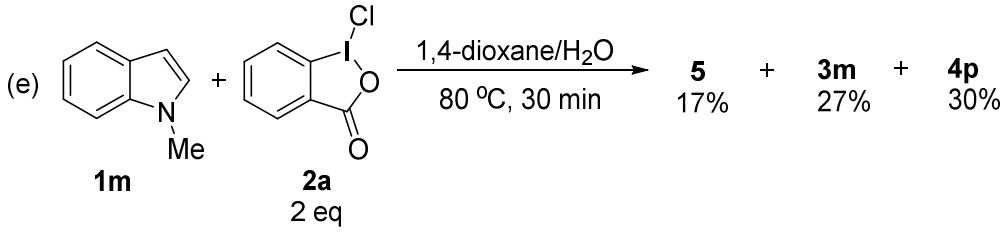


To the solution of DMF (1.5 mL) and TFA (1.0 mL) was added H_2O (0.2 mL). After the mixture cooled to room temperature, 1-methyl-1*H*-indole (65 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (142 mg, 0.5 mmol) were added successively, and then

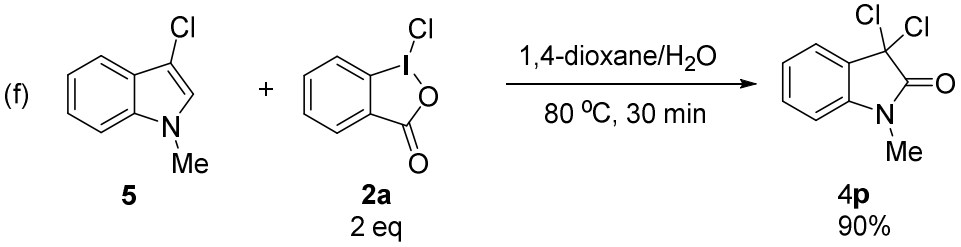
stirred at room temperature for 10 min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexane/ethyl acetate = 50:1 to 40:1) to afford **5** as a yellow oil (75 mg, 90%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.9 Hz, 1H), 7.32 - 7.23 (m, 2H), 7.26 – 7.19 (m, 1H), 7.01 (s, 1H), 3.73 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 135.7, 125.6, 125.2, 122.5, 119.8, 118.3, 109.4, 104.2, 32.9.



To the solution of DMF (1.5 mL) and TFA (1.0 mL) was added H_2O (0.2 mL). After the mixture cooled to room temperature, **5** (81 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (142 mg, 0.5 mmol) were added successively, and then stirred at room temperature for 10 min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexane/ethyl acetate = 20:1 to 10:1) to afford **3m** as a white solid (74 mg, 82%).

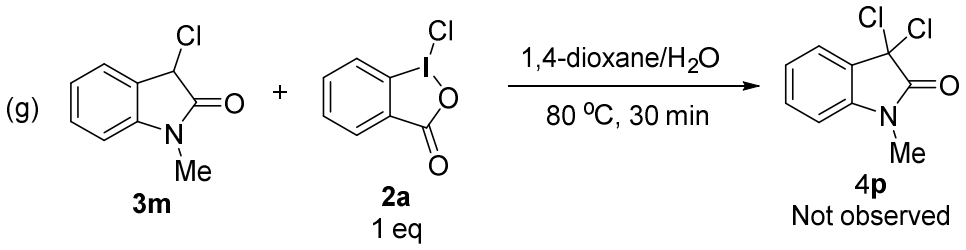


To the solution of 1,4-dioxane (3.0 mL) and H_2O (0.2 mL) was added 1-methyl-1*H*-indole (66 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (283 mg, 1.0 mmol) successively. The reaction mixture was then stirred at 80 °C for 30min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexane/ethyl acetate = 30:1 to 20:1) to afford **5** as a yellow oil (14mg, 17%), **3m** as a white solid (29 mg, 27%) and **4p** as a white solid (27mg, 30%).



To the solution of 1,4-dioxane (3.0 mL) and H_2O (0.2 mL) was added **5** (82 mg, 0.5 mmol) and

1-chloro-1,2-benziodoxol-3-(1*H*)-one (282 mg, 1.0mmol) successively. The reaction mixture was then stirred at 80 °C for 30min. After the completion of reaction, as indicated by TLC, the reaction was then quenched by slow addition of saturated $NaHCO_3$ solution (15 mL) and extracted with ethyl acetate (10 mL x 3). The combined organic phase was washed with brine (10 mL), dried over Na_2SO_4, and evaporated under reduced pressure. The residue was purified by flash chromatography on silica gel (hexane/ethyl acetate = 30:1 to 20:1) to afford **4p** as a white solid (97mg, 90%).



To the solution of 1,4-dioxane (3.0 mL) and H_2O (0.2 mL) was added **3m** (91 mg, 0.5 mmol) and 1-chloro-1,2-benziodoxol-3-(1*H*)-one (142 mg, 0.5mmol) successively. The reaction mixture was then stirred at 80 °C for 30min.The **4p** was not obtained.

4. References

[1] V. Matoušek, E. Pietrasiak, R. Schwenk, A. Togni, *J. Org. Chem.* 2013, **78**, 6763-6768.

[2] M. Stodulski, A. Goetzinger, S. V. Kohlhepp, T. Gulder, *Chem. Commun.* 2014, **50**, 3435-3438.

[3] Y. Wu, X. Peng, B. Luo, F. Wu, B. Liu, F. Song, P. Huang, S. Wen, *Org. Biomol. Chem.* 2014, **12**, 9777-9780.

[4] T. W. Greulich, C. G. Daniliuc, A. Studer, *Org. Lett.* 2015, **17**, 254-257.

[5] R. C. da Silva, I. Chatterjee, E. Escudero-Adan, M. W. Paixao, P. Melchiorre, *Asian J. Org. Chem.* 2014, **3**, 466-469.

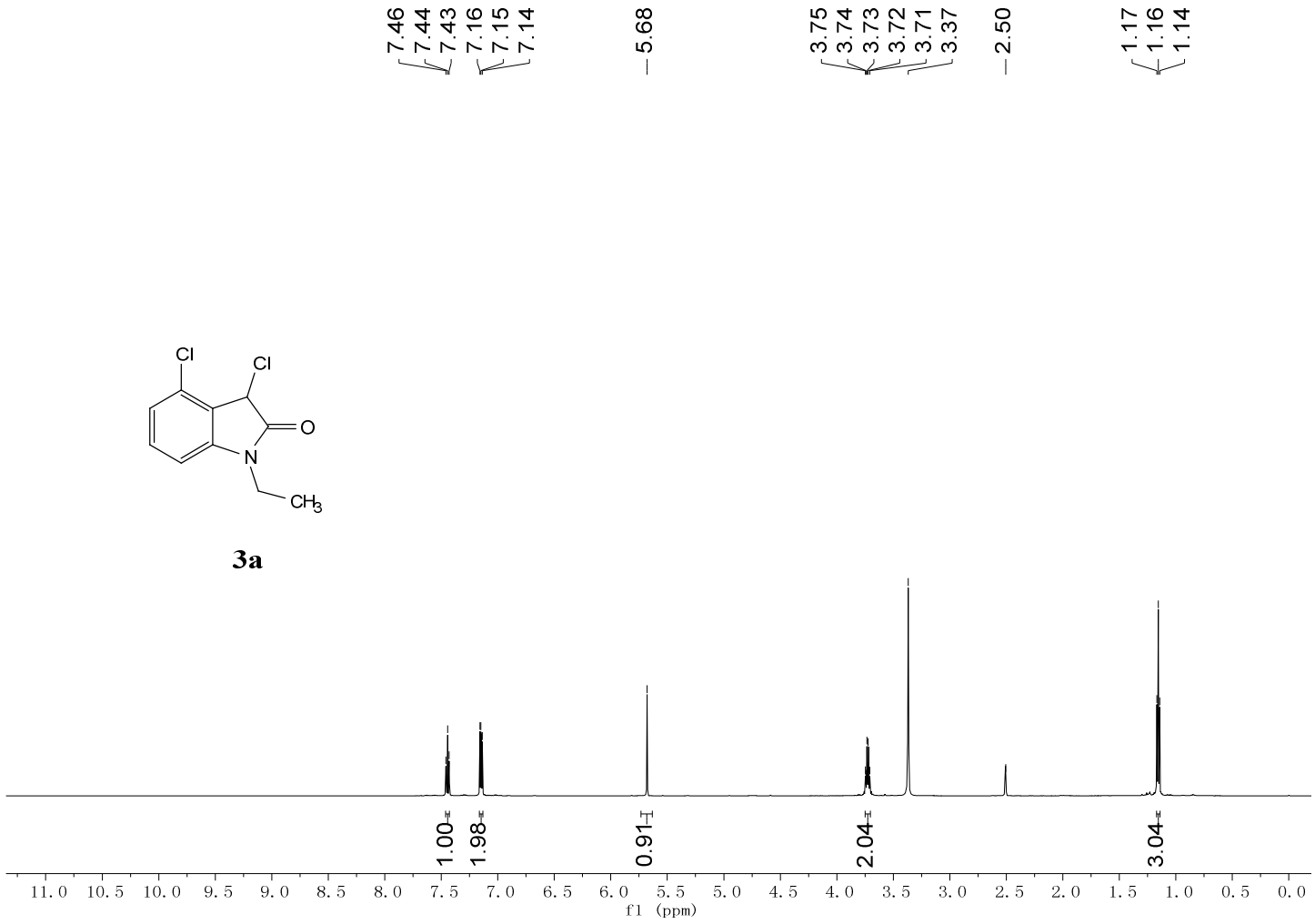
[6] X. Wang, K. Dong, B. Yan, C. Zhang, L. Qiu, X. Xu, *RSC Adv.* 2016, **6**, 70221-70225.

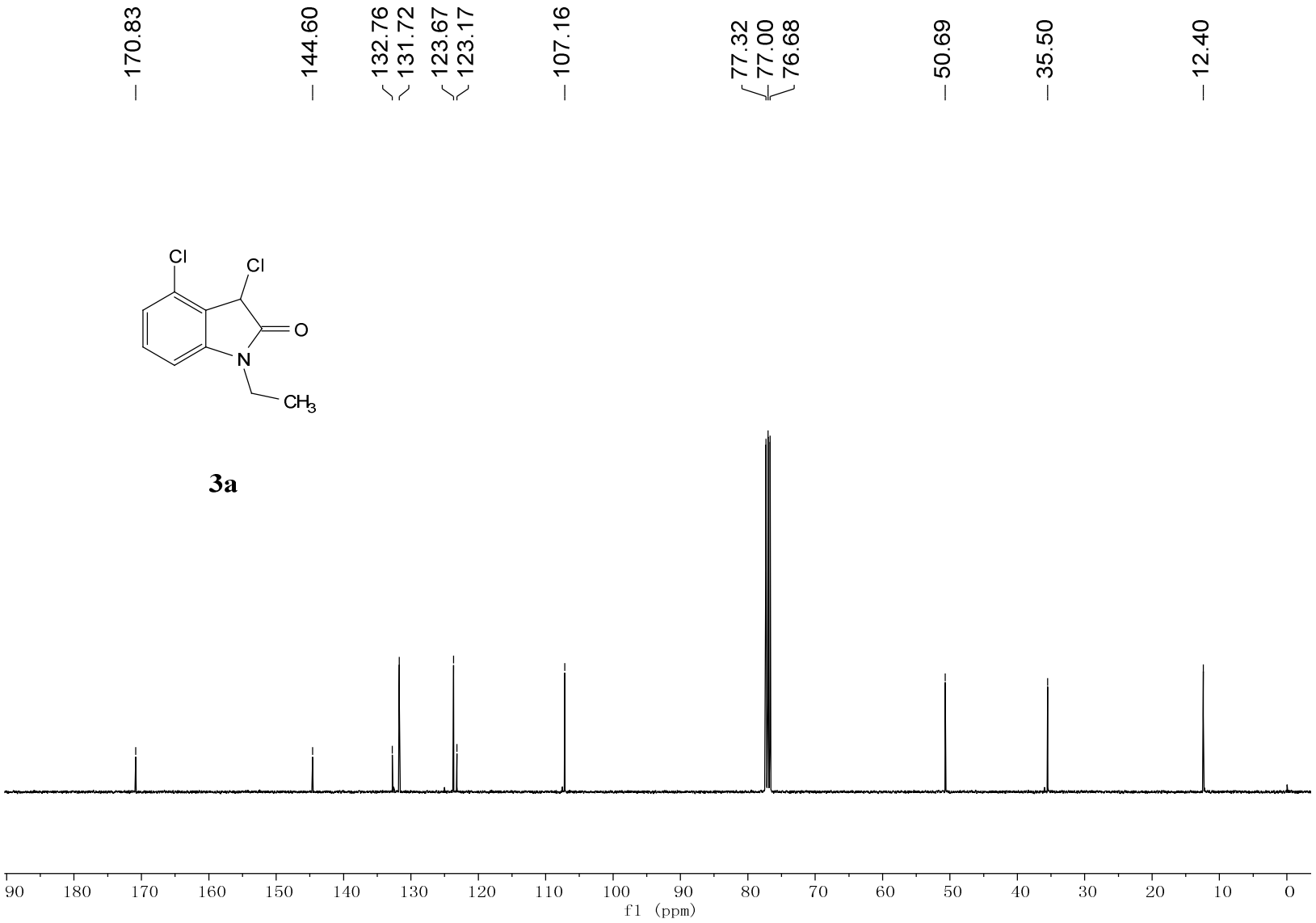
[7] G. K. Murphy, F. Z. Abbas, A. V. Poulton, *Adv. Synth. Catal.* 2014, **356**, 2919-2923.

[8] H. Hamamoto, H. Umemoto, M. Umemoto, C. Ohta, M. Dohshita, Y. Miki, *Synlett* 2010, **2010**, 2593-2596.

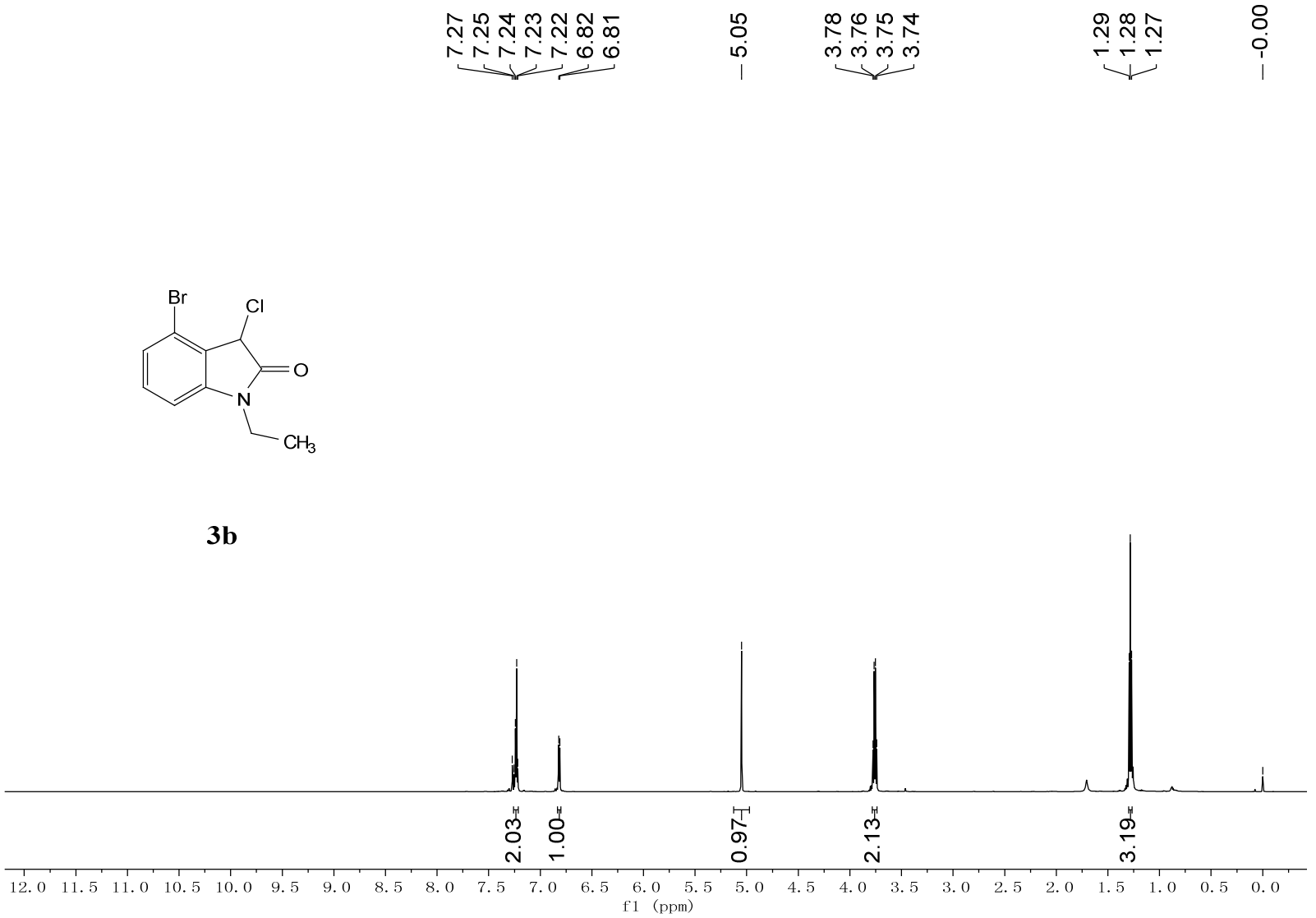
[9] A. N. C. Lötter, R. Pathak, T. S. Sello, M. A. Fernandes, W. A. L. van Otterlo, C. B. de Koning, *Tetrahedron* 2007, **63**, 2263-2274.

5. Copies of ^{1}H, ^{13}C NMR and ^{19}F NMR spectra of **3** ,**4** and **5.**

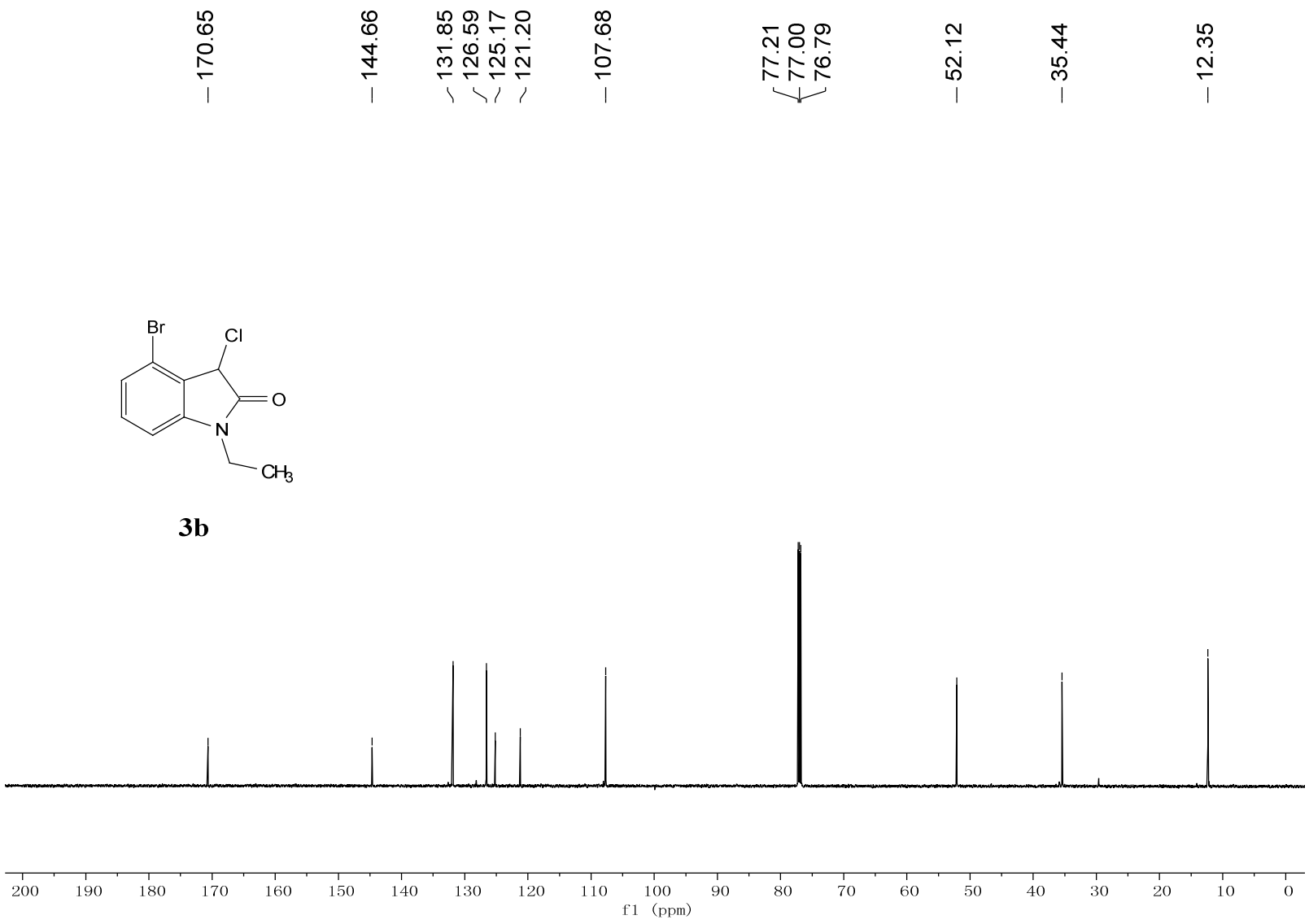



170.83
144.60
132.76
131.72
123.67
123.17
107.16
77.32
77.00
76.68
50.69
35.50
12.40
Cl
Cl
O
N
CH3
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

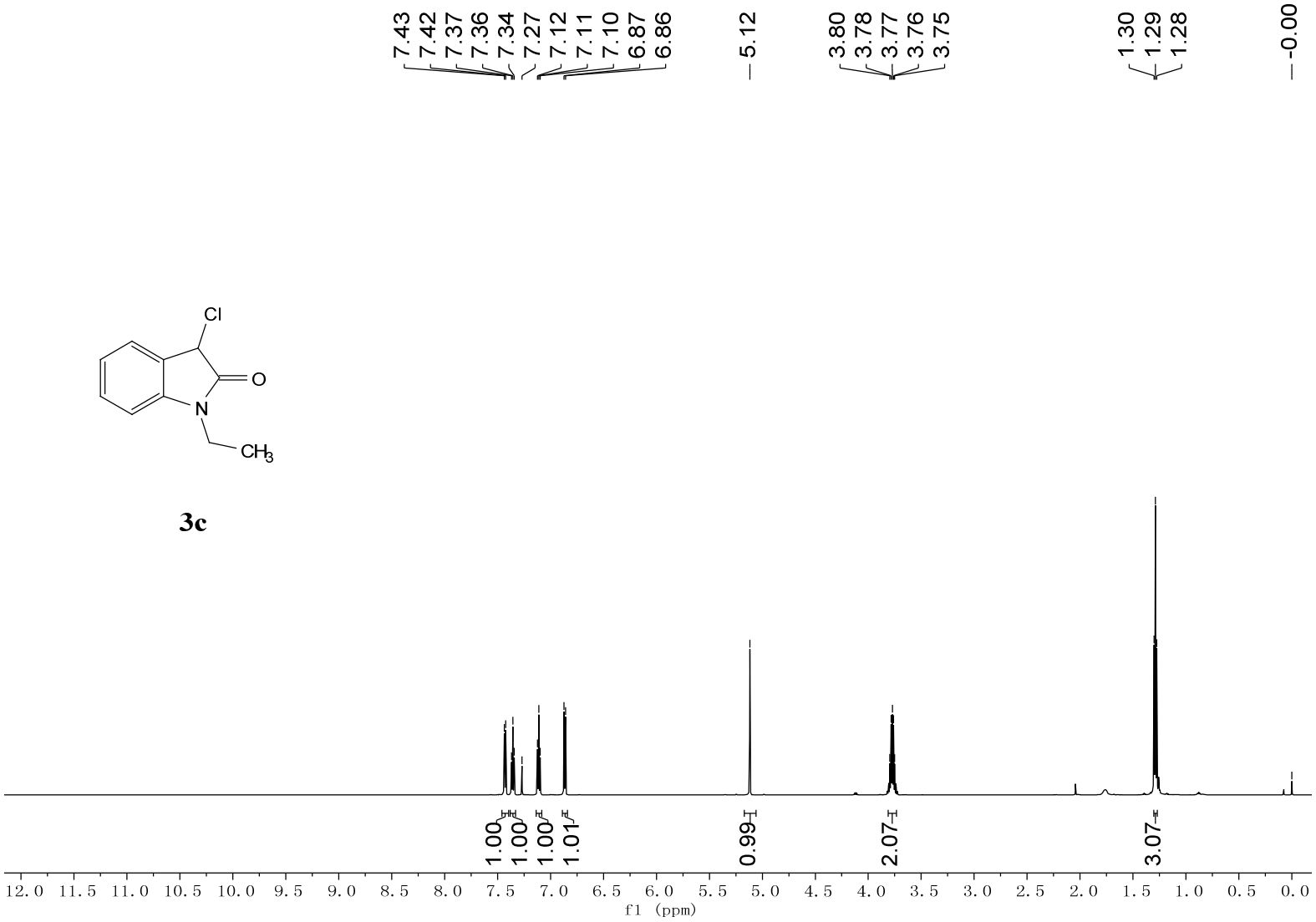
3a


7.27
7.25
7.24
7.23
7.22
6.82
6.81
5.05
3.78
3.76
3.75
3.74
1.29
1.28
1.27
-0.00
Br
Cl
O
N
CH3
2.03
1.00
0.97
2.13
3.19
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

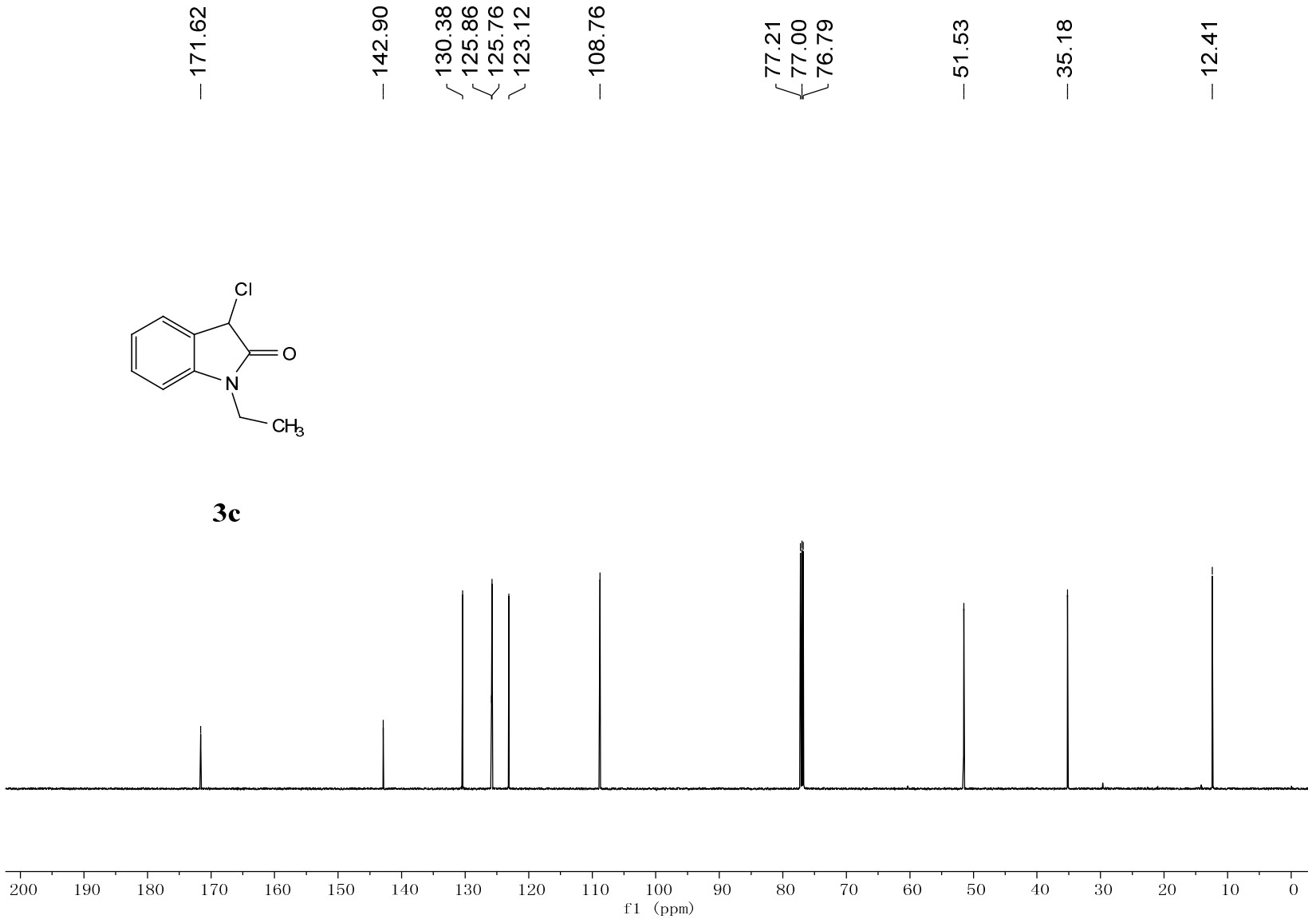
3b


170.65
144.66
131.85
126.59
125.17
121.20
107.68
77.21
77.00
76.79
52.12
35.44
12.35
Br
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

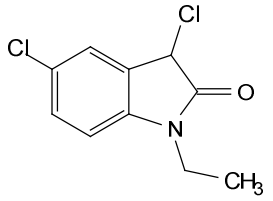
3b


7.43
7.42
7.37
7.36
7.34
7.27
7.12
7.11
7.10
6.87
6.86
5.12
3.80
3.78
3.77
3.76
3.75
1.30
1.29
1.28
0.00
Cl
O
N
CH3
1.00
1.00
1.00
1.01
0.99
2.07
3.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3c

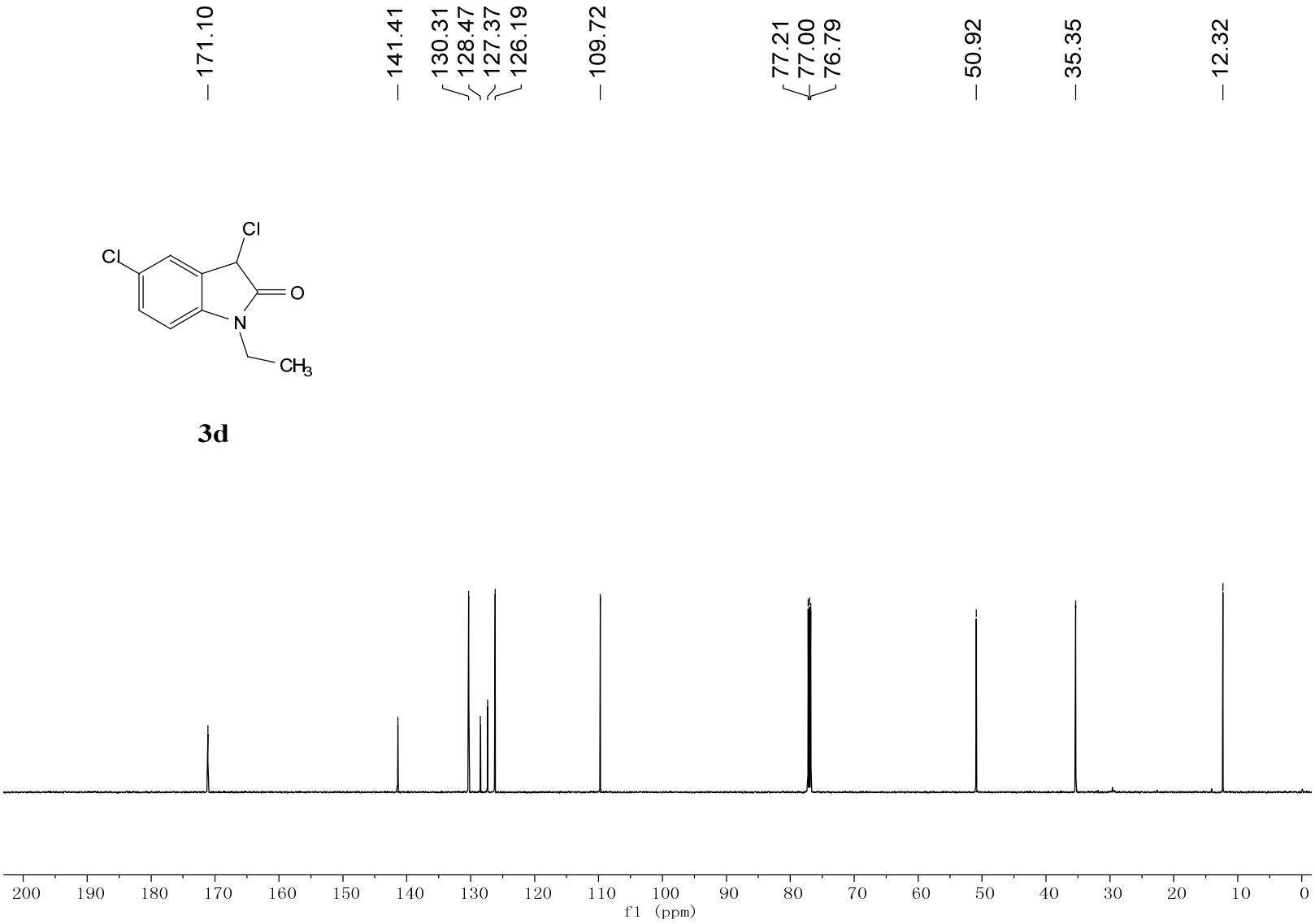

171.62
142.90
130.38
125.86
125.76
123.12
108.76
77.21
77.00
76.79
51.53
35.18
12.41
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3c

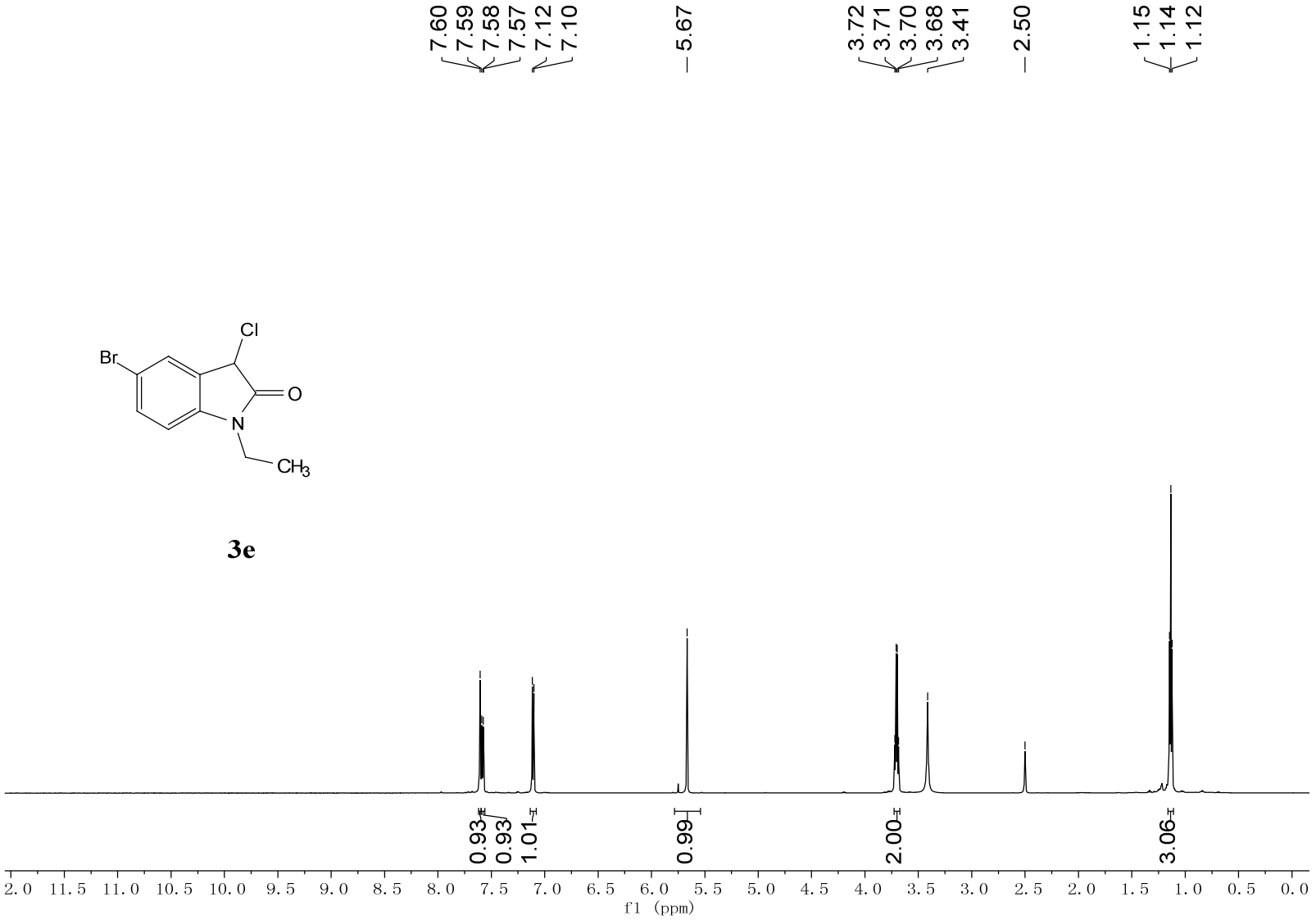

Cl
Cl
O
N
CH3

3d

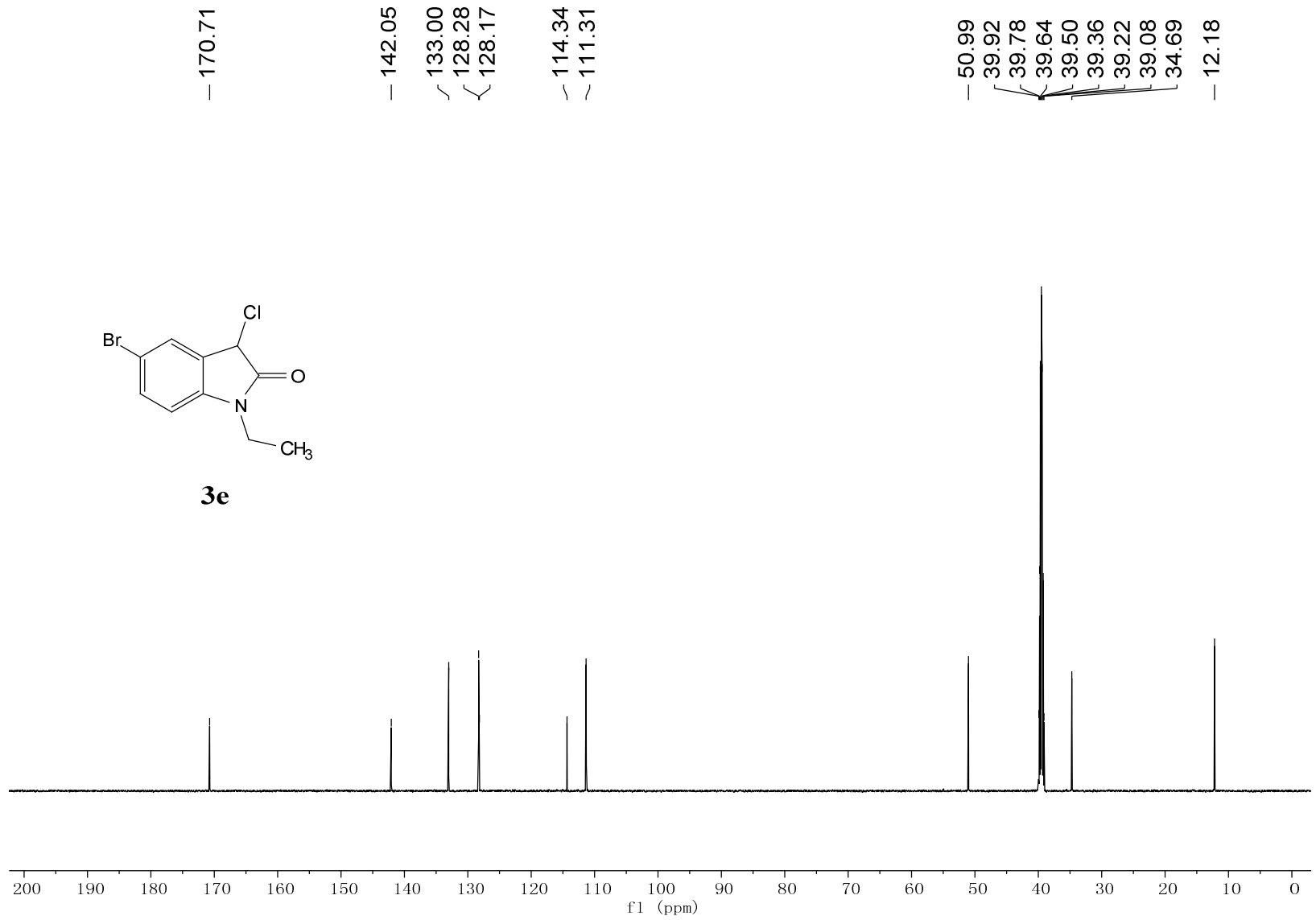
7.41
7.34
7.34
7.33
7.32
7.28
6.81
6.79
5.10
3.79
3.78
3.77
3.76
3.75
3.74
3.72
1.29
1.28
1.27
-0.00
0.95
0.98
1.00
1.00
2.03
3.01
f1 (ppm)


171.10
141.41
130.31
128.47
127.37
126.19
109.72
77.21
77.00
76.79
50.92
35.35
12.32
Cl
Cl
O
N
CH3
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

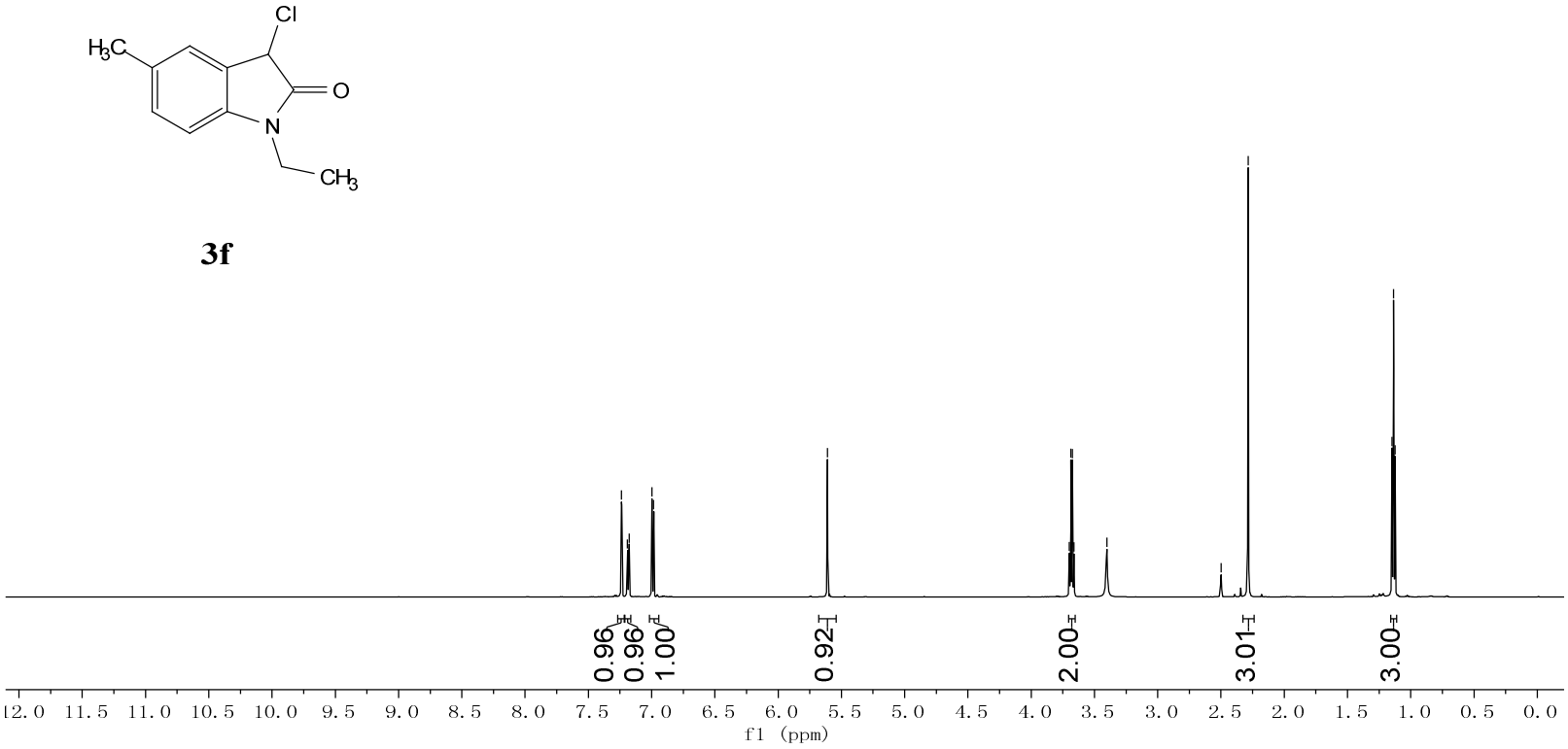
3d


7.60
7.59
7.58
7.57
7.12
7.10
5.67
3.72
3.71
3.70
3.68
3.41
2.50
1.15
1.14
1.12
Cl
Br
O
N
CH3
0.93
0.93
1.01
0.99
2.00
3.06
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

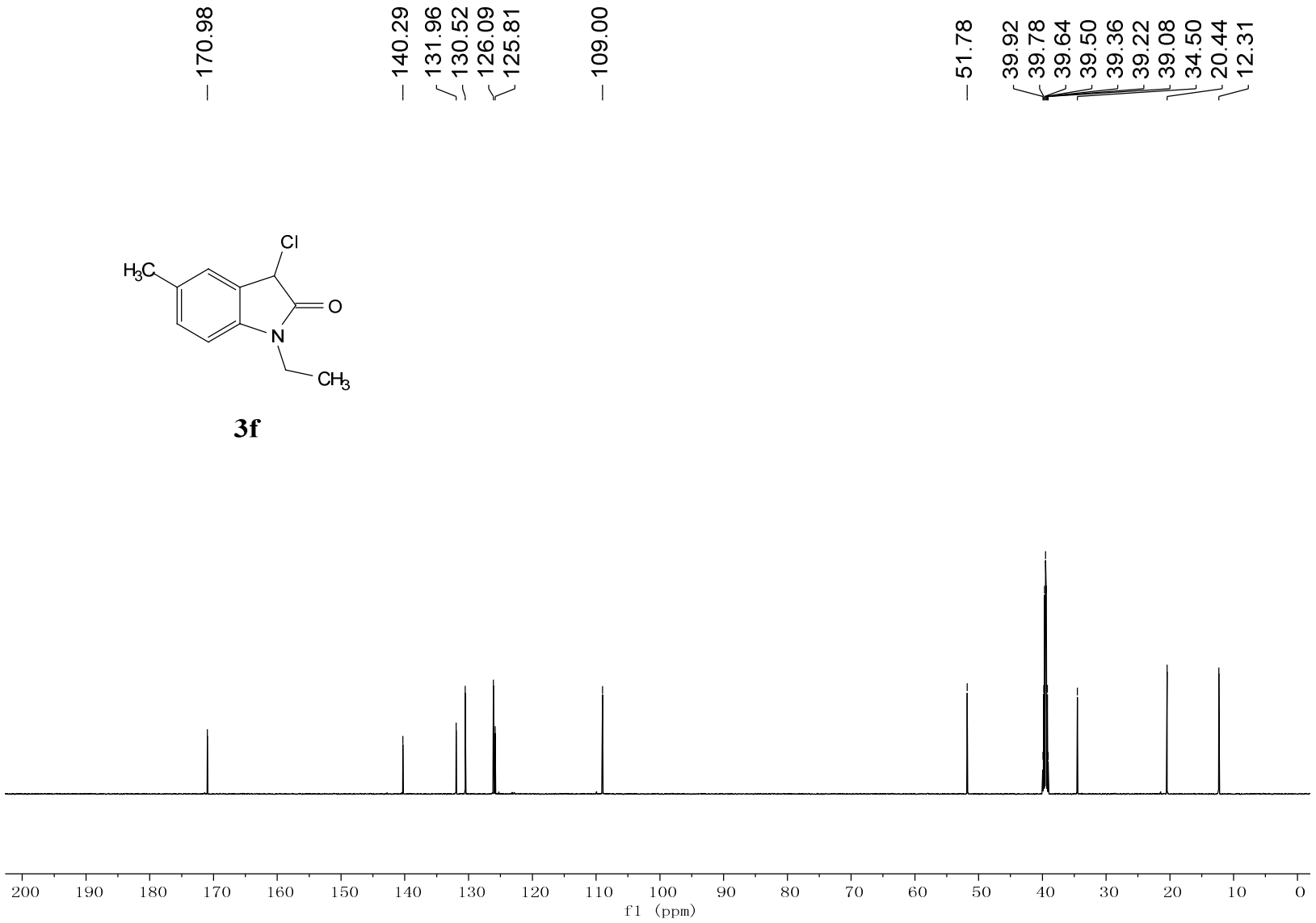
3e


170.71
142.05
133.00
128.28
128.17
114.34
111.31
50.99
39.92
39.78
39.64
39.50
39.36
39.22
39.08
34.69
12.18
Cl
Br
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

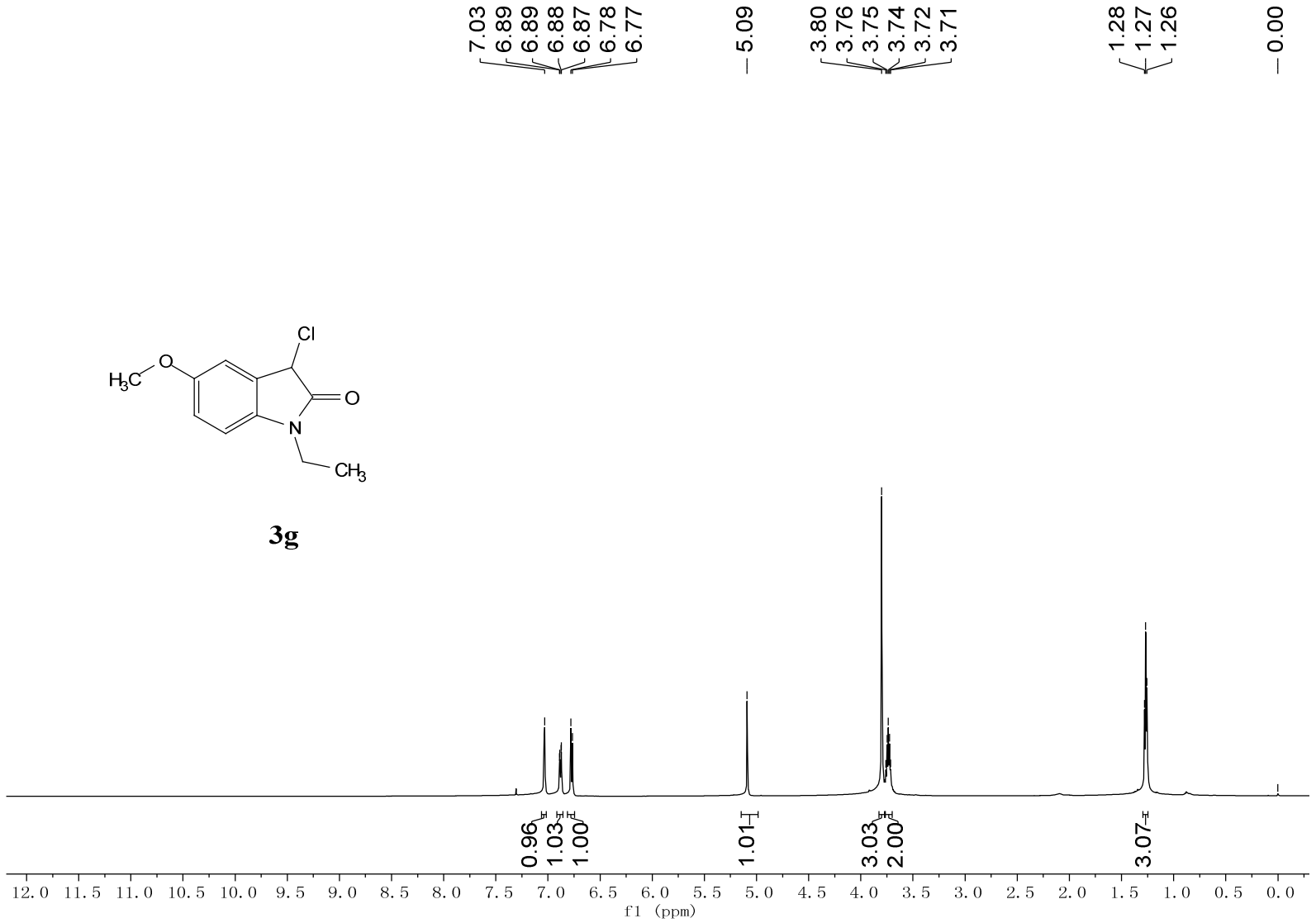
3e


7.24
7.19
7.18
7.00
6.98
5.61
3.70
3.69
3.67
3.66
3.40
2.50
2.29
1.15
1.14
1.12
Cl
H3C
O
N
CH3
0.96
0.96
1.00
0.92
2.00
3.01
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

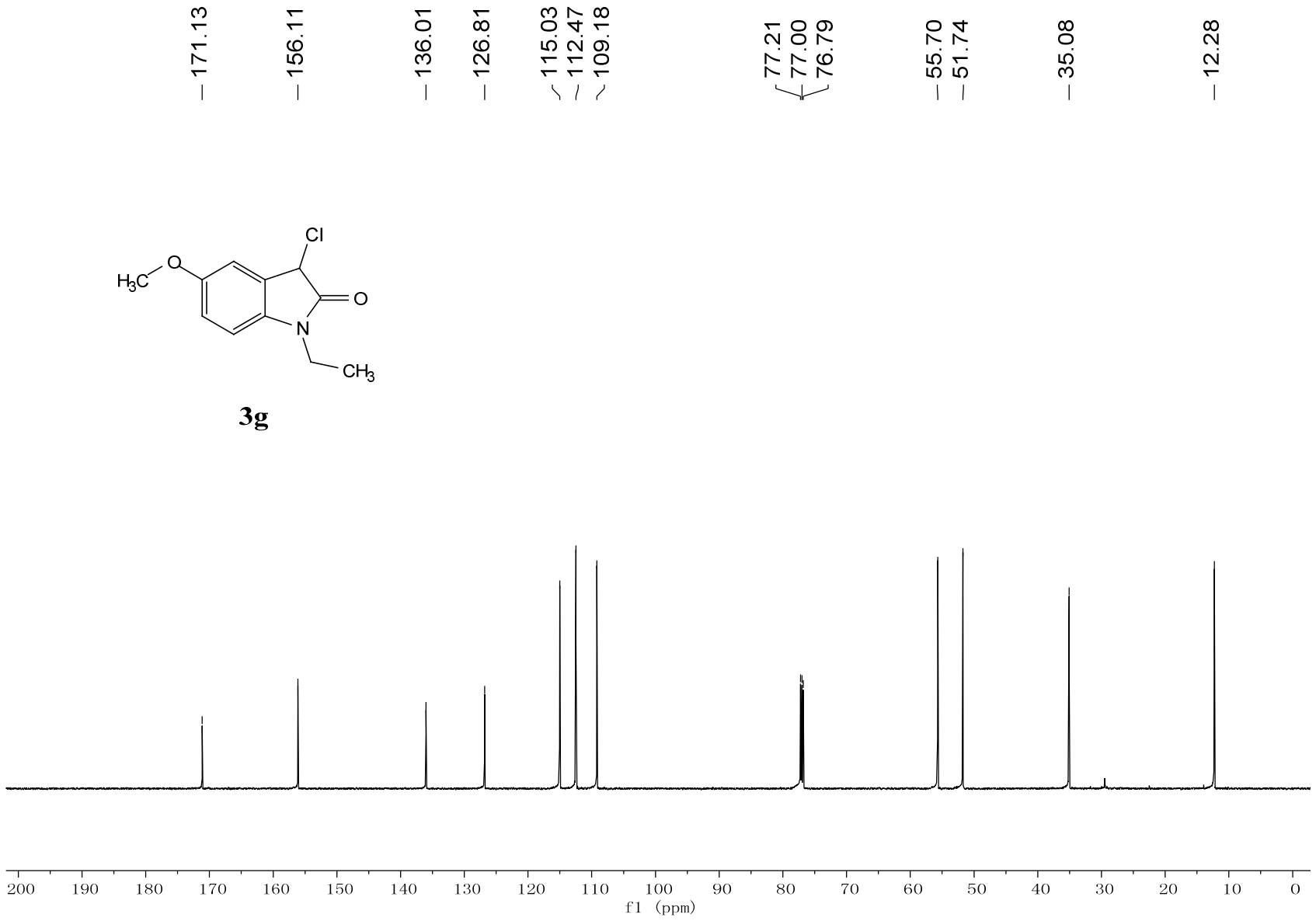
3f


170.98
140.29
131.96
130.52
126.09
125.81
109.00
51.78
39.92
39.78
39.64
39.50
39.36
39.22
39.08
34.50
20.44
12.31
Cl
H3C
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

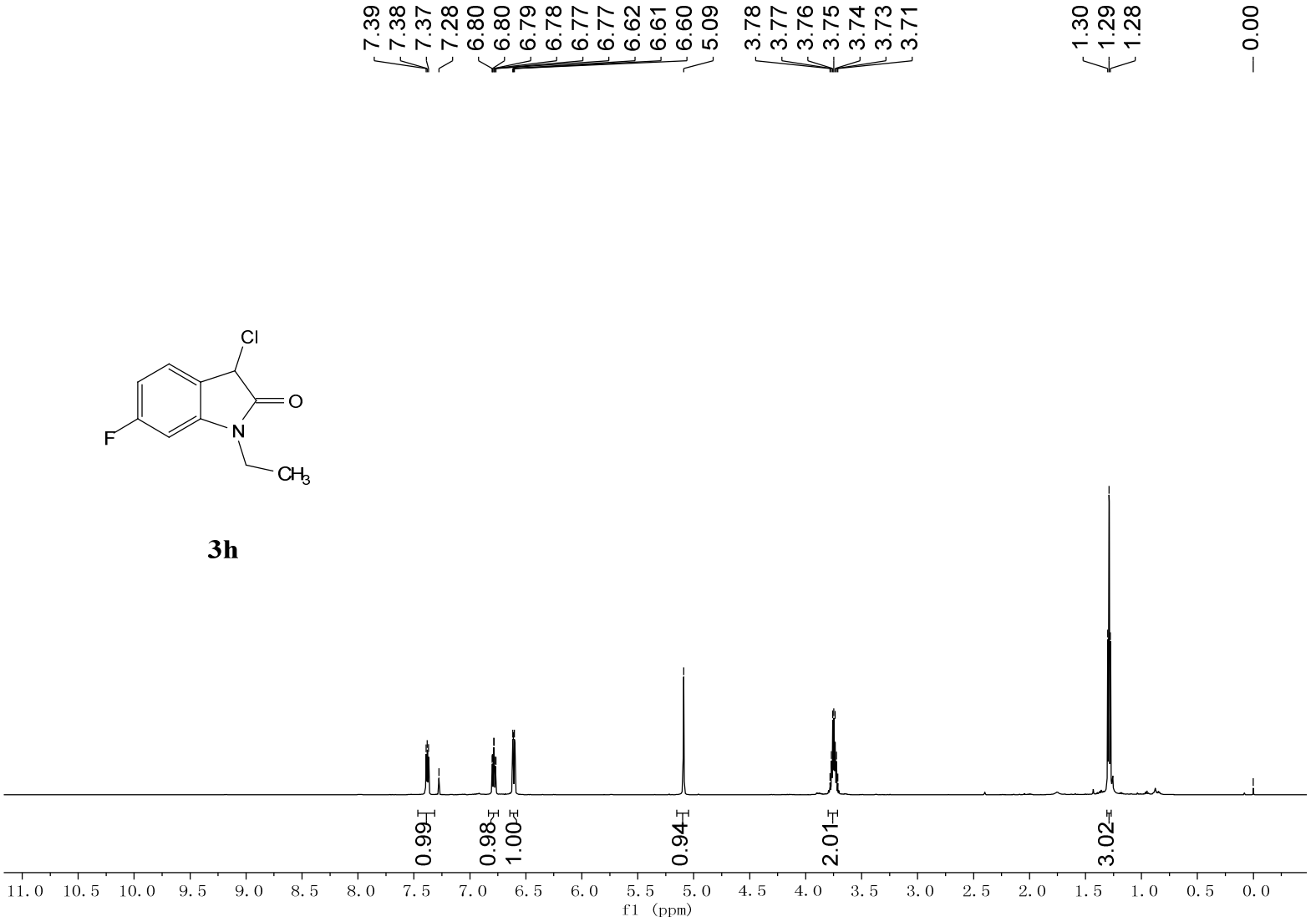
3f


7.03
6.89
6.89
6.88
6.87
6.78
6.77
5.09
3.80
3.76
3.75
3.74
3.72
3.71
1.28
1.27
1.26
0.00
Cl
O
H3C
O
N
CH3
0.96
1.03
1.00
1.01
3.03
2.00
3.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

3g

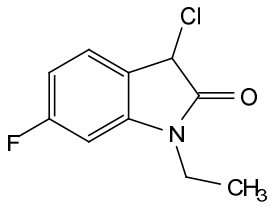

171.13
156.11
136.01
126.81
115.03
112.47
109.18
77.21
77.00
76.79
55.70
51.74
35.08
12.28
Cl
O
H3C
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3g

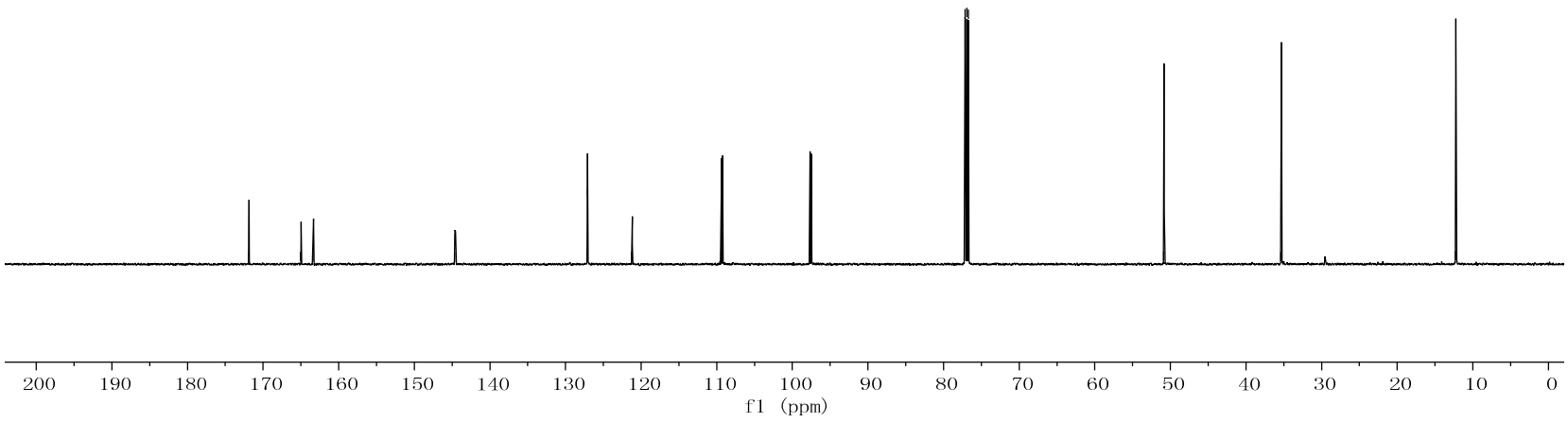

7.39
7.38
7.37
7.28
6.80
6.80
6.79
6.78
6.77
6.77
6.62
6.61
6.60
5.09
3.78
3.77
3.76
3.75
3.74
3.73
3.71
1.30
1.29
1.28
0.00
Cl
O
F
N
CH3
0.99
0.98
1.00
0.94
2.01
3.02
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

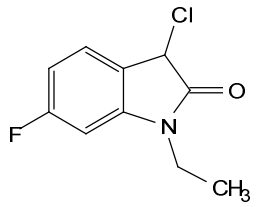
3h

171.92
165.04
163.39
144.68
144.60
127.17
127.10
121.24
121.22
109.43
109.28
97.73
97.55
77.14
76.93
76.72
50.90
35.40
12.33

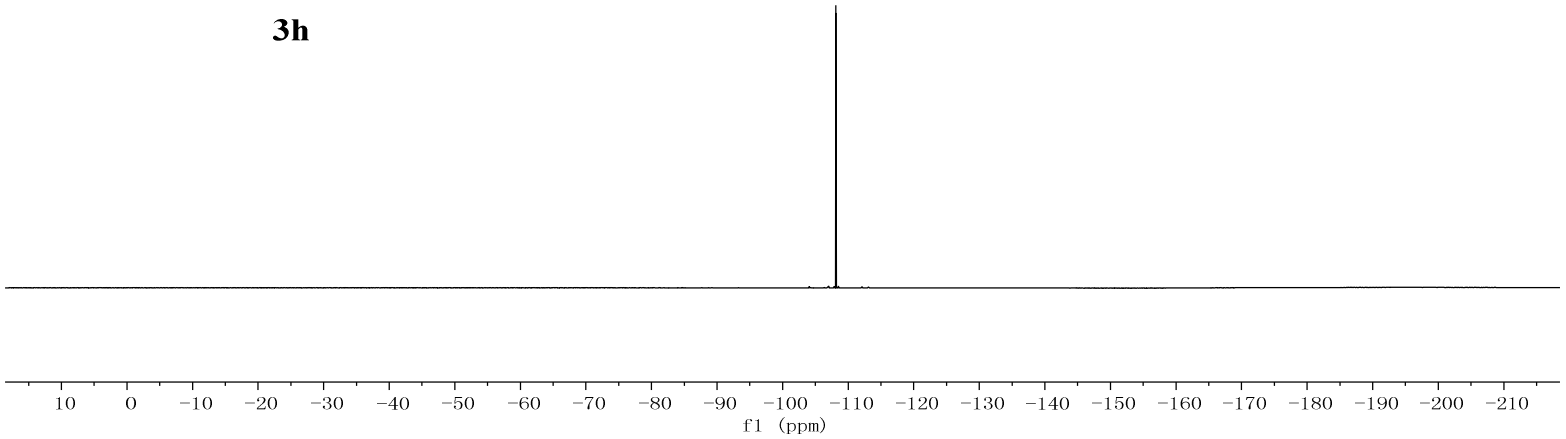


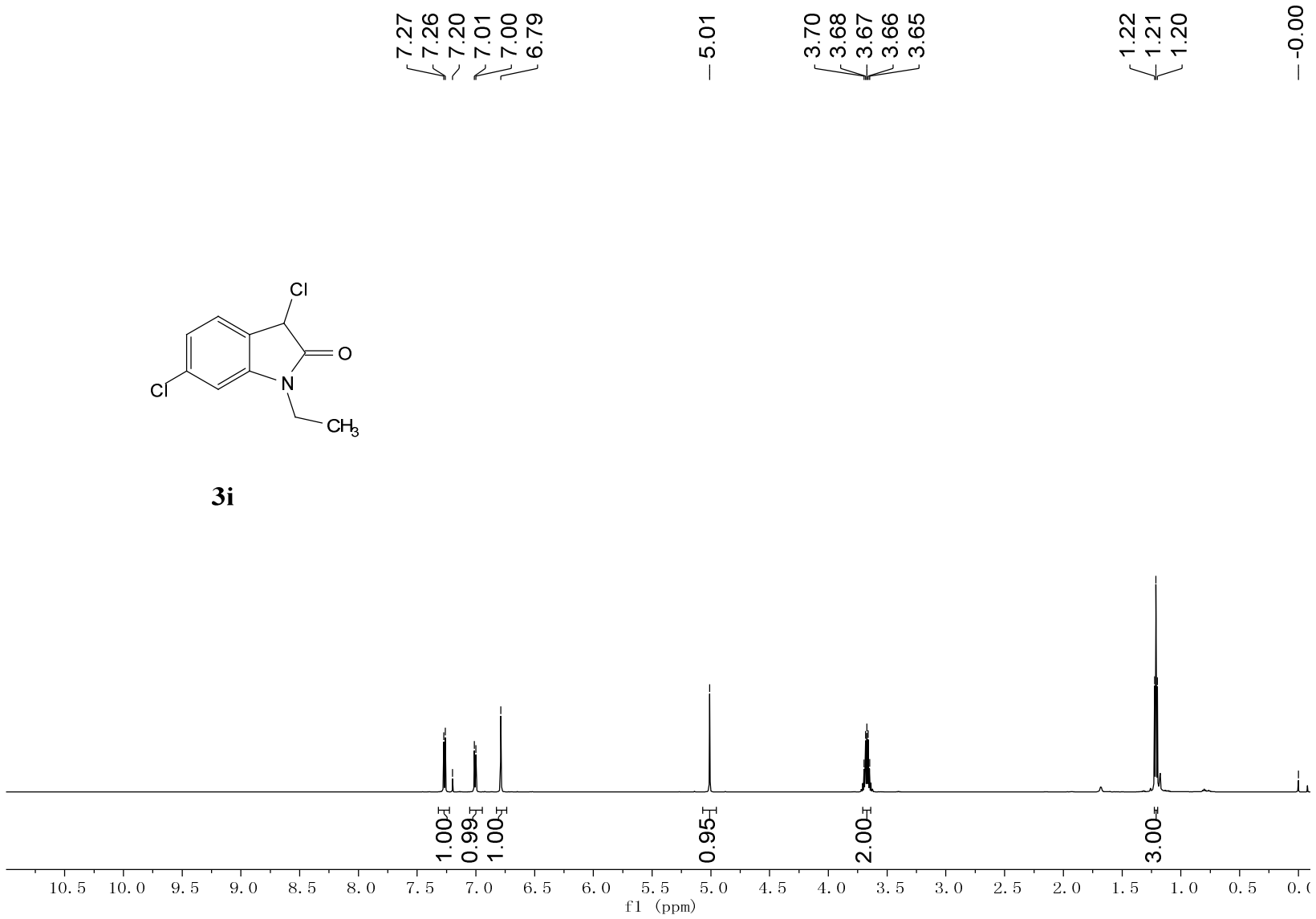
3h



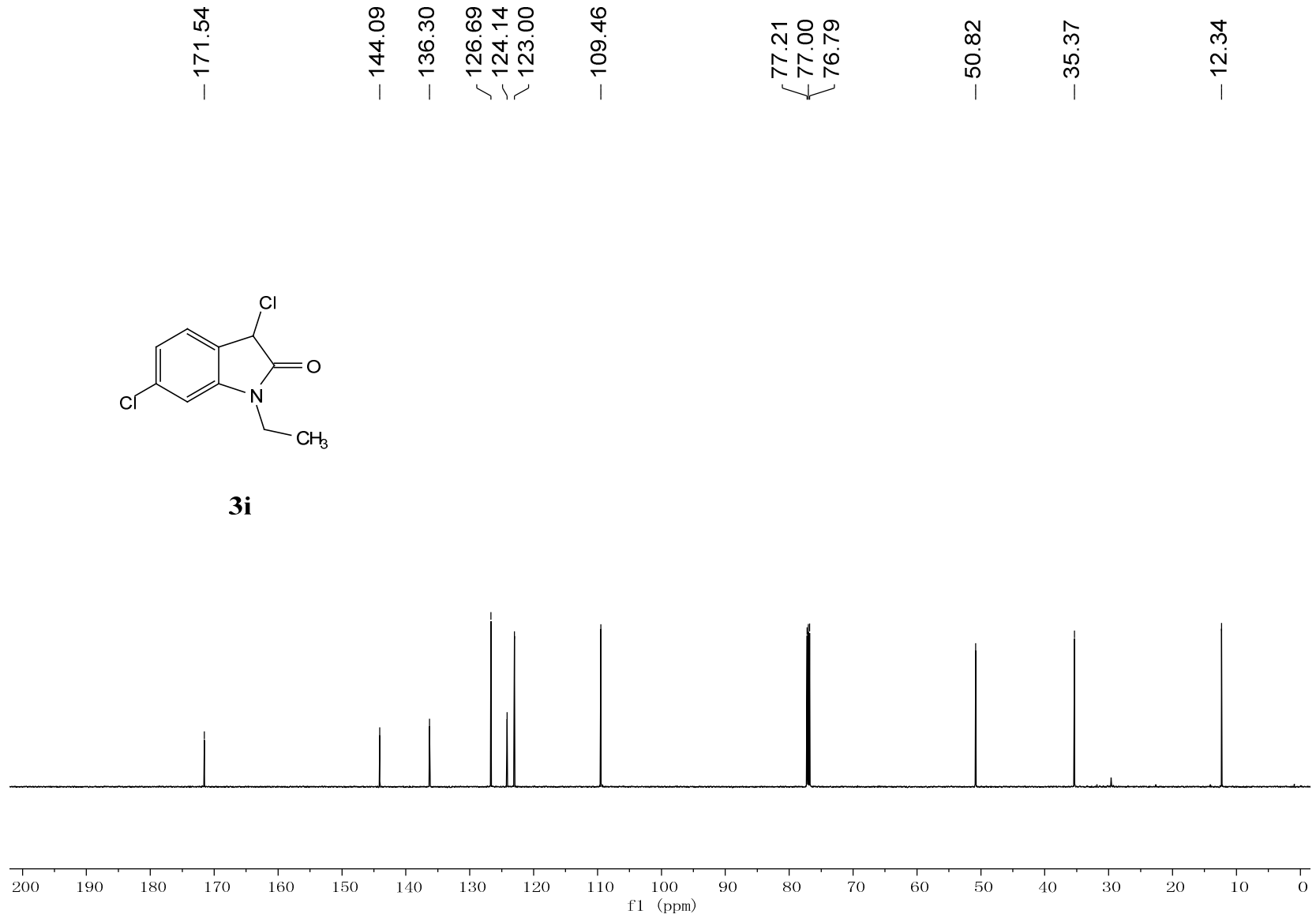

Cl
O
F
N
CH3

3h

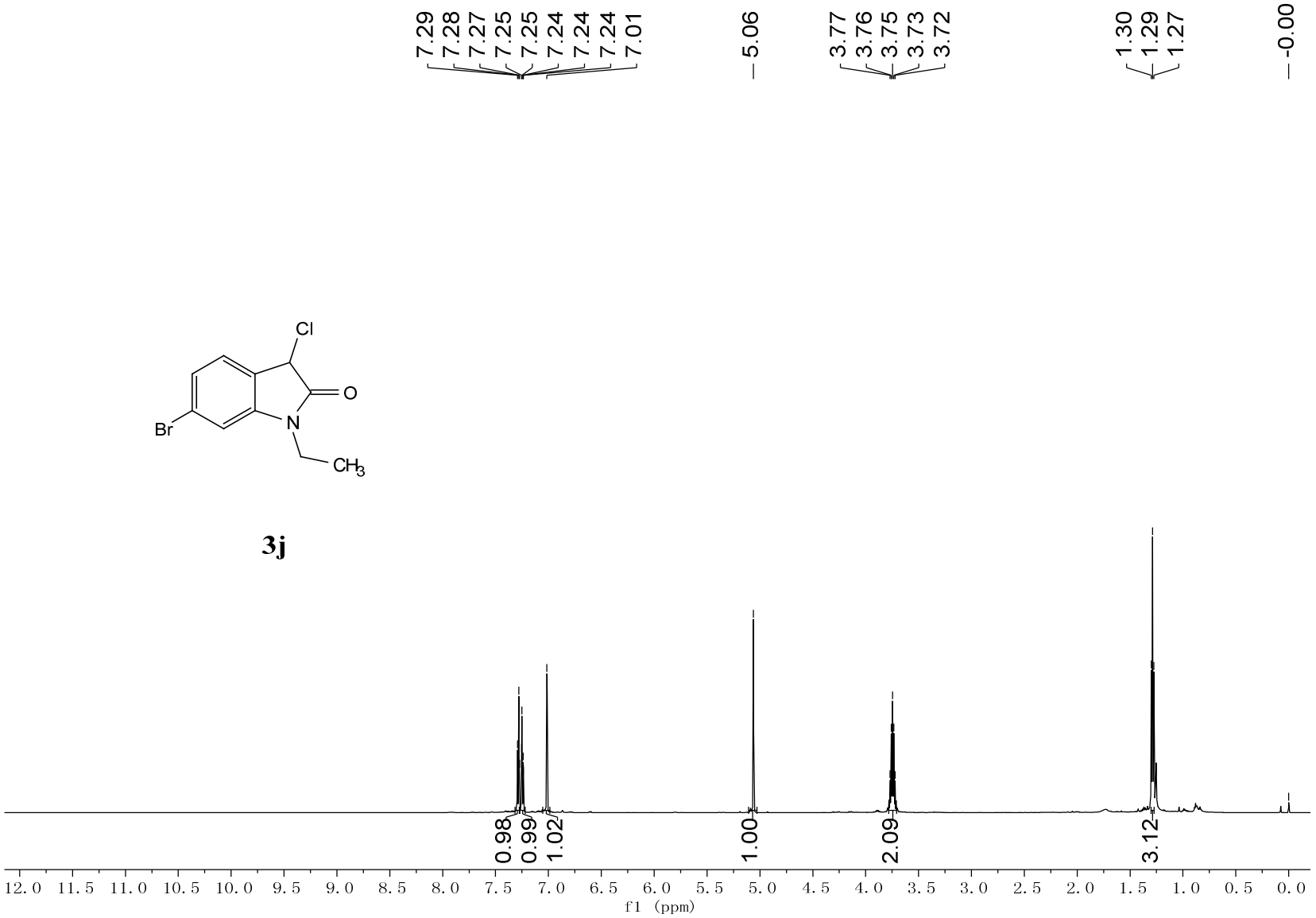

-108.11
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

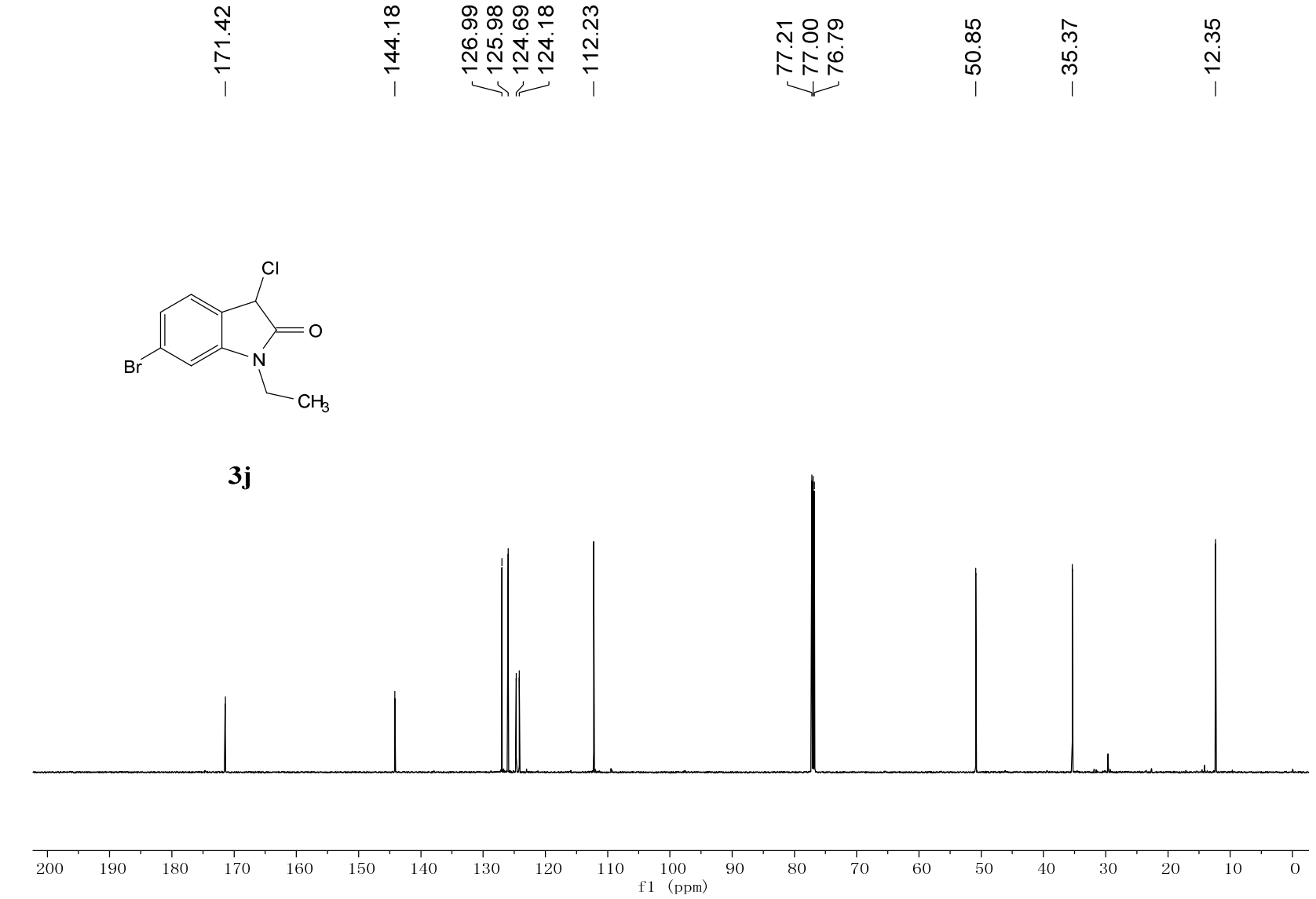

7.27
7.26
7.20
7.01
7.00
6.79
5.01
3.70
3.68
3.67
3.66
3.65
1.22
1.21
1.20
-0.00
Cl
O
Cl
N
CH3
1.00
0.99
1.00
0.95
2.00
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

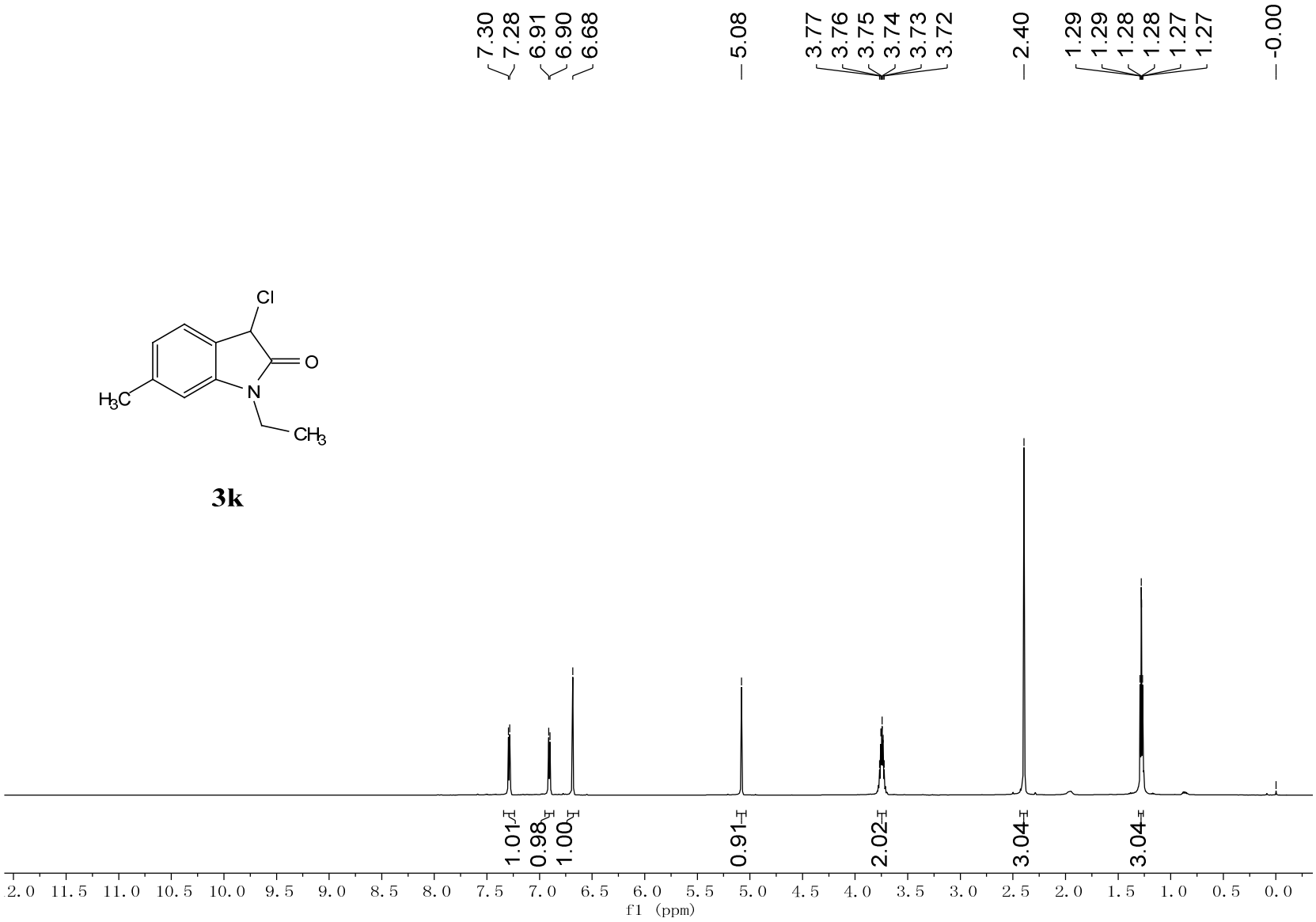
3i


171.54
144.09
136.30
126.69
124.14
123.00
109.46
77.21
77.00
76.79
50.82
35.37
12.34
Cl
O
N
Cl
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

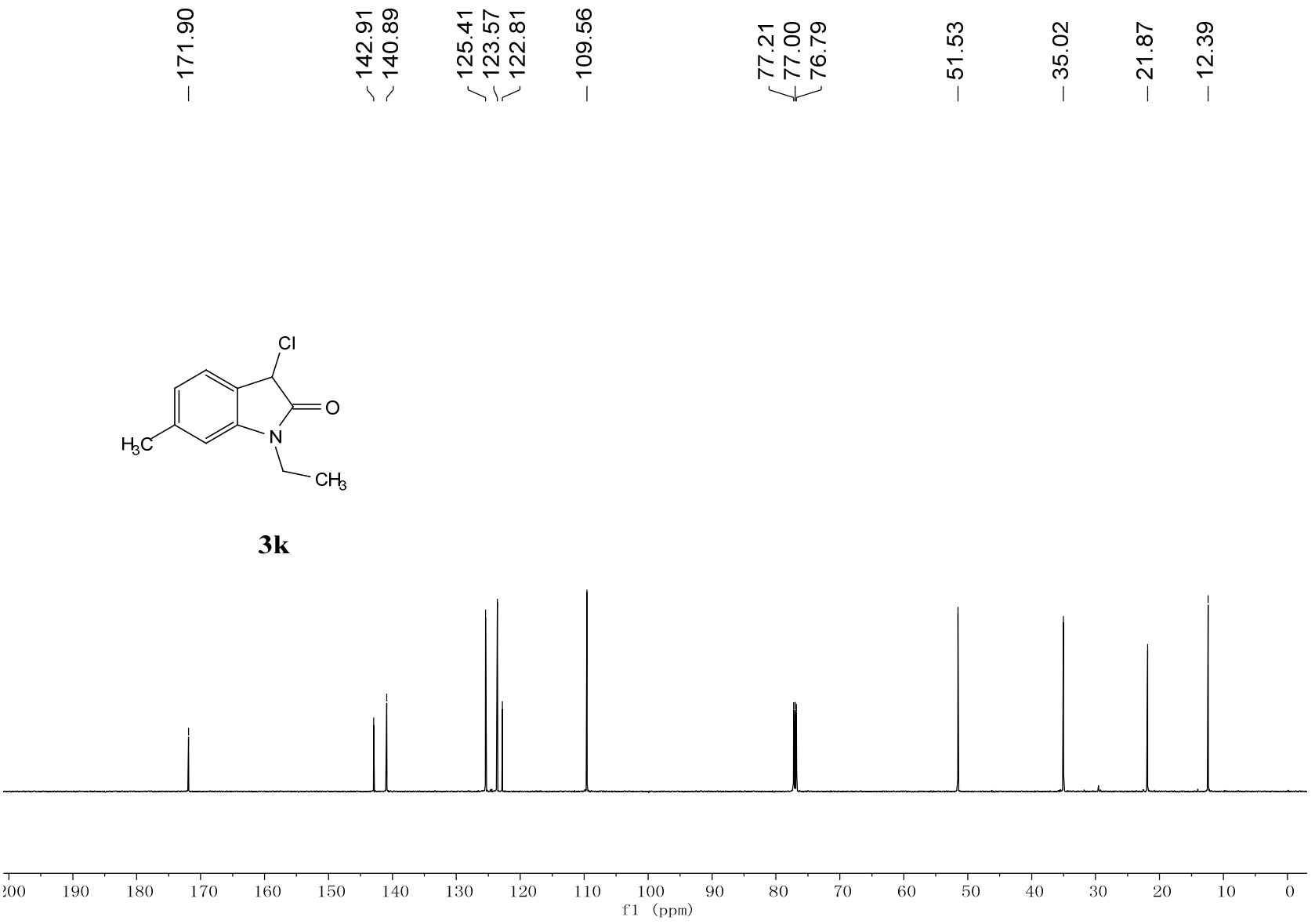
3i


7.29
7.28
7.27
7.25
7.25
7.24
7.24
7.24
7.01
5.06
3.77
3.76
3.75
3.73
3.72
1.30
1.29
1.27
-0.00
Cl
O
N
Br
CH3
3j
0.98
0.99
1.02
1.00
2.09
3.12
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

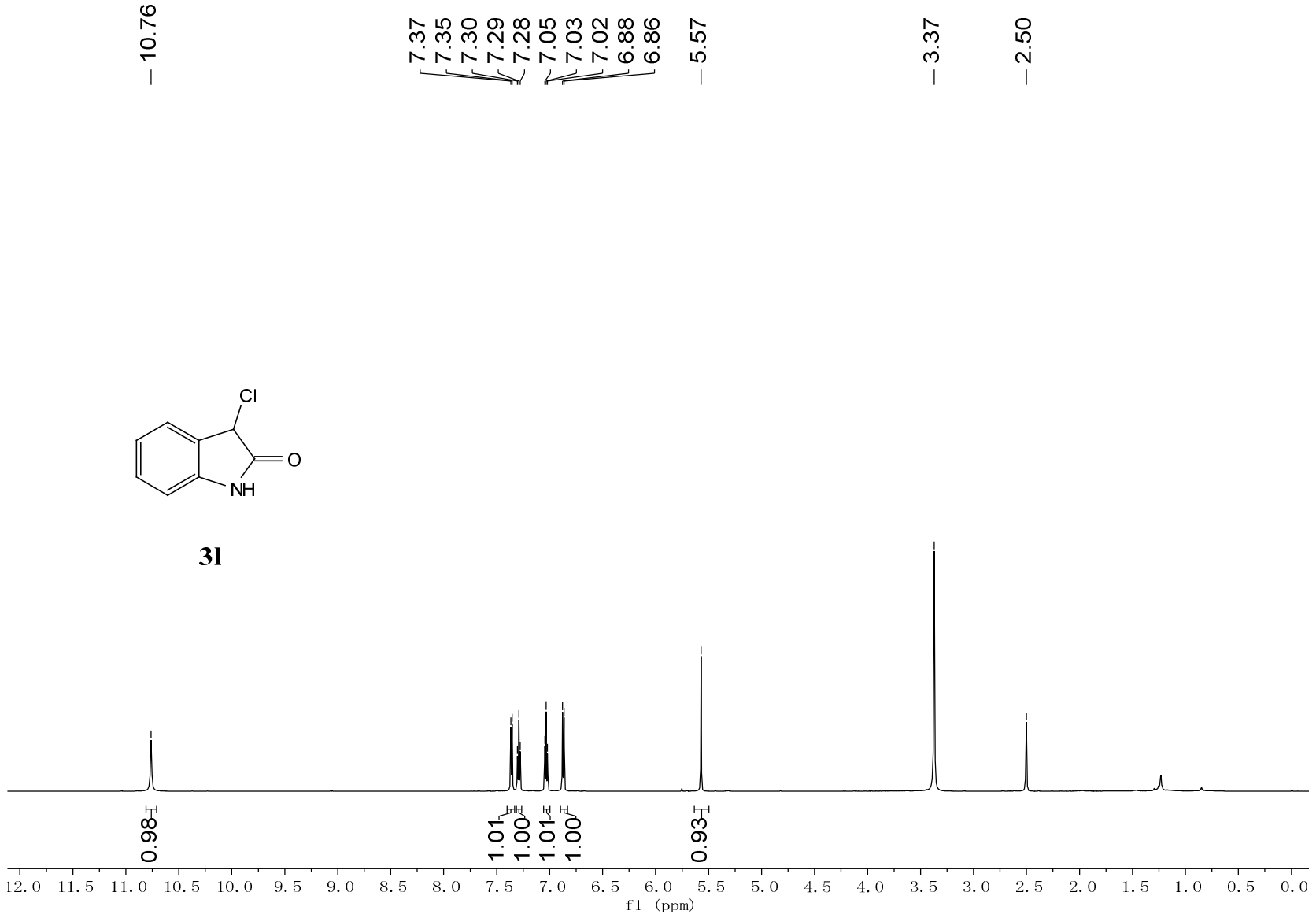

171.42
144.18
126.99
125.98
124.69
124.18
112.23
77.21
77.00
76.79
50.85
35.37
12.35
Cl
O
N
Br
CH3
3j
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.30
7.28
6.91
6.90
6.68
5.08
3.77
3.76
3.75
3.74
3.73
3.72
2.40
1.29
1.29
1.28
1.28
1.27
1.27
0.00
Cl
O
N
H3C
CH3
1.01
0.98
1.00
0.91
2.02
3.04
3.04
f1 (ppm)

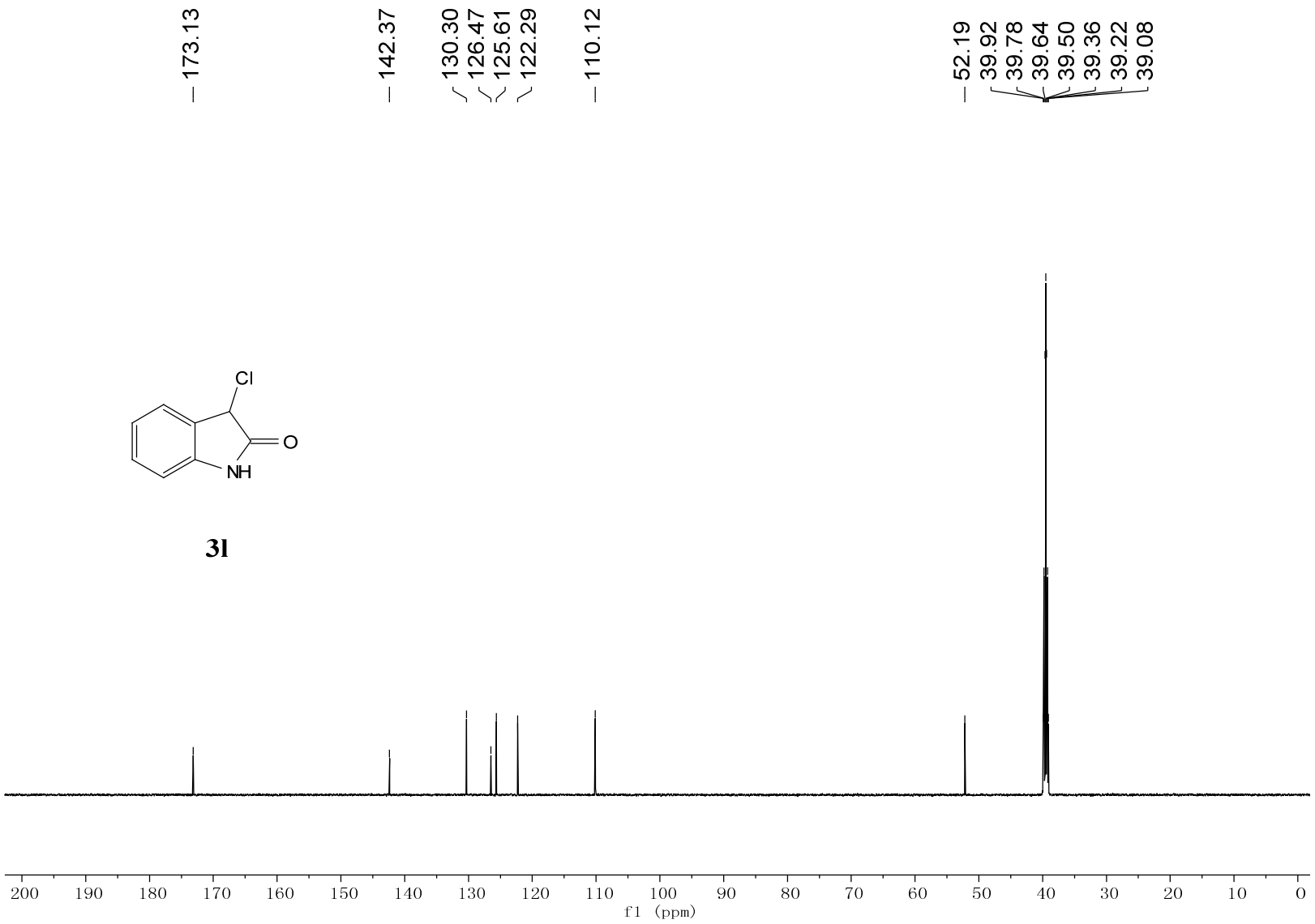
3k


171.90
142.91
140.89
125.41
123.57
122.81
109.56
77.21
77.00
76.79
51.53
35.02
21.87
12.39
Cl
O
N
H3C
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

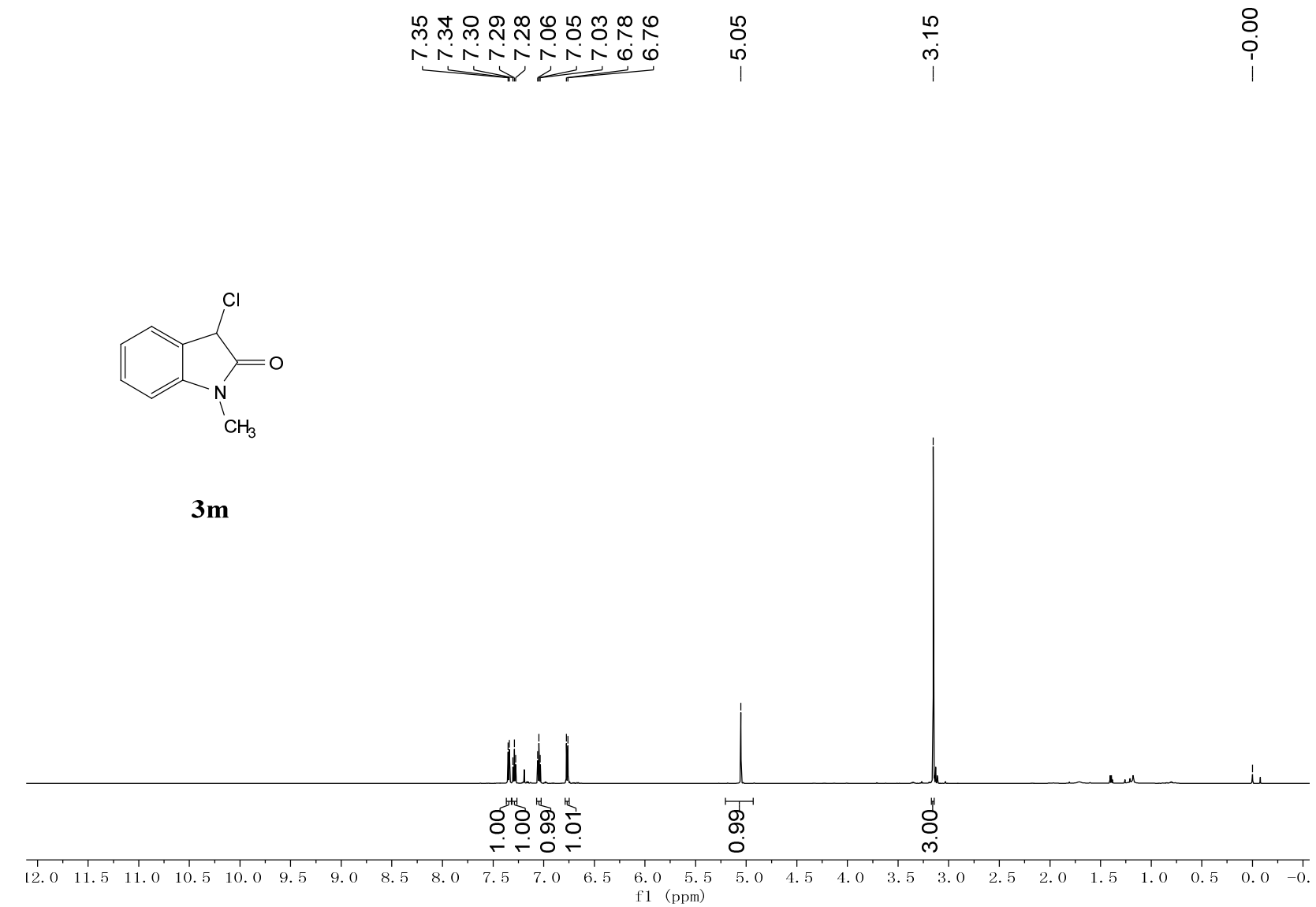
3k


10.76
7.37
7.35
7.30
7.29
7.28
7.05
7.03
7.02
6.88
6.86
5.57
3.37
2.50
Cl
O
NH
0.98
1.01
1.00
1.01
1.00
0.93
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

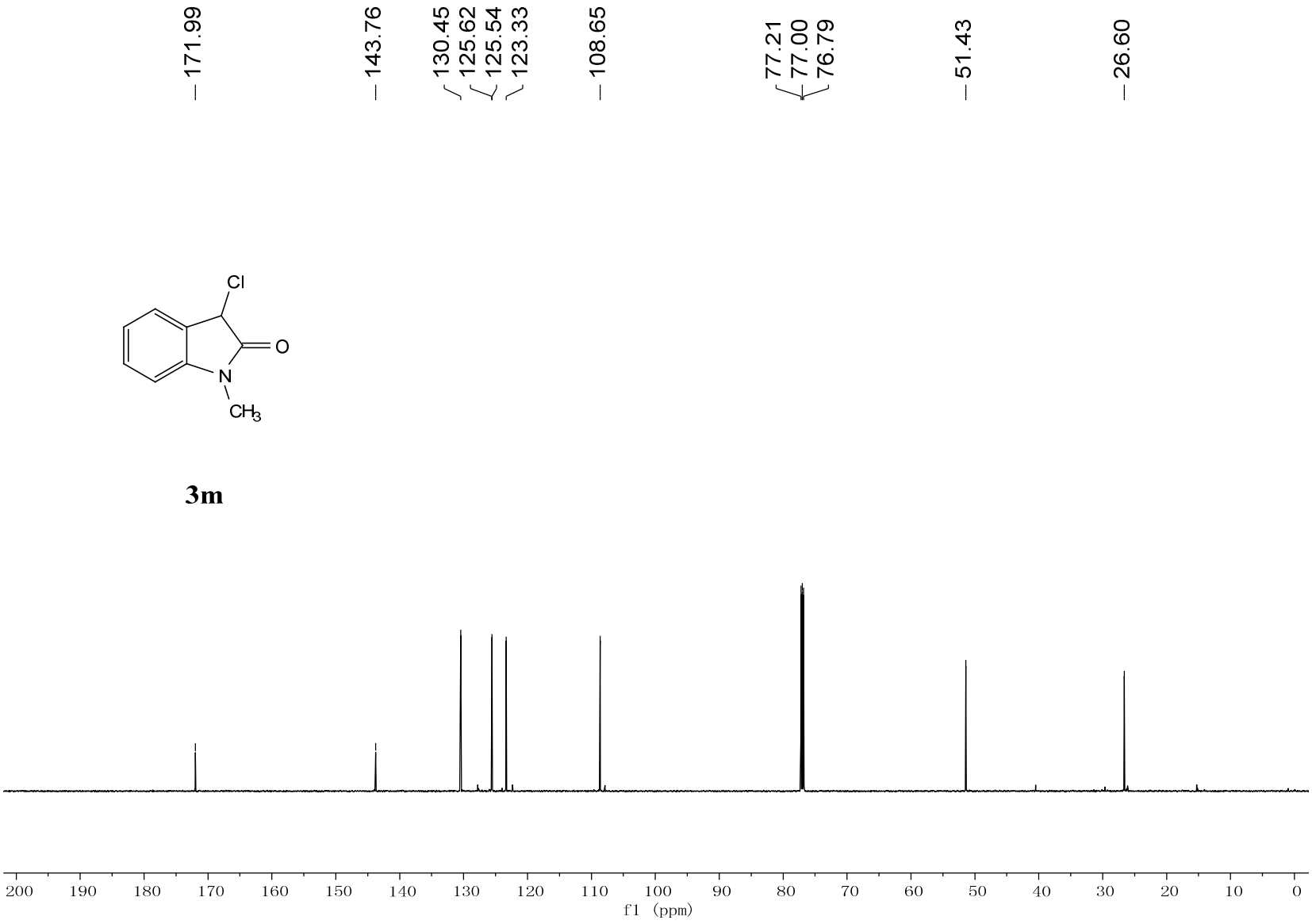
3l


173.13
142.37
130.30
126.47
125.61
122.29
110.12
52.19
39.92
39.78
39.64
39.50
39.36
39.22
39.08
Cl
O
NH
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

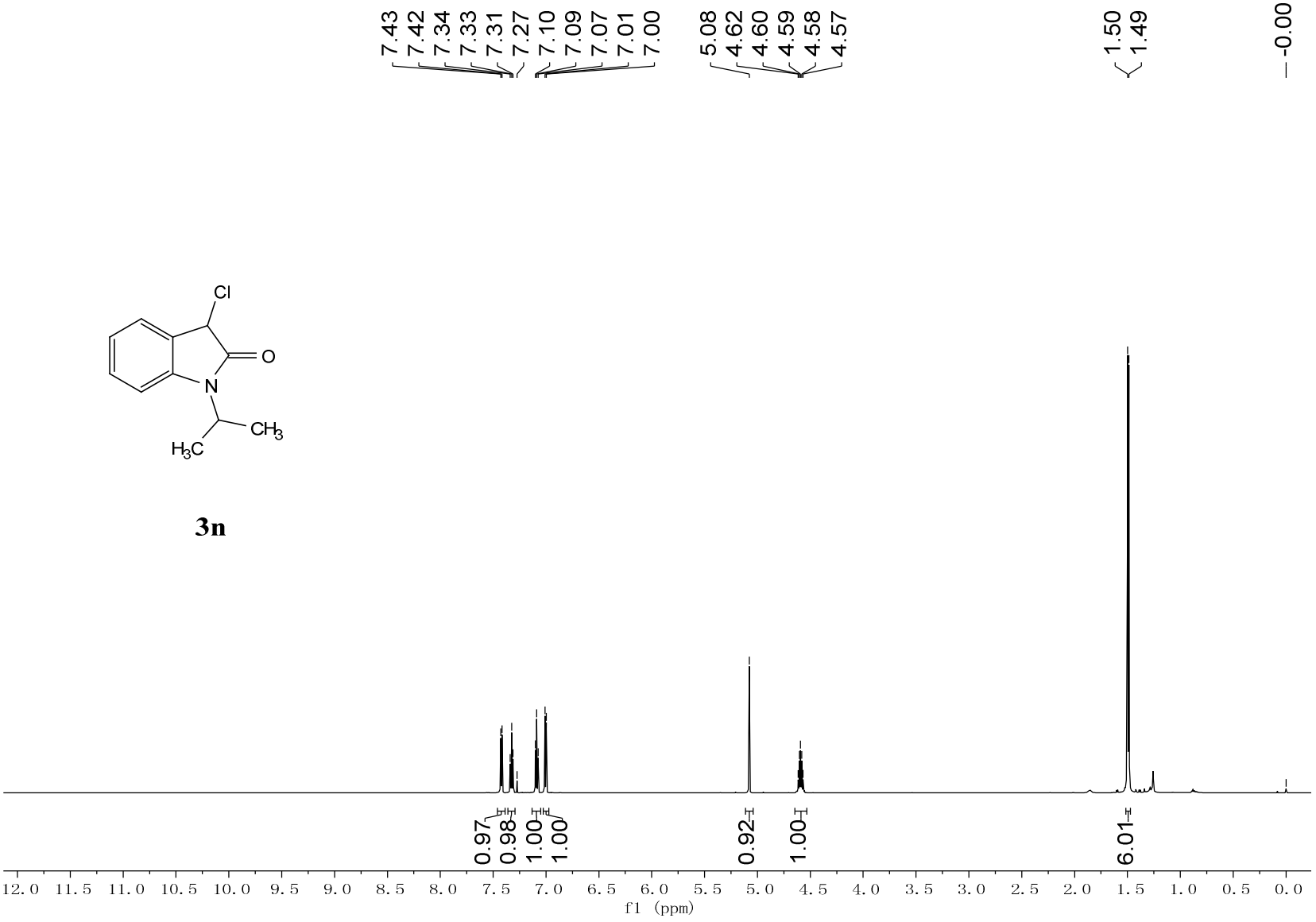
3l


7.35
7.34
7.30
7.29
7.28
7.06
7.05
7.03
6.78
6.76
5.05
3.15
-0.00
Cl
O
N
CH3
1.00
1.00
0.99
1.01
0.99
3.00
f1 (ppm)

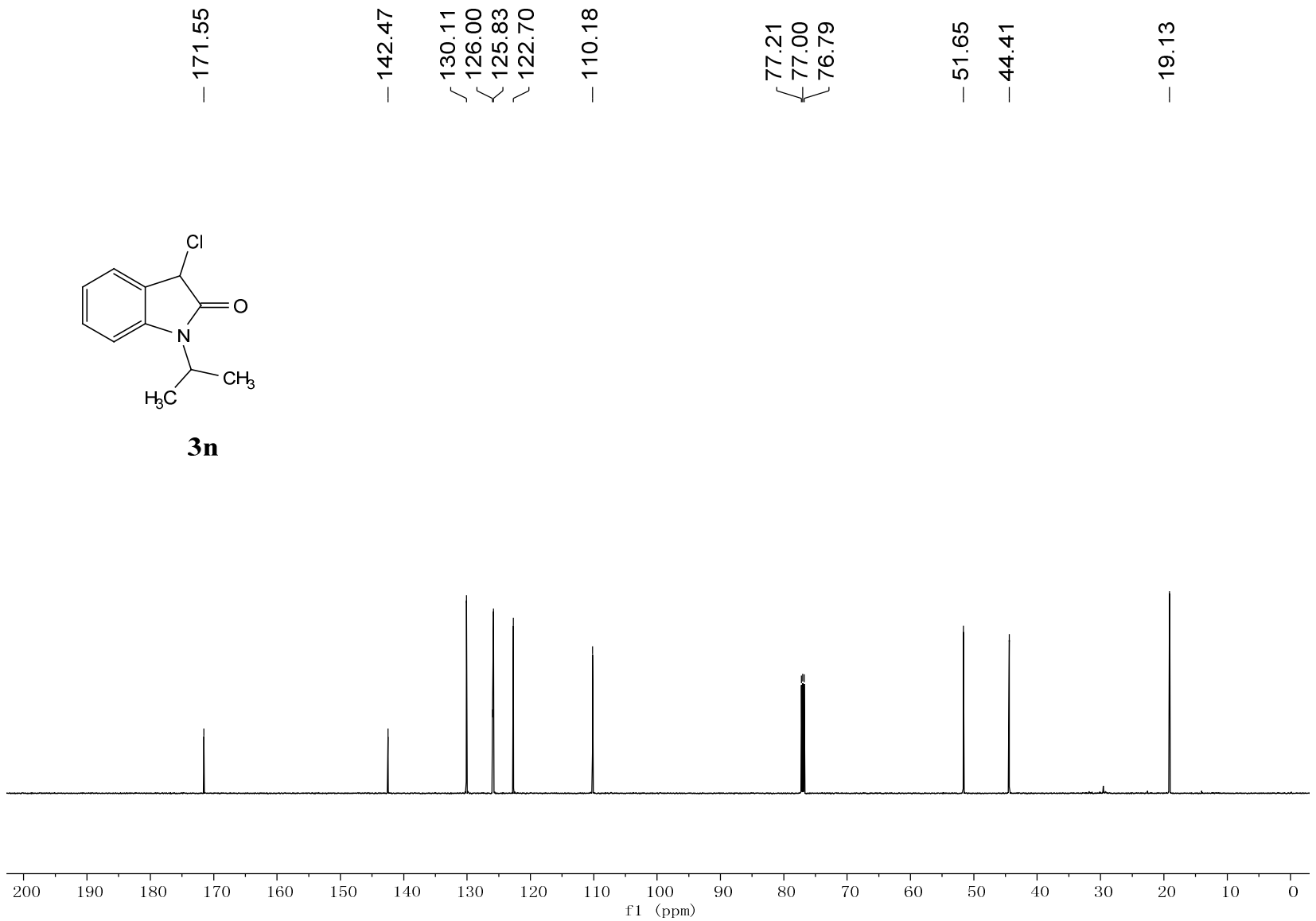
3m


171.99
143.76
130.45
125.62
125.54
123.33
108.65
77.21
77.00
76.79
51.43
26.60
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

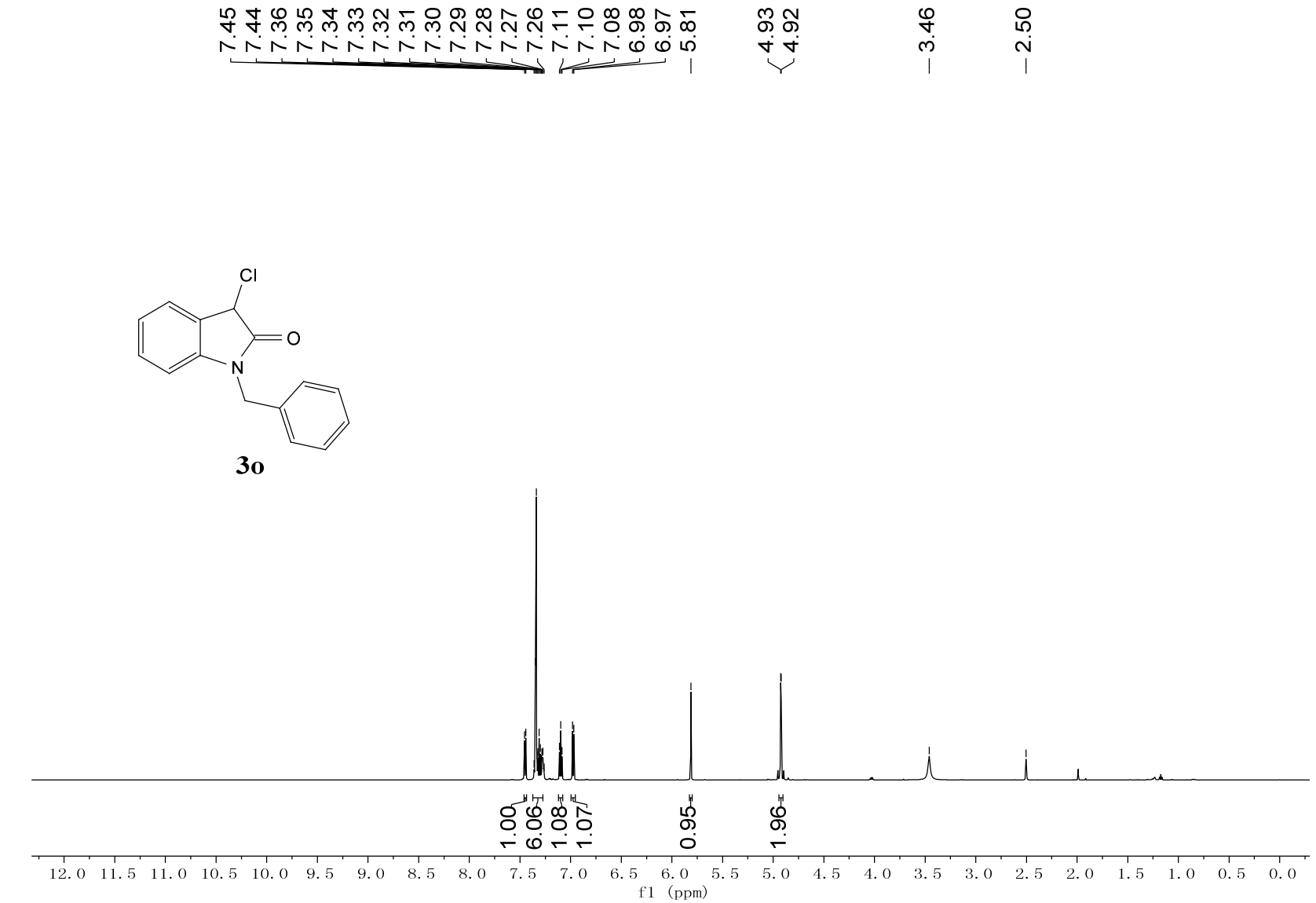
3m


7.43
7.42
7.34
7.33
7.31
7.27
7.10
7.09
7.07
7.01
7.00
5.08
4.62
4.60
4.59
4.58
4.57
1.50
1.49
-0.00
Cl
O
N
CH3
H3C
0.97
0.98
1.00
1.00
0.92
1.00
6.01
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

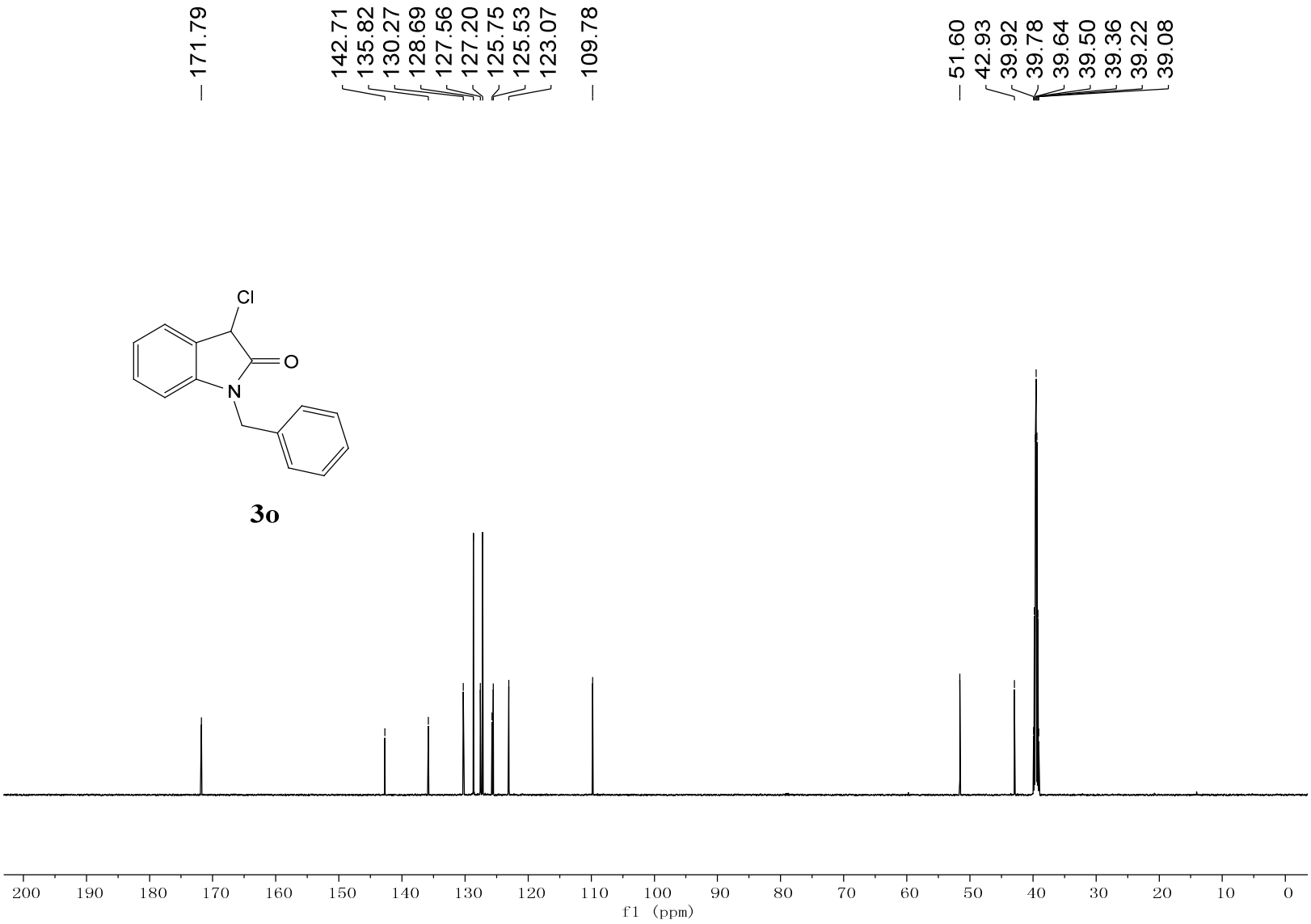
3n

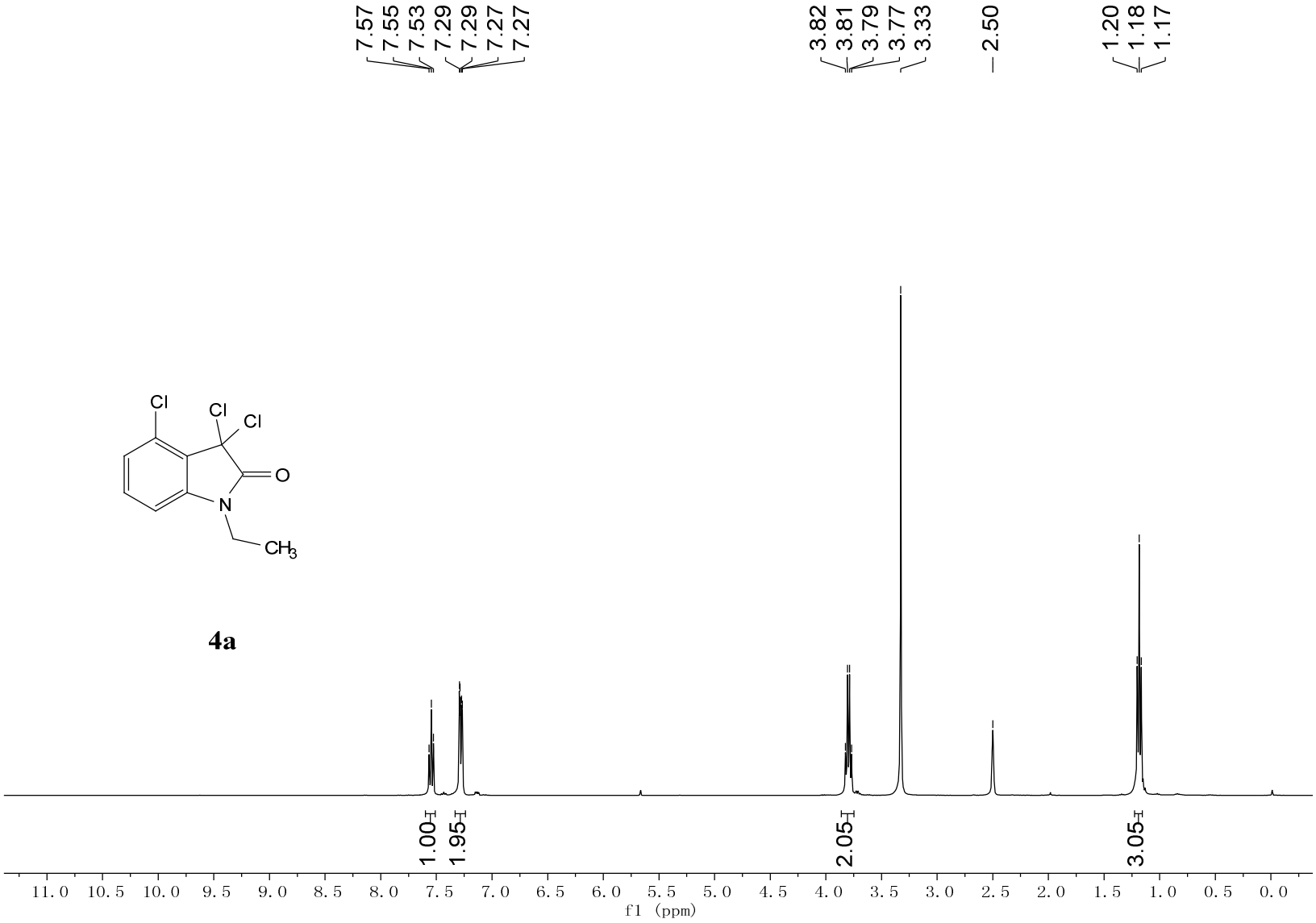

171.55
142.47
130.11
126.00
125.83
122.70
110.18
77.21
77.00
76.79
51.65
44.41
19.13
Cl
O
N
CH3
H3C
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

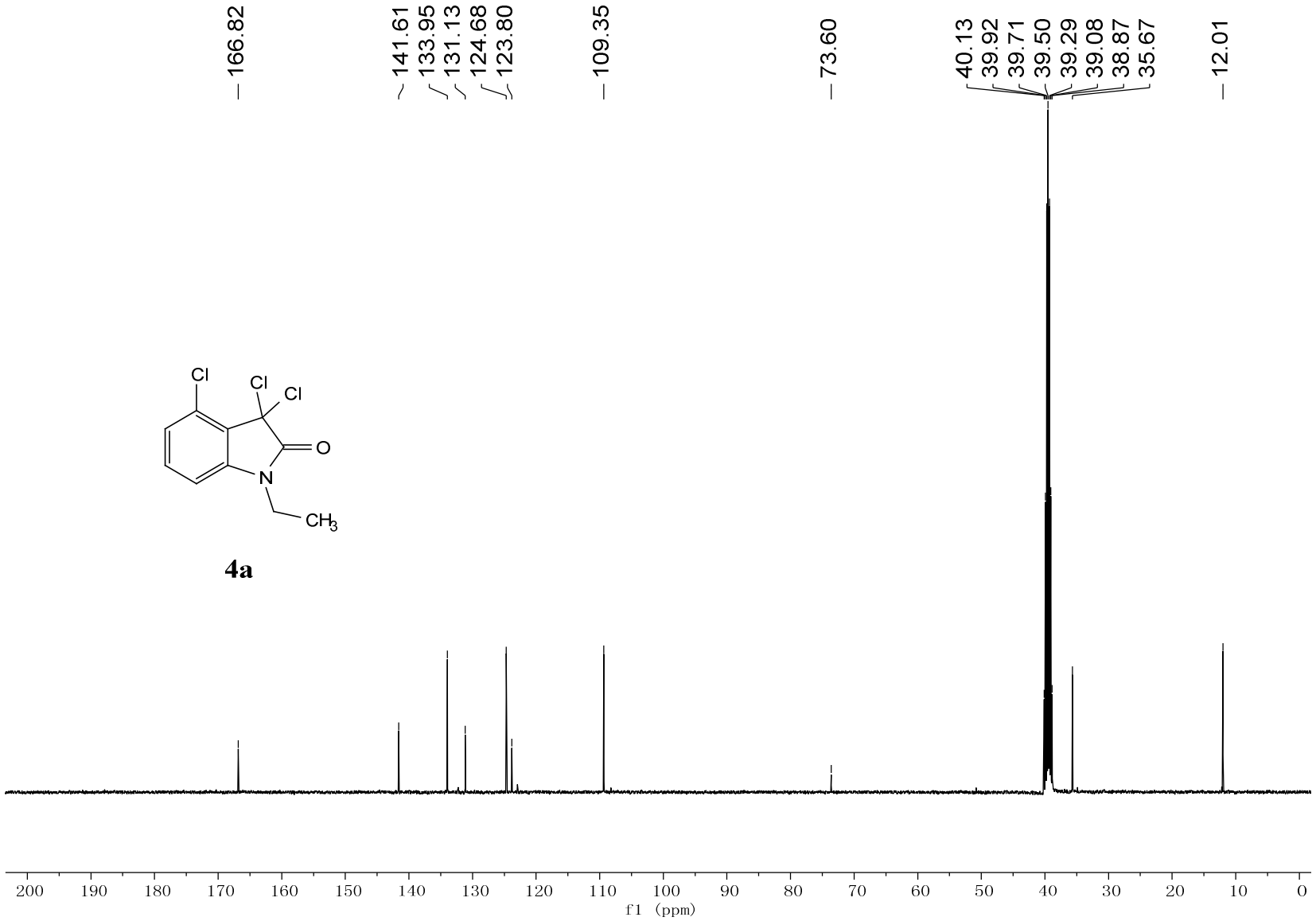
3n


7.45
7.44
7.36
7.35
7.34
7.33
7.32
7.31
7.30
7.29
7.28
7.27
7.26
7.11
7.10
7.08
6.98
6.97
5.81
4.93
4.92
3.46
2.50
Cl
O
N
1.00
6.06
1.08
1.07
0.95
1.96
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

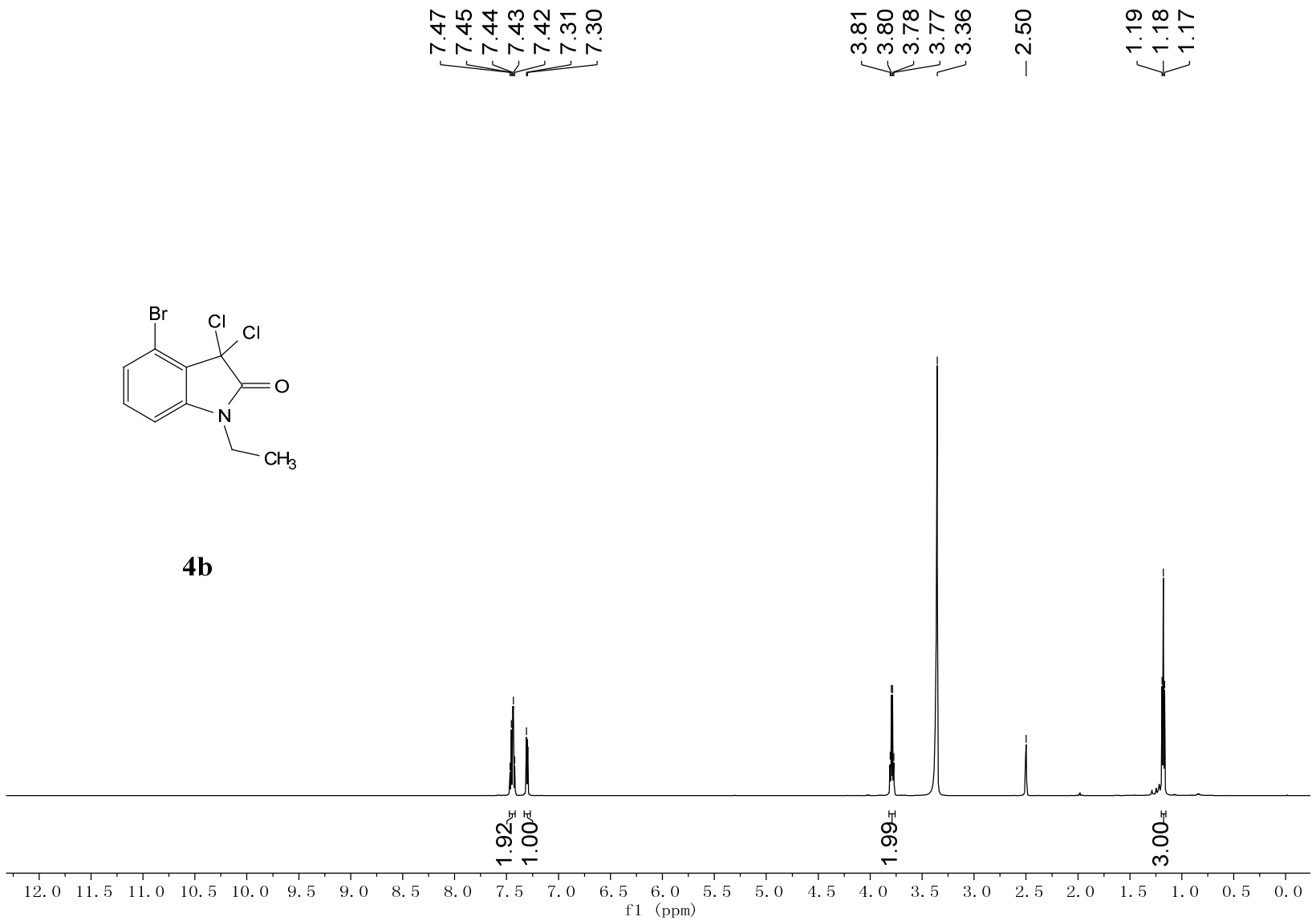
3o

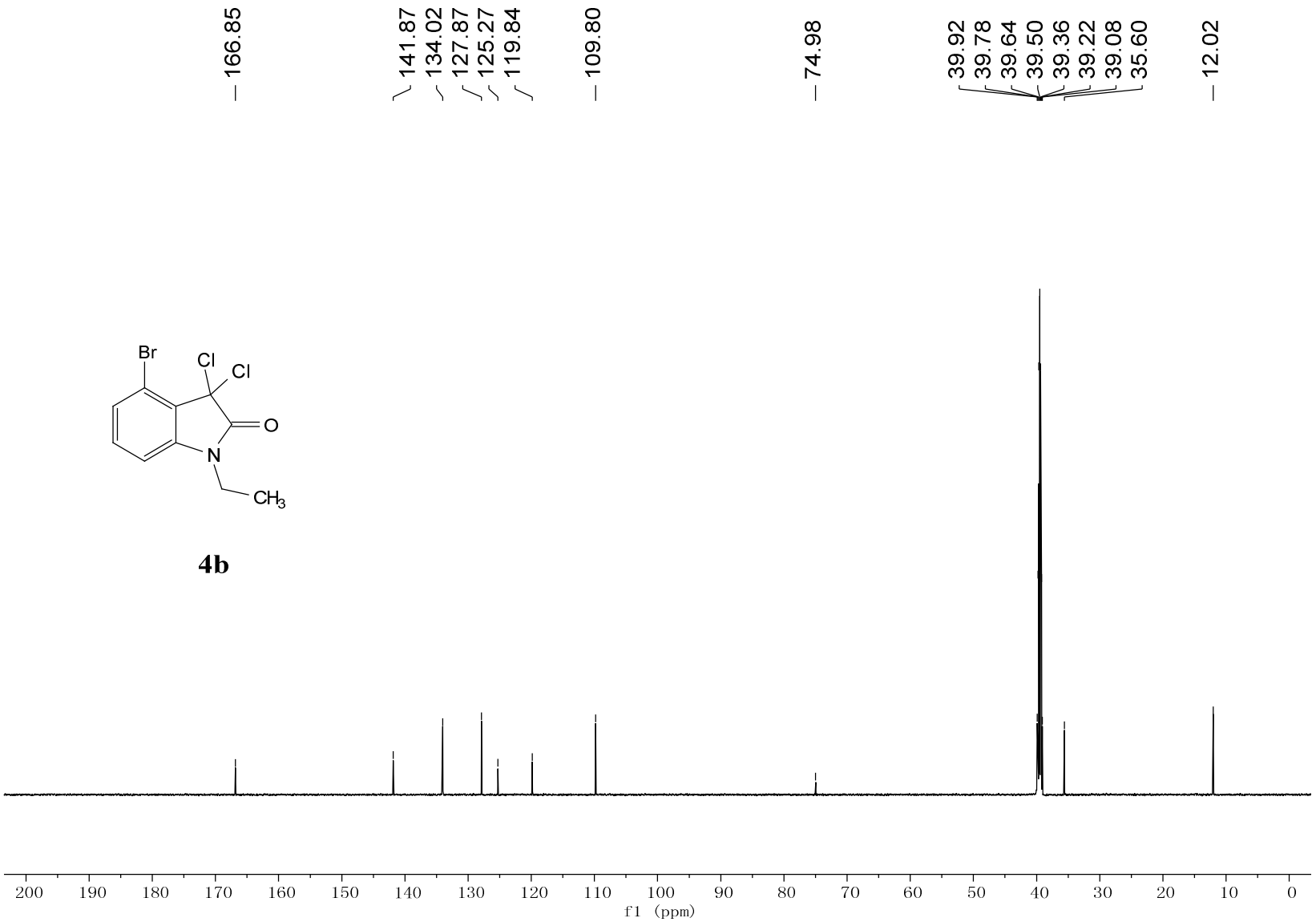

171.79
142.71
135.82
130.27
128.69
127.56
127.20
125.75
125.53
123.07
109.78
51.60
42.93
39.92
39.78
39.64
39.50
39.36
39.22
39.08
Cl
O
N
3o
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.57
7.55
7.53
7.29
7.29
7.27
7.27
3.82
3.81
3.79
3.77
3.33
2.50
1.20
1.18
1.17
Cl
Cl
Cl
O
N
CH3
4a
1.00
1.95
2.05
3.05
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

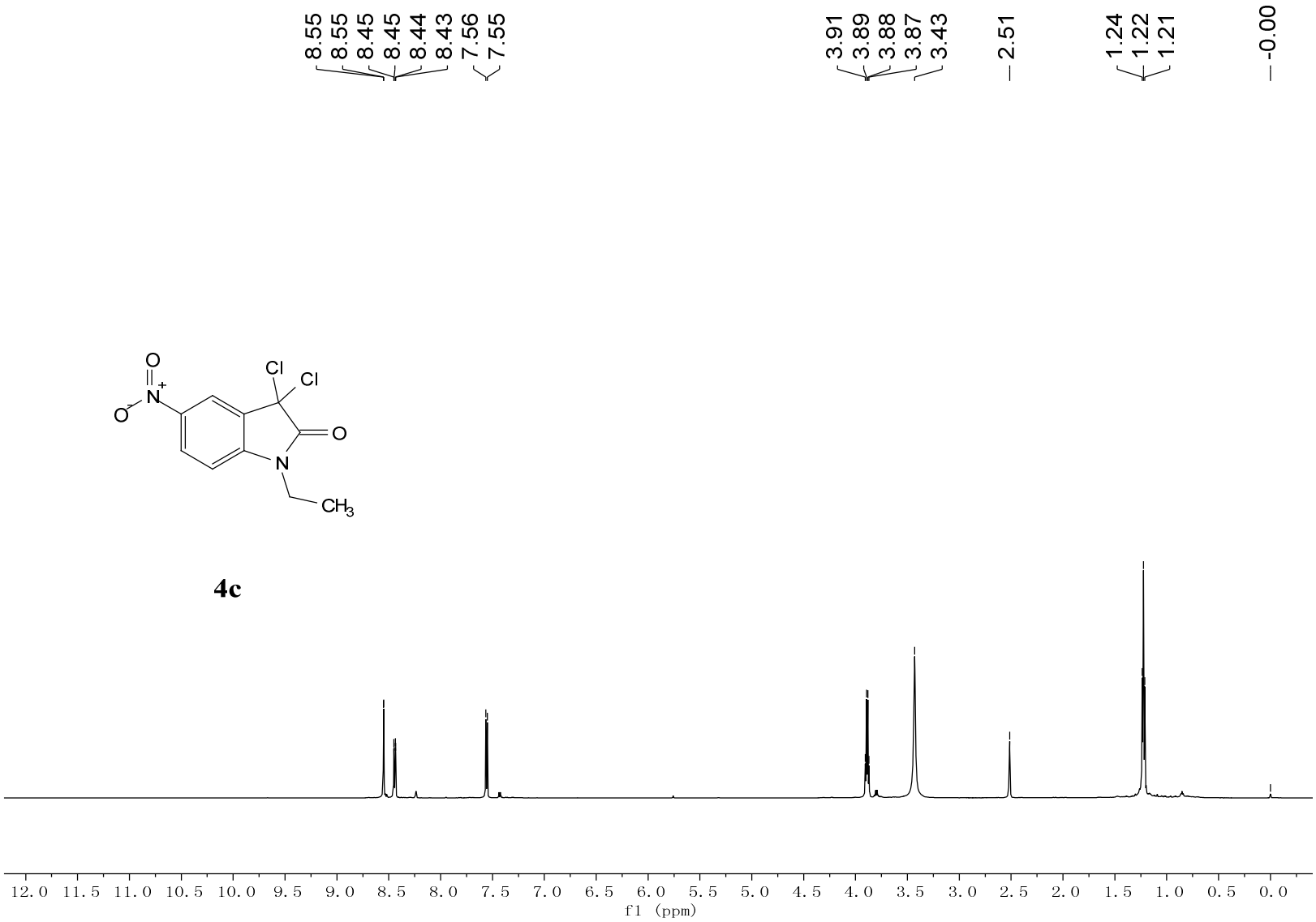

166.82
141.61
133.95
131.13
124.68
123.80
109.35
73.60
40.13
39.92
39.71
39.50
39.29
39.08
38.87
35.67
12.01
Cl
Cl
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4a

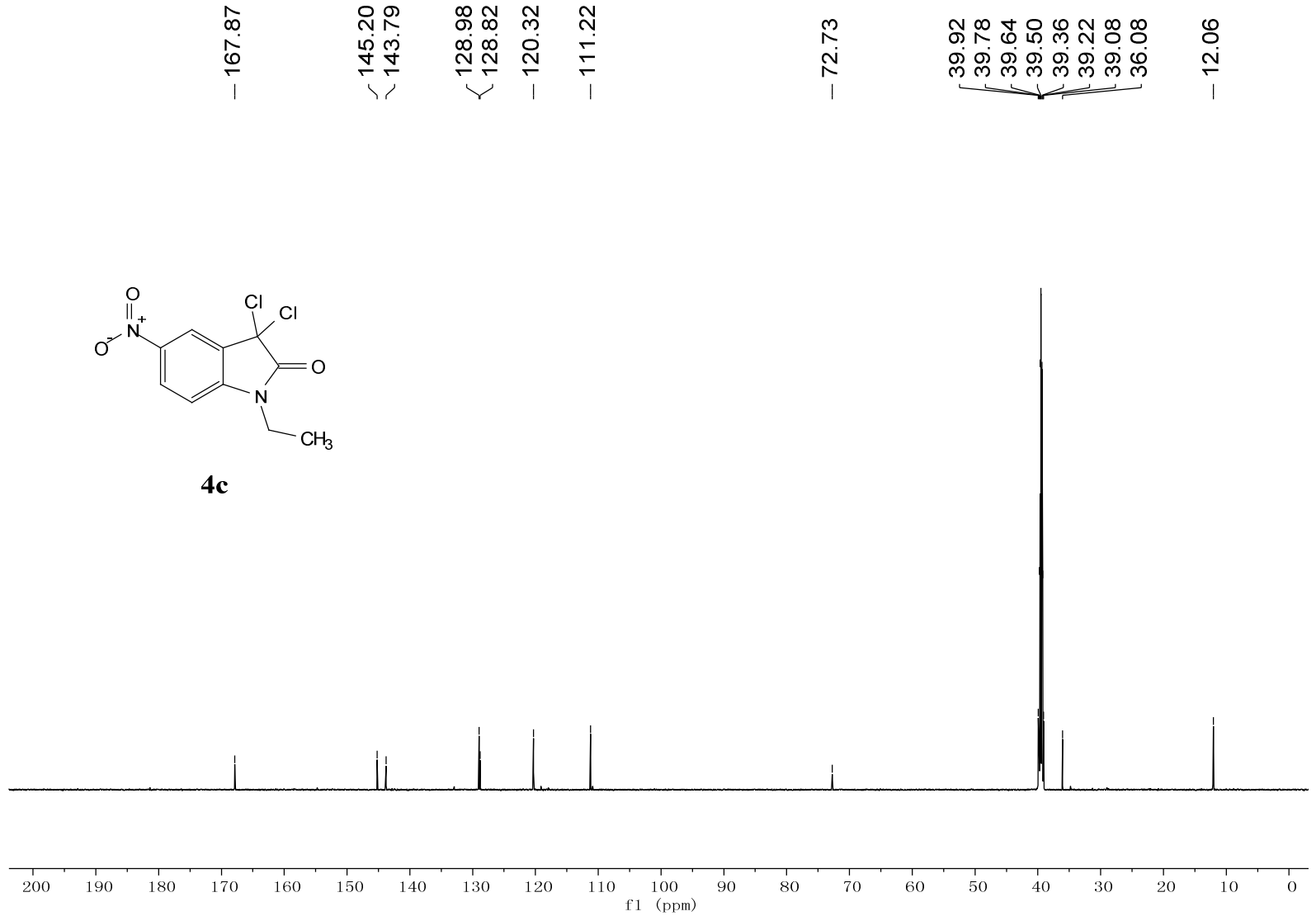

7.47
7.45
7.44
7.43
7.42
7.31
7.30
3.81
3.80
3.78
3.77
3.36
2.50
1.19
1.18
1.17
Br
Cl
Cl
O
N
CH3
4b
1.92
1.00
1.99
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.85
141.87
134.02
127.87
125.27
119.84
109.80
74.98
39.92
39.78
39.64
39.50
39.36
39.22
39.08
35.60
12.02
Br
Cl
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

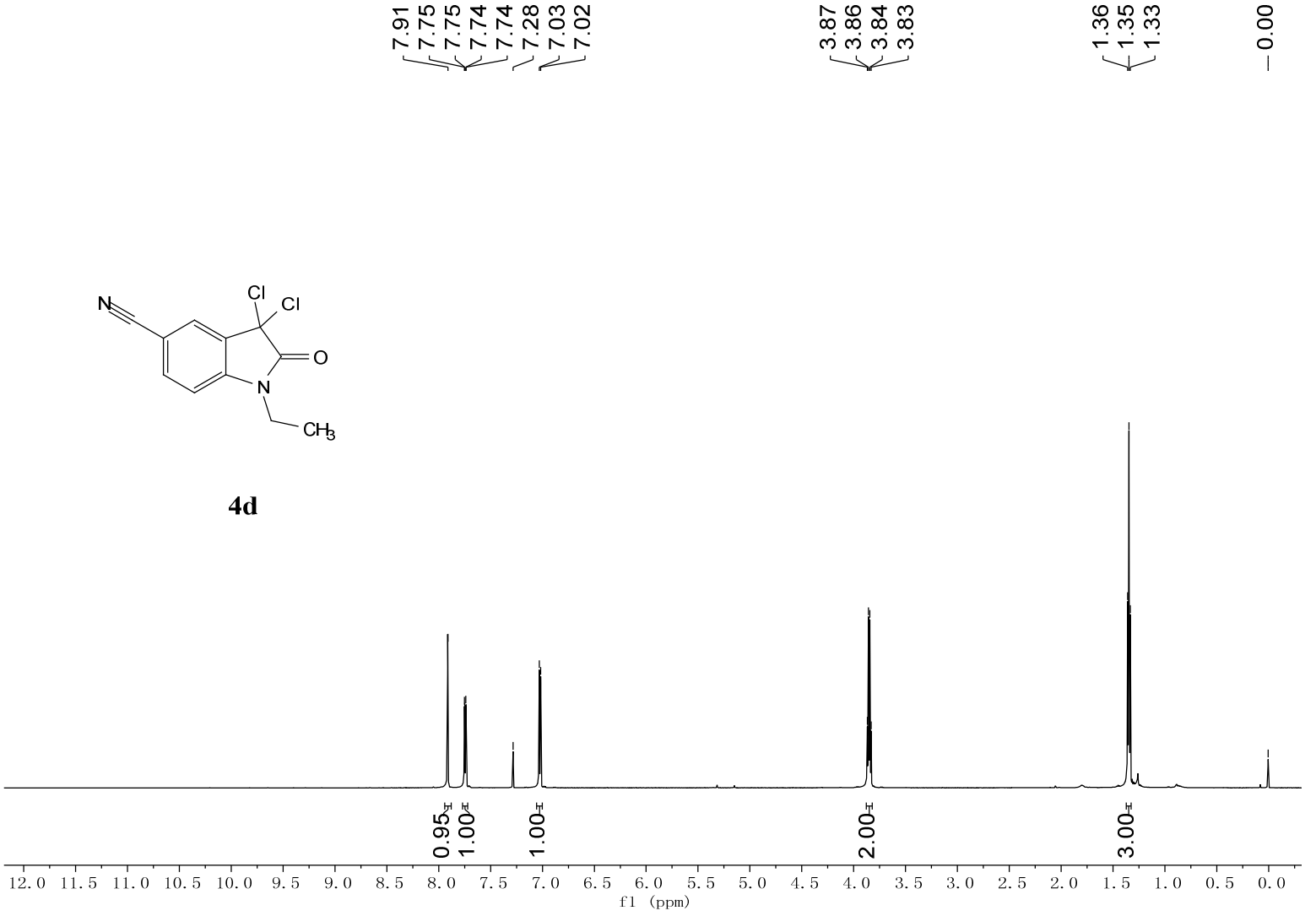
4b


8.55
8.55
8.45
8.45
8.44
8.43
7.56
7.55
3.91
3.89
3.88
3.87
3.43
2.51
1.24
1.22
1.21
-0.00
O
N+
O-
Cl
Cl
O
N
CH3
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

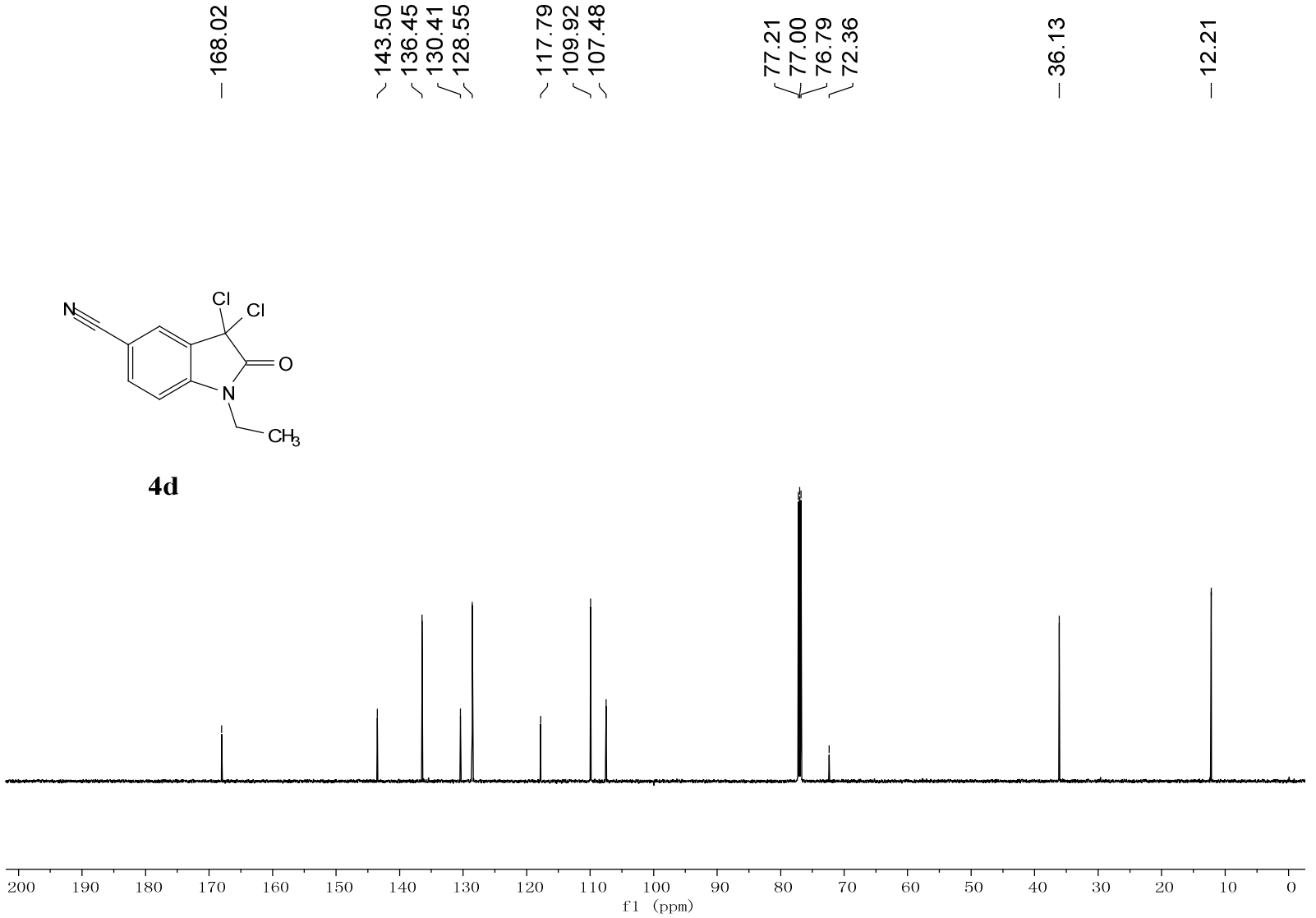
4c


167.87
145.20
143.79
128.98
128.82
120.32
111.22
72.73
39.92
39.78
39.64
39.50
39.36
39.22
39.08
36.08
12.06
O
N+
O-
Cl
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

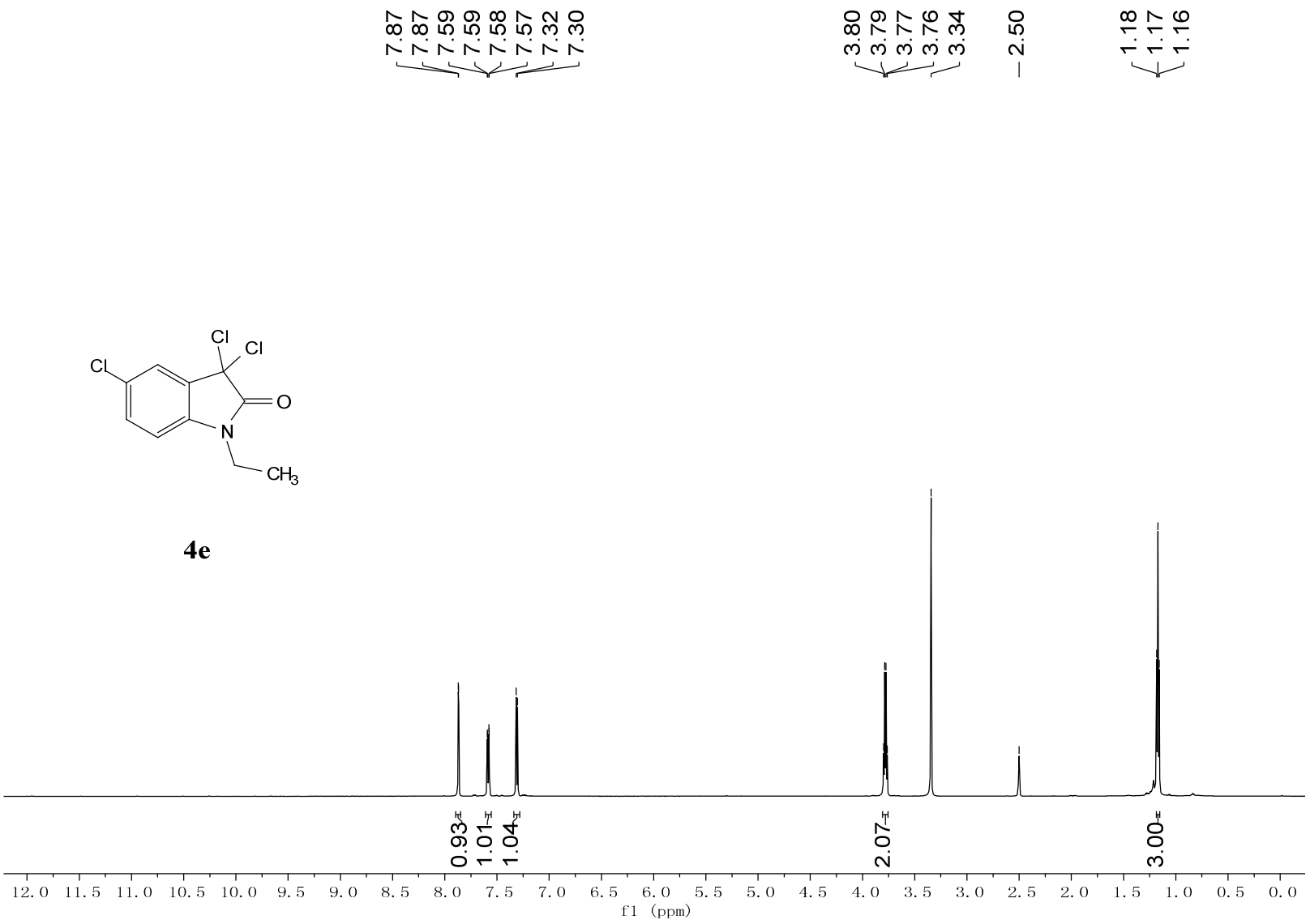
4c


7.91
7.75
7.75
7.74
7.74
7.28
7.03
7.02
3.87
3.86
3.84
3.83
1.36
1.35
1.33
0.00
Cl
Cl
N
O
N
CH3
0.95
1.00
1.00
2.00
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

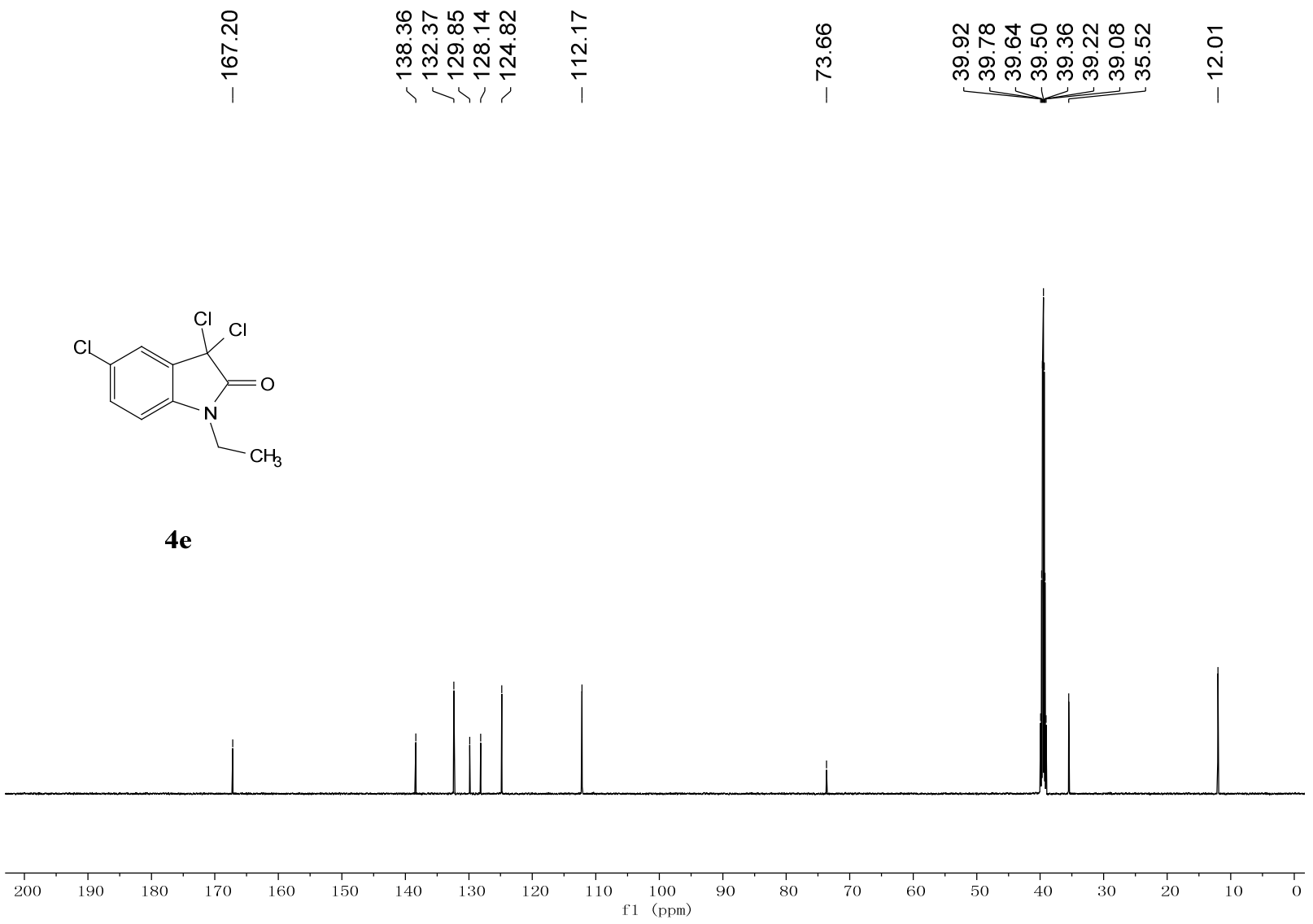
4d


168.02
143.50
136.45
130.41
128.55
117.79
109.92
107.48
77.21
77.00
76.79
72.36
36.13
12.21
Cl
Cl
N
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

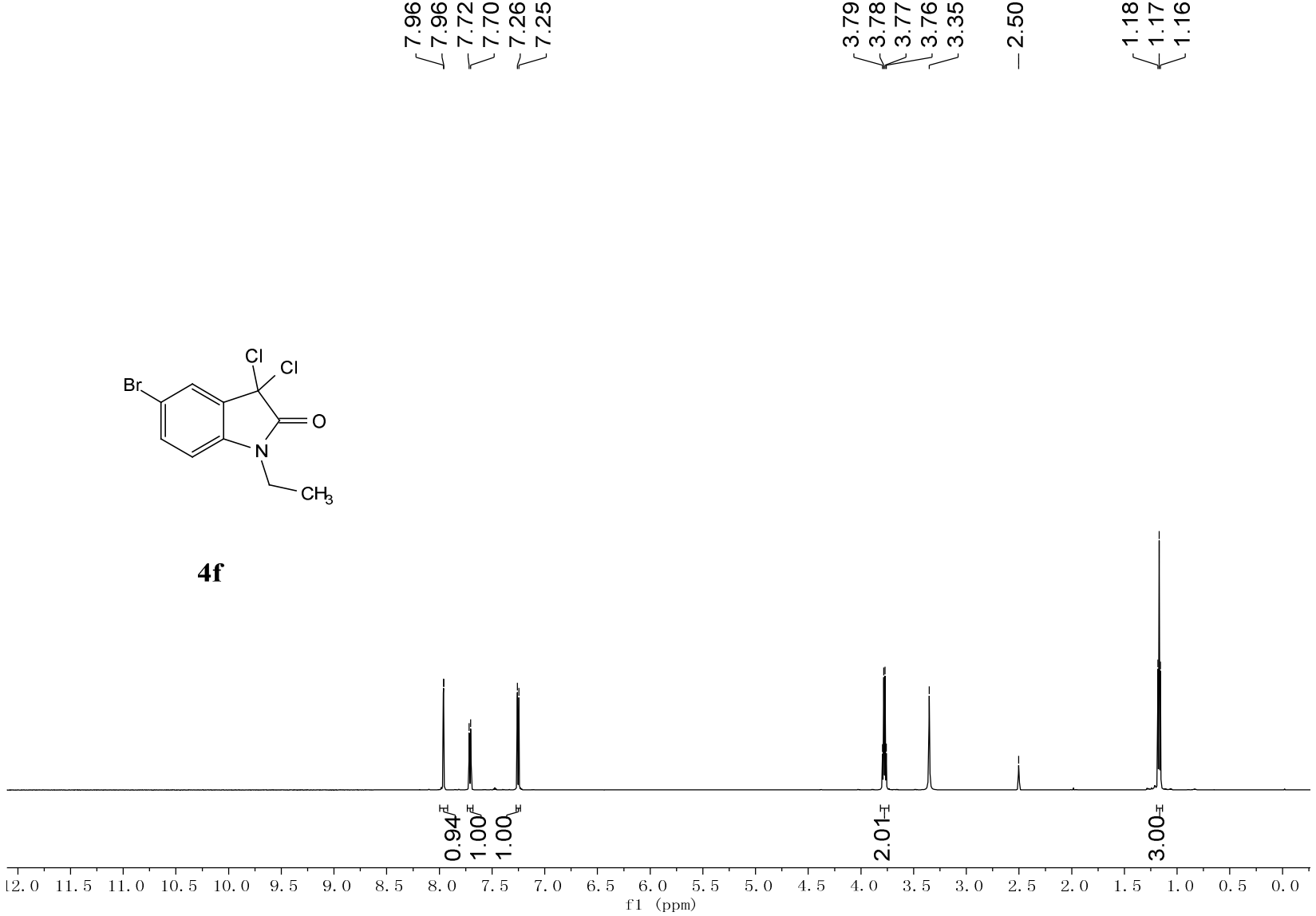
4d


7.87
7.87
7.59
7.59
7.58
7.57
7.32
7.30
3.80
3.79
3.77
3.76
3.34
2.50
1.18
1.17
1.16
Cl
Cl
Cl
O
N
CH3
0.93
1.01
1.04
2.07
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

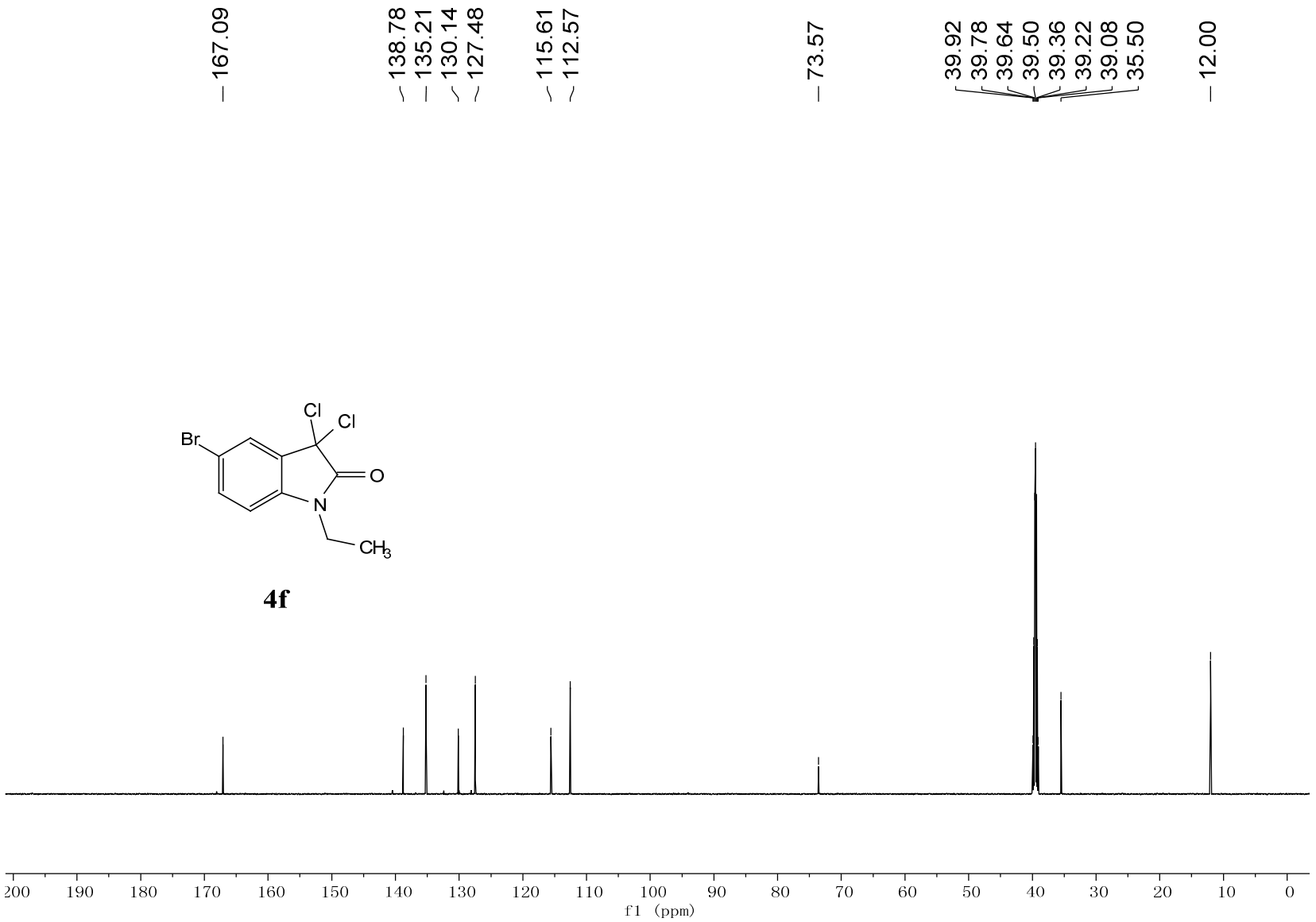
4e


167.20
138.36
132.37
129.85
128.14
124.82
112.17
73.66
39.92
39.78
39.64
39.50
39.36
39.22
39.08
35.52
12.01
Cl
Cl
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

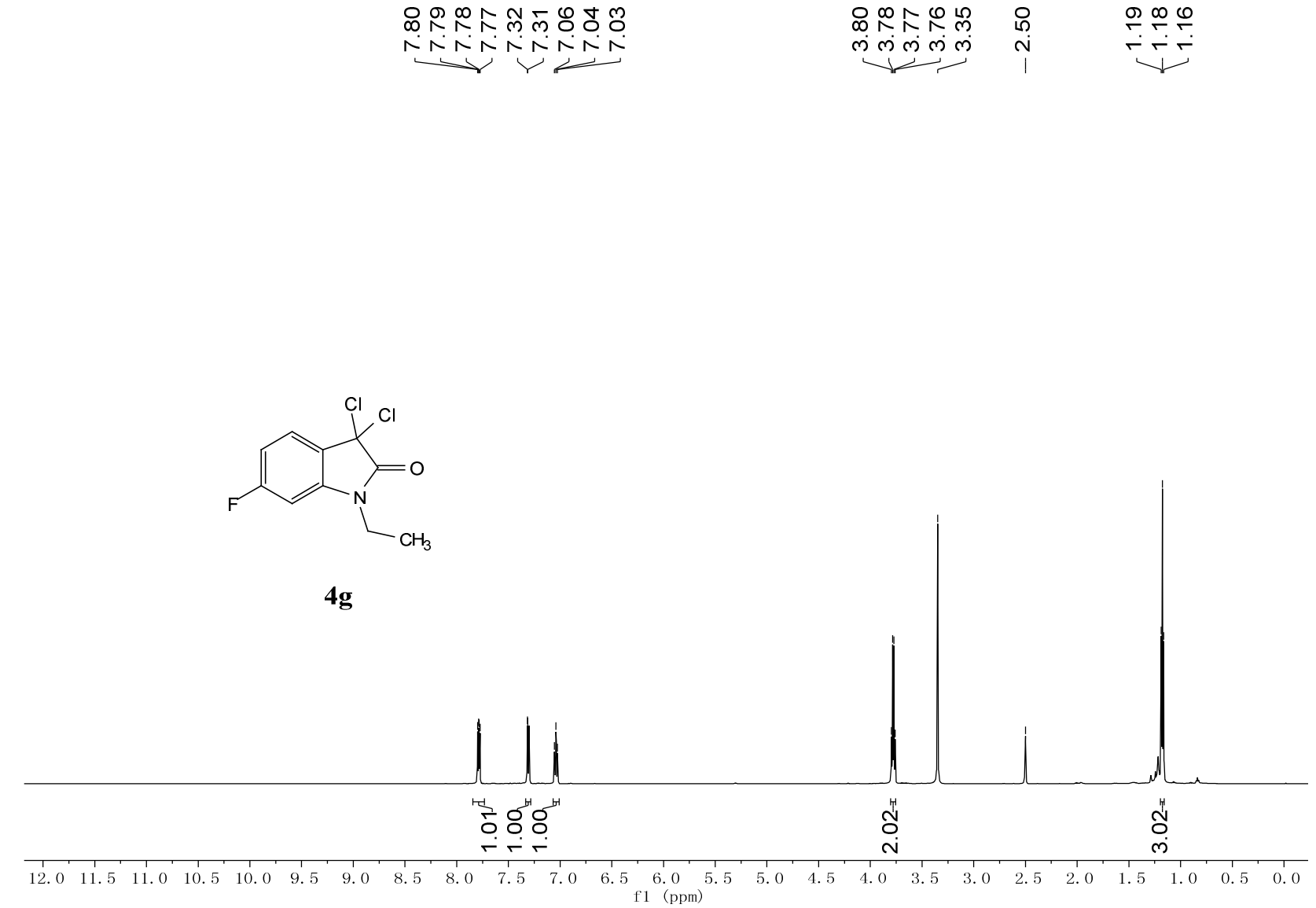
4e

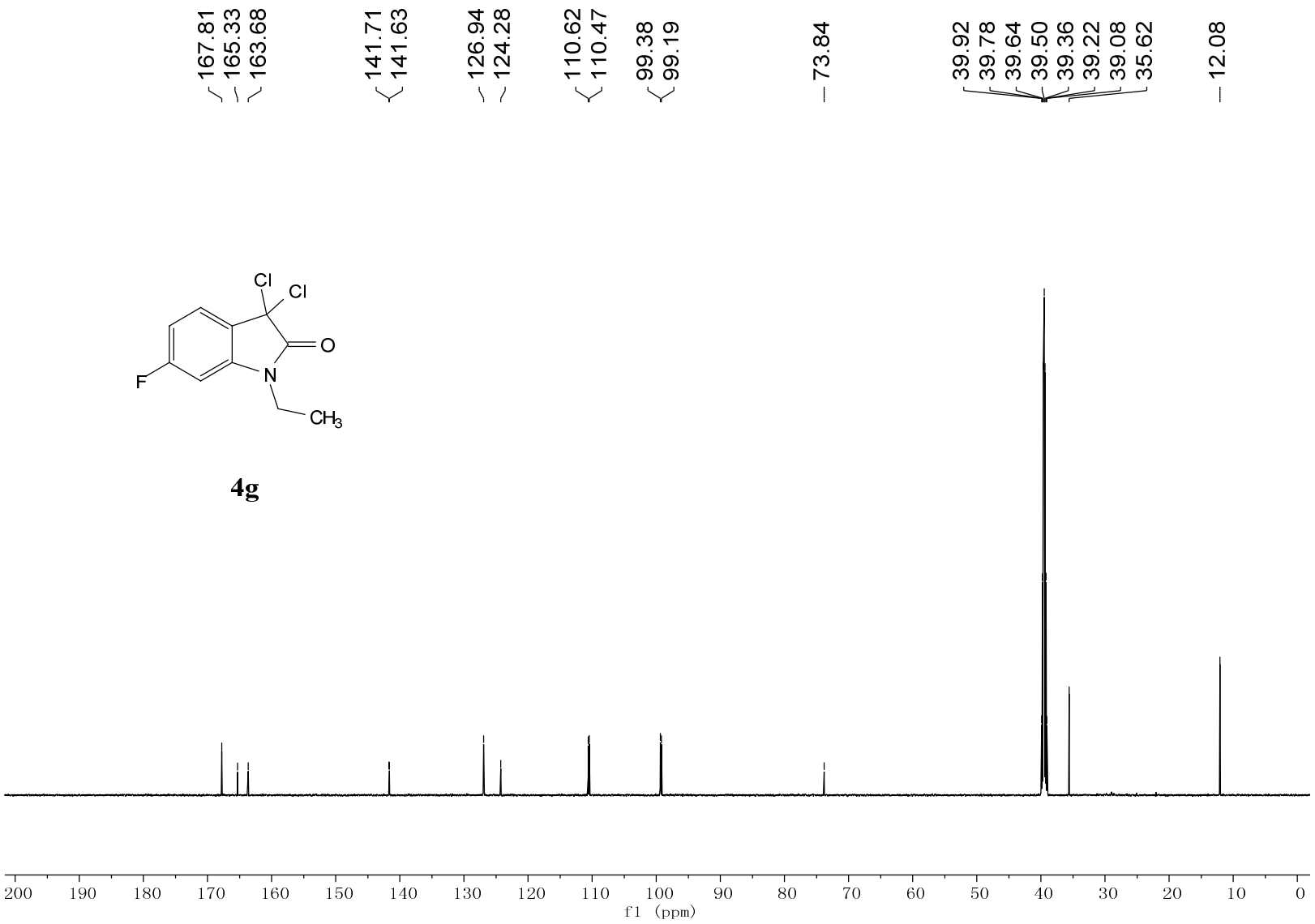

7.96
7.96
7.72
7.70
7.26
7.25
3.79
3.78
3.77
3.76
3.35
2.50
1.18
1.17
1.16
Cl
Cl
Br
O
N
CH3
0.94
1.00
1.00
2.01
3.00
f1 (ppm)

4f

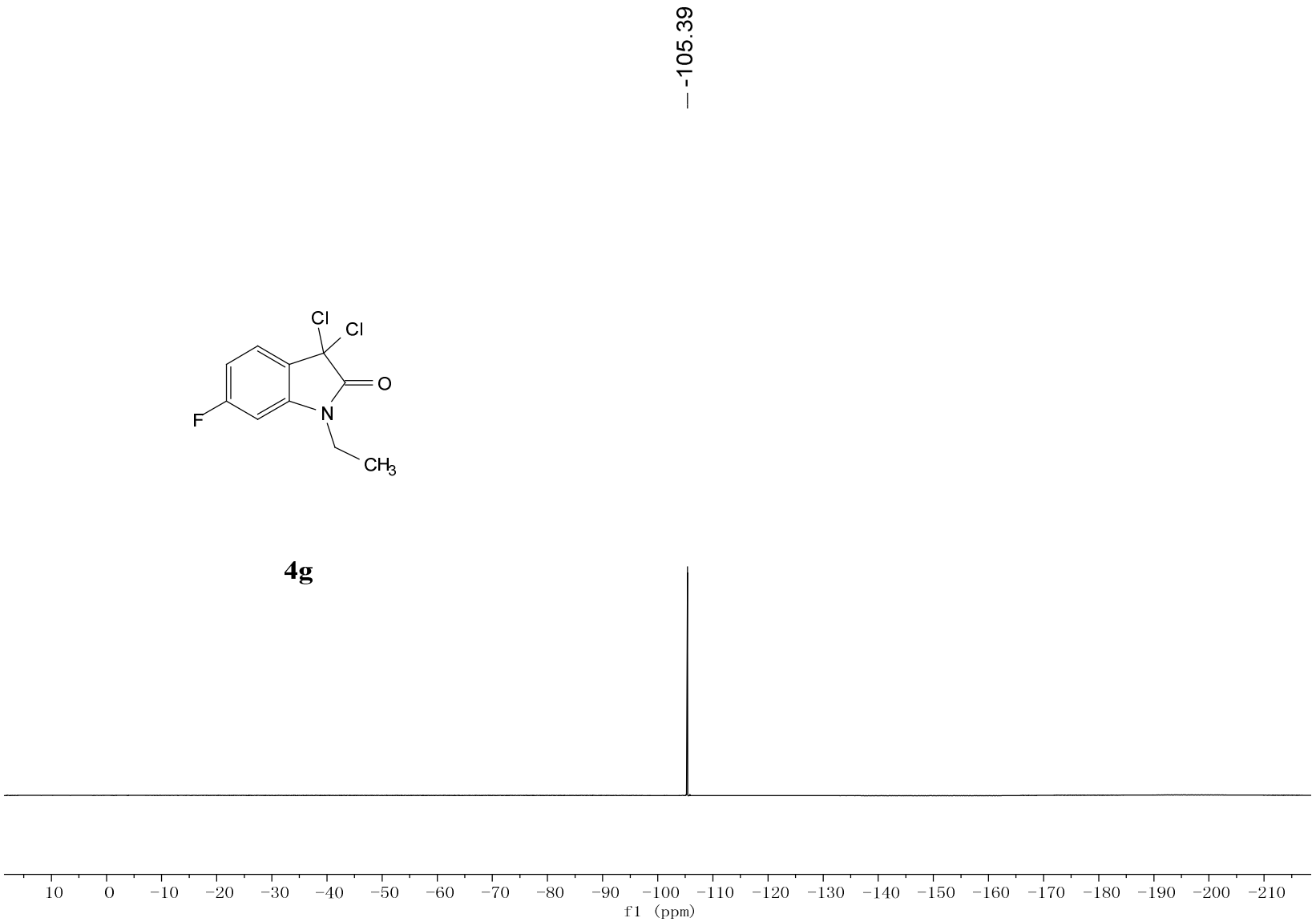

167.09
138.78
135.21
130.14
127.48
115.61
112.57
73.57
39.92
39.78
39.64
39.50
39.36
39.22
39.08
35.50
12.00
Cl
Cl
Br
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4f

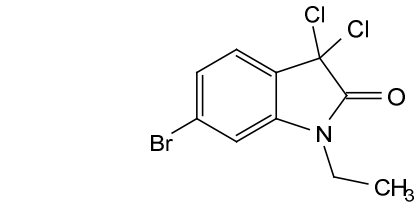

7.80
7.79
7.78
7.77
7.32
7.31
7.06
7.04
7.03
3.80
3.78
3.77
3.76
3.35
2.50
1.19
1.18
1.16
Cl
Cl
O
F
N
CH3
4g
1.01
1.00
1.00
2.02
3.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


167.81
165.33
163.68
141.71
141.63
126.94
124.28
110.62
110.47
99.38
99.19
73.84
39.92
39.78
39.64
39.50
39.36
39.22
39.08
35.62
12.08
Cl
Cl
O
F
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4g

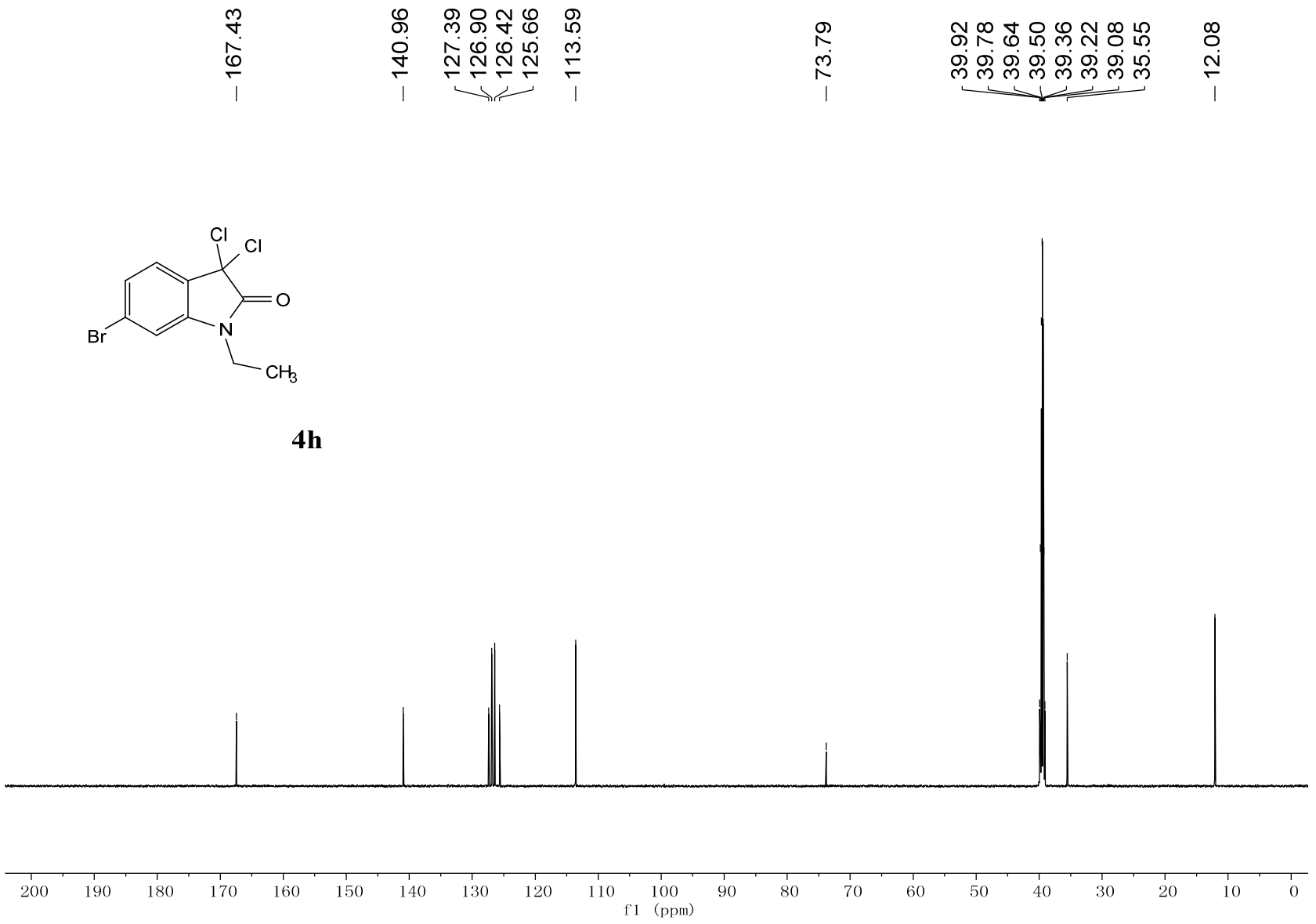

-105.39
Cl
Cl
O
F
N
CH3
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200
−210
f1 (ppm)

4g

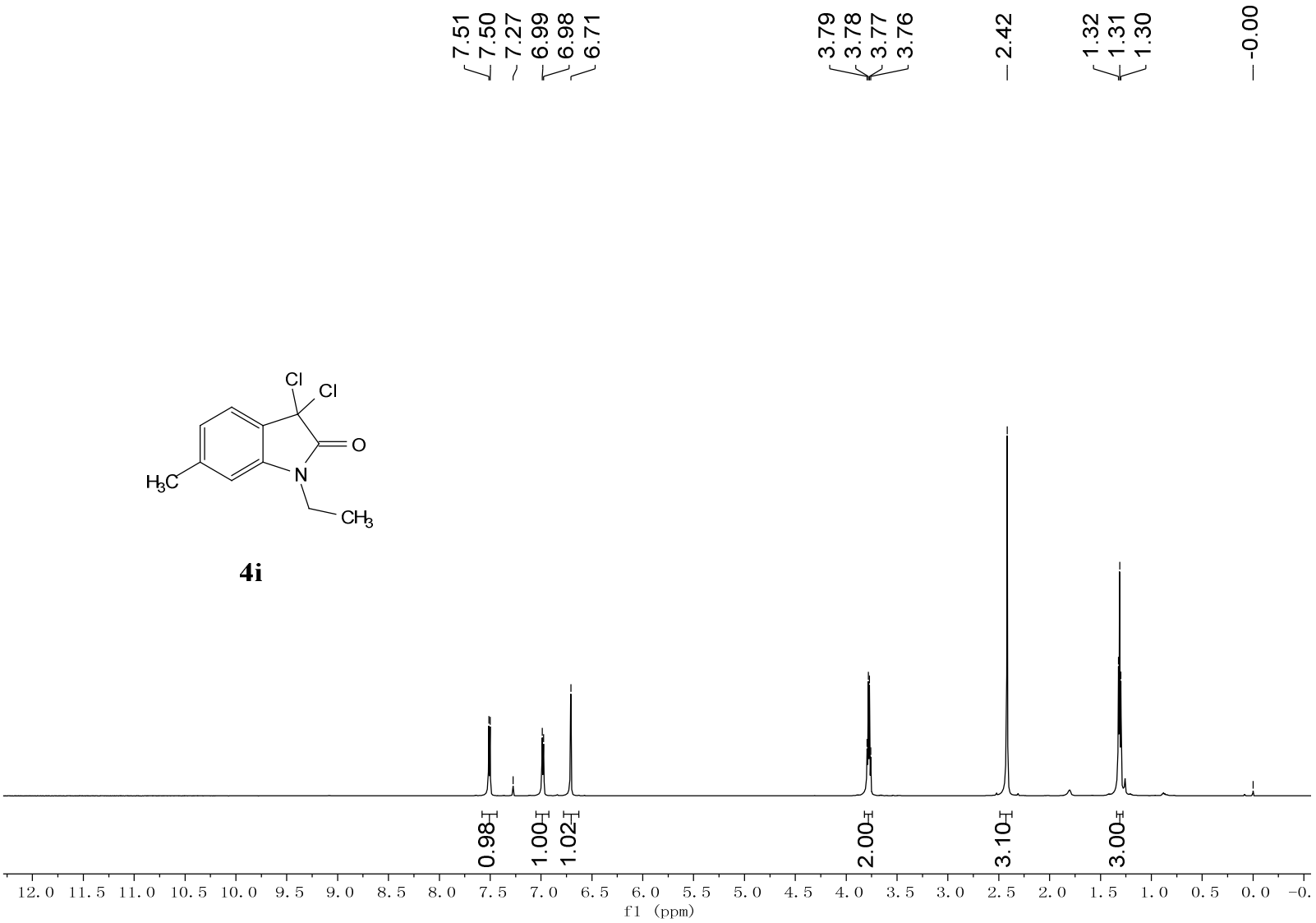

Cl
Cl
O
Br
N
CH3

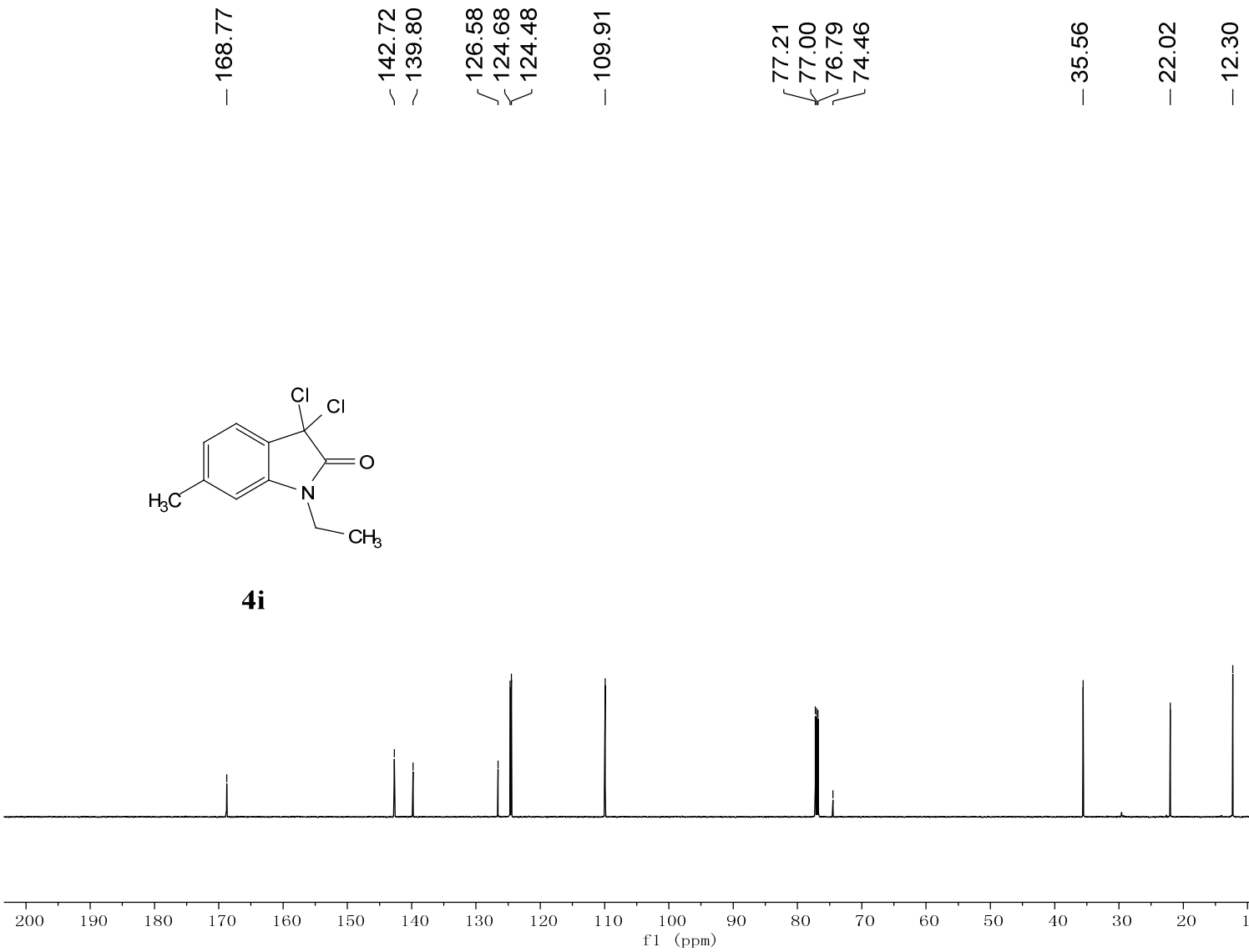
4h

7.68
7.66
7.60
7.60
7.43
7.43
7.42
7.42
3.81
3.79
3.78
3.77
3.35
2.50
1.18
1.17
1.15
1.00
1.00
1.00
2.02
2.99
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

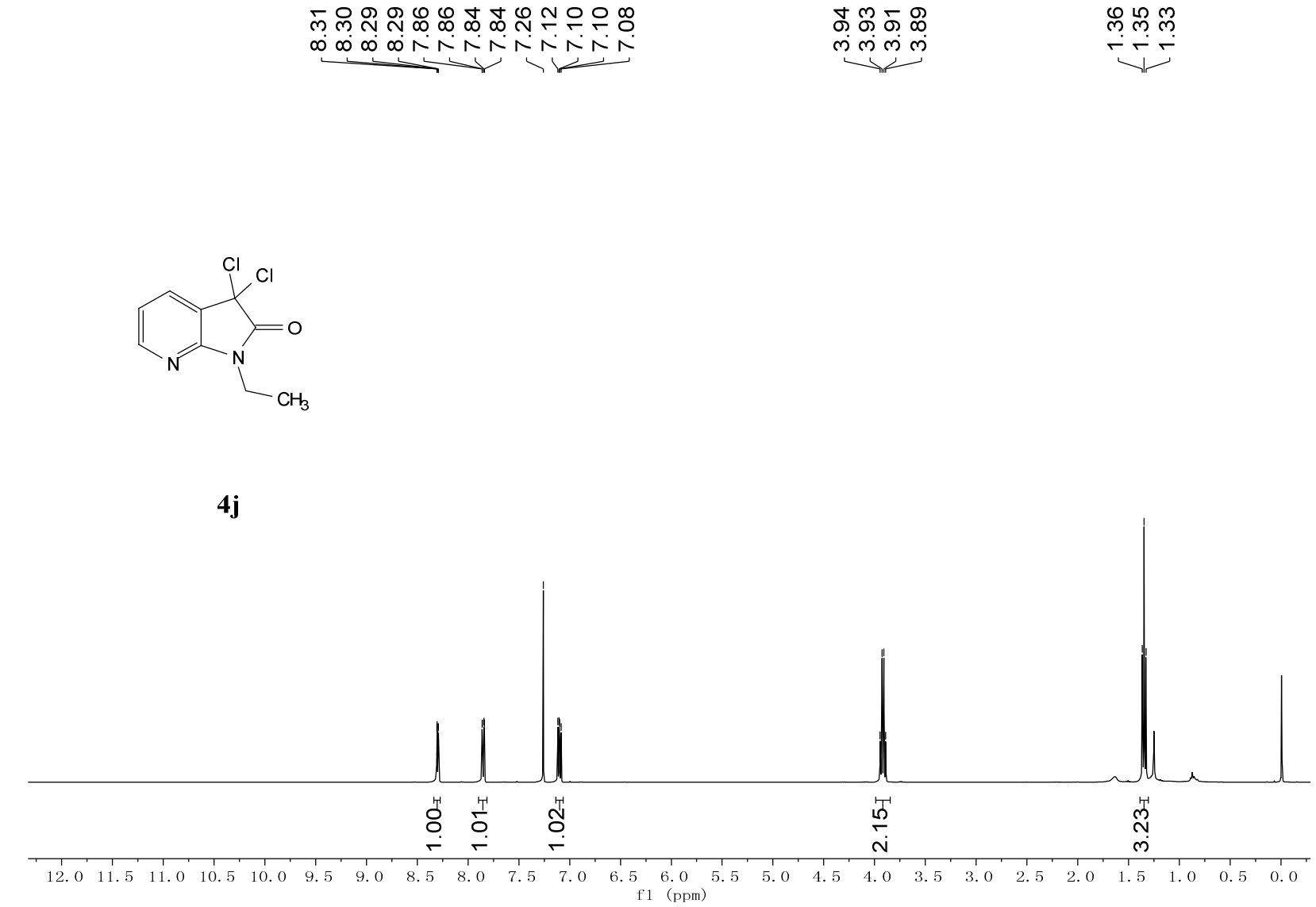

167.43
140.96
127.39
126.90
126.42
125.66
113.59
73.79
39.92
39.78
39.64
39.50
39.36
39.22
39.08
35.55
12.08
Cl
Cl
O
Br
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

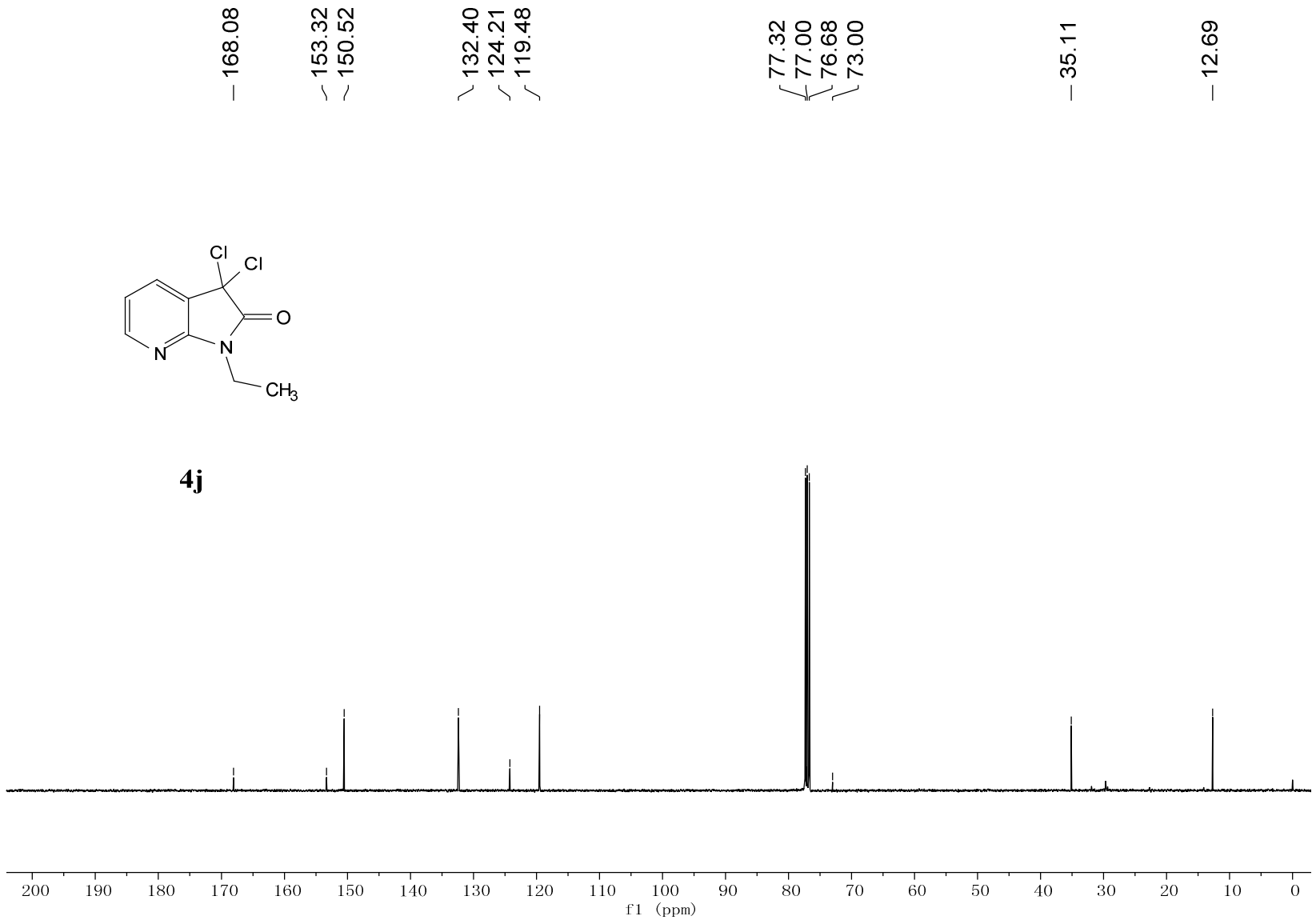
4h

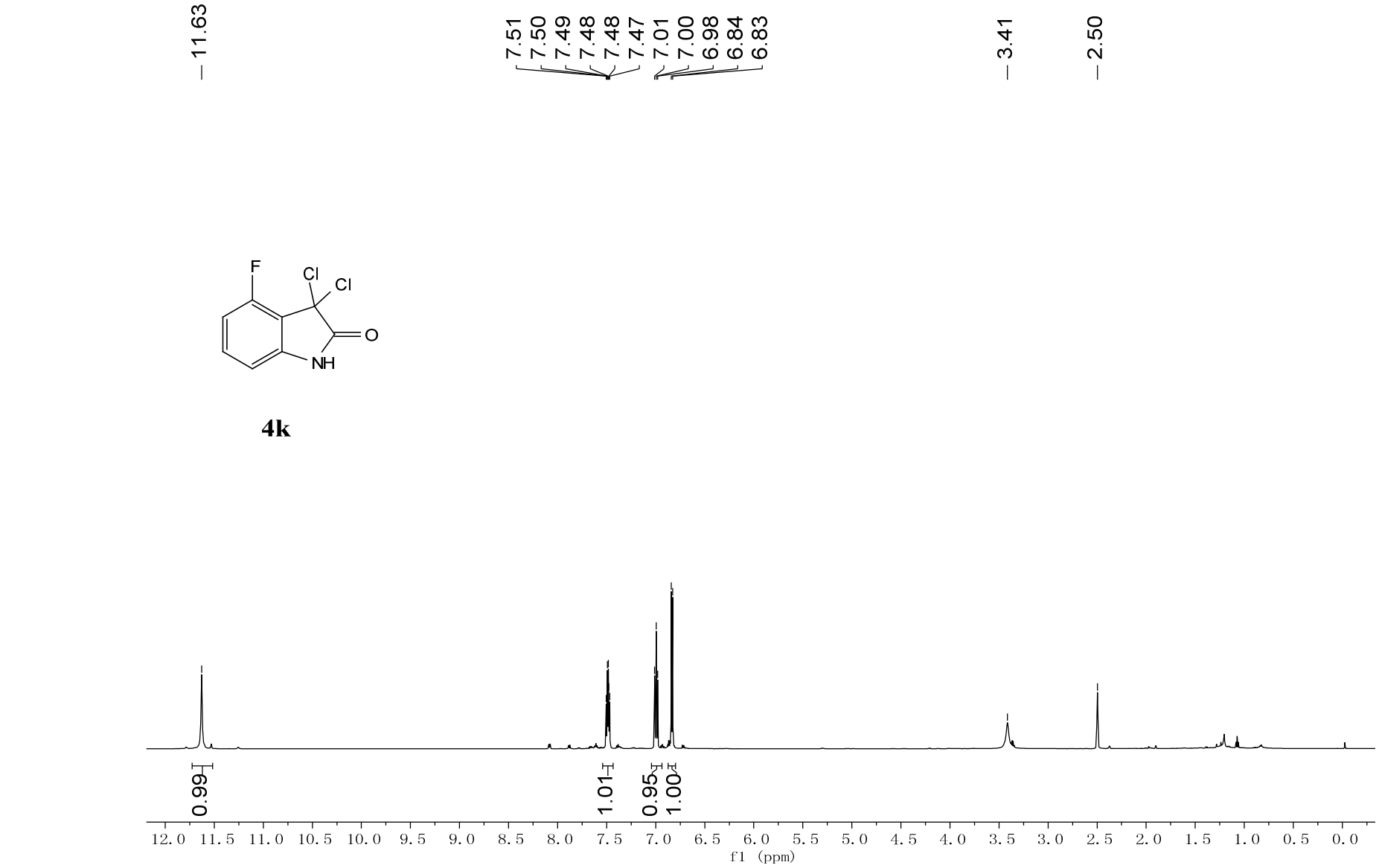

7.51
7.50
7.27
6.99
6.98
6.71
3.79
3.78
3.77
3.76
2.42
1.32
1.31
1.30
-0.00
Cl
Cl
O
N
H3C
CH3
4i
0.98
1.00
1.02
2.00
3.10
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)


168.77
142.72
139.80
126.58
124.68
124.48
109.91
77.21
77.00
76.79
74.46
35.56
22.02
12.30
Cl
Cl
O
N
H3C
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

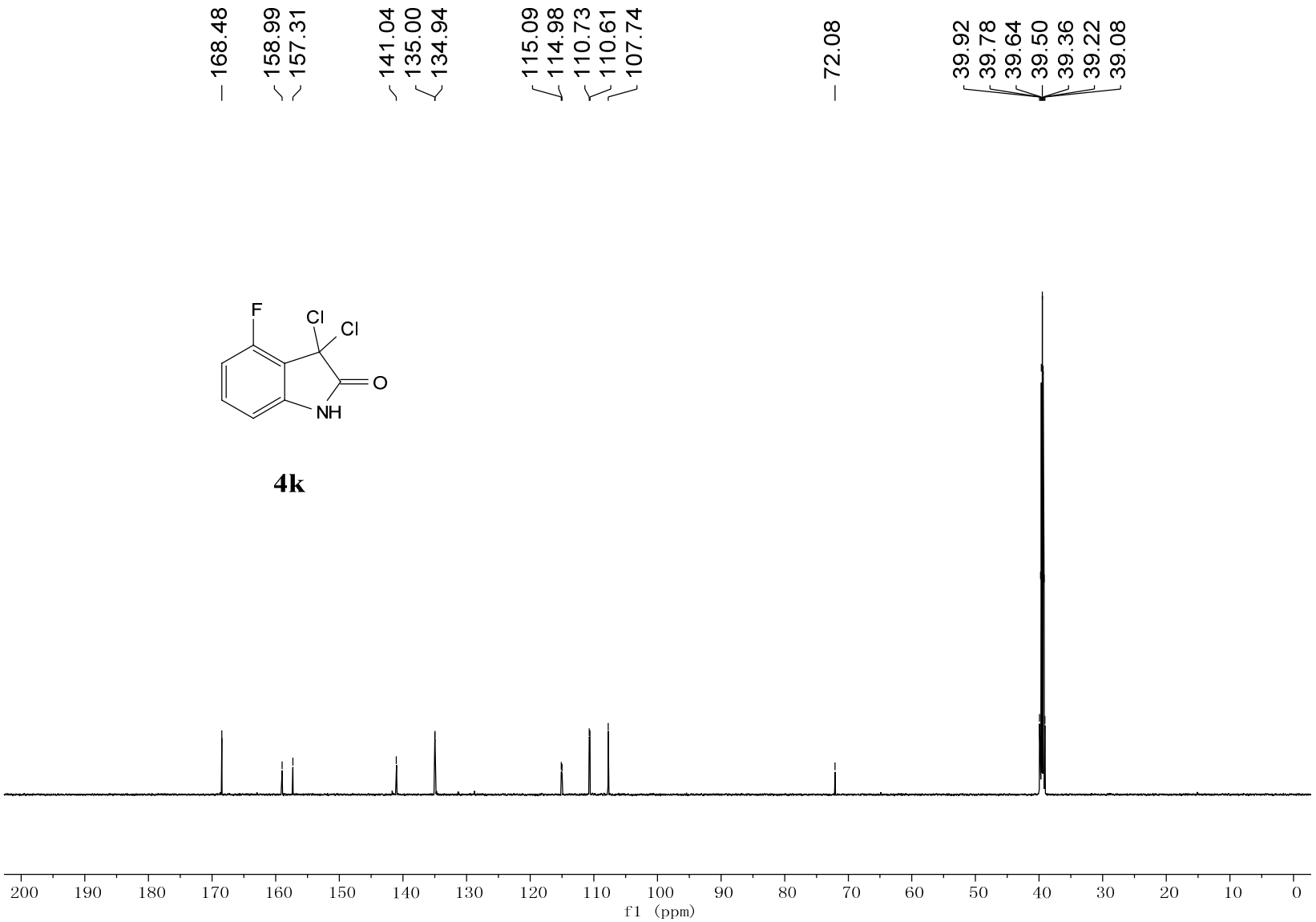
4i

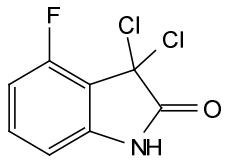

8.31
8.30
8.29
8.29
7.86
7.86
7.84
7.84
7.26
7.12
7.10
7.10
7.08
3.94
3.93
3.91
3.89
1.36
1.35
1.33
Cl
Cl
O
N
N
CH3
4j
1.00
1.01
1.02
2.15
3.23
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.08
153.32
150.52
132.40
124.21
119.48
77.32
77.00
76.68
73.00
35.11
12.69
Cl
Cl
O
N
N
CH3
4j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

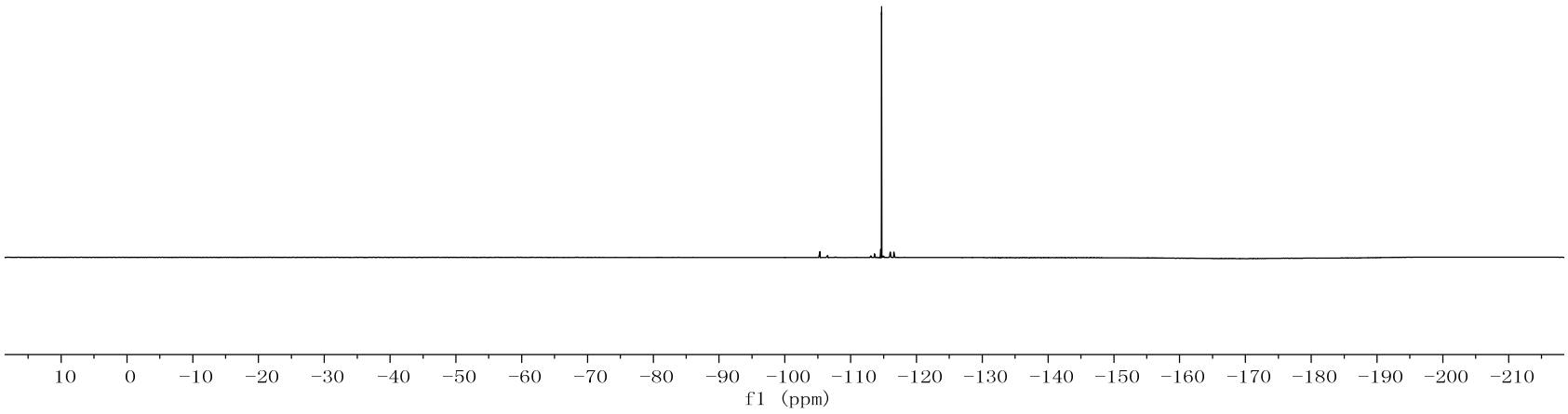

11.63
7.51
7.50
7.49
7.48
7.48
7.47
7.01
7.00
6.98
6.84
6.83
3.41
2.50
F
Cl
Cl
O
NH
0.99
1.01
0.95
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

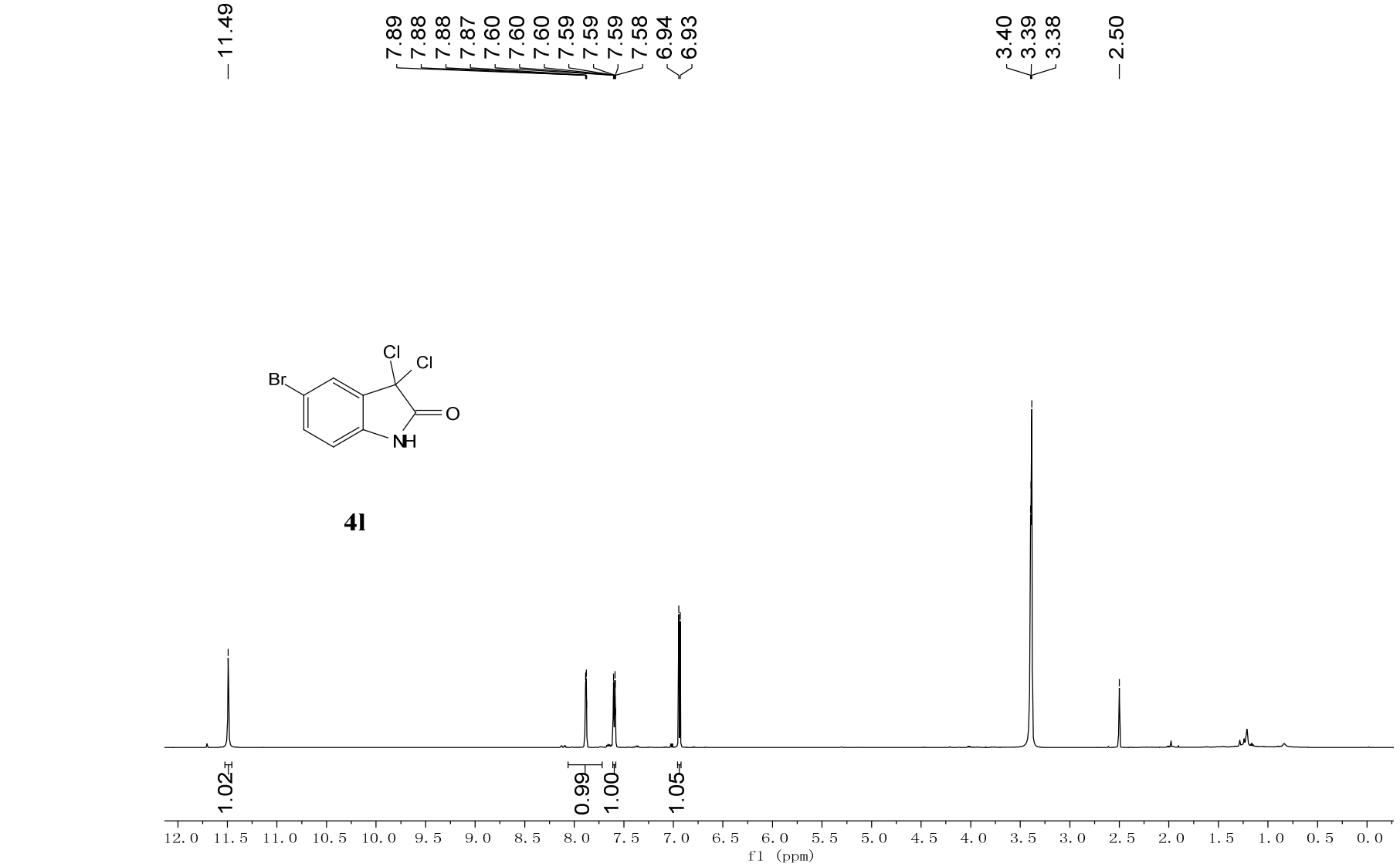
4k


168.48
158.99
157.31
141.04
135.00
134.94
115.09
114.98
110.73
110.61
107.74
72.08
39.92
39.78
39.64
39.50
39.36
39.22
39.08
F
Cl
Cl
O
NH
4k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

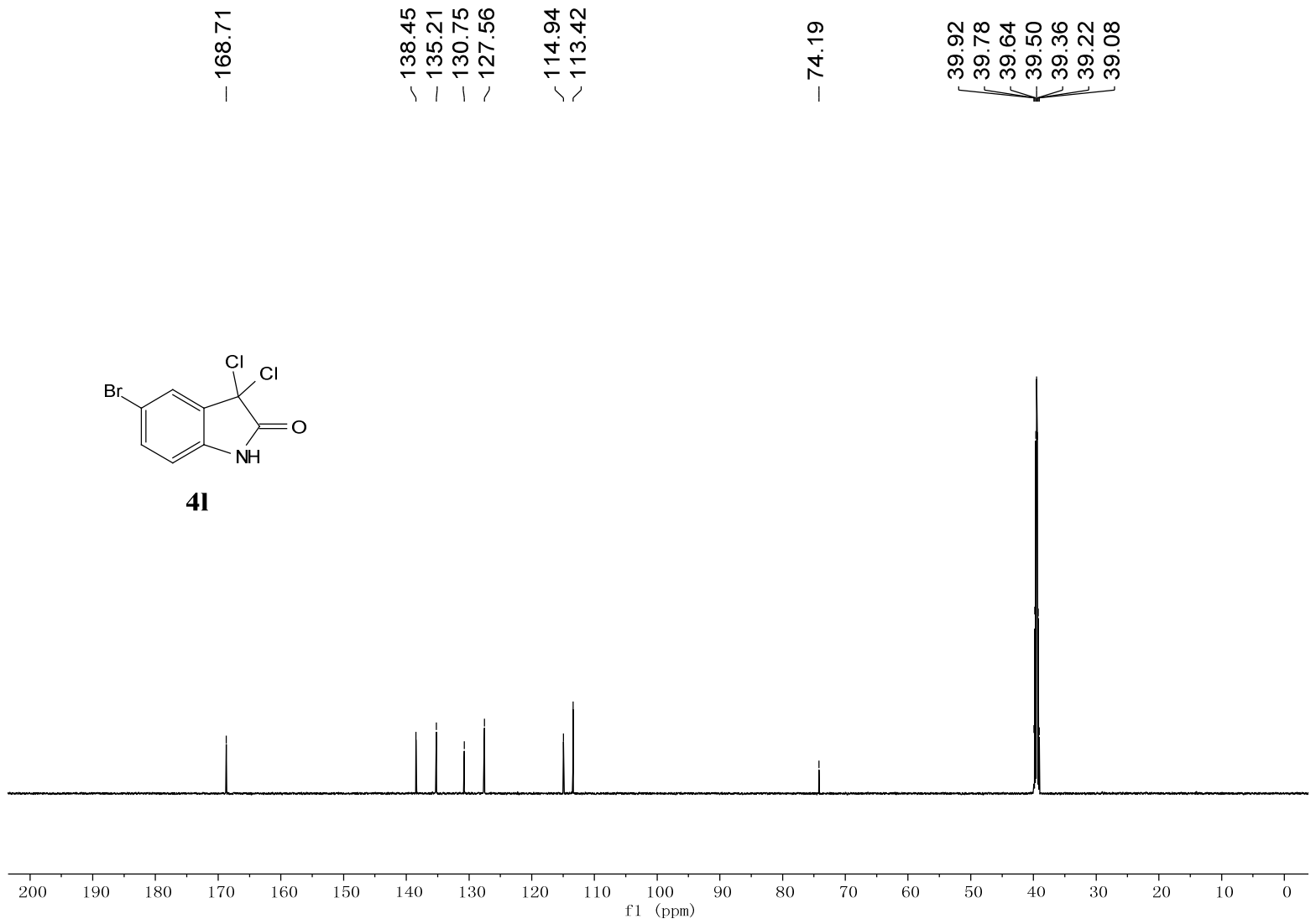

F
Cl
Cl
O
NH

4k

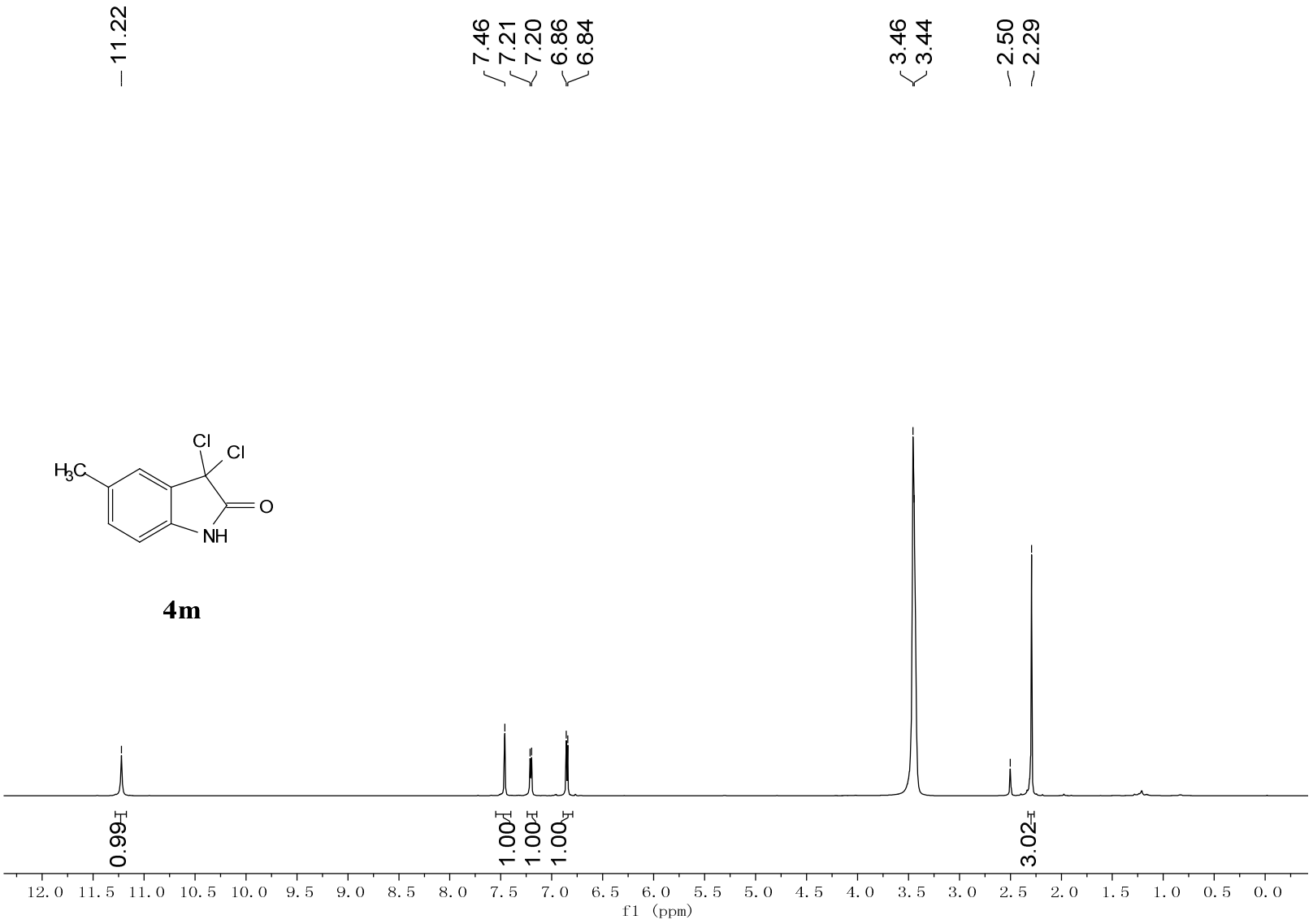

-114.71
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

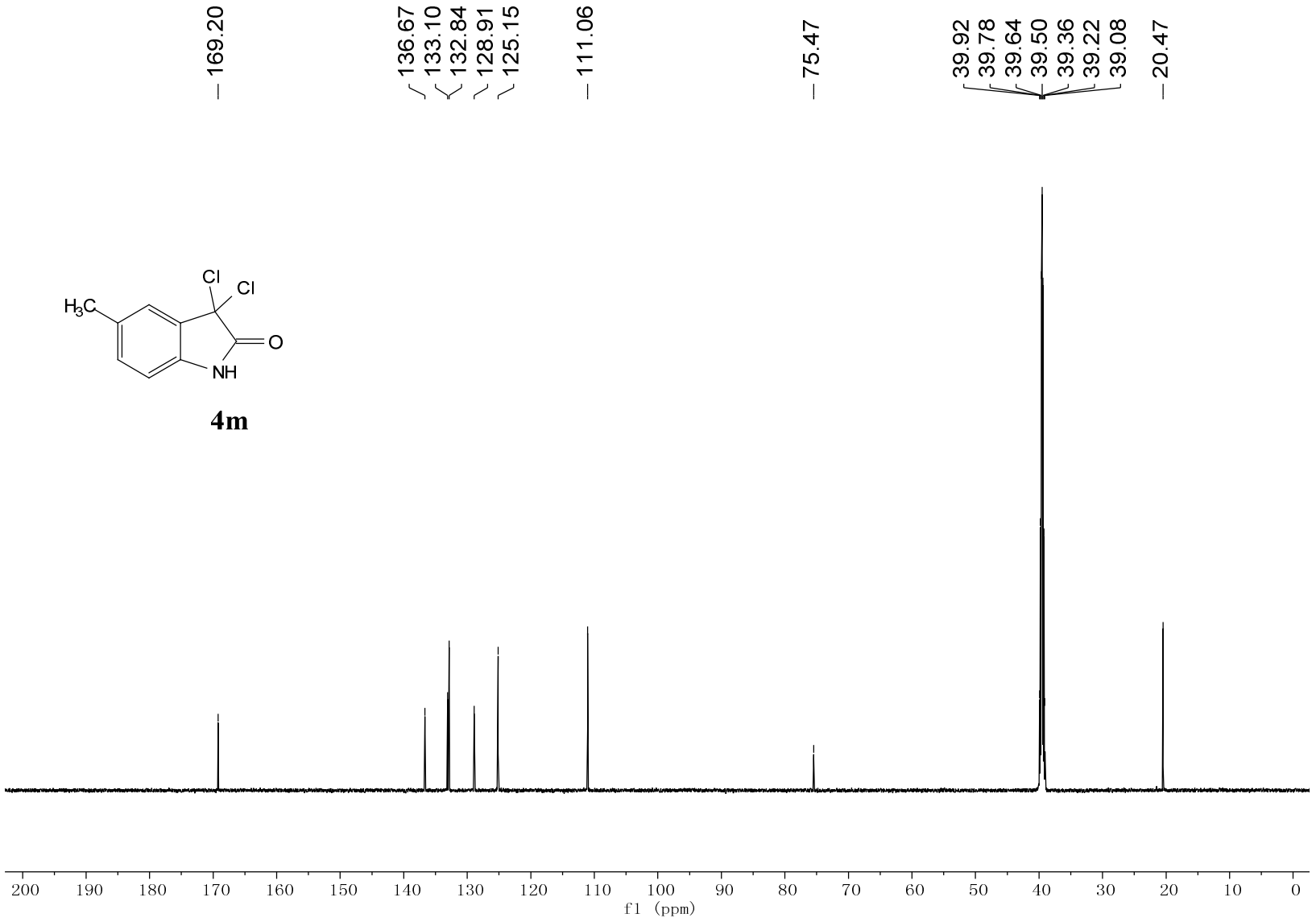

11.49
7.89
7.88
7.88
7.87
7.60
7.60
7.60
7.59
7.59
7.59
7.58
6.94
6.93
3.40
3.39
3.38
2.50
Cl
Cl
Br
O
NH
1.02
0.99
1.00
1.05
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

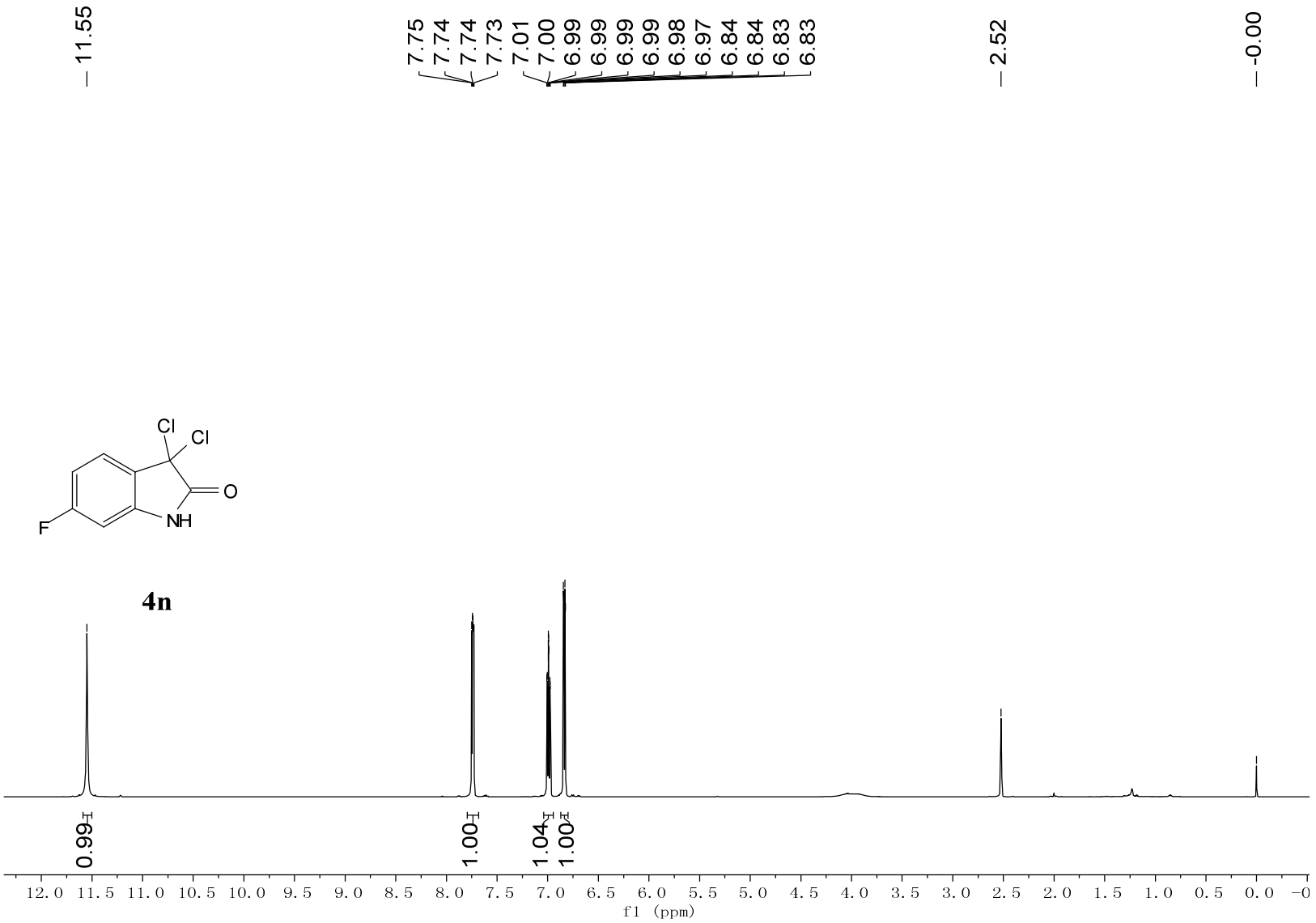
4l

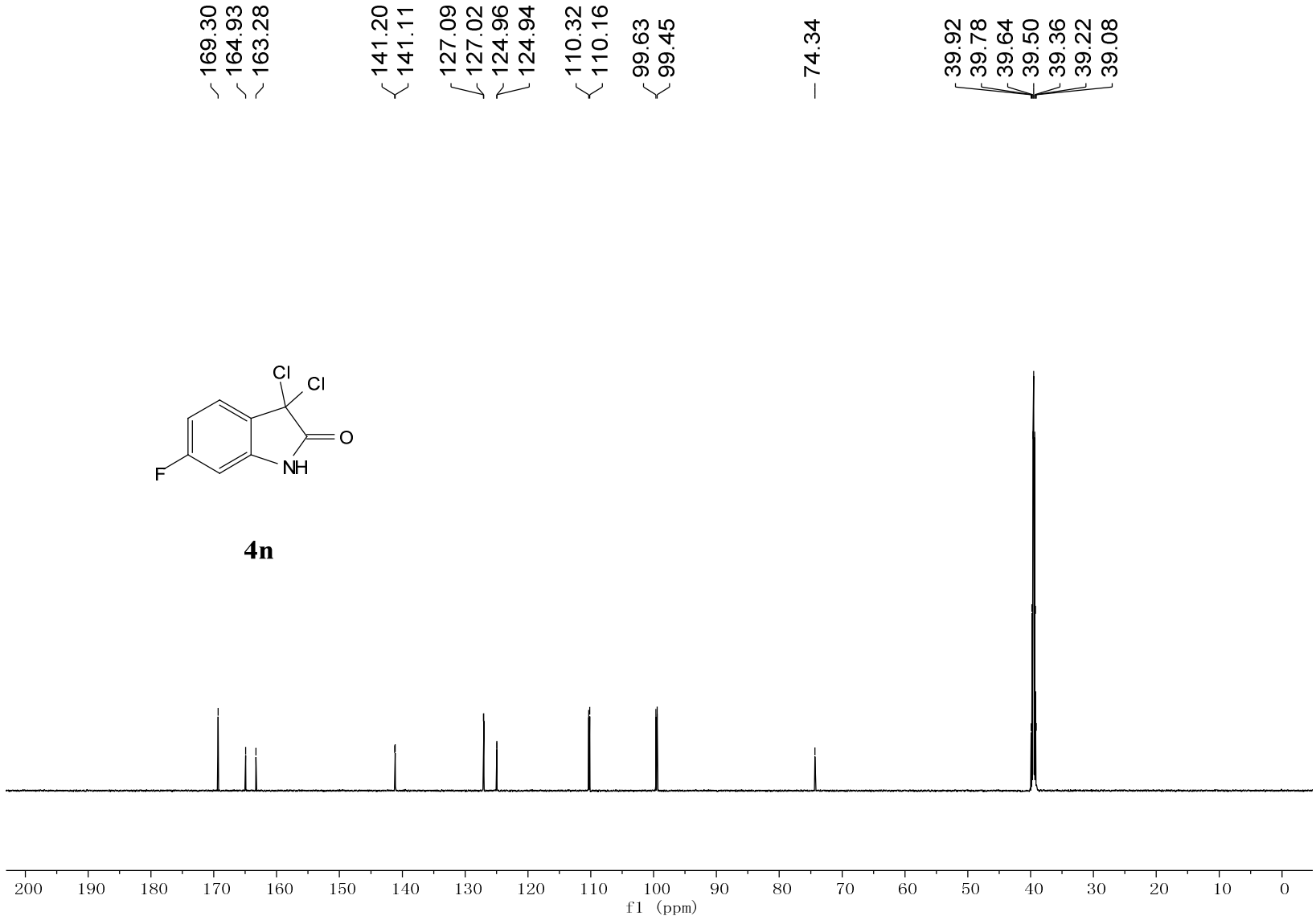

168.71
138.45
135.21
130.75
127.56
114.94
113.42
74.19
39.92
39.78
39.64
39.50
39.36
39.22
39.08
Cl
Cl
Br
O
NH
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4l

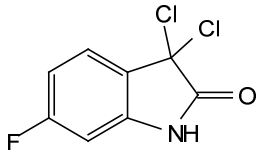

11.22
7.46
7.21
7.20
6.86
6.84
3.46
3.44
2.50
2.29
Cl
Cl
H3C
O
NH
4m
0.99
1.00
1.00
1.00
3.02
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


169.20
136.67
133.10
132.84
128.91
125.15
111.06
75.47
39.92
39.78
39.64
39.50
39.36
39.22
39.08
20.47
Cl
Cl
H3C
O
NH
4m
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


11.55
7.75
7.74
7.74
7.73
7.01
7.00
6.99
6.99
6.99
6.99
6.98
6.97
6.84
6.84
6.83
6.83
2.52
-0.00
Cl
Cl
O
NH
F
4n
0.99
1.00
1.04
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

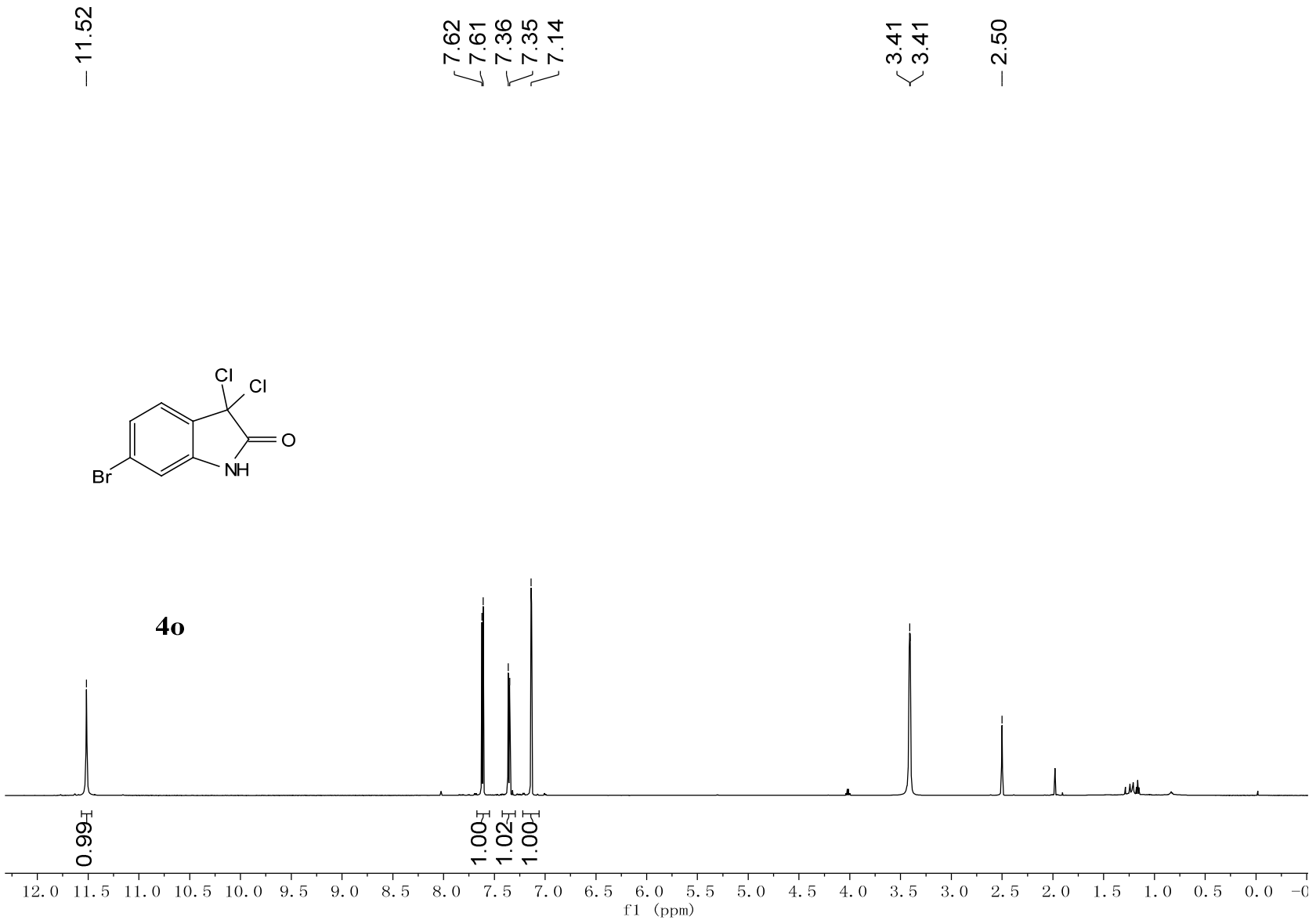

169.30
164.93
163.28
141.20
141.11
127.09
127.02
124.96
124.94
110.32
110.16
99.63
99.45
74.34
39.92
39.78
39.64
39.50
39.36
39.22
39.08
Cl
Cl
O
NH
F
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

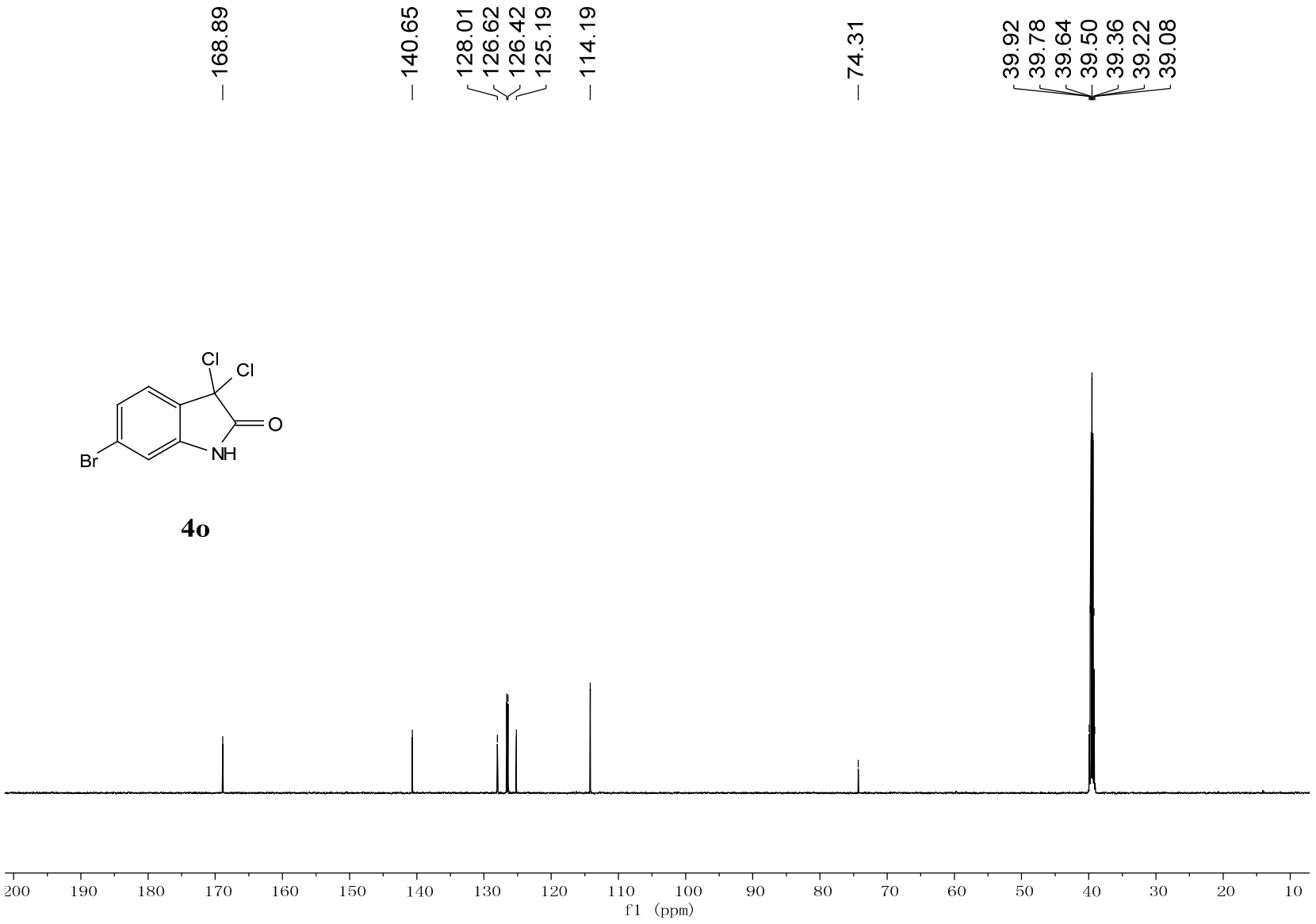
4n


Cl
Cl
O
NH
F

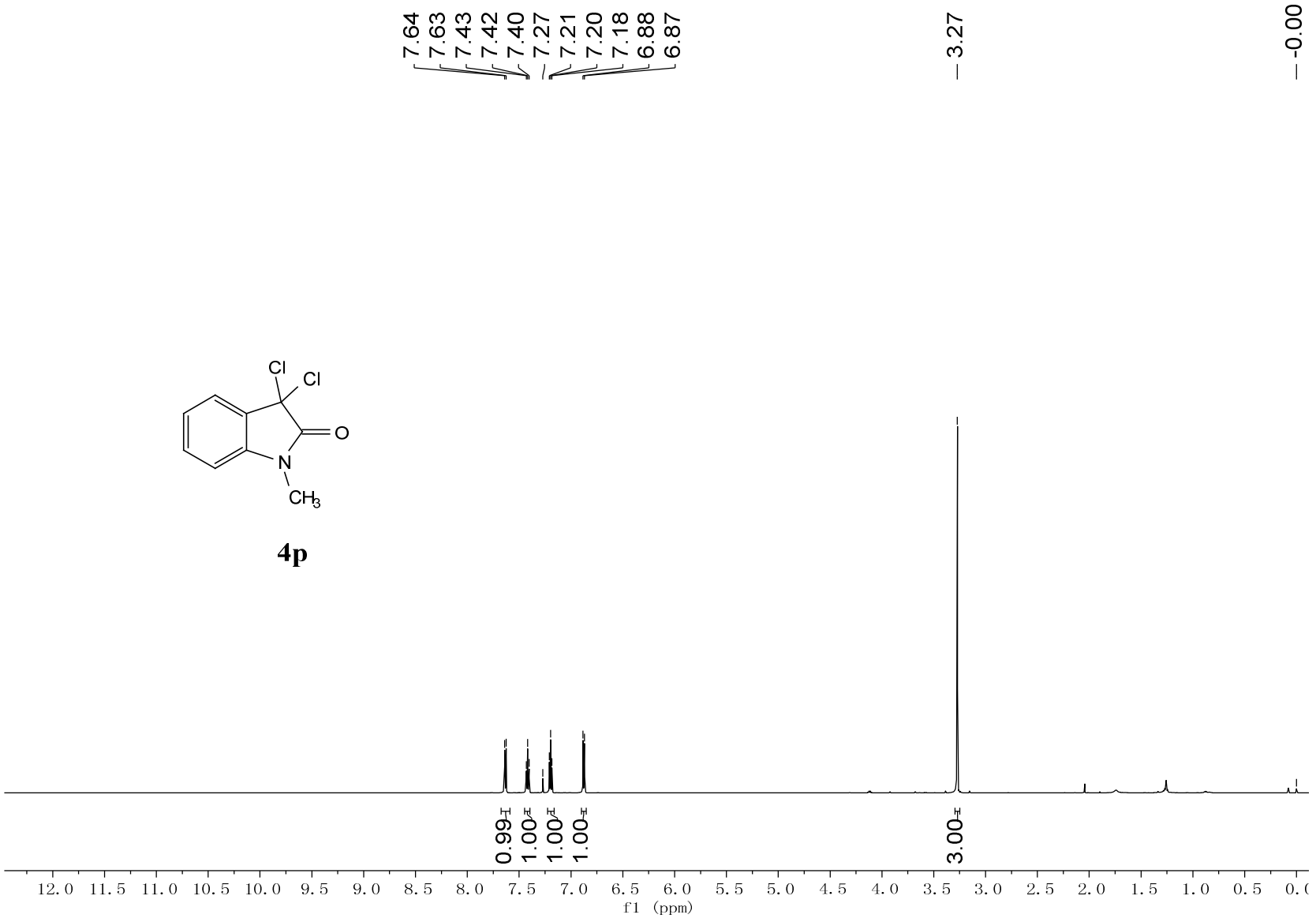
4n

-105.68
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)

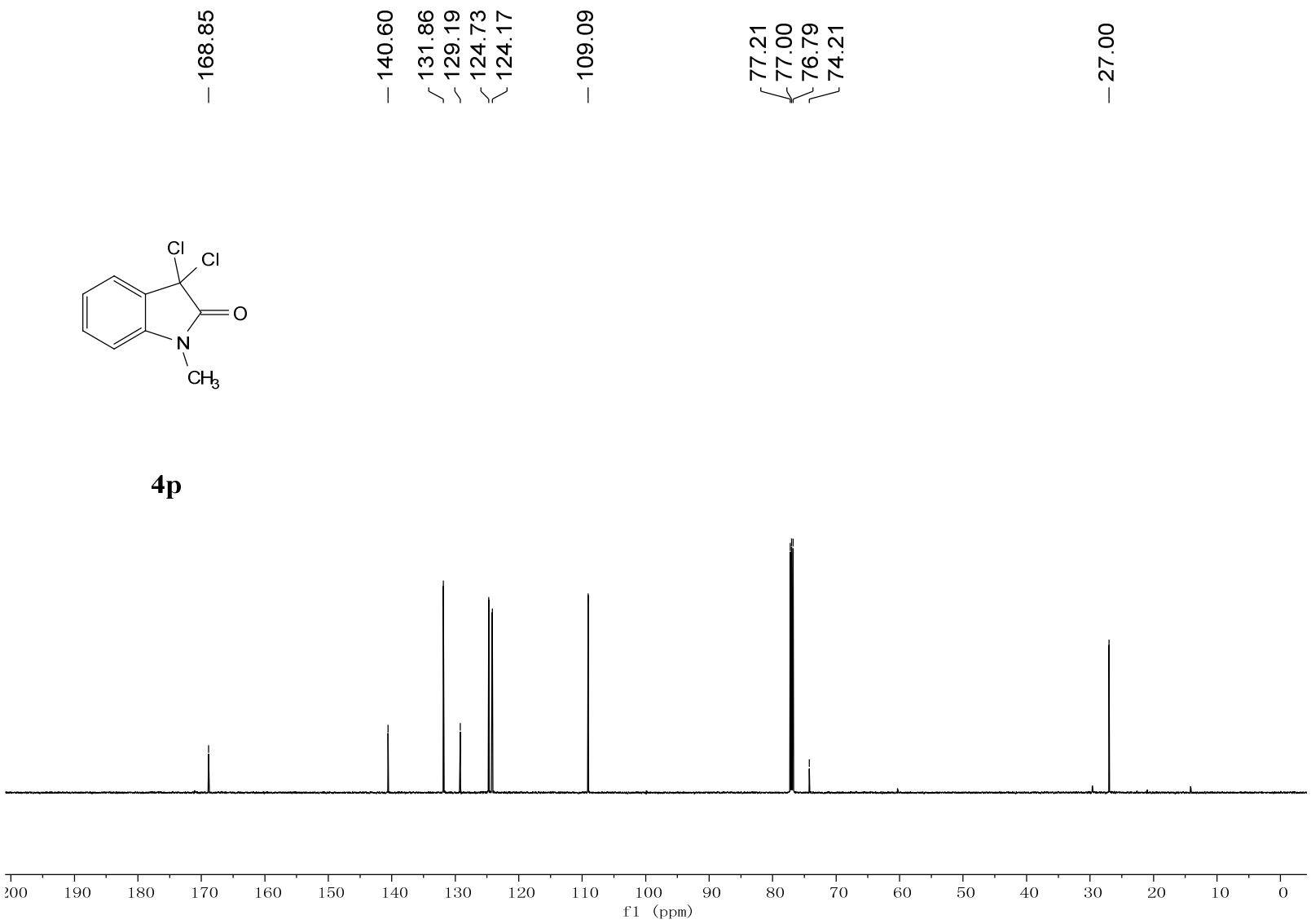

11.52
7.62
7.61
7.36
7.35
7.14
3.41
3.41
2.50
Cl
Cl
O
NH
Br
4o
0.99
1.00
1.02
1.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.89
140.65
128.01
126.62
126.42
125.19
114.19
74.31
39.92
39.78
39.64
39.50
39.36
39.22
39.08
Cl
Cl
O
NH
Br
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

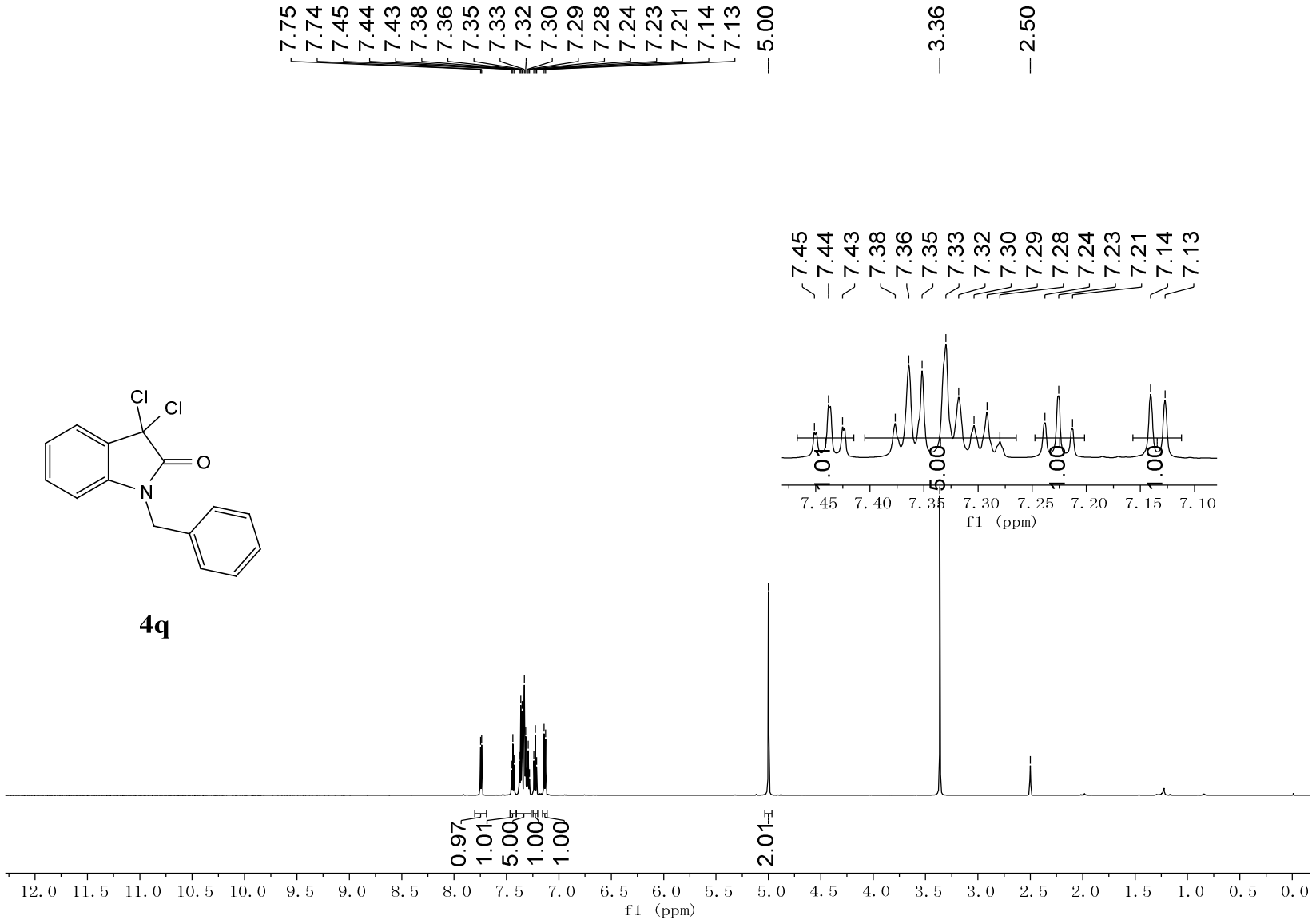
4o

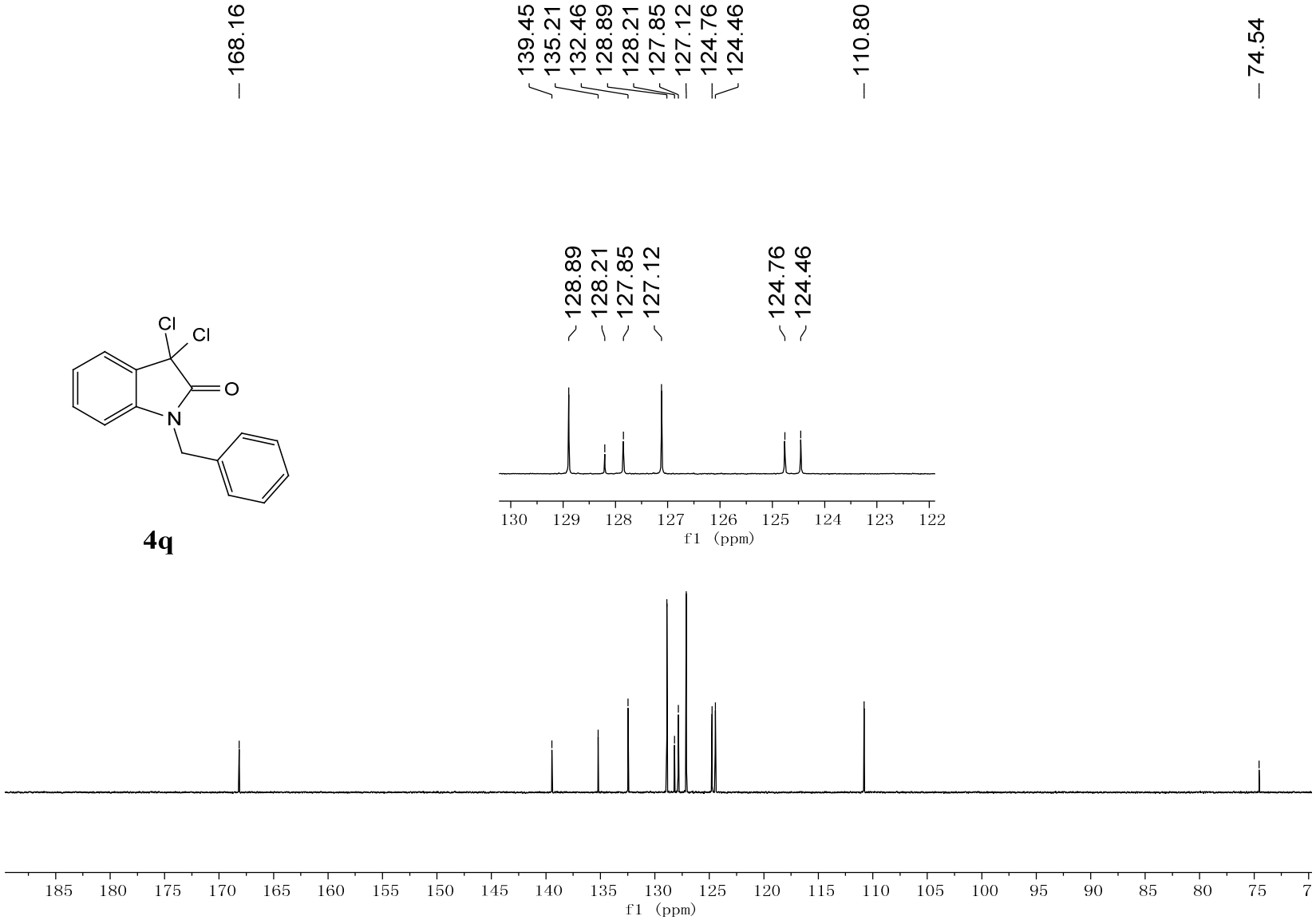

7.64
7.63
7.43
7.42
7.40
7.27
7.21
7.20
7.18
6.88
6.87
3.27
-0.00
Cl
Cl
O
N
CH3
0.99
1.00
1.00
1.00
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

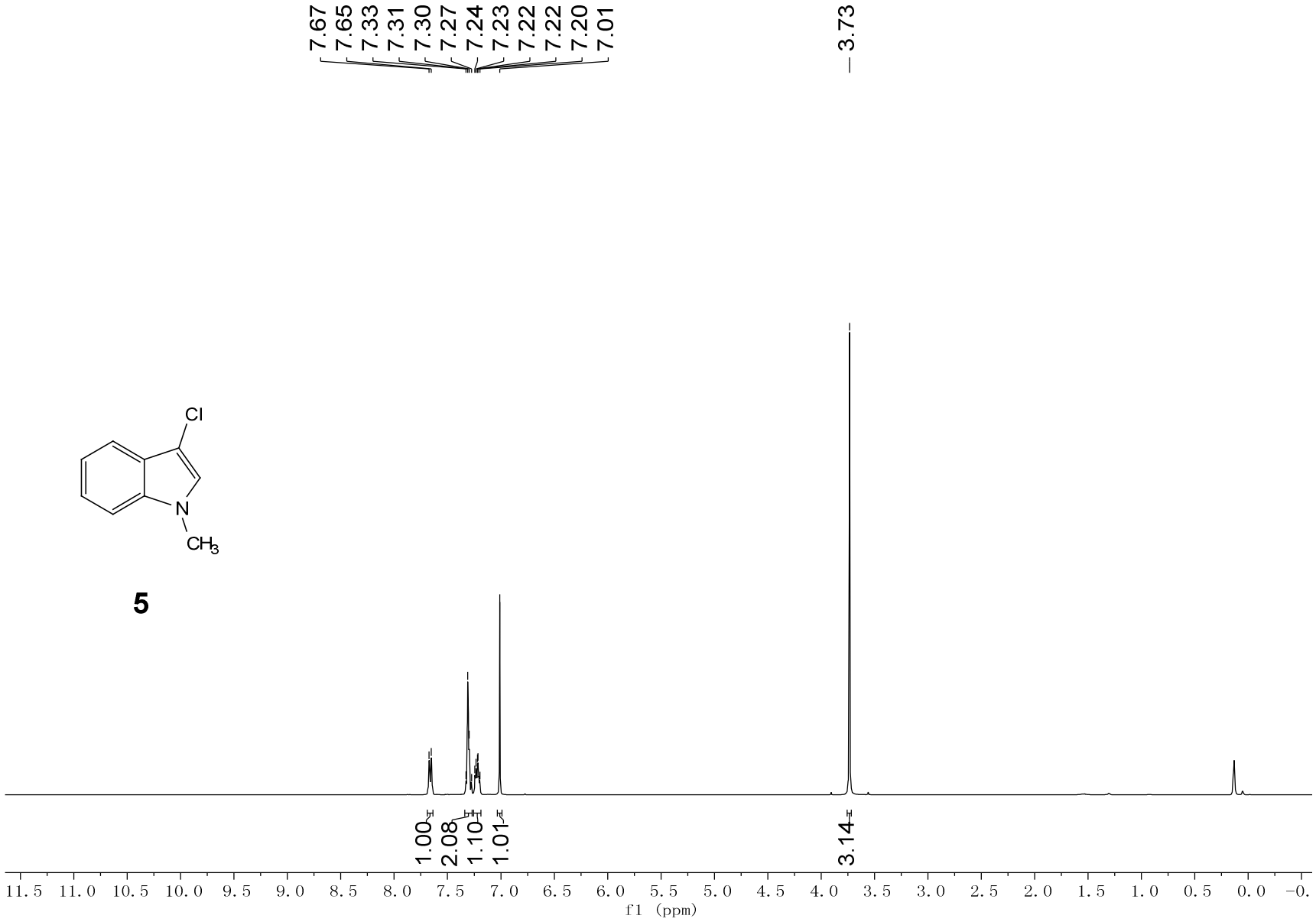
4p

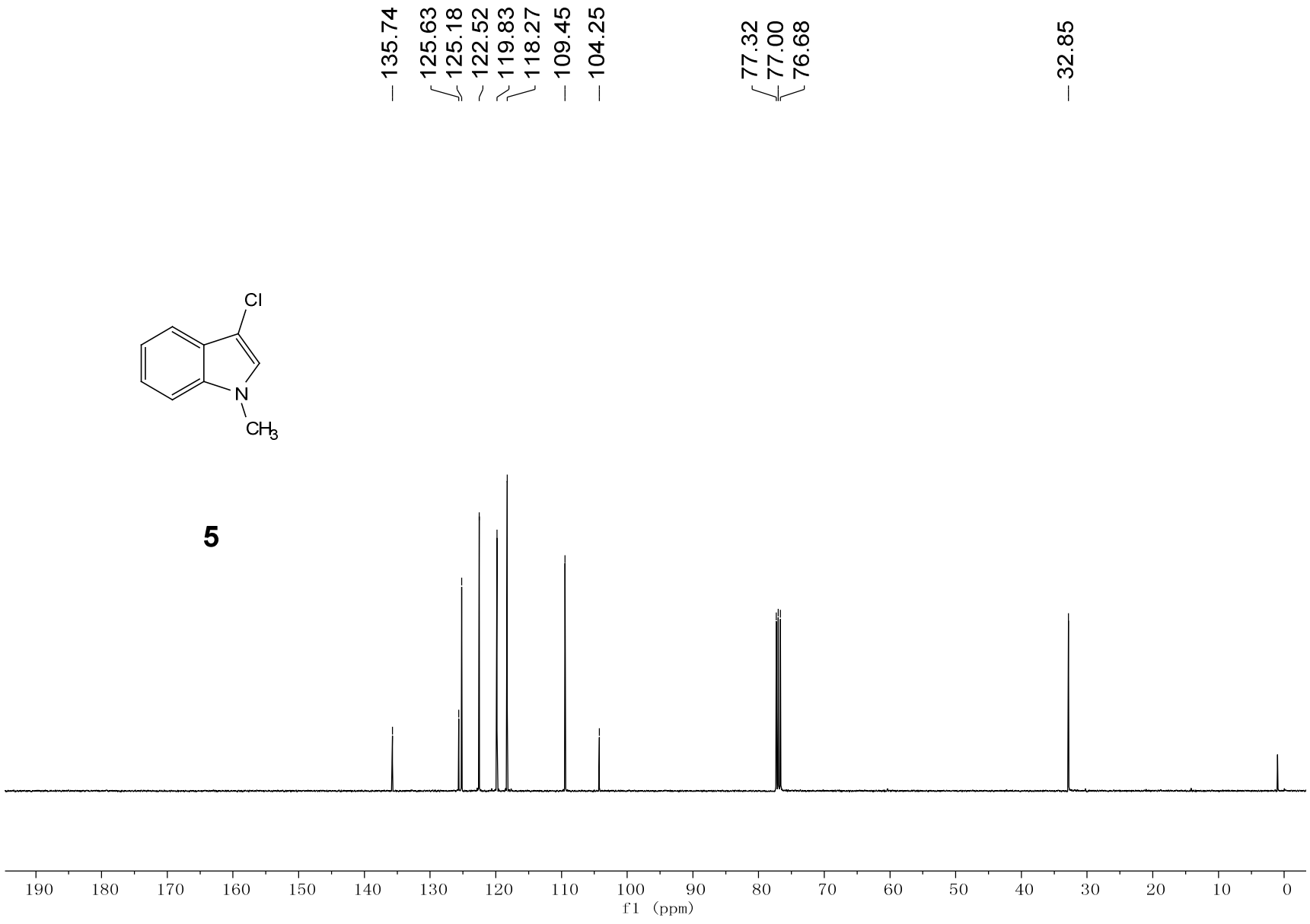

168.85
140.60
131.86
129.19
124.73
124.17
109.09
77.21
77.00
76.79
74.21
27.00
Cl
Cl
O
N
CH3
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

4p


7.75
7.74
7.45
7.44
7.43
7.38
7.36
7.35
7.33
7.32
7.30
7.29
7.28
7.24
7.23
7.21
7.14
7.13
5.00
3.36
2.50
Cl
Cl
O
N
4q
0.97
1.01
5.00
1.00
1.00
2.01
f1 (ppm)


168.16
139.45
135.21
132.46
128.89
128.21
127.85
127.12
124.76
124.46
110.80
74.54
Cl
Cl
O
N
4q
128.89
128.21
127.85
127.12
124.76
124.46
130 129 128 127 126 125 124 123 122
f1 (ppm)
185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 7
f1 (ppm)


7.67
7.65
7.33
7.31
7.30
7.27
7.24
7.23
7.22
7.22
7.20
7.01
3.73
Cl
N
CH3
5
1.00
2.08
1.10
1.01
3.14
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)


135.74
125.63
125.18
122.52
119.83
118.27
109.45
104.25
77.32
77.00
76.68
32.85
Cl
N
CH3
5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)