Electronic Supplementary Material (ESI) for Chemical Communications

Recyclable Copper Catalyzed Nitrogenation of Biphenyl Halides: A Direct Approach to Carbazole

(Supporting Information)

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General remarks

All manipulations were conducted with schlenk tubes. ^{1}H-NMR spectra were recorded on a Bruker AVIII-400 spectrometers. Chemical shifts (in ppm) were calibrated with $CDCl_3$ and DMSO-d6. ^{13}C-NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ and DMSO-d6. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. The starting materials **1** and A21-CuI were prepared according to previously reported literatures.[1, 2]

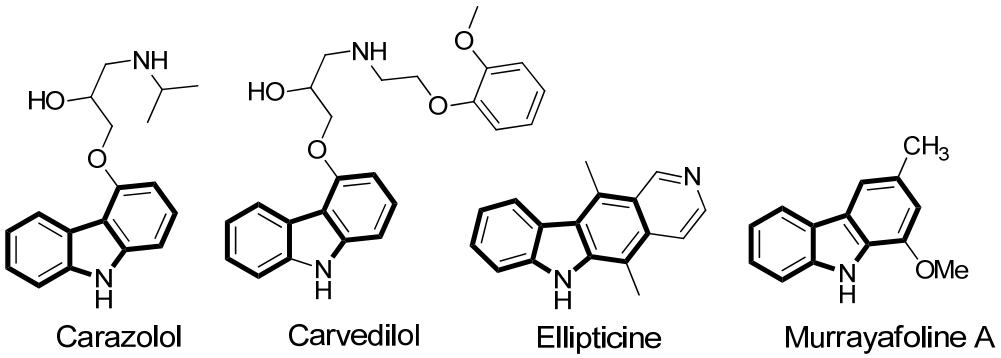
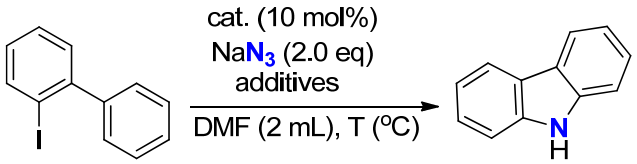


Figure S1. Some biologically active molecules or medicines.

Table S1 Optimization of reaction conditions for nitrogenation of biphenyl halides 2a.[a]

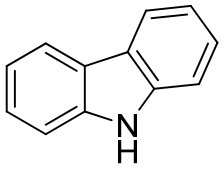


1a **2a**

entry	catalyst	temperature	additives	solvent	yield (%)[b]
1	none	100		DMSO	0
2[c]	CuI	100	NaOH	DMSO	trace
3[c]	CuI	100	NaOH, PEG-400	DMSO	trace
4	A21-CuI	100		DMF	34
5	A21-CuI	100		toluene	0
6	A21-CuI	130		DMF	55
7[d]	CuI	130		DMF	53
9	$Pd(OAc)_2$	130		DMF	0
10	$Rh(O_2CC_7H_{15})_4$	130		DMF	0
11	$RuCl_3$	130		DMF	0
12	$Rh(O_2CC_7H_{15})_4$, A_{21}-CuI	130		DMF	53
13	A21-CuI	130		NMP	57
14	A21-CuI	130		p-xylene	0
15	A21-CuI	130		DMF, PhCl	57
16	A21-CuI, $Pd(PPh_3)_4$	130		DMF	32
17	A_{21}-CuI	130	$NaNO_2$	DMF	60
18	A_{21}-CuI	150		DMF	61
19	A_{21}-CuI	150	4Å MS, $NaNO_2$	DMF	65
20	A21-CuI	130	Na_2CO_3	DMF	43
21	A21-CuI	130	$AlCl_3$	DMF	0
22	A21-CuI	130	$FeCl_2$	DMF	trace
23[e]	A21-CuI	130		DMF	0
24[f]	A21-CuI	130		DMF	0

[a] Reaction conditions: **1a** (0.5 mmol), sodium azide (1.0 mmol), additives, catalyst (10 mol %), in dry DMF (2.0 mL) under Ar for 48 h. [b] Isolated yields. [c] L-proline was added as a ligand. [d] Phen was added as a ligand. [e] $TMSN_3$ was added instead of NaN_3. [f] $TosN_3$ was added instead of NaN_3.

Experimental procedure and characterization data

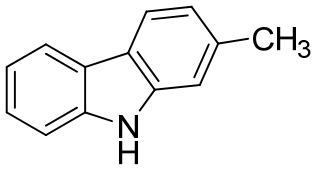


2a

1) 9H-Carbazole (2a)[3]

Typical procedure:

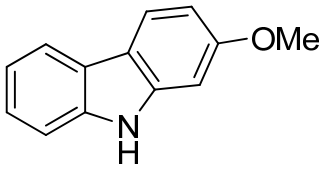
The reaction of 2-iodo-1,1'-biphenyl (**1a**) (0.5 mmol, 140 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h. The resulting mixture was filtered, concentrated and purified by flash chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 10:1) to afford 54.6 mg (65%) of **2a** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.29 (s, 1H), 8.12 (d, *J* = 7.6 Hz, 2H), 7.53 (d, *J* = 8 Hz, 2H), 7.43-7.38 (m, 2H), 7.20-7.14 (m, 2H); ^{13}C NMR (DMSO, 100 MHz): δ = 140.2, 126.0, 122.9, 120.6, 119.0, 111.4 ppm; IR (neat): ν = 3479, 2250, 1242, 823, 761, 624 cm^{-1}. MS (70 eV): m/z (%) 167.0 (M+,100).



2b

2) 2-Methyl-9H-carbazole (2b)[3]

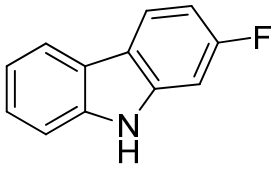
The reaction of 2-iodo-4'-methyl-1,1'-biphenyl (**1b**) (0.5 mmol, 147 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 75.3 mg (83%) of **2b** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.14 (s, 1H), 8.05 (d, *J* = 8.0 Hz, 1H), 7.97 (d, *J* = 7.6 Hz, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.38-7.34 (m, 1H), 7.32 (s, 1H), 7.14 (t, *J* = 7.4 Hz, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 2.50(s, 3H); ^{13}C NMR (DMSO, 100 MHz): δ = 140.7, 140.2, 135.5, 125.4, 123.0, 120.7, 120.5, 120.3, 120.2, 118.9, 111.4, 111.3, 22.2; IR (thin film): 3467, 2251, 1658, 1243, 823, 761, 625 cm^{-1}; MS (70 eV): m/z (%) 181.1 (M+,100).



2c

3) 2-Methoxy-9H-carbazole (2c) [3]

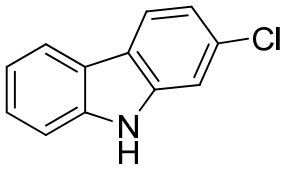
The reaction of 2-iodo-4'-methoxy-1,1'-biphenyl (**1c**) (0.5 mmol, 155 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 79 mg (80%) of **2c** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.14 (s, 1H), 8.00-7.95 (m, 2H), 7.46 (d, J = 8.0 Hz, 1H), 7.33-7.29 (m, 1H), 7.15-7.11 (m, 1H), 7.01 (d, J = 2.0 Hz, 1H), 6.79 (dd, J_1 = 2.0 Hz, J_2 = 8.8 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (DMSO, 100 MHz): δ = 159.0, 141.6, 140.2, 124.6, 123.2, 121.4, 119.7, 119.0, 116.7, 111.1, 108.2, 94.9, 55.7; IR (thin film): 3432, 2251, 1632, 1463, 823, 761, 625 cm^{-1}; MS (70 eV): m/z (%) 197.1 (M+,100).



2d

4) 2-Fluoro-9H-carbazole (2d) [3]

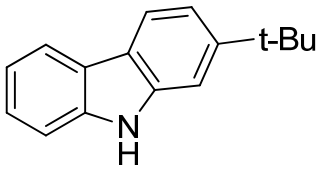
The reaction of 4'-fluoro-2-iodo-1,1'-biphenyl (**1d**) (0.5 mmol, 149 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 57.3 mg (62%) of **2d** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.40 (s, 1H), 8.18-8.06 (m, 2.0 H), 7.50 (d, J = 8.0 Hz, 1H), 7.37 (t, J = 7.4 Hz, 1H), 7.28 (dd, J_1 = 2.0 Hz, J_2 = 10.0 Hz, 1H), 7.17 (t, J = 7.4 Hz, 1H), 7.10-6.80 (m, 1H); ^{13}C NMR (DMSO, 100 MHz): δ = 161.6 (d, J = 23.7 Hz), 140.8 (J = 1.20 Hz), 125.7, 122.5, 121.9 (d, J = 1.12 Hz), 120.4, 119.5 (d, J = 2.15 Hz), 111.5, 106.9 (d, J = 2.41 Hz), 97.8 (d, J = 2.58 Hz); IR (thin film): 3457, 2250, 1462, 823, 761 cm^{-1}; MS (70 eV): m/z (%) 185.1 (M+,100).



2e

5) 2-Chloro-9H-carbazole (2e) [4]

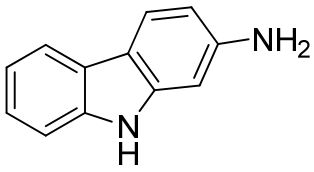
The reaction of 4'-chloro-2-iodo-1,1'-biphenyl (**1e**) (0.5 mmol, 157 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 55.6 mg (55%) of **2e** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.41 (s, 1H), 8.11 (d, *J* = 8.4 Hz, 2H), 7.54-7.51 (m, 2H), 7.45-7.39 (m, 1H), 7.21-7.16 (m, 2H); ^{13}C NMR (DMSO, 100 MHz): δ = 140.8, 140.6, 130.3, 126.4, 122.3, 122.0, 121.8, 120.8, 119.6, 119.1, 111.7, 111.1; IR (thin film): 3457, 2250, 1462, 823, 761 cm^{-1}; MS (70 eV): m/z (%) 201.0 (M+,100).



2f

6) 2-(Tert-butyl)-9H-carbazole (2f) [3]

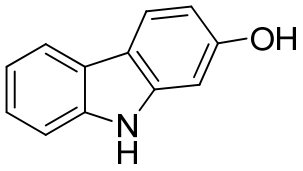
The reaction of 4'-(tert-butyl)-2-iodo-1,1'-biphenyl (**1f**) (0.5 mmol, 168 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 89 mg (79%) of **2f** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.11 (s, 1H), 8.04 (d, *J* = 7.6 Hz, 1H), 7.98 (d, *J* = 8.4 Hz, 1H), 7.50-7.48 (m, 2H), 7.38-7.34 (m, 1H), 7.22 (d, *J* = 8.4 Hz, 1H), 7.14 (t, *J* = 7.6 Hz, 1H); 1.39 (s, 9H); ^{13}C NMR (DMSO, 100 MHz): δ = 149.0, 140.5, 140.4, 125.4, 122.9, 120.5, 120.3, 120.1, 118.8, 117.0, 111.3, 107.6, 35.2, 32.0; IR (thin film): 3450, 2250, 1659, 1462, 823, 761 cm^{-1}; MS (70 eV): m/z (%) 223.1 (M+,100).



2g

13) 9H-Carbazol-2-amine (2g) [6]

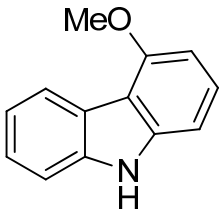
The reaction of 2'-iodo-[1,1'-biphenyl]-4-amine (**1g**) (0.5 mmol, 147.5 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 62 mg (68%) of **2g** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 10.74 (s, 1H), 7.82 (d, *J* = 7.6 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.19-7.15 (m, 1H), 7.02 (t, *J* = 7.6 Hz, 1H), 6.63 (s, 1H), 6.49 (dd, J_1 = 2.0 Hz, J_2 = 8.0 Hz, 1H), 5.18 (brs, 2H); ^{13}C NMR (DMSO, 100 MHz): δ = 148.2, 142.4, 139.7, 124.0, 123.3, 121.0, 118.6, 118.5, 113.6, 110.5, 108.6, 94.8; IR (thin film): 3428, 2251, 1632, 1243, 824, 762, 626 cm^{-1}; MS (70 eV): m/z (%) 182.1 (M+,100).



2h

14) 9H-Carbazol-2-ol (2h) [7]

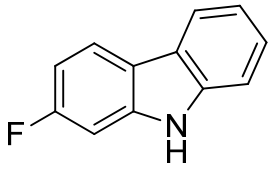
The reaction of 2'-iodo-[1,1'-biphenyl]-4-ol (**1h**) (0.5 mmol, 148 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 33.8 mg (37%) of **2h** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 10.95 (s, 1H), 9.42 (s, 1H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.85 (d, *J* = 7.6 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.07 (t, *J* = 7.6 Hz, 1H), 6.83-6.82 (m, 1H), 6.65-6.62 (m, 1H); ^{13}C NMR (DMSO, 100 MHz): δ = 156.9, 141.9, 140.0, 124.2, 123.4, 121.3, 119.3, 118.8, 115.6, 110.8, 108.7, 96.7; IR (thin film): 3429, 2251, 1659, 824, 762, 625 cm^{-1}; MS (70 eV): m/z (%) 183.1 (M+,100).



2i

7) 4-Methoxy-9H-carbazole (2i) [5]

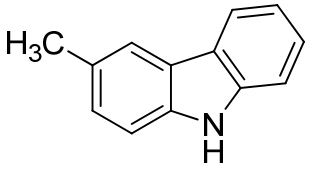
The reaction of 2-iodo-2'-methoxy-1,1'-biphenyl (**1i**) (0.5 mmol, 155 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 73 mg (74%) of **2i** as solid: ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.42 (d, *J* = 7.6 Hz, 1H), 7.92 (s, 1H), 7.45-7.33 (m, 4H), 7.01 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 4.12 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 156.2, 140.8, 138.6, 126.6, 124.9, 123.0, 122.6, 119.5, 112.5, 109.9, 103.5, 100.3, 55.3; IR (thin film): 3403, 3229, 3176, 2959, 1734, 1456 cm^{-1}; MS (70 eV): m/z (%) 310.0 (M+,100).



2d

8) 2-Fluoro-9H-carbazole (2d) [3]

The reaction of 4-fluoro-2-iodo-1,1'-biphenyl (**1j**) (0.5 mmol, 149 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 51.6 mg (56%) of **2d** as solid.

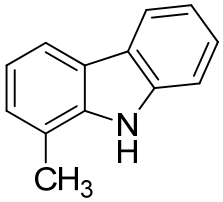


2k

9) 3-Methyl-9H-carbazole (2k) [3]

The reaction of 2-iodo-5-methyl-1,1'-biphenyl (**1k**) (0.5 mmol, 147 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL

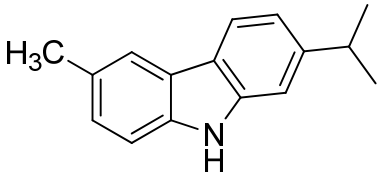
dry DMF at 150 ℃ under argon for 48 h afforded 70.8 mg (78%) of **2k** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.14 (s, 1H), 8.06 (d, *J* = 7.6 Hz, 1H), 7.90 (s, 1H), 7.49 (d, *J* = 8.0 Hz, 1H), 7.42-7.35 (m, 2H), 7.22 (d, *J* = 8.0 Hz, 1H), 7.16-7.12 (m, 1H), 2.48 (s, 3H); ^{13}C NMR (DMSO, 100 MHz): δ = 140.5, 138.5, 127.6, 127.3, 125.8, 123.1, 122.7, 120.5, 120.4, 118.7, 111.3, 111.1, 21.6; IR (thin film): 3464, 2250, 1660, 1242, 823, 761 cm^{-1}; MS (70 eV): m/z (%) 181.1 (M+,100).



2l

10) 4-Methyl-9H-carbazole (2l) [3]

The reaction of 2-iodo-5-methyl-1,1'-biphenyl (**1l**) (0.5 mmol, 147 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 51 mg (56%) of **2l** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 11.20 (s, 1H), 8.09 (d, *J* = 7.6 Hz, 1H), 7.94 (d, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.42-7.38 (m, 1H), 7.21-7.14 (m, 2H), 7.08 (t, *J* = 7.6 Hz, 1H), 2.58(s, 3H); ^{13}C NMR (DMSO, 100 MHz): δ = 140.3, 139.5, 126.4, 125.8, 123.3, 122.5, 120.7, 120.5, 119.1, 119.0, 118.1, 111.5, 17.5; IR (thin film): 3432, 2251, 1659, 1239, 823, 761 cm^{-1}; MS (70 eV): m/z (%) 181.1 (M+,100).

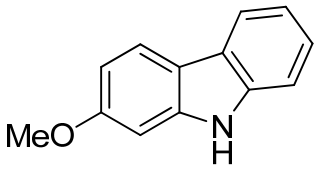


2m

11) 2-Isopropyl-6-methyl-9H-carbazole (2m) [3]

The reaction of 2-iodo-4'-isopropyl-5-methyl-1,1'-biphenyl (**1m**) (0.5 mmol, 168 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 78 mg (70%) of **2m** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 10.98 (s, 1H), 7.93 (d, *J* = 8.0 Hz, 1H),

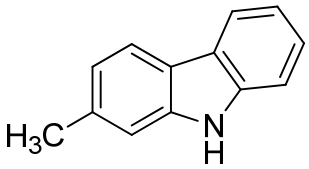
7.84 (s, 1H), 7.38 (d, J = 8.0 Hz, 1H), 7.31 (s, 1H), 7.18 (dd, J_1 = 1.6 Hz, J_2 = 8.0 Hz 1H), 7.19 (dd, J_1 = 1.2 Hz, J_2 = 8.0 Hz 1H), 3.05-3.02(m, 1H), 2.47(s, 3H), 1,30(d, J = 7.6 Hz, 6H); ^{13}C NMR (DMSO, 100 MHz): δ = 146.6, 140.9, 138.6, 127.4, 126.7, 123.2, 120.9, 120.3, 120.1, 117.8, 111.0, 108.5, 34.4, 24.8, 21.6; IR (thin film): 3431, 2251, 1659, 1246, 823, 761 cm^{-1}; MS (70 eV): m/z (%) 223.1 (M+,100).



2c

12) 2-Methoxy-9H-carbazole (2c) [3]

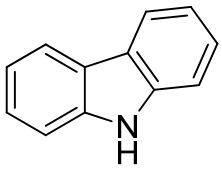
The reaction of 2-iodo-4-methoxy-1,1'-biphenyl (**1n**) (0.5 mmol, 155 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 68 mg (69%) of **2c**.



2b

15) 2-Methyl-9H-carbazole (2b) [3]

The reaction of 2-iodo-4-methyl-1,1'-biphenyl (**1o**) (0.5 mmol, 147 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 48 h afforded 73.3 mg (81%) of **2b**.

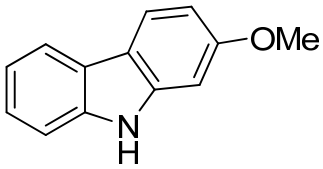


2a

16) 9H-Carbazole (2a) [3]

The reaction of 2-bromo-1,1'-biphenyl (**1p**) (0.5 mmol, 116.5 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry

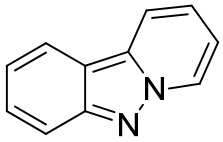
DMF at 150 ℃ under argon for 72 h afforded 50.9 mg (61%) of **2p** as white solid.



2c

17) 2-Methoxy-9H-carbazole (2c) [3]

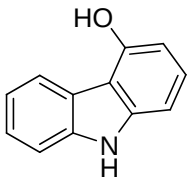
The reaction of 2-bromo-4'-methoxy-1,1'-biphenyl (**1q**) (0.5 mmol, 131 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 72 h afforded 75.8 mg (77%) of **2c**.



2r

18) Pyrido[1,2-b]indazole (2r) [8]

The reaction of 2-(2-iodophenyl)pyridine (**1r**) (0.5 mmol, 140.5 mg), A21-CuI (0.025 mmol, 20mg), NaN_3 (1.0 mmol, 65 mg), 4 Å MS (100 mg) were stirred in 2 mL dry DMF at 150 ℃ under argon for 72 h afforded 53.8 mg (64%) of **2r** as solid: ^{1}H NMR (DMSO, 400 MHz): δ = 9.03 (d, *J* = 6.8 Hz, 1H), 8.42 (d, *J* = 8.0 Hz, 1H), 8.27 (d, *J* = 7.6 Hz, 1H), 7.77 (d, *J* = 7.6 Hz, 1H), 7.55-7.46 (m, 2H), 7.37-7.34 (m, 1H), 7.20(t, *J* = 7.6 Hz, 1H); ^{13}C NMR (DMSO, 100 MHz): δ = 149.5, 135.2, 128.7, 128.6, 123.0, 121.0, 119.7, 118.8, 117.5, 115.5, 115.2; IR (thin film): 3430, 2251, 1645, 824, 761, 625 cm^{-1}; MS (70 eV): m/z (%) 168.1 (M+,100).



3g

19) 9H-Carbazol-4-ol (3g) [9]

The reaction of 4-methoxy-9H-carbazole **2g**) (0.3 mmol, 59 mg), BBr_3 (0.25 mL), were stirred in 2 mL CH_2Cl_2 at -30 ℃ for 3 h afforded 49 mg (90%) of **3g** as solid: ^{1}H

NMR (DMSO, 400 MHz): δ = 11.13 (s, 1H), 10.03 (s, 1H), 8.17 (d, *J* = 8.0 Hz, 1H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.32 (d, *J* = 8.0 Hz, 1H), 7.20-7.11 (m, 2H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.59(d, *J* = 8.0 Hz, 1H); ^{13}C NMR (DMSO, 100 MHz): δ = 153.9, 142.0, 139.4, 139.3, 126.9, 124.7, 122.6, 118.8, 111.5, 110.6, 104.5, 102.4; IR (thin film): 3428, 2251, 1661, 824, 762, 624 cm^{-1}; MS (70 eV): m/z (%) 183.1 (M+,100).

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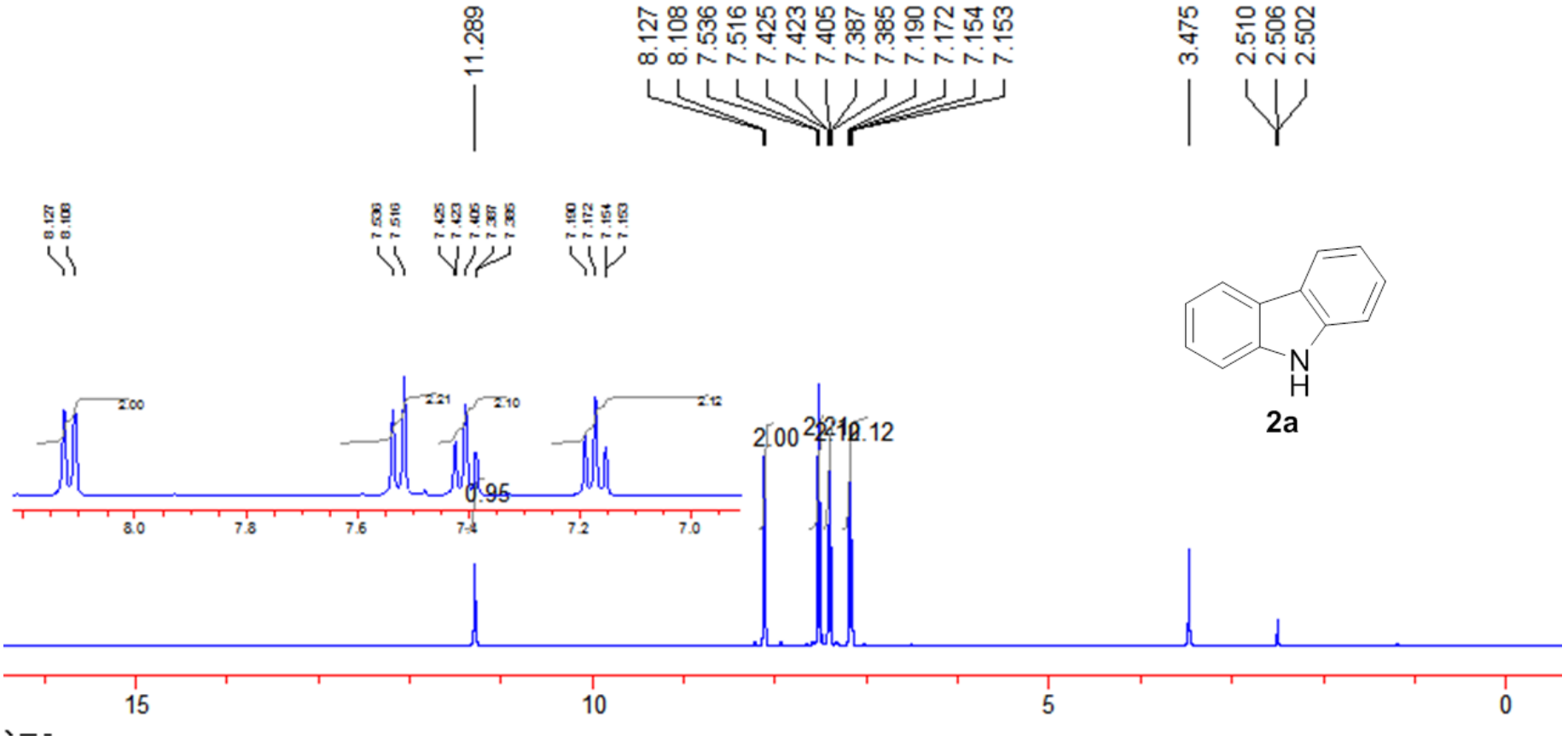
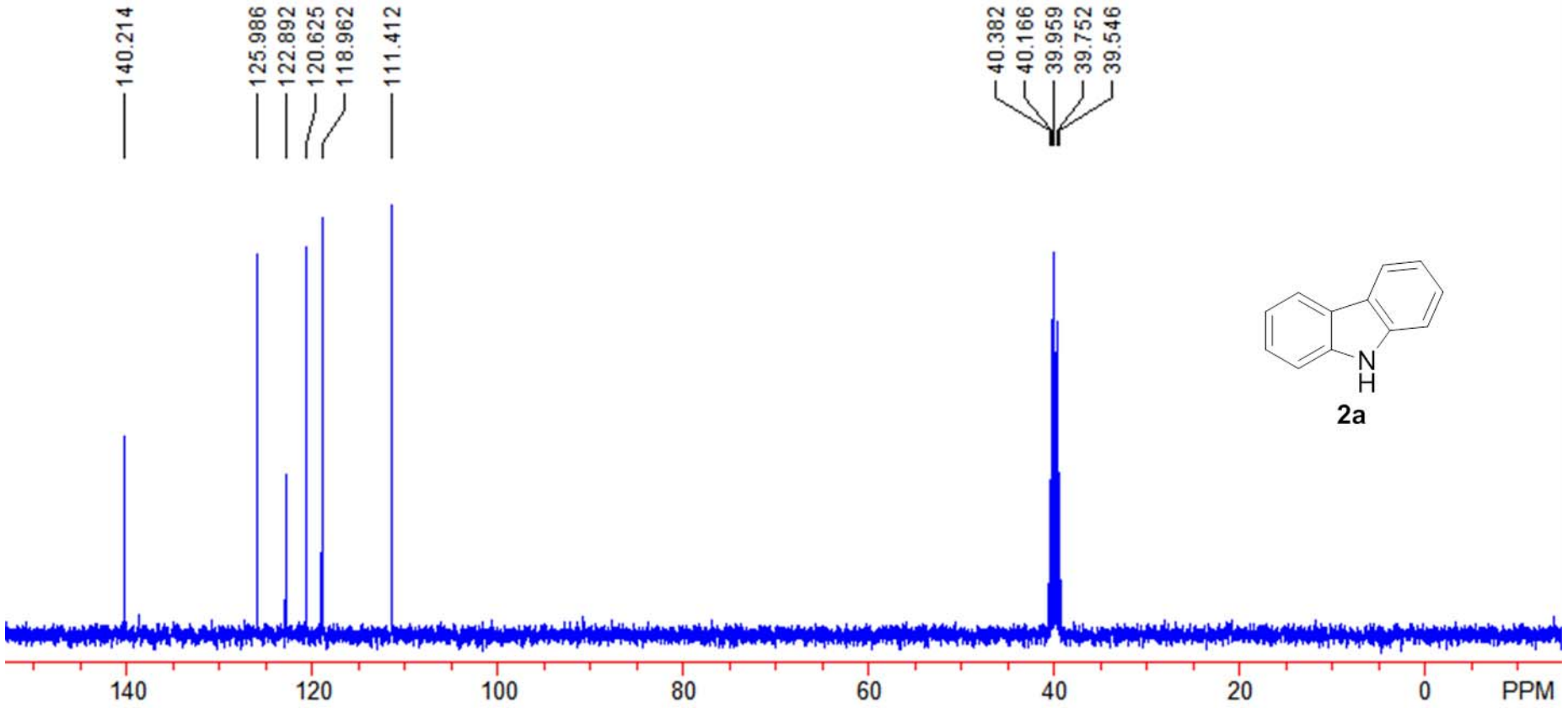
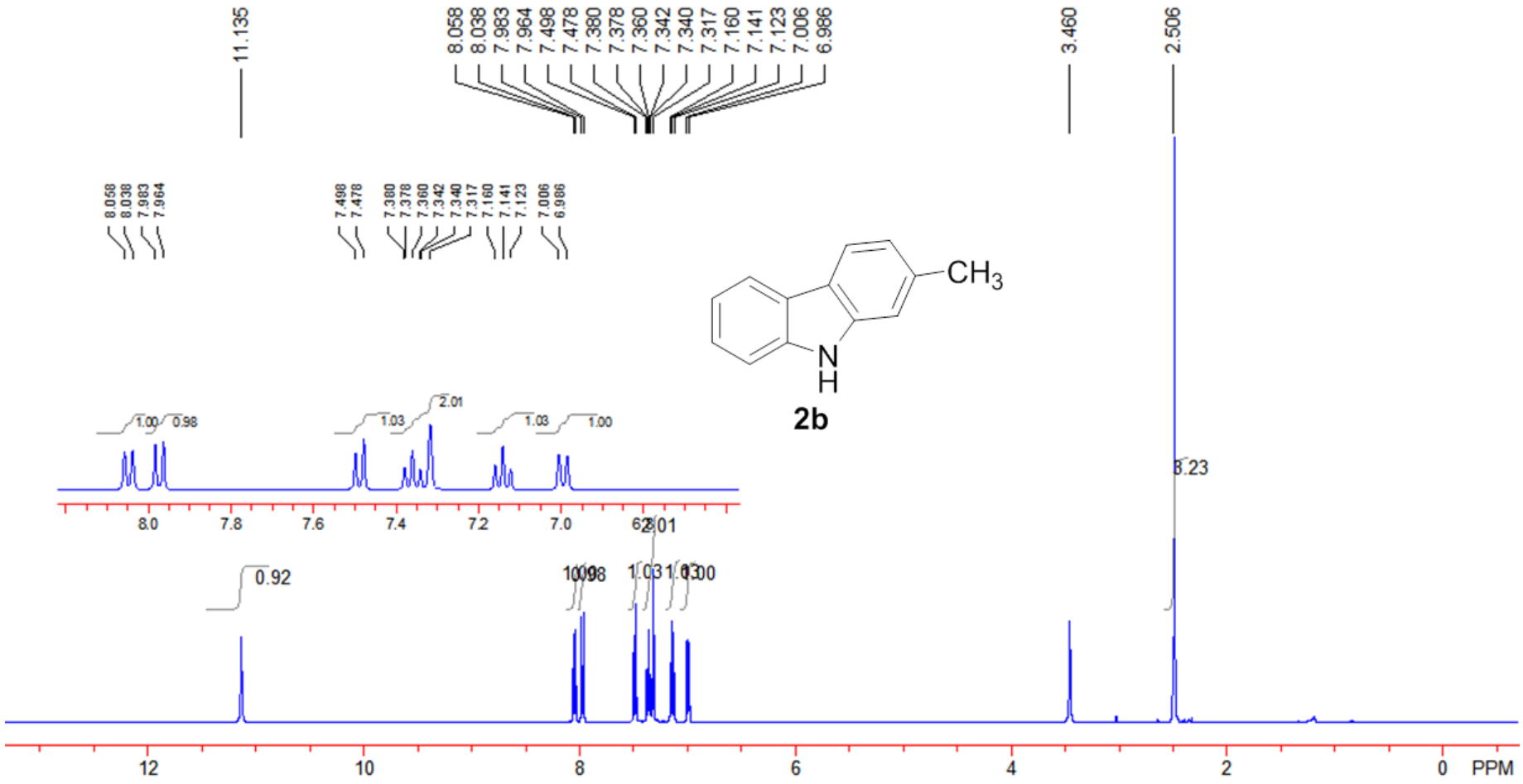
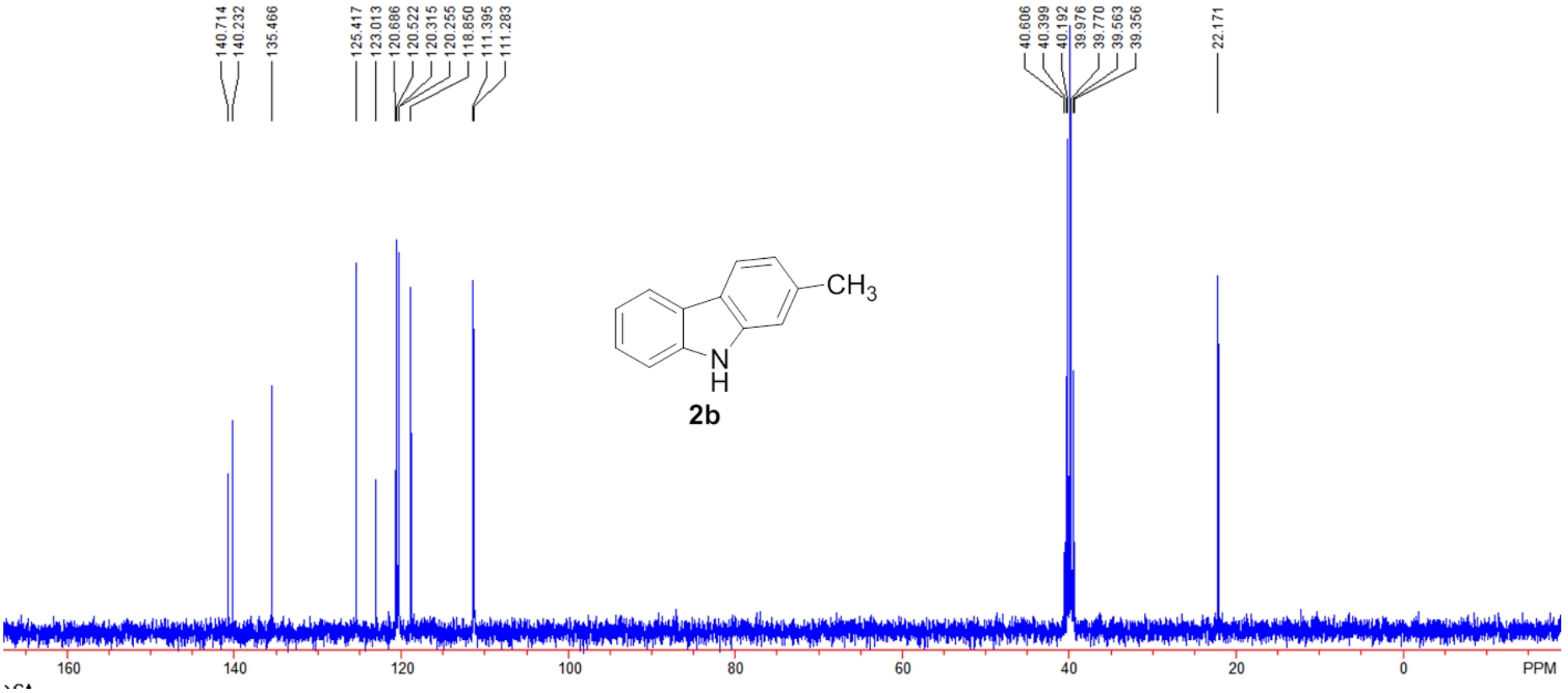
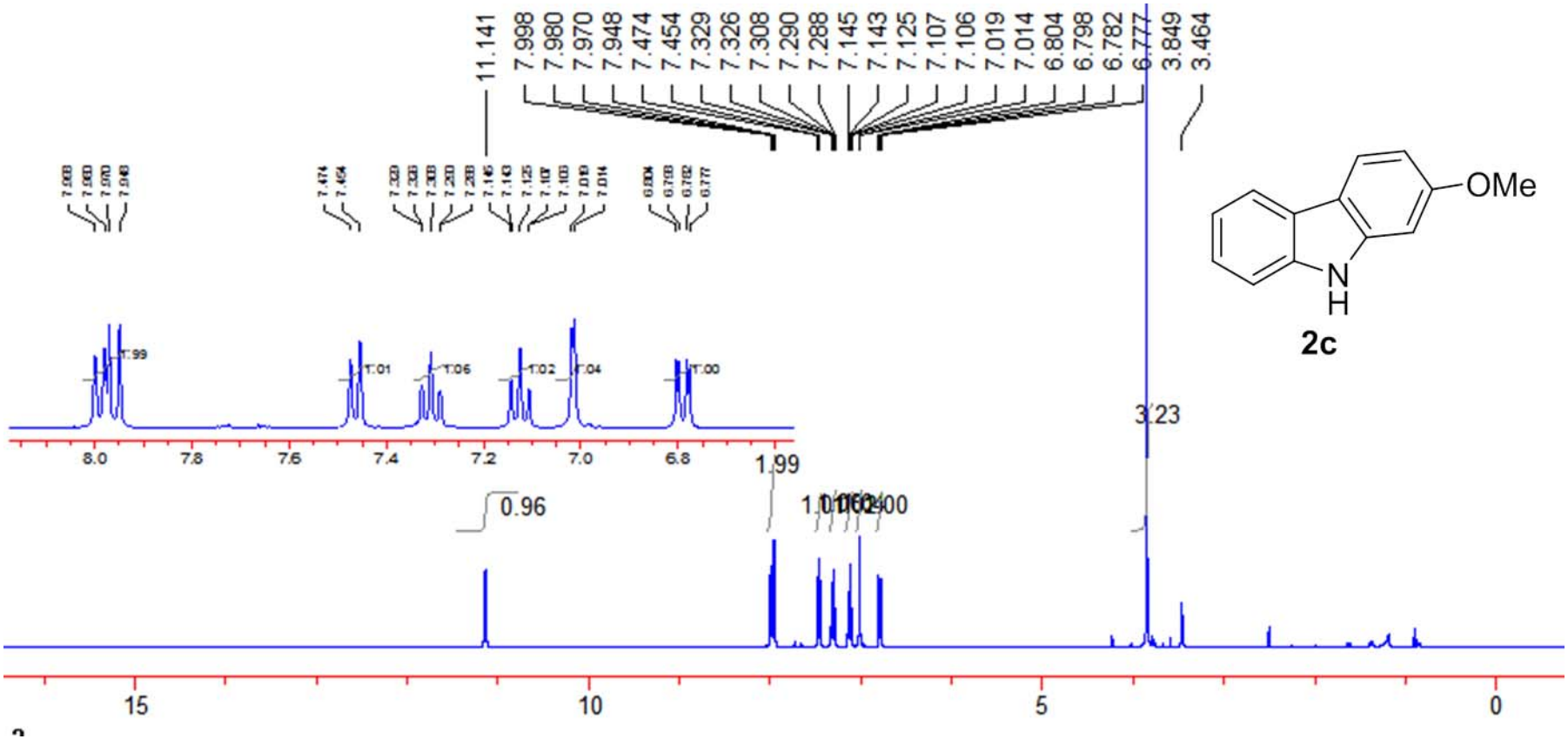


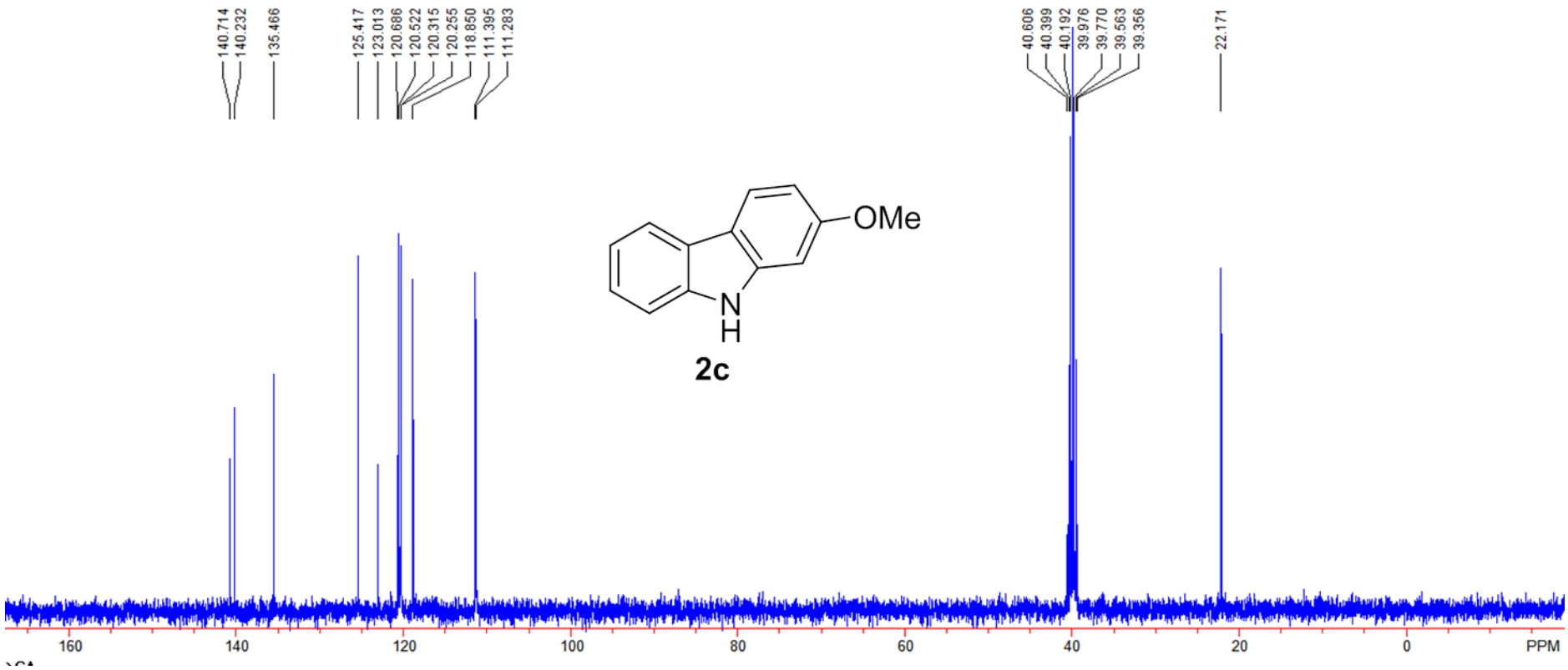




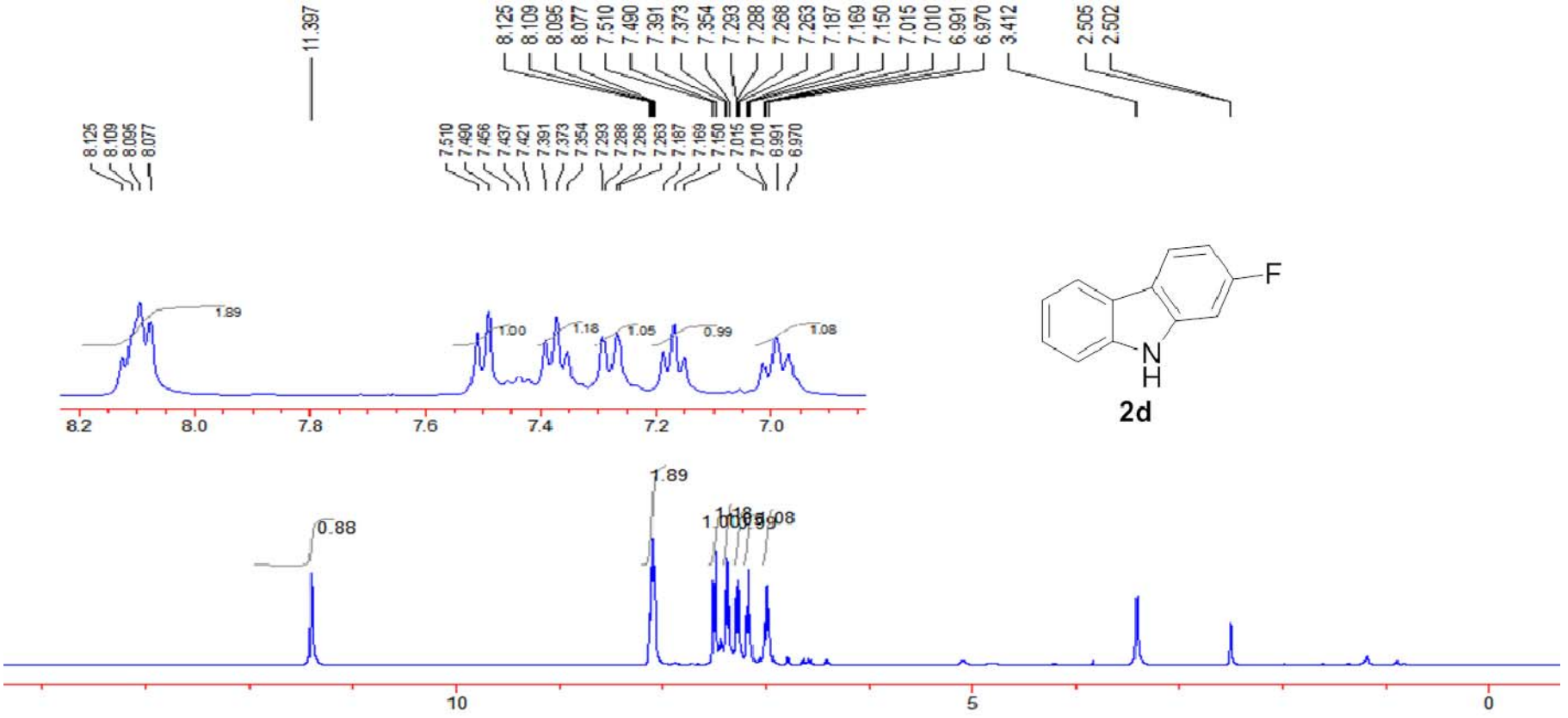


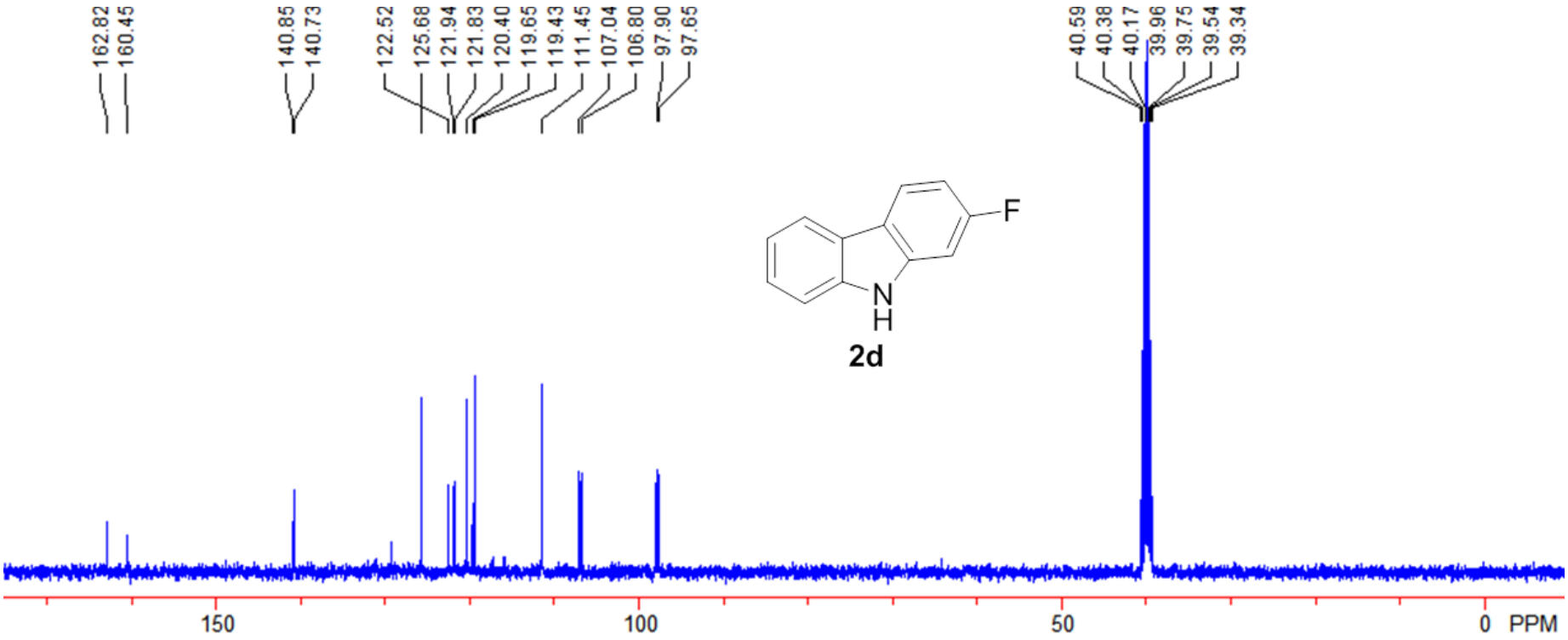
2b



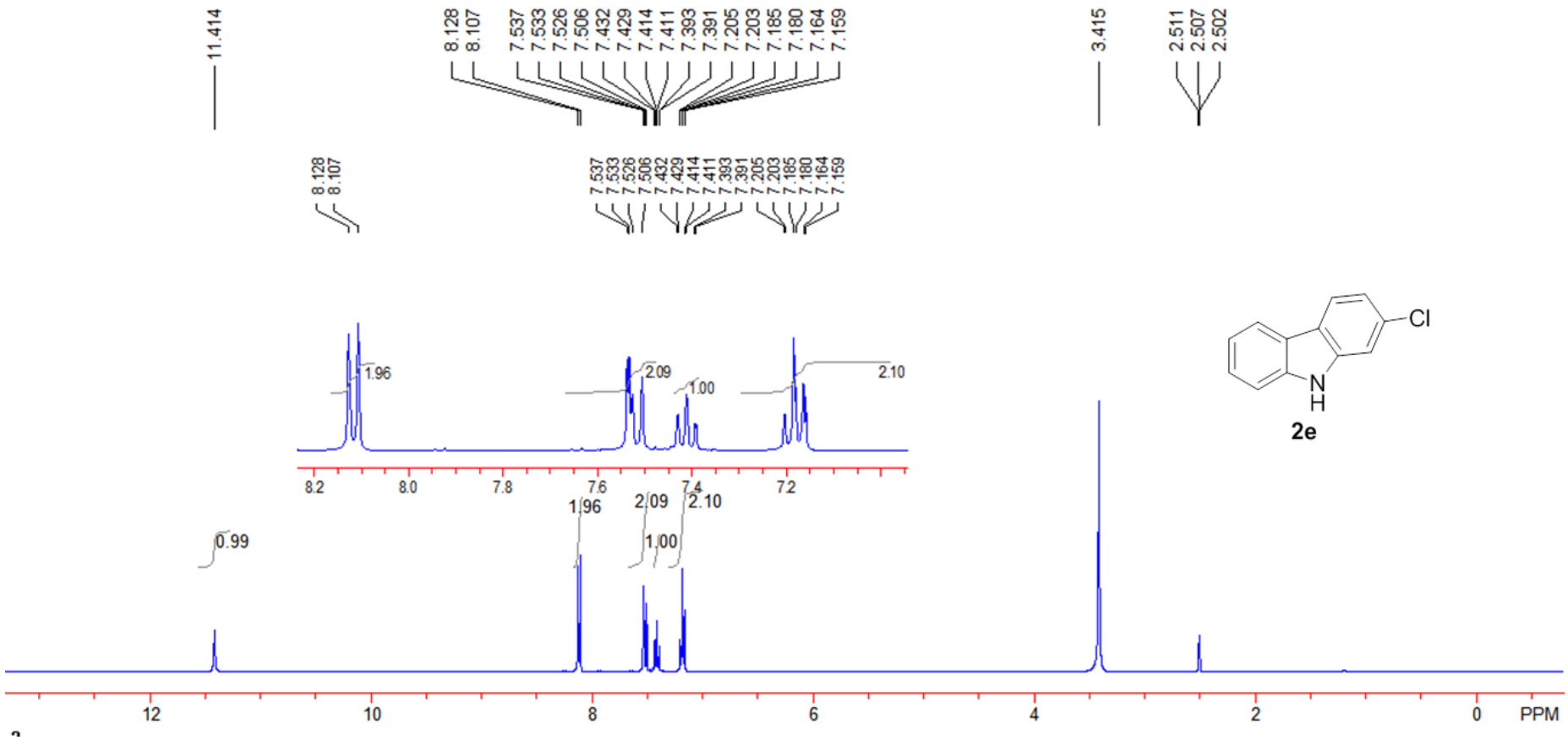


2c





2d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7.185
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2.09
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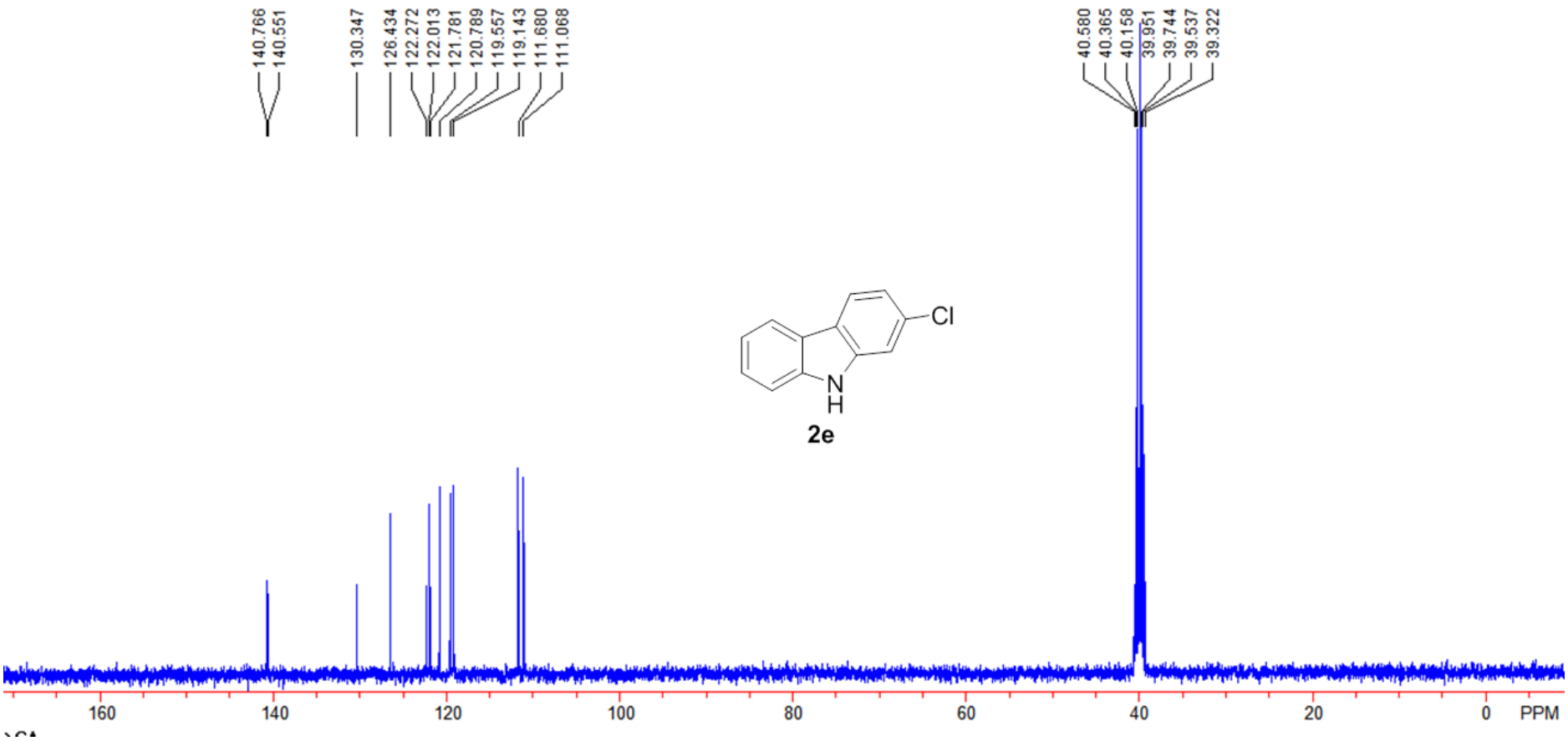
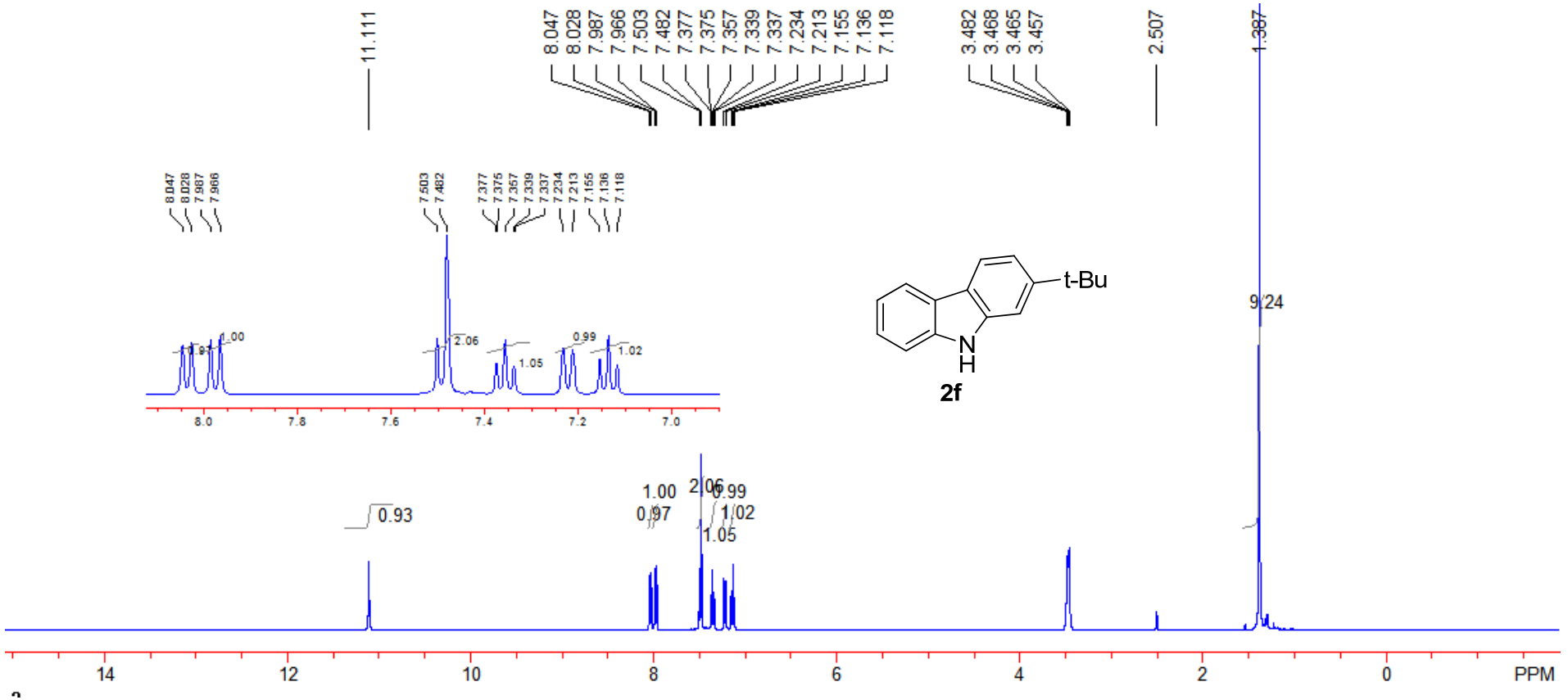
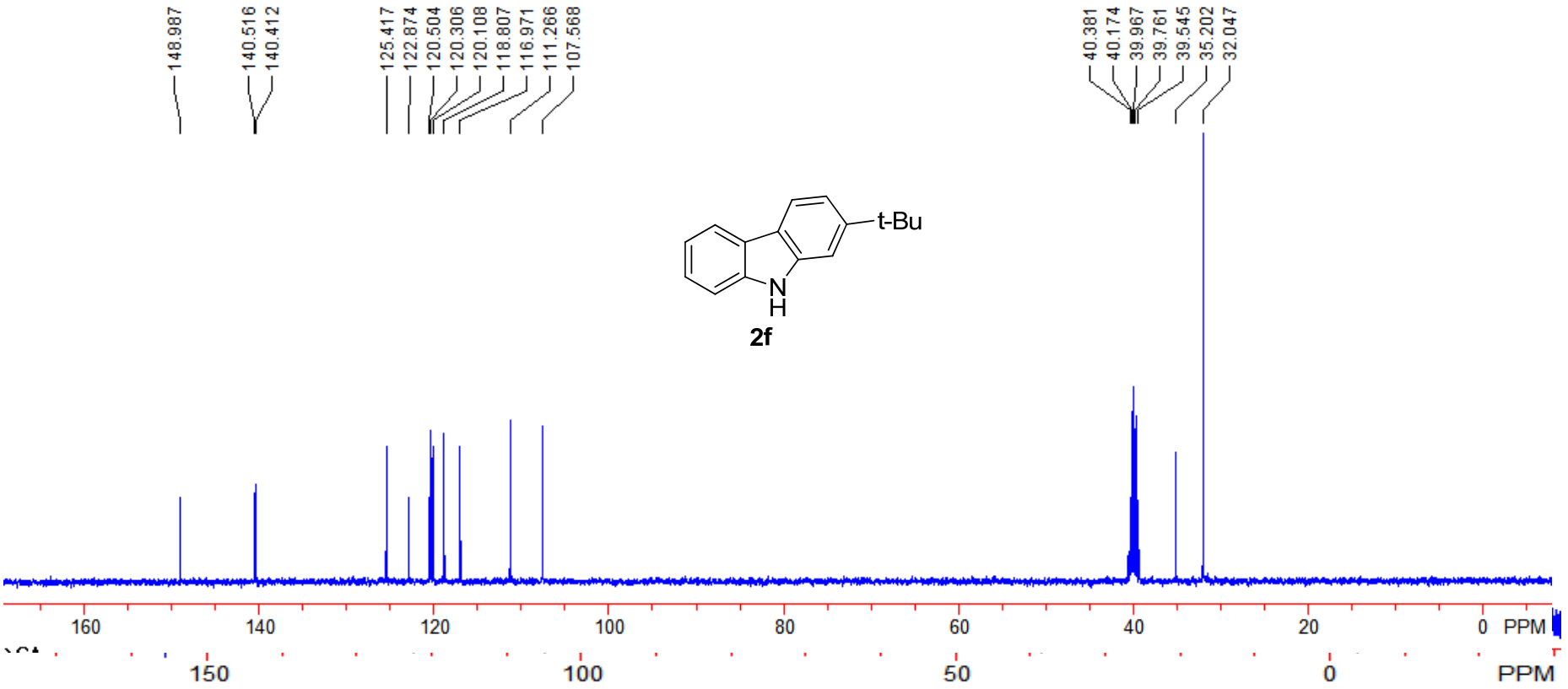
H
2e
PPM







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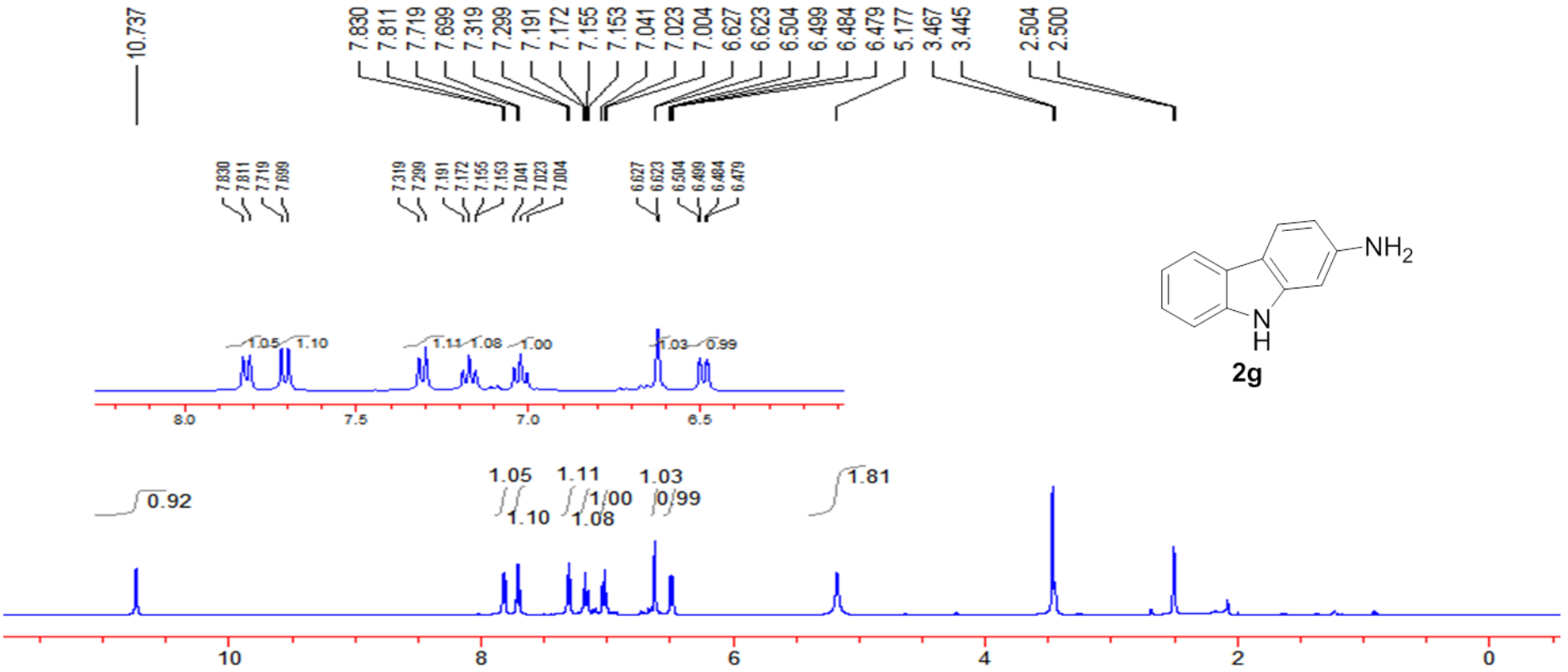

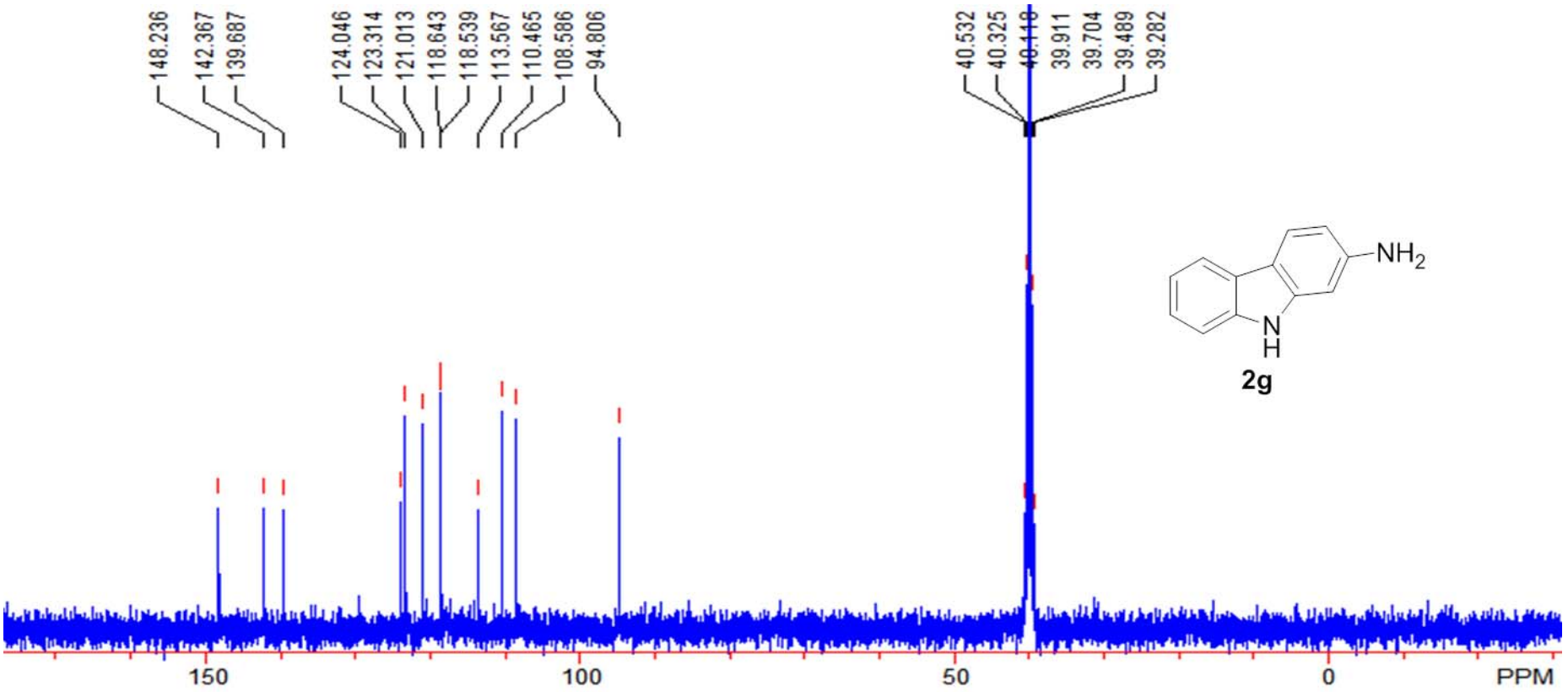

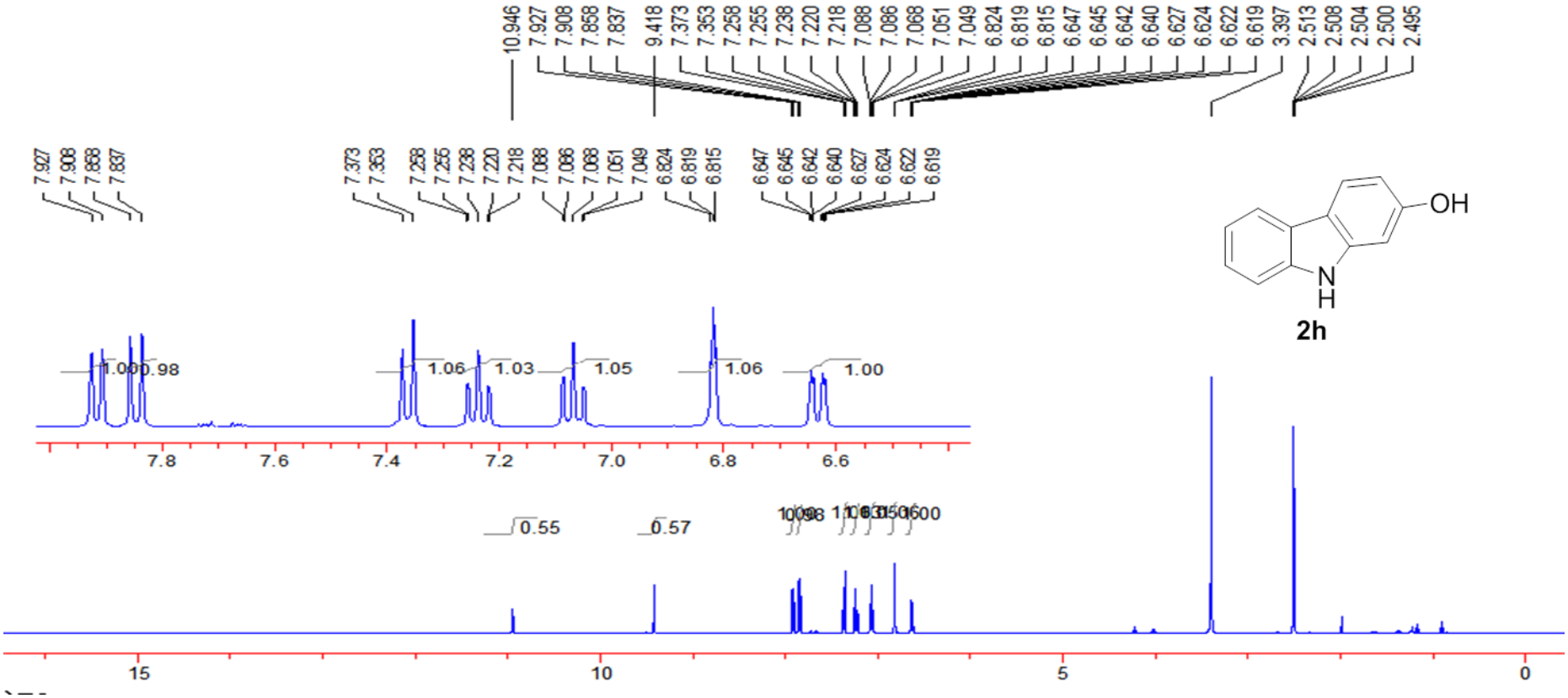
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1.05
1.06
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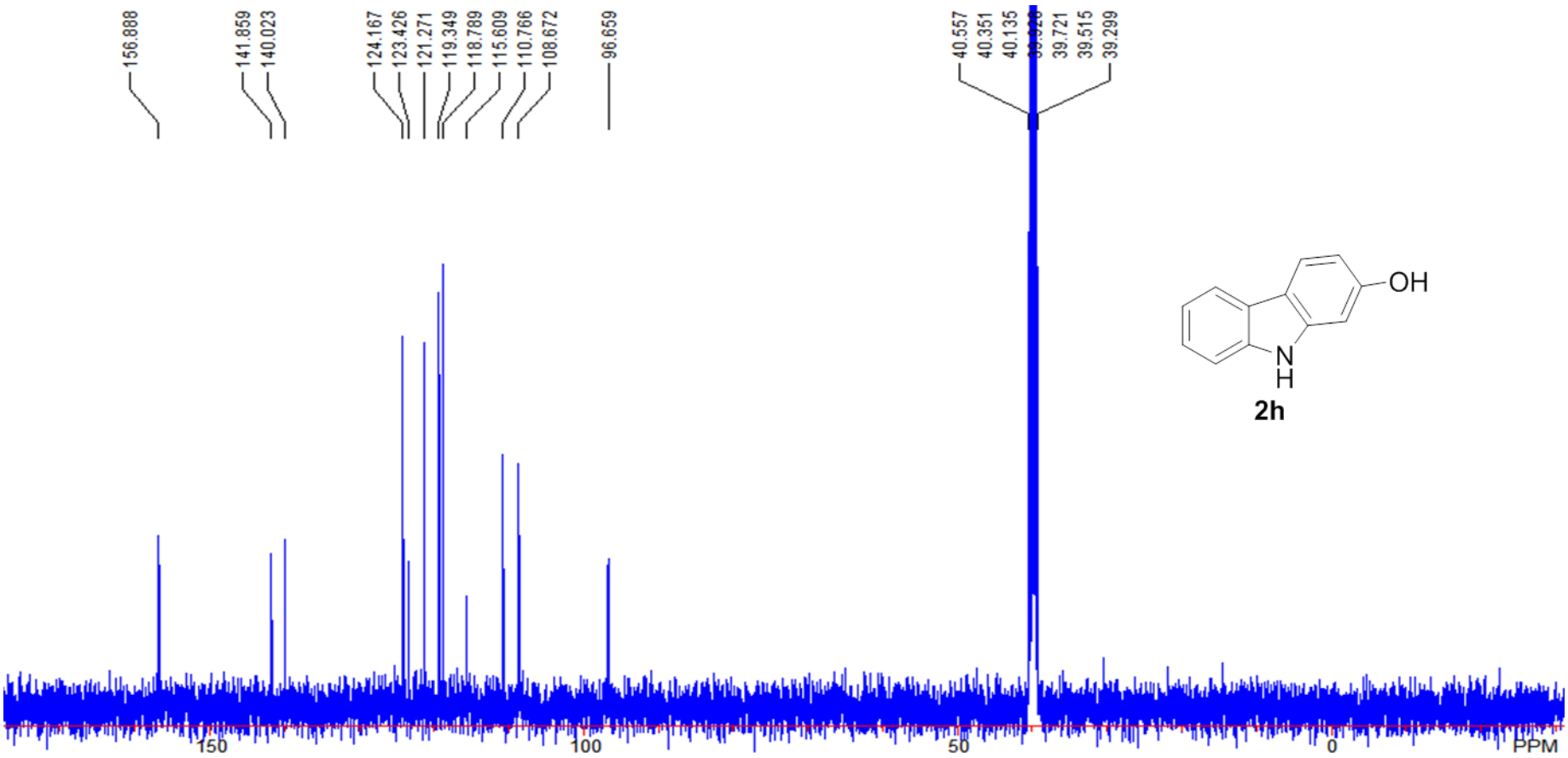

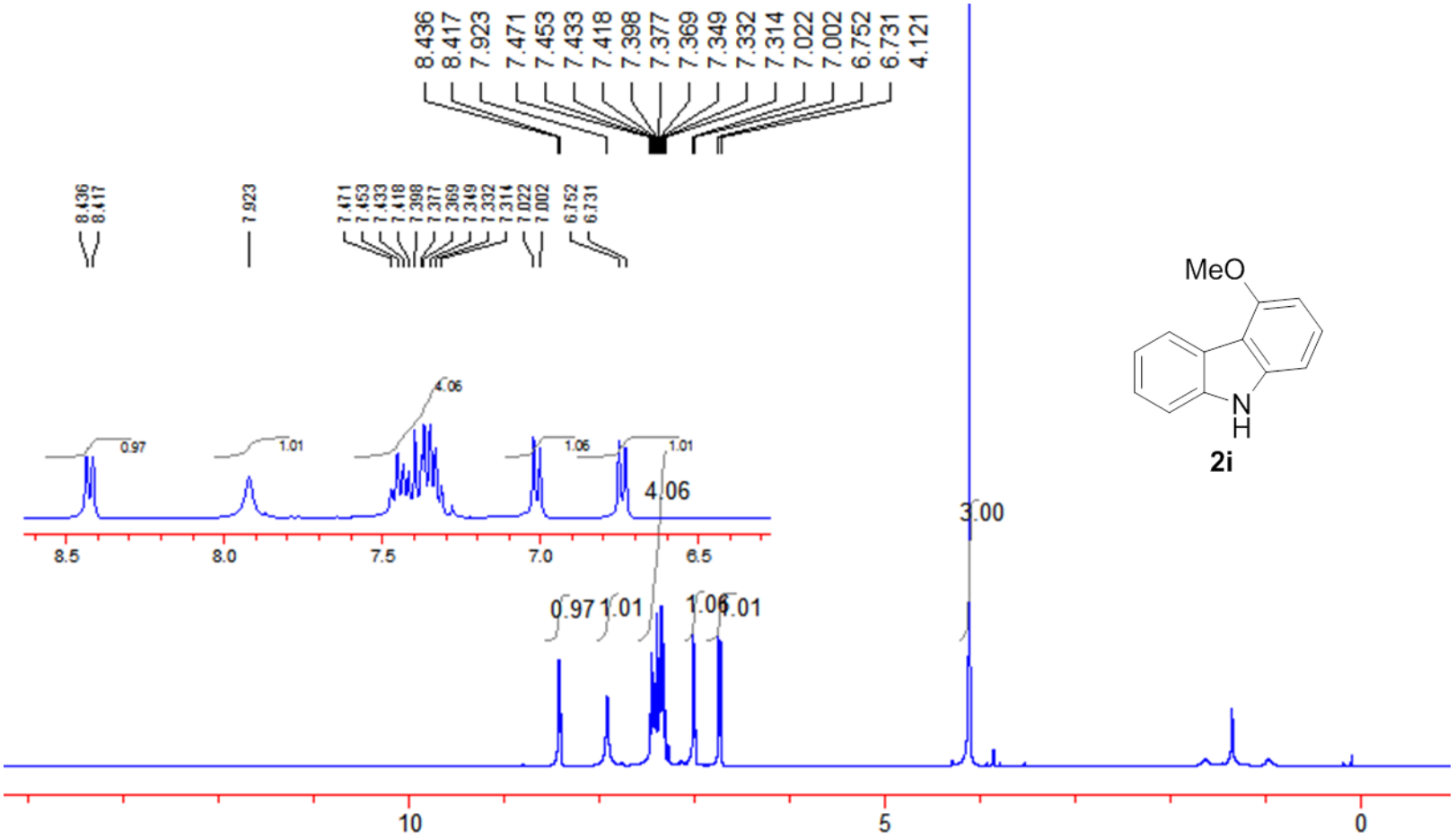

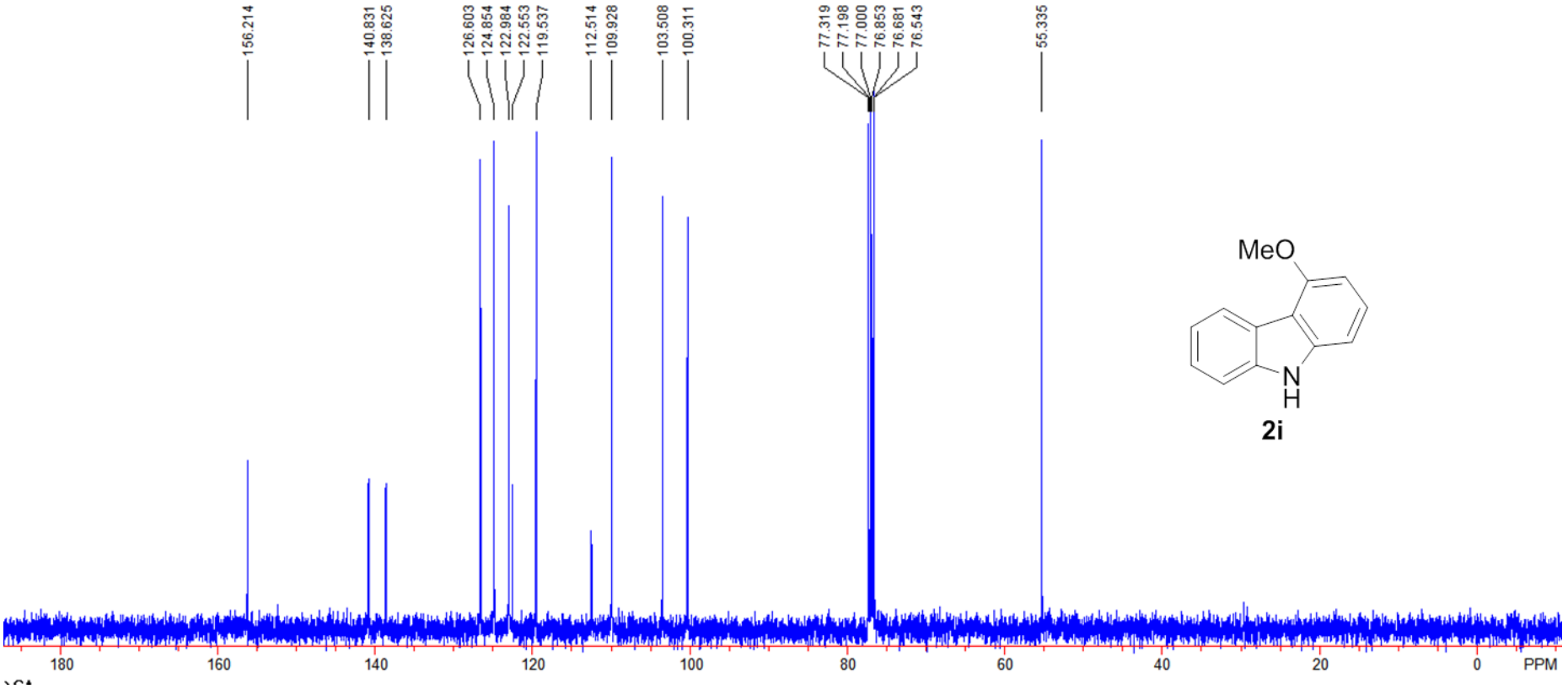
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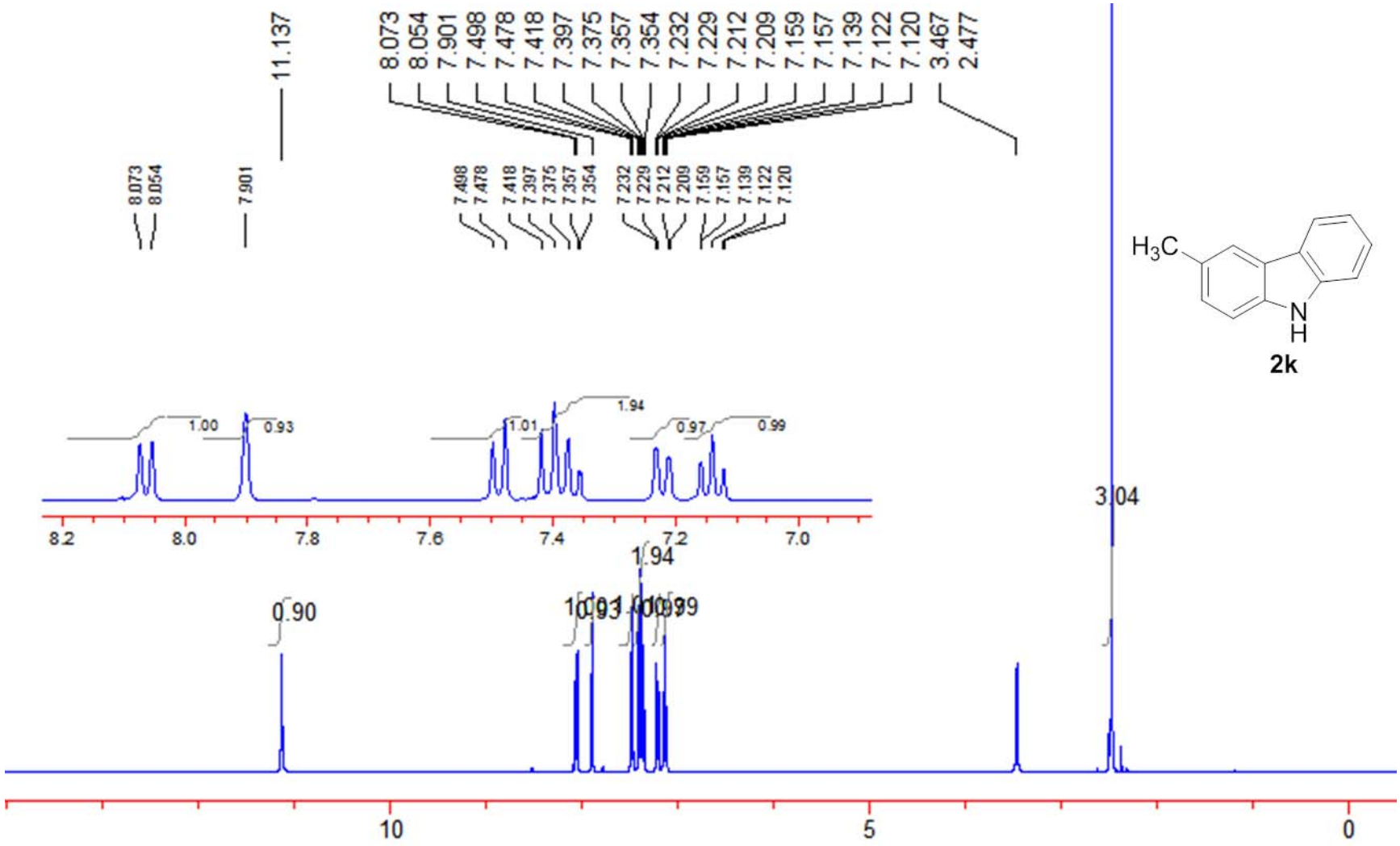

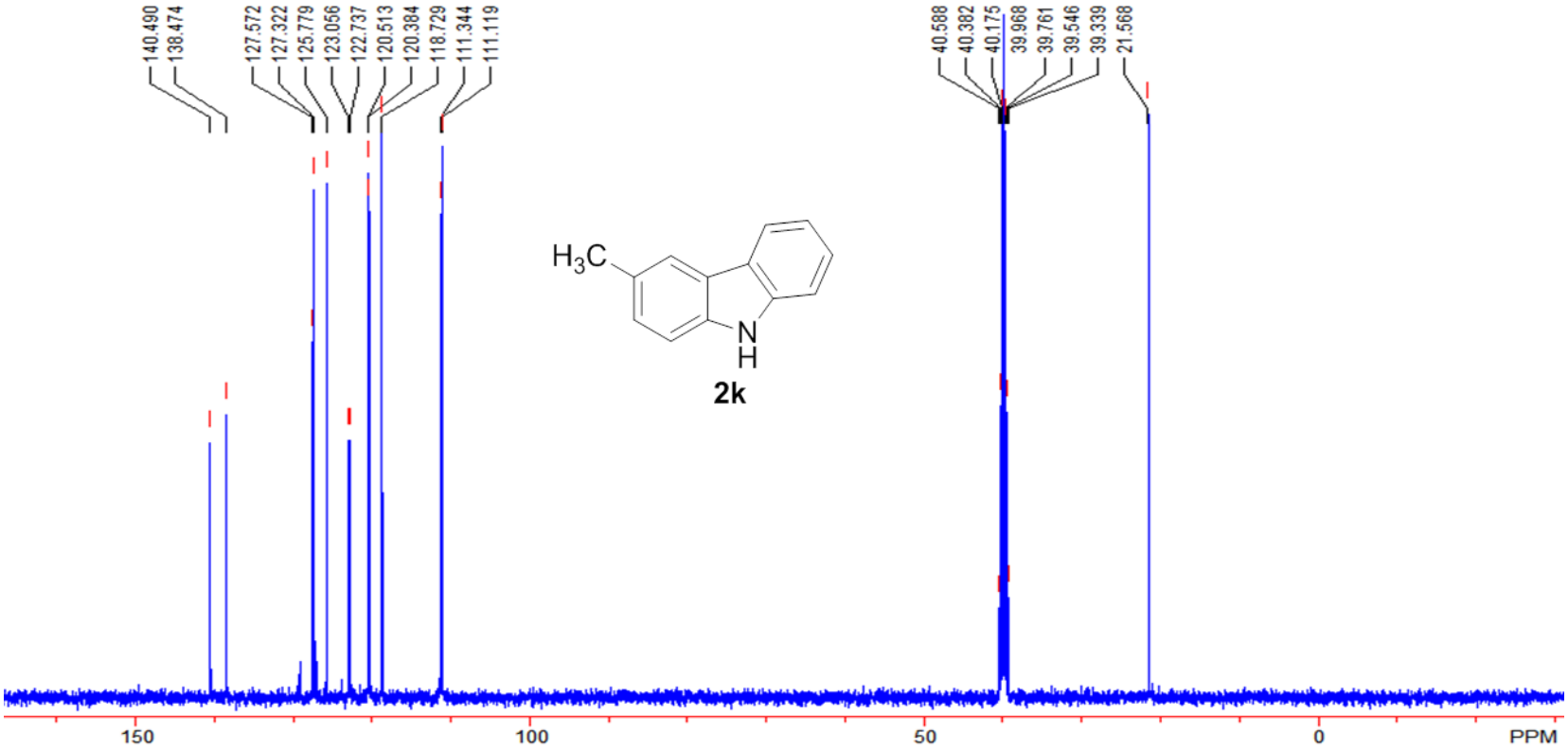
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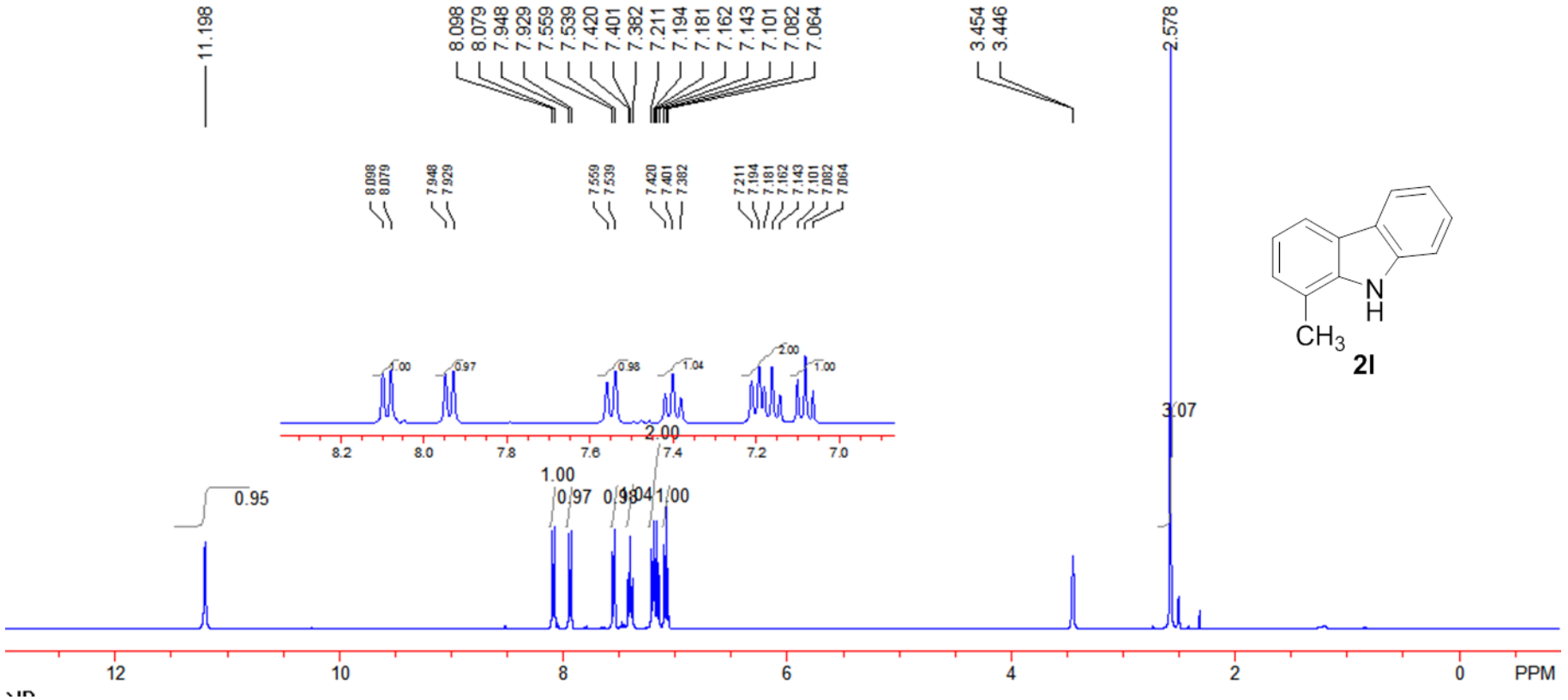
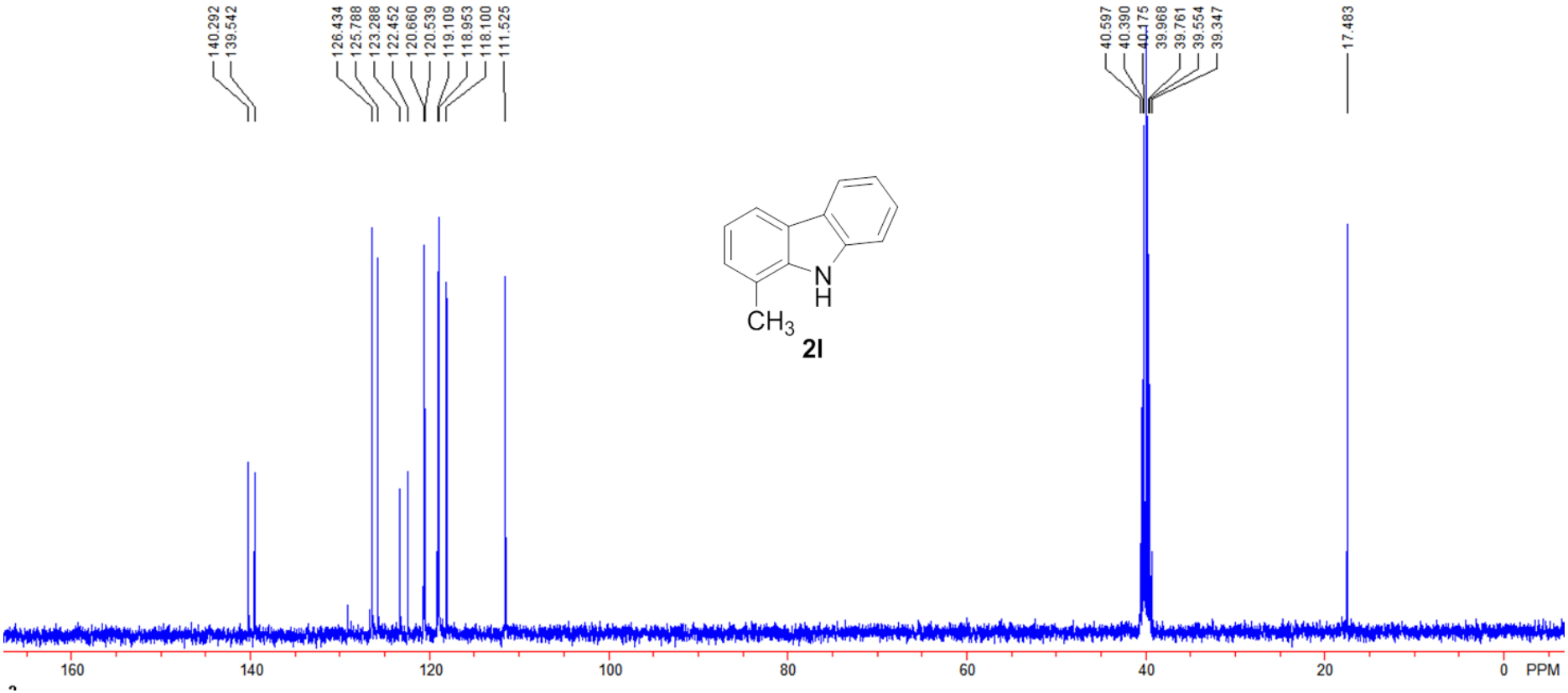
2k





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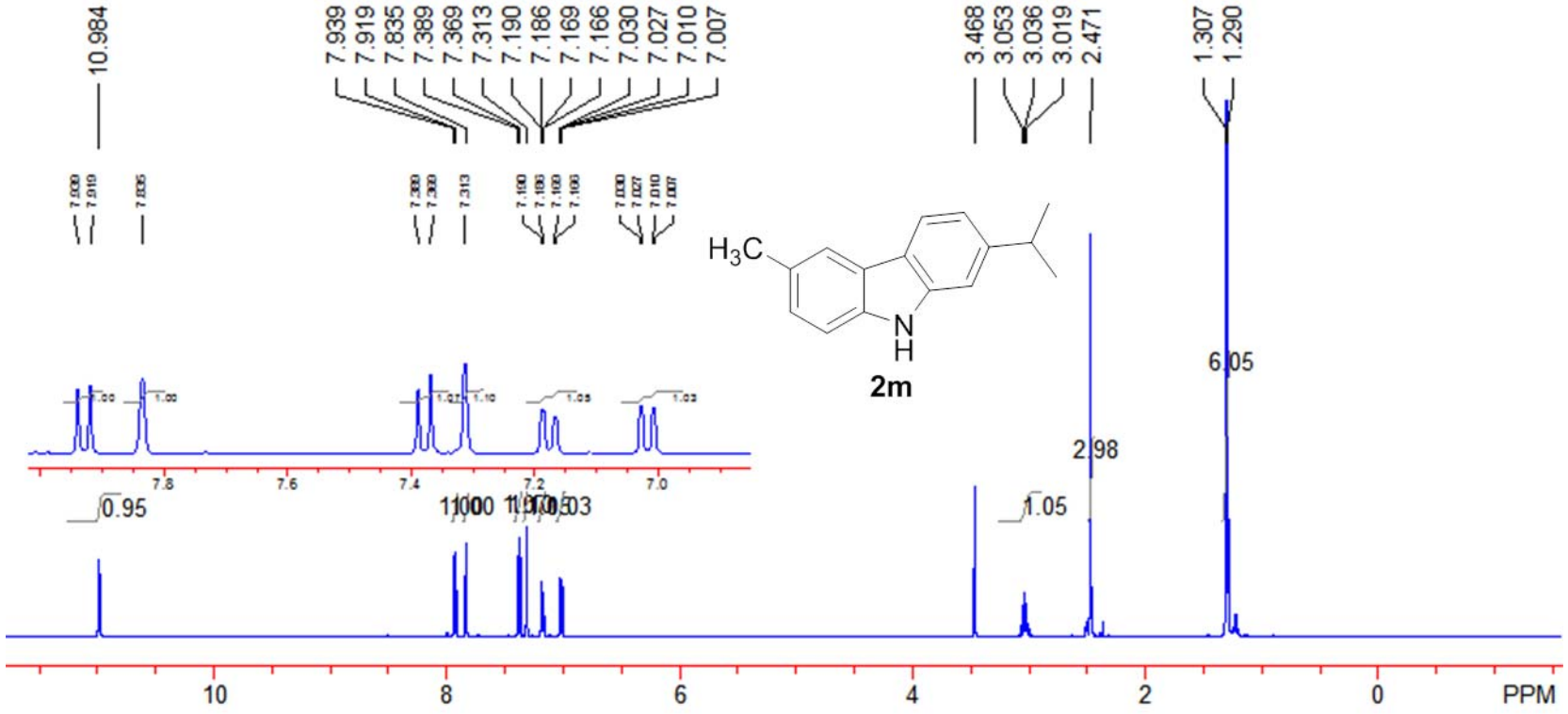

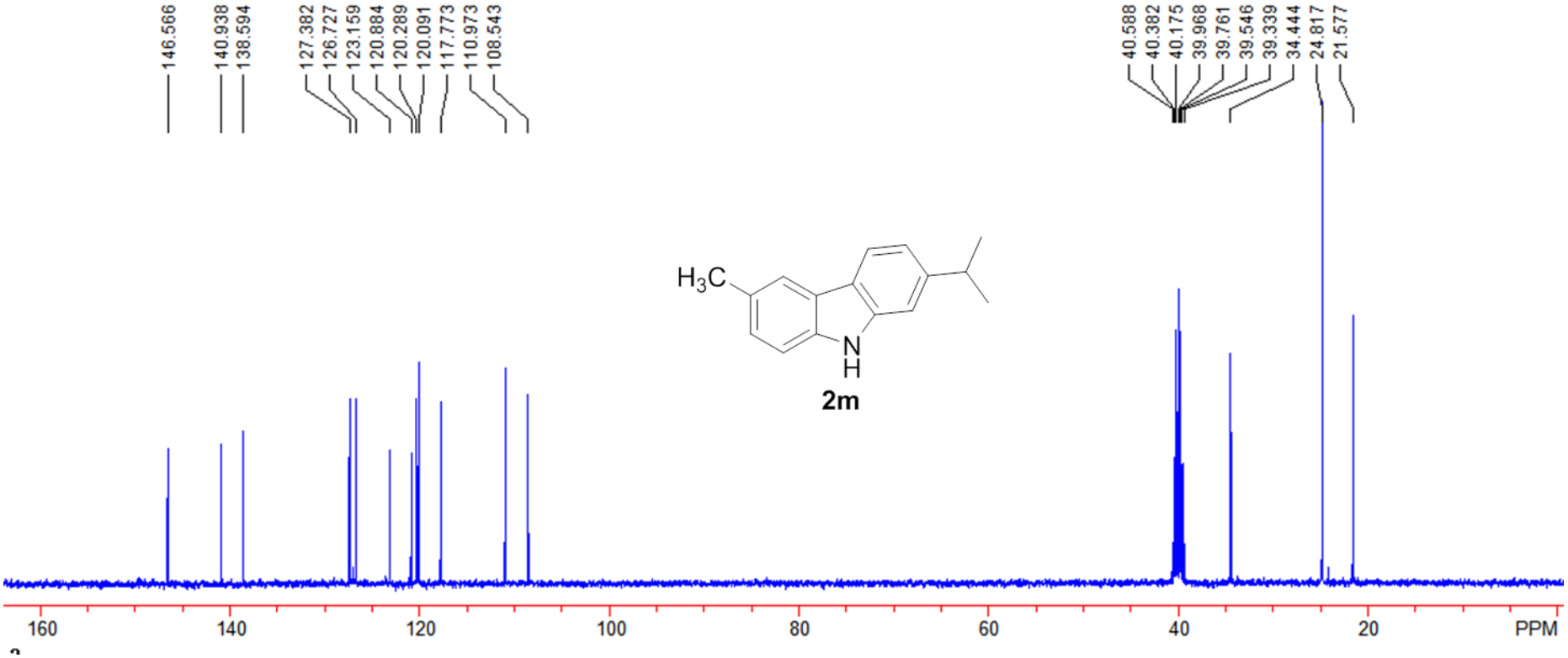

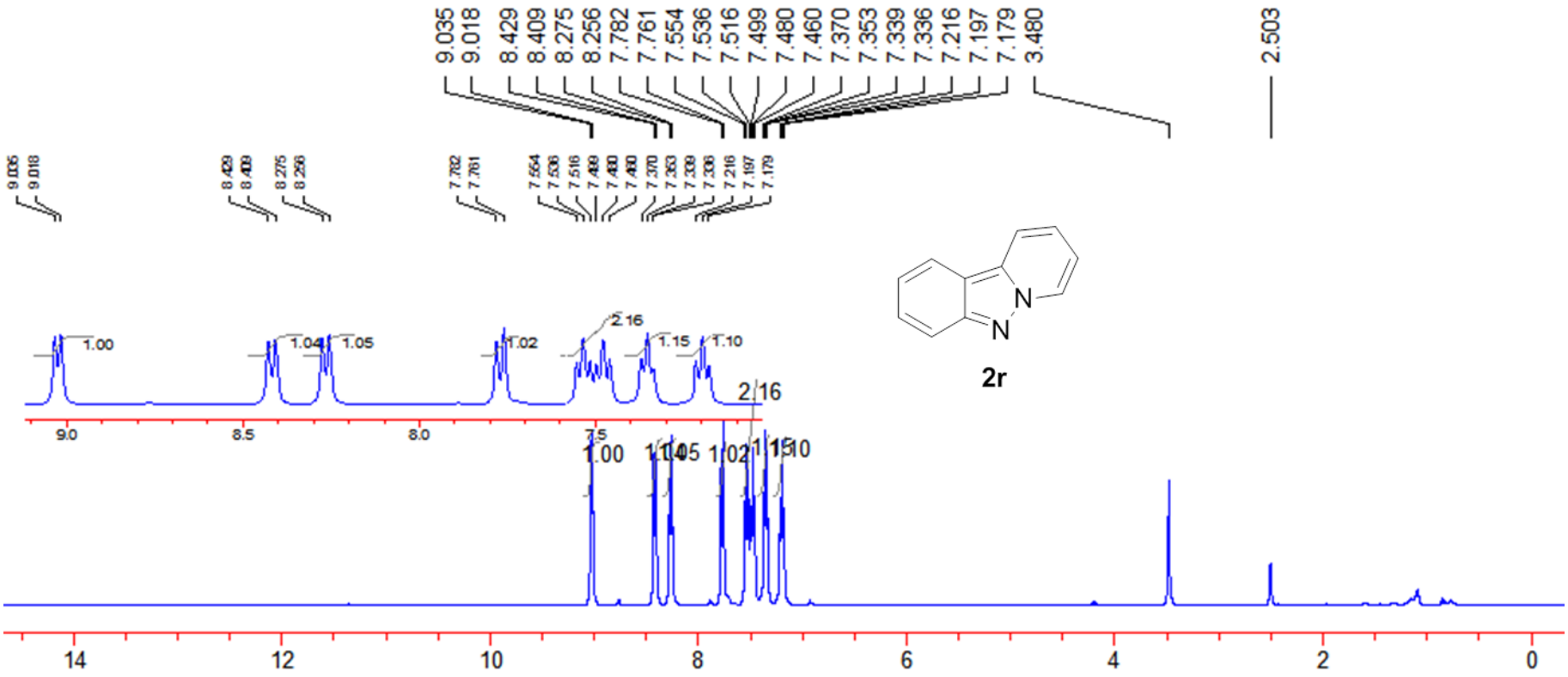

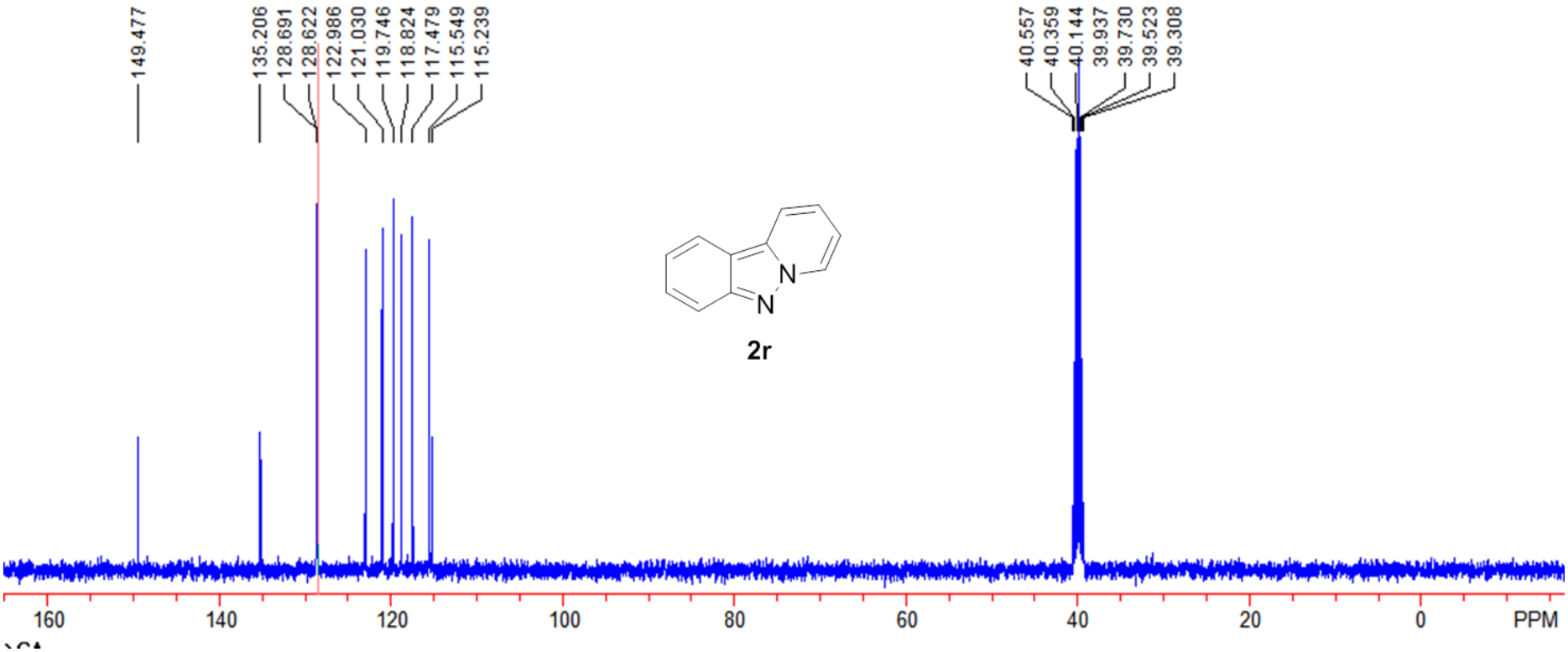

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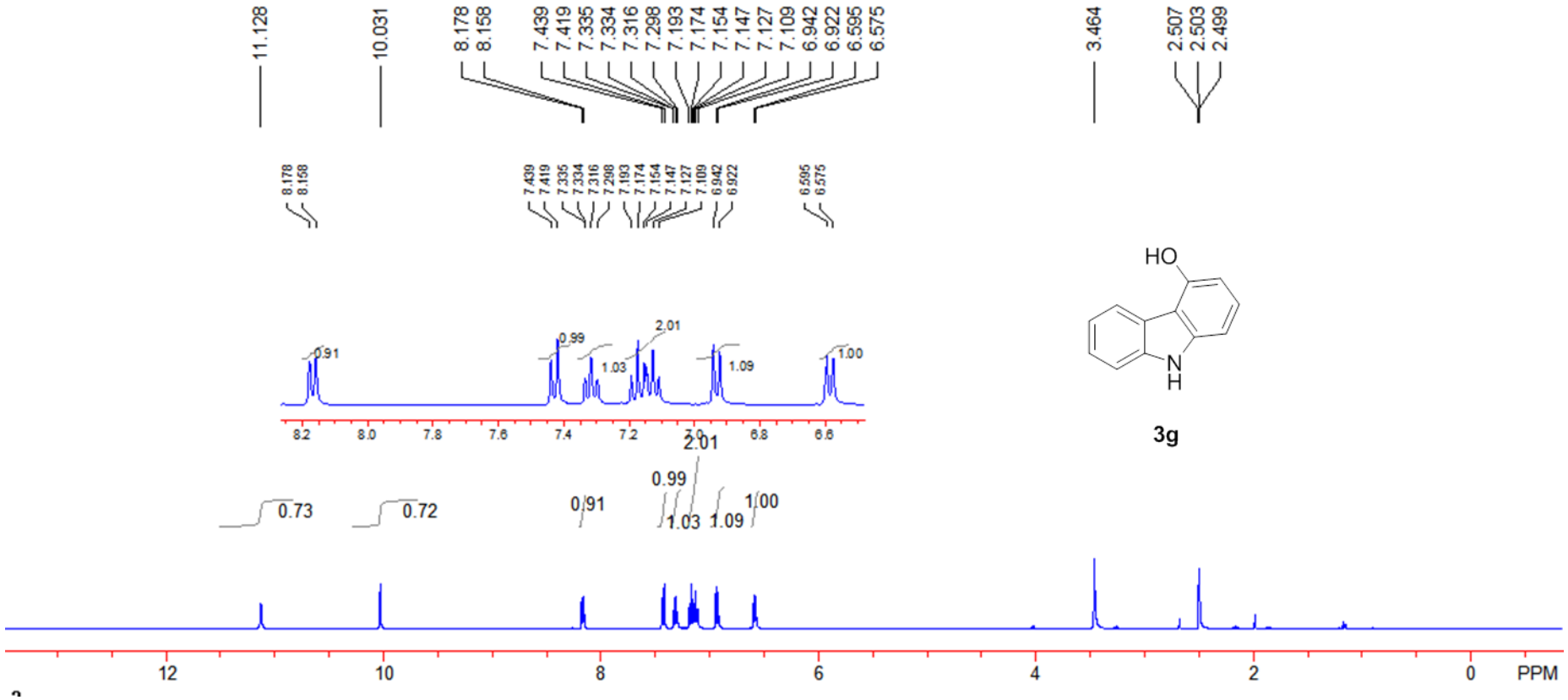

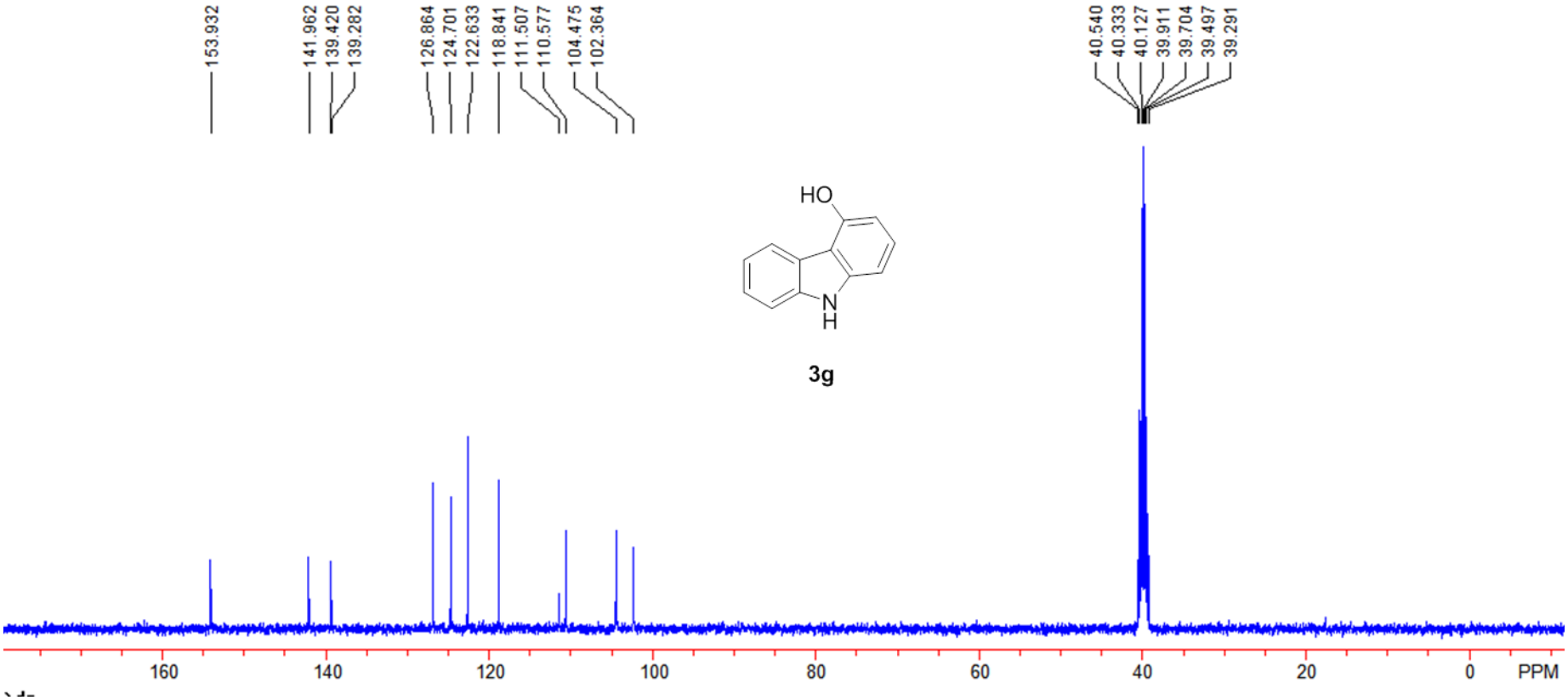

3g