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Supporting Information

for

A Copper(II)-Catalyzed Annulative Formylation of *o*-Alkynylanilines with DMF: Single-Step Strategy for 3-Formyl Indoles

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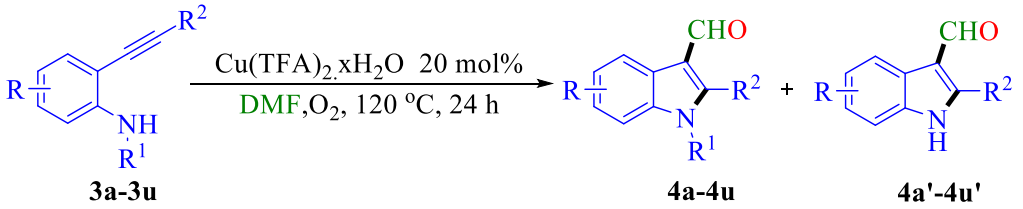
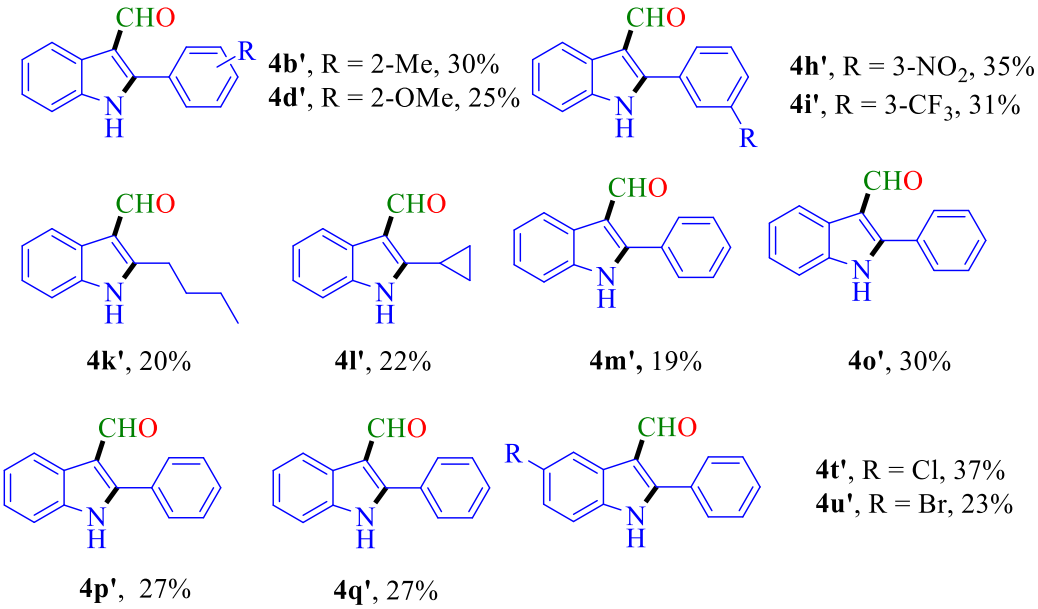
I. General information

All reagents and solvents were purchased from commercial providers (Matrix scientific, Sigma Aldrich, TCI, and Alfa Aesar) and were used without further purification. Cleaned and oven dried glassware were used for reaction. ^{1}H, and ^{13}C NMR spectra were recorded on a 400 MHz Varian Mercury spectrometer or 400 MHz JEOL NMR spectrometer. The spectra were recorded in $CDCl_3$ or DMSO-d_6 as solvent. ^{1}H NMR spectra are reported chemical shifts (δ) in parts per million (ppm) with 7.26 ppm ($CDCl_3$) or 2.50 (DMSO-d_6) solvent peak. ^{13}C NMR spectra are reported chemical shifts (δ) in parts per million (ppm) with 77.23 ppm ($CDCl_3$) or 39.51 ppm (DMSO-d_6) solvent peak. Multiplicities are represented by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), dd (doublet of doublet). Coupling constants (*J*) are reported in Hz. In Academia Sinica, High Resolution Mass Spectrometer (HRMS) (FAB & APCI mode) mass spectra was conducted on a JMS-700 double focusing mass spectrometer. Melting points were determined on an fargo instruments. Reaction was monitored by analytical thin layer chromatography (TLC) which is purchased from Merck, is precoated with silica gel 60 F254. Column chromatography was performed with silica gel 230-400 mesh (purchased from Merck) with mixture of ethyl acetate/hexane or hexane as the eluent.

II. Table S1. *N*-debenzylated product of scheme 4

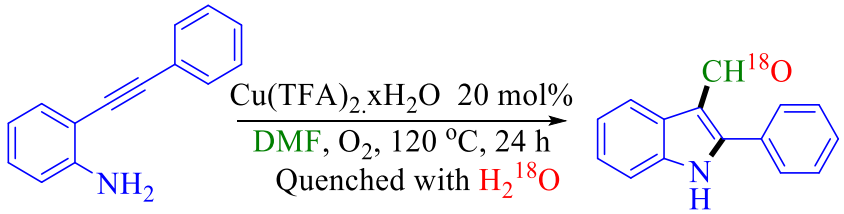




When exploring the scope of *N*-benzylated-2-alkynylaniline derivatives (**Scheme 3**), the debenzylated product was observed along with desired product. The debenzylated product formation, may be due to the presence of oxygen at high temperature [1,2]. The scope of *N*-debenzylated products were showed in **Table S1.**

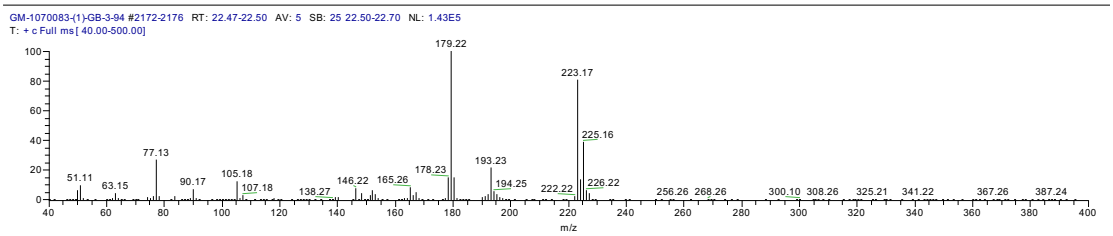
III. Control experiments

1. ^{18}O labelled water experiment



o-phenylethynylaniline (0.3 mmol) in 3 mL of DMF taken in sealed tube and then followed by 20 mol% of $Cu(TFA)_2.xH_2O$ added into the reaction vessel. Then allowed

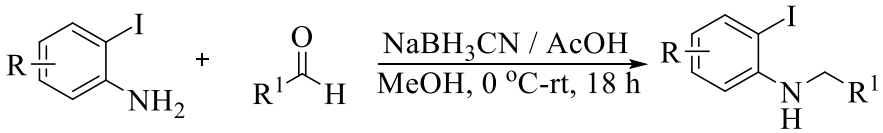
it to stir for 24 h at 120 ℃ under oxygen atmosphere. Upon the consumption of starting material, which is monitored by TLC. The reaction mixture is quenched with $H_2{}^{18}O$ and the formation of ^{18}O labelled aldehyde (m/z: 223) is confirmed by GCMS. The results revealed that the oxygen is coming from water for aldehyde formation.



IV. General procedures for synthesis of *o*-alkynylanilines

All starting materials were synthesized according to the literature [3] and their ^1H NMR spectra were matched with the assigned structures.

Scheme S1: Synthesis of *N*-benzyl-2-iodoaniline derivatives



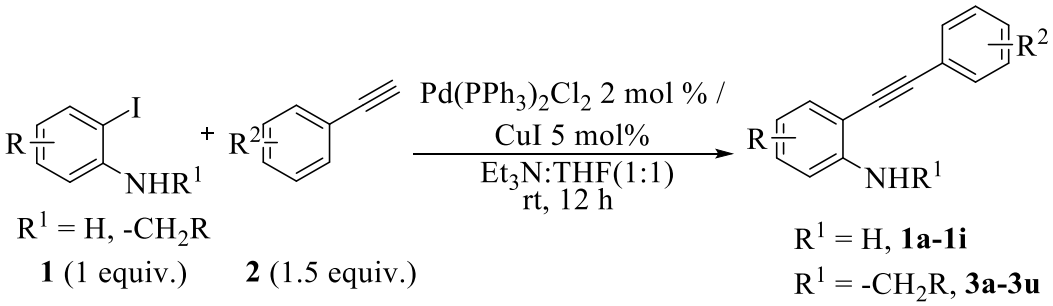
1 (1 equiv.) 2.4 equiv.

Scheme S1

Aniline (1 equiv.) in 5 mL of methanol as a solvent taken in two necked round bottomed flask. Then dropwise addition of acetic acid (4 equiv.) at 0 ℃ to the reaction mixture. $NaBH_3CN$ (2 equiv.) was added and stir for few minutes. Then addition of aldehyde (2.4 equiv.) to the reaction mixture and allowed to stir for 18 h at room temperature under N_2. The consumption of starting materials were checked by TLC and then quenched with ice cold water. The excess amount of MeOH were removed by vacuum. Followed by extraction with ethyl acetate and water. The combined organic layers washed with brine, dried over $MgSO_4$ and concentrated by vacuum. Purified by

column chromatography with 2 % of ethyl acetate in hexane as eluent to afford the desire product.

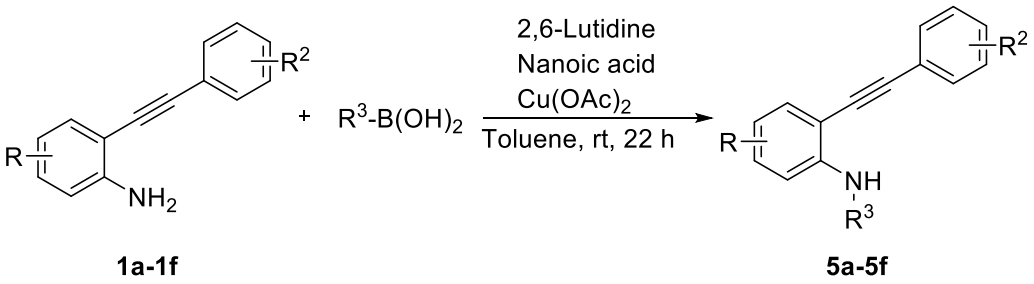
Scheme S2: Procedure for Synthesis of *o*-alkynylaniline derivatives (1a-1i and 3a-3u)



Scheme S2

Two necked round bottom flask was charged with 1 equivalence of 2-iodoaniline or *N*-benzyl-2-iodoaniline in 1:1 equal amount of Et_3N (0.25 M) and THF (0.25 M). To the solution, alkyne (2 equiv.), and $Pd(PPh_3)_2Cl_2$ (2 mol%) were added and stir for 5 minutes. Then 5 mol% of CuI was added and allowed to stir for 8-12 h, upon the fully consumption of starting materials by TLC. The excess amount of Et_3N and THF were removed by vacuum. The crude was purified by column chromatography with 1-2 % ethyl acetate in hexane as eluent to afford the desire product **1a-1i** and **3a-3u**.

Scheme S3: Procedure for Synthesis of *o*-alkynylaniline derivatives (5a-5f)

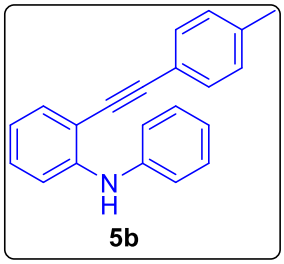


Scheme S3

The starting materials (**5a-5f**) were prepared accordingly the reported literature by Senadi, G. C., et al. In toluene (0.25 M), *o*-(phenylethynyl)aniline derivatives was taken and followed by addition of phenylboronic acid derivatives (1.5 equiv.), 2,6-lutidine (1.1 equiv.), nonanoic acid (0.2 equiv.), and $Cu(OAc)_2$ (0.1 equiv.). Then the resulting solution was allowed to stir (22~ 30 h) in room temperature and the reaction condition was monitored by TLC. Once starting materials were consumed, then the reaction mixture was quenched with water and extracted with ethyl acetate. Finally, the combined organic layer was washed with brine, dried over $MgSO_4$, and concentrated in vacuum. The crude was purified by column chromatography using hexane as eluent to afford the product (**5a-5f**) with 70-80 % yield.

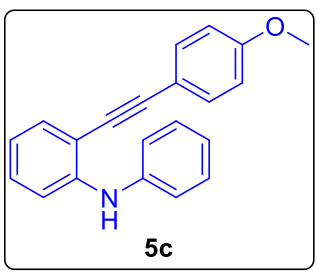
V. Characterization data of *o*-alkynylaniline derivatives

***N*-phenyl-2-(*p*-tolylethynyl)aniline (5b):** The title compound was prepared according to the general procedure (**Scheme S3**) and obtain the desired compound as a yellow liquid in 62 % yield; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.47-7.41 (m, 3H), 7.34-7.31 (m, 1H), 7.27-7.15 (m, 6H), 7.05-7.01 (m, 1H), 6.83-6.79 (m, 1H), 6.53 (s, 1H), 2.37 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 144.9, 141.9, 138.8, 132.7, 131.6, 129.6, 129.5, 129.4, 122.7, 120.5, 120.1, 119.4, 113.6, 110.6, 96.0, 85.2, 21.7; HRMS (m/z, APCI): calcd for $[C_{21}H_{18}N]^+$ $[M+H]^+$ 284.1439, observed 284.1441.



5b

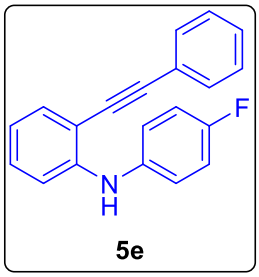
2-((4-methoxyphenyl)ethynyl)-*N*-phenylaniline (5c): The title compound was prepared according to the general procedure (**Scheme S3**) and obtain the desired compound as a yellow liquid in 64 % yield; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.47-7.43 (m, 2H), 7.34-7.29 (m, 2H), 7.26-7.17 (m, 4H), 7.07-7.02 (m, 1H),



5c

6.89-6.86 (m, 2H), 6.82-6.78 (m, 1H), 6.50 (s, 1H), 3.82 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 160.0, 144.8, 142.0, 133.2, 132. 7, 129.6, 129.4, 122.6, 120.4, 119.4, 115.3, 114.3, 113.6, 110.7, 95.8, 84.4, 55.5; HRMS (m/z, FAB$^+$): calcd for $[C_{21}H_{17}NO]^+$ $[M]^+$ 299.1310, observed 299.1309.

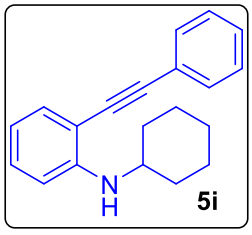
***N*-(4-fluorophenyl)-2-(phenylethynyl)aniline (5e):** The title compound was prepared



5e

according to the general procedure (**Scheme S3**) and obtain the desired compound as a dark brown liquid in 45 % yield; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.55-7.53 (m, 2H), 7.47 (dd, J = 7.6, 1.6 Hz, 1H), 7.38-7.35 (m, 2H), 7.21-7.16 (m, 3H), 7.07-7.02 (m, 3H), 6.82-6.79 (m, 1H), 6.39 (s, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 145.8, 137.7, 132.8, 131.7, 129.8, 128.7, 123.6, 123.5, 123.2, 119.1, 116.4, 116.2, 112.7, 109.7, 95.8, 85.8; HRMS (m/z, FAB$^+$): calcd for $[C_{20}H_{14}FN]^+$ $[M]^+$ 287.1110, observed 287.1112.

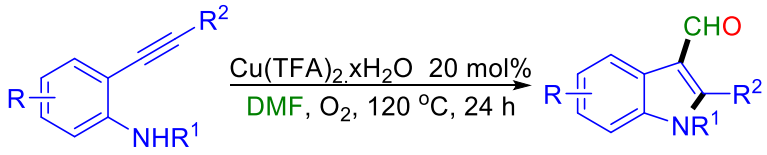
***N*-cyclohexyl-2-(phenylethynyl)aniline (5i)**: The title compound was prepared



5i

according to the general procedure (**Scheme S3**) and obtain the desired compound as a yellow liquid in 52 % yield; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.52-7.49 (m, 2H), 7.38-7.32 (m, 4H), 7.21-7.16 (m, 1H), 6.62 (q, J = 8 Hz, 2H), 4.65 (s, 1H), 3.39-3.34 (m, 1H), 2.10-2.06 (m, 2H), 1.81-1.76 (m, 2H), 1.47-1.24 (m, 6H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 148.3, 132.4, 131.6, 130.1, 128.6, 128.3, 123.7, 116.0, 110.3, 107.6, 95.3, 86.4, 51.4, 33.4, 26.1, 25.0; HRMS (m/z, FAB$^+$): calcd for $[C_{20}H_{21}N]^+$ $[M]^+$ 275.1674, observed 275.1672.

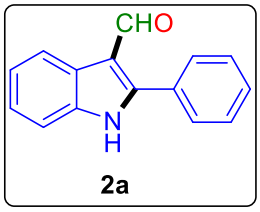
VI. General procedures and characterization data for of 3-formyl indole



1a-1i, 5a-5h, and 3a-3u **2a-2i, 6a-6h , and 4a-4u**

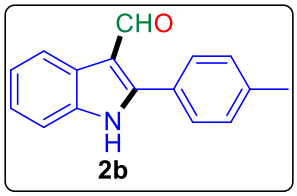
In sealed tube, *o*-alkynylaniline derivatives (0.3 mmol) was taken in 3 mL of DMF. To the stirred solution, $Cu(TFA)_2.xH_2O$ (20 mol%) was added and allowed to stir under O_2 source at 120 °C, until the completion of starting material (~ 24 h). The reaction mixture was quenched with ice cold water and extracted with ethyl acetate. Combined organic layer washed with brine, dried over $MgSO_4$ and concentrated in vacuum. Then the crude material was purified by column chromatography using 20 % EA in hexane as eluent afford to the final product.

2-phenyl-1*H*-indole-3-carbaldehyde (2a):[4] The title compound was prepared according to the general procedure and obtain the desired compound as a yellow solid (34 mg, 52 % yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 11.19 (s, 1H), 9.98 (s, 1H), 8.32-8.30 (m, 1H), 7.63-7.61 (m, 2H), 7.49-7.40 (m, 4H), 7.23-7.20 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 186.8, 149.6, 136.2, 130.5, 130.0, 129.7, 128.9, 126.2, 123.9, 122.8, 122.0, 114.5, 111.7.



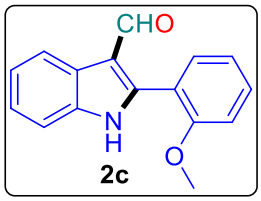
2a

2-(*p*-tolyl)-1*H*-indole-3-carbaldehyde (2b):[4] The title compound was prepared according to the general procedure and obtain the desired compound as a yellow solid (34 mg, 36% yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 11.26 (s, 1H), 10.07 (s, 1H), 8.40-8.37 (m, 1H), 7.60 (d, *J* = 7.4 Hz, 2H), 7.50-7.48 (m, 1H), 7.36 (d, *J* = 7.4 Hz, 2H), 7.31-7.27 (m, 2H), 2.47 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 186.8, 149.9, 140.0, 136.1, 129.8, 129.6, 127.5, 126.3, 123.8, 122.7, 121.9, 114.3, 111.7, 21.4.



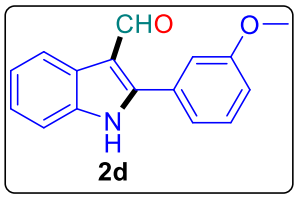
2b

2-(2-methoxyphenyl)-1*H*-indole-3-carbaldehyde (2c): The title compound was prepared according to the general procedure and obtain the desired compound as a yellowish oil (28 mg, 37 % yield); M.P. 125 ºC ~ 127 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.99 (s, 1H), 9.38 (s, 1H), 8.44-8.41 (m, 1H), 7.53-7.42 (m, 3H), 7.32-7.27 (m, 2H), 7.12-7.05 (m, 2H), 3.88 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.2, 157.2, 145.9, 135.4, 133.4, 131.5, 125.7, 124.1, 122.9, 122.1, 121.1, 118.3, 115.3, 116.6, 111.1, 55.9; HRMS (m/z, APCI): calcd for $[C_{16}H_{14}NO_2]^+$ $[M+H]^+$ 252.1025, observed 252.1030.



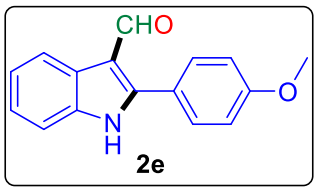
2c

2-(3-methoxyphenyl)-1*H*-indole-3-carbaldehyde (2d):[4] The title compound was prepared according to the general procedure and obtain the desired compound as a brown solid (37 mg, 49% yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 11.30 (s, 1H), 10.00 (s, 1H), 8.28 (t, *J* = 4.8 Hz, 1H), 7.39-7.33 (m, 2H), 7.20-7.13 (m, 3H), 6.97-6.95 (m, 2H), 3.79 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 186.7, 159.8, 149.4, 136.1, 131.7, 130.7, 129.9, 128.3, 126.2, 123.9, 122.7, 122.4, 121.9, 115.4, 114.5, 111.7, 55.5.



2d

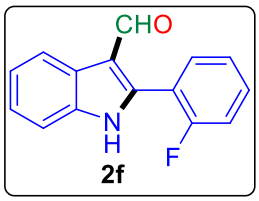
2-(4-methoxyphenyl)-1*H*-indole-3-carbaldehyde (2e):[4] The title compound was



2e

prepared according to the general procedure and obtain the desired compound as a brown solid (30 mg, 40% yield); M.P. 195 ºC ~ 197 ºC; Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 8.65 (s, 1H), 8.42-8.40 (m, 1H), 7.58 (d, *J* = 9.0 Hz, 2H), 7.43-7.40 (m, 1H), 7.33-7.29 (m, 2H), 7.06 (d, *J* = 9.0 Hz, 2H), 3.90 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.0, 161.4, 149.3, 135.5, 131.1, 126.5, 124.4, 123.4, 122.4, 121.5, 114.9, 114.8, 111.1, 55.7.

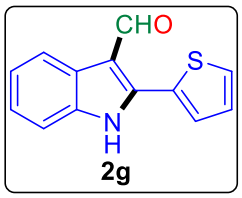
2-(2-fluorophenyl)-1*H*-indole-3-carbaldehyde (**2f**): The title compound was



2f

prepared according to the general procedure and obtain the desired compound as a yellow solid (15 mg, 20 % yield); M.P. 192 ºC ~ 194 ºC; 1H-NMR (400 MHz, $CDCl_3$ 4 drops DMSO-d_6) δ 11.02 (s, 1H), 9.89 (d, *J* = 1.6 Hz, 1H), 8.32-8.29 (m, 1H), 7.54-7.50 (m, 1H), 7.46-7.40 (m, 2H), 7.26-7.17 (m, 5H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.4, 161.2, 142.6, 136.4, 132.8, 131.7, 131.7, 125.7, 124.5, 124.1, 122.8, 122.0, 116.5, 116.3, 115.6, 111.8; HRMS (m/z, FAB^{+}): calcd for $[C_{15}H_{11}FNO]^{+}$ $[M+H]^{+}$ 240.0825, observed 240.0828.

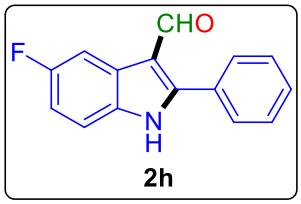
2-(thiophen-2-yl)-1*H*-indole-3-carbaldehyde (2g):[5] The title compound was



2g

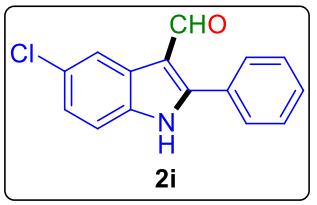
prepared according to the general procedure and obtain the desired compound as a brown solid (17 mg, 25% yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 11.43 (s, 1H), 10.20 (s, 1H), 8.20 (s, 1H), 7.45-7.43 (m, 2H), 7.34-7.31 (m, 1H), 7.15-7.09 (m, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 186.0, 141.8, 136.3, 131.4, 129.2, 128.6, 128.0, 126.3, 124.1, 122.8, 121.7, 114.6, 111.6.

5-fluoro-2-phenyl-1*H*-indole-3-carbaldehyde (2h): The title compound was prepared according to the general procedure and obtain the desired compound as orange solid (27 mg, 38 %); M.P. 278 ºC ~ 280 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 11.53 (s, 1H), 9.91 (s, 1H), 7.95-7.90 (m, 1H), 7.58-7.55 (m, 1H), 7.45-7.41 (m, 2H), 7.31-7.21 (m, 2H), 7.11-7.06 (1H), 6.92-6.86 (m, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 186.3, 150.6, 132.6, 130.1, 129.8, 128.8, 128.4, 126.3, 112.6, 112.0, 111.8, 107.2, 106.9; HRMS (m/z, APCI): calcd for $[C_{15}H_{11}NOF]^+$ $[M+H]^+$ 240.0825, observed 240.0822.



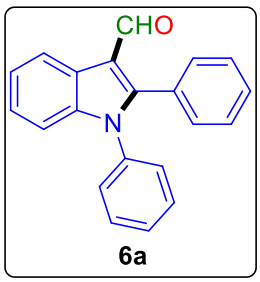
2h

5-chloro-2-phenyl-1*H*-indole-3-carbaldehyde (2i): The title compound was prepared according to the general procedure and obtain the desired compound as an orange oil (38 mg, 50% yield); M.P. 182 ºC ~ 184 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 11.41 (s, 1H), 9.96 (s, 1H), 8.32 (d, J = 2.4 Hz, 1H), 7.63-7.60 (m, 2H), 7.49-7.46 (m, 3H), 7.33 (d, J = 8.4 Hz, 1H), 7.17 (dd, J = 9.2, 2 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$ + 4 drops DMSO-d_6) δ 185.8, 149.7, 134.0, 131.7, 129.5, 129.3, 128.3, 127.8, 126.7, 123.5, 120.9, 113.5, 112.2; HRMS (m/z, APCI): calcd for $[C_{15}H_{11}NOCl]^+$ $[M+H]^+$ 256.0529, observed 256.0533.



2i

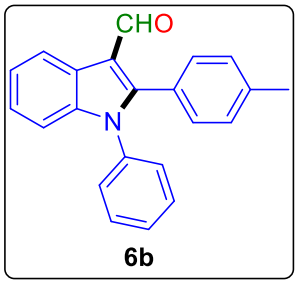
1,2-diphenyl-1*H*-indole-3-carbaldehyde (6a): The title compound was prepared according to the general procedure and obtain the desired compound as a pale yellow solid (49 mg, 55% yield); M.P. 234 ºC ~ 236 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.93 (s, 1H), 8.51 (d, J = 7.6 Hz, 1H), 7.41-7.29 (m, 11H), 7.23-7.20 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.6, 150.9, 138.3, 136.6, 131.4, 129.7, 129.5, 128.9, 128.6,



6a

128.5, 128.3, 125.4, 124.6, 123.9, 122.4, 116.5, 111.1; HRMS (m/z, APCI): calcd for $[C_{21}H_{16}NO]^+$ $[M+H]^+$ 298.1232, observed 298.1235.

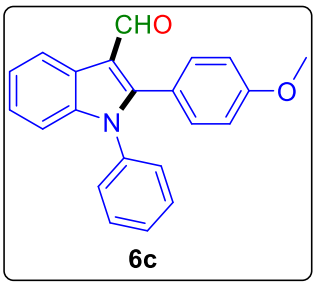
1-phenyl-2-(*p*-tolyl)-1*H*-indole-3-carbaldehyde (6b): The title compound was



6b

prepared according to the general procedure and obtain the desired compound as a yellow solid (44 mg, 48 % yield); M.P. 251 ºC ~ 253 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.93 (s, 1H), 8.50 (d, *J* = 8.0 Hz, 1H), 7.43-7.35 (m, 4H), 7.31-7.28 (m, 1H), 7.23-7.18 (m, 5H), 7.13 (d, *J* = 7.8 Hz, 2H), 2.35 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.7, 151.2, 139.7, 138.3, 136.7, 131.3, 129.6, 129.2, 128.51, 128.3, 125.8, 125.5, 124.5, 123.7, 122.3, 116.4, 111.0, 21.5; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{18}NO]^+$ $[M+H]^+$ 312.1388, observed 312.1385.

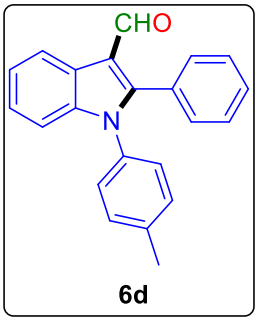
2-(4-methoxyphenyl)-1-phenyl-1H-indole-3-carbaldehyde (6c): The title compound



6c

was prepared according to the general procedure and obtain the desired compound as a yellow solid (32 mg, 33% yield); M.P. 199 ºC ~ 201 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 8.48 (d, *J* = 7.6 Hz, 1H), 7.41-7.33 (m, 4H), 7.29-7.19 (m, 6H), 6.83 (d, *J* = 8.8 Hz, 2H), 3.80 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.6, 160.6, 151.0, 138.3, 136.8, 132.8, 129.7, 128.5, 128.3, 125.5, 124.4, 123.7, 122.3, 120.9, 116.3, 114.0, 111.0, 55.5; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{18}NO_2]^+$ $[M+H]^+$ 328.1338, observed 328.1330.

2-phenyl-1-(*p*-tolyl)-1H-indole-3-carbaldehyde (6d): The title compound was

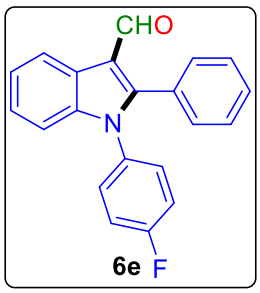


6d

prepared according to the general procedure and obtain the desired compound as a yellow solid (30 mg, 33% yield); M.P. 206 ºC ~ 208 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 8.50 (d, *J* = 8.0 Hz, 1H), 7.39-7.28 (m, 7H), 7.23-7.18 (m, 3H),

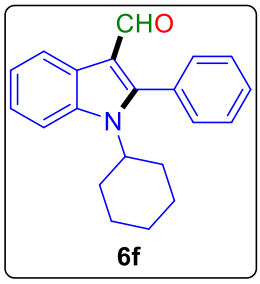
7.09 (d, J = 8.4 Hz, 2H), 2.38 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.6, 151.0, 138.6, 138.5, 134.0, 131.4, 130.3, 129.5, 129.0, 128.4, 128.0, 125.4, 124.5, 123.8, 122.3, 116.4, 111.2, 21.4; HRMS (m/z, APCI): calcd for $[C_{22}H_{18}NO]^+$ $[M+H]^+$ 312,1388, observed 312.1385.

1-(4-fluorophenyl)-2-phenyl-1*H*-indole-3-carbaldehyde (6e): The title compound



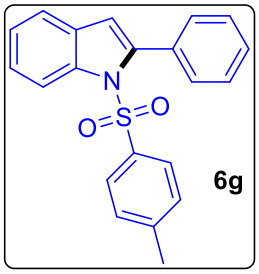
was prepared according to the general procedure and obtain the desired compound as a yellow solid (23 mg, 25% yield); M.P. 212 ºC ~ 214 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.93 (s, 1H), 8.51 (d, J = 8.0 Hz, 1H), 7.40-7.29 (m, 7H), 7.22-7.18 (m, 3H), 7.12-7.08 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.5, 163.4, 150.8, 138.4, 132.6, 131.4, 130.0, 130.0, 129.7, 128.6, 125.3, 124.7, 123.9, 122.5, 116.9, 116.7, 110.8; HRMS (m/z, FAB$^+$): calcd for $[C_{21}H_{15}FNO]^+$ $[M+H]^+$ 316.1138, observed 316.1130.

1-cyclohexyl-2-phenyl-1*H*-indole-3-carbaldehyde (6f): The title compound was

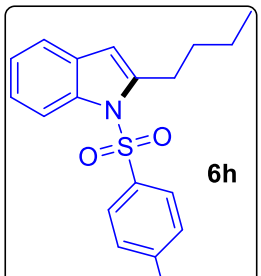


prepared according to the general procedure and obtain the desired compound as a yellow solid (35 mg, 36% yield); M.P. 218 ºC ~ 220 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.58 (s, 1H), 8.47-8.45 (m, 1H), 7.69-7.65 (m, 1H), 7.57-7.52 (m, 3H), 7.46-7.43 (m, 2H), 7.34-7.29 (m, 2H), 4.13-4.05 (m, 1H), 2.35-2.29 (m, 2H), 1.89-1.87 (m, 4H), 1.29-1.21 (m, 4H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.0, 151.6, 135.5, 130.8, 129.9, 129.7, 128.7, 128.0, 126.4, 123.5, 123.0, 122.6, 113.1, 57.3, 31.5, 26.1, 25.4; HRMS (m/z, FAB$^+$): calcd for $[C_{21}H_{22}NO]^+$ $[M+H]^+$ 304.1701, observed 304.1710.

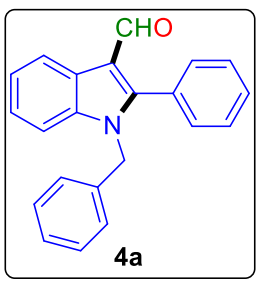
2-phenyl-1-tosyl-1*H*-indole (6g):[8] The title compound was prepared according to the general procedure and obtain the desired compound as a white solid (66 mg, 63%); M.P. 188 ºC ~ 190 ºC; Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.30 (d, *J* = 8.8 Hz, 1H), 7.50-7.47 (m, 2H), 7.44-7.39 (m, 4H), 7.36-7.32 (m, 1H), 7.27-7.23 (m, 3H), 7.02 (d, *J* = 8.0 Hz, 2H), 6.53 (s, 1H), 2.27 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 144.7, 142.3, 138.5, 134.8, 132.6, 130.8, 130.5, 129.4, 128.9, 127.7, 127.0, 125.0, 124.5, 120.1, 116.9, 113.8, 21.7; HRMS (m/z, FAB$^+$): calcd for $[C_{21}H_{17}NO_2S]^+$ $[M]^+$ 347.0980, observed 347.0972.



2-butyl-1-tosyl-1*H*-indole (6h):[8] The title compound was prepared according to the general procedure and obtain the desired compound as a yellow solid (56 mg, 58%); M.P. 188 ºC ~ 190 ºC; Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* =7.2 Hz, 1H), 7.25-7.16 (m, 3H), 6.38 (s, 1H), 2.99 (t, *J* = 7.6 Hz, 2H), 2.32 (s, 3H), 1.75-1.69 (m, 2H), 1.47-1.41 (m, 2H), 0.96 (t, *J* = 7.6 Hz, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 144.8, 142.7, 137.4, 136.4, 130.0, 129.9, 126.4, 123.9, 123.6, 120.2, 115.0, 108.8, 31.1, 28.9, 22.7, 21.7, 14.1.

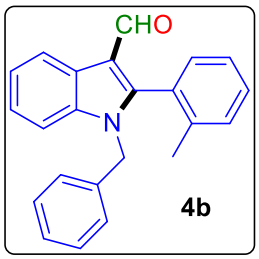


1-benzyl-2-phenyl-1*H*-indole-3-carbaldehyde (4a):[6] The title compound was prepared according to the general procedure and obtain the desired compound as a reddish brown solid (31 mg, 34% yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.66 (s, 1H), 8.36-8.35 (m, 1H), 7.41-7.30 (m, 5H), 7.26-7.22 (m, 1H), 7.17-7.11 (m, 5H), 6.87-6.85 (m, 2H), 5.18 (s,



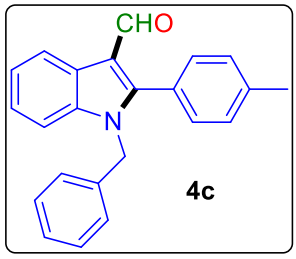
2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.0, 151.9, 137.1, 136.6, 131.0, 130.2, 129.1, 128.9, 128.7, 127.9, 126.2, 125.6, 124.4, 123.6, 122.5, 116.4, 111.0, 48.0.

1-benzyl-2-(*o*-tolyl)-1*H*-indole-3-carbaldehyde (4b): The title compound was



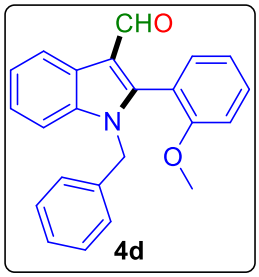
prepared according to the general procedure and obtain the desired compound as a brown solid (46 mg, 48% yield); M.P. 148 ºC ~ 150 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.60 (s, 1H), 8.44 (m, 1H), 7.44-7.40 (m, 1H), 7.35-7.25 (m, 6H), 7.22-7.20 (m, 3H), 6.90-6.88 (m, 2H), 5.13 (dd, J = 38.4, 16.1 Hz, 2H), 2.05 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.5, 151.4, 138.7, 137.1, 136.2, 131.3, 130.6, 130.4, 128.9, 128.4, 128.0, 126.7, 126.1, 125.5, 124.2, 123.5, 122.4, 116.4, 110.9, 47.8, 20.1; HRMS (m/z, APCI): calcd for $[C_{23}H_{20}NO]^+$ $[M+H]^+$ 326.1545, observed 326.1541.

1-benzyl-2-(*p*-tolyl)-1*H*-indole-3-carbaldehyde (4c):[7] The title compound was



prepared according to the general procedure and obtain the desired compound as a yellow solid (62 mg, 64% yield); M.P. 151 ºC ~ 153 ºC; Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.47-8.44 (m, 1H), 7.33-7.29 (m, 3H), 7.28-7.19 (m, 7H), 6.99-6.96 (m, 2H), 5.28 (s, 2H), 2.42 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.1, 152.2, 140.4, 137.1, 136.7, 130.9, 129.6, 129.1, 127.8, 126.2, 125.7, 125.7, 124.3, 123.5, 122.4, 116.3, 110.9, 47.9, 21.6.

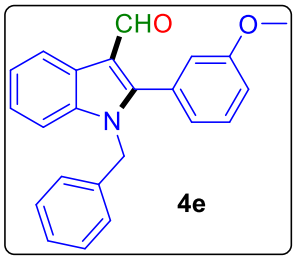
1-benzyl-2-(2-methoxyphenyl)-1*H*-indole-3-carbaldehyde (4d): The title compound



was prepared according to the general procedure and obtain the desired compound as a yellow solid (57 mg, 56% yield); M.P. 156 ºC ~ 158 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.55 (s, 1H), 8.33-8.29 (m, 1H), 7.39-7.34 (m, 1H), 7.21-7.17 (m, 2H), 7.12-7.08 (m, 5H), 6.94-6.81 (m, 4H), 5.05 (q, J = 16.0 Hz, 2H), 3.51 (s, 3H); ^{13}C-NMR (100

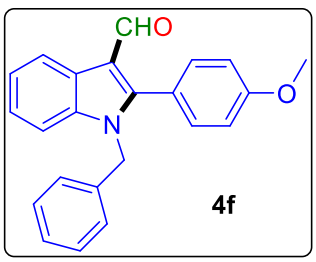
MHz, $CDCl_3$) δ 186.9, 158.1, 149.2, 137.2, 136.6, 133.4, 132.0, 128.8, 127.6, 126.6, 125.8, 124.0, 123.2, 122.3, 120.8, 117.6, 116.4, 111.3, 111.0, 55.5, 48.3; HRMS (m/z, FAB^+): calcd for $[C_{23}H_{20}NO_2]^+$ $[M+H]^+$ 342.1494, observed 342.1487.

1-benzyl-2-(3-methoxyphenyl)-1*H*-indole-3-carbaldehyde (4e): The title compound



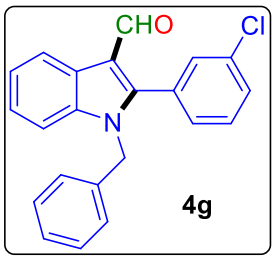
was prepared according to the general procedure and obtain the desired compound as a brown solid (47 mg, 40% yield); M.P. 155 ºC ~ 156 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.70 (s, 1H), 8.36-8.34 (m, 1H), 7.29-7.22 (m, 2H), 7.20 -7.12 (m, 5H), 6.94-6.86 (m, 4H), 6.78 (m, 1H), 5.19 (s, 2H), 3.56 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.1, 159.7, 151.7, 137.2, 136.8, 129.9, 129.1, 127.9, 126.2, 125.6, 124.5, 123.6, 123.4, 122.5, 116.3, 116.1, 110.9, 55.4, 48.0; HRMS (m/z, FAB^+): calcd for $[C_{23}H_{20}NO_2]^+$ $[M+H]^+$ 342.1494, observed 342.1491.

benzyl-2-(4-methoxyphenyl)-1*H*-indole-3-carbaldehyde (4f): The title compound



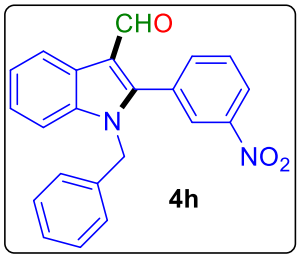
was prepared according to the general procedure and obtain the desired compound as a yellow solid (47 mg, 46% yield); M.P. 128 ºC ~ 130 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 8.47-8.43 (m, 1H), 7.36-7.31 (m, 3H), 7.28-7.20 (m, 5H), 7.01-6.97 (m, 4H), 5.28 (s, 2H), 3.86 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.1, 161.1, 152.0, 137.1, 136.8, 132.3, 129.1, 127.9, 126.1, 125.7, 124.3, 123.5, 122.4, 120.7, 116.3, 114.4, 110.9, 55.6, 47.9; HRMS (m/z, FAB^+): calcd for $[C_{23}H_{20}NO_2]^+$ $[M+H]^+$ 342.1494, observed 342.1487.

1-benzyl-2-(3-chlorophenyl)-1*H*-indole-3-carbaldehyde (4g): The title compound



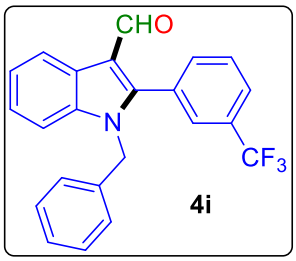
was prepared according to the general procedure and obtain the desired compound as a yellow solid (46 mg, 44% yield); M.P. 204 ºC ~ 206 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.46 (d, J = 7.7 Hz, 1H), 7.51-7.49 (m, 1H), 7.42-7.25 (m, 9H), 6.96-6.94 (m, 2H), 5.28 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.5, 149.7, 137.2, 136.4, 134.9, 131.0, 130.6, 130.4, 130.1, 129.2, 129.2, 128.1, 126.2, 125.5, 124.8, 123.8, 122.6, 116.7, 110.9, 48.0; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{17}ClNO]^+$ $[M+H]^+$ 346.0999, observed 346.0992.

1-benzyl-2-(3-nitrophenyl)-1*H*-indole-3-carbaldehyde (4h): The title compound



was prepared according to the general procedure and obtain the desired compound as a yellow oil (45 mg, 42% yield); ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.48-8.25 (m, 3H), 7.75-7.63 (m, 2H), 7.41-7.30 (m, 3H), 7.29-7.24 (m, 3H), 6.92 (dd, J = 6.6, 2.9 Hz, 2H), 5.30 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 185.6, 148.0, 147.6, 137.1, 136.5, 135.8, 130.4, 129.8, 129.1, 128.0, 125.8, 125.5, 125.1, 124.9, 124.7, 123.8, 122.3, 116.9, 110.7, 47.8; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{17}N_2O_3]^+$ $[M+H]^+$ 357.1239, observed 357.1241.

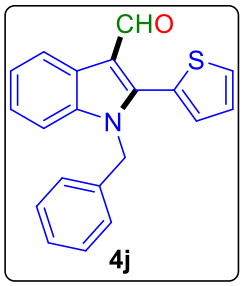
1-benzyl-2-(3-(trifluoromethyl)phenyl)-1*H*-indole-3-carbaldehyde (4i): The title



compound was prepared according to the general procedure and obtain the desired compound as a yellow solid (37 mg, 32% yield); M.P. 162 ºC ~ 164 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.74 (s, 1H), 8.46 (d, J = 8.0 Hz, 1H), 7.77-7.76 (m, 1H), 7.62-7.58 (m, 3H), 7.37-7.24 (m, 6H), 6.93-6.91 (m, 2H), 5.26 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.3, 149.3, 137.3, 136.3, 134.2, 131.6, 129.8, 129.5, 129.3,

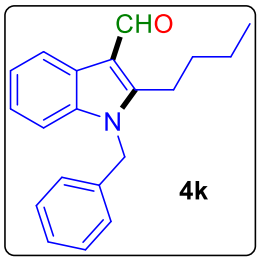
128.2, 127.8, 127.8, 126.9, 126.1, 125.4, 124.9, 123.9, 122.6, 116.9, 110.9, 48.1; HRMS (m/z, FAB$^+$): calcd for $[C_{23}H_{17}F_3NO]^+$ $[M+H]^+$ 380.1262, observed 380.1258.

1-benzyl-2-(thiophen-2-yl)-1*H*-indole-3-carbaldehyde (4j): The title compound was



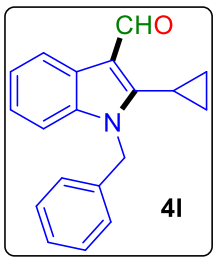
prepared according to the general procedure and obtain the desired compound as a white solid (57 mg, 60% yield); M.P. 208 ºC ~ 210 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.97 (s, 1H), 8.46 (d, J = 8.0 Hz, 1H), 7.57 (dd, J = 5.1, 1.3 Hz, 1H), 7.36-7.23 (m, 6H), 7.20-7.15 (m, 2H), 7.02-7.00 (m, 2H), 5.39 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.9, 143.6, 137.4, 136.6, 131.8, 129.9, 129.2, 128.2, 128.0, 127.9, 126.1, 125.5, 124.8, 123.7, 122.5, 117.7, 110.9, 48.0; HRMS (m/z, APCI): calcd for $[C_{20}H_{16}NOS]^+$ $[M+H]^+$ 318.0953, observed 318.0958.

1-benzyl-2-butyl-1*H*-indole-3-carbaldehyde (4k):[8] The title compound was



prepared according to the general procedure and obtain the desired compound as a brown solid (27 mg, 30% yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.15 (s, 1H), 8.26 (d, J = 8.0 Hz, 1H), 7.24-7.13 (m, 7H), 6.92 (d, J = 6.0 Hz, 2H), 5.31 (s, 2H), 3.00 (t, J = 7.6 Hz, 2H), 1.54-1.47 (m, 2H), 1.37-1.29 (m, 2H), 0.82 (t, J = 7.6 Hz, 3H); ^{13}C-NMR (400 MHz, $CDCl_3$) δ 184.6, 152.2, 137.0, 136.2, 129.2, 128.1, 126.0, 123.6, 123.2, 121.3, 114.6, 110.2, 46.9, 32.8, 24.6, 22.7, 13.9.

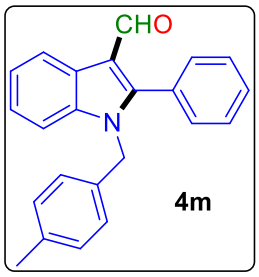
1-benzyl-2-cyclopropyl-1*H*-indole-3-carbaldehyde (4l): The title compound was



prepared according to the general procedure and obtain the desired compound as a red solid (27 mg, 33% yield); M.P. 115 ºC ~ 117 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.47 (s, 1H), 8.38 (d, J = 7.7 Hz, 1H), 7.31- 7.16 (m, 6H), 7.01-6.99 (m, 2H), 5.57 (s, 2H), 1.92-1.84

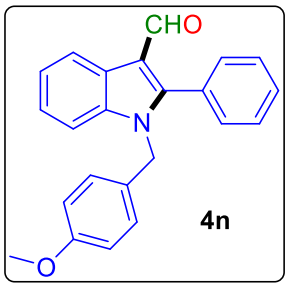
(m, 1H), 1.20-1.13 (m, 2H), 0.93-0.88 (m, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.0, 151.6, 136.7, 136.6, 129.2, 127.9, 126.1, 125.5, 123.9, 123.3, 122.3, 116.1, 110.1, 47.5, 6.6, 6.3; HRMS (m/z, FAB$^+$): calcd for $[C_{19}H_{18}NO]^+$ $[M+H]^+$ 276.1388, observed 276.1389.

1-(4-methylbenzyl)-2-phenyl-1*H*-indole-3-carbaldehyde (4m): The title compound



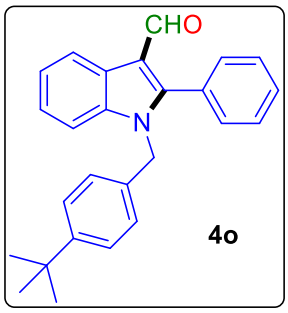
was prepared according to the general procedure and obtain the desired compound as a white solid (50 mg, 51% yield); M.P. 148 ºC ~ 150 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.46 (d, *J* = 7.8 Hz, 1H), 7.52-7.42 (m, 5H), 7.36-7.32 (m, 1H), 7.30-7.22 (m, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 5.25 (s, 2H), 2.30 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.1, 151.9, 137.6, 137.1, 133.6, 131.0, 130.1, 129.8, 128.8, 128.8, 126.1, 125.7, 124.4, 123.6, 122.4, 116.4, 111.0, 47.8, 21.3; HRMS (m/z, FAB$^+$): calcd for $[C_{23}H_{20}NO]^+$ $[M+H]^+$ 326.1545, observed 326.1538.

1-(4-methoxybenzyl)-2-phenyl-1H-indole-3-carbaldehyde (4n): The title compound

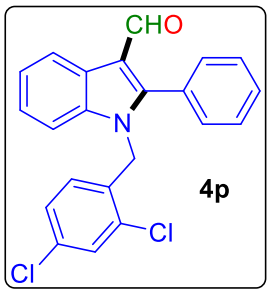


was prepared according to the general procedure and obtain the desired compound as a yellow solid (45 mg, 44% yield); M.P. 138 ºC ~ 140 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 8.47-8.45 (m, 1H), 7.52-7.41 (m, 5H), 7.35-7.24 (m, 3H), 6.88 (d, *J* = 8.0, 2H), 6.78 (d, *J* = 8.0, 2H), 5.22 (s, 2H), 3.74 (s, 3H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.0, 159.2, 151.8, 137.1, 131.0, 130.1, 128.8, 128.6, 127.5, 125.6, 124.4, 123.5, 122.4, 116.3, 114.5, 114.0, 111.0, 55.4, 47.4; HRMS (m/z, FAB$^+$): calcd for $[C_{23}H_{20}NO_2]^+$ $[M+H]^+$ 342.1494, observed 342.1487.

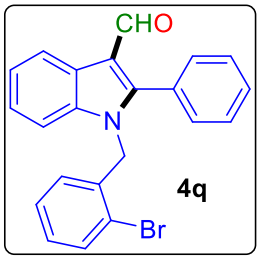
1-(4-(tert-butyl)benzyl)-2-phenyl-1H-indole-3-carbaldehyde (4o): The title compound was prepared according to the general procedure and obtain the desired compound as a pale yellow solid (60 mg, 54% yield); M.P. 180 ºC ~ 182 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.46 (d, *J* = 8.0 Hz, 1H), 7.53-7.45 (m, 5H), 7.37-7.33 (m, 1H), 7.31-7.26 (m, 4H), 6.91 (d, *J* = 8.0 Hz, 2H), 5.27 (s, 2H), 1.28 (s, 9H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.1, 151.9, 150.9, 137.1, 133.5, 131.1, 130.1, 128.8, 128.8, 126.0, 126.0, 125.7, 124.3, 123.5, 122.4, 116.3, 111.1, 47.7, 34.7, 31.5; HRMS (m/z, FAB$^+$): calcd for $[C_{26}H_{26}NO]^+$ $[M+H]^+$ 368.2014, observed 368.2008.



1-(2,4-dichlorobenzyl)-2-phenyl-1*H*-indole-3-carbaldehyde (4p): The title compound was prepared according to the general procedure and obtain the desired compound as a yellow solid (34 mg, 30% yield); M.P. 188 ºC ~ 190 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 8.47 (d, *J* = 8.0 Hz, 1H), 7.52-7.45 (m, 10H), 6.50 (d, *J* = 8.0 Hz, 1H), 5.28 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.9, 151.5, 136. 7, 134.3, 132.7, 132.6, 130.6, 130.3, 129.7, 128.9, 128.1, 127.9, 127.8, 125.5, 124.6, 123.8, 122.6, 116.6, 110.4, 45.3; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{16}Cl_2NO]^+$ $[M+H]^+$ 380.0609, observed 380.0608.

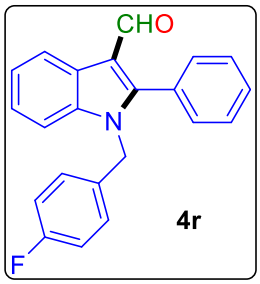


1-(2-bromobenzyl)-2-phenyl-1*H*-indole-3-carbaldehyde (4q): The title compound was prepared according to the general procedure and obtain the desired compound as a white solid (43 mg, 37% yield); M.P. 184 ºC ~ 186 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 1H), 8.49 (d, *J* = 7.8 Hz, 1H), 7.60-7.58 (m, 1H), 7.53-7.45 (m, 3H), 7.43-7.35 (m, 3H), 7.31-7.27(m, 1H), 7.17-7.14 (m, 3H), 6.59-6.56 (m, 1H), 5.31 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.1, 151.8, 136.9, 135.7, 133.2, 130.8, 130.3, 129.4, 129.0,



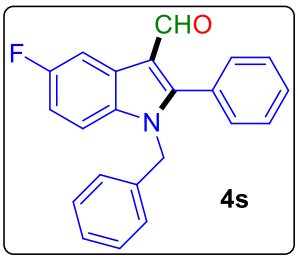
128.4, 128.2, 127.3, 125.6, 124.7, 123.8, 122.6, 121.8, 116.6, 110.8, 48.4; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{17}BrNO]^+$ $[M+H]^+$ 390.0494, observed 390.0485.

1-(4-fluorobenzyl)-2-phenyl-1*H*-indole-3-carbaldehyde (4r): The title compound



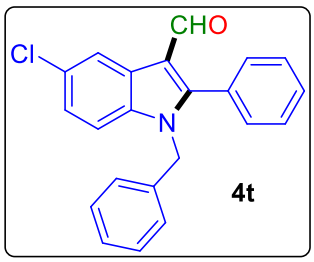
was prepared according to the general procedure and obtain the desired compound as a brown solid (46 mg, 47% yield); M.P. 234 ºC ~ 236 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.48-8.46 (m, 1H), 7.56-7.46 (m, 3H), 7.42-7.40 (m, 2H), 7.36-7.28 (m, 2H), 7.25-7.23 (m, 1H), 6.98 - 6.91 (m, 4H), 5.26 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 187.0, 163.6, 151.6, 137.0, 132.4, 131.0, 130.3, 128.9, 127.93, 127.9, 125.7, 124.5, 123.7, 122.6, 116.2, 116.0, 110.8, 47.3; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{17}FNO]^+$ $[M+H]^+$ 330.1294, observed 330.1285.

1-benzyl-5-fluoro-2-phenyl-1H-indole-3-carbaldehyde (4s): [6] The title compound



was prepared according to the general procedure and obtain the desired compound as a yellow solid (39 mg, 40% yield); M.P. 171 ºC ~ 173 ºC; Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.74 (s, 1H), 8.14 (dd, J = 8.2, 4.1 Hz, 1H), 7.53-7.42 (m, 5H), 7.29-7.26 (m, 3H), 7.13 (dd, J = 8.9, 4.2 Hz, 1H), 7.01 (dd, J = 9.0, 2.6 Hz, 1H), 6.98-6.94 (m, 2H), 5.28 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.7, 161.5, 152.7, 136.3, 133.5, 130.9, 130.4, 129.2, 129.0, 128.5, 128.1, 126.1, 112.8, 112.6, 111.9, 108.1, 107.8, 48.2.

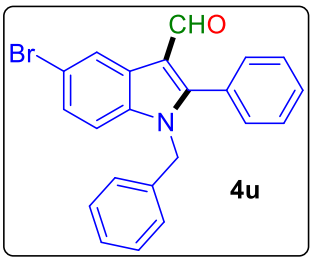
1-benzyl-5-chloro-2-phenyl-1*H*-indole-3-carbaldehyde(4t): [6] The title compound



was prepared according to the general procedure and obtain the desired compound as a brown solid (56 mg, 55% yield); Spectroscopic data is matched with previous literature; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 8.44 (s, 1H), 7.52-

7.40 (m, 5H), 7.30-7.18 (m, 4H), 7.11 (d, J = 8.8 Hz, 1H), 6.93 (d, J = 6.8 Hz, 2H), 5.26 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.6, 152.4, 136.1, 135.3, 130.8, 130.3, 129.4, 129.1, 128.9, 128.1, 128.0, 126.5, 126.0, 124.6, 121.9, 115.7, 111.9, 48.0.

benzyl-5-bromo-2-phenyl-1*H*-indole-3-carbaldehyde (4u): The title compound was



prepared according to the general procedure and obtain the desired compound as a yellow solid (58 mg, 50% yield); M.P. 186 ºC ~ 188 ºC; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 1H), 8.61 (s, 1H), 7.52-7.45 (m, 3H), 7.41 (d, J = 7.2 Hz, 2H), 7.34-7.31 (m, 1H), 7.26-7.24 (m, 3H), 7.06 (dd, J = 8.8, 2.4 Hz, 1H), 6.93 (d, J = 6.4 Hz, 2H), 5.26 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 186.6, 152.2, 136.0, 135.6, 130.8, 130.3, 129.1, 128.9, 128.1, 128.0, 127.3, 127.0, 126.0, 125.0, 117.0, 115.6, 112.3, 48.0; HRMS (m/z, FAB$^+$): calcd for $[C_{22}H_{17}BrNO]^+$ $[M+H]^+$ 390.0494, observed 390.0504.

VII. References

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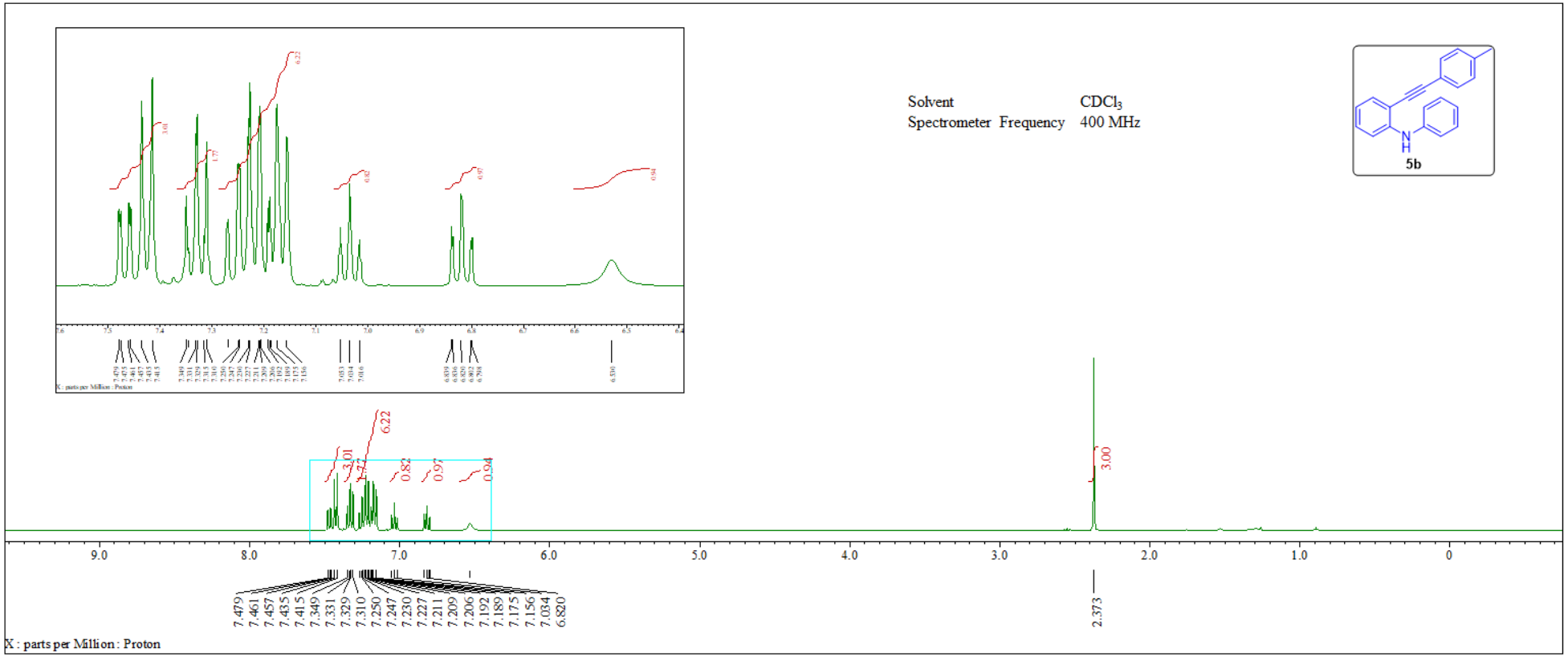
VIII. X-ray data analysis

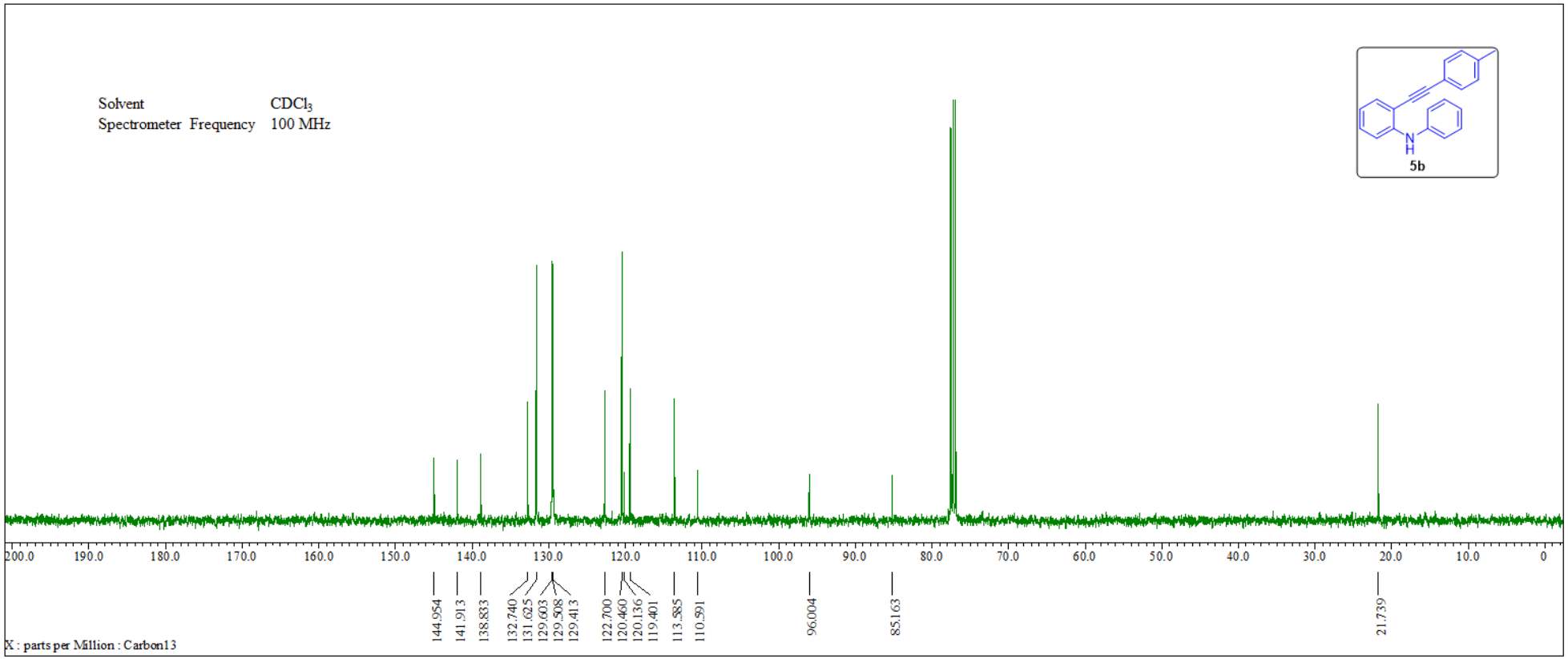
2a **2a**

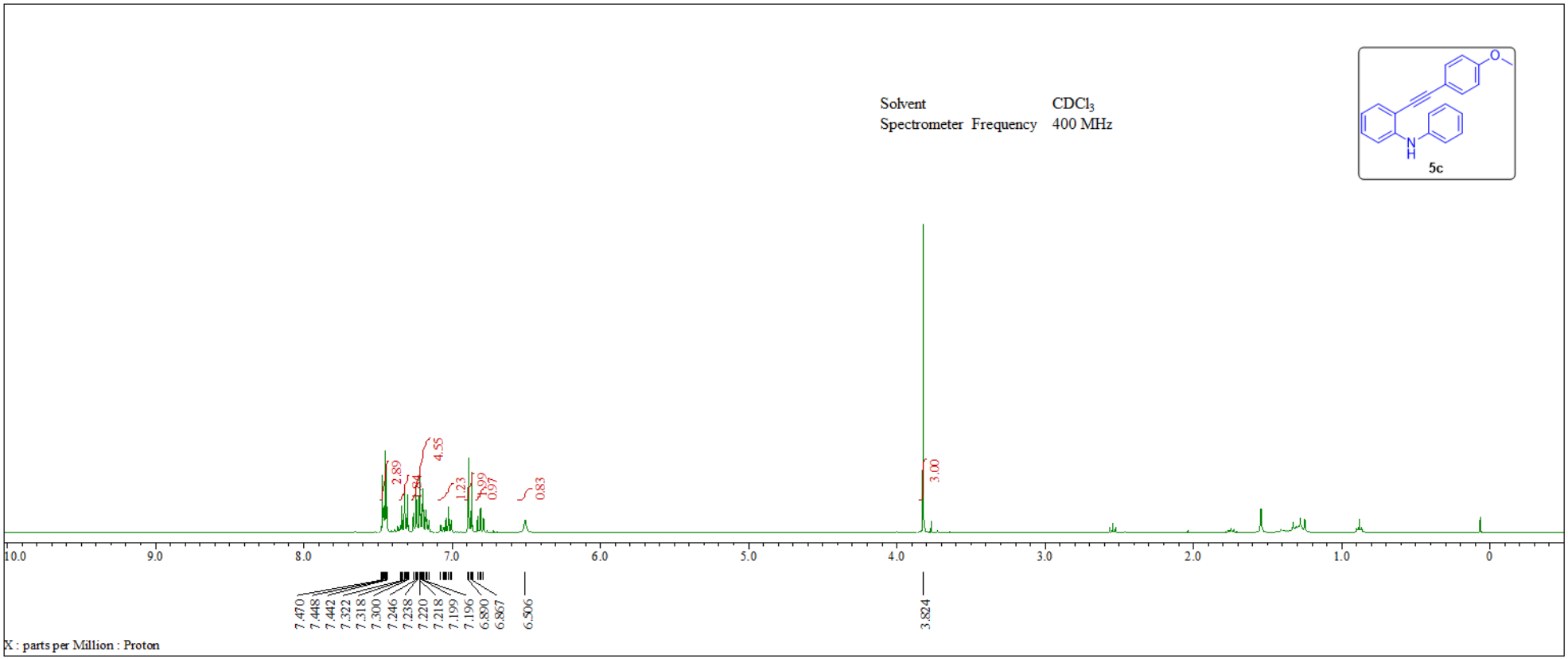
Table S2. Crystal data and structure refinement for 2a.

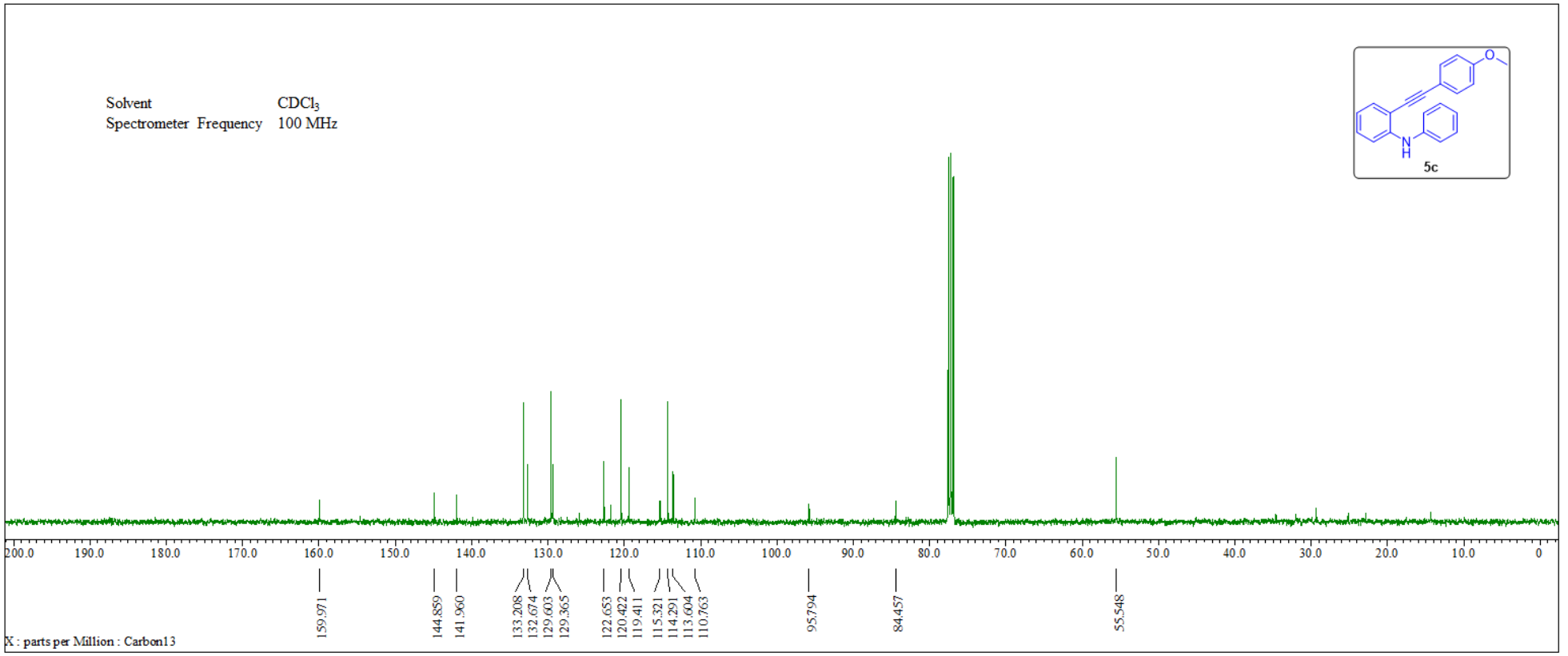
Identification code	**2a**	
Empirical formula	$C_{15}H_{11}NO$	
Formula weight	221.25	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbcn	
Unit cell dimensions	a = 22.6720(20) Å	α= 90°.
	b = 14.2171(16) Å	β= 90°.
	c = 6.8368(6) Å	γ = 90°.
Volume	2203.7(4) $Å^3$	
Z	8	
Density (calculated)	1.334 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	928	
Crystal size	0.40 x 0.15 x 0.13 mm^3	
Theta range for data collection	2.865 to 29.303°.	
Index ranges	-31<=h<=22, -18<=k<=9, -8<=l<=8	
Reflections collected	6932	
Independent reflections	2591 [R(int) = 0.0339]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.98371	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2591 / 0 / 155	
Goodness-of-fit on F^2	1.031	
Final R indices [I>2sigma(I)]	R1 = 0.0519, wR2 = 0.1215	
R indices (all data)	R1 = 0.0749, wR2 = 0.1344	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.250 and -0.258 e.$Å^-$	

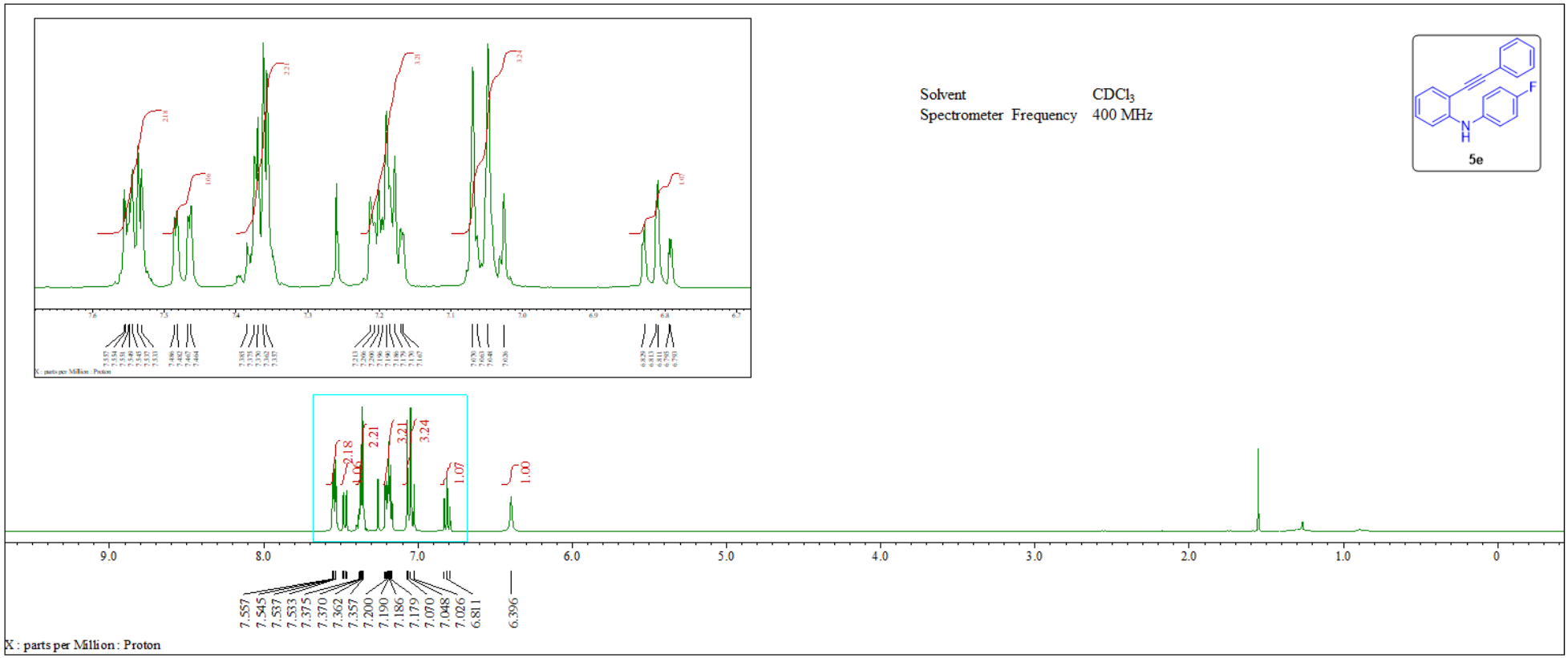
IX. ^{1}H, and ^{13}C Spectra

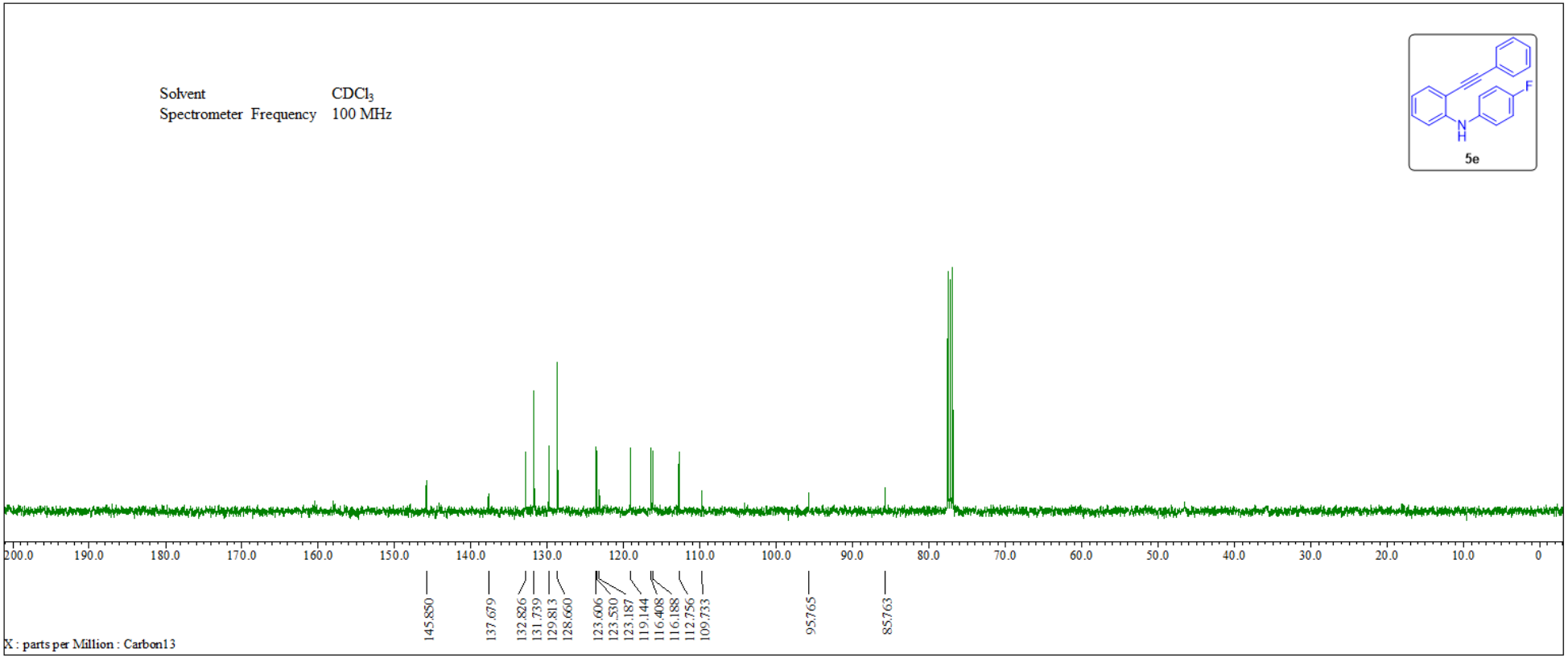


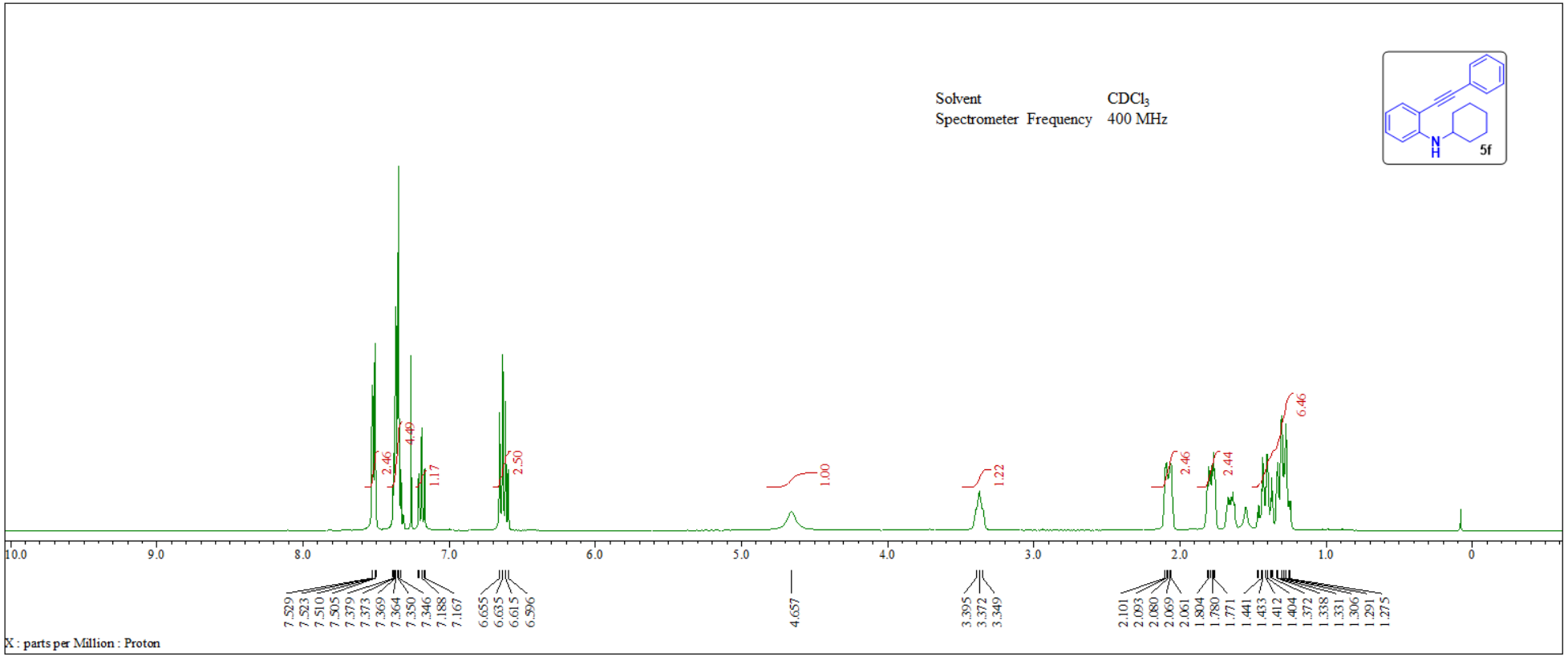

Solvent CDCl3
Spectrometer Frequency 100 MHz
5b
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
144.954 141.913 138.833 132.740 131.625 129.603 129.508 129.413 122.700 120.460 120.136 119.401 113.585 110.591 96.004 85.163 21.739
X : parts per Million : Carbon13

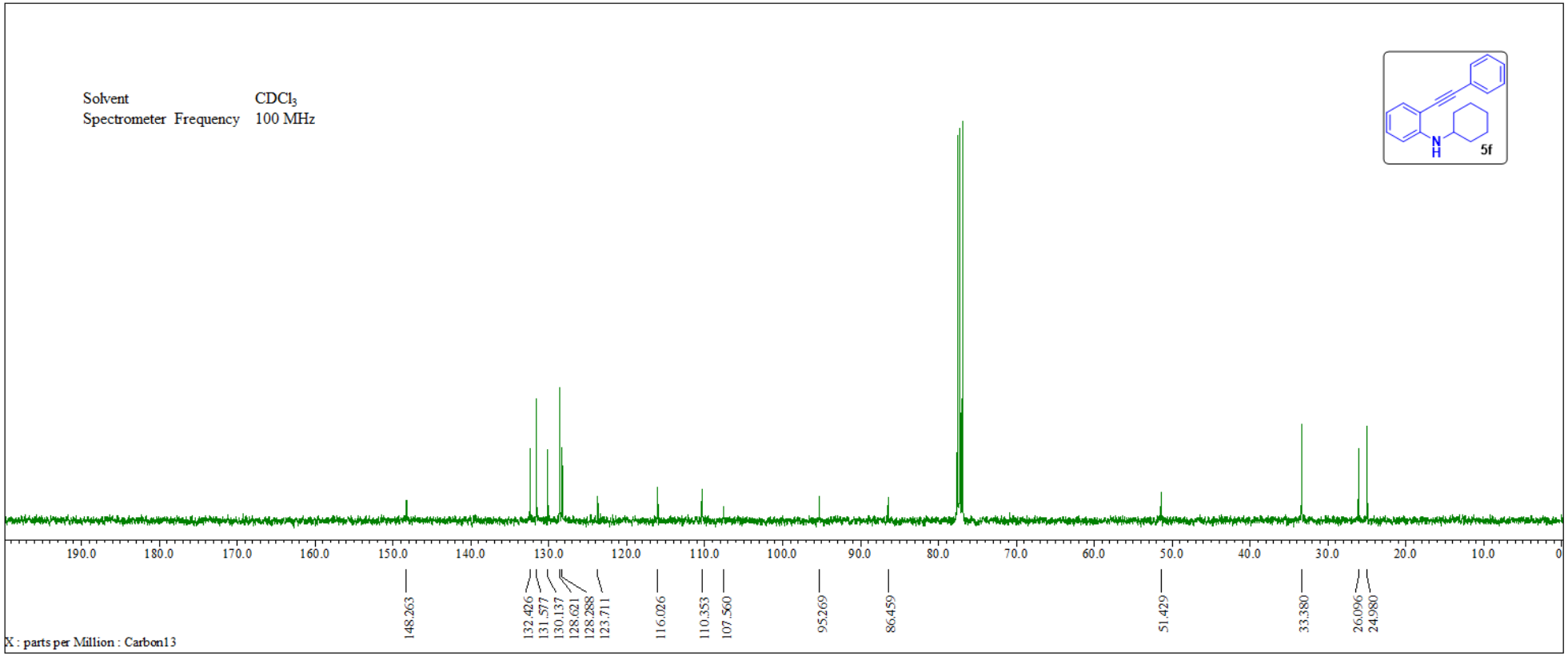

Solvent CDCl3
Spectrometer Frequency 400 MHz
5c
4.55
2.89
1.84
1.23
0.99
0.97
0.83
3.00
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.470
7.448
7.442
7.322
7.318
7.300
7.246
7.238
7.220
7.218
7.199
7.196
6.890
6.867
6.506
3.824
X : parts per Million : Proton

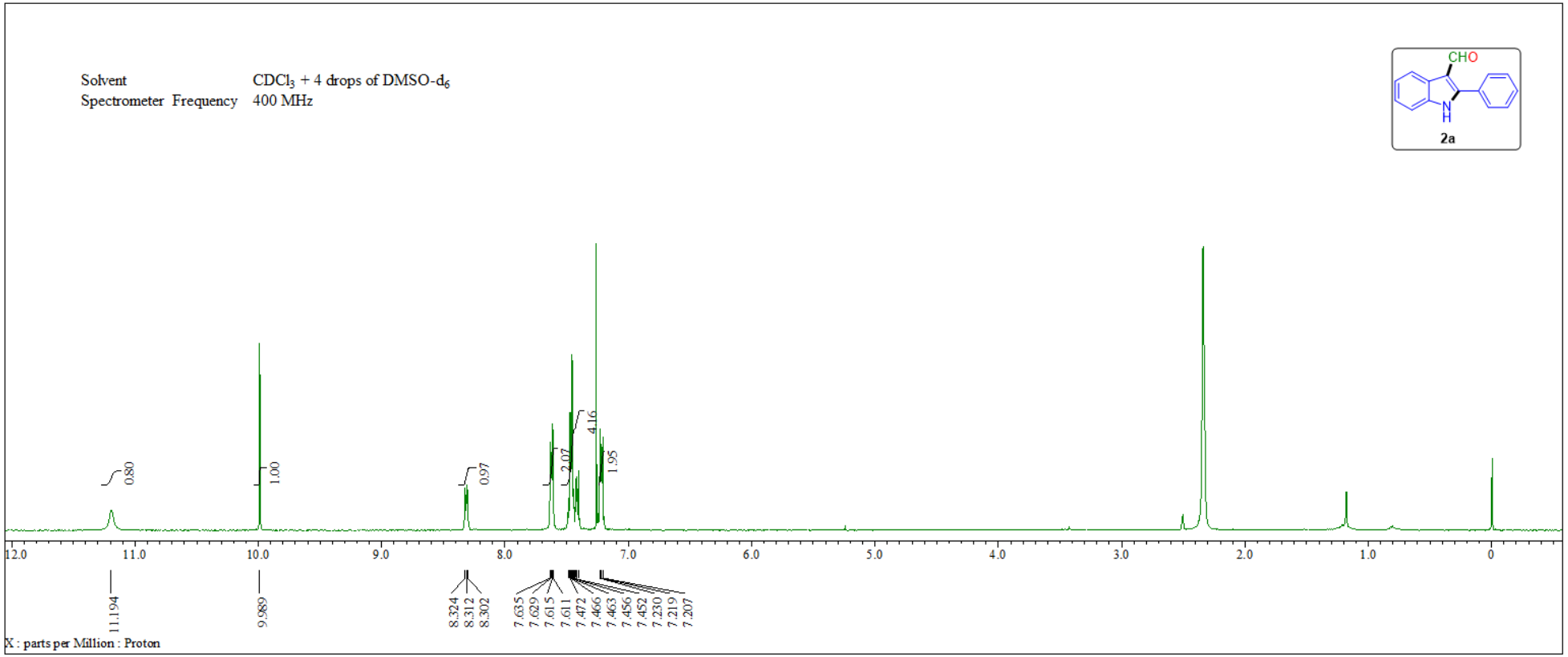

Solvent CDCl3
Spectrometer Frequency 100 MHz
5c
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
159.971
144.859
141.960
133.208
132.674
129.603
129.365
122.653
120.422
119.411
115.321
114.291
113.604
110.763
95.794
84.457
55.548
X : parts per Million : Carbon13

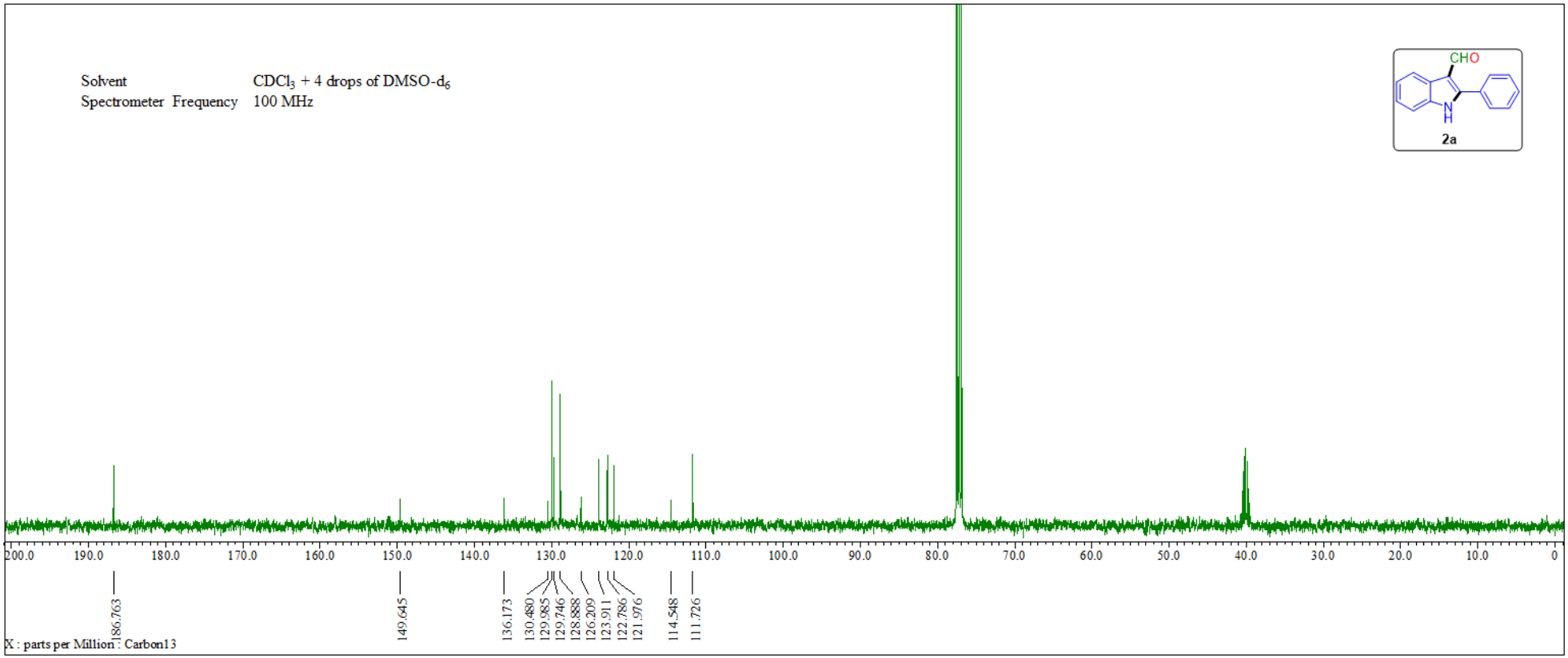

Solvent CDCl3
Spectrometer Frequency 400 MHz
5e
F
N
H
2.18
2.21
3.21
3.24
1.07
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.557
7.545
7.537
7.533
7.375
7.370
7.362
7.357
7.200
7.190
7.186
7.179
7.070
7.048
7.026
6.811
6.396
X : parts per Million : Proton

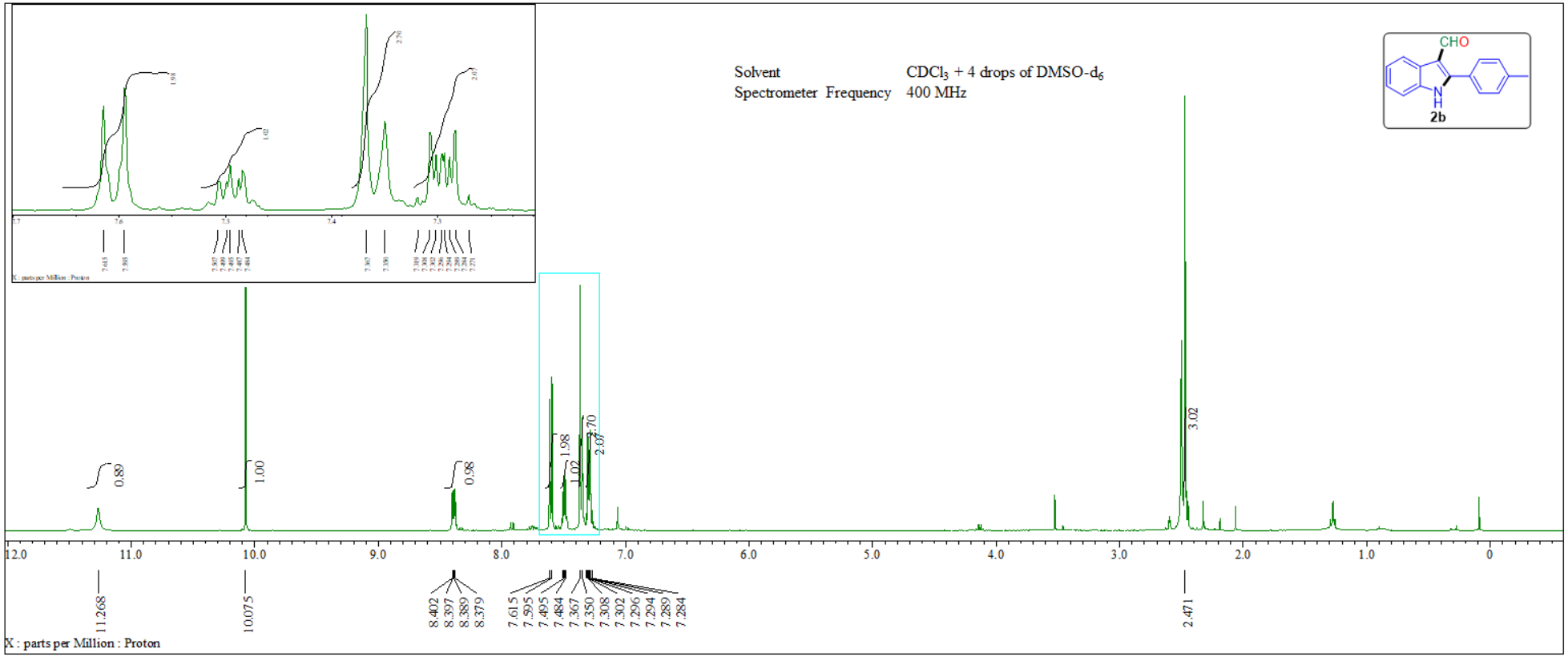

Solvent CDCl3
Spectrometer Frequency 100 MHz
F
N
H
5e
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
145.850
137.679
132.826
131.739
129.813
128.660
123.606
123.530
123.187
119.144
116.408
116.188
112.756
109.733
95.765
85.763
X : parts per Million : Carbon13

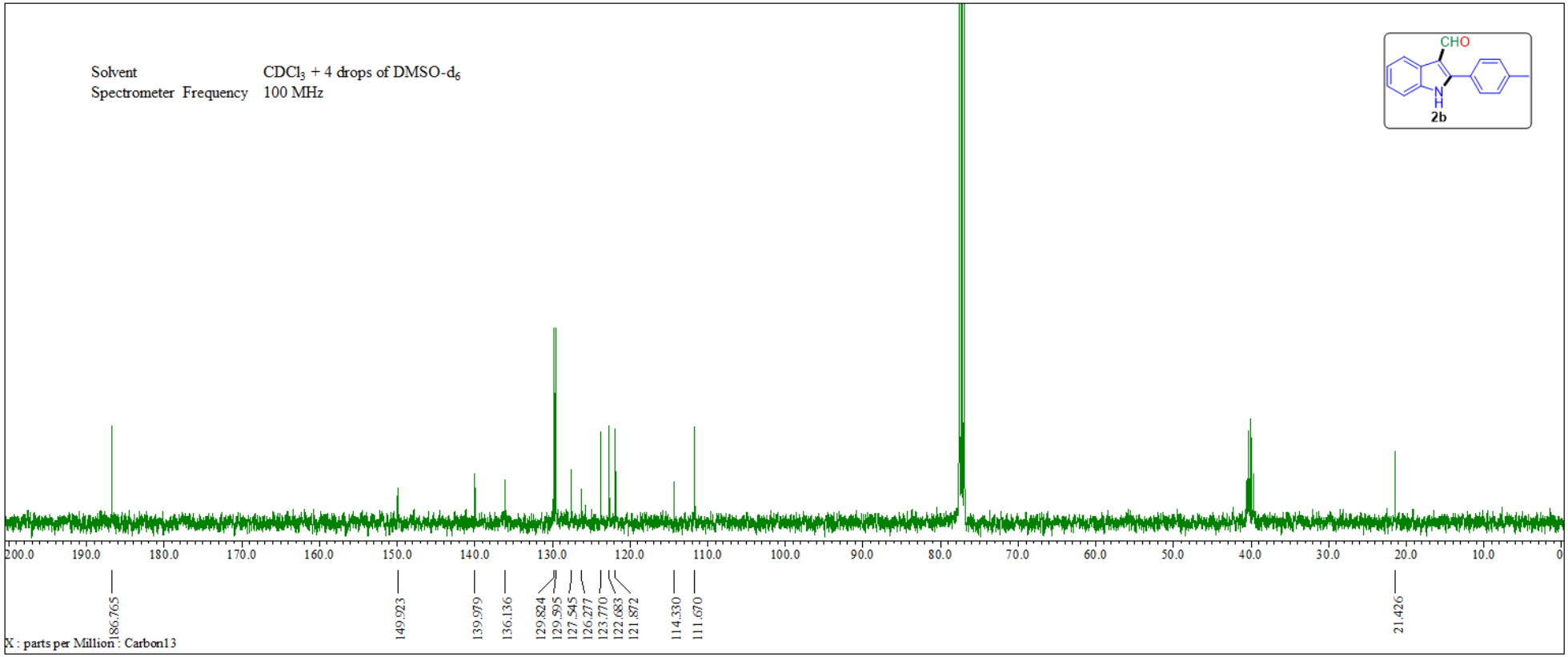

Solvent CDCl3
Spectrometer Frequency 400 MHz
5f
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
2.46
4.49
1.17
2.50
1.00
1.22
2.46
2.44
6.46
7.529
7.523
7.510
7.505
7.379
7.373
7.369
7.364
7.350
7.346
7.188
7.167
6.655
6.635
6.615
6.596
4.657
3.395
3.372
3.349
2.101
2.093
2.080
2.069
2.061
1.804
1.780
1.771
1.441
1.433
1.412
1.404
1.372
1.338
1.331
1.306
1.291
1.275
X : parts per Million : Proton

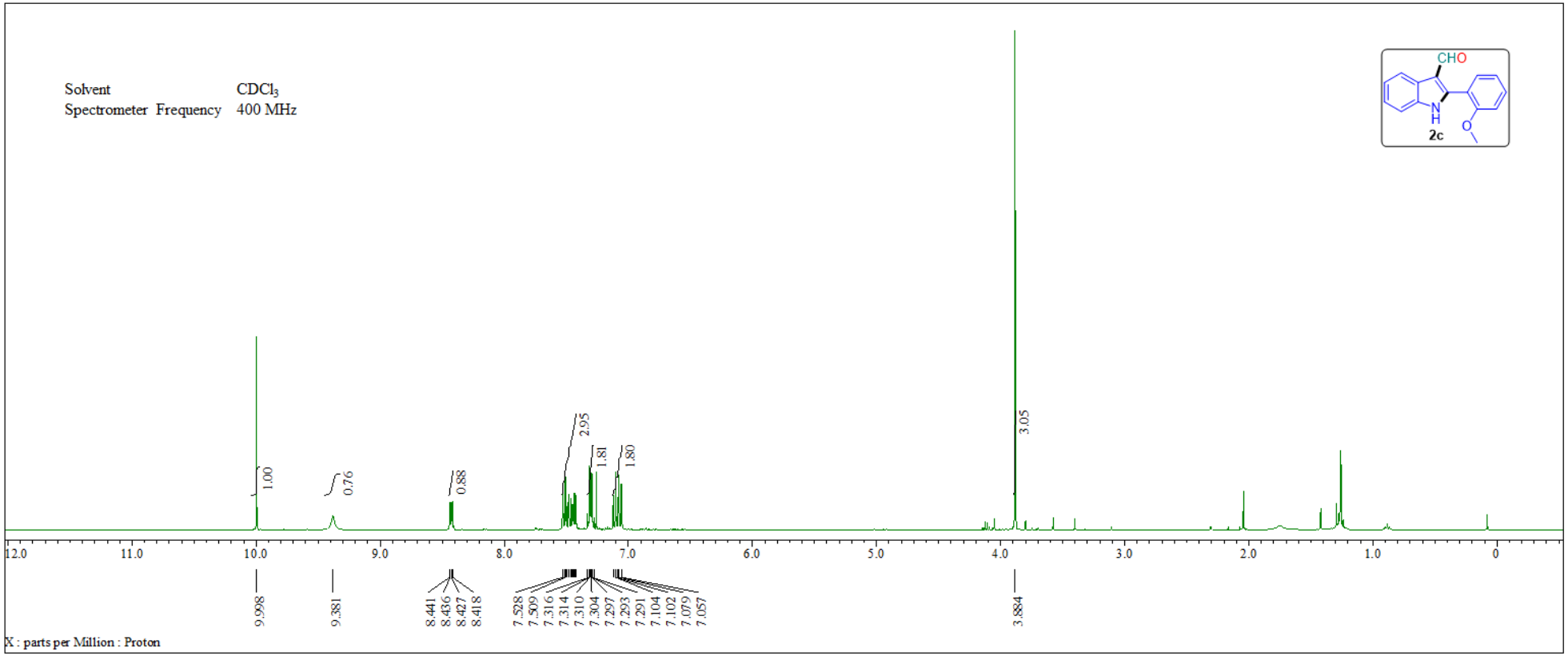

Solvent CDCl3
Spectrometer Frequency 100 MHz
5f
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
148.263
132.426
131.577
130.137
128.621
128.288
123.711
116.026
110.353
107.560
95.269
86.459
51.429
33.380
26.096
24.980
X : parts per Million : Carbon13

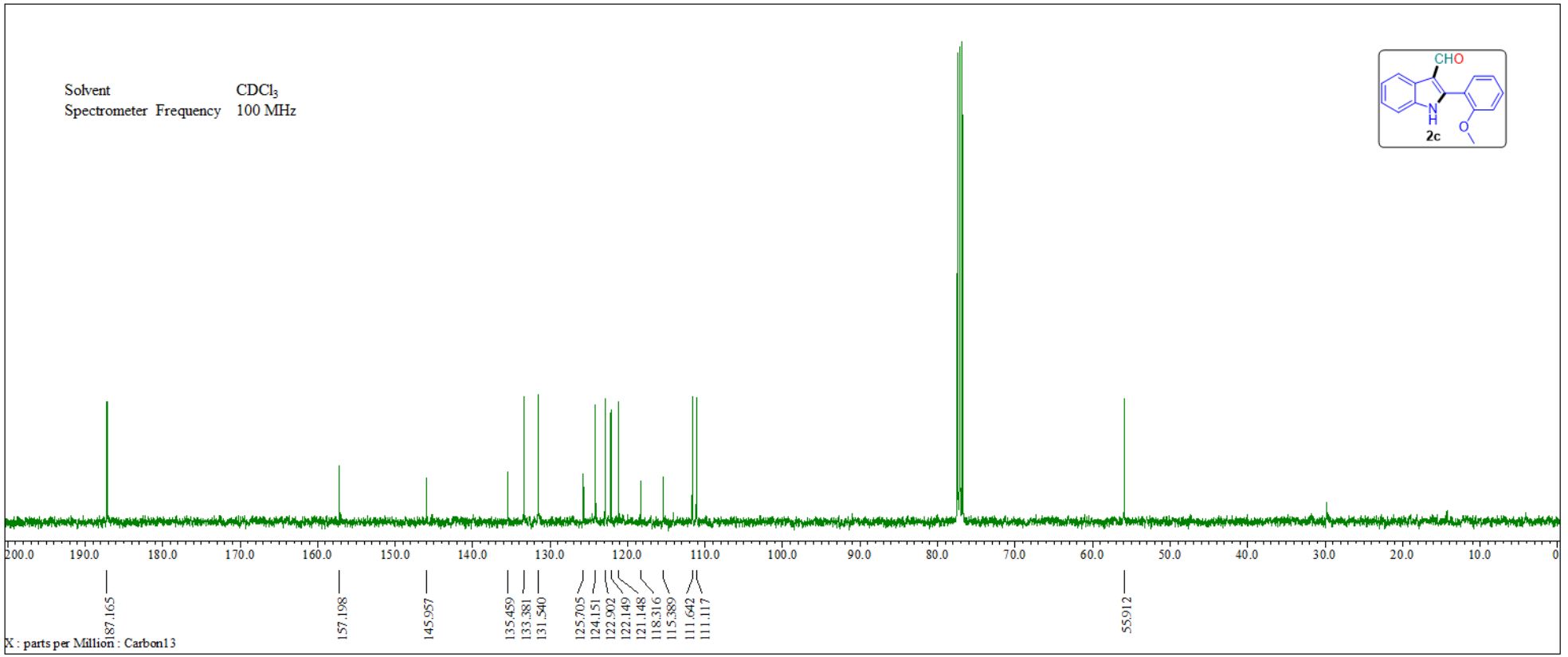

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 400 MHz
CHO
N
H
2a
0.80
1.00
0.97
2.07
4.16
1.95
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.194
9.989
8.324
8.312
8.302
7.635
7.629
7.615
7.611
7.472
7.466
7.463
7.456
7.452
7.230
7.219
7.207
X : parts per Million : Proton

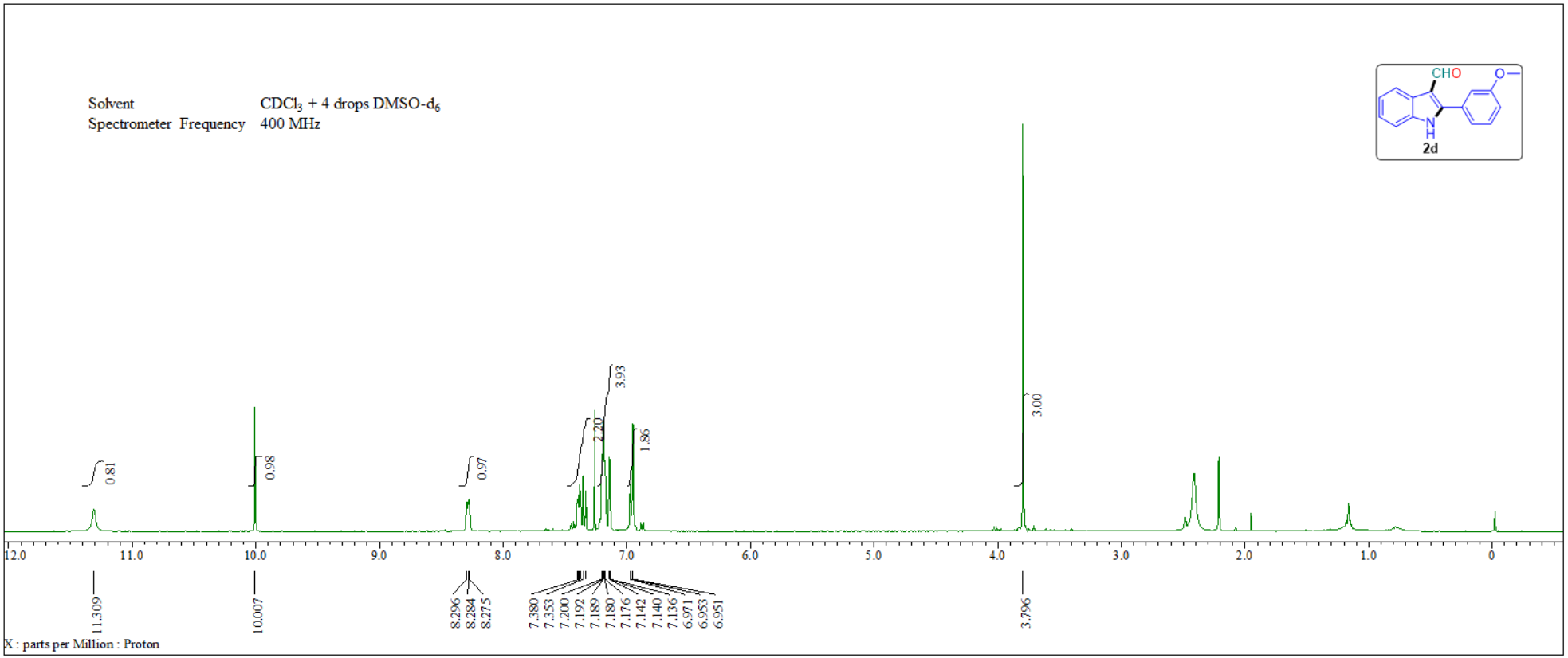

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 100 MHz
CHO
N
H
2a
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.763
149.645
136.173
130.480
129.985
129.746
128.888
126.209
123.911
122.786
121.976
114.548
111.726
X : parts per Million : Carbon13

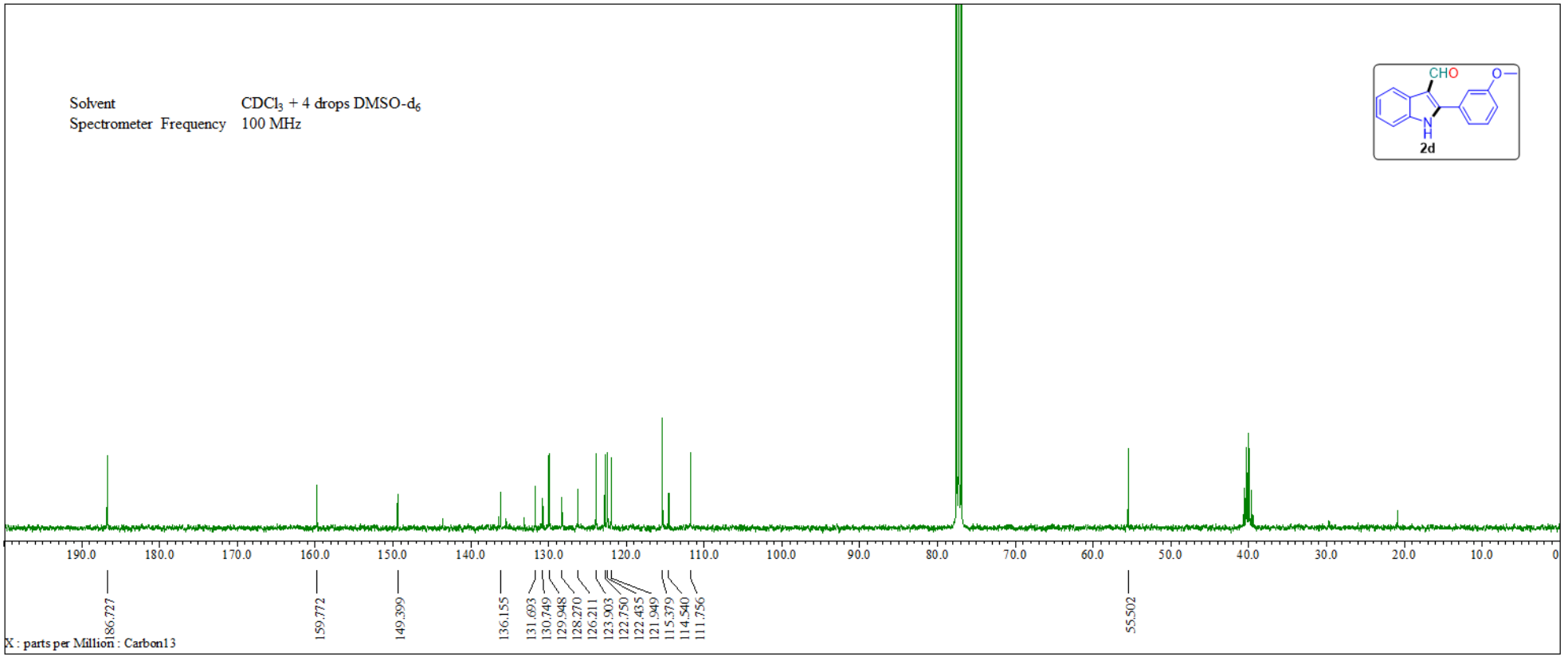

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 400 MHz
CHO
N
H
2b
0.89
1.00
0.98
1.98
1.02
2.70
2.67
3.02
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.268
10.075
8.402
8.397
8.389
8.379
7.615
7.595
7.495
7.484
7.367
7.350
7.308
7.302
7.296
7.294
7.289
7.284
2.471
X : parts per Million : Proton

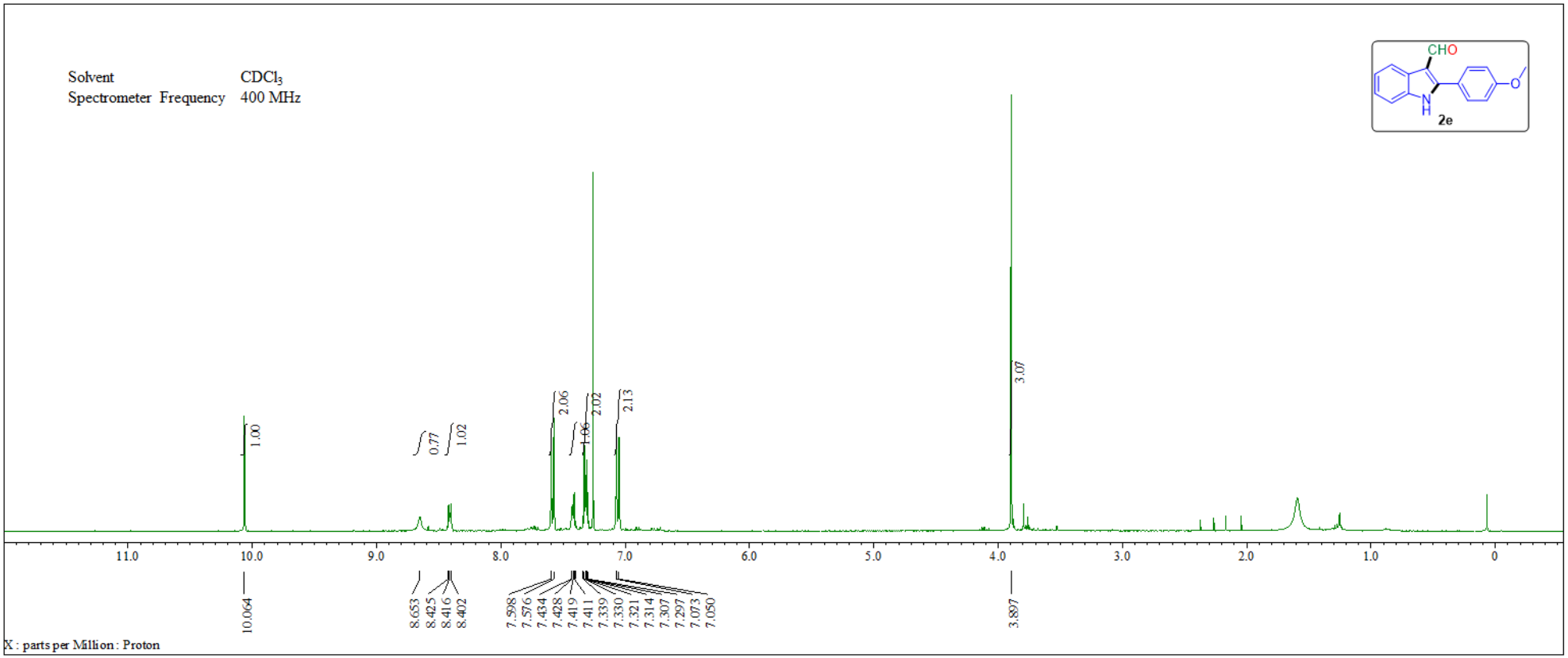

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 100 MHz
CHO
N
H
2b
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.765
149.923
139.979
136.136
129.824
129.595
127.545
126.277
123.770
122.683
121.872
114.330
111.670
21.426
X : parts per Million : Carbon13

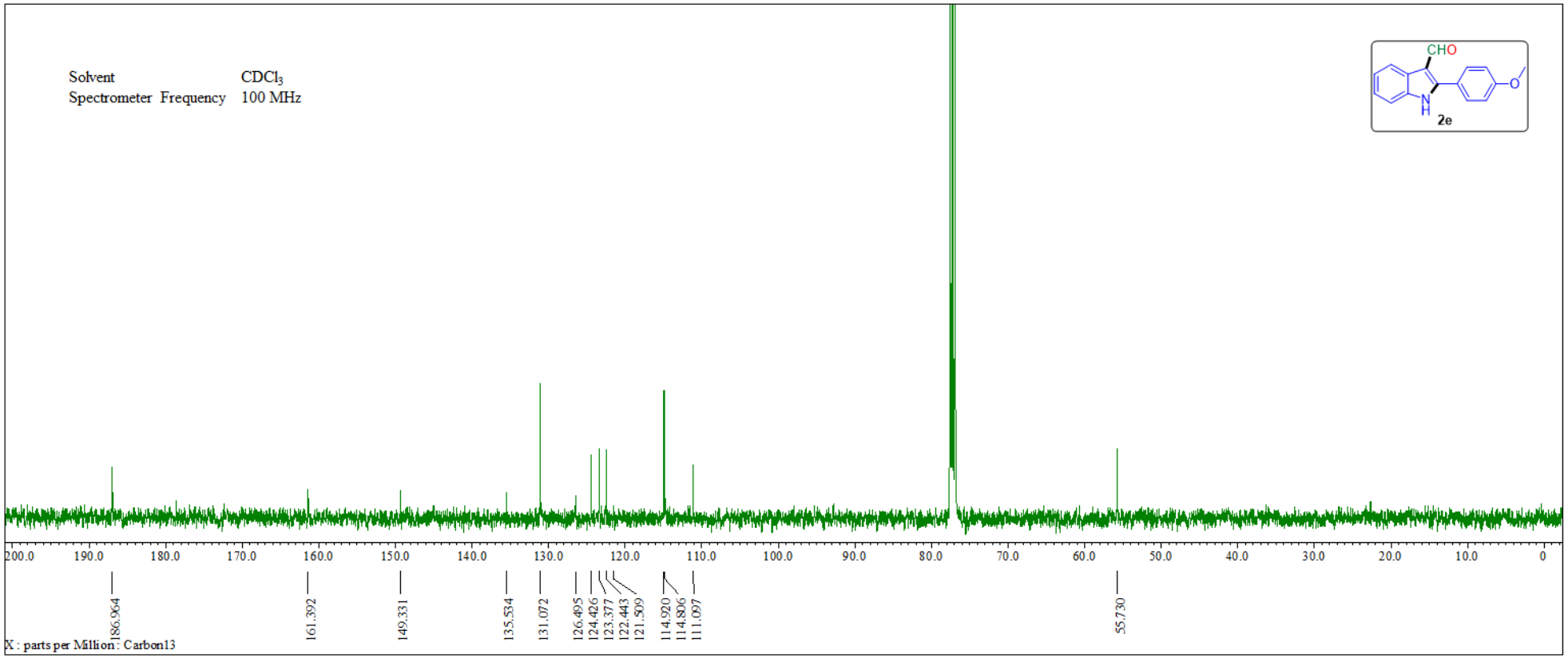

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
H
O
2c
1.00
0.76
0.88
2.95
1.81
1.80
3.05
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.998
9.381
8.441
8.436
8.427
8.418
7.528
7.509
7.316
7.314
7.310
7.304
7.297
7.293
7.291
7.104
7.102
7.079
7.057
3.884
X : parts per Million : Proton

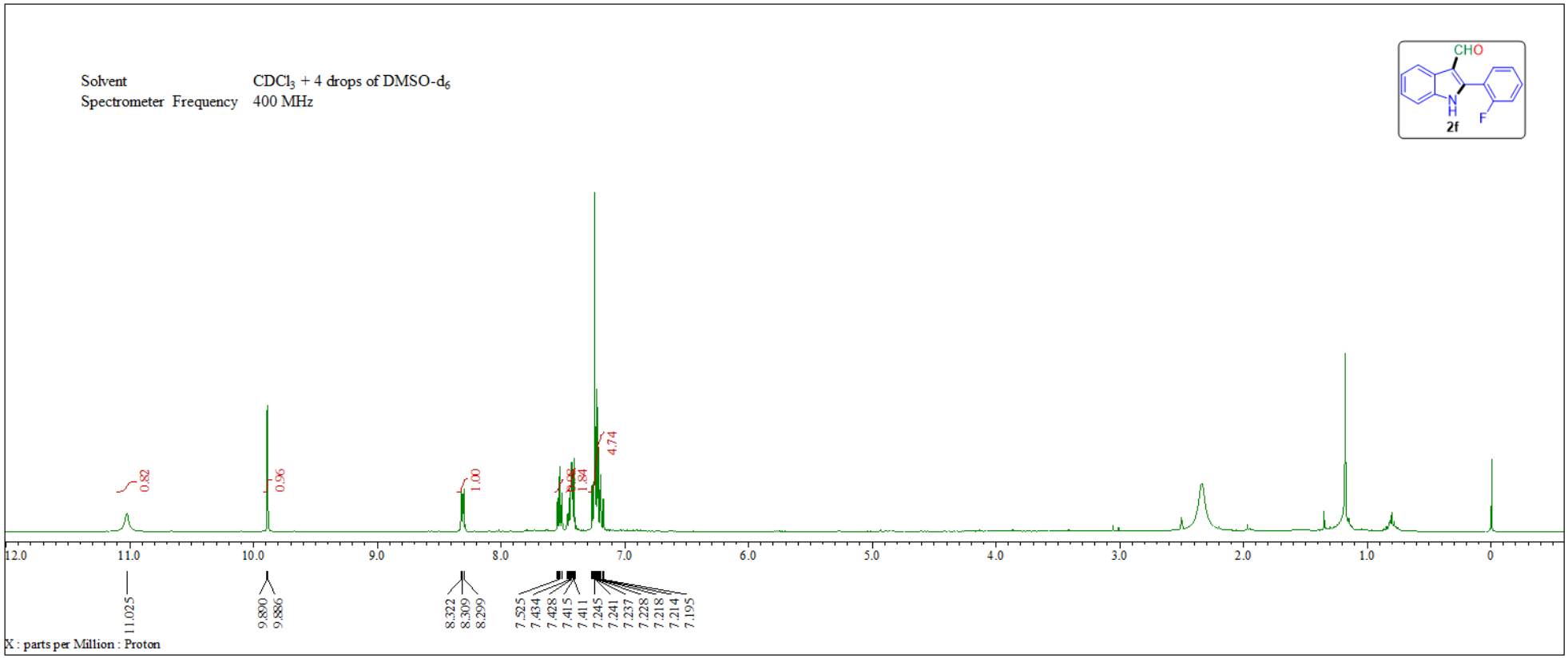

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
H
O
2c
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
187.165
157.198
145.957
135.459
133.381
131.540
125.705
124.151
122.902
122.149
121.148
118.316
115.389
111.642
111.117
55.912
X : parts per Million : Carbon13

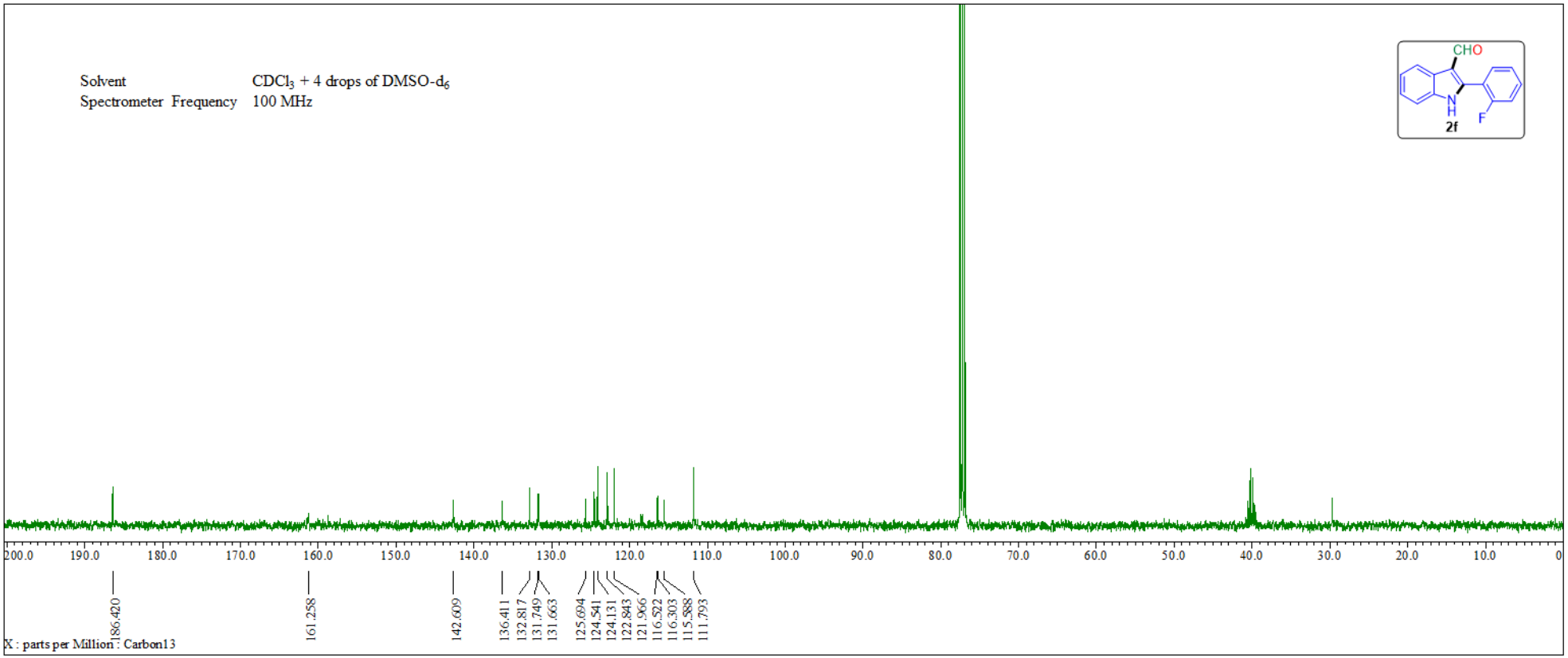

Solvent CDCl3 + 4 drops DMSO-d6
Spectrometer Frequency 400 MHz
CHO
O
N
H
2d
0.81
0.98
0.97
2.20
3.93
1.86
3.00
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.309
10.007
8.296
8.284
8.275
7.380
7.353
7.200
7.192
7.189
7.180
7.176
7.142
7.140
7.136
6.971
6.953
6.951
3.796
X : parts per Million : Proton

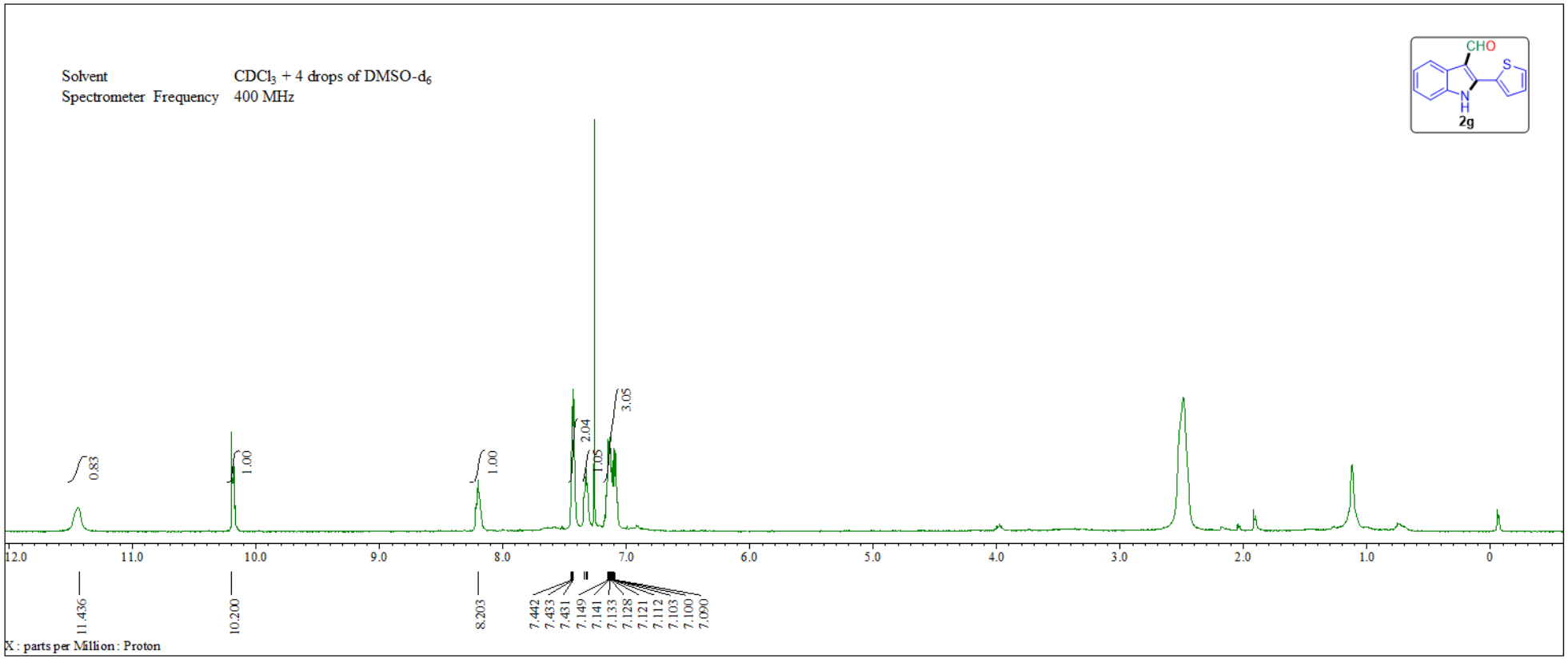

Solvent CDCl3 + 4 drops DMSO-d6
Spectrometer Frequency 100 MHz
CHO
O
N
H
2d
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
186.727
159.772
149.399
136.155
131.693
130.749
129.948
128.270
126.211
123.903
122.750
122.435
121.949
115.379
114.540
111.756
55.502
X : parts per Million : Carbon13

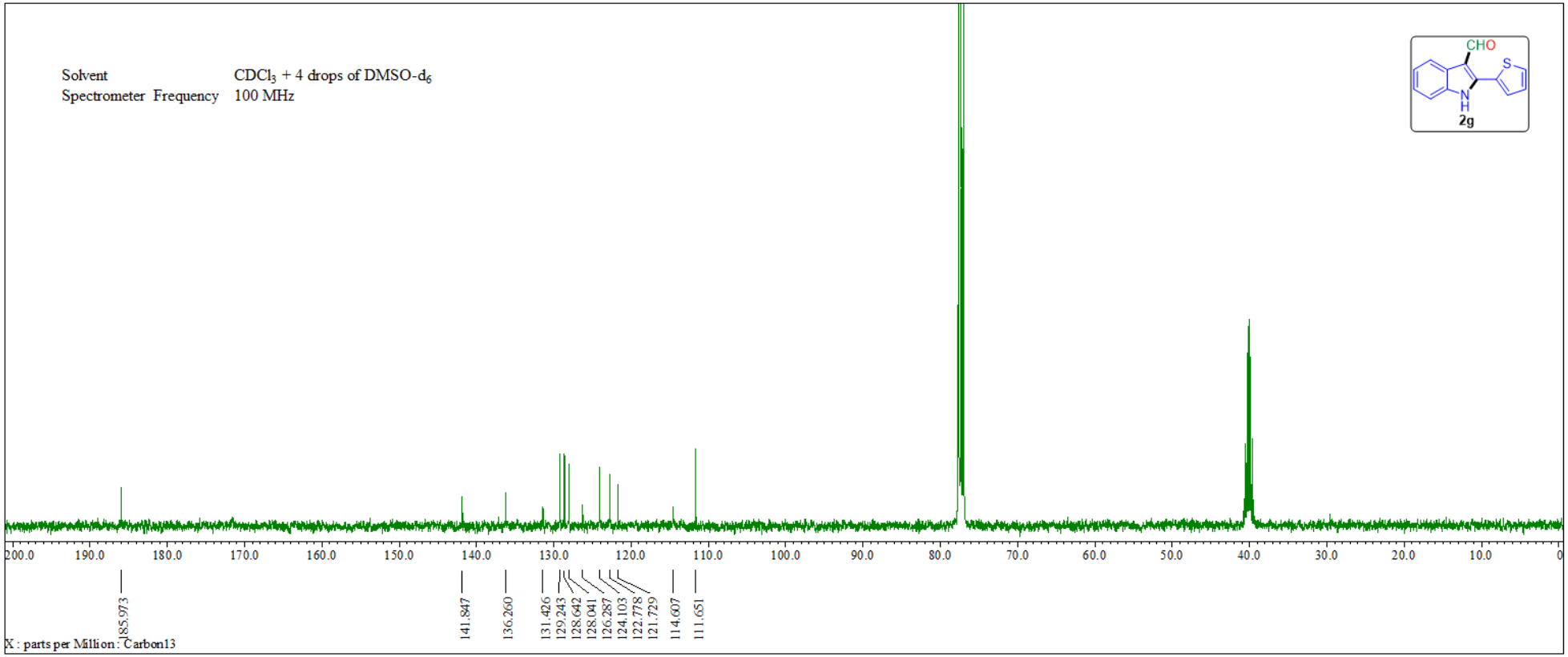

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
O
N
H
2e
1.00
0.77
1.02
2.06
1.06
2.02
2.13
3.07
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.064
8.653
8.425
8.416
8.402
7.598
7.576
7.434
7.428
7.419
7.411
7.339
7.330
7.321
7.314
7.307
7.297
7.073
7.050
3.897
X : parts per Million : Proton

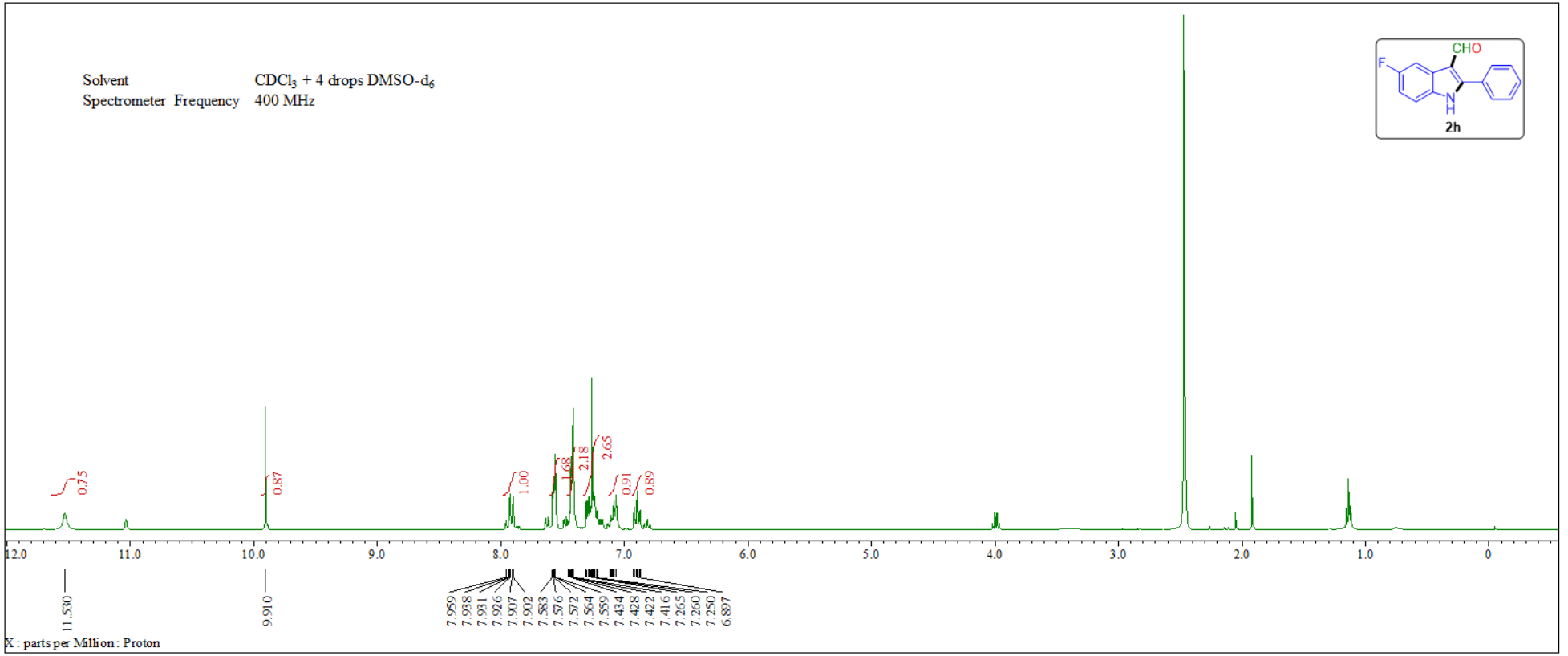

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
O
N
H
2e
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.964
161.392
149.331
135.534
131.072
126.495
124.426
123.377
122.443
121.509
114.920
114.806
111.097
55.730
X : parts per Million : Carbon13

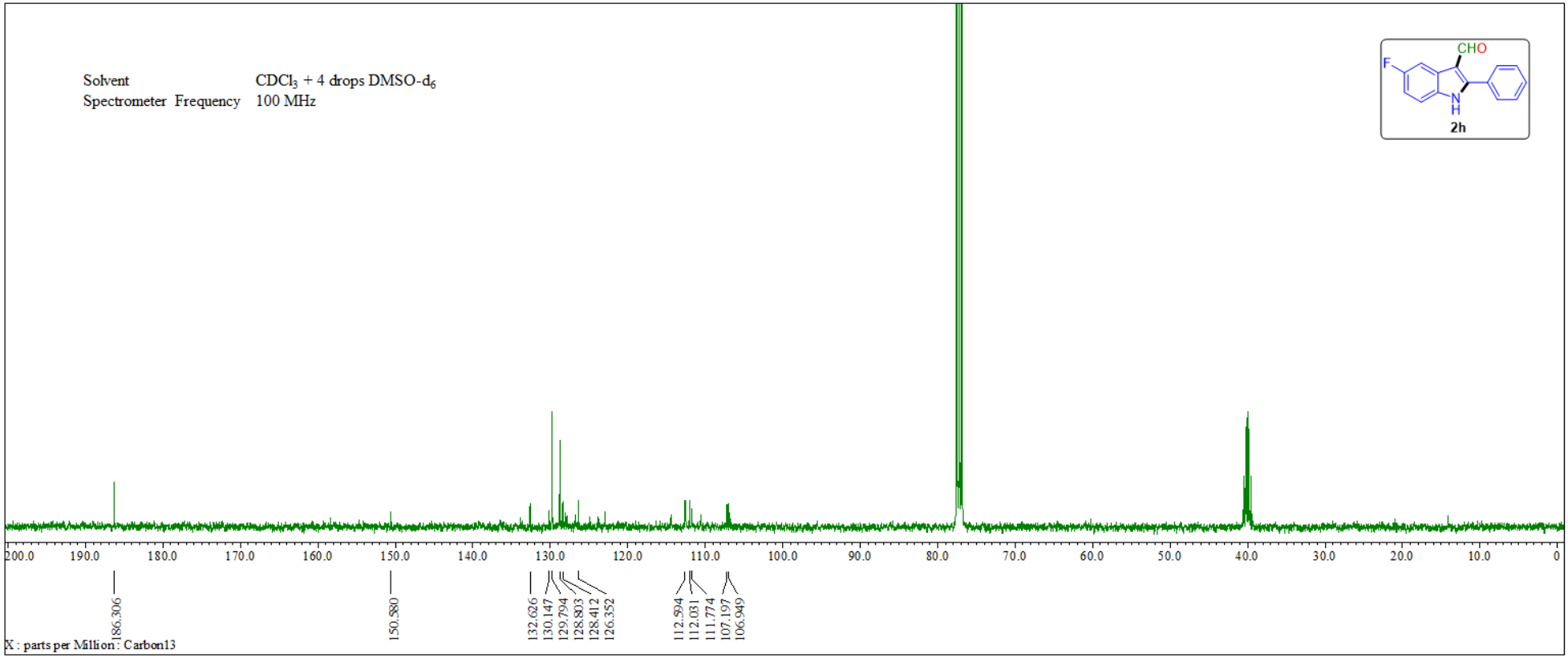

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 400 MHz
CHO
N
H
F
2f
0.82
0.96
1.00
1.84
4.74
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.025
9.890
9.886
8.322
8.309
8.299
7.525
7.434
7.428
7.415
7.411
7.245
7.241
7.237
7.228
7.218
7.214
7.195
X : parts per Million : Proton

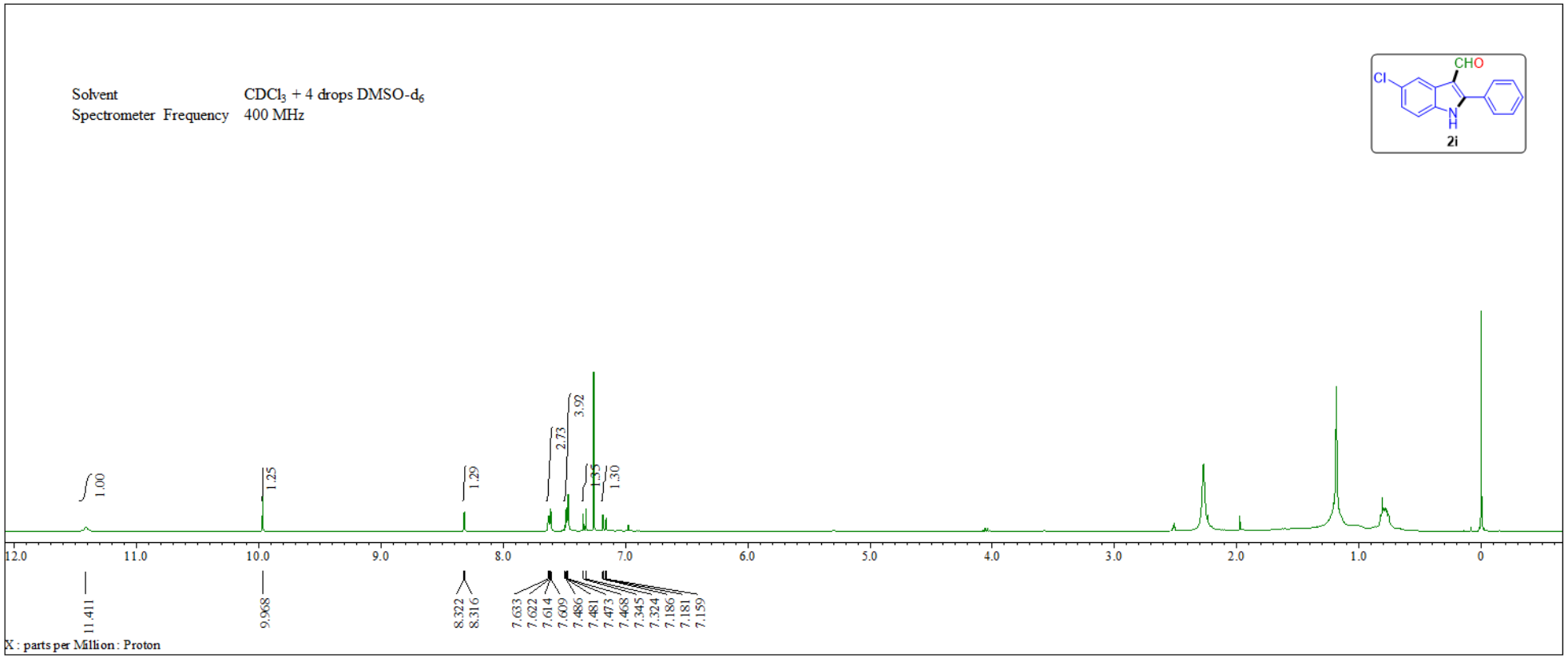

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 100 MHz
CHO
N
H
F
2f
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.420
161.258
142.609
136.411
132.817
131.749
131.663
125.694
124.541
124.131
122.843
121.966
116.522
116.303
115.588
111.793
X : parts per Million : Carbon13

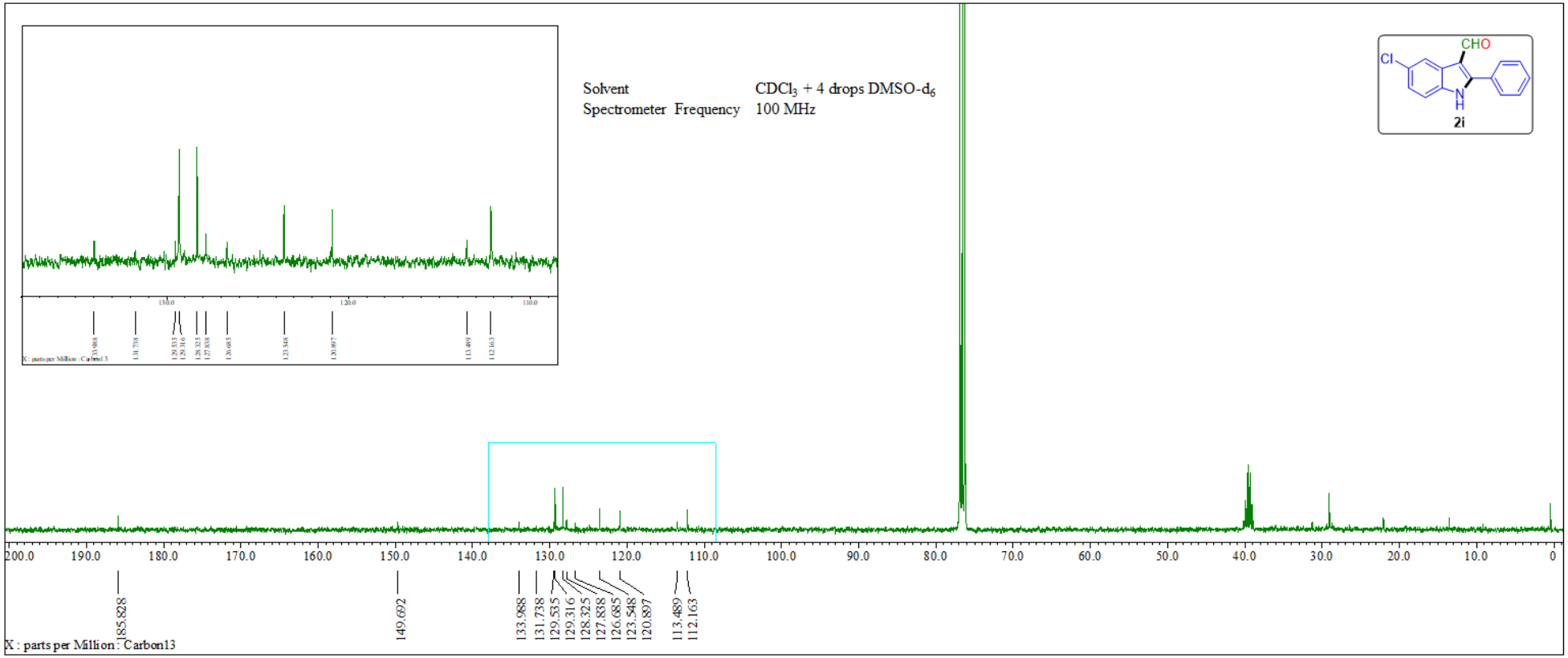

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 400 MHz
CHO
S
N
H
2g
0.83
1.00
1.00
2.04
1.05
3.05
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.436
10.200
8.203
7.442
7.433
7.431
7.149
7.141
7.133
7.128
7.121
7.112
7.103
7.100
7.090
X : parts per Million : Proton

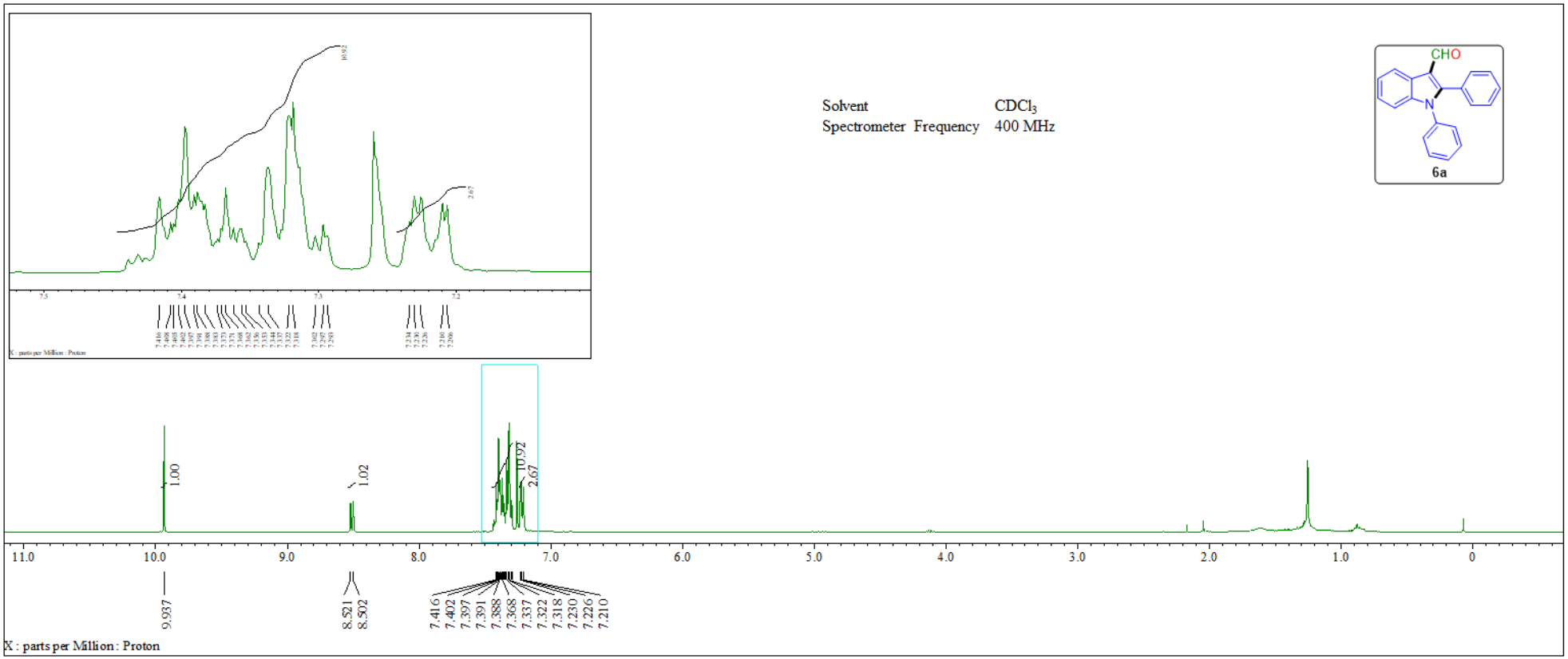

Solvent CDCl3 + 4 drops of DMSO-d6
Spectrometer Frequency 100 MHz
CHO
S
N
H
2g
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
185.973
141.847
136.260
131.426
129.243
128.642
128.041
126.287
124.103
122.778
121.729
114.607
111.651
X : parts per Million : Carbon13

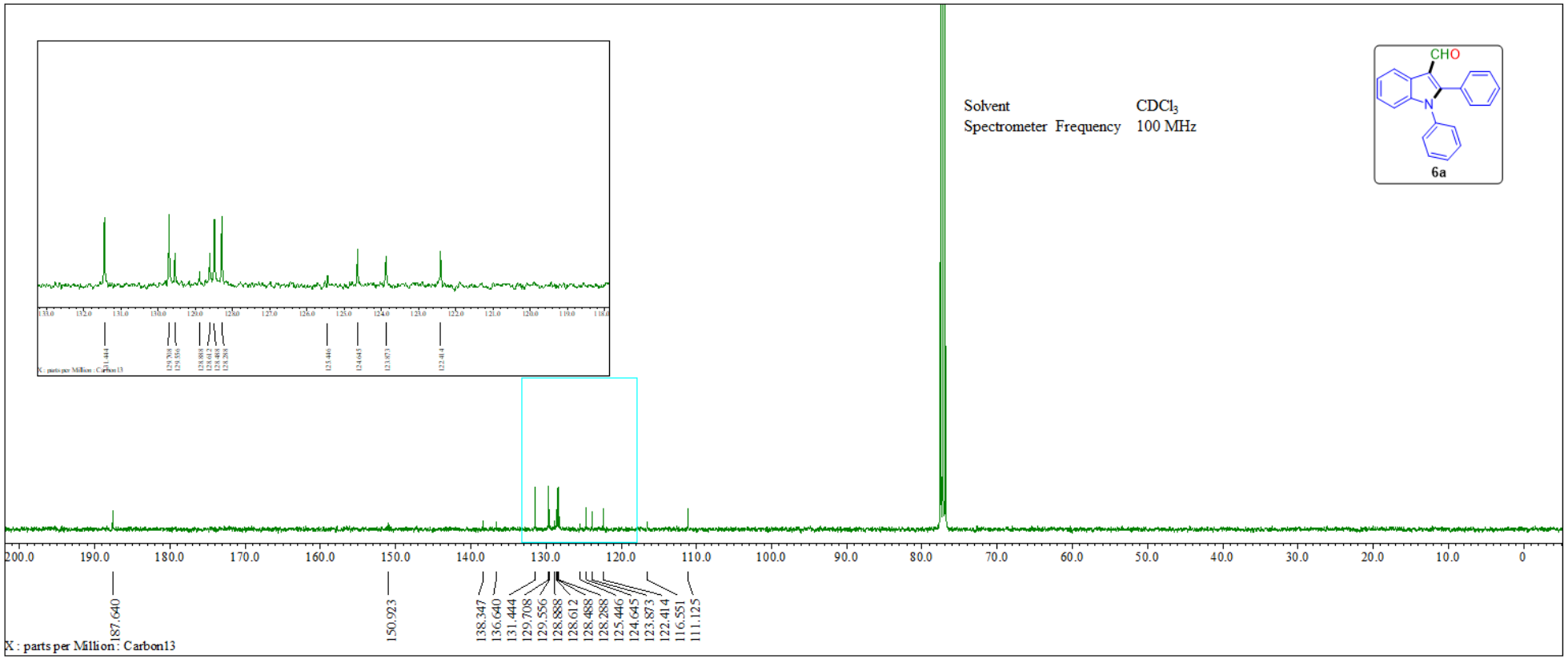

Solvent CDCl3 + 4 drops DMSO-d6
Spectrometer Frequency 400 MHz
CHO
F
N
H
2h
0.75
0.87
1.00
1.68
2.18
2.65
0.91
0.89
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.530
9.910
7.959
7.938
7.931
7.926
7.907
7.902
7.583
7.576
7.572
7.564
7.559
7.434
7.428
7.422
7.416
7.265
7.260
7.250
6.897
X : parts per Million : Proton

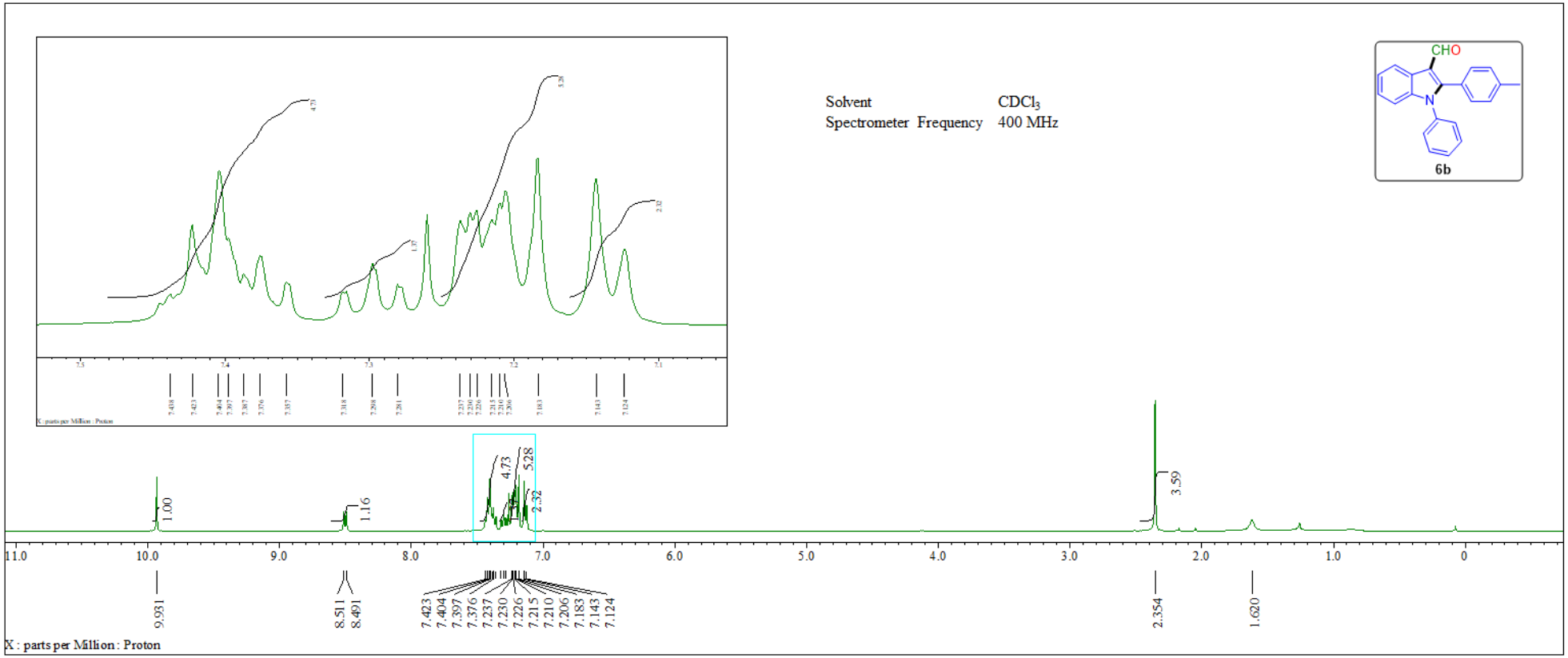

Solvent CDCl3 + 4 drops DMSO-d6
Spectrometer Frequency 100 MHz
CHO
F
N
H
2h
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.306
150.380
132.626
130.147
129.794
128.803
128.412
126.352
112.594
112.031
111.774
107.197
106.949
X : parts per Million : Carbon13

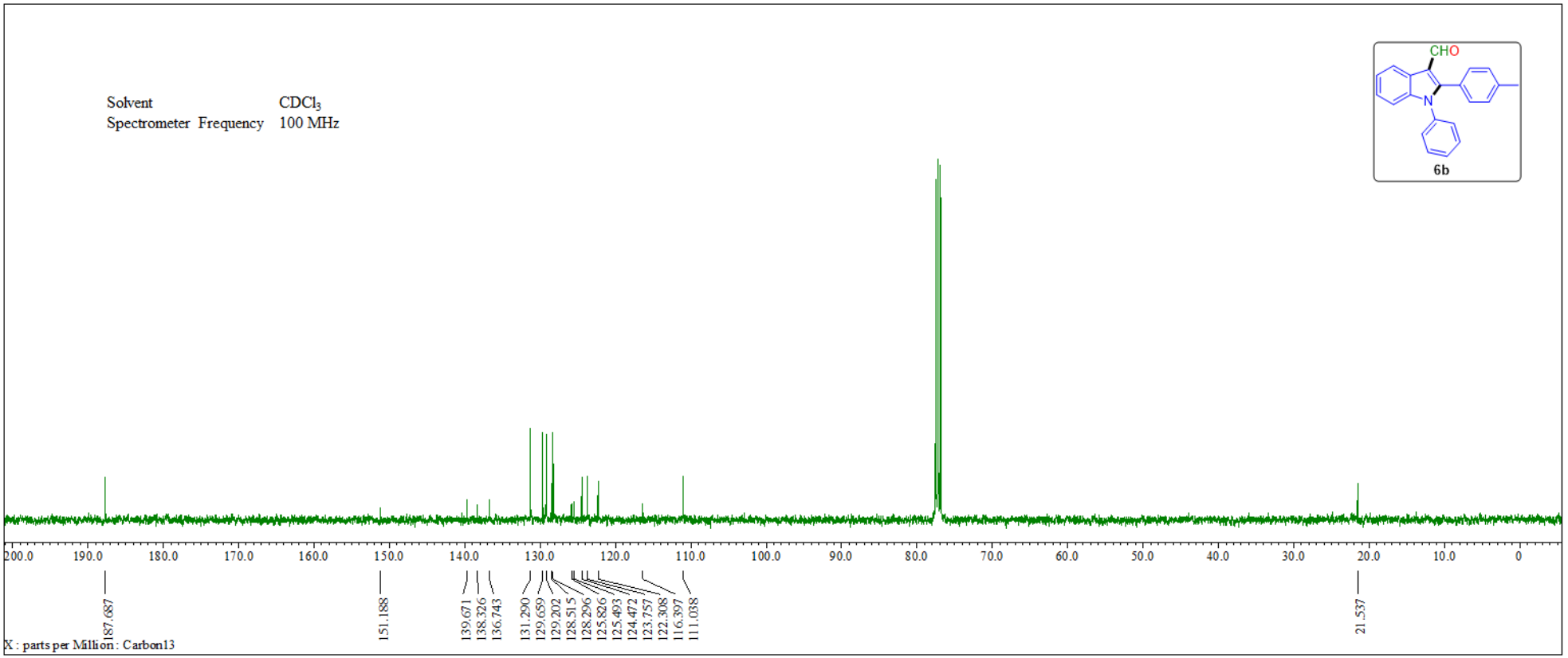

Solvent CDCl3 + 4 drops DMSO-d6
Spectrometer Frequency 400 MHz
CHO
Cl
N
H
2i
1.00
1.25
1.29
2.73
3.92
1.35
1.30
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
11.411
9.968
8.322
8.316
7.633
7.622
7.614
7.609
7.486
7.481
7.473
7.468
7.345
7.324
7.186
7.181
7.159
X : parts per Million : Proton

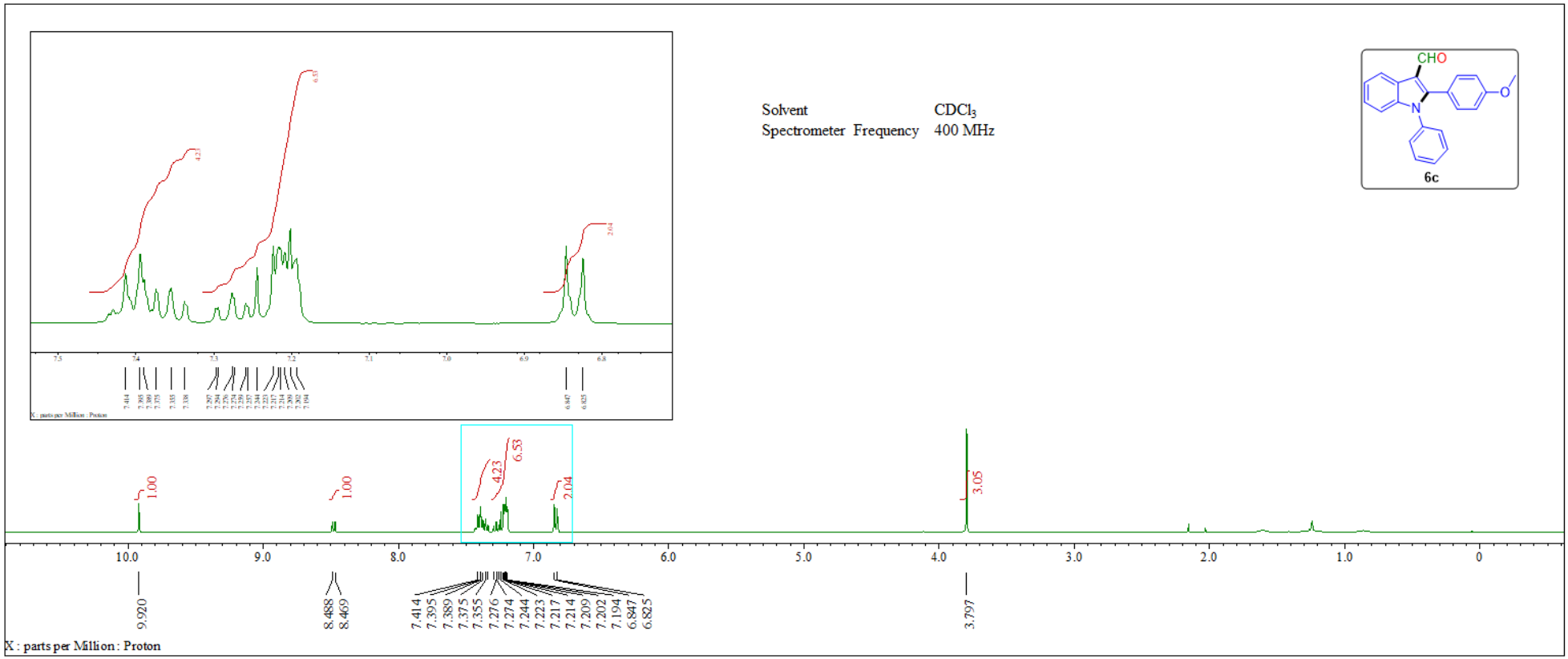

Solvent CDCl3 + 4 drops DMSO-d6
Spectrometer Frequency 100 MHz
CHO
Cl
N
H
2i
185.828
149.692
133.988
131.738
129.535
129.316
128.325
127.838
126.685
123.548
120.897
113.489
112.163
X : parts per Million : Carbon13

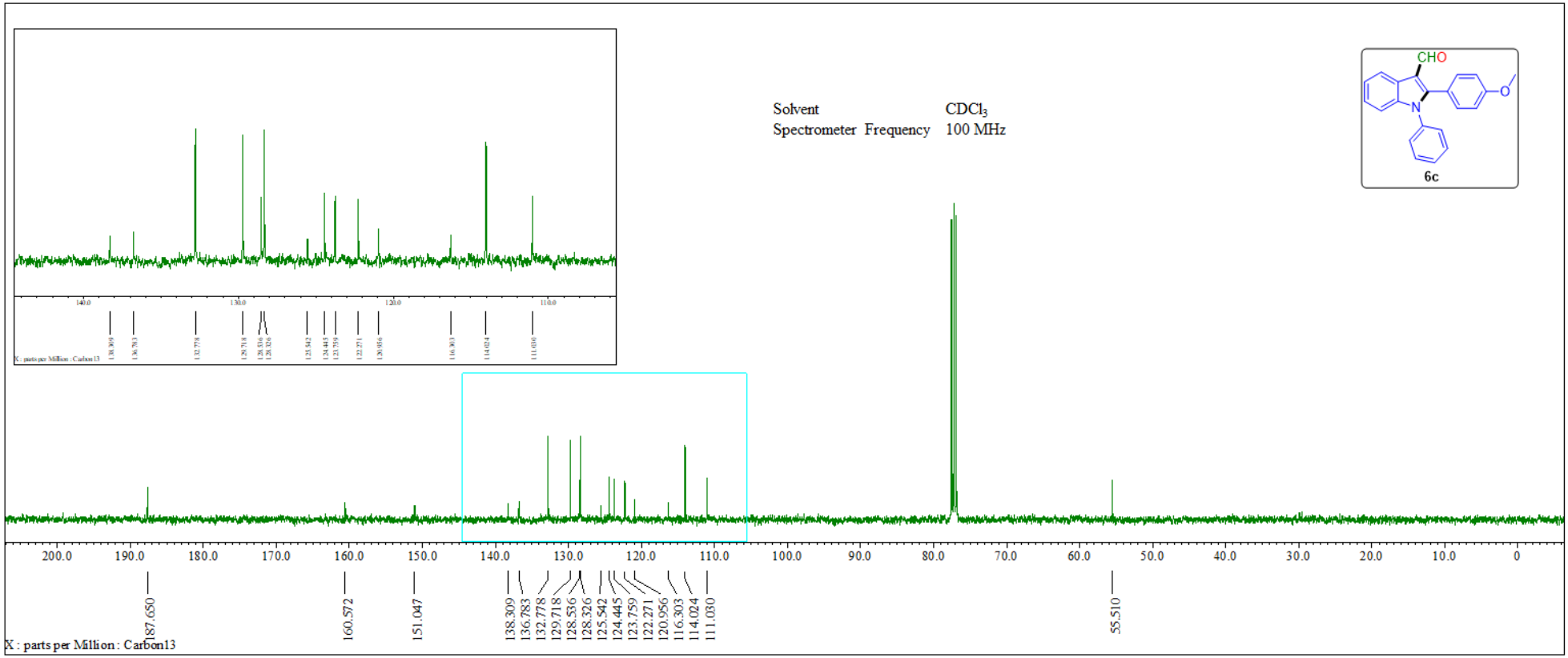

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
6a
1.00
1.02
10.92
2.67
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.937
8.521
8.502
7.416
7.402
7.397
7.391
7.388
7.368
7.337
7.322
7.318
7.230
7.226
7.210
X : parts per Million : Proton

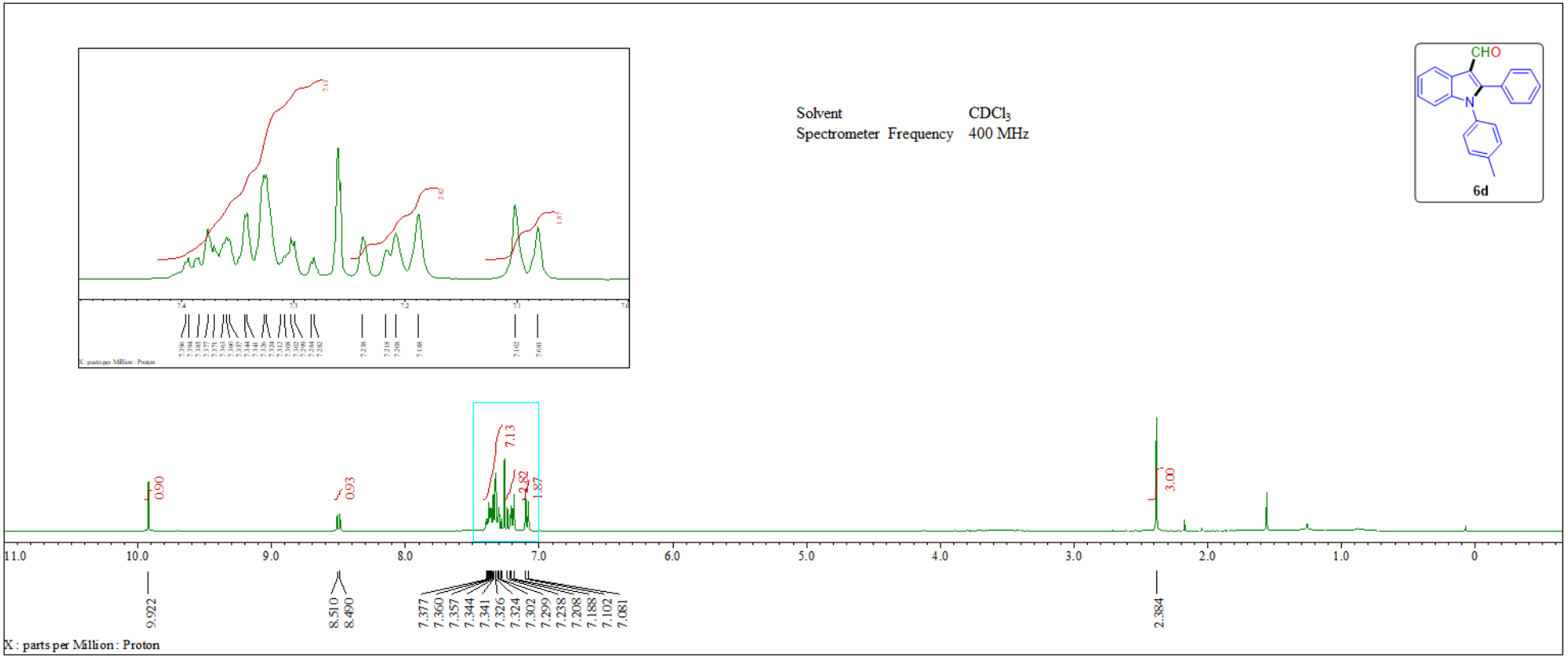

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
6a
187.640
150.923
138.347
136.640
131.444
129.708
129.556
128.888
128.612
128.488
128.288
125.446
124.645
123.873
122.414
116.551
111.125
X : parts per Million : Carbon13

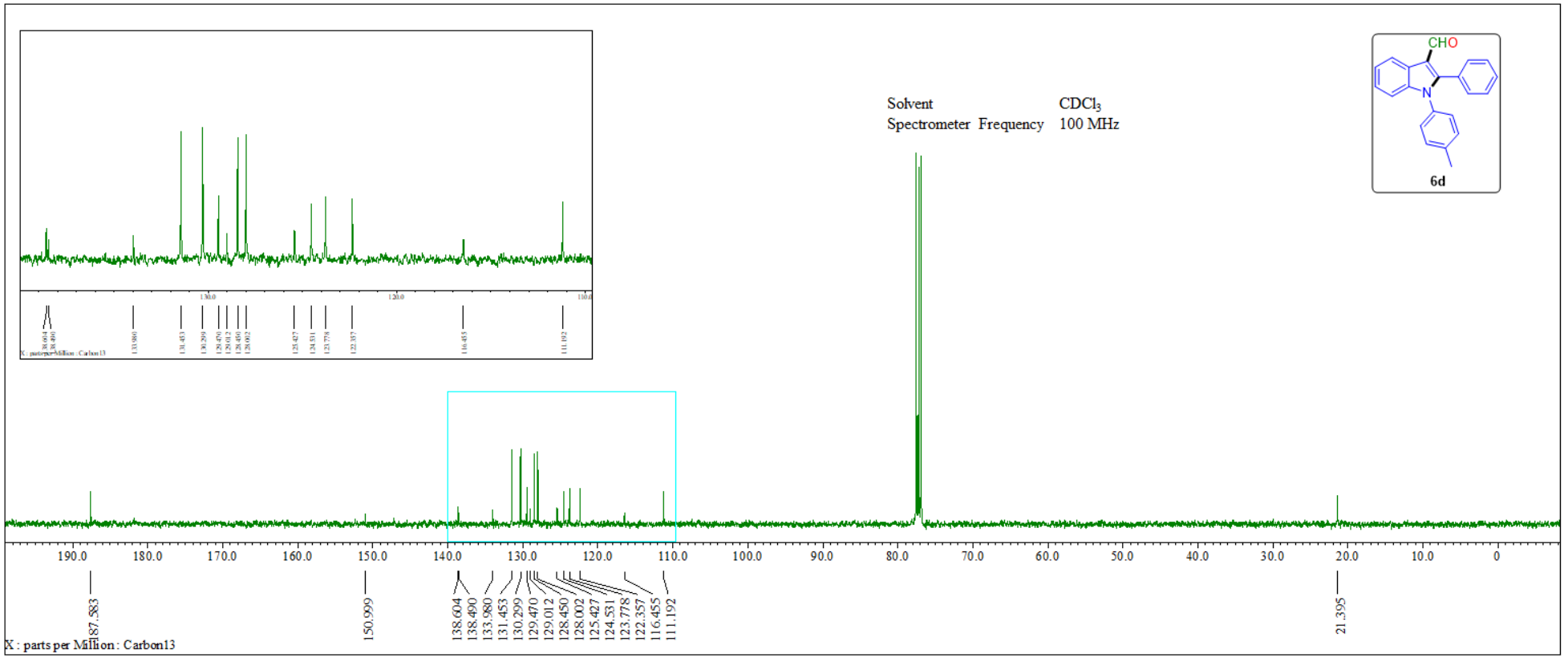

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
6b
1.00
1.16
4.73
5.28
2.32
3.59
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.931
8.511
8.491
7.423
7.404
7.397
7.376
7.237
7.230
7.226
7.215
7.210
7.206
7.183
7.143
7.124
2.354
1.620
X : parts per Million : Proton

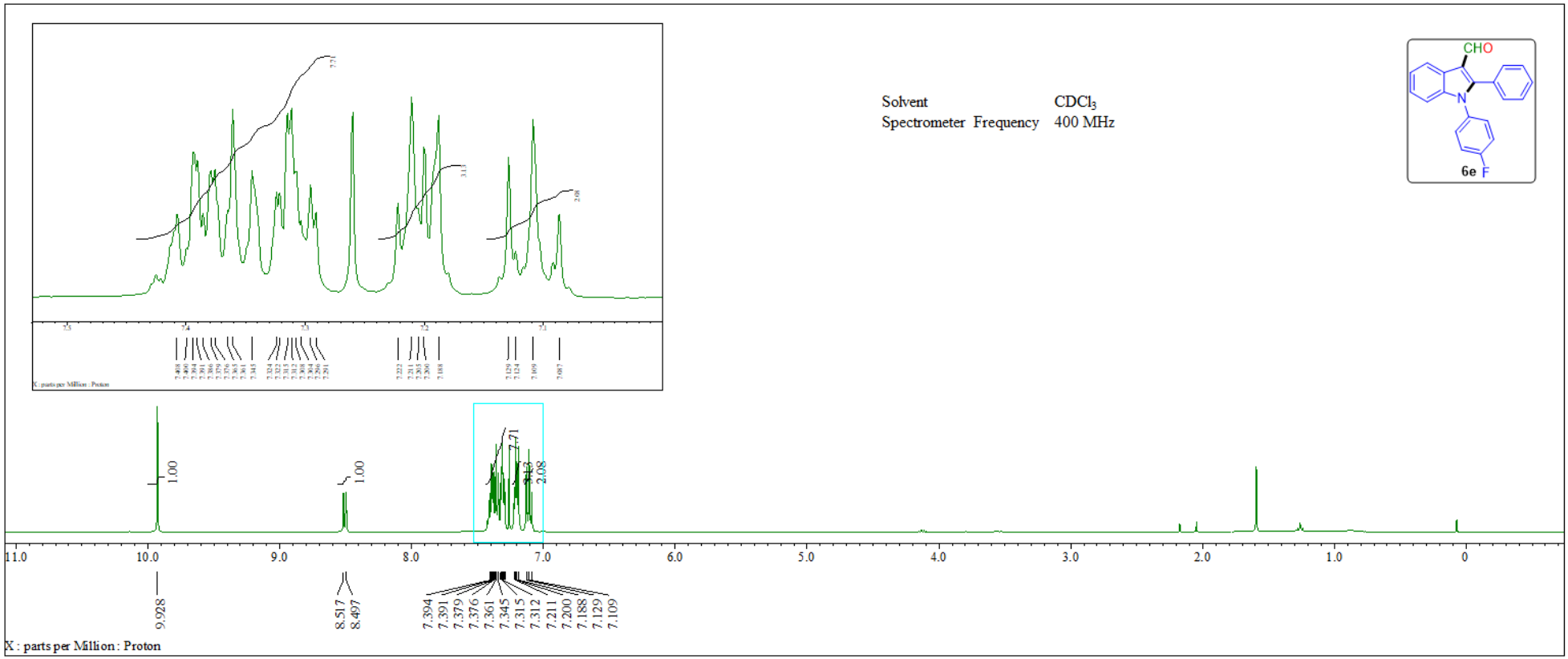

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
6b
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
187.687
151.188
139.671
138.326
136.743
131.290
129.659
129.202
128.515
128.296
125.826
125.493
124.472
123.757
122.308
116.397
111.038
21.537
X : parts per Million : Carbon13

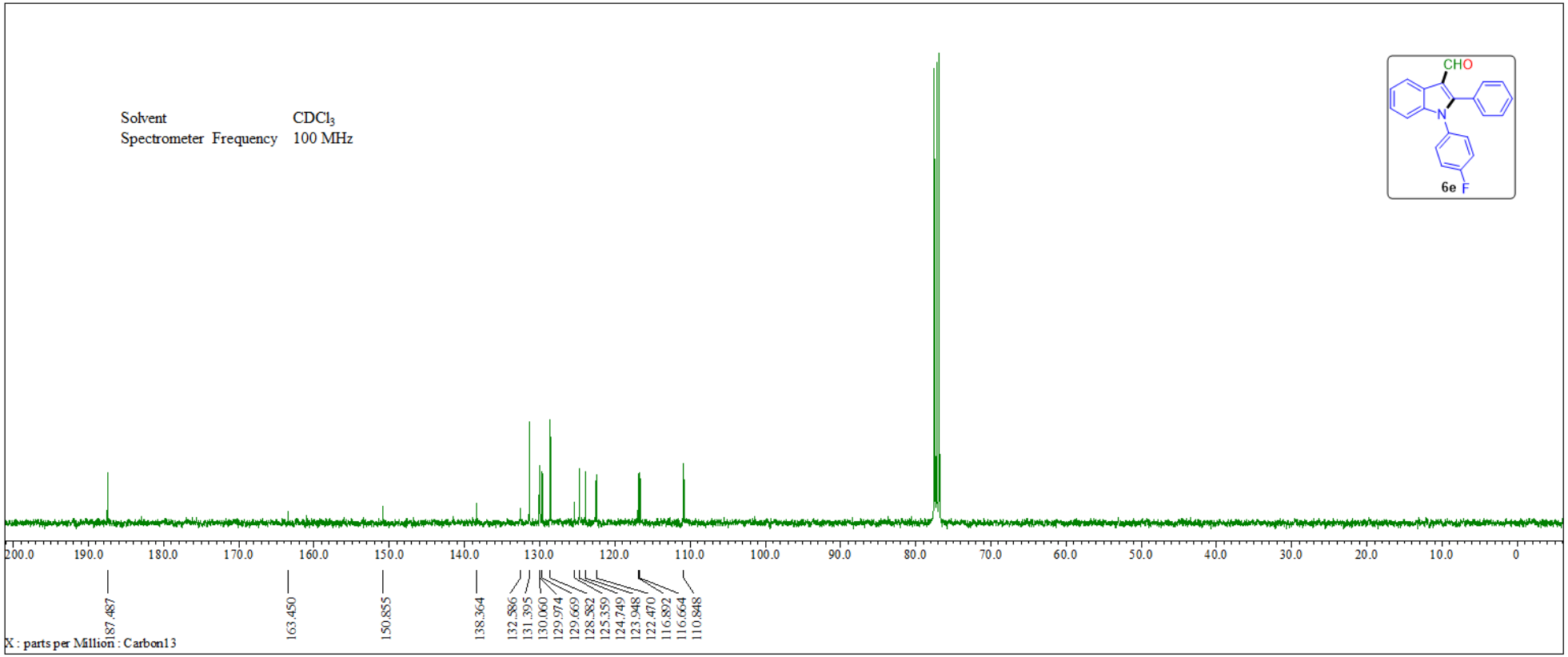

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
O
N
6c
1.00
1.00
4.23
6.53
2.04
3.05
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.920
8.488
8.469
7.414
7.395
7.389
7.375
7.355
7.276
7.274
7.244
7.223
7.217
7.214
7.209
7.202
7.194
6.847
6.825
3.797
X : parts per Million : Proton

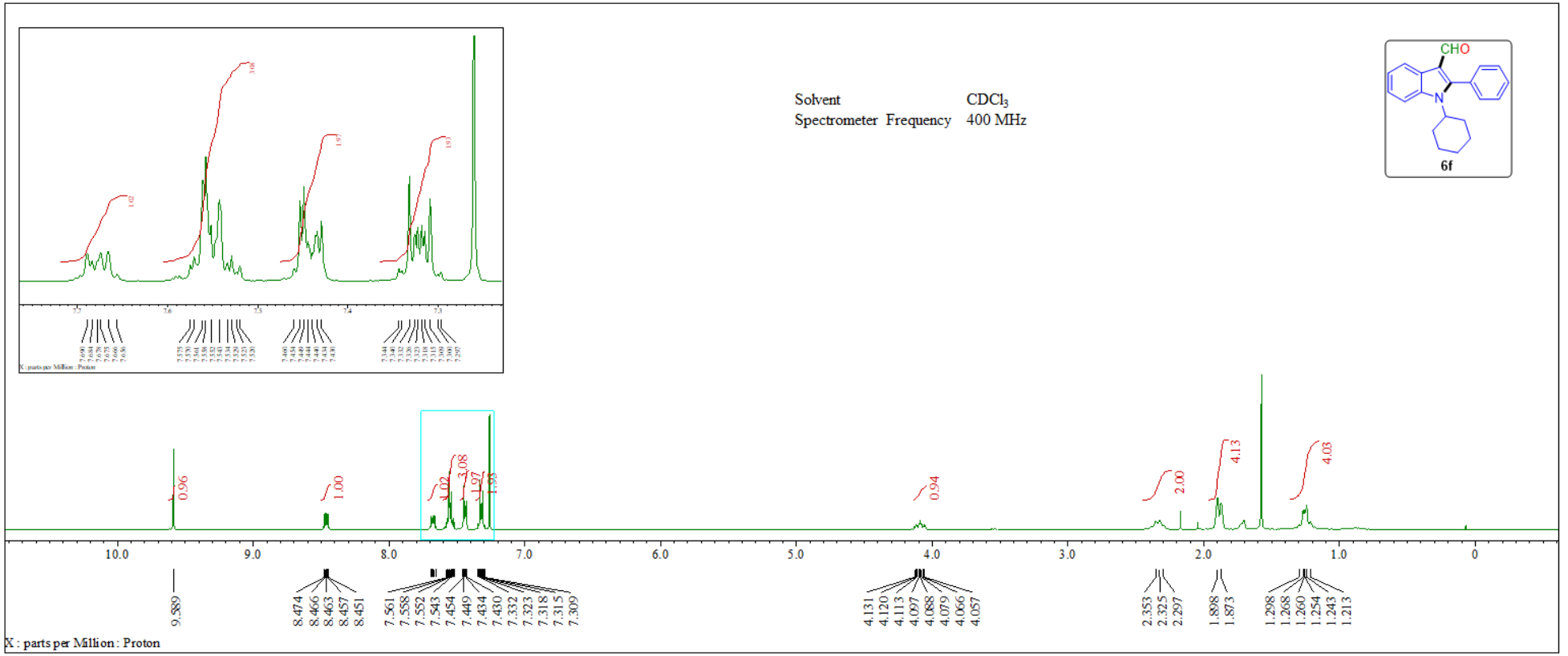

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
O
N
6c
X : parts per Million : Carbon13
187.650
160.572
151.047
138.309
136.783
132.778
129.718
128.536
128.326
125.542
124.445
123.759
122.271
120.956
116.303
114.024
111.030
55.510

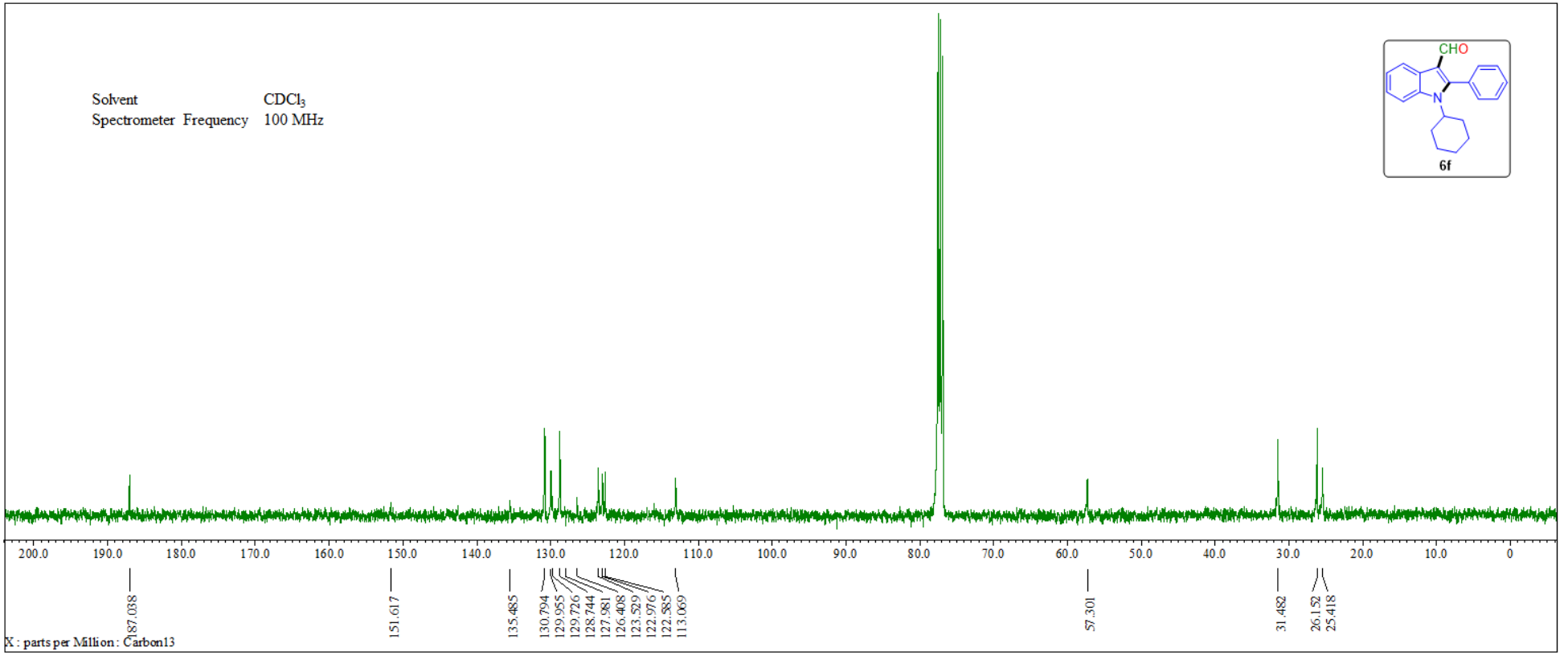

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
6d
0.90
0.93
7.13
2.82
1.87
3.00
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.922
8.510
8.490
7.377
7.360
7.357
7.344
7.341
7.326
7.324
7.302
7.299
7.238
7.208
7.188
7.102
7.081
2.384
X : parts per Million : Proton


CHO
N
6d
Solvent CDCl3
Spectrometer Frequency 100 MHz
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
187.583
150.999
138.604
138.490
133.580
131.453
130.299
129.470
129.012
128.450
128.002
125.427
124.531
123.778
122.357
116.455
111.192
21.395
X : parts per Million : Carbon13

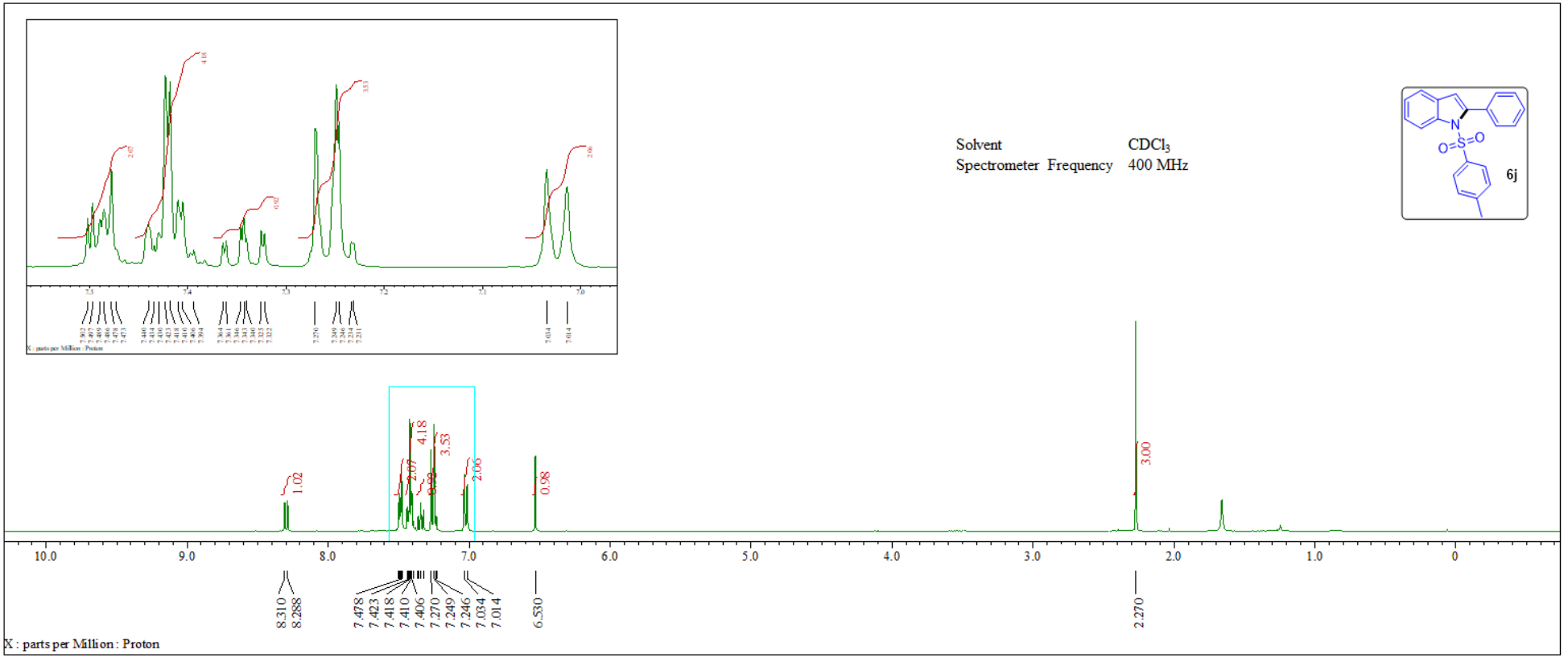

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
6e F
1.00
1.00
3.13
2.08
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.928
8.517
8.497
7.394
7.391
7.379
7.376
7.361
7.345
7.315
7.312
7.211
7.200
7.188
7.129
7.109
X : parts per Million : Proton


Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
6e F
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
187.487
163.450
150.855
138.364
132.586
131.395
130.060
129.974
129.669
128.582
125.359
124.749
123.948
122.470
116.892
116.664
110.848
X : parts per Million : Carbon13

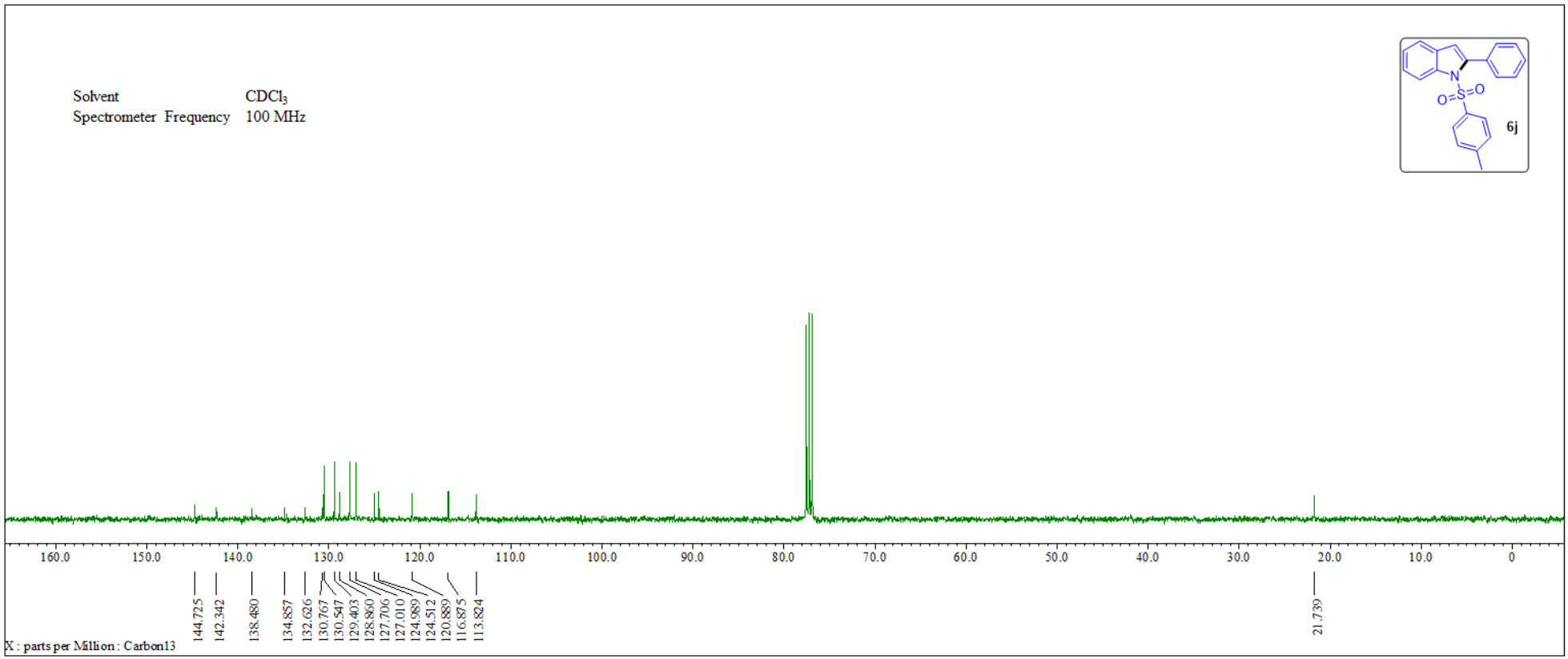

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
6f
0.96
1.00
1.02
3.08
1.97
1.98
0.94
2.00
4.13
4.03
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.589
8.474
8.466
8.463
8.457
8.451
7.561
7.558
7.552
7.543
7.454
7.449
7.434
7.430
7.332
7.323
7.318
7.315
7.309
4.131
4.120
4.113
4.097
4.088
4.079
4.066
4.057
2.353
2.325
2.297
1.898
1.873
1.298
1.268
1.260
1.254
1.243
1.213
X : parts per Million : Proton

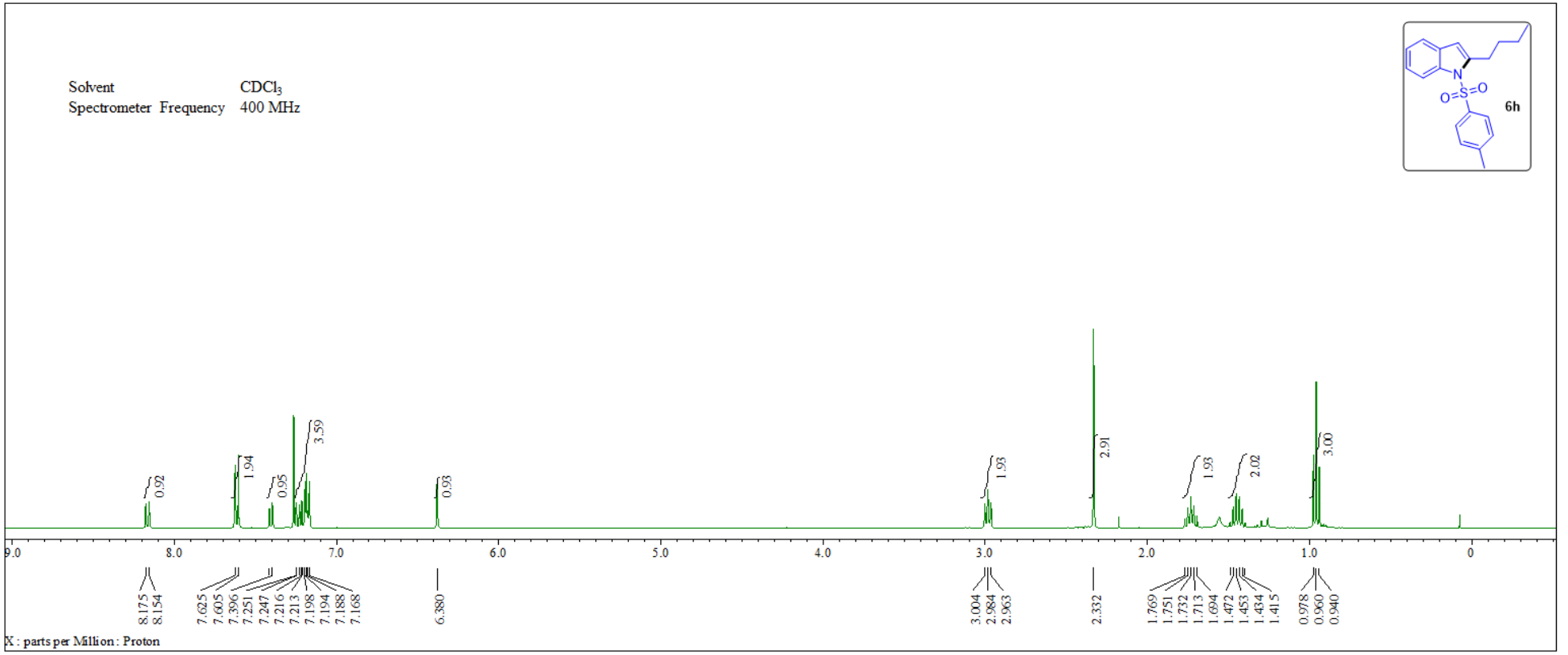

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
6f
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
187.038
151.617
135.485
130.794
129.955
129.726
128.744
127.981
126.408
123.529
122.976
122.585
113.069
57.301
31.482
26.152
25.418
X : parts per Million : Carbon13

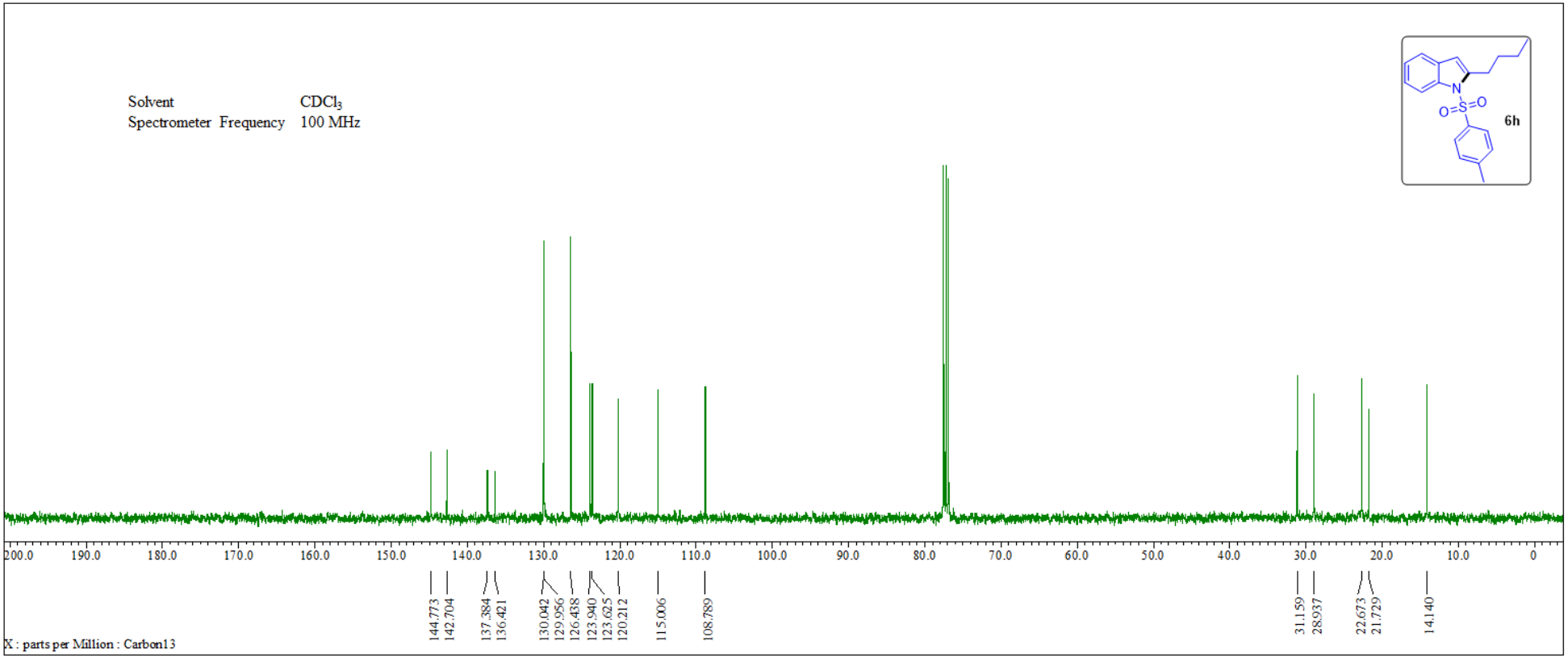
Compound code: **6g**

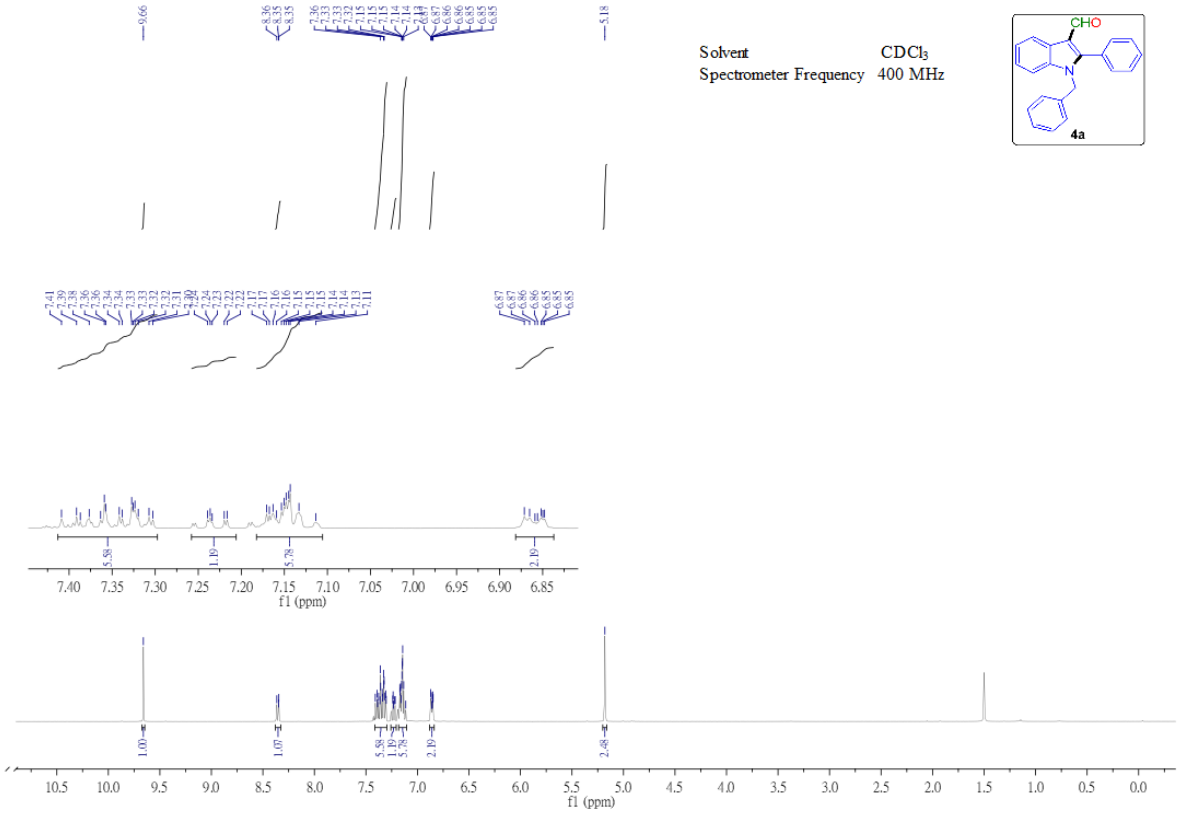


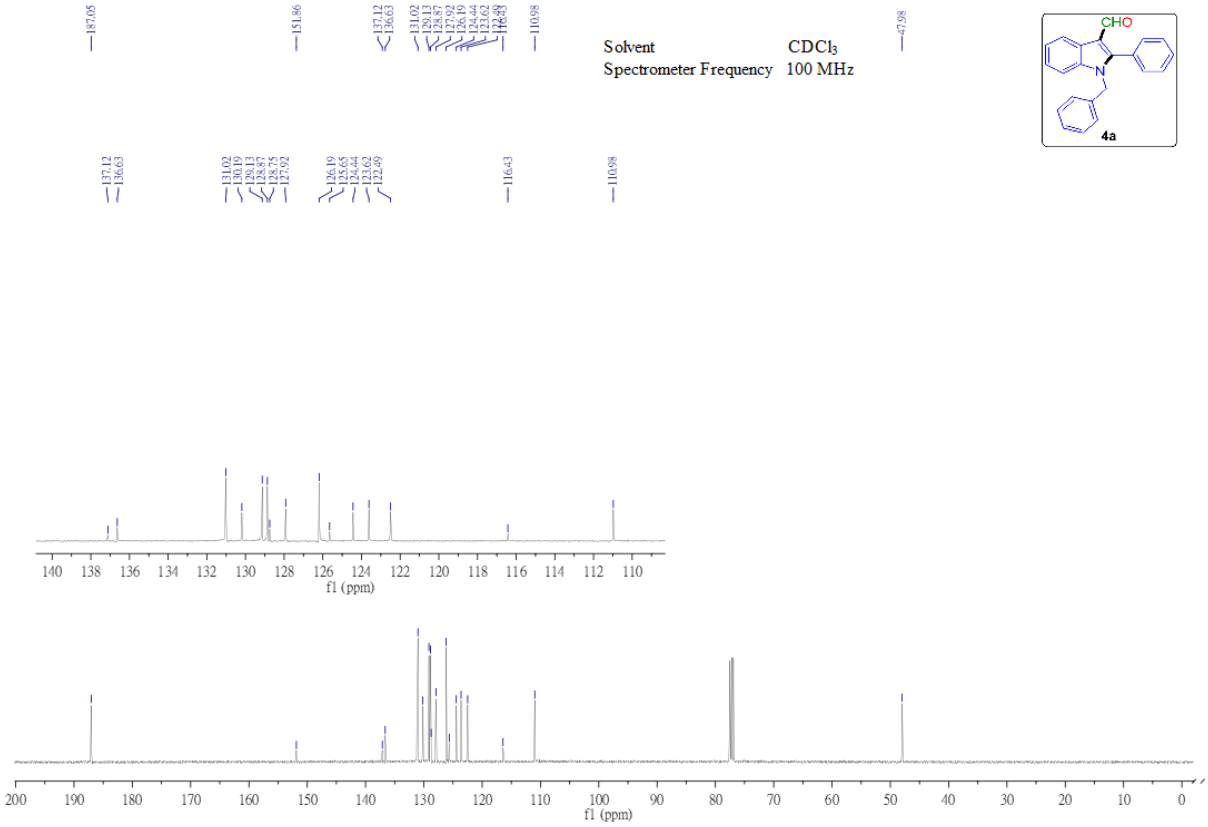
Compound code: **6g**

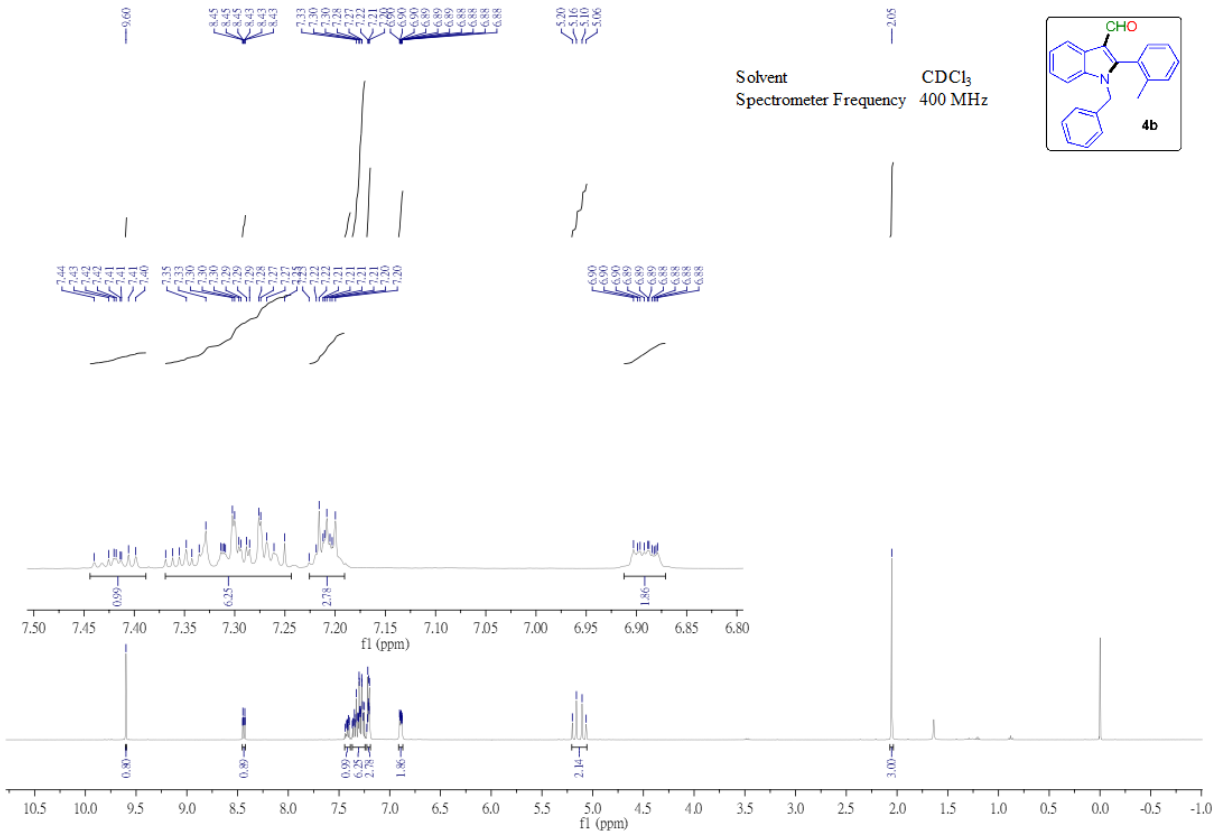


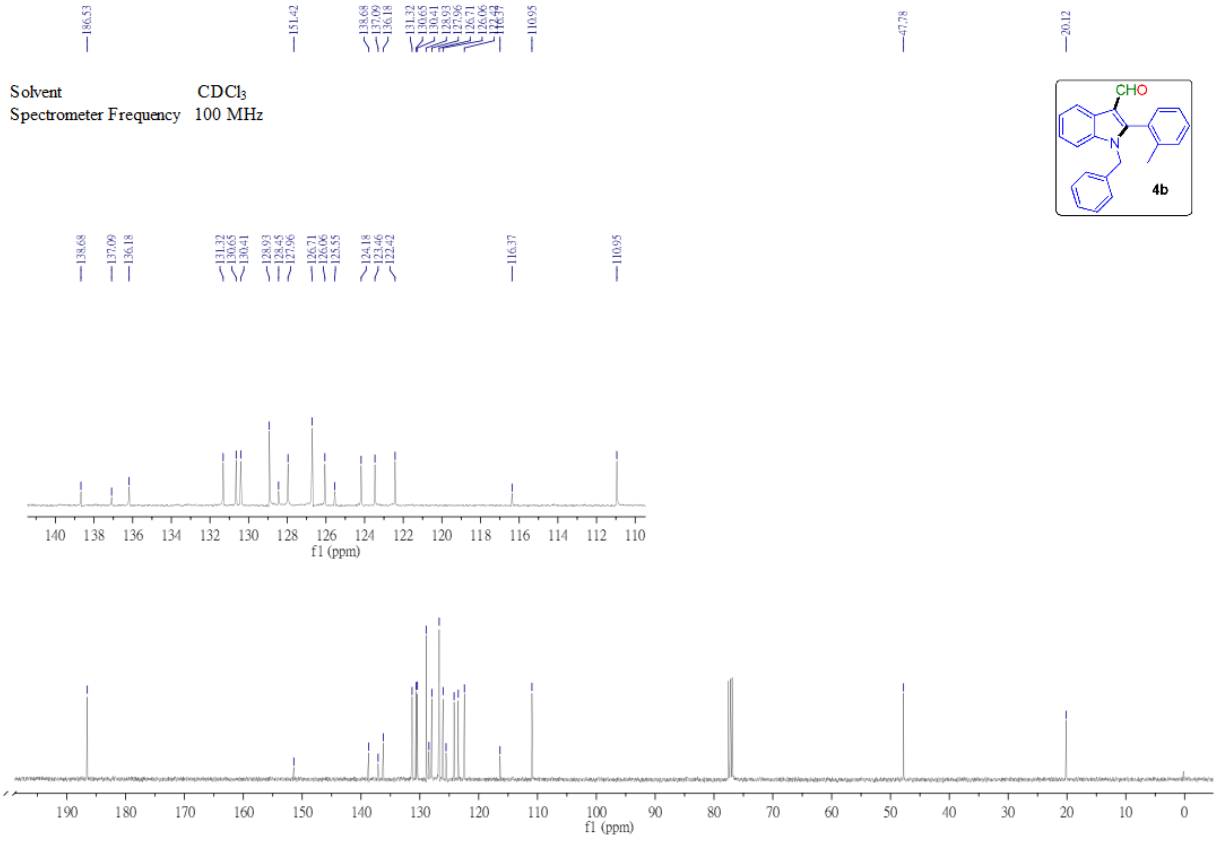

Solvent CDCl3
Spectrometer Frequency 400 MHz
6h
0.92
1.94
0.95
3.59
0.93
1.93
2.91
1.93
2.02
3.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
8.175
8.154
7.625
7.605
7.396
7.251
7.247
7.216
7.213
7.198
7.194
7.188
7.168
6.380
3.004
2.984
2.963
2.332
1.769
1.751
1.732
1.713
1.694
1.472
1.453
1.434
1.415
0.978
0.960
0.940
X : parts per Million : Proton

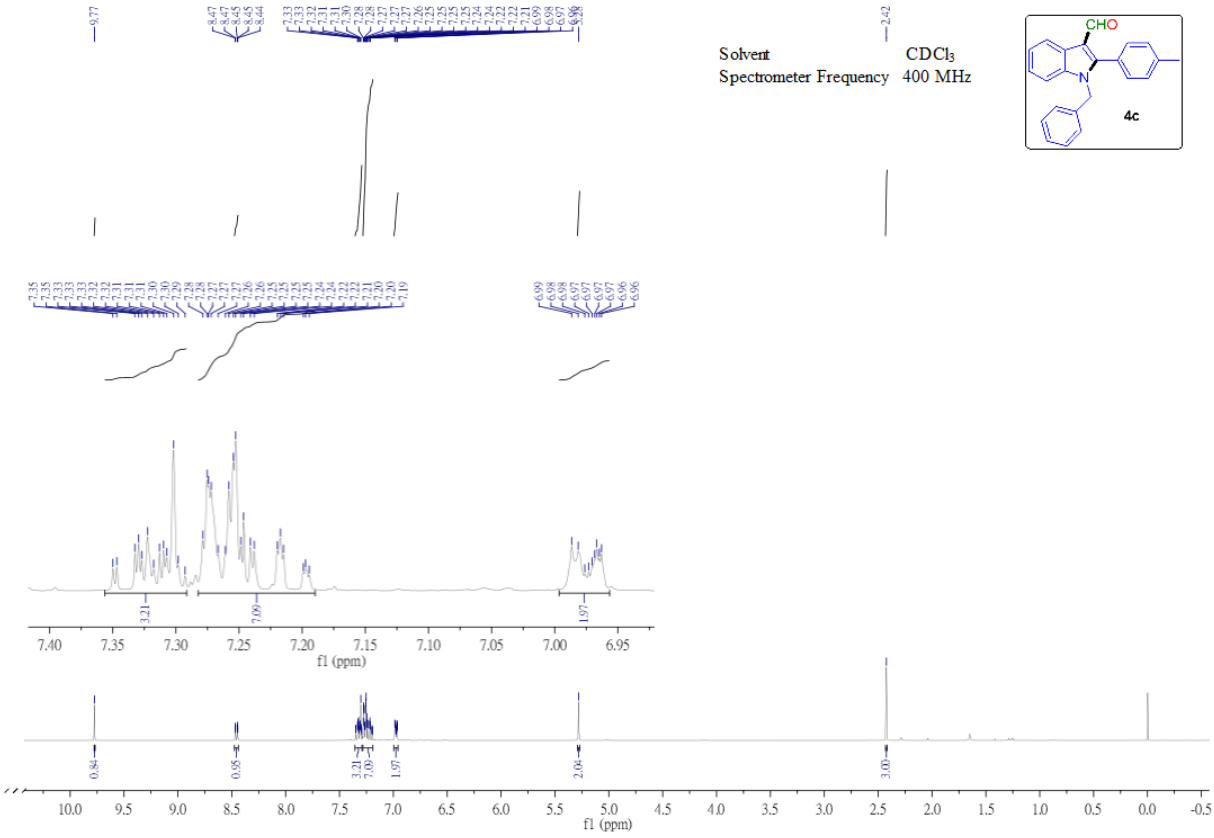

Solvent CDCl3
Spectrometer Frequency 100 MHz
6h
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
144.773
142.704
137.384
136.421
130.042
129.956
126.438
123.940
123.625
120.212
115.006
108.789
31.159
28.937
22.673
21.729
14.140
X : parts per Million : Carbon13

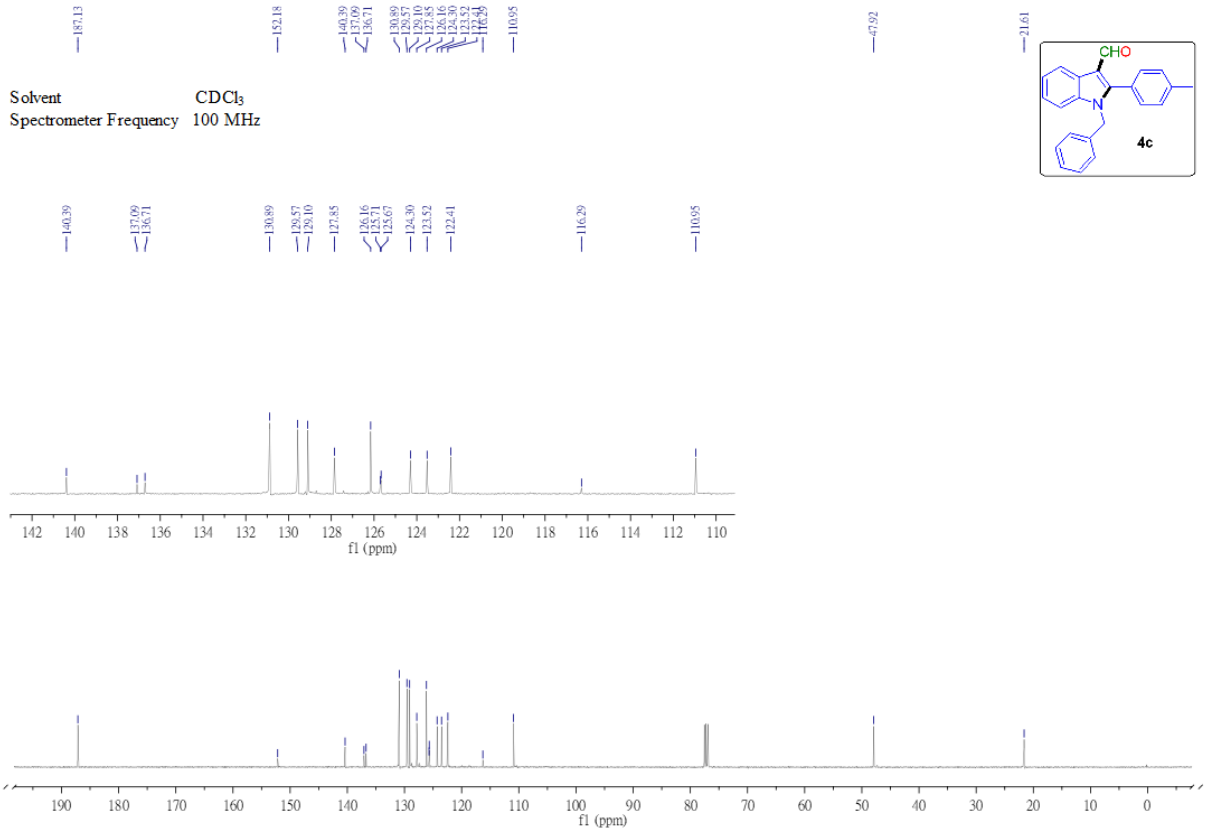

9.66
8.36
8.35
8.35
7.36
7.33
7.33
7.33
7.32
7.15
7.15
7.15
7.14
7.14
7.14
6.87
6.87
6.86
6.86
6.85
6.85
6.85
5.18
7.41
7.39
7.38
7.36
7.36
7.34
7.34
7.33
7.33
7.33
7.32
7.32
7.31
7.30
7.24
7.24
7.23
7.22
7.22
7.22
7.17
7.17
7.16
7.16
7.15
7.15
7.15
7.14
7.14
7.13
7.11
6.87
6.87
6.86
6.86
6.85
6.85
6.85
5.58
1.89
5.78
2.19
7.40 7.35 7.30 7.25 7.20 7.15 7.10 7.05 7.00 6.95 6.90 6.85
f1 (ppm)
1.00
1.07
5.81
1.89
5.78
2.19
2.48
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)
Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
4a

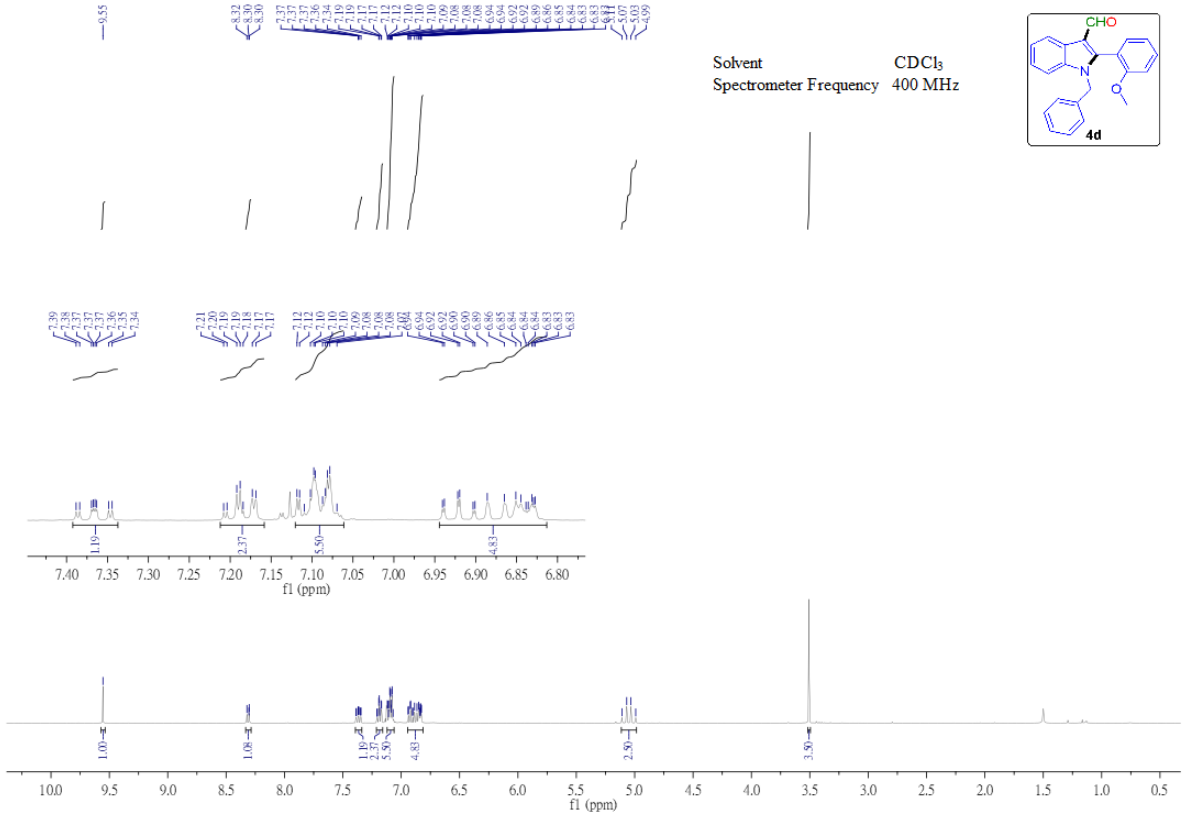

187.05
151.86
137.12
136.63
131.02
130.19
129.13
128.87
127.92
126.19
125.65
124.44
123.62
122.49
116.43
110.98
47.98
137.12
136.63
131.02
130.19
129.13
128.87
128.75
127.92
126.19
125.65
124.44
123.62
122.49
116.43
110.98
140 138 136 134 132 130 128 126 124 122 120 118 116 114 112 110
f1 (ppm)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
4a

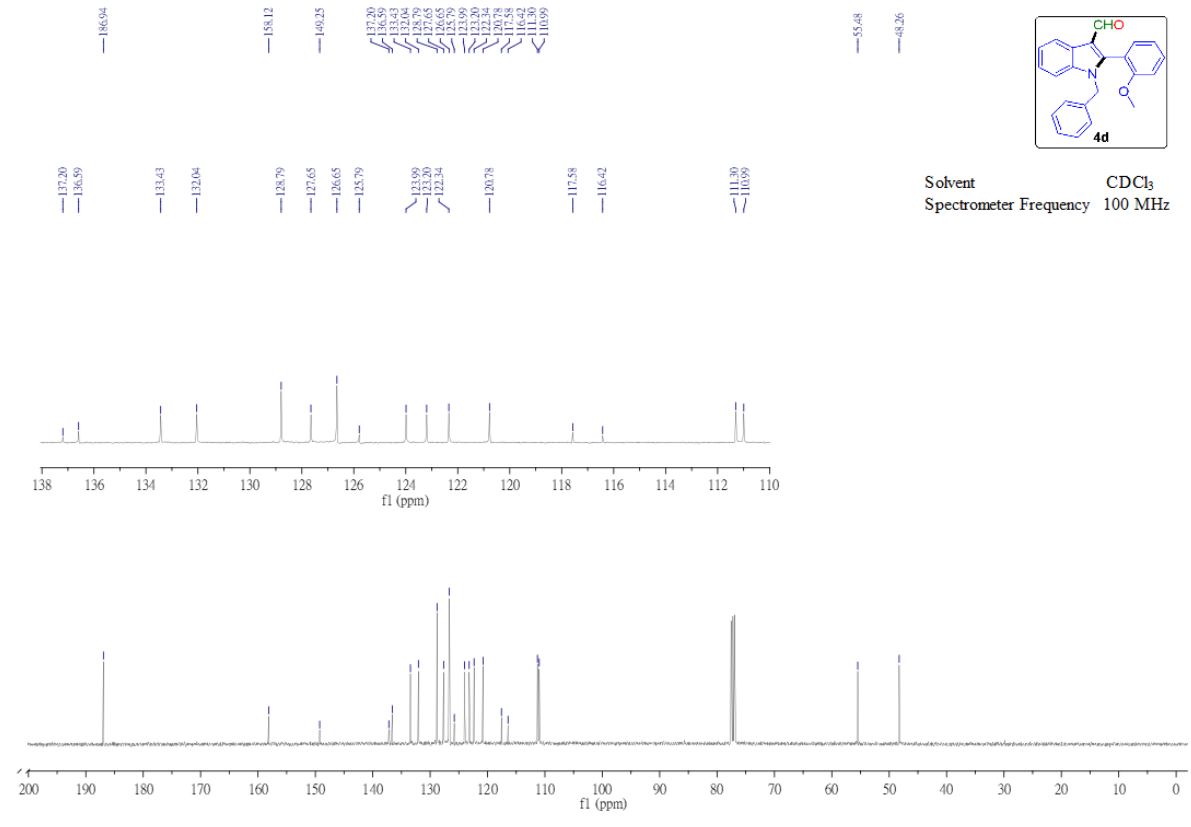

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
4b
f1 (ppm)

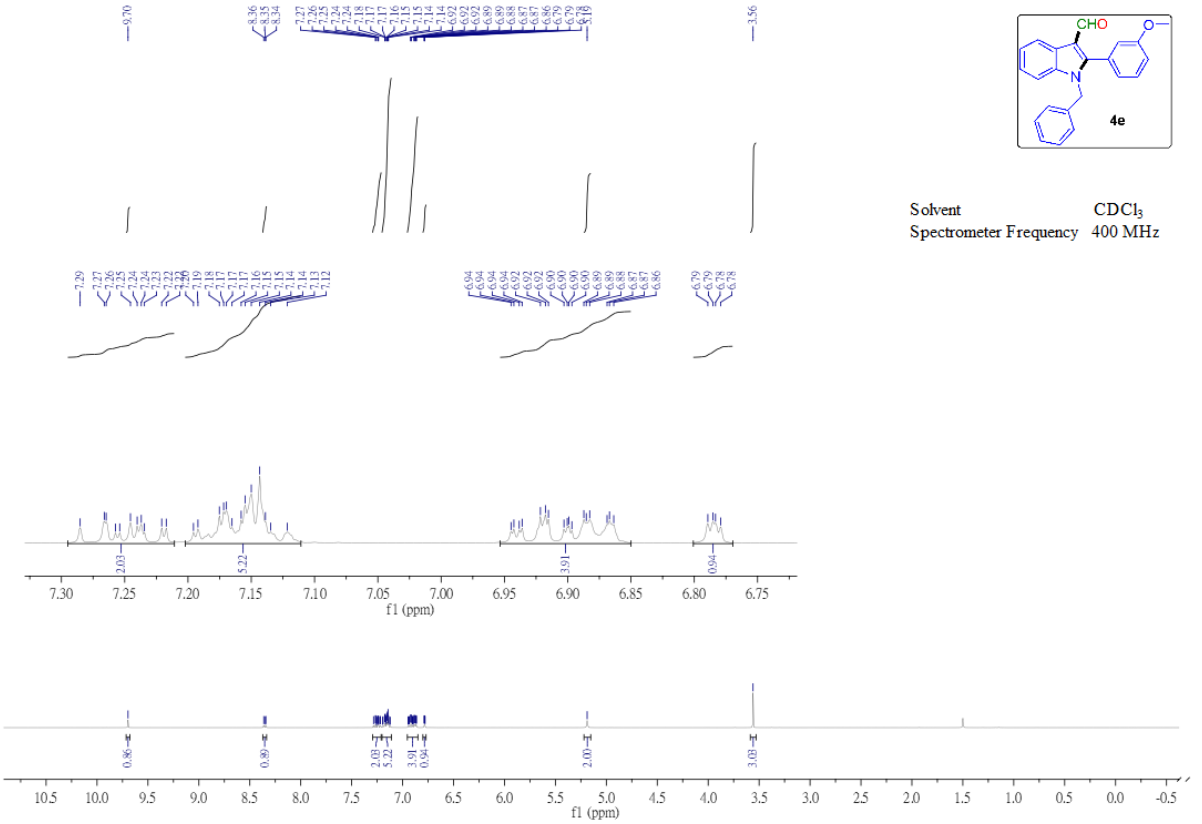

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
4b
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
4c
f1 (ppm)

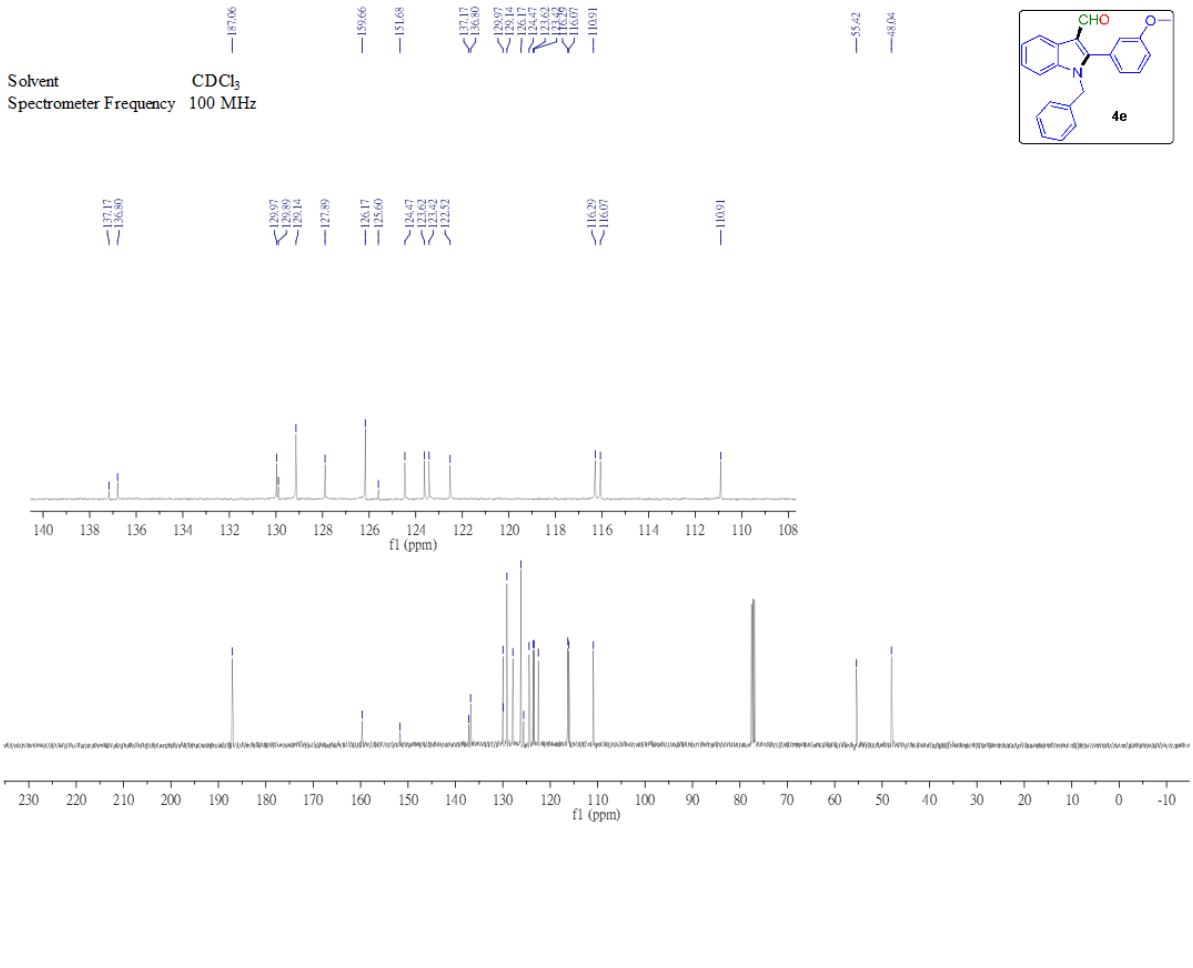

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
4c
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 400 MHz
4d
f1 (ppm)

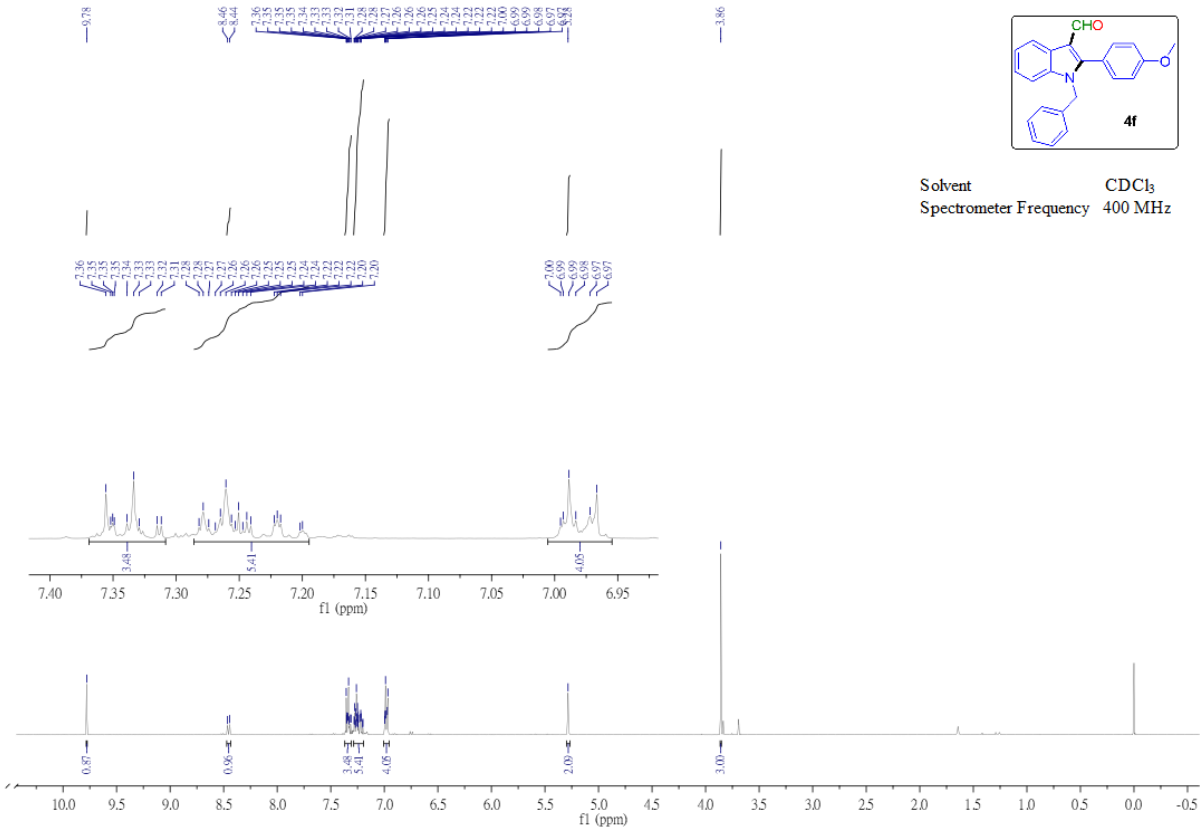

Solvent CDCl3
Spectrometer Frequency 100 MHz
4d
f1 (ppm)



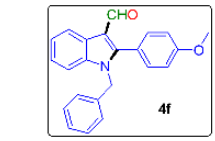
Solvent $CDCl_3$
Spectrometer Frequency 400 MHz



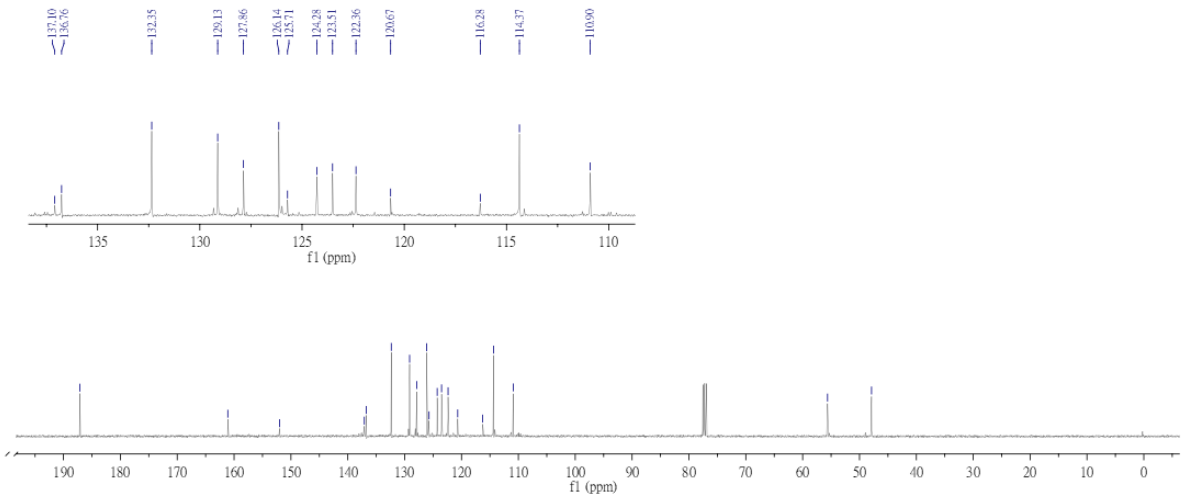
Solvent $CDCl_3$
Spectrometer Frequency 100 MHz

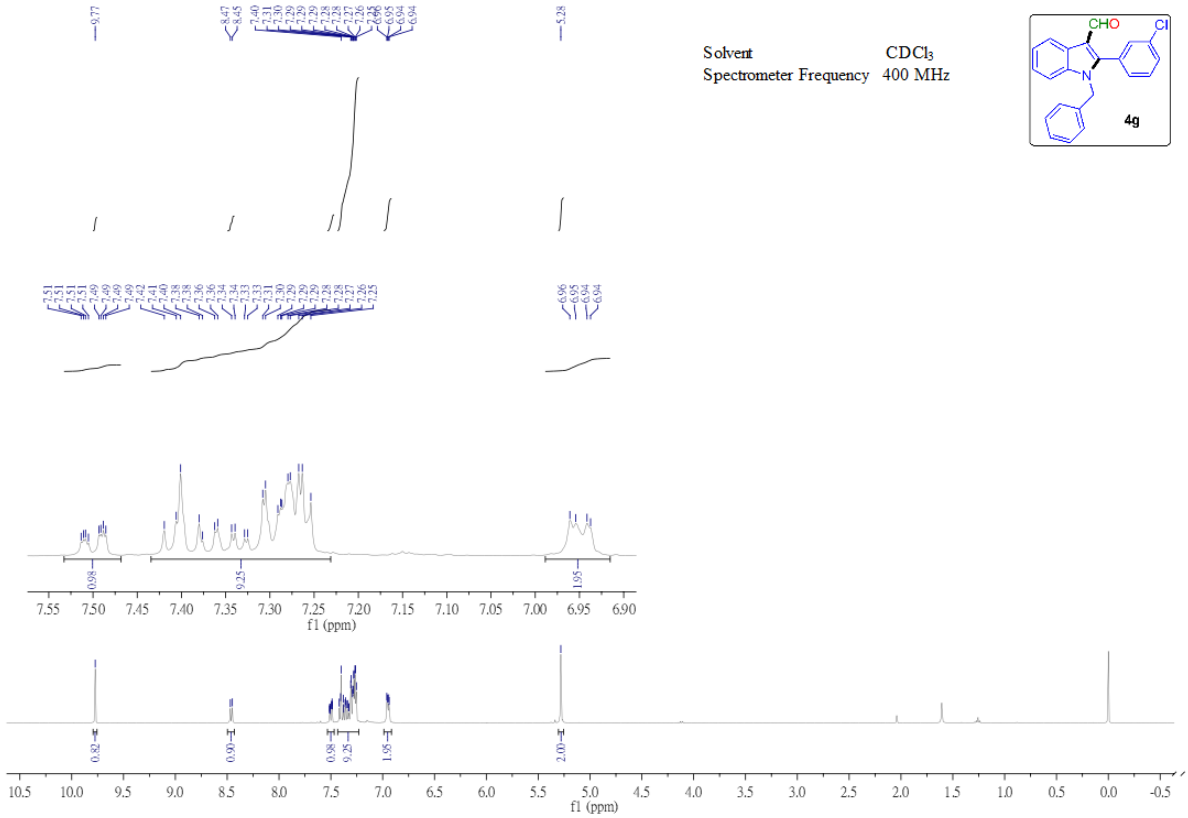


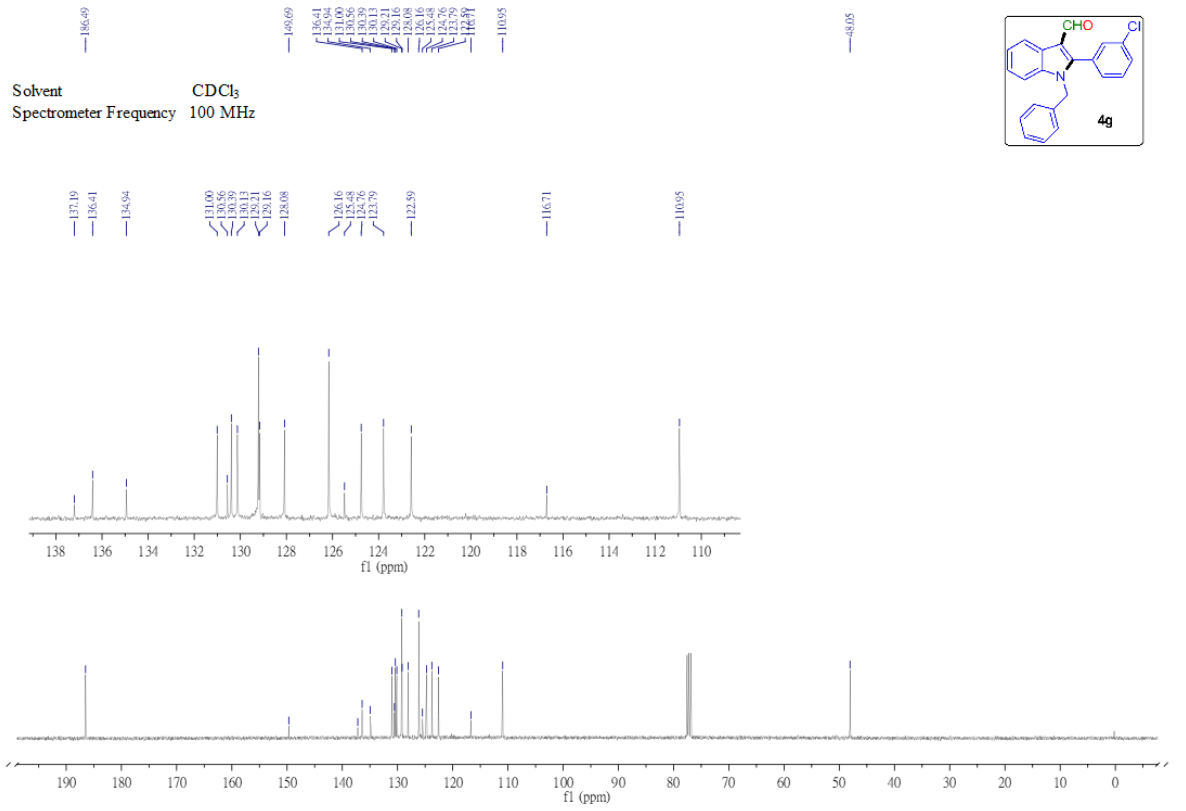
Solvent $CDCl_3$
Spectrometer Frequency 400 MHz

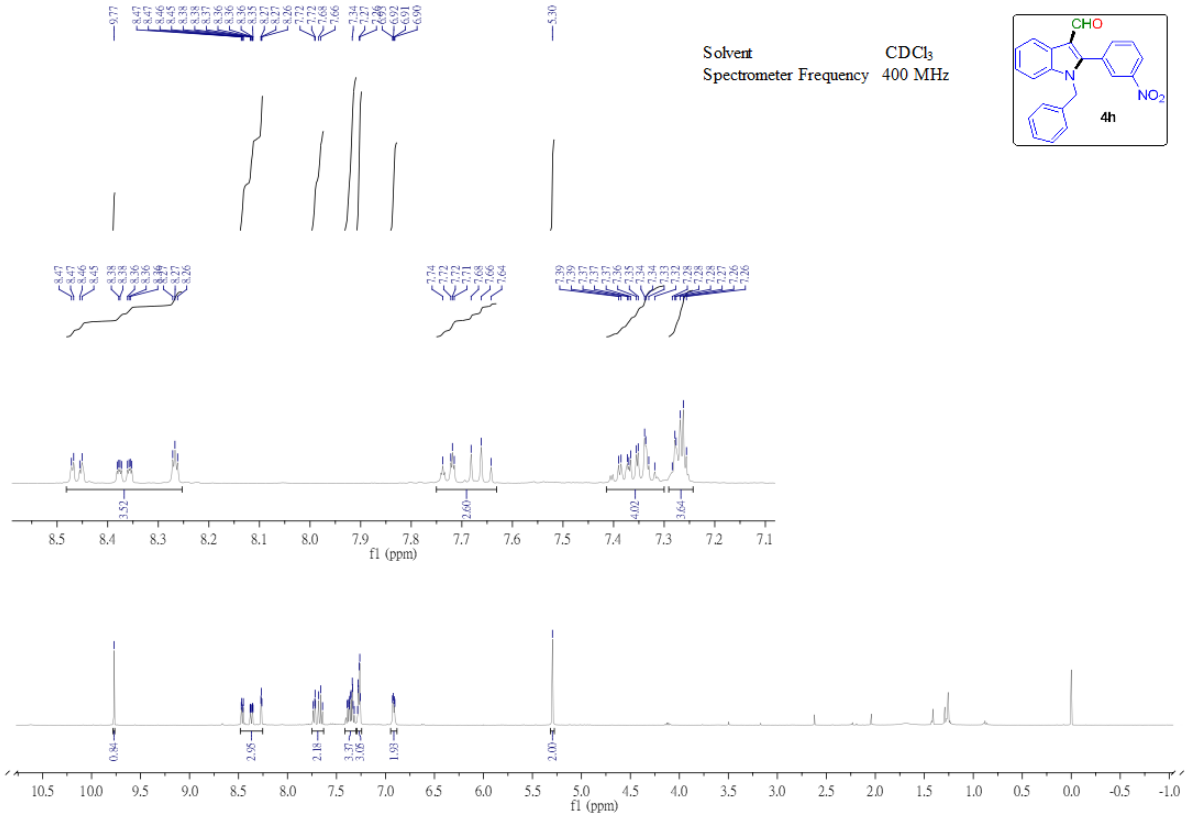


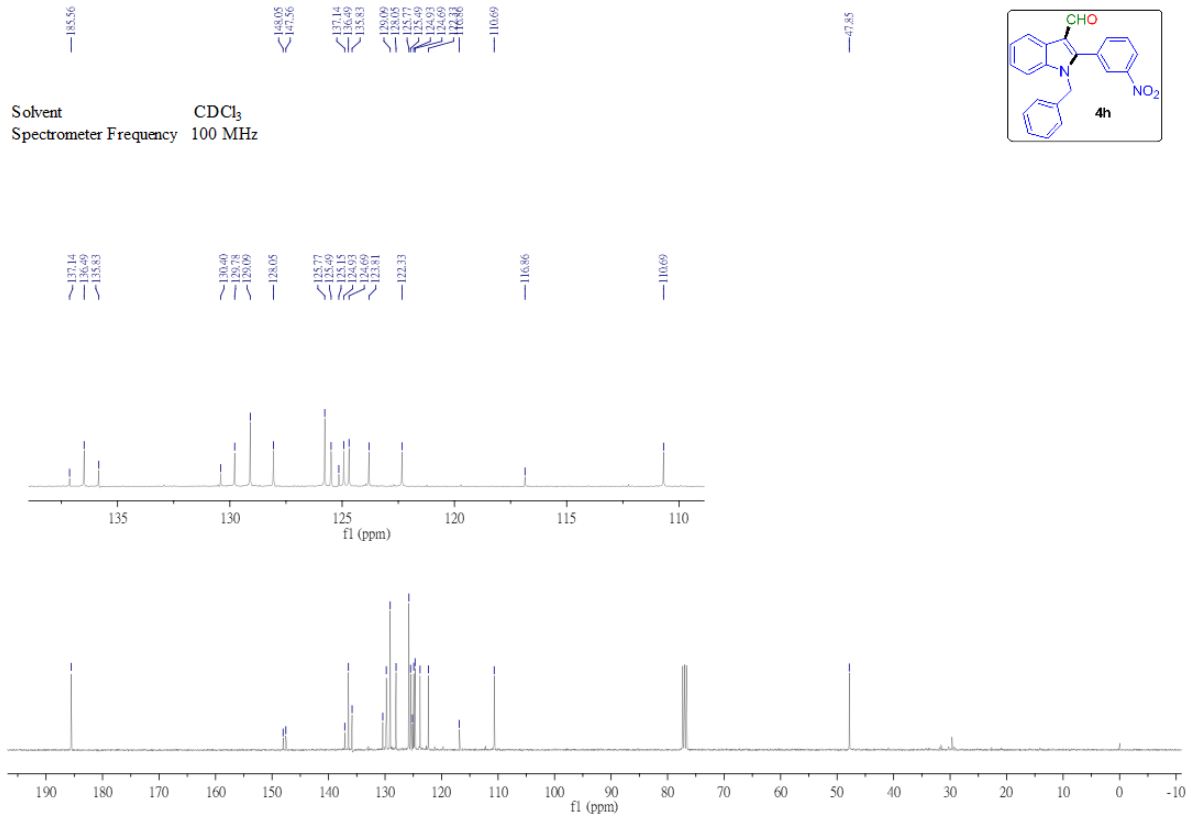
Solvent $CDCl_3$
Spectrometer Frequency 100 MHz

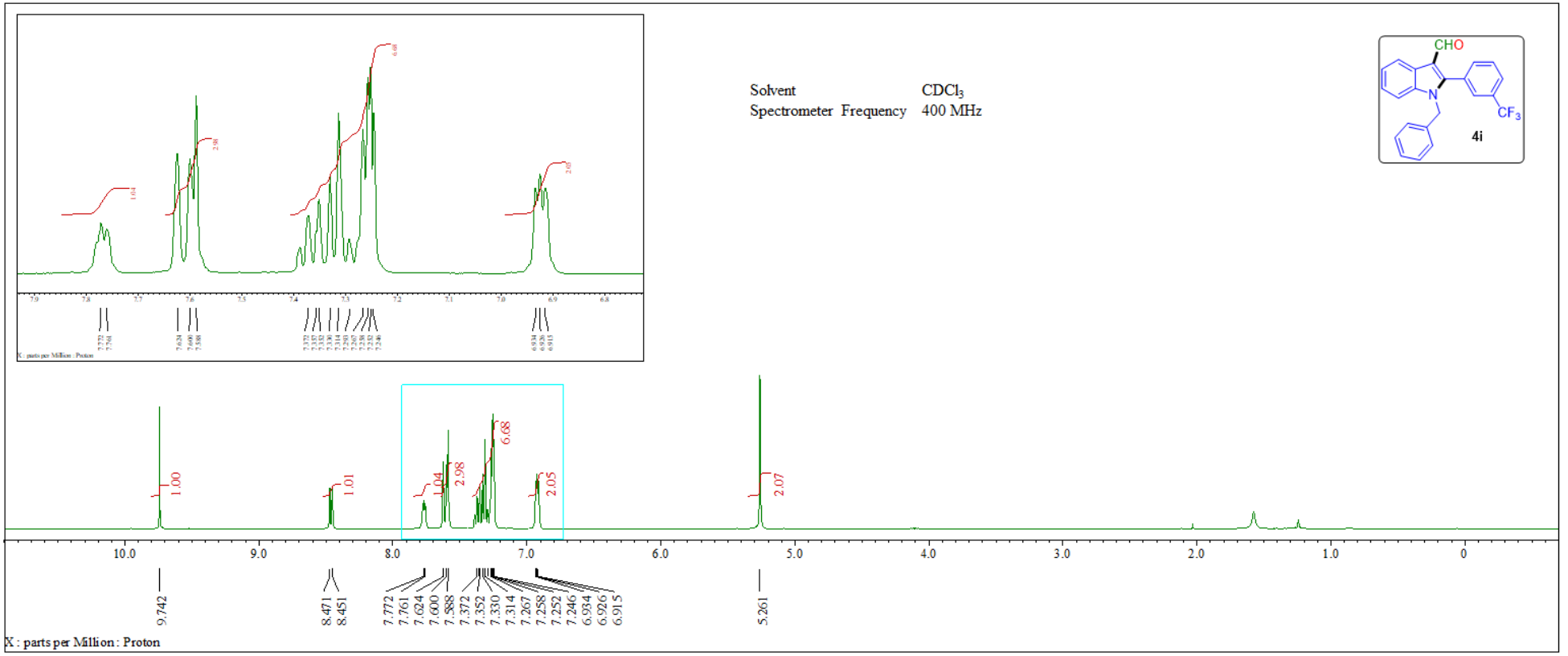


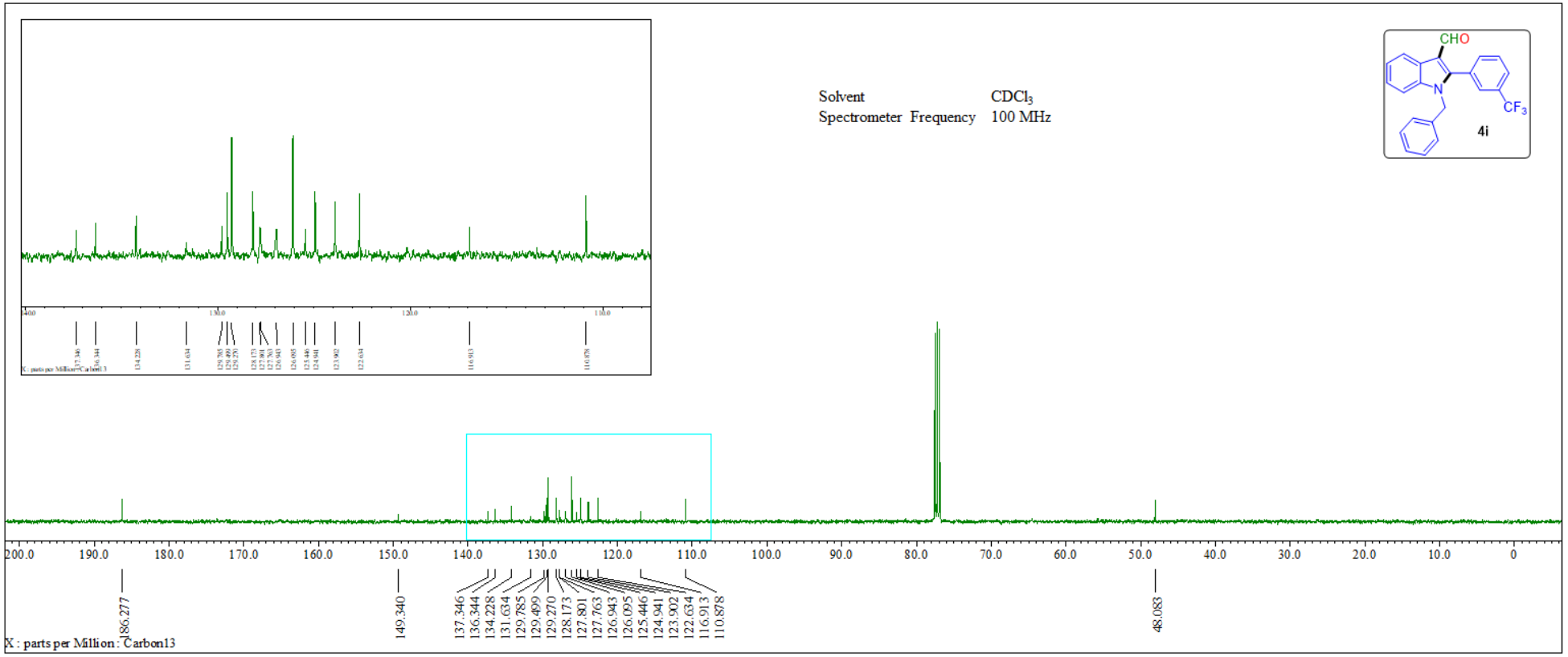

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
Cl
N
4g
f1 (ppm)

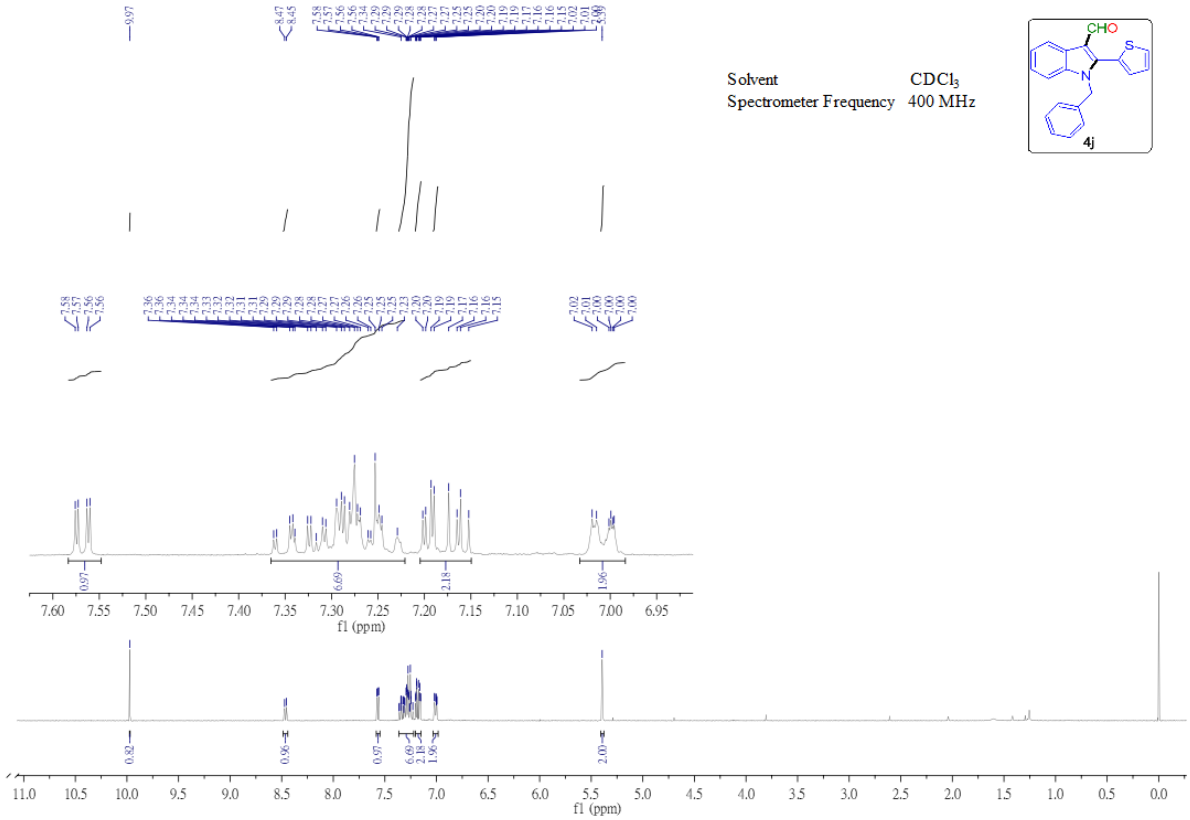

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
Cl
N
4g
f1 (ppm)

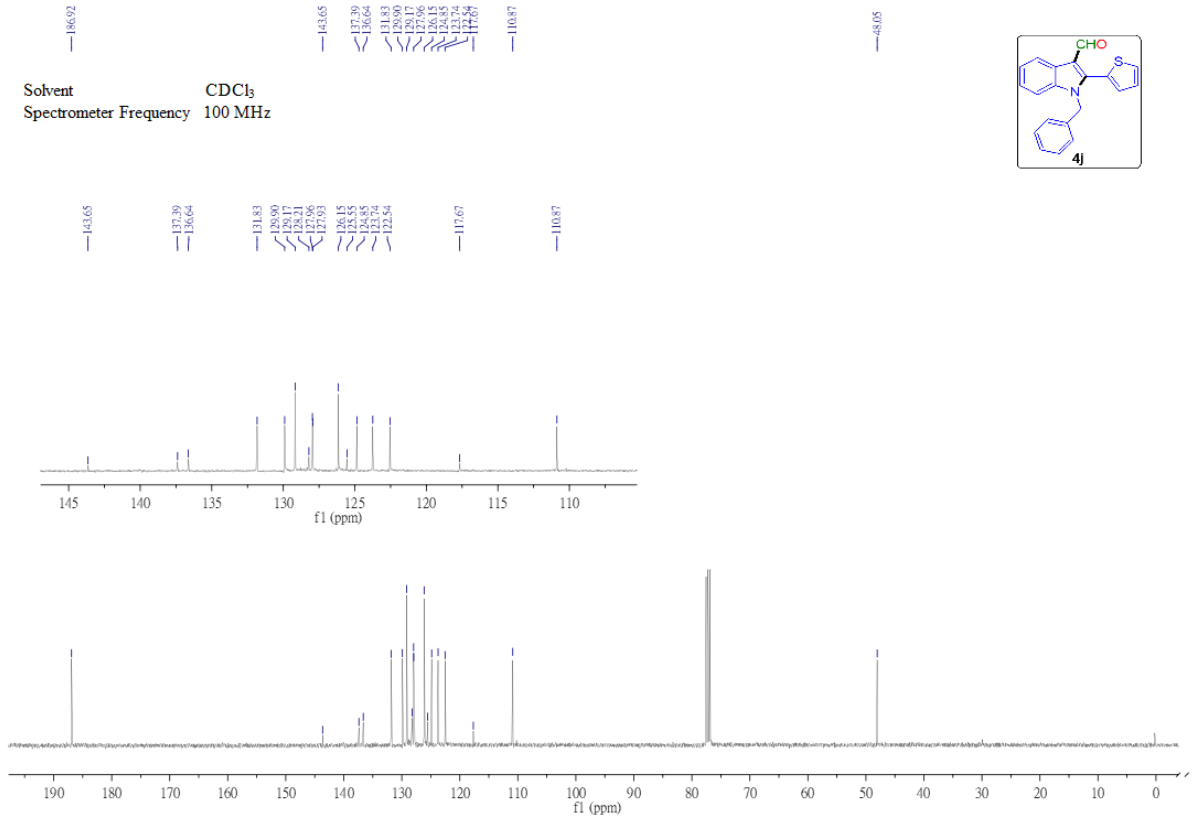

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
NO2
4h
f1 (ppm)

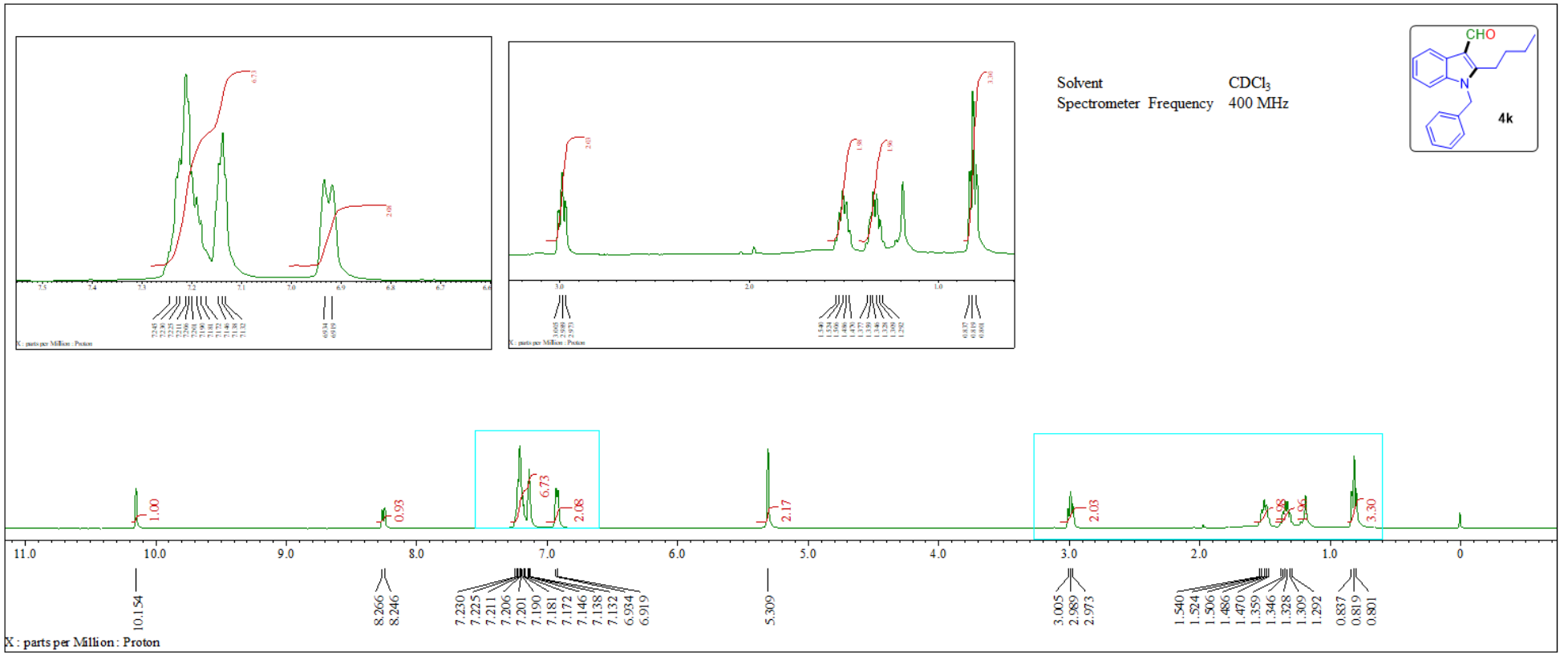

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
NO2
4h
f1 (ppm)

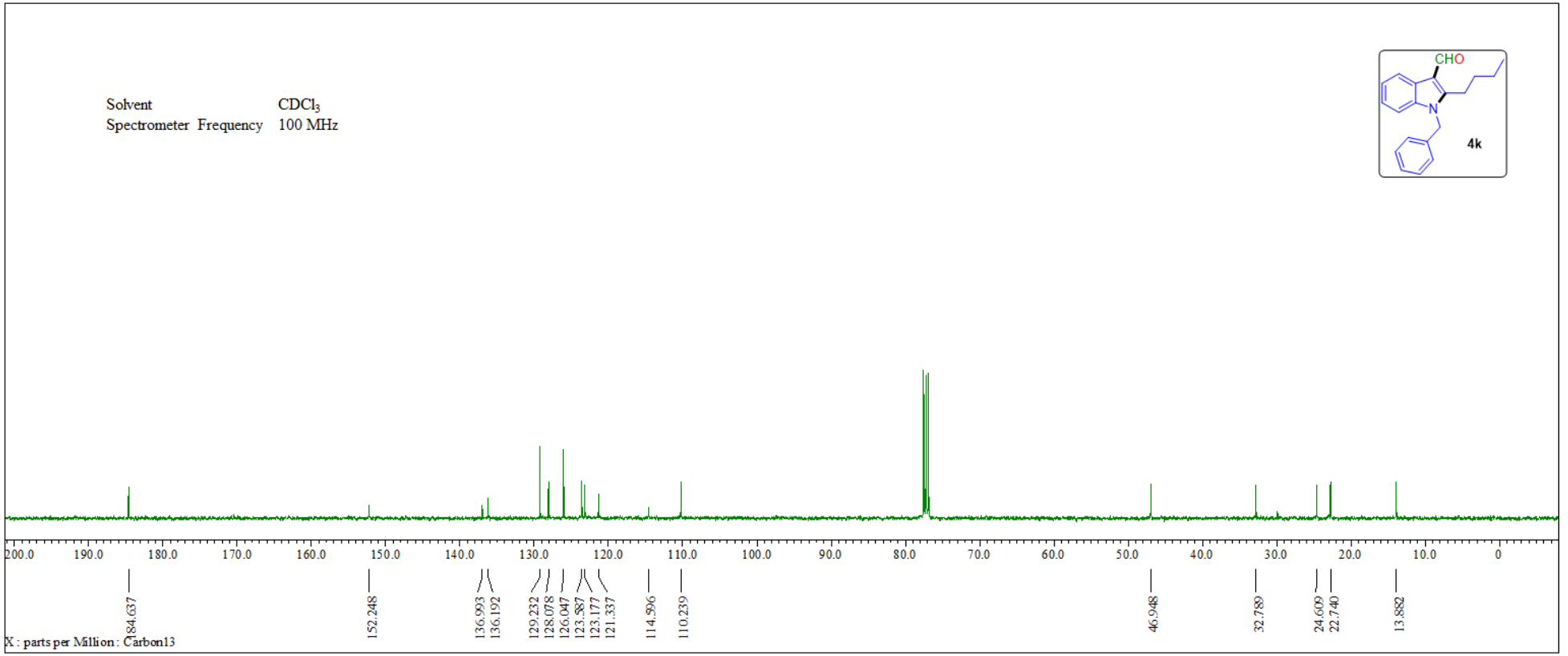

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
CF3
4i
1.00
1.01
1.04
2.98
6.68
2.05
2.07
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.742
8.471
8.451
7.772
7.761
7.624
7.600
7.588
7.372
7.352
7.330
7.314
7.267
7.258
7.252
7.246
6.934
6.926
6.915
5.261
X : parts per Million : Proton

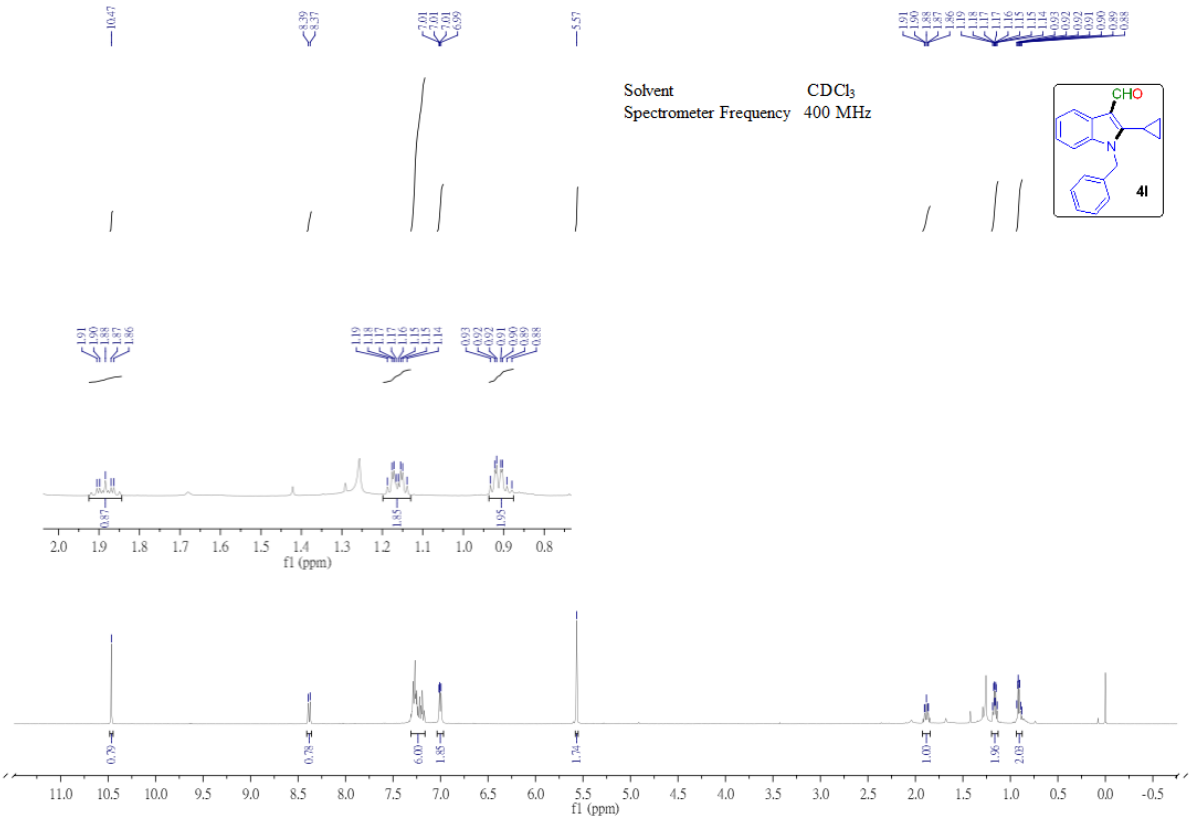

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
CF3
4i
186.277
149.340
137.346
136.344
134.228
131.634
129.785
129.499
129.270
128.173
127.801
127.763
126.943
126.095
125.446
124.941
123.902
122.634
116.913
110.878
48.083
X : parts per Million : Carbon13

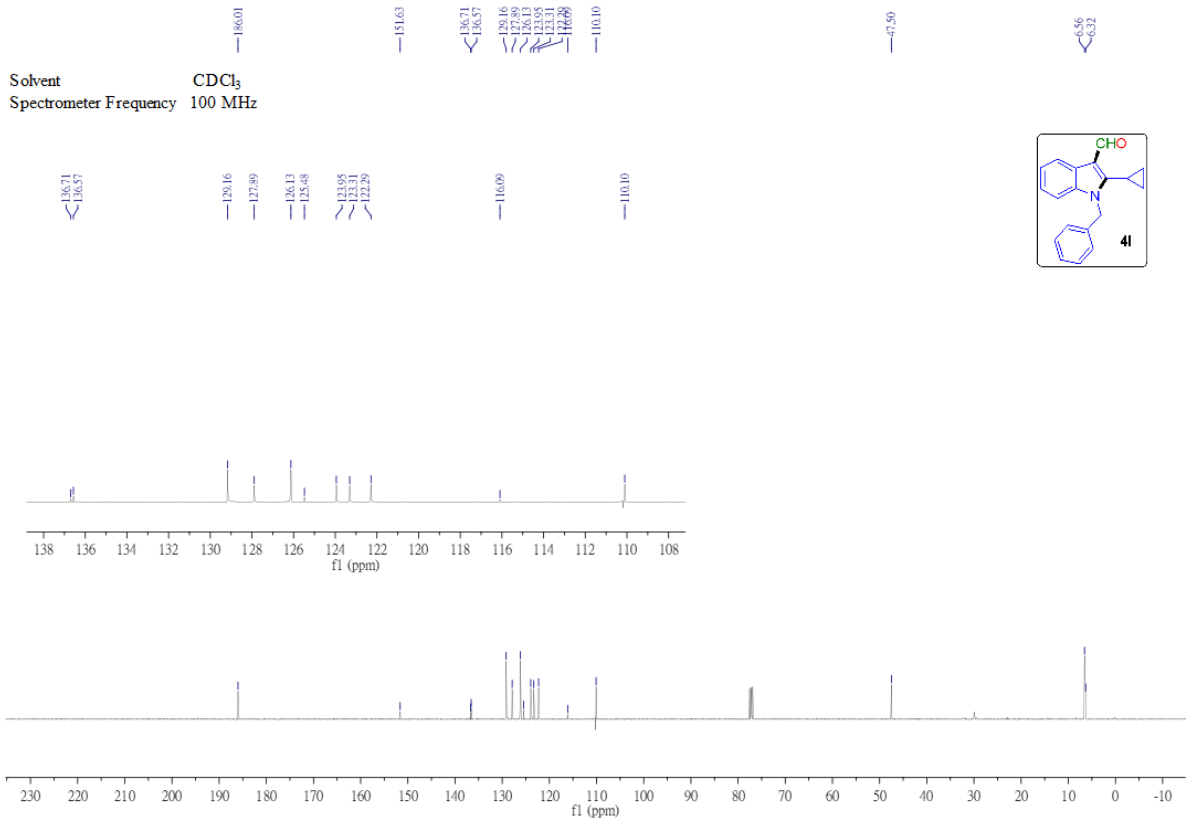

Solvent CDCl3
Spectrometer Frequency 400 MHz
4j
f1 (ppm)

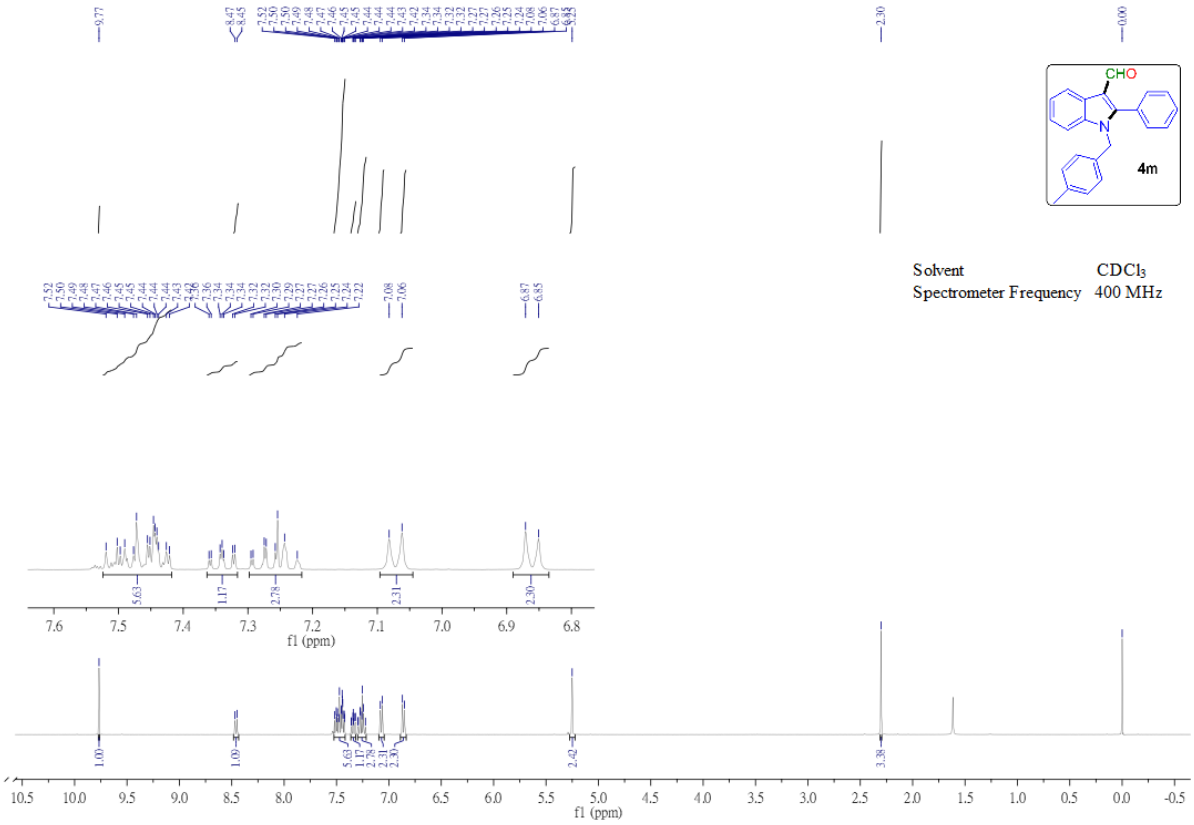

Solvent CDCl3
Spectrometer Frequency 100 MHz
4j
f1 (ppm)

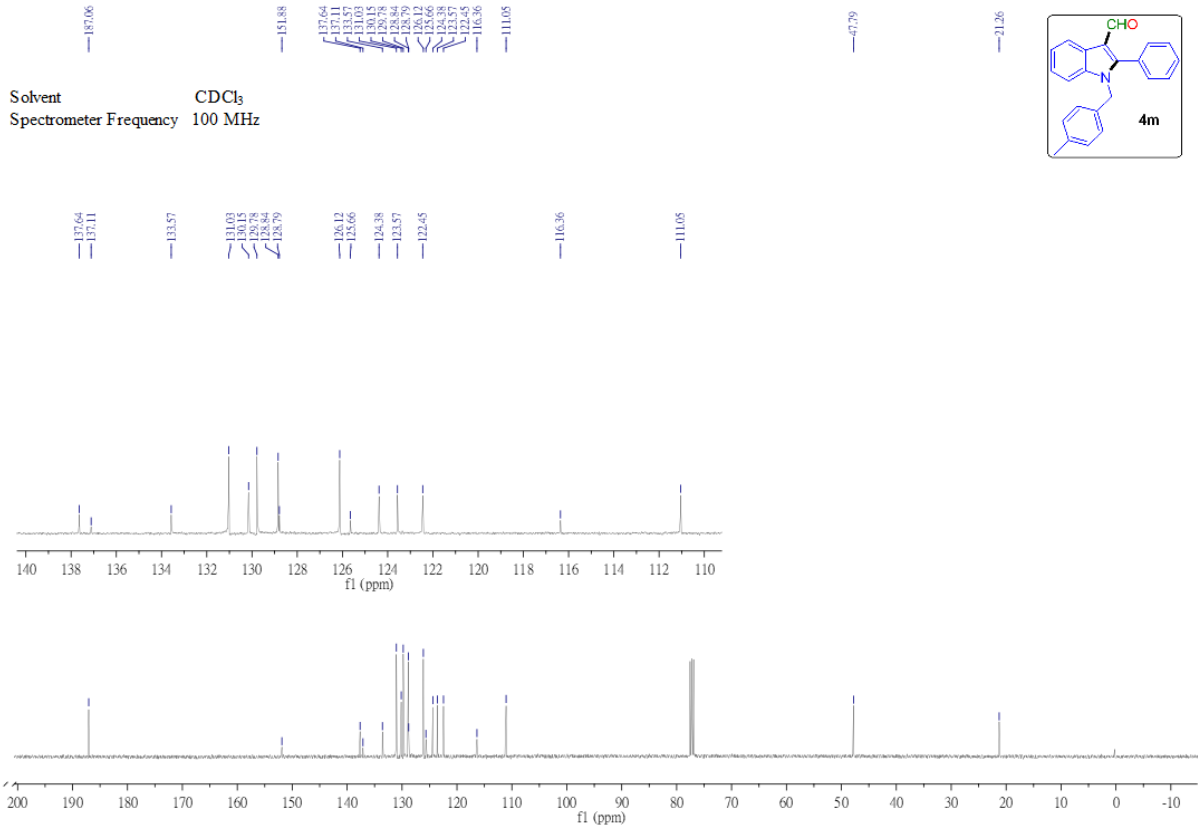

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
4k
1.00
0.93
6.73
2.08
2.17
2.03
3.30
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
10.154
8.266
8.246
7.230
7.225
7.211
7.206
7.201
7.190
7.181
7.172
7.146
7.138
7.132
6.934
6.919
5.309
3.005
2.989
2.973
1.540
1.524
1.506
1.486
1.470
1.359
1.346
1.328
1.309
1.292
0.837
0.819
0.801
X : parts per Million : Proton

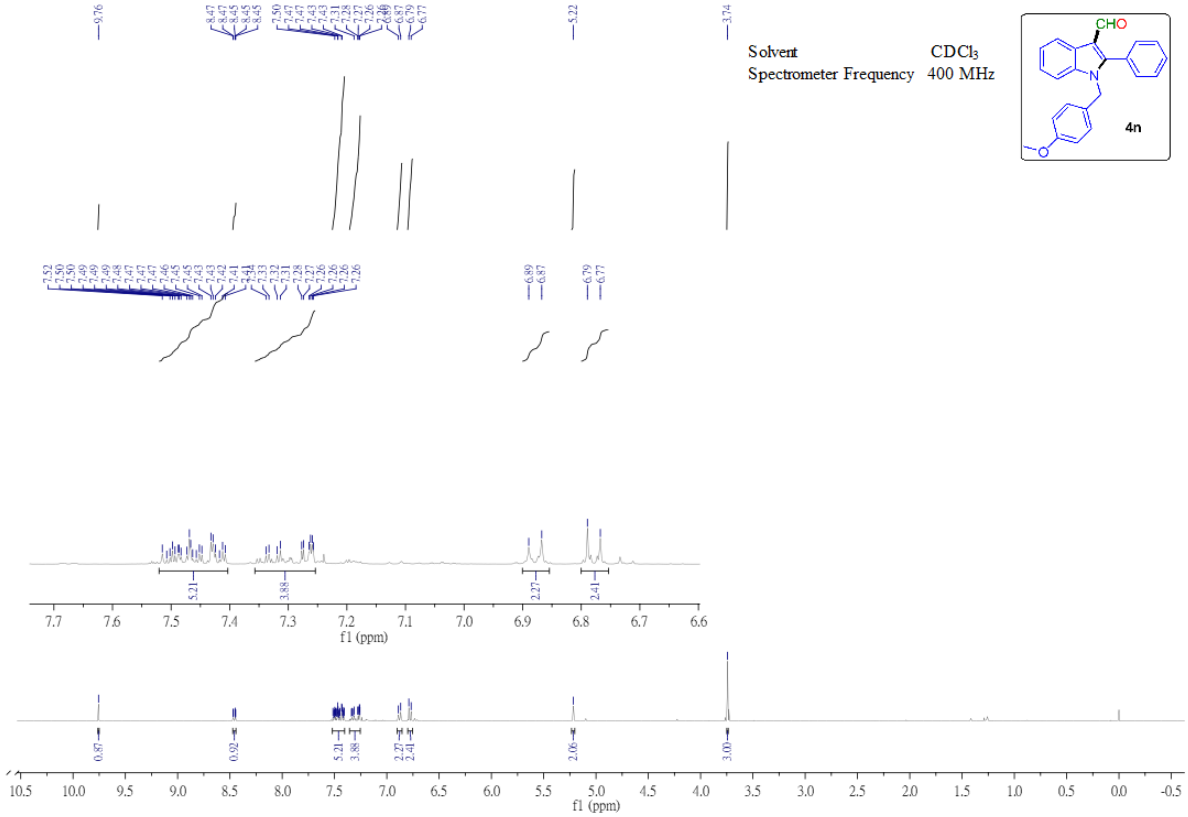

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
4k
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
184.637
152.248
136.993
136.192
129.232
128.078
126.047
123.887
123.177
121.337
114.596
110.239
46.948
32.789
24.609
22.740
13.882
X : parts per Million : Carbon13


Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
4l
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
4l
f1 (ppm)


CHO
4m
Solvent CDCl3
Spectrometer Frequency 400 MHz
f1 (ppm)


CHO
4m
Solvent CDCl3
Spectrometer Frequency 100 MHz
f1 (ppm)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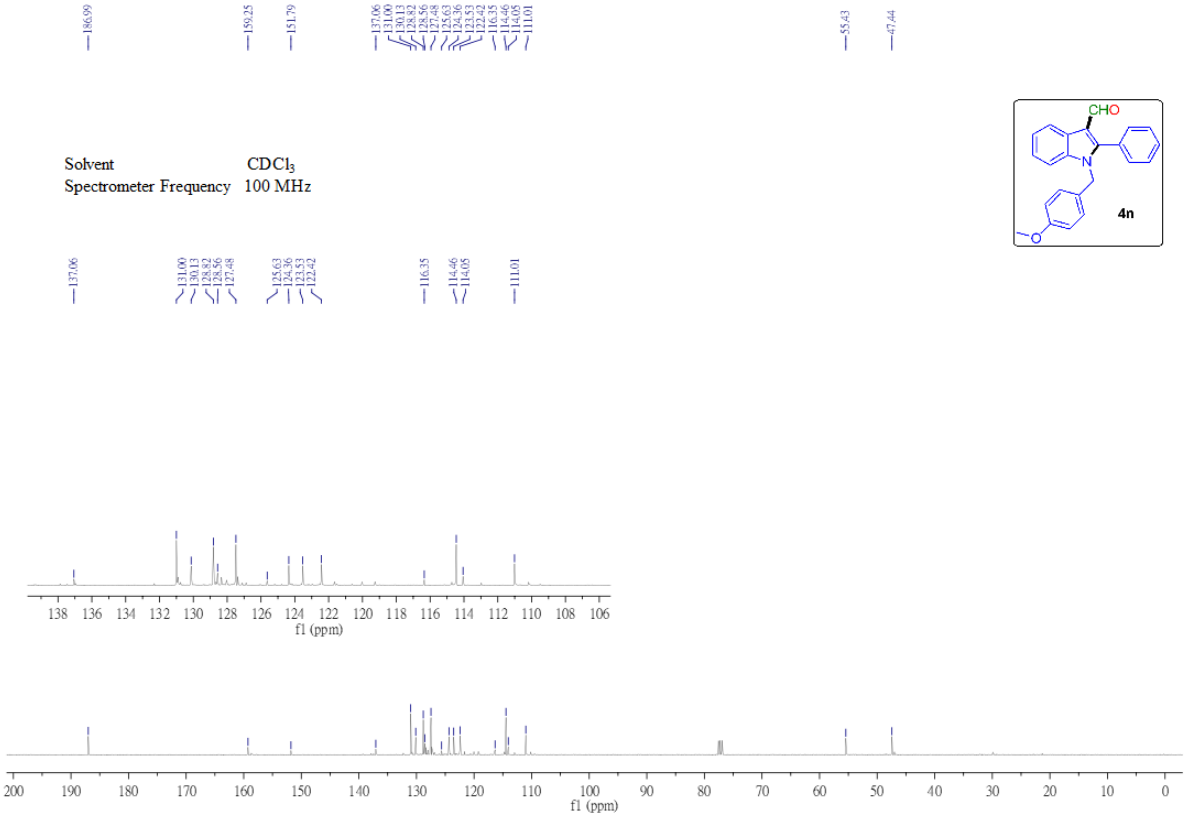
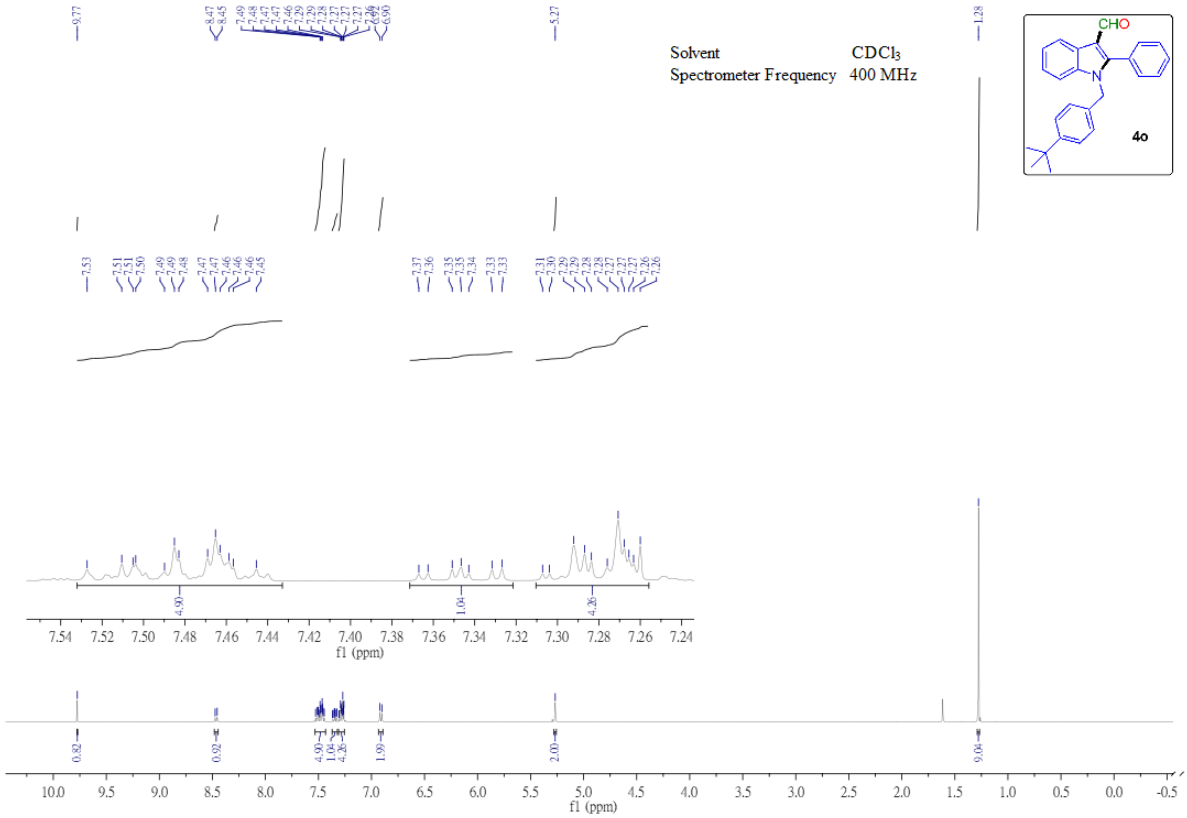
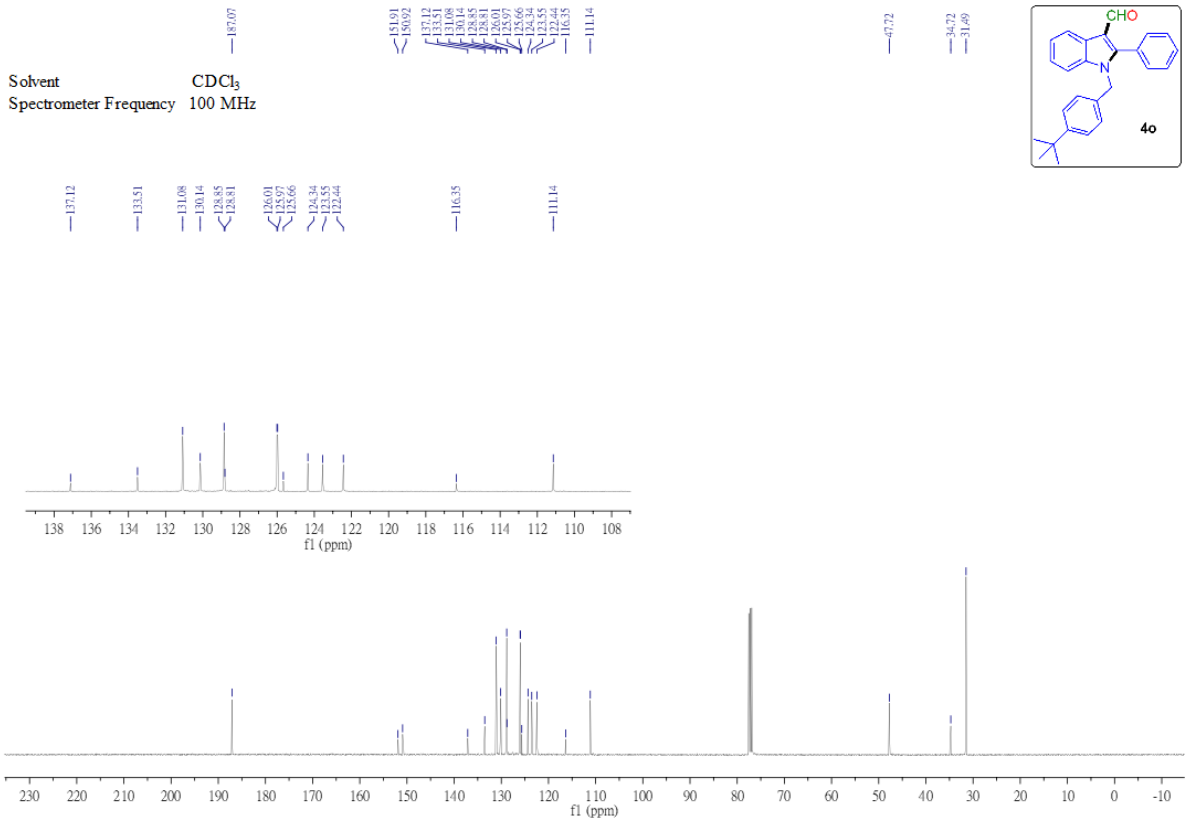
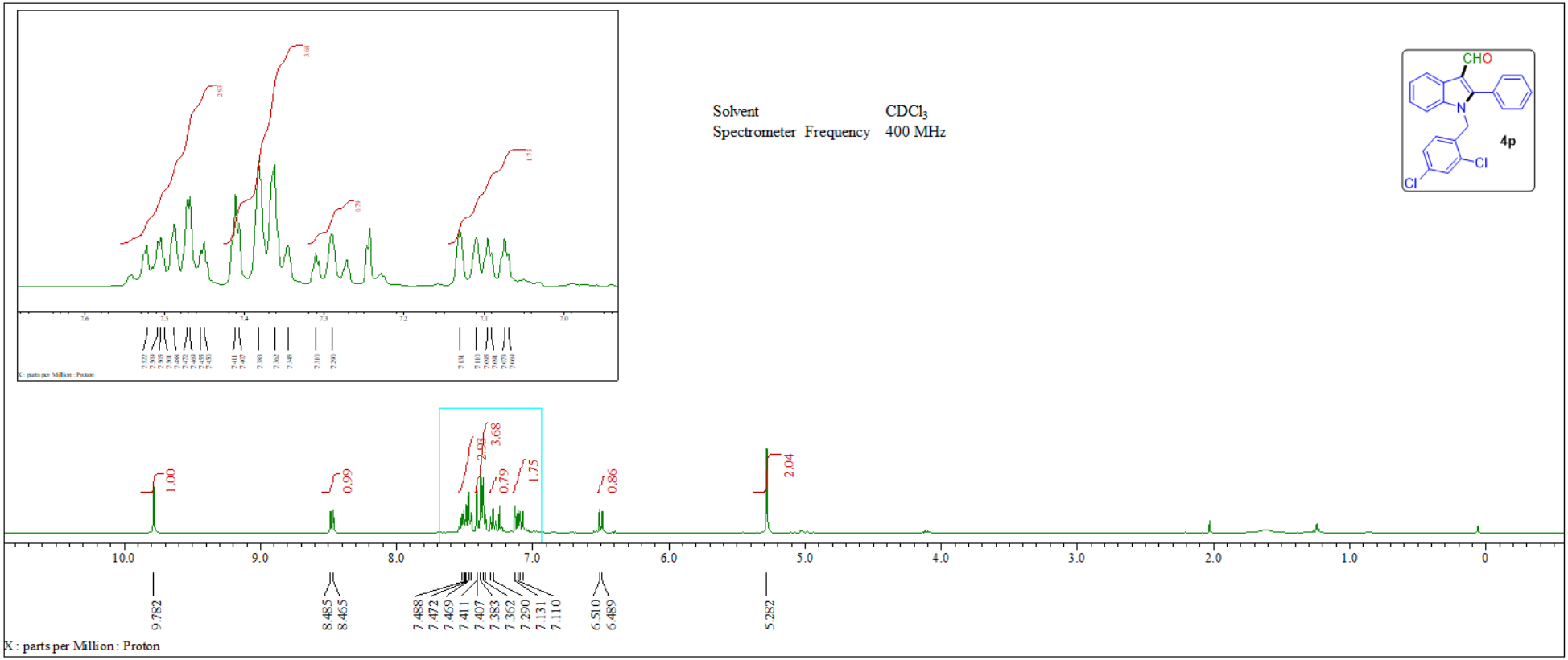
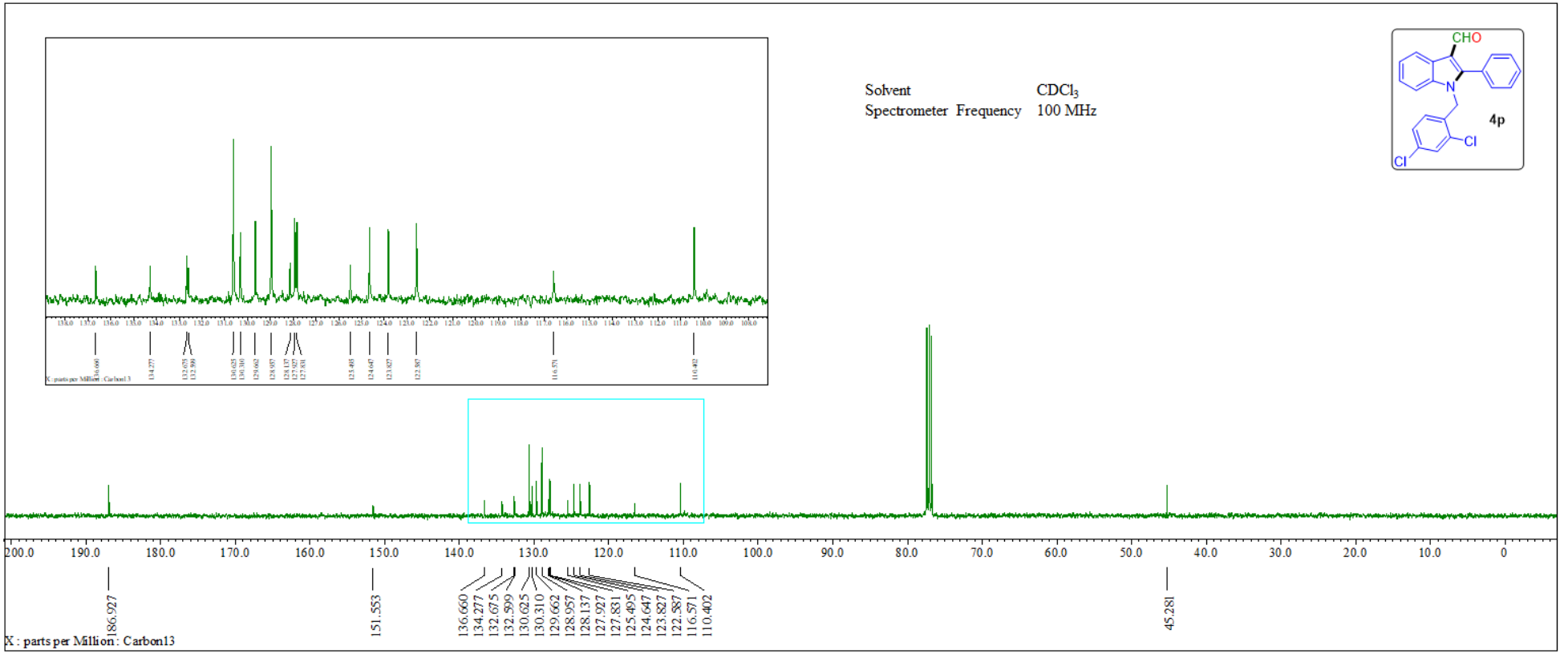
Spectrometer Frequency 400 MHz
CHO
4n
f1 (ppm)

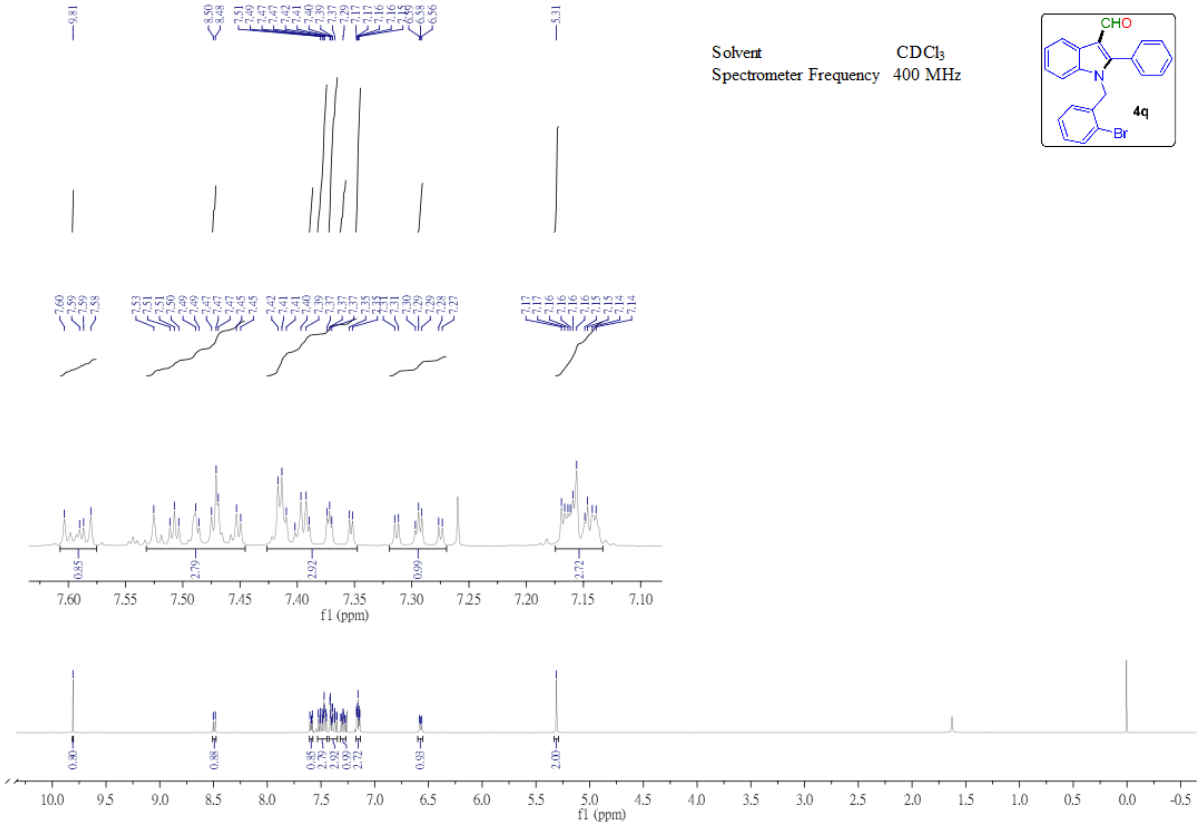

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
4n
f1 (ppm)

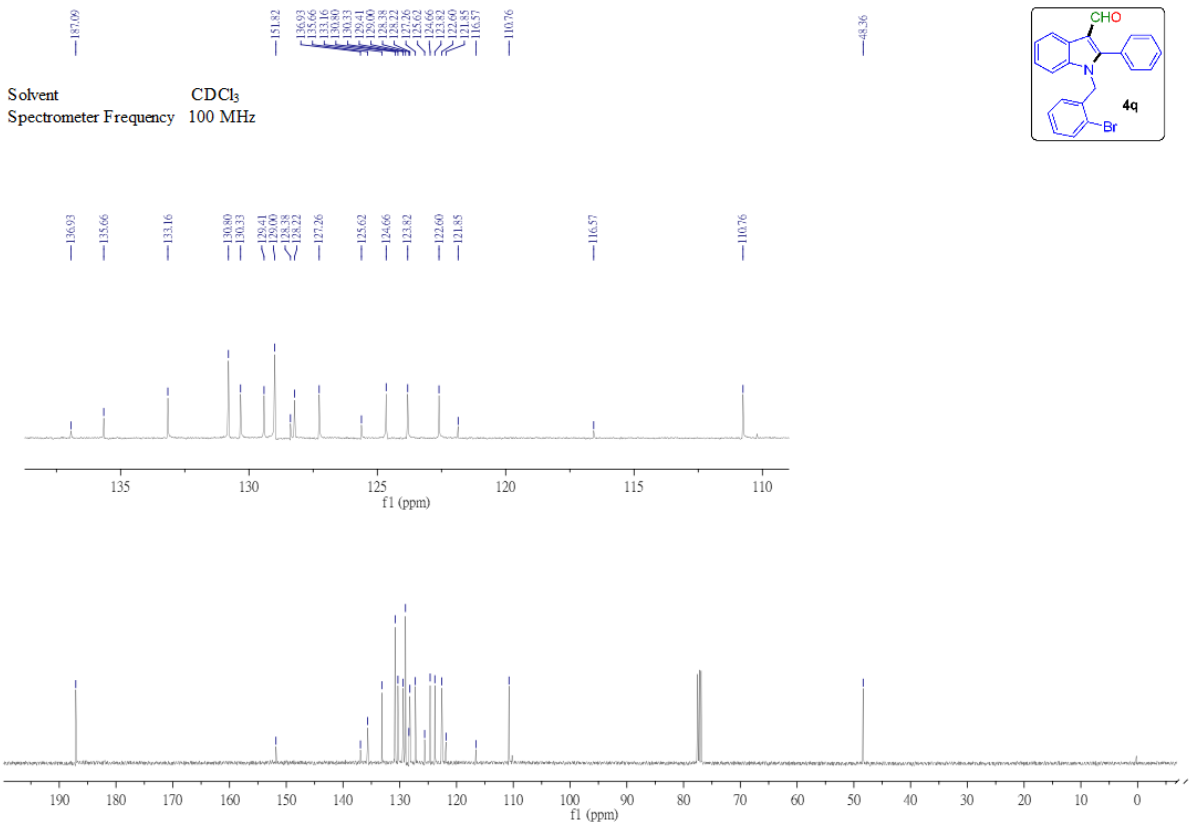

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
4o
f1 (ppm)

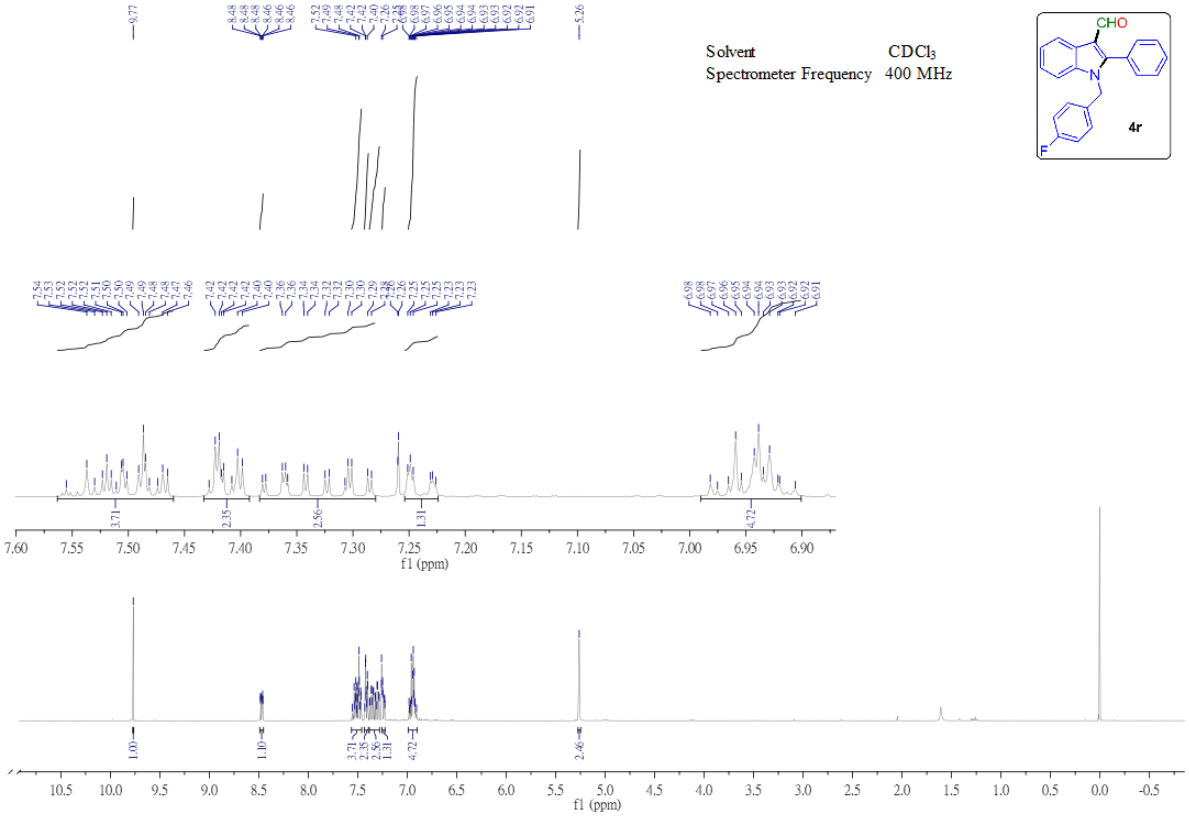

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
4o
f1 (ppm)

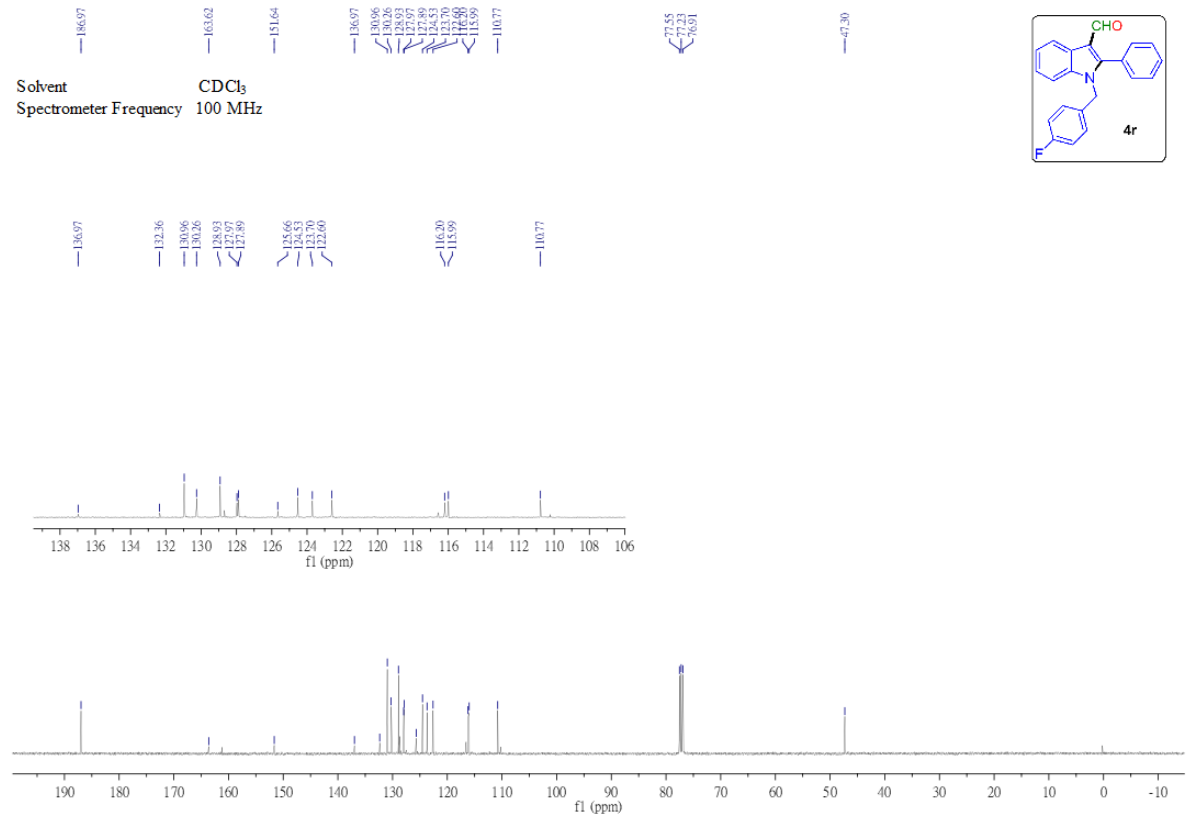

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
Cl
Cl
4p
1.00
0.99
2.93
3.68
0.79
1.75
0.86
2.04
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.782
8.485
8.465
7.488
7.472
7.469
7.411
7.407
7.383
7.362
7.290
7.131
7.110
6.510
6.489
5.282
X : parts per Million : Proton

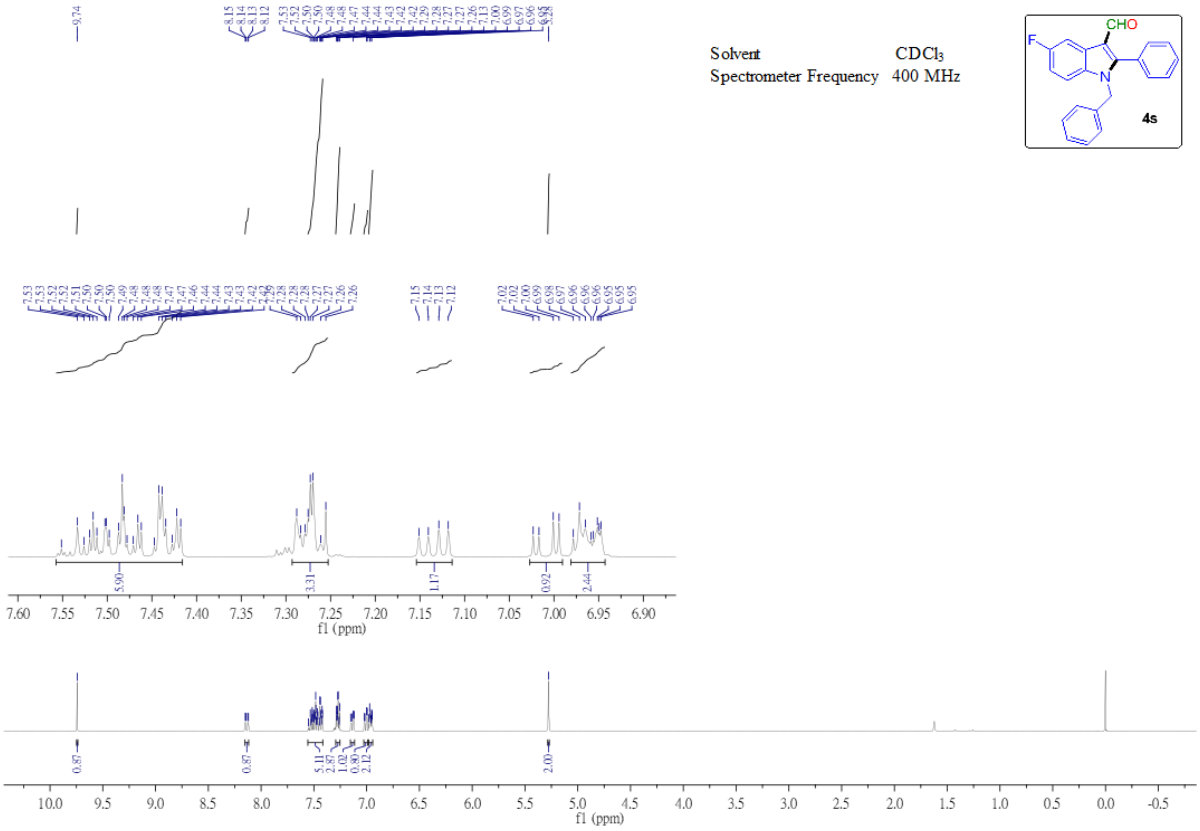

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
Cl
Cl
4p
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.927
151.553
136.660
134.277
132.675
132.599
130.625
130.310
129.662
128.957
128.137
127.927
127.831
125.495
124.647
123.827
122.587
116.571
110.402
45.281
X : parts per Million : Carbon13

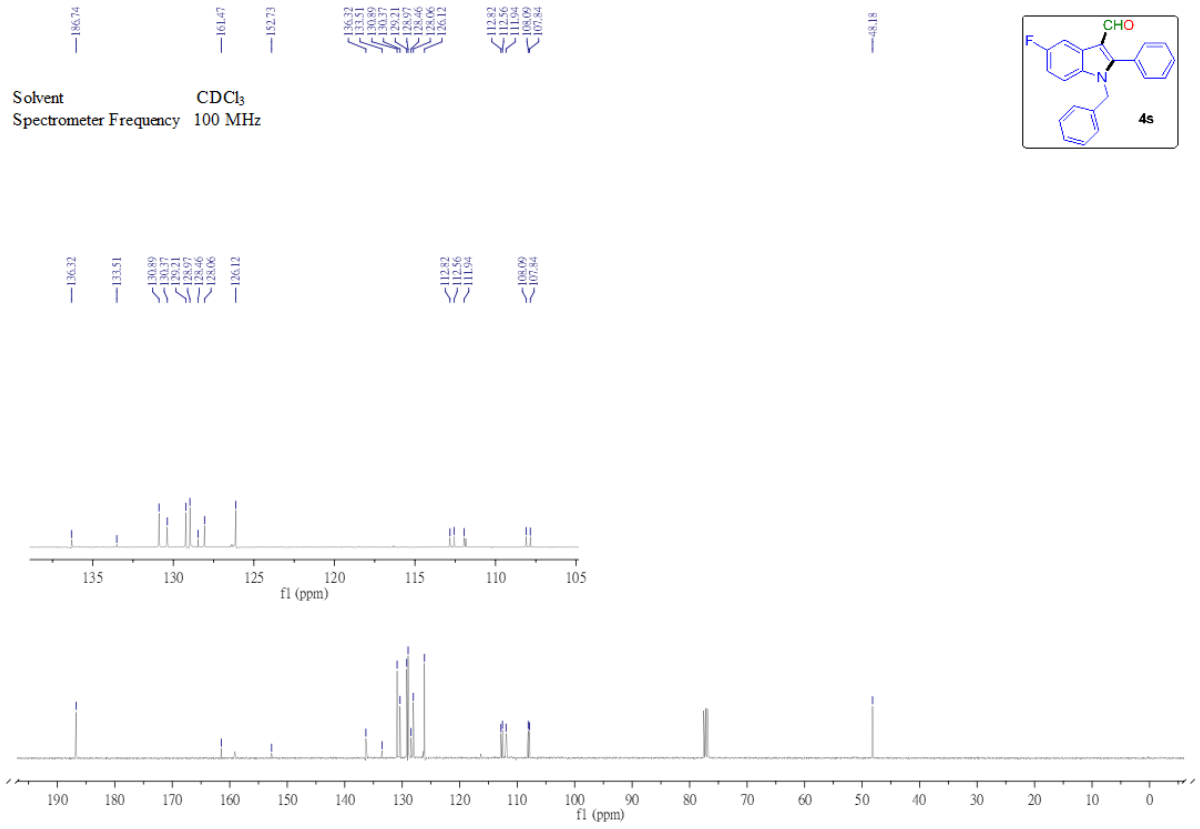

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
N
Br
4q
f1 (ppm)

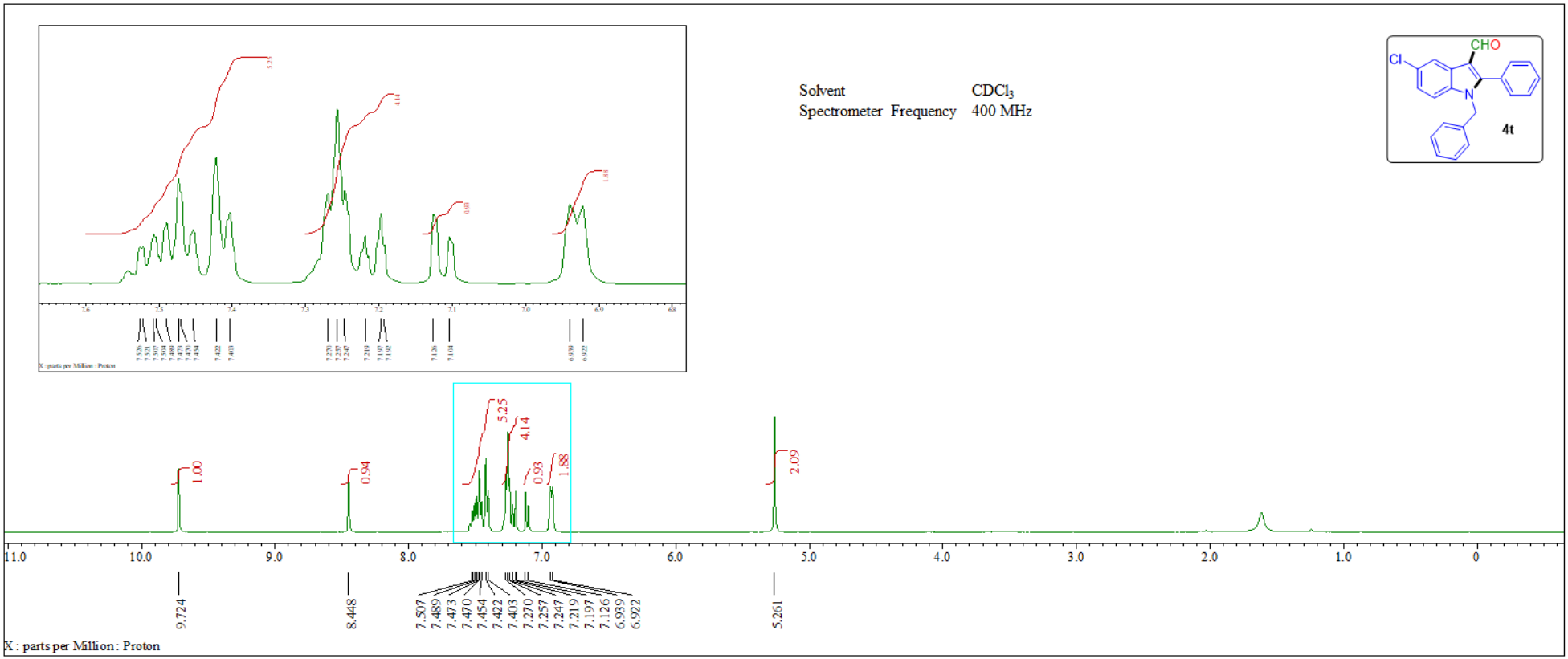

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
N
Br
4q
f1 (ppm)

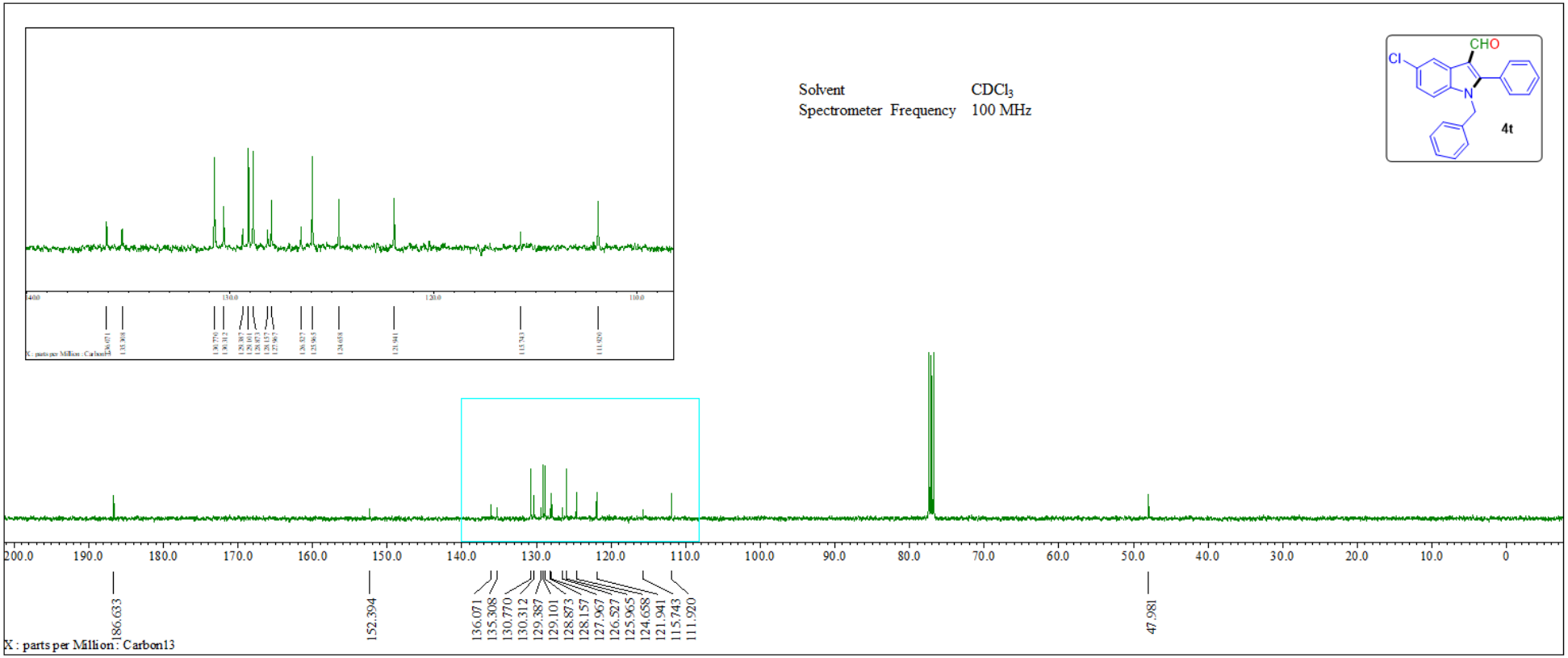

Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
4r
F
f1 (ppm)

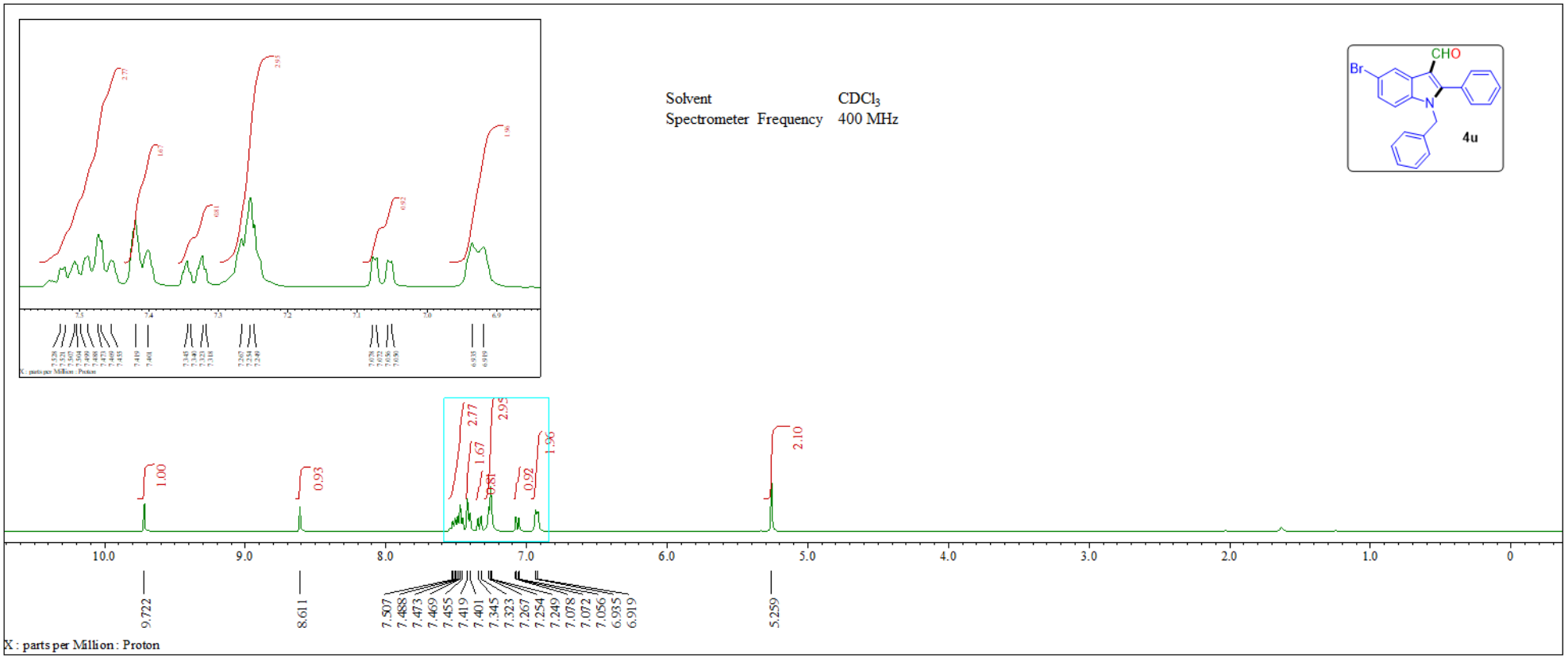

Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
4r
F
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 400 MHz
4s
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 100 MHz
4s
f1 (ppm)


Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
Cl
N
4t
1.00
0.94
5.25
4.14
0.93
1.88
2.09
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.724
8.448
7.507
7.489
7.473
7.470
7.454
7.422
7.403
7.270
7.257
7.247
7.219
7.197
7.126
6.939
6.922
5.261
X : parts per Million : Proton


Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
Cl
N
4t
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
186.633
152.394
136.071
135.308
130.770
130.312
129.387
129.101
128.873
128.157
127.967
126.527
125.965
124.658
121.941
115.743
111.920
47.981
X : parts per Million : Carbon13


Solvent CDCl3
Spectrometer Frequency 400 MHz
CHO
Br
N
4u
1.00
0.93
2.77
2.95
1.67
0.81
0.92
1.96
2.10
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
9.722
8.611
7.507
7.488
7.473
7.469
7.455
7.419
7.401
7.345
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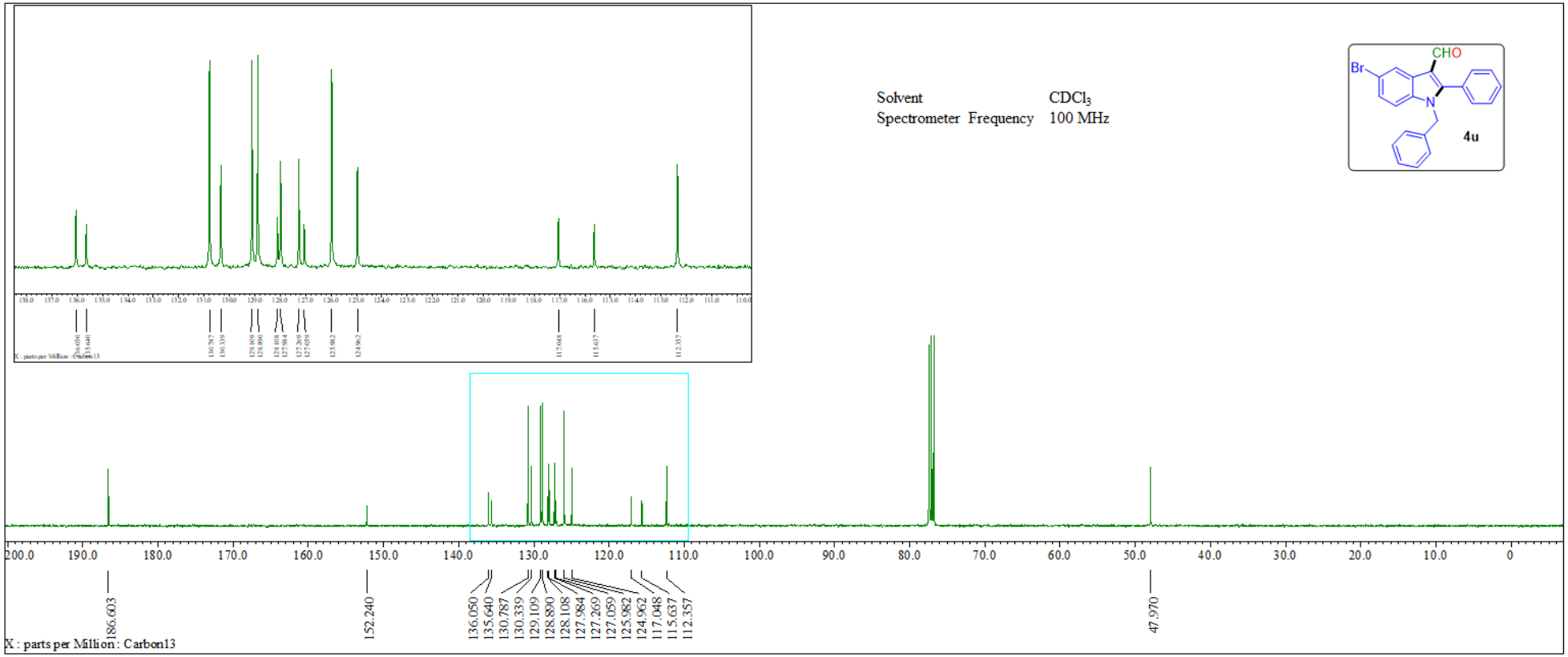
7.249
7.078
7.072
7.056
6.935
6.919
5.259
X : parts per Million : Proton


Solvent CDCl3
Spectrometer Frequency 100 MHz
CHO
Br
N
4u
186.603
152.240
136.050
135.640
130.787
130.339
129.109
128.890
128.108
127.584
127.269
127.059
125.982
124.962
117.048
115.637
112.357
47.970
X : parts per Million : Carbon13

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) rp101521

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: rp101521

```
Bond precision:  C-C = 0.0024 A          Wavelength=0.71073

Cell:            a=22.672(2)     b=14.2171(16)      c=6.8368(6)
                 alpha=90        beta=90            gamma=90
Temperature:     150 K
```

	Calculated	Reported
Volume	2203.7(4)	2203.7(4)
Space group	P b c n	P b c n
Hall group	-P 2n 2ab	-P 2n 2ab
Moiety formula	C15 H11 N O	C15 H11 N O
Sum formula	C15 H11 N O	C15 H11 N O
Mr	221.25	221.25
Dx,g cm-3	1.334	1.334
Z	8	8
Mu (mm-1)	0.084	0.084
F000	928.0	928.0
F000’	928.38	
h,k,lmax	31,19,9	31,18,8
Nref	3030	2591
Tmin,Tmax	0.985,0.989	0.984,1.000
Tmin’	0.967	

```
Correction method= # Reported T Limits: Tmin=0.984 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.855          Theta(max)= 29.303

R(reflections)= 0.0519( 1928)     wR2(reflections)= 0.1344( 2591)

S = 1.031                 Npar= 155
```

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level A

PLAT183_ALERT_1_A Missing _cell_measurement_reflns_used Value .... Please Do !
PLAT184_ALERT_1_A Missing _cell_measurement_theta_min Value ..... Please Do !
PLAT185_ALERT_1_A Missing _cell_measurement_theta_max Value ..... Please Do !

Alert level C

PLAT906_ALERT_3_C Large K Value in the Analysis of Variance ..... 6.555 Check

Alert level G

PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..... 1 Report
PLAT093_ALERT_1_G No s.u.’s on H-positions, Refinement Reported as mixed Check
PLAT720_ALERT_4_G Number of Unusual/Non-Standard Labels ..... 1 Note
PLAT898_ALERT_4_G Second Reported H-M Symbol in CIF Ignored ..... ! Check
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min). 2 Note
PLAT912_ALERT_4_G Missing # of FCF Reflections Above STh/L= 0.600 386 Note
PLAT953_ALERT_1_G Reported (CIF) and Actual (FCF) Hmax Differ by . 1 Units
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 14 Info

3 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
1 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
8 **ALERT level G** = General information/check it is not something unexpected

5 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
2 ALERT type 3 Indicator that the structure quality may be low
3 ALERT type 4 Improvement, methodology, query or suggestion
1 ALERT type 5 Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 20/08/2018; check.def file version of 20/08/2018

Datablock rp101521 - ellipsoid plot