Supplementary Information

2D-MoS_2 photocatalyzed Cross Dehydrogenative Coupling reaction synchronized with Hydrogen Evolution Reaction

*Yarabhally R Girish, Komal Jaiswal, Pranav Prakash and Mrinmoy De**

Department of Organic Chemistry, Indian Institute of Science, Bangalore 560 012, Karnataka, India

*To whom correspondence should be addressed. E-mail: md@iisc.ac.in

Contents:

1. General procedure for the preparation of *N*-Phenyl-1,2,3,4-tetrahydroisoquinoline.

Briefly Copper (I) iodide (0.21g, 1.13 mmol) and potassium phosphate (4.8 g, 22.6 mmol) were taken into the RB flask filled with argon baloon. 2-Propanol (10 mL), ethylene glycol (1.24 mL, 22.6 mmol), 1,2,3,4-tetrahydro-isoquinoline (2 g, 11.3 mmol) and iodobenzene (1.26 mL, 11.3 mmol) were added successively at room temperature. The reaction mixture was heated at 90 °C for 24 h and then allowed to cool to room temperature. Dichloromethane (30 mL) and water (25 mL) were then added to the reaction mixture and extracted with dichloromethane (3x 30 mL). The organic layer was dried over sodium sulfate. The solvent was concentrated under reduced pressure and purified by column chromatography on silica gel (hexane/ethyl acetate=95:5), to give the desired product 3a with 40% isolated yields.

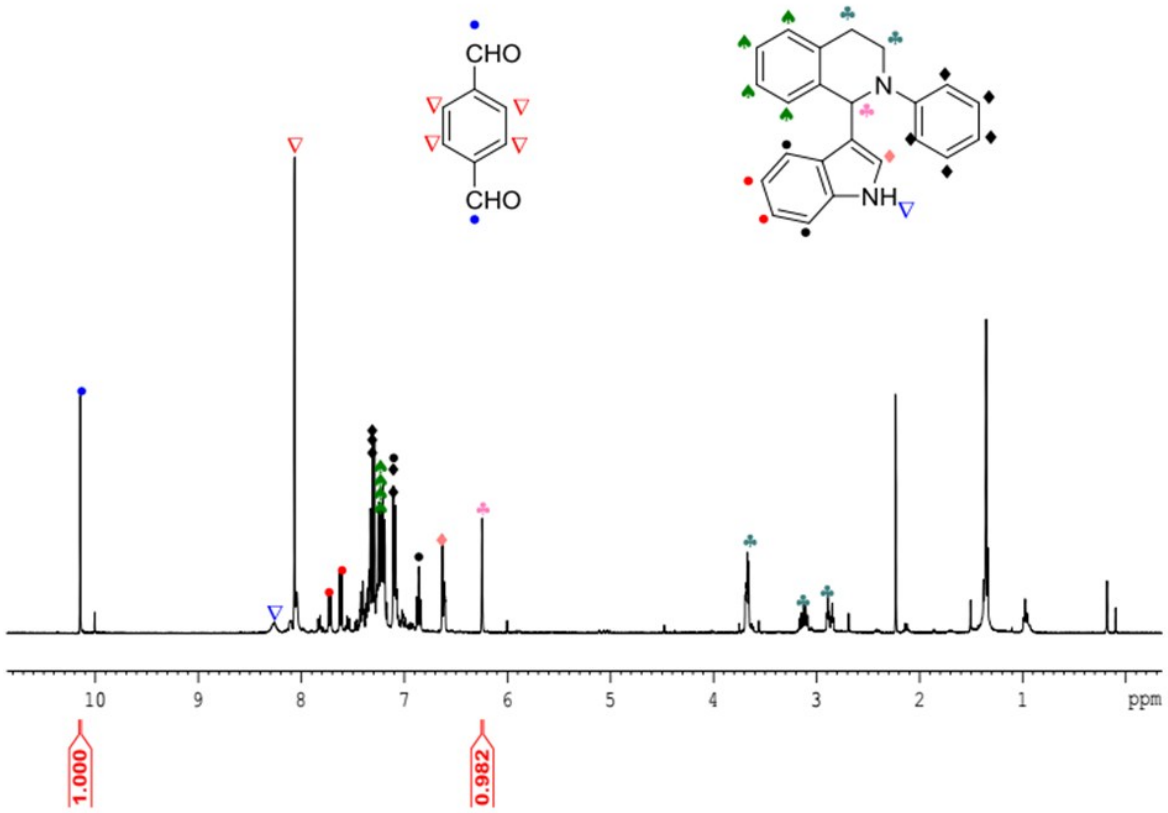


Figure S1: Crude ^{1}H NMR spectra of reaction mixture in presence of terephthalaldehyde as reference.

2. Table S1. Solvent ratio optimization

$MoS_2/H_2O/CH_3CN$ (v/v mL)	1:2:0	1.25:2:0	1.25: 2:1	1.25:2:0.5
Yield	90	72	98	91

3. Detection of H_2 gas formation during CDC reaction.

For H_2 gas detection, reactions were carried out under argon atmosphere. The RB flask containing reaction mixture was sealed and then the reaction mixture was irradiated for 5 h by a 525 nm green LED at room temperature under argon atmosphere. The H_2 gas evolved during the reaction was injected into an argon-purged chamber. From this chamber the gas flows through the leak valve to ultra-high vacuum (~10^{-9} torr) chamber, which is connected to mass spectrometry (SRS RGA 100) where mass of the gas can be detected. In addition to this as a control experiment, we have also performed the reaction in absence of light and we found that there is no H_2 gas formation which confirms that light is essential for the CDC reaction.

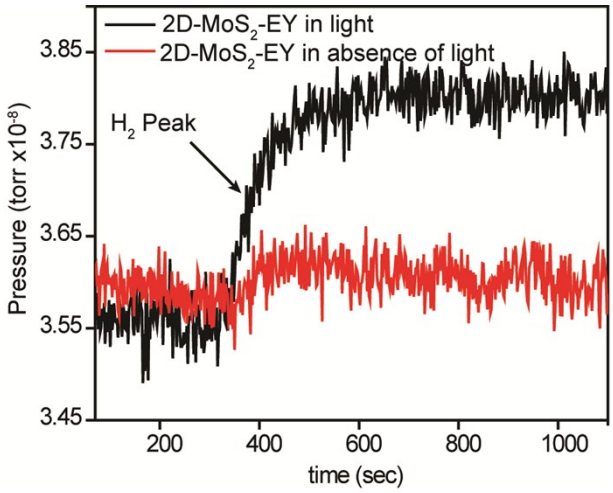
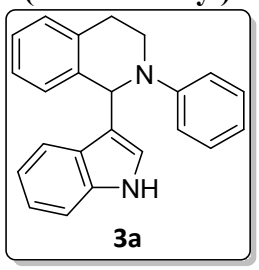


Figure S2: Hydrogen evolution of 2D-MoS_2-EY under light in presence of *N*-Phenyl-1,2,3,4-tetrahydroisoquinoline and in absence of light.

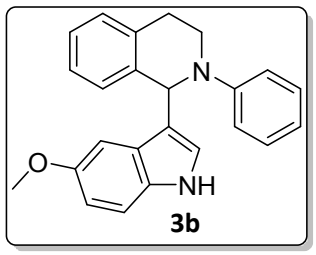
4. Characterization data for CDC reaction of indole derivatives with *N*-pheny-l ,2,3,4-tetrahydroisoquinoline derivatives.

1-(1H-Indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3a)[2]; (Pale yellow solid); *Rf* (20% EtOAc/Hexane) 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.92 (s, 1H), 7.55 - 7.53 (d, *J* = 7.6 Hz, 1H), 7.31 - 7.29 (d, *J* = 8.0 Hz, 1H), 7.27 - 7.20 (m, 3H), 7.16 - 7. 15 (m, 4H), 7.03- 7.00 (m, 3H), 6.78 - 6.74 (t, *J* =7.4 Hz, 1H), 6.63 -6.62 (d, *J* = 2.4 Hz, 1H), 6.16 (s, 1H), 3.63 - 3.60 (m, 2H), 3.09 - 3.02 (m, 1H), 2.82 - 2.76 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 149.7, 137.3, 136.5, 135.5, 128.7, 128.0, 126.6, 126.4, 125.6, 124.1, 122.0, 120.0, 119.5, 119.2, 118.0, 115.7, 111.0, 56.5, 42.2, 26.5; HRESI-MS (m/z): Calculated for $C_{23}H_{20}N_2$ (M + H): 325.1705, Found (M + H): 325.1703



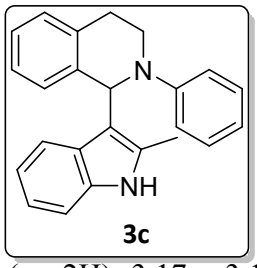
3a

1-(1-methyl-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3b)[2]; Pale yellow solid; *Rf* (20% EtOAc/Hexane) 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.81 (s, 1H), 7.24 - 7.19 (m, 4H), 7.17 - 7.15 (m, 3H), 7.02 - 7.00 (d, *J* = 8.0 Hz, 2H), 6.87 (s, 1H), 6.80- 6.76 (m, 2H), 6.57 (s, 1H), 6.13 (s, 1H), 3.64 (s, 3H), 3.60 - 3.57 (m, 2H), 3.10 - 3.02 (m, 1H), 2.83 - 2.77 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 153.3, 149.9, 137.4, 135.5, 131.5, 129.1, 128.7, 127.9, 126.8, 126.6, 125.6, 124.9, 118.6, 118.2, 116.1, 112.2, 111.5, 101.8, 56.7, 55.6, 42.0, 26.8; HRESI-MS (m/z): Calculated for $C_{24}H_{22}N_2O$ (M + H): 355.1732, Found (M + H): 339.1861.



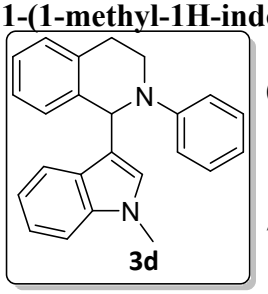
3b

1-(2-methyl-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3c)[4]: Orange solid; *Rf* (20% EtOAc/Hexane) 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.72 (s, 1H), 7.22 – 7.19 (m, 5H), 7.12 – 7.09 (m, 3H), 7.07 – 7.04 (m, 3H), 6.96 – 6.92 (t, *J* = 7.4 Hz, 1H), 6.89 – 6.86 (t, *J* = 7.2 Hz, 1H), 6.01 (s, 1H), 3.75 – 3.63 (m, 2H), 3.17 – 3.13 – 3.02 (m, 2H), 2.04 (t, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = δ 150.9, 137.9, 135.2, 134.8, 128.8, 128.65, 128.62, 128.2, 126.2, 126.0, 120.7, 120.2, 119.4, 119.43, 119.1, 113.3, 110.0, 57.1, 45.8, 29.6, 27.9; HRESI-MS (m/z): Calculated for $C_{24}H_{22}N_2$ (M + H): 338.1783, Found (M + H): 338.1780.



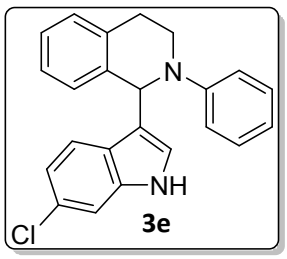
3c

1-(1-methyl-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3d)[4]: Yellow solid; *Rf* (20% EtOAc/Hexane) 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 7.59 (d, *J* = 8Hz, 1H), 7.36 – 7.35 (d, *J* = 6 Hz, 1H), 7.34 – 7.27 (m, 3H), 7.25 – 7.21 (m, 4H), 7.09 – 7.07 (t, *J* = 7.6 Hz, 3H), 6.85 – 6.81 (t, J = 7.2 Hz, 1H), 6.56(s, 1H), 6.24 (s, 1H), 3.71 – 3.70 (m, 5H), 3.17 – 3.09 (m, 1H), 2.92 – 2.84 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = δ 149.7, 137.6, 137.3, 135.5, 129.2, 128.8, 128.0, 126.8, 126.6, 125.6, 121.6, 120.1, 119.1, 117.9, 117.6, 115.6, 109.1, 56. 6, 42.1, 32.6, 26.6; HRESI-MS (m/z): Calculated for $C_{24}H_{22}N_2$ (M + H): 339.1861, Found (M + H): 339.1861.



3d

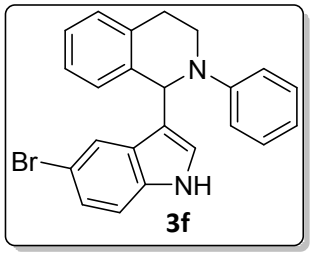
1-(6-chloro-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline(3e)[4]; (Pale yellow solid); *Rf* (20% EtOAc/Hexane) 0.6; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.93 (s, 1H), 7.39 – 7.37 (d, *J* = 8.8 Hz, 1H), 7.17 – 7.15 (m, 7H), 7.00 – 6.95 (m, 3H), 6.80 - 6.76 (t, *J* = 7.0 Hz, 1H), 6.59 (s, 1H), 6.10 (s, 1H), 3.58 – 3.54 (m, 2H), 3.06 – 3.01 (m, 1H), 2.79 - 2.75 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 149.7, 137.0, 136.8, 135.4, 129.1, 128.8, 128.0, 127.9, 126.7, 125.7, 125.0, 124.7, 121.0, 120.3, 119.4,



3e

118.4, 116.1, 110.8, 112.4, 56.6, 42.3, 29.6, 26.6; HRESI-MS (m/z): Calculated for $C_{23}H_{19}ClN_2$ (M + H): 358.1237, Found (M + H): 358.1235.

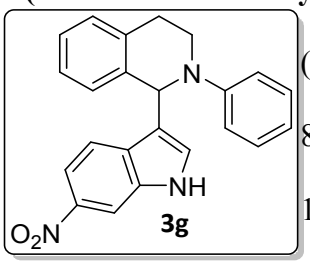
1-(5-bromo-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3f)[3]; (Pale yellow solid);



3f

Rf (20% EtOAc/Hexane) 0.5; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.94 (s, 1H), 7.59 (s, 1H), 7.24 - 7.17 (m, 4H), 7.17 – 7.15 (m, 4H), 7.00 - 6.99 (d, *J* = 8.0 Hz, 2H), 6.82- 6.78 (t, *J* = 7.2 Hz, *J* = 7.2 Hz, 1H), 6.66 (s, 1H), 6.06 (s, 1H), 3.59 – 3.55 (m, 2H), 3.08 – 2.97 (m, 1H), 2.81 - 2.76 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ ppm = 149.8, 136.9, 136.6, 135.3, 135.1, 129.1, 128.8, 128.0, 127.9, 126.7, 125.7, 125.3, 124.9, 122.6, 119.0, 118.7, 116.4, 112.9, 112.4, 56.6, 42.5, 26.5; HRESI-MS (m/z): Calculated for $C_{23}H_{19}BrN_2$ (M + H): 403.0732, Found (M + H): 403.0731.

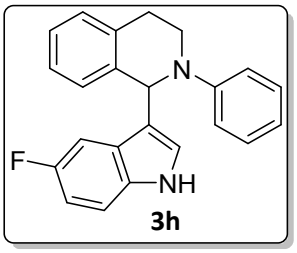
1-(6-nitro-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3g); (Pale yellow solid); *Rf*



3g

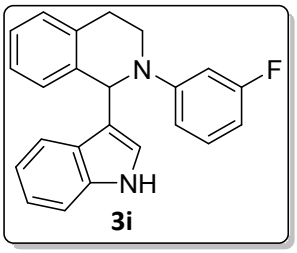
(20% EtOAc/Hexane) 0.6; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 8.60 (s, 1H), 8.30 (s, 1H), 7.92 – 7.90 (d, *J* = 8.8 Hz, 1H), 7.53 – 7.51 (d, *J* = 8.8 Hz, 1H), 7.26 – 7.22 (m, 6H), 7.05 – 7.03 (d, *J* = 8.0 Hz, 2H), 6.96 (s, 1H), 6.88 - 6.86 (t, *J* = 7.2 Hz, 1H), 6.17 (s, 1H), 3.63 – 3.55 (m, 2H), 3.13 – 3.07 (m, 1H), 2.86 - 2.81 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 149.7, 136.5, 135.4, 135.0, 131.1, 129.6, 129.2, 129.0, 127.8, 127.0, 125.9, 120.3, 120.1, 119.1, 116.7, 115.1, 107.9, 56.6, 42.6, 30.8, HRESI-MS (m/z): Calculated for $C_{23}H_{19}ClN_2$ (M + H): 369.1477, Found (M + H): 369.1479.

1-(5-fluoro-1H-indol-3-yl)-2-phenyl-1,2,3,4-tetrahydroisoquinoline (3h); (white solid); *Rf* (28% DCM/Hexane) 0.16; mp: 125-127°C (lit.6 118-120°C) ; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.95 (s, 1H), 7.22 – 7.15 (m, 5H), 7.14 (m, 3H), 7.01 – 6.99 (d, *J* = 8.0 Hz, 3H), 6.90 – 6.86 (t, *J* = 8.8 Hz, 1H), 6.80 – 6.76 (t, *J* = 7.2 Hz, 1H), 6.66 (s, 1H), 6.60 (s, 1H), 3.59 - 3.56 (m, 2H), 3.07 – 3.01 (m, 1H), 2.80 - 2.76 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 149.6, 136.9, 135.3, 133.0, 129.2, 128.8, 128.0, 127.9, 126.7, 125.8, 125.7, 118.7, 116.3, 111.4, 110.6, 110.3, 105.1, 104.9, 56.8, 42.5, 26.6; HRESI-MS (m/z): Calculated for $C_{23}H_{19}FN_2$ (M + H): 343.1611, Found (M + H): 343.1613.



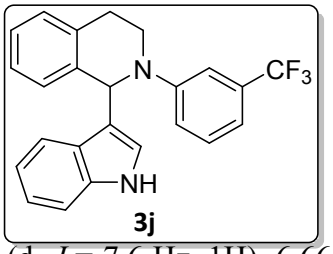
3h

2-(3-fluorophenyl)-1-(1H-indol-3-yl)-1,2,3,4-tetrahydroisoquinoline (3i); (white solid); *Rf* (24% DCM/Hexane) 0.4; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.94 (s, 1H), 7.56 - 7.54 (d, *J* = 8.0 Hz, 1H), 7.33 - 7.31 (d, *J* = 8.4 Hz, 2H), 7.24 – 7.12 (m, 5H), 7.06 – 7. 04 (t, *J* = 8.0 Hz, 1H), 6.79- 6.76 (dd, *J* = 2.4 Hz, *J* = 2.4 Hz, 1H), 6.69 – 6.66 (d, *J* =14 Hz, 2H), 6.45 – 6.41 (dt, *J* = 2.4 Hz, *J* = 2.0 Hz, *J* = 2.4 Hz, 1H), 6.14 (s, 1H), 3.63 - 3.58 (m, 2H), 3.09 - 3.05 (m, 1H), 3.04 – 3.02 (m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ ppm = 136.9, 136.5, 135.3, 130.2, 130.1, 128.7, 127.9, 126.8, 126.1, 125.8, 124.0, 122.2, 119.8, 119.7, 118.8, 111.0, 110.4, 101.9, 101.6, 77.3, 76.9, 76.6, 56.4, 42.1, 26.5; HRESI-MS (m/z): Calculated for $C_{23}H_{19}FN_2$ (M + H): 343.1611, Found (M + H): 343.1613.



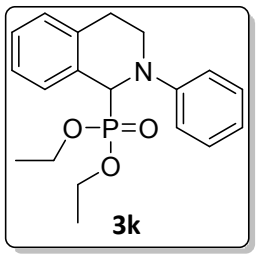
3i

1-(1H-indol-3-yl)-2-(3-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroisoquinoline (3j); (white solid); *Rf* (30% DCM/Hexane) 0.7; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.92 (s, 1H), 7.54 - 7.52 (d, *J* = 8.0 Hz, 1H), 7.33 - 7.31 (d, *J* = 8.0 Hz, 3H), 7.24 – 7.16 (m, 6H), 7.07 – 7. 03 (t, *J* = 7.6 Hz, 1H), 7.00- 6.99 (d, *J* = 7.6 Hz, 1H), 6.66 (s, 1H), 6.20 (s, 1H), 3.70 - 3.62 (m, 2H), 3.11 - 3.03 (m, 1H), 2.90 - 2.84(m, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ ppm = 149.6, 136.9, 136.6, 135.2, 129.6, 128.8, 127.9, 126.9, 126.1, 125.9, 124.1, 122.2, 119.7, 118.5, 118.0, 111.1, 56.5, 42.2, 29.7 ; HRESI-MS (m/z): Calculated for $C_{24}H_{19}F_3N_2$ (M+): 392.1500, Found (M+): 391.1499.



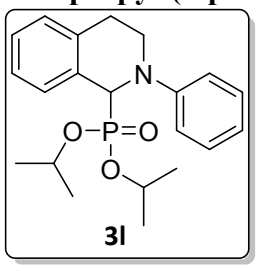
3j

Diethyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (3k); : Red oily liquid; Rf (30% EtOAc/Hexane) 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = δ 7.37 – 7.36 (d, *J* = 6.8 Hz, 1H), 7.26 – 7.13 (m, 5H), 6.98 – 6.96 (d, *J* = 8.0 Hz, 2H), 6.80 – 6.77 (t, *J* = 7.2 Hz, 1H), 5.21 – 5.16 (d, *J* = 20 Hz, 1H), 4.09 – 3.88 (m, 5H), 3.64 – 3.61 (m, 1H), 3.05 – 3.00 (m, 2H), 1.26 – 1.22 (t, *J* = 7.0 Hz, 3H), 1.15 – 1.11 (t, *J* = 7.0 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 149.4 (d, *J* = 6 Hz), 136.3 (d, *J* = 5.3 Hz), 130.5, 129.1, 128.7, 128.1, 128.0, 127.44, 127.40, 125.86, 125.83, 118.4, 114.7, 63.38 – 63.30 (d, *J* = 32 Hz), 62.39 – 62.31 (d, *J* = 31.6 Hz), 59.5 – 57.9 (d, *J* = 158.3 Hz), 43.4, 26.7, 16.43 – 16.35 (d, *J* = 7.9 Hz), 16.35 – 16.29 (d, *J* = 5.8 Hz); HRESI-MS (m/z): Calculated for $C_{19}H_{24}NO_3P$ (M + Na): 368.1391, Found (M + Na): 368.1392.



3k

Diisopropyl (2-phenyl-1,2,3,4-tetrahydroisoquinolin-1-yl)phosphonate (3l) : Red oily liquid; Yield - 74%; Rf (30% EtOAc/Hexane) 0.2; **^{1}H NMR** (400 MHz, $CDCl_3$): δ = δ 7.36 – 7.34 (d, *J* = 6.8 Hz, 1H), 7.34 – 7.12 (m, 5H), 6.97 – 6.95 (d, *J* = 8.0 Hz, 2H), 6.79 – 6.75 (t, *J* = 7.2 Hz, 1H), 5.19 – 5.14 (d, *J* = 20 Hz, 1H),



3l

4.08 – 3.96 (m, 2H), 3.91 – 3.84 (m, 1H), 3.64 – 3.58 (m, 1H), 3.04 – 2.97 (m, 2H), 1.41 – 1.39 (d, *J* = 8.0 Hz, 2H), 1.27 – 1.21 (m, 7H), 1.12 – 1.10 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$): δ 149.37 – 149.31 (d, *J* = 5.8 Hz), 136.4 – 136.3 (d, *J* = 5.6 Hz), 136.4 (d, *J* = 5.6 Hz), 130.5, 129.0, 128.7, 128.6, 128.0,128.05, 127.4, 127.3, 125.83, 125.80, 118.4, 114.7, 63.3 – 63.2 (d, *J* = 7.2 Hz), 62.3 – 62.2 (d, *J* = 7.7 Hz),59.5, 57.9, 43.4, 31.5, 30.2, 29.6, 26.7, 16.4, 16.3, 16.2; HRESI-MS (m/z): Calculated for $C_{21}H_{28}NO_3P$ (M + Na): 396.1704, Found (M + Na): 396.1705.

References:

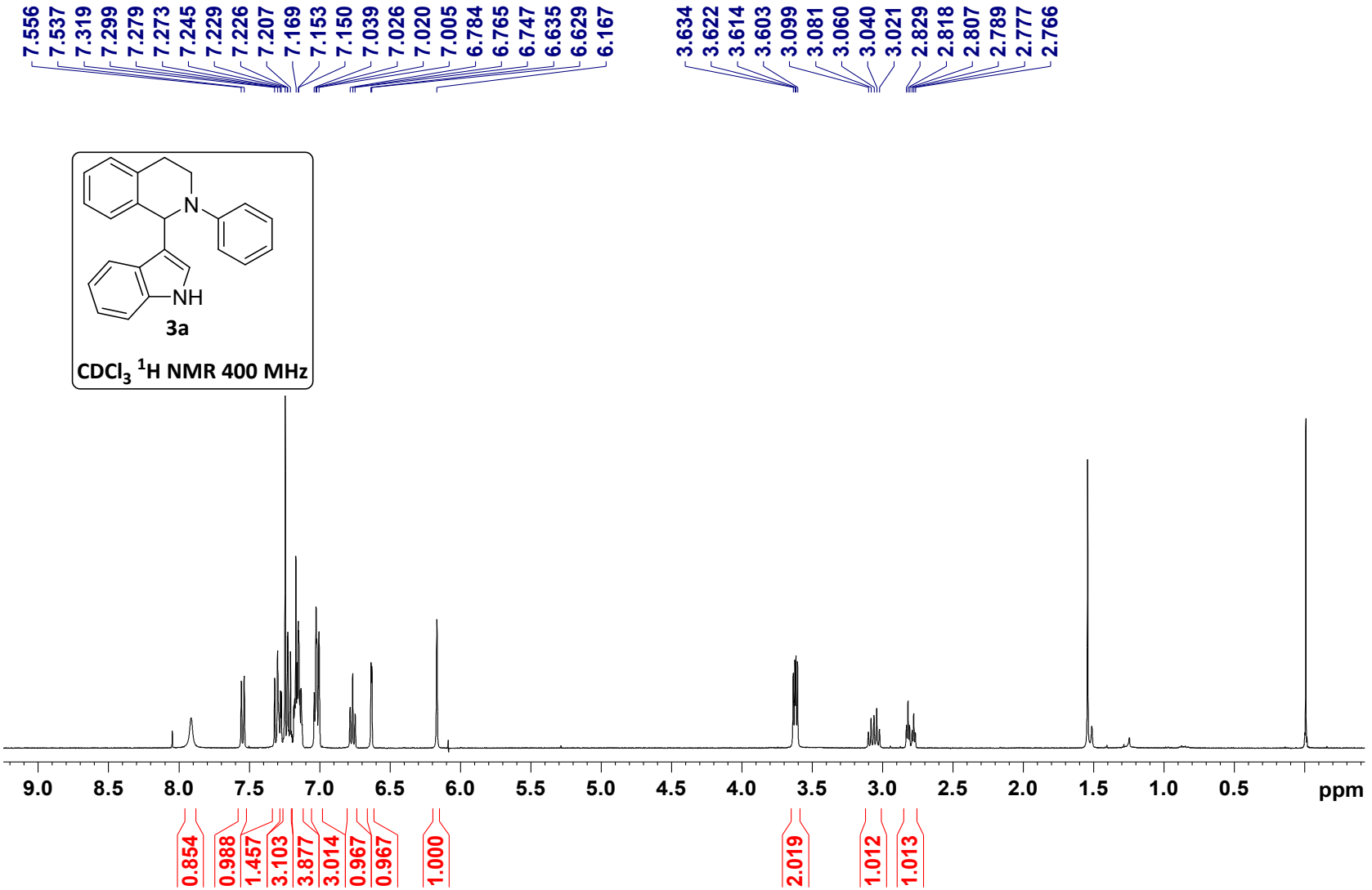
[1] Liu, L.; Zhang, S.; Fu, X.; Yan, C.H., *Chem. Commun*. **2011**, 47, 10148-10150.

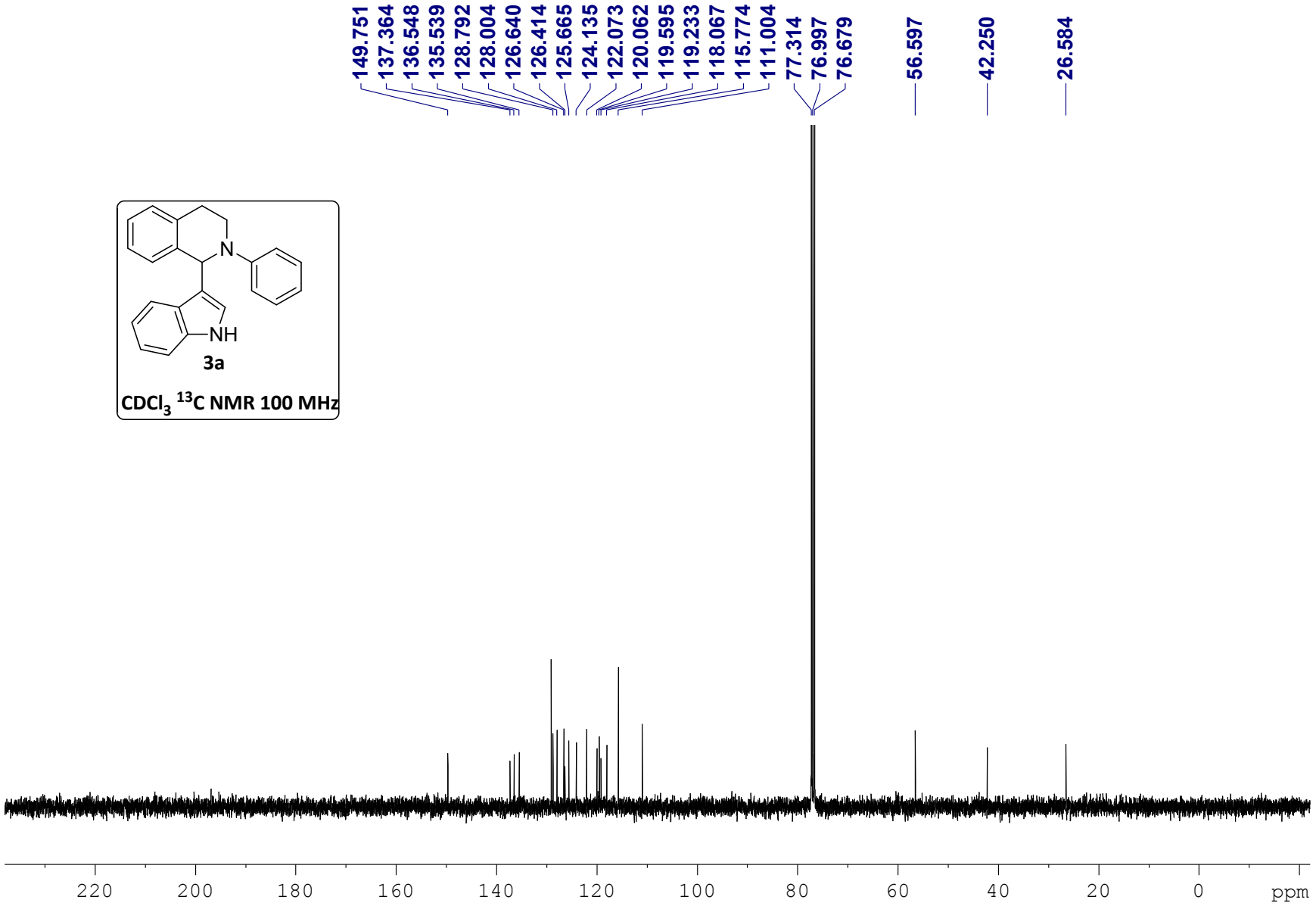
[2] Li, Z.; Li, C. J. *J. Am. Chem. Soc*. **2005**, 127, 6968-6969.

[3] Alagiri, K.; Prabhu, K. R. *Org. Biomol. Chem.* **2012**, *10*, 835.

[4] Zhong, J-J.; Meng, Q-Y.; Liu, B.; Li, X-B.; Gao, X-W.; Lei, T.; Wu, C. J.; Li, Z-J.; Tung, C-H.; Wu, L-Z. *Org. Lett.* **2014** *16* (7), 1988-1991

5. NMR spectral data:

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118.067
115.774
111.004
77.314
76.997
76.679
56.597
42.250
26.584
N
NH
3a
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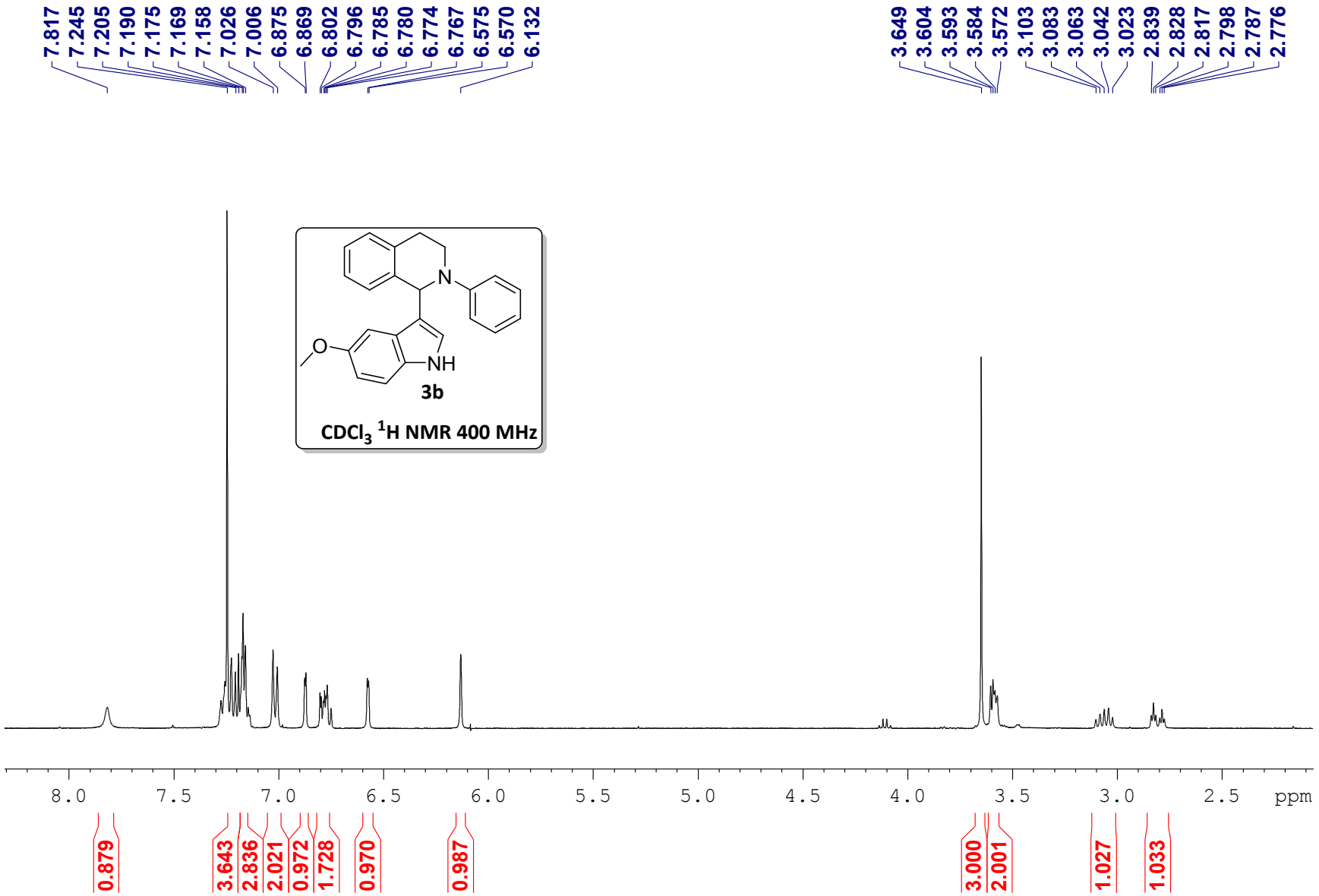
200
180
160
140
120
100
80
60
40
20
0
ppm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7.205
7.190
7.175
7.169
7.158
7.026
7.006
6.875
6.869
6.802
6.796
6.785
6.780
6.774
6.767
6.575
6.570
6.132
3.649
3.604
3.593
3.584
3.572
3.103
3.083
3.063
3.042
3.023
2.839
2.828
2.817
2.798
2.787
2.776
N
O
NH
3b
CDCl3 1H NMR 400 MHz
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
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2.021
0.972
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0.970
0.987
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2.001
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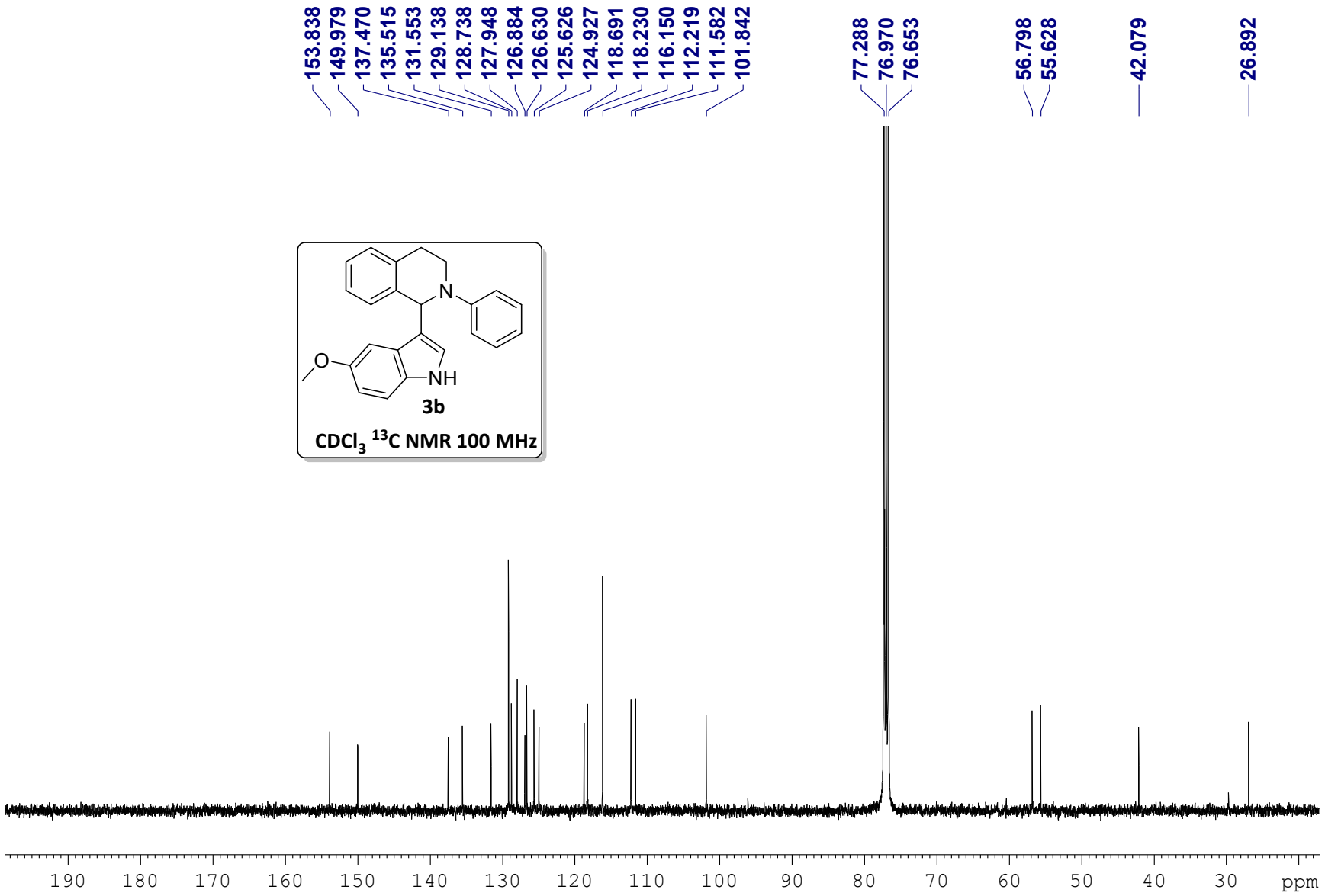
1.033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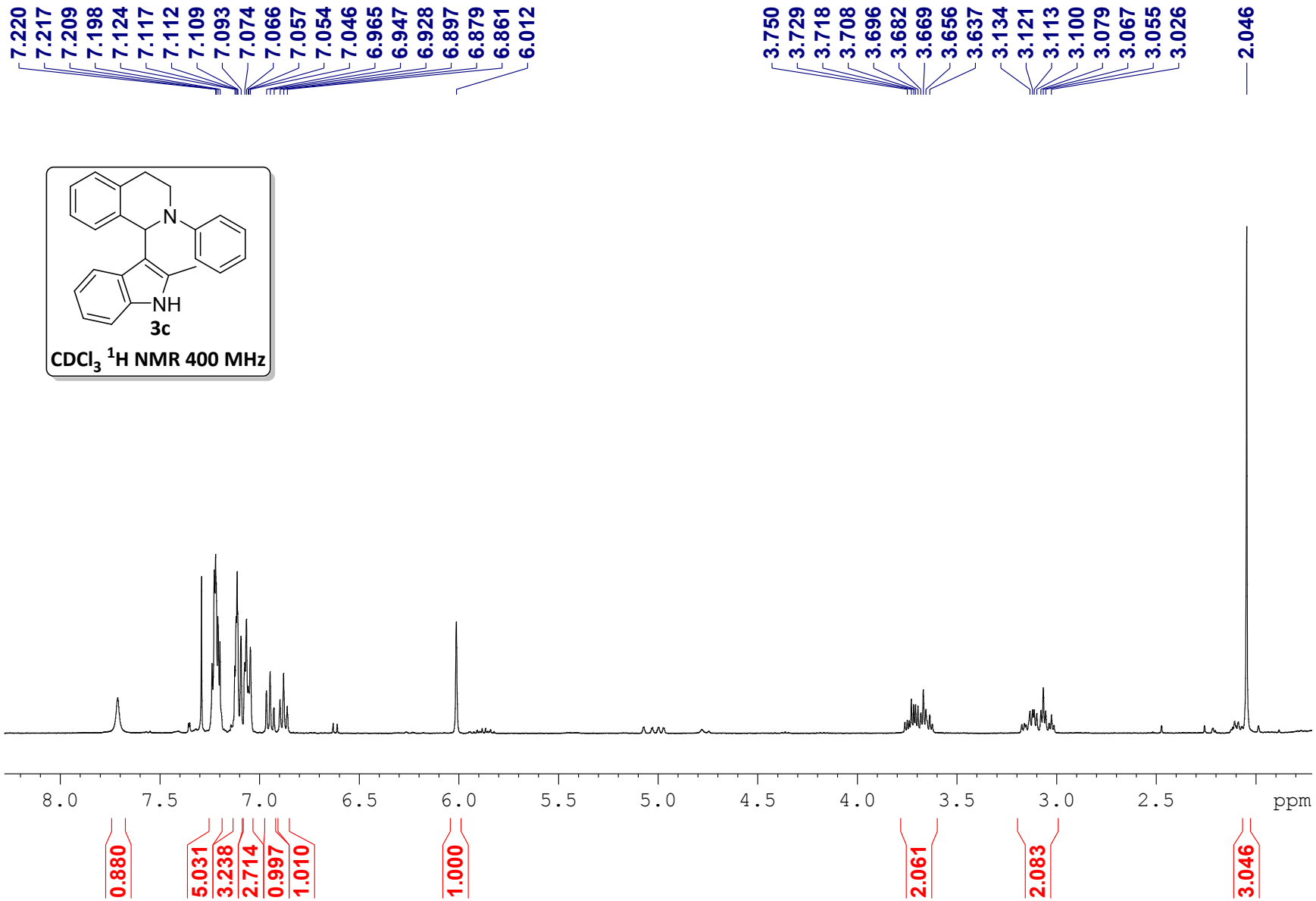
118.691
118.230
116.150
112.219
111.582
101.842
77.288
76.970
76.653
56.798
55.628
42.079
26.892
N
O
NH
3b
CDCl3 13C NMR 100 MHz
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm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7.217
7.209
7.198
7.124
7.117
7.112
7.109
7.093
7.074
7.066
7.057
7.054
7.046
6.965
6.947
6.928
6.897
6.879
6.861
6.012
3.750
3.729
3.718
3.708
3.696
3.682
3.669
3.656
3.637
3.134
3.121
3.113
3.100
3.079
3.067
3.055
3.026
2.046
N
NH
3c
CDCl3 1H NMR 400 MHz
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
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5.031
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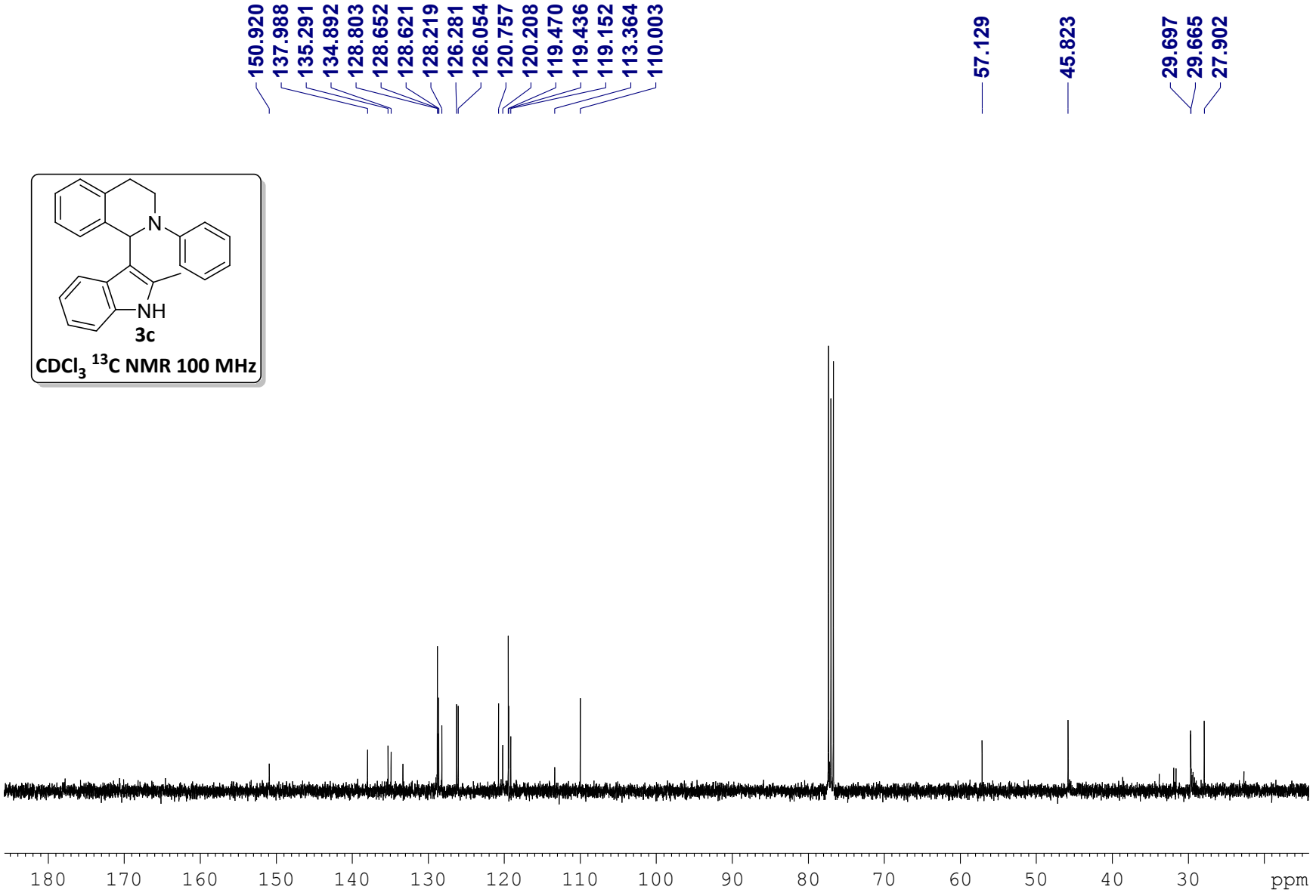
2.714
0.997
1.010
1.000
2.061
2.083
3.046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135.291
134.892
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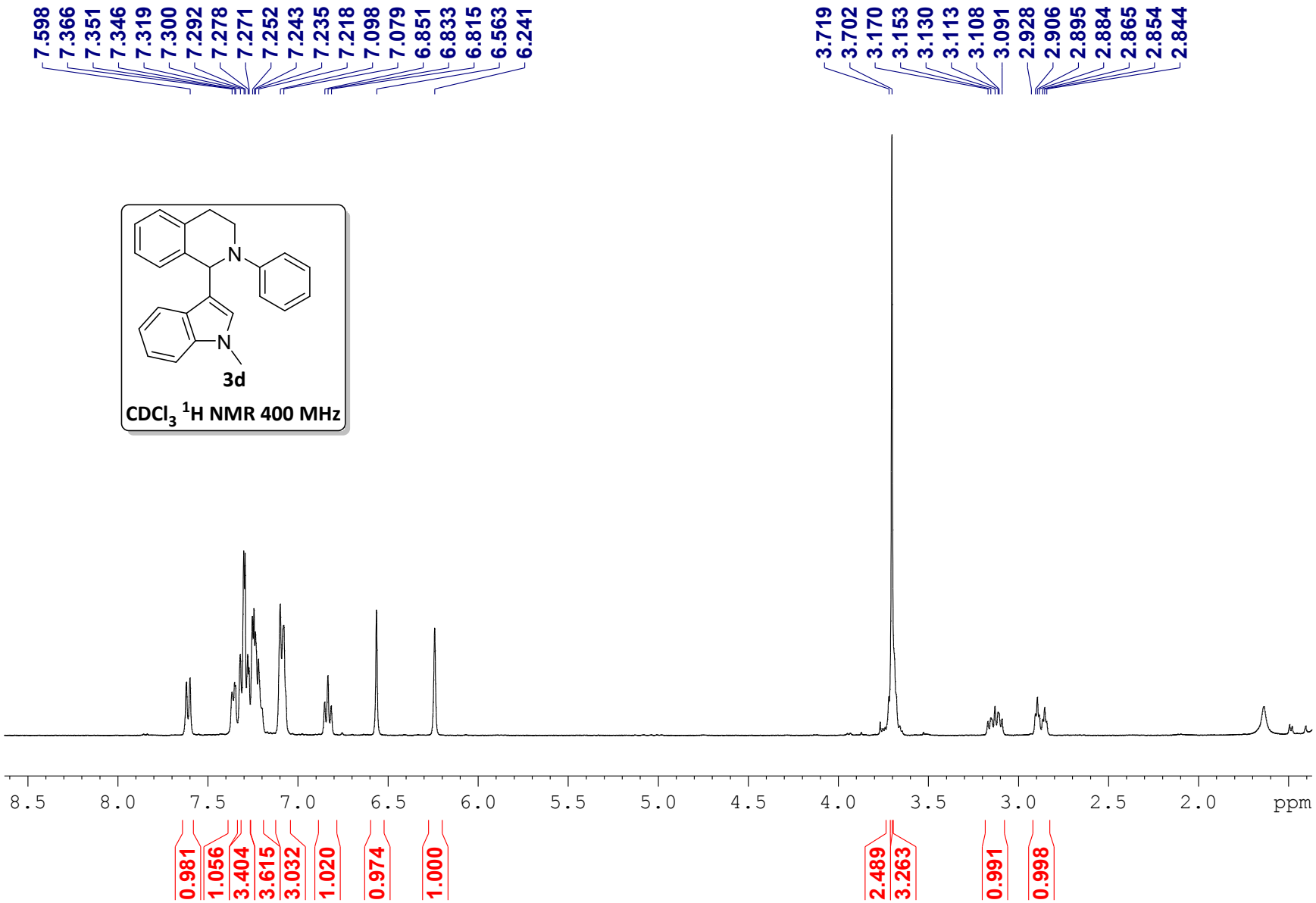
128.621
128.219
126.281
126.054
120.757
120.208
119.470
119.436
119.152
113.364
110.003
57.129
45.823
29.697
29.665
27.902
N
NH
3c
CDCl3 13C NMR 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm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7.366
7.351
7.346
7.319
7.300
7.292
7.278
7.271
7.252
7.243
7.235
7.218
7.098
7.079
6.851
6.833
6.815
6.563
6.241
3.719
3.702
3.170
3.153
3.130
3.113
3.108
3.091
2.928
2.906
2.895
2.884
2.865
2.854
2.844
N
N
3d
CDCl3 1H NMR 400 MHz
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
ppm
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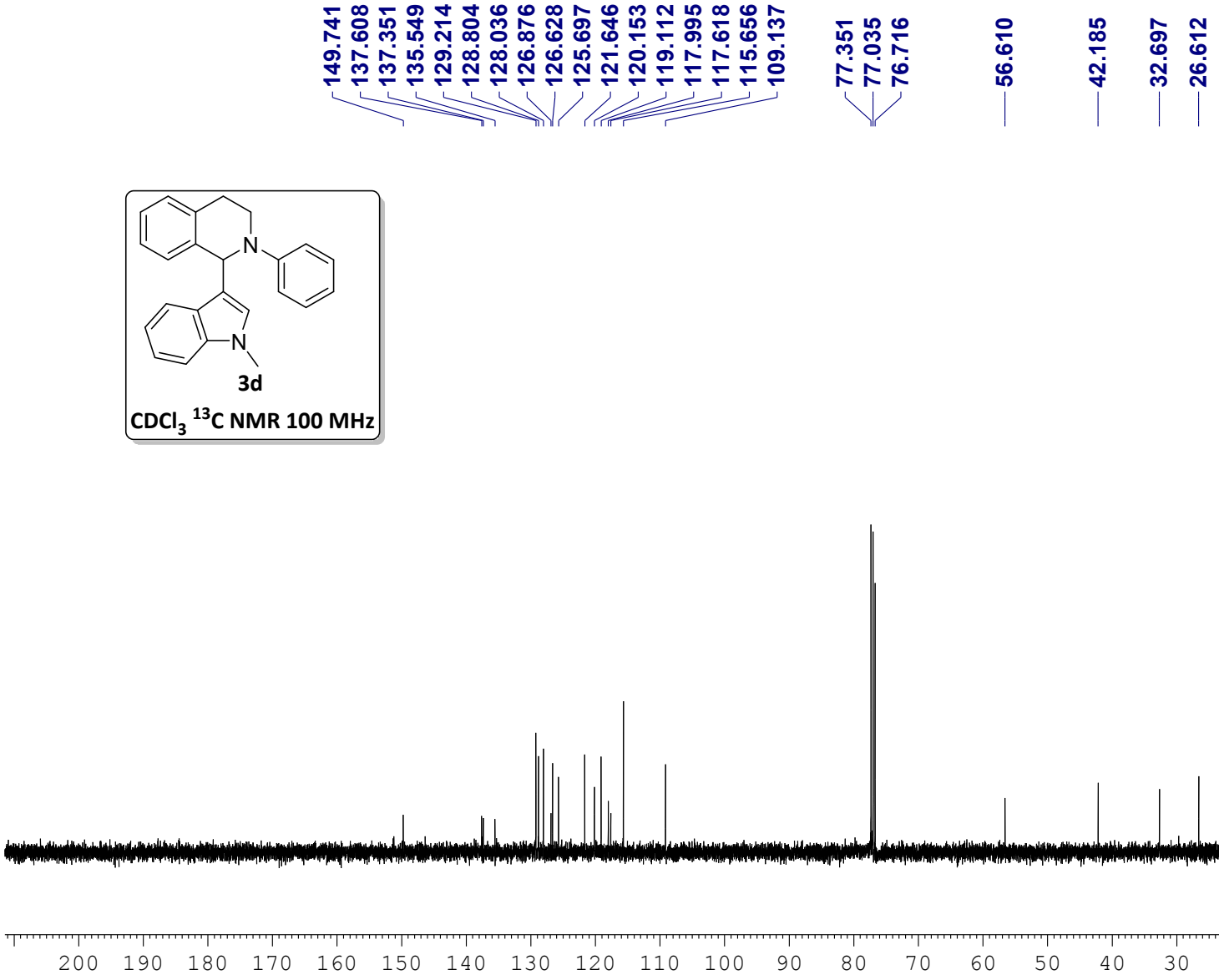
3.615
3.032
1.020
0.974
1.000
2.489
3.263
0.991
0.998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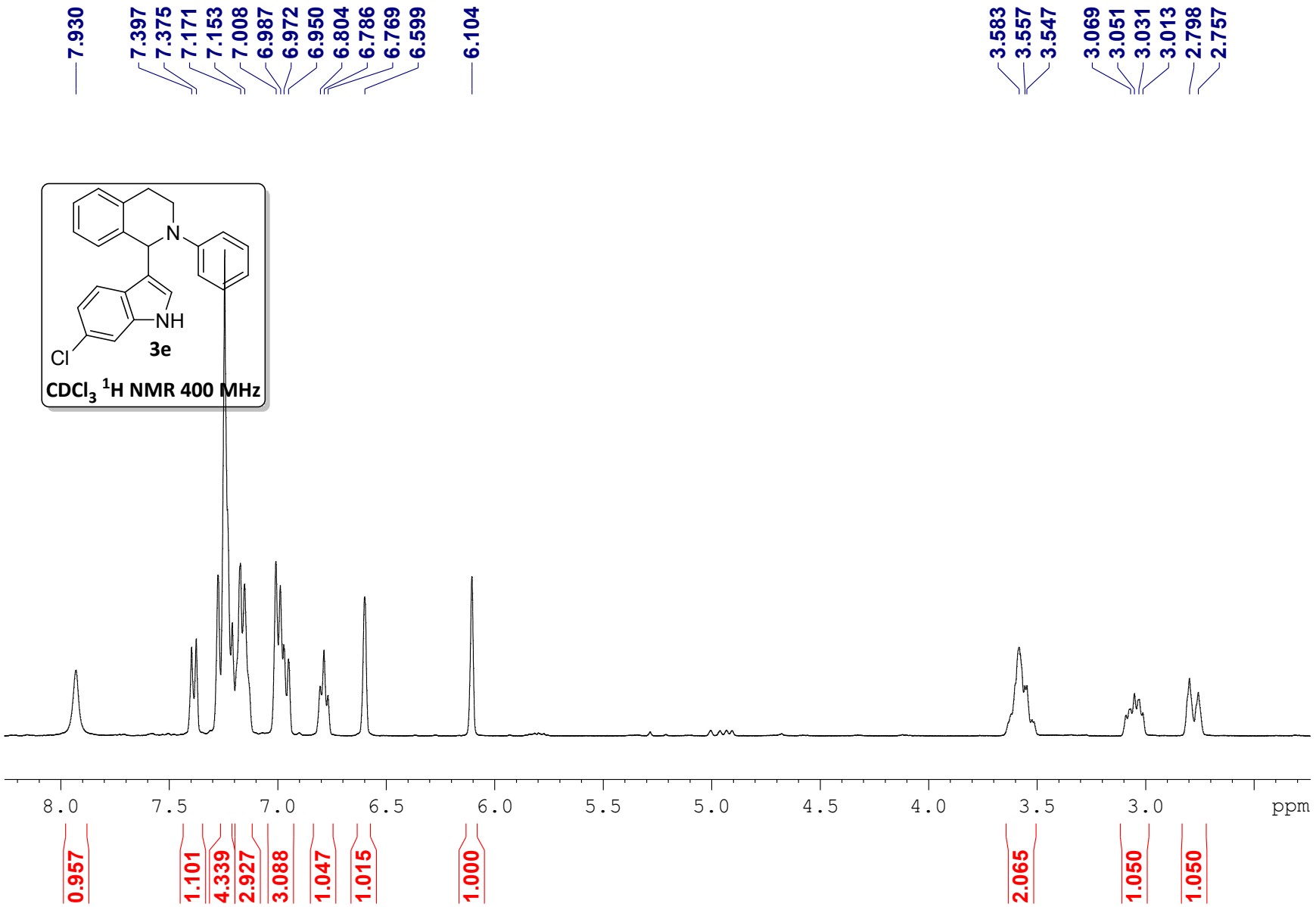
137.351
135.549
129.214
128.804
128.036
126.876
126.628
125.697
121.646
120.153
119.112
117.995
117.618
115.656
109.137
77.351
77.035
76.716
56.610
42.185
32.697
26.612
N
N
3d
CDCl3 13C NMR 100 MHz
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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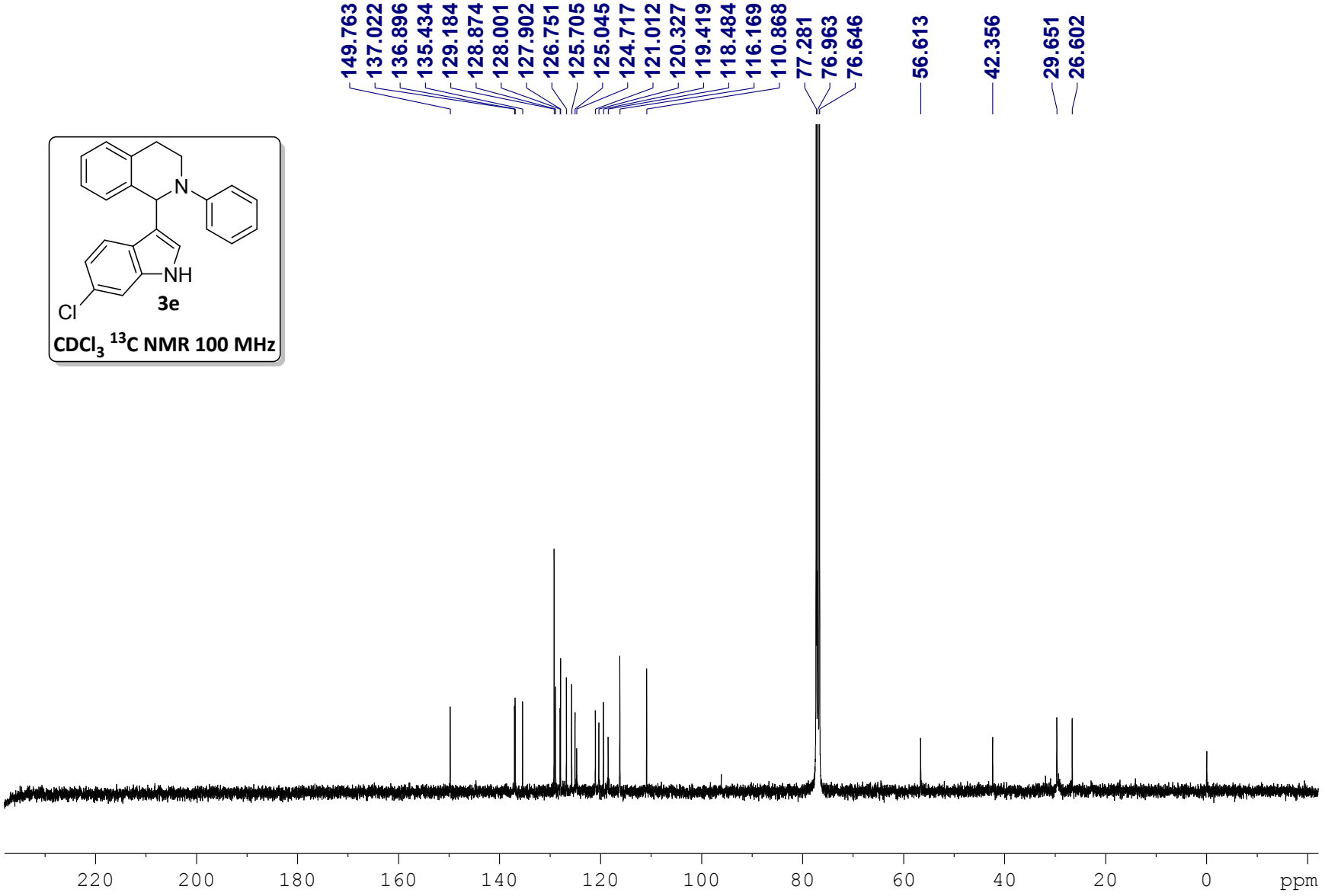
7.397
7.375
7.171
7.153
7.008
6.987
6.972
6.950
6.804
6.786
6.769
6.599
6.104
3.583
3.557
3.547
3.069
3.051
3.031
3.013
2.798
2.757
N
NH
Cl
3e
CDCl3 1H NMR 400 MHz
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
ppm
0.957
1.101
4.339
2.927
3.088
1.047
1.015
1.000
2.065
1.050
1.050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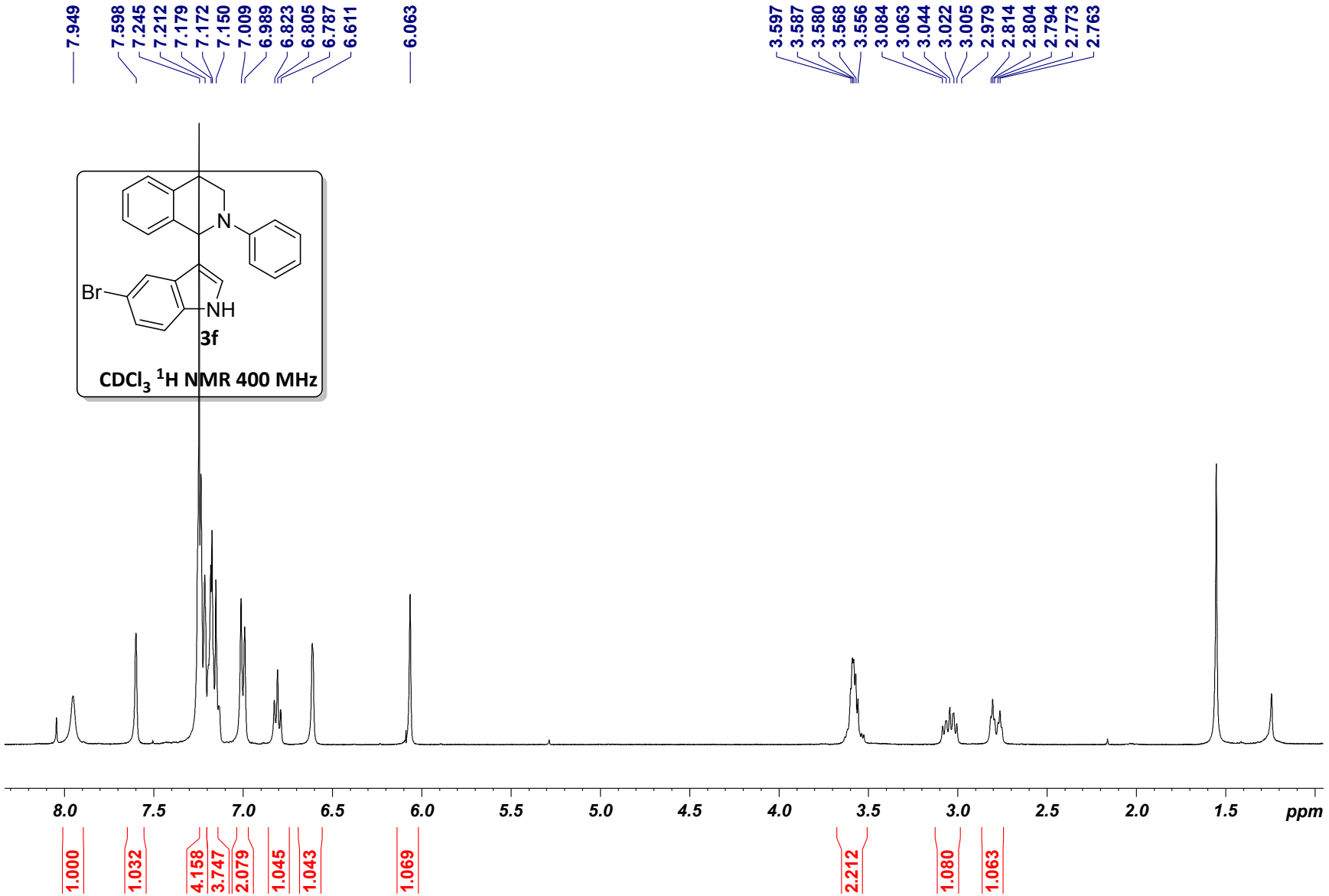
136.896
135.434
129.184
128.874
128.001
127.902
126.751
125.705
125.045
124.717
121.012
120.327
119.419
118.484
116.169
110.868
77.281
76.963
76.646
56.613
42.356
29.651
26.602
N
NH
Cl
3e
CDCl3 13C NMR 100 MHz
220
200
180
160
140
120
100
80
60
40
20
0
ppm

2d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7.598
7.245
7.212
7.179
7.172
7.150
7.009
6.989
6.823
6.805
6.787
6.611
6.063
3.597
3.587
3.580
3.568
3.556
3.084
3.063
3.044
3.022
3.005
2.979
2.814
2.804
2.794
2.773
2.763
N
Br
NH
3f
CDCl3 1H NMR 400 MHz
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
ppm
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4.158
3.747
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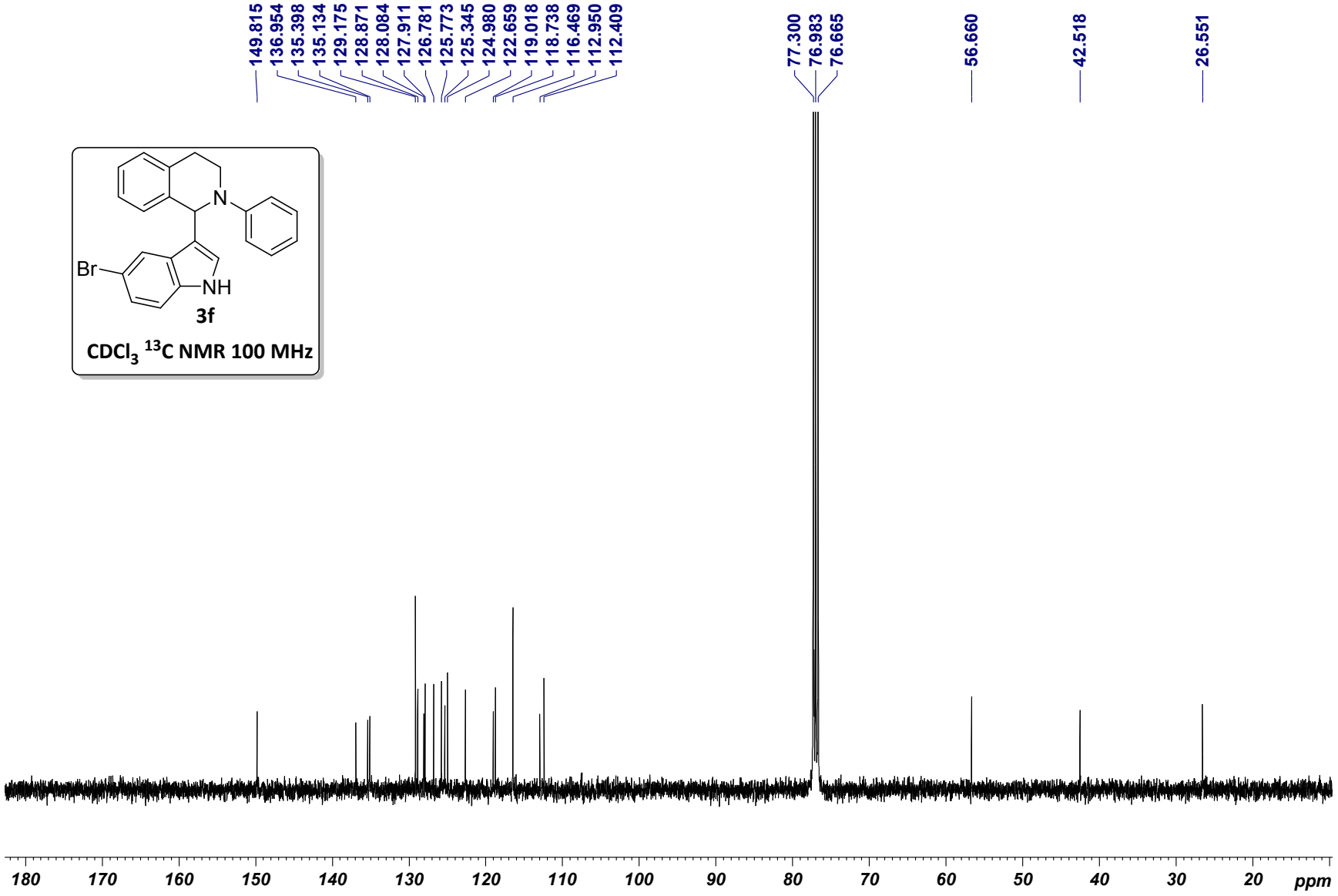
1.045
1.043
1.069
2.212
1.080
1.063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136.954
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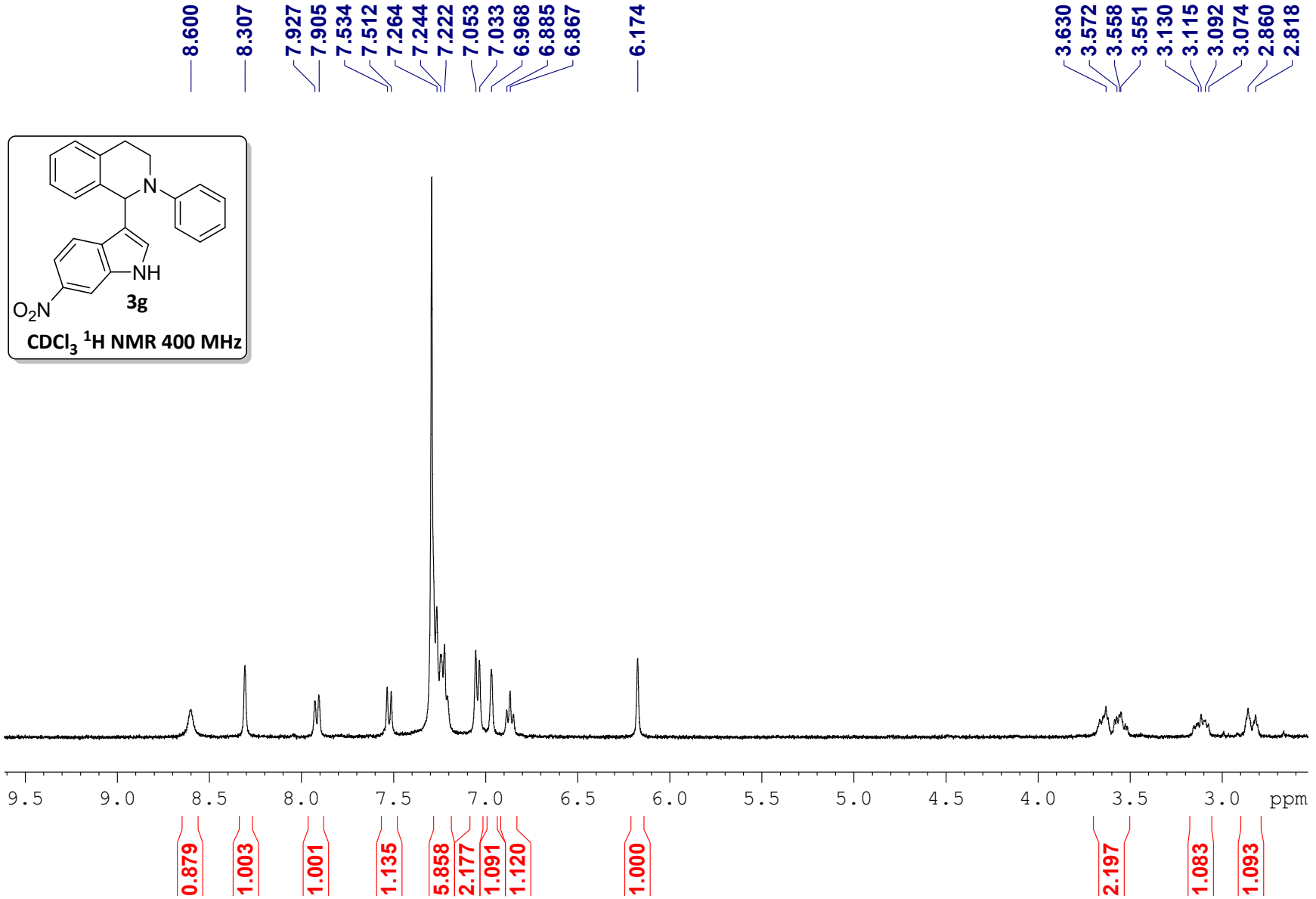
135.134
129.175
128.871
128.084
127.911
126.781
125.773
125.345
124.980
122.659
119.018
118.738
116.469
112.950
112.409
77.300
76.983
76.665
56.660
42.518
26.551
Br
NH
3f
CDCl3 13C NMR 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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8.307
7.927
7.905
7.534
7.512
7.264
7.244
7.222
7.053
7.033
6.968
6.885
6.867
6.174
3.630
3.572
3.558
3.551
3.130
3.115
3.092
3.074
2.860
2.818
N
NH
O2N
3g
CDCl3 1H NMR 400 MHz
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
ppm
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1.001
1.135
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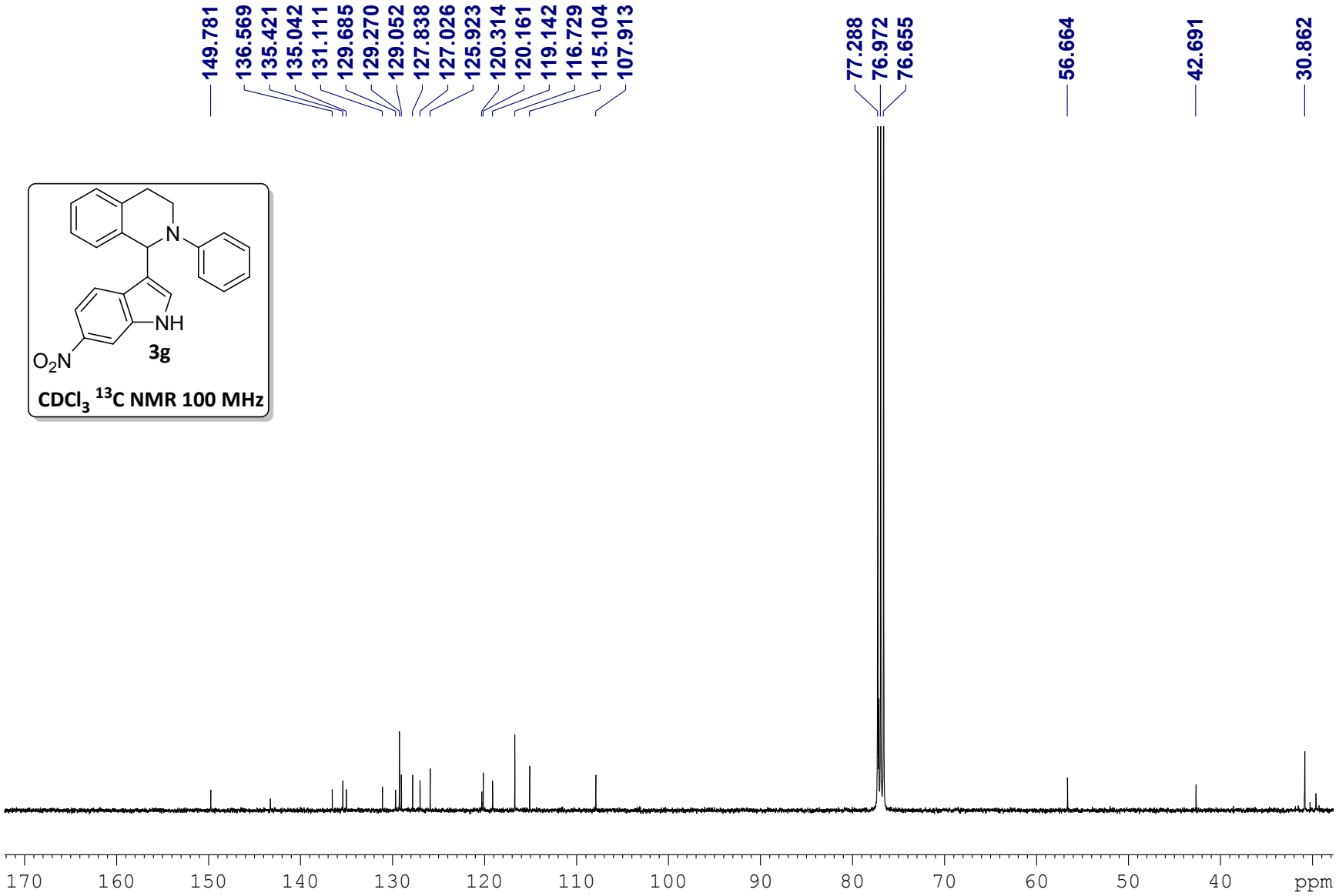
1.120
1.000
2.197
1.083
1.093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135.421
135.042
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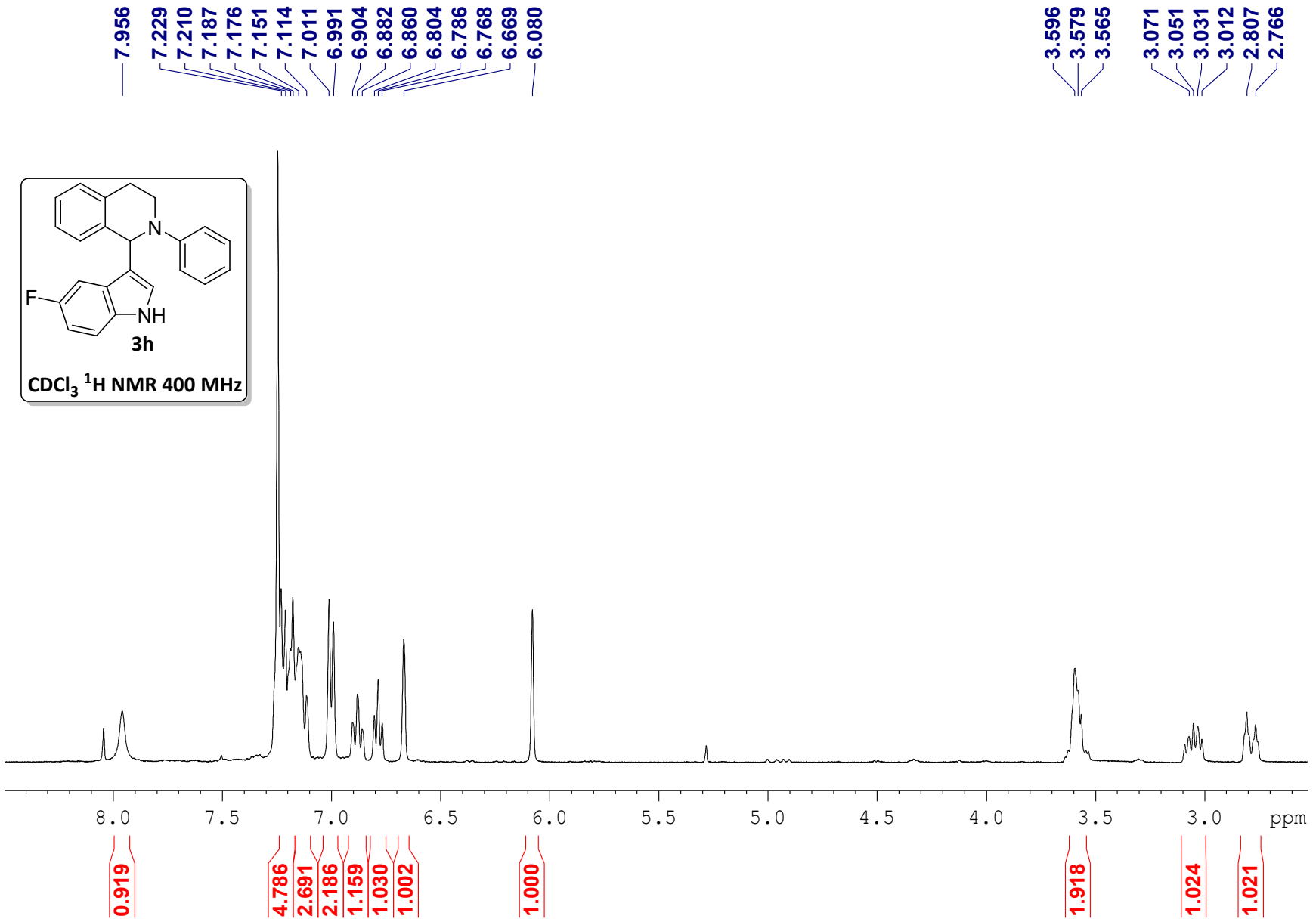
129.685
129.270
129.052
127.838
127.026
125.923
120.314
120.161
119.142
116.729
115.104
107.913
77.288
76.972
76.655
56.664
42.691
30.862
N
NH
O2N
3g
CDCl3 13C NMR 100 MHz
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm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7.210
7.187
7.176
7.151
7.114
7.011
6.991
6.904
6.882
6.860
6.804
6.786
6.768
6.669
6.080
3.596
3.579
3.565
3.071
3.051
3.031
3.012
2.807
2.766
F
NH
N
3h
CDCl3 1H NMR 400 MHz
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7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
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1.002
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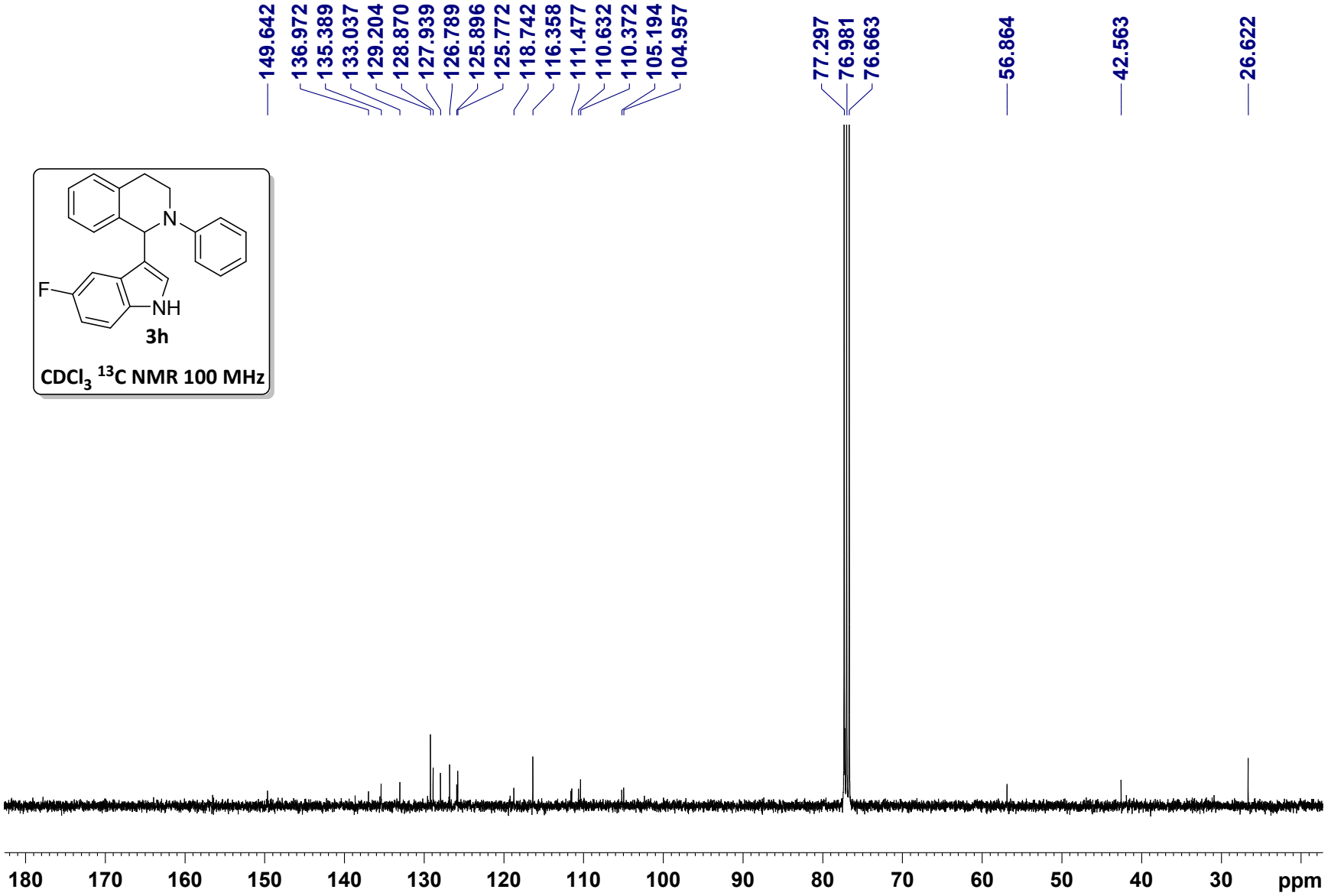
1.021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125.772
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110.632
110.372
105.194
104.957
77.297
76.981
76.663
56.864
42.563
26.622
F
NH
N
3h
CDCl3 13C NMR 100 MHz
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170
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140
130
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110
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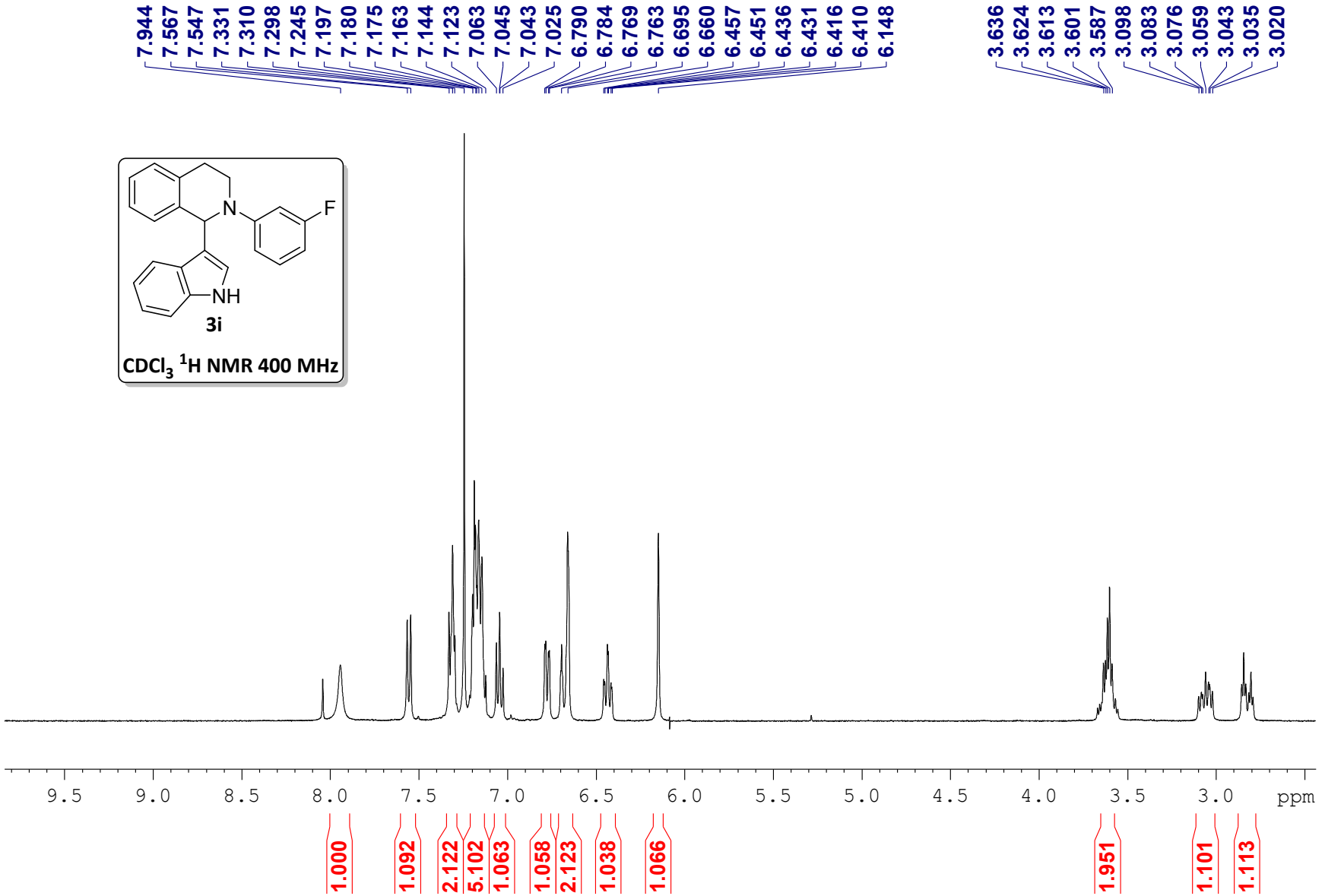
90
80
70
60
50
40
30
ppm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7.197
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7.144
7.123
7.063
7.045
7.043
7.025
6.790
6.784
6.769
6.763
6.695
6.660
6.457
6.451
6.436
6.431
6.416
6.410
6.148
3.636
3.624
3.613
3.601
3.587
3.098
3.083
3.076
3.059
3.043
3.035
3.020
N
F
NH
3i
CDCl3 1H NMR 400 MHz
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
ppm
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1.038
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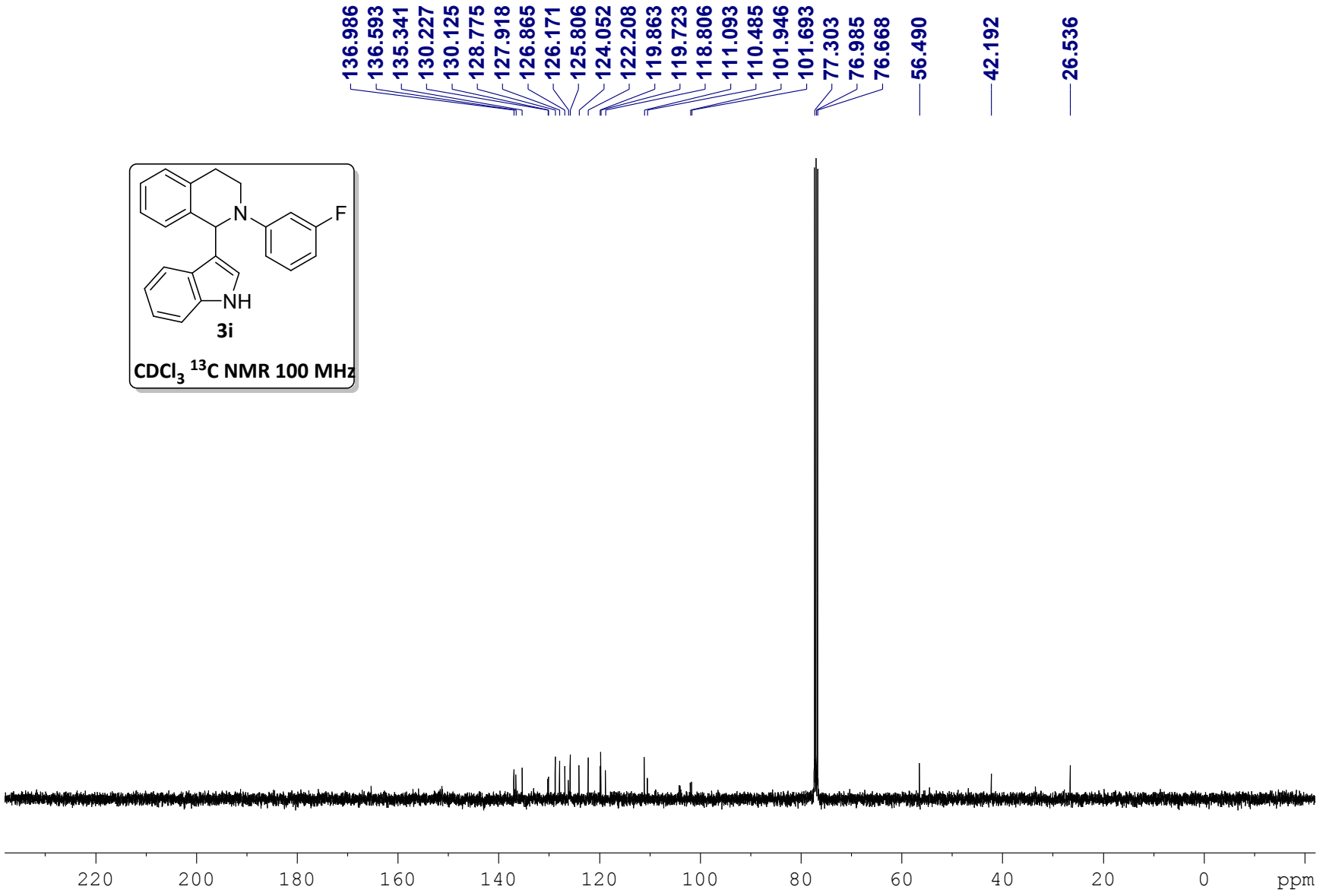
1.101
1.113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122.208
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119.723
118.806
111.093
110.485
101.946
101.693
77.303
76.985
76.668
56.490
42.192
26.536
N
F
NH
3i
CDCl3 13C NMR 100 MHz
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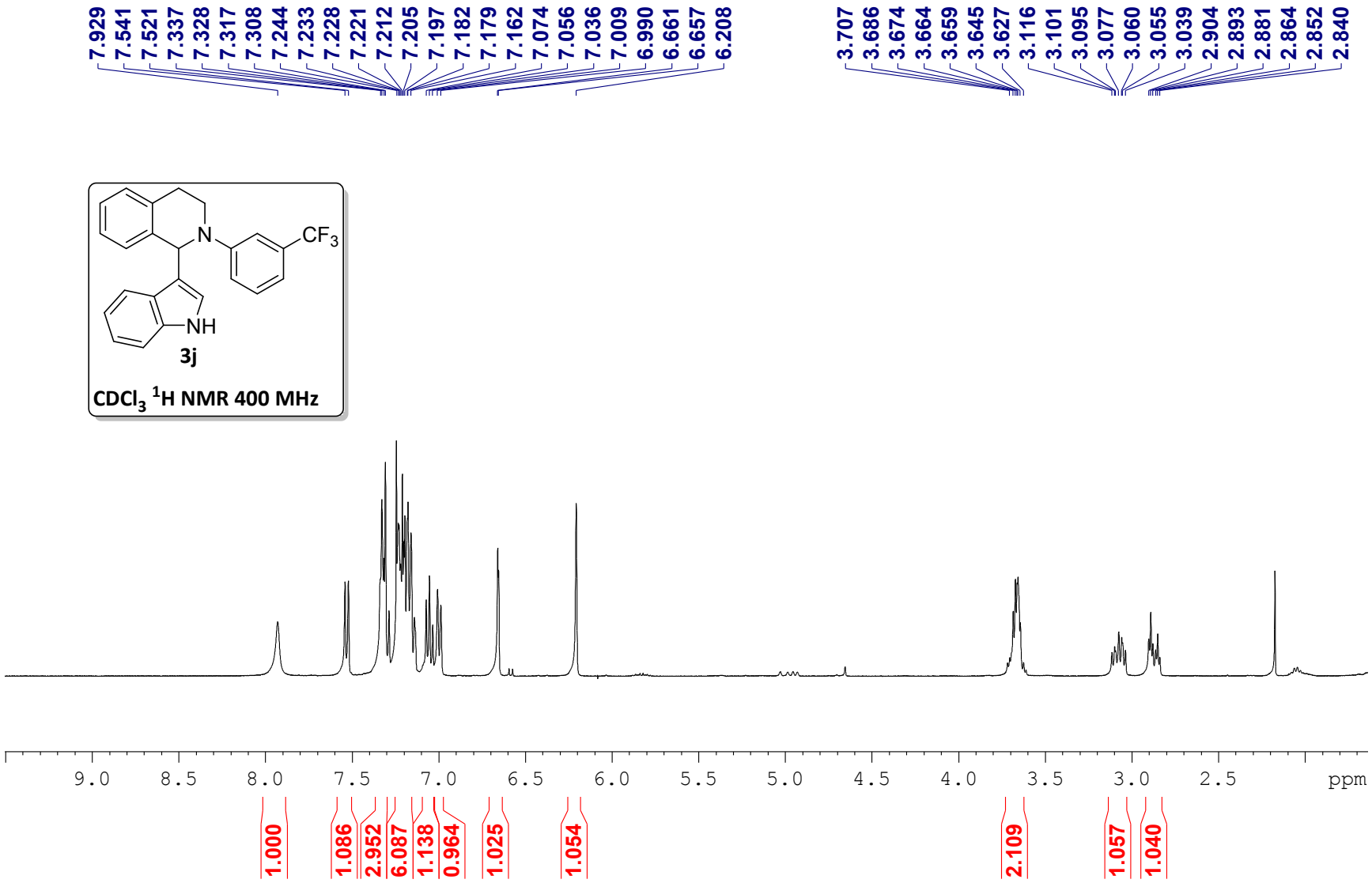
200
180
160
140
120
100
80
60
40
20
0
ppm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7.337
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7.317
7.308
7.244
7.233
7.228
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7.212
7.205
7.197
7.182
7.179
7.162
7.074
7.056
7.036
7.009
6.990
6.661
6.657
6.208
3.707
3.686
3.674
3.664
3.659
3.645
3.627
3.116
3.101
3.095
3.077
3.060
3.055
3.039
2.904
2.893
2.881
2.864
2.852
2.840
N
CF3
NH
3j
CDCl3 1H NMR 400 MHz
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
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2.109
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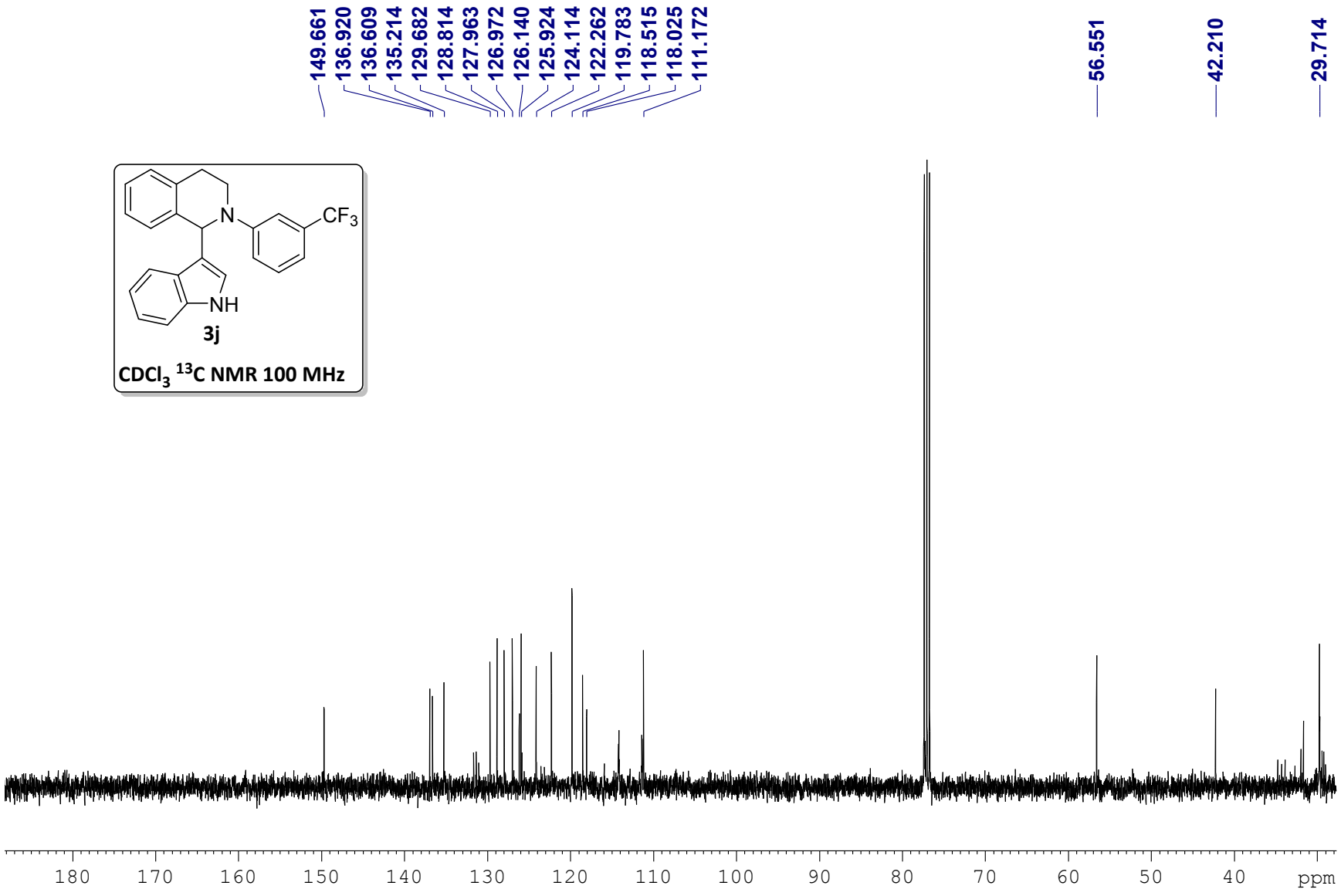
1.040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136.609
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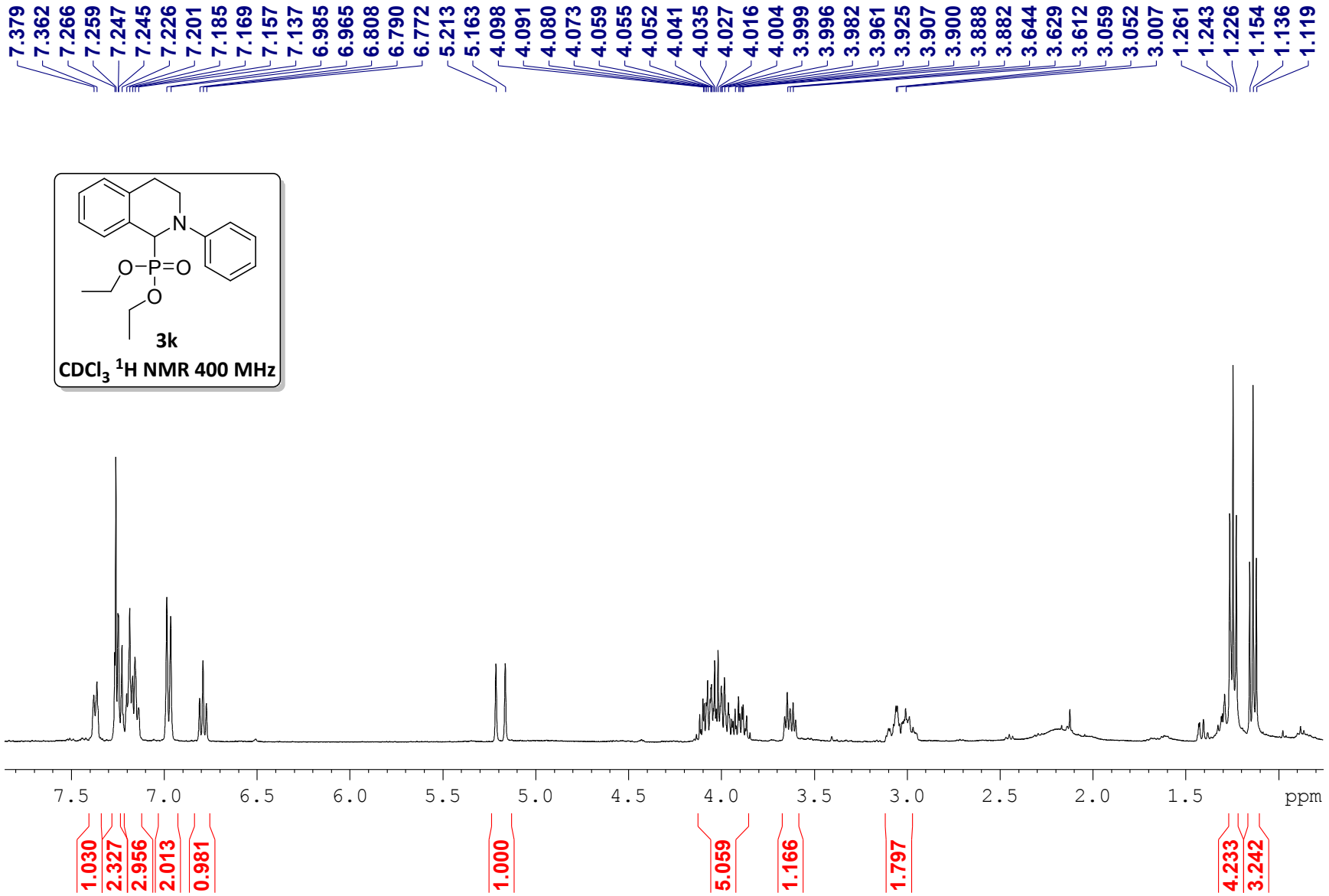
128.814
127.963
126.972
126.140
125.924
124.114
122.262
119.783
118.515
118.025
111.172
56.551
42.210
29.714
N
CF3
NH
3j
CDCl3 13C NMR 100 MHz
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
ppm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7.226
7.201
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7.169
7.157
7.137
6.985
6.965
6.808
6.790
6.772
5.213
5.163
4.098
4.091
4.080
4.073
4.059
4.055
4.052
4.041
4.035
4.027
4.016
4.004
3.999
3.996
3.982
3.961
3.925
3.907
3.900
3.888
3.882
3.644
3.629
3.612
3.059
3.052
3.007
1.261
1.243
1.226
1.154
1.136
1.119
3k
CDCl3 1H NMR 400 MHz
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
ppm
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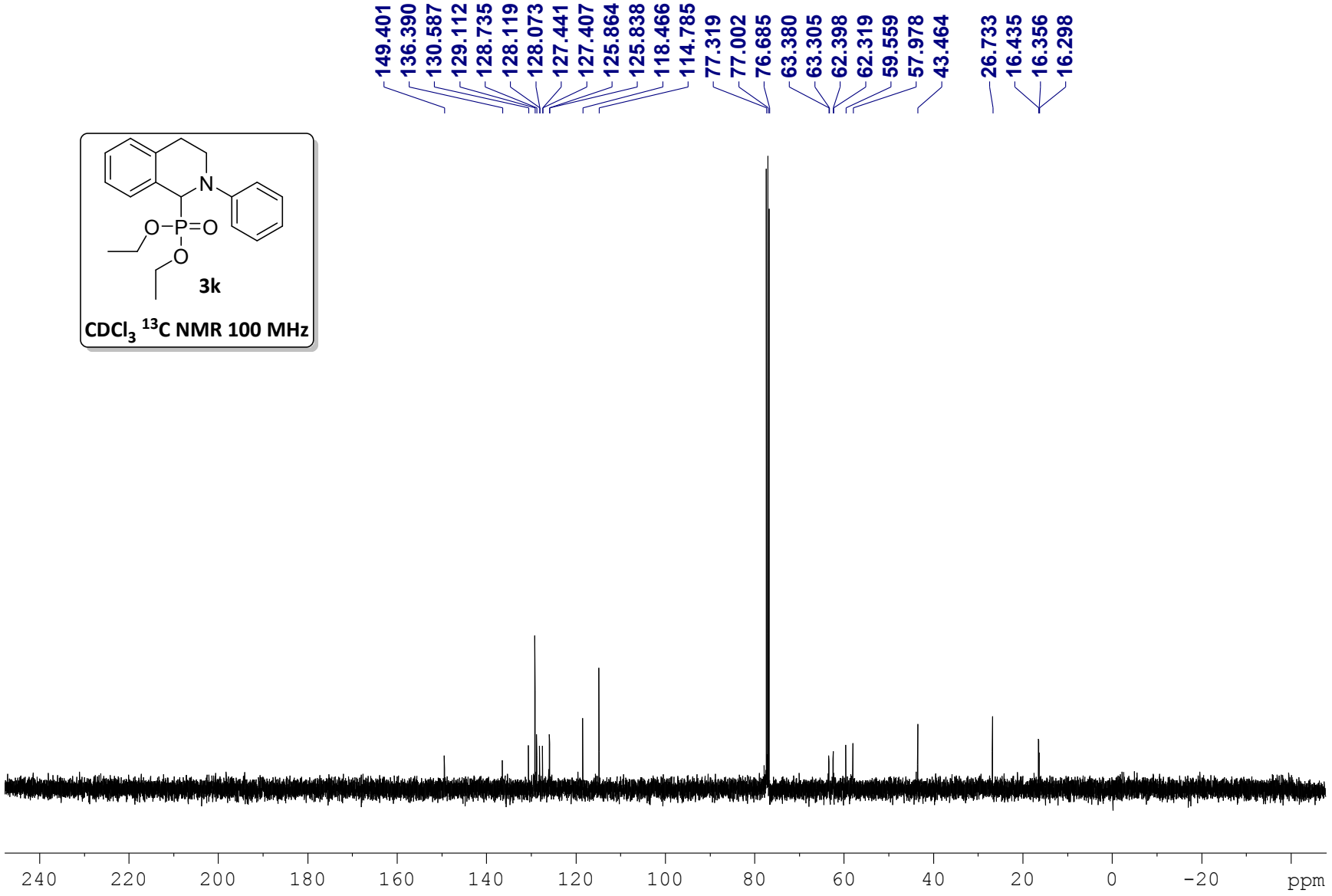
3.242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125.838
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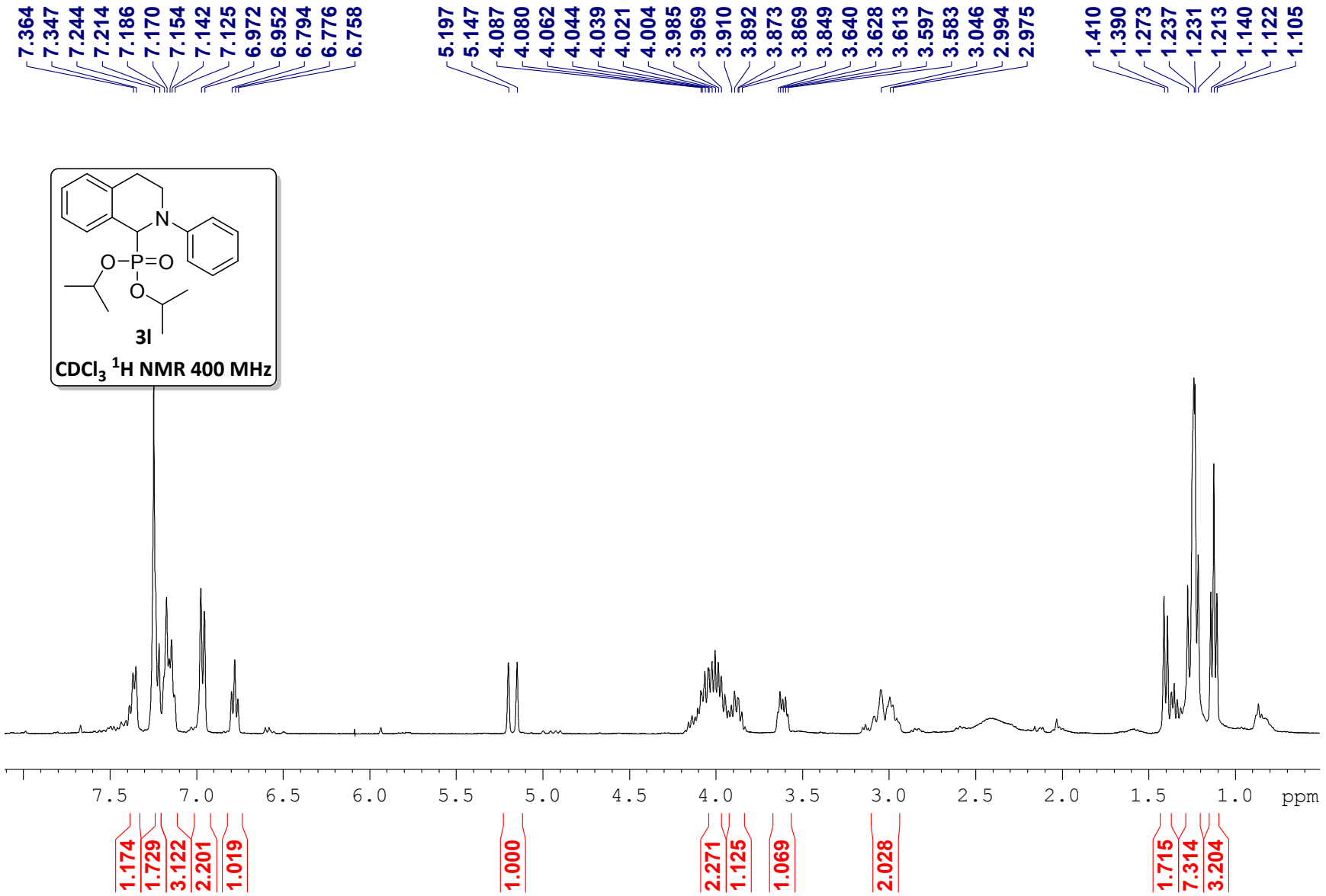
114.785
77.319
77.002
76.685
63.380
63.305
62.398
62.319
59.559
57.978
43.464
26.733
16.435
16.356
16.298
N
O−P=O
O
3k
CDCl3 13C NMR 100 MHz
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm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4.080
4.062
4.044
4.039
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4.004
3.985
3.969
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3.640
3.628
3.613
3.597
3.583
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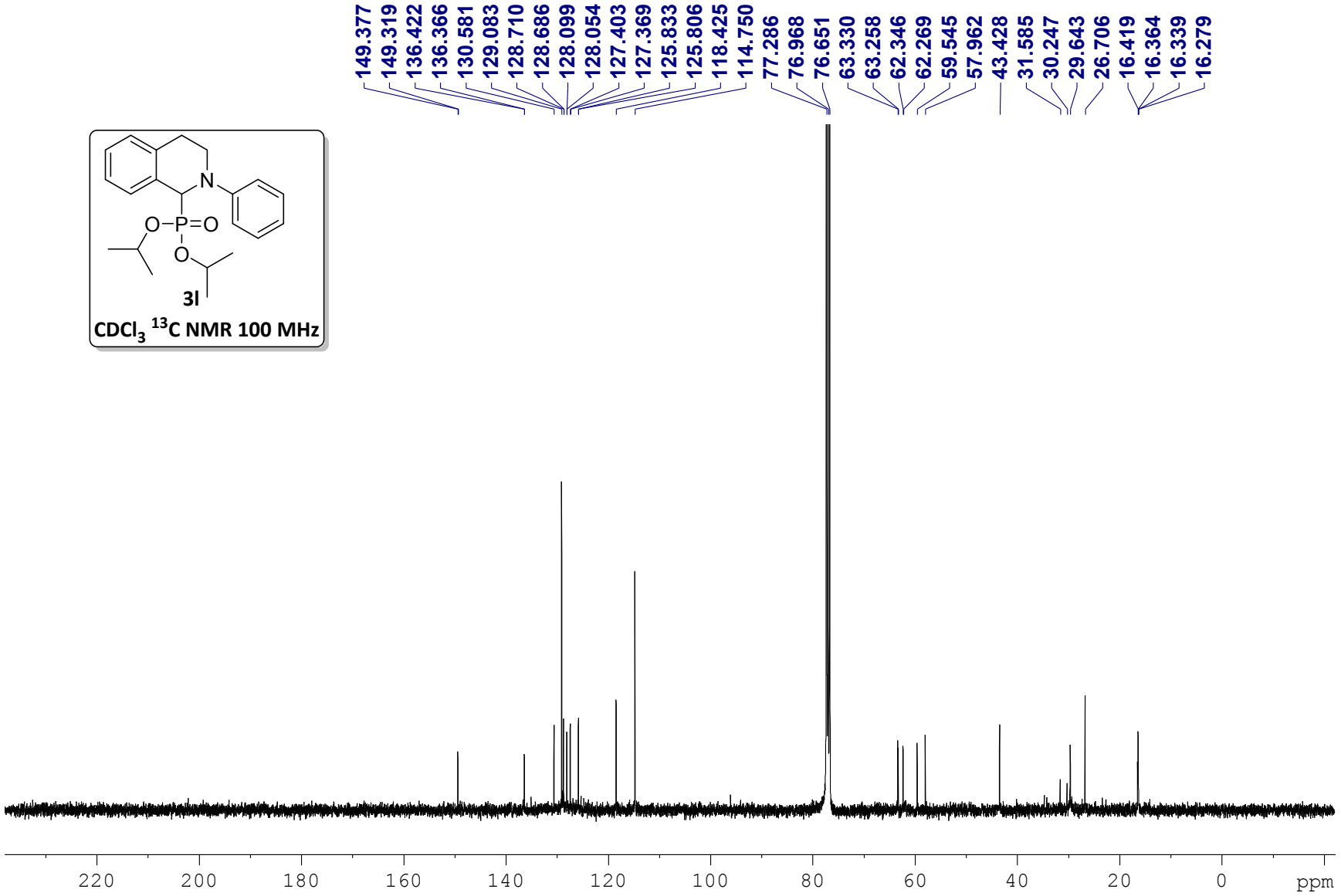
1.390
1.273
1.237
1.231
1.213
1.140
1.122
1.105
N
O−P=O
O
3l
CDCl3 1H NMR 400 MHz
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.174
1.729
3.122
2.201
1.019
1.000
2.271
1.125
1.069
2.028
1.715
7.314
3.204


149.377
149.319
136.422
136.366
130.581
129.083
128.710
128.686
128.099
128.054
127.403
127.369
125.833
125.806
118.425
114.750
77.286
76.968
76.651
63.330
63.258
62.346
62.269
59.545
57.962
43.428
31.585
30.247
29.643
26.706
16.419
16.364
16.339
16.279
O−P=O
O
N
3l
CDCl3 13C NMR 100 MHz
220
200
180
160
140
120
100
80
60
40
20
0
ppm