Supporting Infomation

Iron(III)-Catalyzed Selective N−O Bond Cleavage to Prepare Tetrasubstituted Pyridines and 3,5-Disubstituted Isoxazolines from N-Vinyl-α,β-Unsaturated Ketonitrones

Chun-Hua Chen, Qing-Yan Wu, Cui Wei, Cui Liang, Gui-Fa Su*, and Dong-Liang Mo*

State Key Laboratory for Chemistry and Molecular Engineering of Medicinal Resources, Ministry of Science and Technology of China; School of Chemistry and Pharmaceutical Sciences, Guangxi Normal University, 15 Yu Cai Road, Guilin 541004, China.

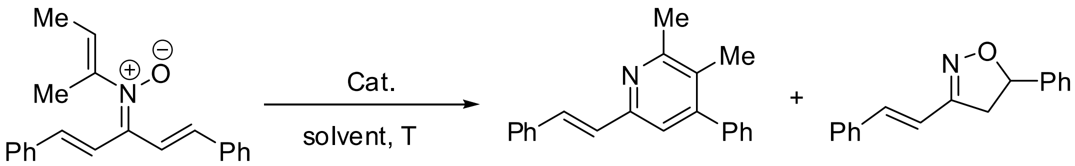
Contents

1. General Experimental Information

[1]H NMR and [13]C NMR spectra were recorded at ambient temperature using 400, 500 or 600 MHz spectrometers. The data are reported as follows: chemical shift in ppm from internal tetramethylsilane on the δ scale, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. High resolution mass spectra were acquired on an LTQ FT spectrometer, and were obtained by peak matching. Melting points are reported uncorrected. Analytical thin layer chromatography was performed on 0.25 mm extra hard silica gel plates with UV254 fluorescent indicator. Chromatography was performed using with 300-400 mesh silica gel (SiO_2). Unless otherwise noted, all reagents and solvents were obtained from commercial sources and, where appropriate, purified prior to use.

2. Optimization tables for the formation of 2a and 3a

Table S1. Optimization Conditions for formation of **2a**.[a]



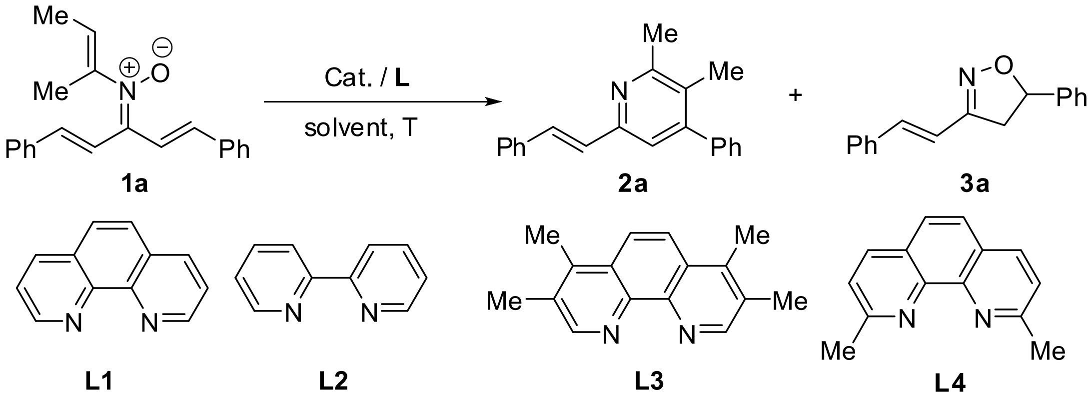
1a **2a** **3a**

entry	Cat.	solvent	T (°C)	**2a**%[b]	**3a**%[b]
1	-	THF	80	<5	<5
2	$FeCl_3$•$6H_2O$	THF	80	33	<5
3	$FeCl_3$	THF	80	39	<5
4	$FeCl_3$	MeCN	80	30	<5
5	$FeCl_3$	toluene	80	38	<5
6	$FeCl_3$	DCE	80	24	<5
7	$FeCl_3$	DMSO	80	27	<5
8	$FeCl_3$	dioxane	80	21	<5
9	$FeCl_3$	MeOH	80	46	<5
10	$FeCl_3$	*t*-BuOH	80	51	<5
11	$FeCl_3$	*i*-PrOH	80	56	<5
12[c]	$FeCl_3$	*i*-PrOH	80	65	<5
13[d]	$FeCl_3$	*i*-PrOH	80	35	<5
14[e]	$FeCl_3$	*i*-PrOH	80	52	<5
15[c]	$FeCl_3$	*i*-PrOH	70	58	<5
16[c]	$FeCl_3$	*i*-PrOH	100	64	<5

17	$FeCl_2$	*i*-PrOH	80	17	<5
18	$Fe(OTf)_2$	*i*-PrOH	80	32	<5
19	$Fe(OTf)_3$	*i*-PrOH	80	33	<5
20	CuCl	*i*-PrOH	80	<5	<5
21	$Pd(OAc)_2$	*i*-PrOH	80	11	<5

[a]Reaction conditions: **1a** (0.2 mmol), Cat. (20 mol% unless noted), solvent (2 mL), 80 °C, 12−24 h; [b]Isolated yield; [c]3Å MS (200 mg) was added; [d]4Å MS (200 mg) was added; [e]5Å MS (200 mg) was added.

Table S2. Optimization Conditions for formation of **3a**.[a]

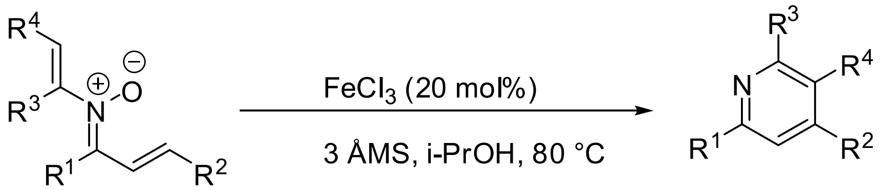


entry	Cat. / **L**	solvent	T (°C)	**2a**%[b]	**3a**%[b]
1	$FeCl_3$/**L1**	*i*-PrOH	80	10	31
2	$FeCl_3$/**L1**	MeCN	80	14	38
3	$FeCl_3•6H_2O$/ **L1**	MeCN	80	18	43
4	$FeCl_3•6H_2O$/ **L1**	DCE	80	15	26
5	$FeCl_3•6H_2O$/ **L1**	toluene	80	<5	60
6	$FeCl_3•6H_2O$/ **L1**	DMSO	80	<5	57
7	$FeCl_3•6H_2O$/ **L1**	DMF	80	<5	63
8	$FeCl_3•6H_2O$/ **L1**	dioxane	80	<5	78
9	$FeCl_3•6H_2O$/ **L1**	THF	80	<5	79
10	$FeCl_3•6H_2O$/ **L1**	THF	60	<5	75
11	$FeCl_3•6H_2O$/ **L1**	THF	40	<5	50
12	$FeCl_3•6H_2O$/ **L1**	THF	25	<5	<5
13	$FeCl_3•6H_2O$/ **L1**	THF	100	<5	58
14[c]	$FeCl_3•6H_2O$/ **L1**	THF	80	<5	78
15[d]	$FeCl_3•6H_2O$/ **L1**	THF	80	<5	59
16	$FeCl_3•6H_2O$/ **L2**	THF	80	<5	32
17	$FeCl_3•6H_2O$/ **L3**	THF	80	<5	73
18	$FeCl_3•6H_2O$/ **L4**	THF	80	<5	71
19	$Fe(ClO_4)_3•9H_2O$/ **L1**	THF	80	<5	36
20	$Fe(OTf)_3$/ **L1**	THF	80	<5	24
21	$FeCl_2$/ **L1**	THF	80	<5	<5

22	$Fe(OTf)_2$/ **L1**	THF	80	<5	<5
23	$Cu(OAc)_2$/**L1**	MeCN	80	10	<5
24	CuCl/**L1**	MeCN	80	6	<5
25	$Pd(OAc)_2$/**L1**	MeCN	80	<5	23

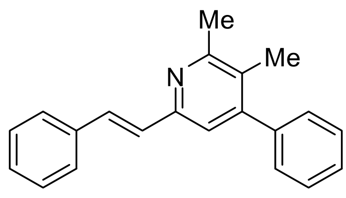
[a]Reaction conditions: **1a** (0.2 mmol), Cat. (20 mol% unless noted), **L** (40 mol% unless noted), solvent (2 mL), 25 °C, 7−24 h; [b]Isolated yield; [c] $FeCl_3•6H_2O$ (20 mol%), **L1** (60 mol%); [d] $FeCl_3•6H_2O$ (10 mol%), **L1** (20 mol%).

3. Synthesis of tetrasubstituted pyridines 2



1 **2**

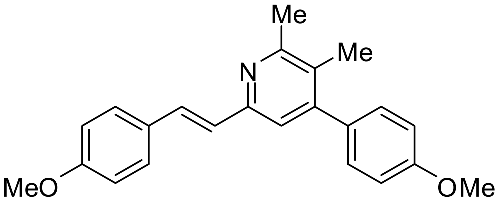
General procedure A: A Teflon-sealed flask was charged with nitrone **1** (0.2 mmol), $FeCl_3$ (7 mg, 20 mol%), 3ÅMS (200 mg) under N_2 atmosphere. *i*-PrOH (2 mL) was then added via syringe and the reaction vessel was once again sealed with a Teflon cap. The reaction mixture was stirred at 25 °C for 5 min and then heated at 80 °C for 12–15 h until nitrone **1** was consumed completely (monitored by TLC). At this time, the solvent was removed under reduced pressure and the crude product was purified by flash column chromatography (the crude residue was dry loaded on silica gel, 1/50 to 1/20, ethyl acetate/petroleum ether) to afford tetrasubstituted pyridines **2**.



2a

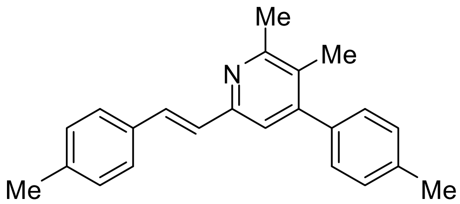
(*E*)-2,3-Dimethyl-4-phenyl-6-styrylpyridine (2a)[1], light yellow solid, 0.036 g, 65% yield. Mp: 101−102 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49−7.45 (m, 3H), 7.39−7.32 (m, 3H), 7.29−7.17 (m, 5H), 7.12−7.08 (m, 2H), 2.54 (s, 3H), 2.11 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.7, 151.9, 149.9, 140.0, 136.7, 131.5, 128.7, 128.6, 128.4, 128.3, 127.9, 127.7, 127.5, 126.9, 120.4, 23.6, 16.2; HRMS (ESI) *m/z* calcd for

$C_{21}H_{20}N$ $(M+H)^+$ 286.1590, found 286.1578.



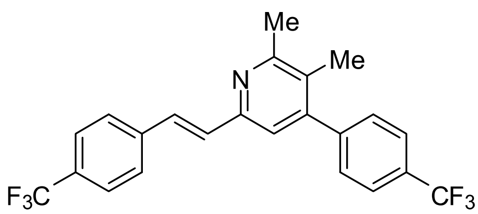
2b

(*E*)-4-(4-Methoxyphenyl)-6-(4-methoxystyryl)-2,3-dimethylpyridine (2b), light yellow solid, 0.016 g, 23% yield. Mp: 115−116 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44−7.40 (m, 3H), 7.19−7.17 (m, 2H), 7.06 (s, 1H), 6.99 (d, J = 16.4 Hz, 1H), 6.92−6.90 (m, 2H), 6.83−6.81 (m, 2H), 3.79 (s, 3H), 3.75 (s, 3H), 2.53 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.6, 159.2, 157.4, 152.2, 149.6, 132.4, 131.1, 129.9, 129.8, 128.2, 127.2, 126.2, 120.2, 114.1, 113.7, 55.3, 55.3, 23.6, 16.2; IR (thin film) 2976, 2899, 1651, 1453, 1380, 1327, 1087, 973, 881, 692 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{23}H_{24}NO_2$ $(M+H)^+$ 346.1802, found 346.1778.



2c

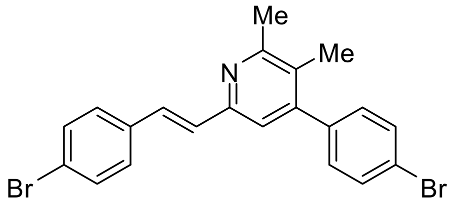
(*E*)-2,3-Dimethyl-6-(4-methylstyryl)-4-*p*-tolylpyridine (2c), yellow oil , 0.035 g, 56% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44−7.36 (m, 3H), 7.18−7.11 (m, 4H), 7.08−7.02 (m, 4H), 2.52 (s, 3H), 2.33 (s, 3H), 2.26 (s, 3H), 2.10 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.5, 152.1, 149.9, 137.9, 137.4, 137.1, 134.2, 131.4, 129.3, 128.9, 128.6, 127.4, 127.3, 126.8, 120.3, 23.6, 21.2, 21.2, 16.1; IR (thin film) 2955, 2924, 2856, 1583, 1513, 1456, 1380, 971, 821, 742 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{23}H_{24}N$ $(M+H)^+$ 314.1903, found 314.1882.



2d

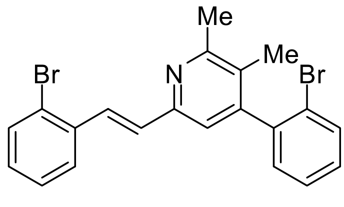
(*E*)-2,3-Dimethyl-4-(4-(trifluoromethyl)phenyl)-6-(4-(trifluoromethyl)styryl)pyri

dine (2d), yellow oil, 0.045 g, 53% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91−7.85 (m, 2H), 7.83−7.74 (m, 5H), 7.60 (d, J = 8.0 Hz, 2H), 7.40−7.36 (m, 1H), 7.29−7.28 (m, 1H), 2.78 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.3, 151.4, 148.6, 143.5, 141.7, 140.3, 138.0, 130.3, 130.1 (q, J = 31.9 Hz), 129.6 (q, J = 236.1 Hz), 129.1, 127.2, 127.0, 125.9 (q, J = 36.3 Hz), 125.6 (q, J = 27.4 Hz), 125.3 (q, J = 270.5 Hz), 120.7, 23.6, 16.1; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.5; IR (thin film) 2926, 2857, 1657, 1613, 1585, 1325, 1016, 968, 841, 685 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{23}H_{18}F_6N$ $(M+H)^+$ 422.1338, found 422.1311.



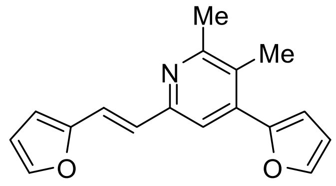
2e

(*E*)-4-(4-Bromophenyl)-6-(4-bromostyryl)-2,3-dimethylpyridine (2e), light yellow solid, 0.051 g, 58% yield. Mp: 102−103 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.60−7.58 (m, 2H), 7.52−7.47 (m, 3H), 7.42−7.41 (m, 2H), 7.20−7.16 (m, 3H), 7.13−7.10 (m, 1H), 2.62 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.9, 151.6, 148.9, 138.7, 135.8, 131.8, 131.6, 130.6, 130.4, 128.5, 128.4, 127.7, 122.1, 121.9, 120.4, 23.5, 16.1; IR (thin film) 2923, 1592, 1486, 1453, 1381, 1070, 1006, 962, 823 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{21}H_{18}Br_2N$ $(M+H)^+$ 441.9801, found 441.9771.



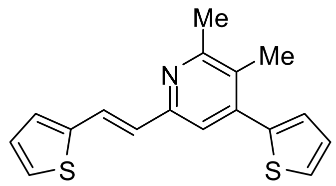
2f

(*E*)-4-(2-Bromophenyl)-6-(2-bromostyryl)-2,3-dimethylpyridine (2f), light yellow solid, 0.047 g, 54% yield. Mp: 118−119 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.86 (d, J = 15.6 Hz, 1H), 7.73−7.68 (m, 2H), 7.58 (d, J = 7.8 Hz, 1H), 7.42−7.39 (m, 1H), 7.32−7.26 (m, 2H), 7.22−7.21 (m, 1H), 7.18−7.11 (m, 3H), 2.64 (s, 3H), 2.06 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.5, 151.6, 140.7, 136.7, 133.1, 132.8, 130.2, 129.4, 129.2, 127.6, 127.4, 127.0, 124.4, 122.7, 120.0, 23.3, 15.9; IR (thin film) 2977, 2899 , 1649, 1453, 1381, 1275, 1087, 1048, 880, 692 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{21}H_{18}Br_2N(M+H)^+$ 441.9801, found 441.9780.



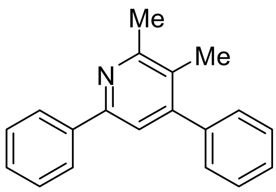
2g

(*E*)-4-(Furan-2-yl)-6-(2-(furan-2-yl)vinyl)-2,3-dimethylpyridine (2g), yellow oil, 0.025 g, 47% yield. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.57 (s, 1H), 7.47−7.42 (m, 3H), 7.08 (d, J = 15.5 Hz, 1H), 6.70 (d, J = 3.5 Hz, 1H), 6.55−6.54 (m, 1H), 6.44−6.42 (m, 2H), 2.62 (s, 3H), 2.42 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 158.1, 153.2, 151.8, 151.6, 142.9, 142.5, 137.5, 126.3, 125.6, 119.2, 117.8, 111.7, 111.6, 111.3, 109.9, 23.9, 16.6; IR (thin film) 3119, 2924, 2856, 1640, 1597, 1454, 1014, 965, 885, 738 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{16}NO_2$ $(M+H)^+$ 266.1176, found 266.1162.



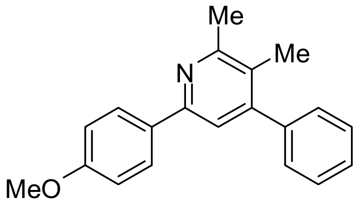
2h

(*E*)-2,3-Dimethyl-4-(thiophen-2-yl)-6-(2-(thiophen-2-yl)vinyl)pyridine(2h), light yellow solid, 0.031 g, 53% yield. Mp: 72−73 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, J = 16.0 Hz, 1H), 7.42−7.41 (m, 1H), 7.23−7.21 (m, 2H), 7.15−7.13 (m, 3H), 7.02−7.00 (m, 1H), 6.98 (d, J = 16.0 Hz, 1H), 2.62 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.1, 151.6, 142.5, 142.3, 140.8, 127.7, 127.6, 127.6, 127.4, 127.3, 127.2, 126.3, 125.1, 124.8, 120.9, 23.8, 16.4; IR (thin film) 3071, 2922, 2855, 1583, 1543, 1454, 1387, 958, 854, 698 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{16}NS_2$ $(M+H)^+$ 298.0719, found 298.0699.



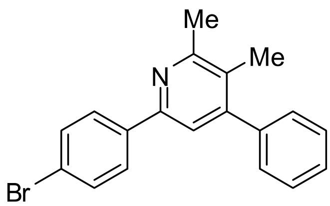
2i

2,3-Dimethyl-4,6-diphenylpyridine (2i)[1], light yellow solid, 0.038 g, 74% yield. Mp: 57−58 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.01−7.99 (m, 2H), 7.48−7.35 (m, 9H), 2.68 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.7, 153.6, 150.3, 140.2, 139.5, 128.8, 128.6, 128.5, 128.3, 127.7, 127.4, 126.8, 119.4, 23.6, 16.0; HRMS (ESI) *m/z* calcd for $C_{19}H_{18}N$ $(M+H)^+$ 260.1434, found 260.1417.



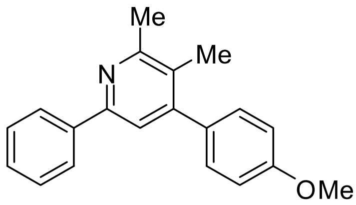
2j

6-(4-Methoxyphenyl)-2,3-dimethyl-4-phenylpyridine (2j), light yellow solid, 0.038 g, 65% yield. Mp: 80−81 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.97−7.95 (m, 2H), 7.48−7.45 (m, 2H), 7.42−7.39 (m, 2H), 7.35−7.34 (m, 2H), 6.98−6.97 (m, 2H) , 3.85 (s, 3H), 2.66 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 160.1, 157.5, 153.3, 150.2, 140.3, 132.2, 128.8, 128.3, 128.0, 127.6, 126.6, 118.6, 114.0, 55.3, 23.6, 16.0; IR (thin film) 2925, 1609, 1452, 1376, 1253, 1168, 974, 837, 776, 703 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{20}H_{20}NO$ $(M+H)^+$ 290.1539, found 290.1526.



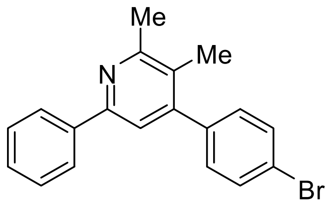
2k

6-(4-Bromophenyl)-2,3-dimethyl-4-phenylpyridine (2k), light yellow solid, 0.045 g, 67% yield. Mp: 88−89 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90−7.88 (m, 2H), 7.57−7.55 (m, 2H), 7.49−7.42 (m, 4H), 7.35−7.32 (m, 2H), 3.85 (s, 3H), 2.66 (s, 3H), 2.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.9, 152.4, 150.3, 140.1, 138.5, 131.7, 128.7, 128.4, 128.3, 127.8, 127.8, 122.8, 119.0, 23.7, 16.1; IR (thin film) 2924, 1663, 1589, 1547, 1492, 1451, 1008, 829, 771, 702 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{17}BrN$ (M+H)$^+$ 338.0539, found 338.0523.



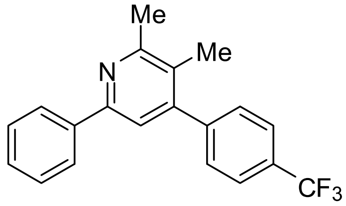
2l

4-(4-Methoxyphenyl)-2,3-dimethyl-6-phenylpyridine (2l), light yellow solid, 0.029 g, 50% yield. Mp: 99−100 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, *J* = 4.8 Hz, 2H), 7.46−7.44 (m, 3H), 7.39−7.36 (m, 1H), 7.30 (d, *J* = 5.6 Hz, 2H), 7.01 (d, *J* = 6.0 Hz, 2H), 3.88 (s, 3H), 2.66 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.2, 157.7, 153.6, 149.8, 139.7, 132.5, 130.0, 128.6, 128.3, 127.5, 126.8, 119.5, 113.8, 55.3, 23.7, 16.1; IR (thin film) 2924 , 1609, 1510, 1455, 1380, 1244, 1170, 826, 687 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{20}H_{20}NO$ (M+H)$^+$ 290.1539, found 290.1526.



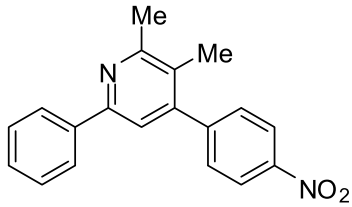
2m

4-(4-Bromophenyl)-2,3-dimethyl-6-phenylpyridine (2m), light yellow solid, 0.041 g, 61% yield. Mp: 89−90 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.00 (d, *J* = 4.8 Hz, 2H), 7.61 (d, *J* = 5.6 Hz, 2H), 7.46−7.44 (m, 2H), 7.40−7.38 (m, 2H), 7.23 (d, *J* = 5.2 Hz, 2H), 2.67 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.9, 153.8, 149.0, 139.3, 139.1, 131.5, 130.4, 128.6, 128.5, 127.2, 126.8, 122.0, 119.0, 23.6, 16.0; IR (thin film) 2924, 1595, 1455, 1377, 1325, 1166, 1069, 826, 775, 670 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{17}BrN$ (M+H)$^+$ 338.0539, found 338.0518.



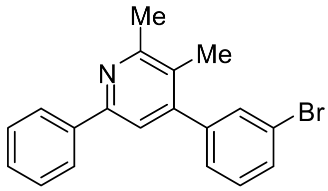
2n

2,3-Dimethyl-6-phenyl-4-(4-(trifluoromethyl)phenyl)pyridine (2n), light yellow solid, 0.039 g, 60% yield. Mp: 79−80 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.01−7.99 (m, 2H), 7.75 (d, J = 9.6 Hz, 2H), 7.48−7.45 (m, 4H), 7.42−7.38 (m, 2H), 2.69 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 158.0, 153.9, 148.9, 143.9, 139.2, 130.1 (q, J = 39.5 Hz), 129.2, 128.7, 128.6, 127.1(q, J = 267.8 Hz), 126.8, 125.4 (q, J = 33.0 Hz), 125.2, 118.9, 23.6, 16.0; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.5; IR (thin film) 3066, 2927, 1665, 1577, 1325, 1166, 1067, 838, 776, 695 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{20}H_{17}F_3N$ $(M+H)^+$ 328.1308, found 328.1291.



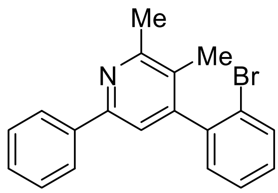
2o

2,3-Dimethyl-4-(4-nitrophenyl)-6-phenylpyridine (2o), light yellow solid, 0.038 g, 62% yield. Mp: 178−179 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.34 (d, J = 8.8 Hz, 2H), 8.01−7.98 (m, 2H), 7.53−7.51 (m, 2H), 7.47−7.39 (m, 4H), 2.68 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.2, 154.0, 147.9, 147.7, 146.9, 139.1, 129.8, 128.7, 128.7, 126.8, 126.7, 123.7, 118.5, 23.6, 16.0; IR (thin film) 2925, 1655, 1512, 1460, 1344, 1167, 973, 841, 693 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{17}N_2O_2$ $(M+H)^+$ 305.1285, found 305.1264.



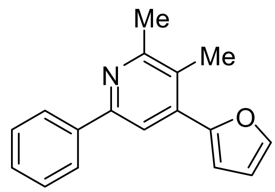
2p

4-(3-Bromophenyl)-2,3-dimethyl-6-phenylpyridine (2p), light yellow solid, 0.052 g, 76% yield. Mp: 98−99 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.91 (d, J = 8.0 Hz, 2H), 7.47−7.17 (m, 8H), 2.58 (s, 3H), 2.12 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.9, 153.8, 148.6, 142.3, 139.3, 131.7, 130.8, 129.9, 128.6, 128.5, 127.4, 127.2, 126.8, 122.4, 119.0, 23.6, 16.0; IR (thin film) 2960, 1652, 1489, 1450, 1325, 1186, 1070, 827, 773, 688 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{17}BrN$ $(M+H)^+$ 338.0539, found 338.0524.



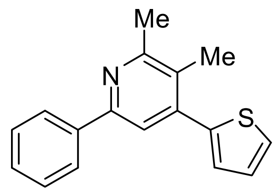
2q

4-(2-Bromophenyl)-2,3-dimethyl-6-phenylpyridine (2q), light yellow solid, 0.039 g, 58% yield. Mp: 106−107 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 (d, J = 7.2 Hz, 2H), 7.62 (d, J = 8.0 Hz, 1H), 7.38−7.27 (m, 5H), 7.21−7.14 (m, 2H), 2.59 (s, 3H), 2.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.6, 153.6, 149.3, 141.0, 139.5, 132.8, 130.3, 129.3, 128.6, 128.5, 128.0, 127.4, 126.8, 122.8, 118.9, 23.5, 15.7; IR (thin film) 3058, 2923, 2855, 1659, 1585, 1432, 1379, 1026, 979, 692 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{17}BrN$ $(M+H)^+$ 338.0539, found 338.0518.



2r

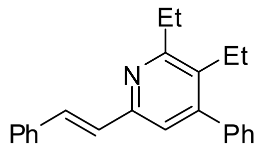
4-(Furan-2-yl)-2,3-dimethyl-6-phenylpyridine (2r), light yellow solid, 0.033 g, 67% yield. Mp: 64−65 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, J = 7.6 Hz, 2H), 7.85 (s, 1H), 7.59−7.58 (m, 1H), 7.49−7.45 (m, 2H), 7.41−7.39 (m, 1H), 6.74 (d, J = 3.6 Hz, 1H), 6.57−6.56 (m, 1H), 2.68 (s, 3H), 2.47 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.0, 154.0, 151.8, 142.9, 139.6, 137.8, 128.6, 128.4, 126.8, 125.6, 116.0, 111.6, 111.4, 23.9, 16.5; IR (thin film) 3056, 2924, 2856, 1664, 1595, 1553, 1451, 1026, 741, 694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{16}NO$ $(M+H)^+$ 250.1226, found 250.1209.



2s

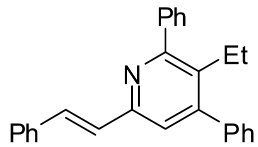
2,3-Dimethyl-6-phenyl-4-(thiophen-2-yl)pyridine (2s), light yellow solid, 0.039 g, 74% yield. Mp: 62−63 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03−8.00 (m, 2H), 7.59 (s, 1H), 7.49−7.37 (m, 4H), 7.16−7.14 (m, 2H), 2.69 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (100

MHz, $CDCl_3$): δ 158.1, 153.8, 142.5, 141.0, 139.4, 128.6, 128.5, 127.7, 127.6, 127.3, 126.8, 126.3, 119.7, 23.9, 16.3; IR (thin film) 2925, 1640, 1459, 1379, 1165, 974, 1048, 880, 692 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{16}NS$ $(M+H)^+$ 266.0998, found 266.0980.



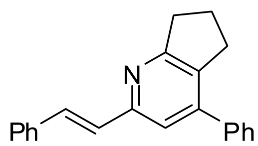
2ac

(*E*)-2,3-Diethyl-4-phenyl-6-styrylpyridine (2ac), yellow oil, 0.037 g, 59% yield. ^1H NMR (400 MHz, $CDCl_3$): δ 7.71−7.69 (m, 3H), 7.58−7.53 (m, 3H), 7.50−7.32 (m, 7H), 3.11 (q, *J* = 14.4 Hz, 2H), 2.78 (q, *J* = 14.4 Hz, 2H), 1.58 (t, *J* = 7.2 Hz, 3H), 1.18 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.5, 152.0, 150.4, 140.4, 137.0, 133.1, 131.7, 128.6, 128.5, 128.4, 128.2, 127.9, 127.5, 126.9, 120.5, 28.2, 21.7, 15.2, 14.1; IR (thin film) 3028, 2964, 2871, 1581, 1495, 1449, 1383, 969, 756, 699 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{23}H_{24}N$ $(M+H)^+$ 314.1903, found 314.1881.



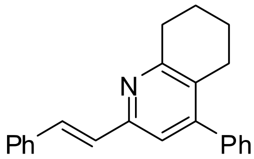
2ad

(*E*)-3-Ethyl-2,4-diphenyl-6-styrylpyridine (2ad), yellow oil, 0.033 g, 45% yield. ^1H NMR (400 MHz, $CDCl_3$): δ 7.47−7.44 (m, 5H), 7.39−7.28 (m, 8H), 7.24−7.13 (m, 5H), 2.57 (q, *J* = 14.0 Hz, 2H), 0.67 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.7, 152.3, 151.0, 141.5, 140.1, 136.8, 133.6, 132.3, 128.8, 128.6, 128.5, 128.3, 128.3, 128.1, 128.0, 127.7, 127.7, 127.0, 121.5, 22.3, 14.8; IR (thin film) 3058, 2966, 2873, 1688, 1579, 1449, 1212, 970, 754, 693 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{27}H_{24}N$ $(M+H)^+$ 362.1903, found 362.1884.



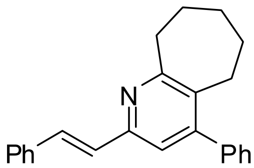
2ae

(*E*)-4-Phenyl-2-styryl-6,7-dihydro-5H-cyclopenta[b]pyridine (2ae)[2], light yellow solid, 0.027 g, 46% yield. Mp: 98−99 °C; ^{1}H NMR (600 MHz, $CDCl_3$): *δ* 7.54 (d, *J* = 16.2 Hz, 1H), 7.49 (d, *J* = 7.2 Hz, 2H), 7.41−7.36 (m, 4H), 7.32−7.30 (m, 1H), 7.27−7.25 (m, 2H), 7.18−7.12 (m, 3H), 3.03 (d, *J* = 7.8 Hz, 2H), 2.93 (d, *J* = 7.2 Hz, 2H), 2.05−2.00 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): *δ* 166.5, 154.3, 145.7, 138.7, 136.9, 133.3, 131.5, 128.9, 128.6, 128.5, 128.2, 128.1, 127.9, 126.9, 119.4, 34.6, 30.7, 23.5.



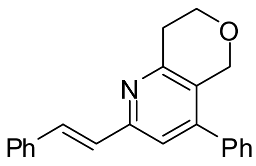
2af

(*E*)-4-Phenyl-2-styryl-5,6,7,8-tetrahydroquinoline (2af)[1], light yellow solid, 0.032 g, 52% yield. ^{1}H NMR (500 MHz, $CDCl_3$): *δ* 7.57 (d, *J* = 7.5 Hz, 2H), 7.52 (d, *J* = 16.0 Hz, 1H), 7.46−7.38 (m, 3H), 7.37−7.32 (m, 4H), 7.28−7.25 (m, 1H) , 7.21 (d, *J* = 17.0 Hz, 2H), 3.05 (t, *J* = 6.5 Hz, 2H), 2.64 (t, *J* = 6.0 Hz, 2H), 1.93−1.91 (m, 2H), 1.75−1.73 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): *δ* 157.5, 152.5, 150.0, 139.5, 136.9, 131.7, 128.7, 128.6, 128.5, 128.3, 128.0, 127.7, 126.9, 119.8, 33.2, 27.4, 23.0, 23.0.



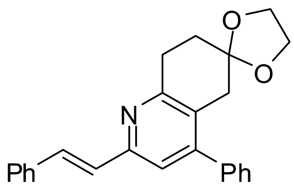
2ag

(*E*)-4-Phenyl-2-styryl-6,7,8,9-tetrahydro-5H-cyclohepta[b]pyridine (2ag), light yellow solid, 0.036 g, 52% yield; Mp: 109−110 °C; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.47−7.44 (m, 3H), 7.35−7.33 (m, 2H), 7.30−7.28 (m, 1H), 7.26−7.23 (m, 2H), 7.20−7.19 (m, 2H), 7.17−7.14 (m, 1H) , 7.10 (d, *J* = 15.6 Hz, 1H), 7.05(s, 1H), 3.08 (t, *J* = 11.4 Hz, 2H), 2.64 (t, *J* = 10.8 Hz, 2H), 1.79−1.76 (m, 2H), 1.72−1.69 (m, 2H), 1.54−1.52 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 164.0, 151.5, 149.3, 140.1, 136.9, 134.2, 131.6, 128.7, 128.6, 128.3, 128.2, 127.9, 127.5, 126.9, 120.5, 39.5, 32.2, 29.5, 27.9, 26.6; IR (thin film) 2922, 2851, 1638, 1583, 1444, 1383, 971, 757, 706, 689 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{24}H_{24}N$ $(M+H)^+$ 326.1903, found 326.1881.



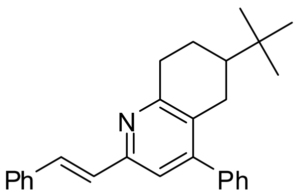
2ah

(*E*)-4-Phenyl-2-styryl-7,8-dihydro-5H-pyrano[4,3-b]pyridine (2ah), light yellow solid, 0.024 g, 38% yield. Mp: 108−109 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51−7.47 (m, 3H), 7.40−7.34 (m, 3H), 7.30−7.27 (m, 2H), 7.22−7.21 (m, 3H), 7.15−7.11 (m, 2H), 4.59 (s, 2H), 4.03 (t, J = 5.6 Hz, 2H), 3.07 (t, J = 5.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 153.8, 153.7, 147.5, 137.7, 136.7, 132.6, 128.7, 128.6, 128.4, 128.3, 128.1, 127.9, 127.0, 126.6, 120.2, 66.5, 65.7, 32.2; IR (thin film) 3049, 2923, 2847, 1637, 1581, 1388, 1098, 968, 760, 695 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{20}NO$ $(M+H)^+$ 314.1539, found 314.1528.



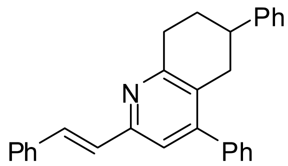
2ai

(*E*)-4'-Phenyl-2'-styryl-7',8'-dihydro-5'H-spiro[[1,3]dioxolane-2,6'-quinoline] (2ai), yellow oil, 0.042 g, 56% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (d, J = 7.6 Hz, 2H), 7.45 (d, J = 12.4 Hz, 1H), 7.39−7.32 (m, 3H), 7.29−7.17 (m, 5H), 7.13−7.09 (m, 2H), 3.92−3.87 (m, 2H), 3.85−3.82 (m, 2H), 3.18 (t, J = 6.8 Hz, 2H), 2.79 (s, 2H), 2.03 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.0, 153.2, 150.3, 139.0, 136.8, 132.2, 128.6, 128.4, 128.3, 128.1, 127.9, 127.0, 125.7, 120.0, 107.9, 64.5, 38.1, 37.2, 33.8, 31.5, 29.7; IR (thin film) 3057, 2952, 2883, 1735, 1582, 1450, 1098, 971, 761,701 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{25}H_{24}NO_2$ $(M+H)^+$ 370.1802, found 370.1786.



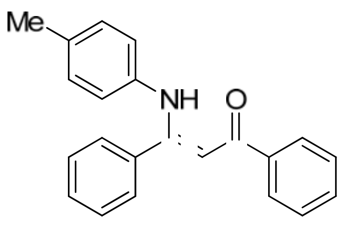
2aj

(*E*)-6-tert-Butyl-4-phenyl-2-styryl-5,6,7,8-tetrahydroquinoline (2aj), light yellow solid, 0.043 g, 59% yield. Mp: 129−130 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46−7.43 (m, 2H), 7.39−7.36 (m, 1H), 7.35−7.27 (m, 3H), 7.24−7.20 (m, 4H), 7.16−7.13 (m, 1H), 7.11 (d, J = 16.0 Hz, 1H), 7.06 (s, 1H), 3.10−3.05 (m, 1H), 2.92−2.88 (m, 1H), 2.59 (d, J = 16.4 Hz, 1H), 2.32−2.25 (m, 1H), 2.01−1.98 (m, 1H), 1.41−1.28 (m, 2H), 0.77 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.5, 152.4, 150.2, 139.5, 136.9, 131.6, 128.7, 128.6, 128.5, 128.4, 128.3, 127.9, 127.7, 126.9, 119.9, 44.6, 41.2, 34.1, 32.4, 28.6, 27.5, 27.2, 24.3; IR (thin film) 3028, 2959, 2868, 1634, 1580, 1537, 1446, 972, 759, 696 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{27}H_{30}N$ $(M+H)^+$ 368.2373, found 368.2359.



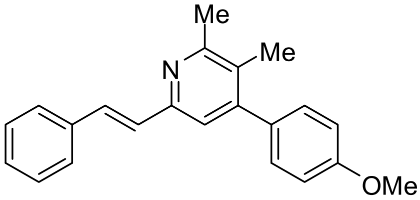
2ak

(*E*)-4,6-Diphenyl-2-styryl-5,6,7,8-tetrahydroquinoline (2ak), light yellow solid, 0.044 g, 57% yield. Mp: 153−154 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51−7.44 (m, 3H), 7.35−7.27 (m, 5H), 7.24−7.19 (m, 5H), 7.17−7.13 (m, 5H), 3.21−3.07 (m, 2H), 2.88−2.68 (m, 3H), 2.16−2.00 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.8, 152.9, 150.2, 145.9, 139.3, 136.9, 132.1, 128.7, 128.5, 128.4, 128.4, 128.2, 128.1, 128.0, 127.8, 127.0, 126.8, 126.3, 120.1, 40.7, 35.6, 33.5, 30.1; IR (thin film) 3026, 2925, 2860, 1631, 1579, 1536, 1444, 967, 758, 700 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{29}H_{26}N$ $(M+H)^+$ 388.2060, found 388.2044.



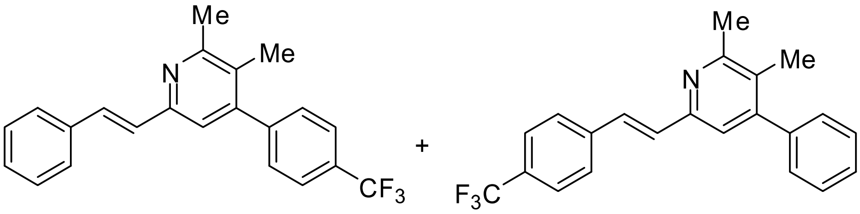
2am

(Z)-1,3-Diphenyl-3-(*p*-tolylamino)prop-2-en-1-one (6)[3], light yellow solid, 0.016 g, 26% yield. Mp: 127−128 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 12.82 (s, 1H), 7.89 (d, J = 6.8 Hz, 2H), 7.39−7.23 (m, 7H), 6.85 (d, J = 8.0 Hz, 2H), 6.62 (d, J = 8.4 Hz, 2H), 5.98 (s, 1H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 189.4, 161.7, 139.9, 136.8, 135.9, 133.8, 131.1, 129.5, 129.3, 128.5, 128.4, 128.3, 127.2, 123.2, 96.5, 20.7;



2v

(*E*)-6-(4-Methoxystyryl)-2,3-dimethyl-4-phenylpyridine (2v), light yellow solid, 0.029 g, 45% yield. Mp: 92−93 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57−7.53 (m, 3H), 7.36−7.34 (m, 2H), 7.27−7.25 (m, 3H), 7.18−7.16 (m, 2H), 6.99−6.98 (m, 2H), 3.86 (s, 3H), 2.61 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.2, 157.6, 151.9, 149.6, 137.0, 132.3, 131.4, 129.9, 128.6, 128.4, 127.9, 127.7, 126.9, 120.6, 113.7, 55.3, 23.6, 16.2; IR (thin film) 2925, 1605, 1510, 1451, 1248, 1174, 1032, 971, 831, 694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{22}NO$ $(M+H)^+$ 316.1696, found 316.1681.

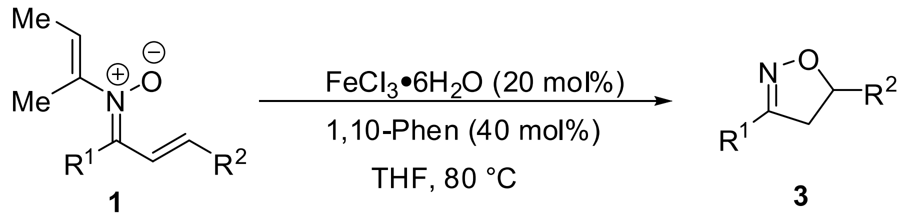


2w (*a/b* = 1:3)

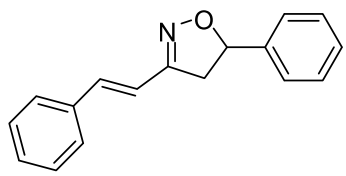
Pyridine (2w, *a/b* = 1:3**)**, light yellow oil, 0.037 g, 52% yield; *major isomer*: ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.60−7.56 (m, 5H), 7.47−7.43 (m, 3H), 7.32−7.31 (m, 2H), 7.25−7.23 (m, 1H), 7.18−7.16 (m, 1H), 2.63 (s, 3H), 2.20 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.9, 152.1, 148.5, 143.7, 139.8, 132.0, 130.6 (q, *J* = 258.3 Hz), 129.6, 129.1, 128.7, 128.3, 127.9, 126.9, 125.6, 125.3, 120.0, 23.6, 16.1; *minor isomer*: ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.74−7.63 (m, 5H), 7.42−7.40 (m, 3H), 7. 37−7.34 (m, 2H), 7.28−7.27 (m, 1H), 7.15−7.13 (m, 1H), 2.63 (s, 3H), 2.17 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 157.8, 151.1, 150.1, 143.7, 139.8, 132.0 129.9, 129.4, 129.0 (q, *J* = 263.4 Hz), 128.4, 128.1, 127.8, 126.9, 125.5, 125.3, 121.0, 23.6, 16.2; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.5; IR (thin film) 3051, 2926, 2859, 1613, 1584, 1453, 1324, 1122, 969, 699 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{19}F_3N$ $(M+H)^+$

354.1464, found 354.1458.

4. Synthesis of 3,5-disubstituted isoxazolines 3

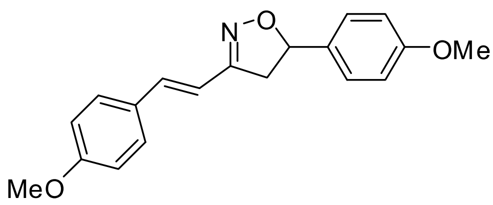


General procedure B: A Teflon-sealed flask was charged with *N*-vinyl nitrone **1** (0.2 mmol), $FeCl_3$•$6H_2O$ (11 mg, 20 mol%), 1,10-Phen (15 mg, 40 mol%) under N_2 atmosphere. THF (2 mL) was then added via syringe and the reaction vessel was once again sealed with a Teflon cap. The reaction mixture was stirred at 25 °C for 5 min and then heated at 80 °C for 7–15 h until nitrone **1** was consumed completely (monitored by TLC). At this time, the solvent was removed under reduced pressure and the crude product was purified by flash column chromatography (the crude residue was dry loaded on silica gel, 1/50 to 1/20, ethyl acetate/petroleum ether) to afford 3,5-disubstituted isoxazolines **3**.



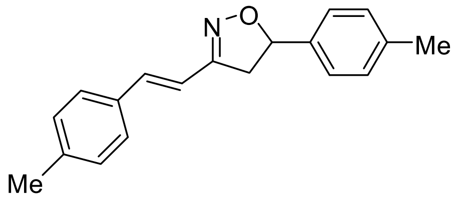
3a

(*E*)-5-Phenyl-3-styryl-4,5-dihydroisoxazole (3a)[4], light yellow solid, 0.040 g, 79% yield. Mp: 83−84 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35−7.33 (m, 2H), 7.26−7.19 (m, 8H), 7.03 (d, J = 16.4 Hz, 1H), 6.63 (d, J = 16.4 Hz, 1H), 5.57 (dd, J = 10.4 Hz, 8.4 Hz, 1H), 3.52 (dd, J = 16.4 Hz, 7.2 Hz, 1H), 3.09 (dd, J = 16.4 Hz, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.2, 140.7, 136.5, 135.6, 128.9, 128.7, 128.6, 128.1, 126.9, 125.7, 117.7, 82.5, 41.5; HRMS (ESI) *m/z* calcd for $C_{17}H_{16}NO$ $(M+H)^+$ 250.1226, found 250.1217.



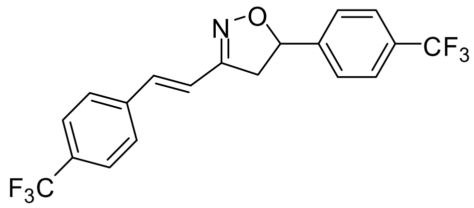
3b

(*E*)-5-(4-Methoxyphenyl)-3-(4-methoxystyryl)-4,5-dihydroisoxazole (3b), light yellow solid, 0.031 g, 51% yield. Mp: 135−136 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, J = 8.8 Hz, 2H), 7.32−7.28 (m, 2H), 7.01 (d, J = 16.4 Hz, 1H), 6.92−6.89 (m, 4H), 6.70 (d, J = 16.4 Hz, 1H), 5.63 (dd, J = 10.8 Hz, 8.8 Hz, 1H), 3.83 (s, 3H), 3.81 (s, 3H); 3.60 (dd, J = 16.4 Hz, 10.8 Hz, 1H), 3.20 (dd, J = 16.4 Hz, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 159.6, 157.6, 136.1, 132.7, 128.5, 128.3, 127.3, 115.8, 114.3, 114.1, 82.4, 55.3, 55.2, 41.5; IR (thin film) 2959, 2840, 1604, 1510, 1463, 1244, 1175, 1027, 908, 817 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{20}NO_3$ $(M+H)^+$ 310.1438, found 310.1423.



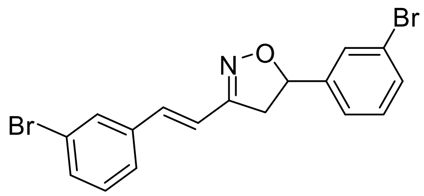
3c

(*E*)-3-(4-Methylstyryl)-5-*p*-tolyl-4,5-dihydroisoxazole (3c), light yellow solid, 0.040 g, 72% yield. Mp: 127−128 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, J = 8.0 Hz, 2H), 7.27 (d, J = 8.0 Hz, 2H), 7.19−7.16 (m, 4H), 7.09 (d, J = 16.4 Hz, 1H), 6.71 (d, J = 16.4 Hz, 1H), 5.66 (dd, J = 10.4 Hz, 8.0 Hz, 1H), 3.62 (dd, J = 16.4 Hz, 10.8 Hz, 1H), 3.20 (dd, J = 16.4 Hz, 8.4 Hz, 1H), 2.36 (s, 3H), 2.35 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.4, 139.1, 138.0, 137.8, 136.5, 133.0, 129.6, 129.4, 126.9, 125.9, 117.0, 82.6, 41.6, 21.3, 21.1; IR (thin film) 3031, 2922, 2857, 1608, 1511, 1451, 1368, 1111, 964, 811 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{20}NO$ $(M+H)^+$ 278.1539, found 278.1518.



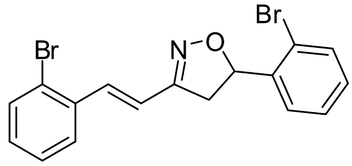
3d

(*E*)-5-(4-(Trifluoromethyl)phenyl)-3-(4-(trifluoromethyl)styryl)-4,5-dihydroisoxazole (3d), light yellow solid, 0.051 g, 66% yield. Mp: 113−114 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.66−7.61 (m, 4H), 7.56 (d, J = 8.0 Hz, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.22 (d, J = 16.4 Hz, 1H), 6.77 (d, J = 16.8 Hz, 1H), 5.80 (dd, J = 11.2 Hz, 7.6 Hz, 1H), 3.74 (dd, J = 16.4 Hz, 11.2 Hz, 1H), 3.21 (dd, J = 16.4 Hz, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7, 144.7, 138.9, 135.2, 131.2 (q, J = 97.7 Hz), 130.8 (q, J = 32.1 Hz), 128.4 (q, J = 37.2 Hz), 127.1, 126.0, 125.9 (q, J = 10.9 Hz), 125.8, 125.3 (q, J = 270.5 Hz), 120.0, 81.9, 41.7; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.6, −62.7; IR (thin film) 3081, 2938, 1618, 1419, 1327, 1126, 1066, 910, 834, 736 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{14}F_6NO$ $(M+H)^+$ 386.0974, found 386.0956.



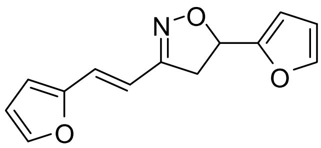
3t

(*E*)-5-(3-Bromophenyl)-3-(3-bromostyryl)-4,5-dihydroisoxazole (3t), light yellow solid, 0.064 g, 76% yield. Mp: 94−95 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.59 (s, 1H), 7.52 (s, 1H), 7.46−7.43 (m, 2H), 7.39 (d, J = 8.0 Hz, 1H), 7.30−7.21 (m, 3H), 7.12 (d, J = 16.4 Hz, 1H), 6.66 (d, J = 16.4 Hz, 1H), 5.68 (dd, J = 11.2 Hz, 8.0 Hz, 1H), 3.66 (dd, J = 16.4 Hz, 11.2 Hz, 1H), 3.18 (dd, J = 16.4 Hz, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.7, 143.0, 137.7, 135.2, 131.9, 131.4, 130.4, 130.3, 129.9, 128.8, 125.4, 124.3, 123.0, 122.9, 119.0, 81.8, 41.6; IR (thin film) 2926, 1650, 1460, 1373, 1167, 973, 901, 786, 693 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{14}Br_2NO$ $(M+H)^+$ 405.9437, found 405.9433.



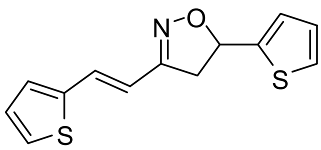
3f

(*E*)-5-(2-Bromophenyl)-3-(2-bromostyryl)-4,5-dihydroisoxazole (3f), light yellow solid, 0.067 g, 80% yield. Mp: 102−103 °C; ^{1}H NMR (600 MHz, $CDCl_3$): *δ* 7.61−7.56 (m, 3H), 7.53 (dd, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.20−7.14 (m, 2H), 7.10 (*J* = 16.8 Hz, 1H), 7.06 (d, *J* = 16.2 Hz, 1H), 5.99 (dd, *J* = 11.4 Hz, 7.2 Hz, 1H), 3.87 (dd, *J* = 16.2 Hz, 10.8 Hz, 1H), 3.11 (dd, *J* = 16.8 Hz, 7.2 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): *δ* 157.0, 140.3, 135.3, 135.2, 133.2, 132.7, 130.1, 129.4, 127.8, 127.7, 126.9, 126.7, 124.3, 120.9, 120.1, 81.8, 41.3; IR (thin film) 3050, 2954, 2863, 1655, 1620, 1465, 1434, 954, 896, 749 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{14}Br_2NO$ $(M+H)^+$ 405.9437, found 405.9424.



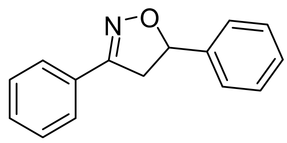
3g

(*E*)-5-(Furan-2-yl)-3-(2-(furan-2-yl)vinyl)-4,5-dihydroisoxazole (3g), light yellow solid, 0.035 g, 76% yield. Mp: 92−93 °C; ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.45−7.42 (m, 2H), 7.01 (d, *J* = 16.4 Hz, 1H), 6.59 (d, *J* = 16.0 Hz, 1H), 6.46−6.43 (m, 2H), 6.41−6.40 (m, 1H), 6.37−6.35 (m, 1H), 5.66−5.61 (m, 1H), 3.42−3.40 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): *δ* 157.0, 151.8, 151.6, 143.6, 143.2, 123.7, 115.9, 111.9, 111.2, 110.5, 108.8, 75.7, 37.3; IR (thin film) 3123, 2924, 2856, 1629, 1546, 1499, 1364, 1016, 906, 751 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{13}H_{12}NO_3$ $(M+H)^+$ 230.0812, found 230.0802.



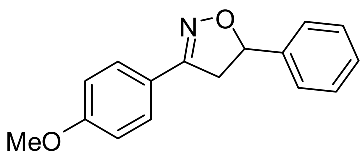
3h

(*E*)-5-(Thiophen-2-yl)-3-(2-(thiophen-2-yl)vinyl)-4,5-dihydroisoxazole (3h), light yellow solid, 0.041 g, 78% yield. Mp: 84−85 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31−7.29 (m, 2H), 7.13−7.12 (m, 1H), 7.10 (d, J = 3.6 Hz, 1H), 7.03−6.98 (m, 2H), 6.95 (d, J = 16.4 Hz, 1H), 6.91 (d, J = 16.0 Hz, 1H) 5.91 (dd, J = 10.8 Hz, 8.4 Hz, 1H), 3.61 (dd, J = 16.4 Hz, 10.8 Hz, 1H), 3.32 (dd, J = 16.0 Hz, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.1, 143.1, 140.9, 129.4, 128.0, 127.9, 126.9, 126.6, 125.8, 125.5, 116.9, 78.4, 41.4; IR (thin film) 2924, 1663, 1589, 1547, 1492, 1451, 1008, 829, 771, 702 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{13}H_{12}NOS_2$ $(M+H)^+$ 262.0355, found 262.0350.



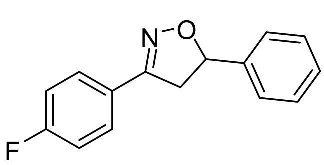
3i

3,5-Diphenyl-4,5-dihydroisoxazole (3i)[4], light yellow solid, 0.044 g, 98% yield. Mp: 62−63 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.75−7.65 (m, 2H), 7.42−7.41 (m, 7H), 7.34−7.33 (m, 1H), 5.77 (dd, J = 10.5 Hz, 8.5 Hz, 1H), 3.82 (dd, J = 16.5 Hz, 11.0 Hz, 1H), 3.38 (dd, J = 16.5 Hz, 8.0 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.1, 140.9, 130.1, 129.4, 128.7, 128.7, 128.2, 126.7, 125.8, 82.5, 43.1.



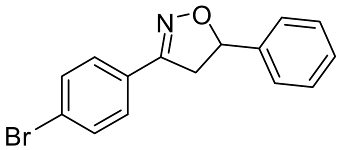
3j

3-(4-Methoxyphenyl)-5-phenyl-4,5-dihydroisoxazole (3j)[5], light yellow solid, 0.041 g, 81% yield. Mp: 93−94 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.65−7.63 (m, 2H), 7.42−7.32 (m, 5H), 6.94−6.91 (m, 2H), 5.73 (dd, J = 10.8 Hz, 8.4 Hz, 1H), 3.84 (s, 3H), 3.79 (dd, J = 10.8 Hz, 8.4 Hz, 1H), 3.35 (dd, J = 16.4 Hz, 8.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.0, 155.6, 141.0, 128.7, 128.2, 128.1, 125.8, 122.0, 114.1, 82.2, 55.3, 43.4.



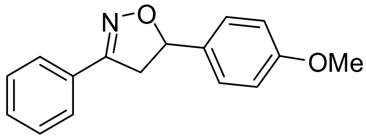
3u

3-(4-Fluorophenyl)-5-phenyl-4,5-dihydroisoxazole (3u)[5], light yellow solid, 0.035 g, 72% yield. Mp: 83−84 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71−7.67 (m, 2H), 7.41−7.33 (m, 5H), 7.12−7.08 (m, 2H), 5.77 (dd, J = 10.8 Hz, 8.4 Hz, 1H), 3.79 (dd, J = 11.2 Hz, 8.8 Hz, 1H), 3.35 (dd, J = 16.4 Hz, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.9 (d, J = 249.3 Hz), 155 (d, J = 15.0 Hz), 140.7, 128.7, 128.7, 128.6, 125.8, 125.7 (d, J = 37.0 Hz), 115.9 (d, J = 21.9 Hz), 82.6, 43.1; ^{19}F NMR (100 MHz, $CDCl_3$): δ −109.8.



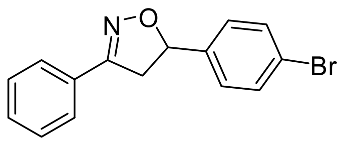
3k

3-(4-Bromophenyl)-5-phenyl-4,5-dihydroisoxazole (3k)[6], light yellow solid, 0.049 g, 82% yield. Mp: 131−132 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57−7.52 (m, 4H), 7.39−7.33 (m, 5H), 5.78 (dd, J = 11.2 Hz, 8.4 Hz, 1H), 3.79 (dd, J = 16.8 Hz, 10.8 Hz, 1H), 3.35 (dd, J = 16.8 Hz, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.3, 140.6, 131.9, 128.8, 128.4, 128.3, 128.1, 125.8, 124.4, 82.8, 42.9.



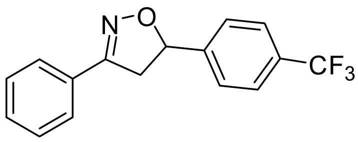
3l

5-(4-Methoxyphenyl)-3-phenyl-4,5-dihydroisoxazole (3l)[5], light yellow solid, 0.034 g, 68% yield. Mp: 96−97 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.71−7.70 (m, 2H), 7.43−7.41 (m, 3H), 7.33 (d, J = 8.4 Hz, 2H), 6.92 (d, J = 9.0 Hz, 2H), 5.71 (dd, J = 11.4 Hz, 9.0 Hz, 1H), 3.81 (s, 3H), 3.75 (dd, J = 16.8 Hz, 10.8 Hz, 1H), 3.35 (dd, J = 16.2 Hz, 8.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 159.6, 156.2, 132.7, 130.0, 129.5, 128.7, 127.3, 126.7, 114.1, 82.5, 55.3, 42.8.



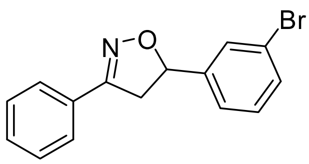
3m

5-(4-Bromophenyl)-3-phenyl-4,5-dihydroisoxazole (3m)[6], light yellow solid, 0.045 g, 75% yield. Mp: 114−115 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68−7.66 (m, 2H), 7.50 (d, J = 8.4 Hz, 2H), 7.44−7.40 (m, 3H), 7.27 (d, J = 8.4 Hz, 2H), 5.71 (dd, J = 10.8 Hz, 8.0 Hz, 1H), 3.81 (dd, J = 16.4 Hz, 10.8 Hz, 1H), 3.31 (dd, J = 16.4 Hz, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.0, 140.0, 131.8, 130.2, 129.2, 128.7, 127.5, 126.7, 122.1, 81.7, 43.1.



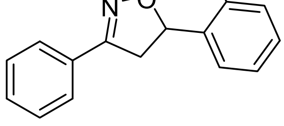
3n

3-Phenyl-5-(4-(trifluoromethyl)phenyl)-4,5-dihydroisoxazole (3n)[6], light yellow solid, 0.048 g, 83% yield. Mp: 137−138 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.70−7.68 (m, 2H), 7.65−7.63 (m, 2H), 7.53−7.51 (m, 2H), 7.44−7.40 (m, 3H), 5.83 (dd, J = 11.2 Hz, 8.0 Hz, 1H), 3.88 (dd, J = 16.8 Hz, 11.2 Hz, 1H), 3.35 (dd, J = 16.4 Hz, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.0, 145.0, 130.8 (q, J = 32.1 Hz), 130.3, 129.0, 128.8, 126.8, 126.1, 125.8 (q, J = 6.5 Hz), 125.3 (q, J = 270.5 Hz), 81.6, 43.3; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.6.



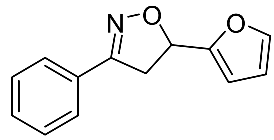
3p

5-(3-Bromophenyl)-3-phenyl-4,5-dihydroisoxazole (3p)[7], light yellow solid, 0.047 g, 78% yield. Mp: 83−84 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62−7.60 (m, 2H), 7.47 (s, 1H), 7.38−7.33 (m, 4H), 7.25 (d, J = 7.6 Hz, 1H), 7.19−7.15 (m, 1H), 5.65 (dd, J = 10.4 Hz, 8.0 Hz, 1H), 3.76 (dd, J = 16.8 Hz, 11.2 Hz, 1H), 3.27 (dd, J = 16.4 Hz, 7.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.0, 143.3, 131.3, 130.4, 130.3, 129.2, 128.9, 128.8, 126.8, 124.4, 122.8, 81.6, 43.3.



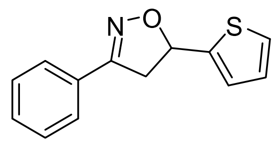
3q

5-(2-Bromophenyl)-3-phenyl-4,5-dihydroisoxazole (3q)[6], light yellow solid, 0.052 g, 86% yield. Mp: 69−70 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.69−7.68 (m, 2H), 7.58−7.57 (m, 2H), 7.41−7.40 (m, 3H), 7.35−7.32 (m, 1H), 7.19−7.16 (m, 1H), 6.01 (dd, J = 11.2 Hz, 6.8 Hz, 1H), 3.99 (dd, J = 16.8 Hz, 11.2 Hz, 1H), 3.22 (dd, J = 16.8 Hz, 6.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.0, 140.6, 132.7, 130.2, 129.3, 129.2, 128.7, 127.8, 126.8, 126.7, 120.8, 81.4, 42.9.



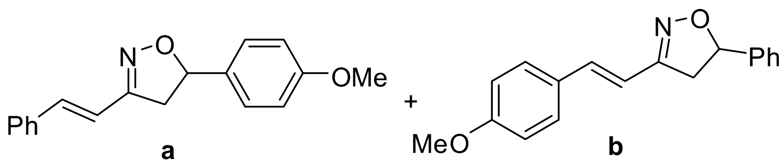
3r

5-(Furan-2-yl)-3-phenyl-4,5-dihydroisoxazole (3r)[7], light yellow solid, 0.035 g, 82% yield. Mp: 70−71 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72−7.70 (m, 2H), 7.46−7.36 (m, 4H), 6.43−6.37 (m, 2H), 5.77−5.69 (m, 1H), 3.63−3.61 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.2, 151.7, 143.1, 130.1, 129.3, 128.7, 126.7, 110.5, 108.7, 75.6, 38.8.



3s

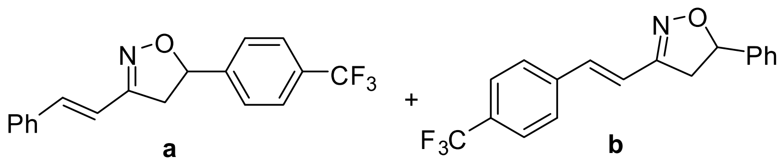
3-Phenyl-5-(thiophen-2-yl)-4,5-dihydroisoxazole (3s)[7], light yellow solid, 0.039 g, 86% yield. Mp: 56−57 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73−7.70 (m, 2H), 7.43−7.42 (m, 3H), 7.31−7.29 (m, 1H), 7.12−7.11 (m, 1H), 7.01−6.99 (m, 1H), 5.98 (dd, J = 10.8 Hz, 8.0 Hz, 1H), 3.79 (dd, J = 15.6 Hz, 10.4 Hz, 1H), 3.51 (dd, J = 17.2 Hz, 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 143.3, 130.2, 129.2, 128.7, 126.9, 126.7, 125.7, 125.4, 78.3, 42.9.



3v (*a/b* = 1:1)

Isoxazoline (3v, *a/b* = 1:1**)**, light yellow solid, 0.039 g, 69% yield. Mp: 110−111 °C;

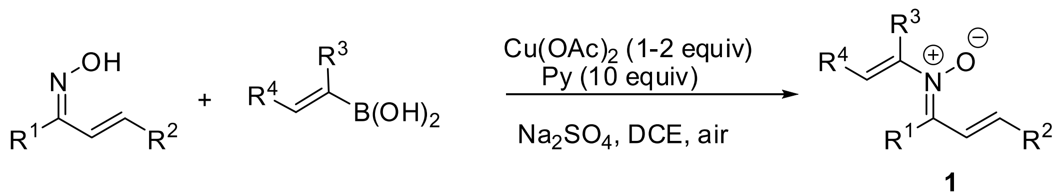
one isomer: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (s, 1H), 7.42−7.37 (m, 6H), 7.16 (d, J = 16.4 Hz, 1H), 6.93−6.91 (m, 2H), 6.76 (d, J = 16.4 Hz, 1H), 5.64−5.61 (m, 1H), 3.81 (s, 3H), 3.59−3.55 (m, 1H), 3.22−3.20 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3, 157.4, 136.4, 135.7, 128.8, 128.7, 128.3, 127.3, 125.8, 117.9, 114.1, 82.5, 55.3, 41.3; *another isomer*: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (s, 1H), 7.35−7.29 (m, 6H), 7.02 (d, J = 16.4 Hz, 1H), 6.90−6.89 (m, 2H), 6.71 (d, J = 16.4 Hz, 1H), 5.69−5.66 (m, 1H), 3.83 (s, 3H), 3.65−3.61 (m, 1H), 3.18−3.15 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.6, 140.9, 136.2, 132.6, 128.9, 128.5, 128.1, 126.9, 125.8, 115.6, 114.3, 82.4, 55.3, 41.7; IR (thin film) 2925, 1649, 1604, 1512, 1456, 1251, 1175, 911, 824, 755 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{18}H_{18}NO_2$ $(M+H)^+$ 280.1338, found 280.1330.



3w (*a/b* = 1:1)

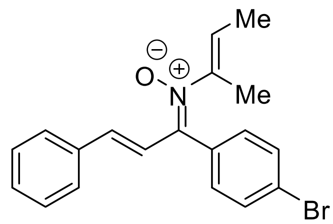
Isoxazoline (3w, *a/b* = 1:1**)**, light yellow solid, 0.038 g, 60% yield. Mp: 114−115 °C; *one isomer*: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.64−7.60 (m, 4H), 7.37−7.37 (m, 2H), 7.34−7.31 (m, 3H), 7.22 (d, J = 16.4 Hz, 1H), 6.71(s, 1H), 5.76−5.73 (m, 1H), 3.66−3.60 (m, 1H), 3.23−3.19 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.1, 144.9, 139.1, 139.1, 134.7, 130.6 (q, J = 32.1 Hz), 129.1, 128.9, 126.9, 126.0, 125.7, 125.3 (q, J = 269.8 Hz), 120.4, 82.9, 41.9; *another isomer:* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55−7.50 (m, 4H), 7.48−7.44 (m, 3H), 7.37−7.37 (m, 2H), 7.13 (d, J = 16.4 Hz, 1H), 6.75 (s, 1H), 5.71−5.69 (m, 1H), 3.72−3.67 (m, 1H), 3.18−3.14 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 140.5, 137.1, 135.5, 130.6 (q, J = 32.1 Hz), 128.8, 128.3, 127.0, 125.8, 125.7, 125.3 (q, J = 269.8 Hz), 117.4, 81.6, 41.4; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.5, −62.6; IR (thin film) 2926, 1615, 1421, 1330, 1109, 1070, 962, 902, 696 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{18}H_{15}F_3NO$ $(M+H)^+$ 318.1106, found 318.1095.

5. **Synthesis of *N*-vinyl nitrones 1.**



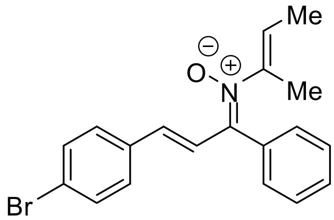
General procedure C: A scintillation vial was charged with oxime (0. 3 mmol, 1.0 equiv), alkenyl boronic acid (0.9 mmol, 3 equiv), $Cu(OAc)_2$ (2 equiv), and anhydrous Na_2SO_4 (8.0 equiv). These solids were diluted with DCE to form a 0.1 M solution of oxime. Pyridine (10.0 equiv) was added to the resulting slurry via syringe. The scintillation vial was then capped with a septum pierced with a ventilation needle and the reaction mixture was stirred at 25 °C for 12 h. DCE and pyridine were removed under reduced pressure and the crude reaction mixture was purified by medium pressure chromatography (2:1; ethyl acetate:hexanes) to give nitrone **1**.

N-vinyl nitrones **1a-j**, **1l**, **1n**, **1o**, **1t**, **1ab-ag**, **1ak** were prepared according to literature methods,[8] and their spectral data matched literature values.



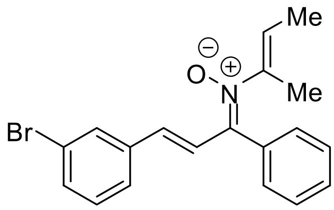
1k (*E/Z* = 8:1)

(2*E*,*NE*)-*N*-((*E*)-1-(4-Bromophenyl)-3-phenylallylidene)but-2-en-2-amine oxide (1k, ***E/Z*** **= 8:1)**, yellow solid, 0.049 g, 46% yield. Mp: 119−120 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, J = 16.4 Hz, 1H), 7.58–7.56 (m, 2H), 7.48–7.46 (m, 2H), 7.32–7.28 (m, 2H), 7.14–7.12 (m, 2H), 6.54 (d, J = 16.4 Hz, 1H), 5.44–5.42 (m, 1H), 1.92 (s, 3H), 1.47 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 147.7, 141.8, 138.4, 135.1, 133.0, 131.6, 129.5, 128.9, 128.6, 128.3, 123.5, 122.8, 122.2, 14.9, 12.2; IR (thin film) 3059 , 2925, 2857, 1664, 1486, 1221, 1071, 970, 813, 699 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{19}BrNO$ $(M+H)^+$ 356.0645, found 356.0644.



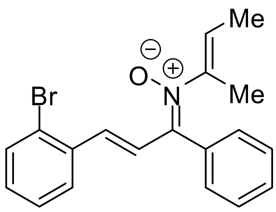
1m (*E/Z* = 8:1)

(2*E*,*NE*)-*N*-((*E*)-3-(4-Bromophenyl)-1-phenylallylidene)but-2-en-2-amine oxide (1m, *E/Z* = 8:1**)**, yellow liquid. 0.064 g, 61% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 16.0 Hz, 1H), 7.40–7.33 (m, 4H), 7.23–7.18 (m, 4H), 6.41 (d, J = 16.4 Hz, 1H), 5.34–5.34 (m, 1H), 1.83 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 147.7, 141.8, 138.4, 135.1, 132.9, 131.6, 129.5, 128.9, 128.7, 128.3, 123.6, 122.8, 122.2, 55.0, 14.9, 12.2; IR (thin film) 3039, 2923, 1664, 1551, 1466, 1223, 1068, 967, 773 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{19}BrNO$ $(M+H)^+$ 356.0645, found 356.0639.



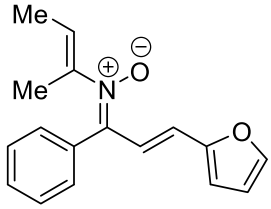
1p (*E/Z* = 8:1)

(2*E*,*NE*)-*N*-((*E*)-3-(3-Bromophenyl)-1-phenylallylidene)but-2-en-2-amine oxide (1p, *E/Z* = 8:1**)**, yellow solid, 0.056 g, 53% yield. Mp: 123−124 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 17.2 Hz, 1H), 7.50–7.44 (m, 1H), 7.42–7.22 (m, 5H), 7.14–7.02 (m, 2H), 6.40 (d, J = 16.8 Hz, 1H), 5.33–5.30 (m, 1H), 1.83 (s, 3H), 1.34 (d, J = 6.0 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 147.6, 141.8, 138.3, 138.1, 132.9, 131.6, 130.2, 130.0, 129.5, 129.0, 128.4, 125.6, 123.7, 122.9, 122.7, 15.0, 12.2; IR (thin film) 3039, 2923, 1664, 1551, 1466, 1223, 1068, 967, 773 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{19}BrNO$ $(M+H)^+$ 356.0645, found 356.0639.



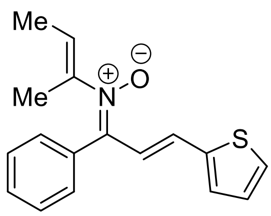
1q (*E*/*Z* = 8:1)

(2*E*,*NE*)-*N*-((*E*)-3-(2-Bromophenyl)-1-Phenylallylidene)but-2-en-2-amine oxide (1q, *E*/*Z* = 8:1**)**, yellow liquid. 0.053 g, 50% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 16.4 Hz, 1H), 7.73–7.71 (m, 1H), 7.47–7.32 (m, 4H), 7.19–7.18 (m, 2H), 7.03–7.01 (m, 1H), 6.90 (d, J = 16.4 Hz, 1H), 5.37–5.36 (m, 1H), 1.84 (s, 3H), 1.35 (d, J = 6.0 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 147.9, 141.9, 138.5, 135.9, 132.9, 132.8, 129.9, 129.5, 128.9, 128.3, 127.5, 127.2, 124.6, 123.9, 123.7, 14.9, 12.2; IR (thin film) 3059, 2923, 2858, 1665, 1502, 1462, 1228, 1022, 970, 754 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{19}BrNO$ $(M+H)^+$ 356.0645, found 356.0643.



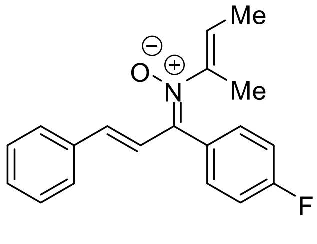
1r (*E*/*Z* = 5:1)

(2*E*,*NZ*)-*N*-((*E*)-3-(Furan-2-yl)-1-phenylallylidene)but-2-en-2-amine oxide (1r, *E*/*Z* = 5:1**)**, light yellow solid, 0.043 g, 54% yield. Mp: 120−121 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (d, J = 16.0 Hz, 1H), 7.42−7.35 (m, 4H), 7.17−7.16 (m, 2H), 6.36−6.35 (m, 3H), 5.36−5.34 (m, 1H), 1.85 (s, 3H), 1.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.7, 147.6, 143.7, 141.8, 133.2, 129.5, 128.8, 128.3, 126.3, 123.5, 119.8, 112.1, 111.9, 14.9, 12.2; IR (thin film) 2922, 2852, 1652, 1587, 1445, 1264, 1230, 841, 698 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{17}H_{18}NO_2$ $(M+H)^+$ 268.1332, found 268.1329.



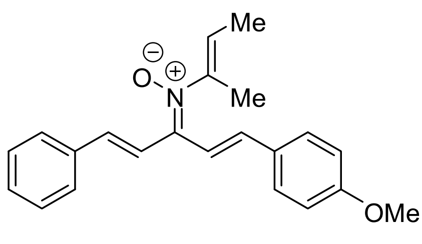
1s (*E*/*Z* = 7:1)

(2*E*,*NZ*)-*N*-((*E*)-1-Phenyl-3-(thiophen-2-yl)allylidene)but-2-en-2-amine oxide (1s, *E*/*Z* = 7:1**)**, light yellow solid, 0.047 g, 55% yield. Mp: 118−119 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74 (d, J = 16.0 Hz, 1H), 7.40−7.33 (m, 3H), 7.19−7.14 (m, 3H), 6.93−6.87 (m, 2H), 6.62 (d, J = 16.0 Hz, 1H), 5.32 (s, 1H), 1.82 (s, 3H), 1.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.6, 142.0, 141.7, 133.0, 132.5, 129.5, 128.9, 128.6, 128.4, 127.7, 127.1, 123.5, 120.9, 14.9, 12.2; IR (thin film) 2929, 2852, 1638, 1502, 1383, 1230, 1072, 698 cm^{-1} HRMS (ESI) *m/z* calcd for $C_{17}H_{18}NOS$ $(M+H)^+$ 284.1104, found 284.1100.



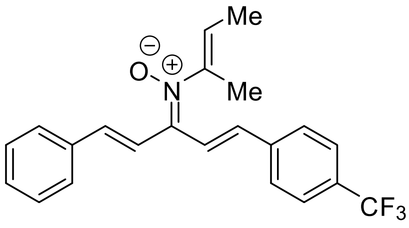
1u (*E/Z* = 8:1**)**

(2*E*,*NE*)-*N*-((*E*)-1-(4-Fluorophenyl)-3-phenylallylidene)but-2-en-2-amine oxide (1u, *E/Z* = 8:1**)**, yellow solid, 0.034 g, 38% yield. Mp: 120−121 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, J = 16.4 Hz, 1H), 7.36–7.35 (m, 2H), 7.18–7.13 (m, 4H), 7.03–7.01 (m, 2H), 6.54 (d, J = 16.4 Hz, 1H), 5.38–5.28 (m, 1H), 1.82 (d, J = 3.6 Hz, 3H), 1.34 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100MHz, $CDCl_3$): δ 163.8 (d, J = 248.6 Hz), 141.7, 140.0, 135.9, 131.5, 131.4, 129.0, 128.5, 127.3, 123.6, 121.5, 115.6, 115.4, 14.9, 12.1; ^{19}F NMR (100 MHz, $CDCl_3$): δ −111.2; IR (thin film) 3057, 2921, 2857, 1601, 1513, 1226, 1157, 975, 837, 761 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{19}H_{19}FNO$ $(M+H)^+$ 296.1445, found 296.1445.



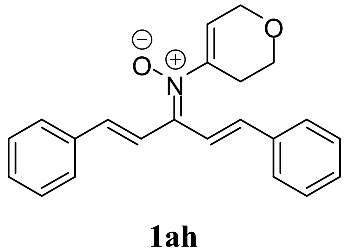
1v (*E/Z* = 1:1)

(2*E*,*NE*)-*N*-((1*E*,4*E*)-1-(4-Methoxyphenyl)-5-phenylpenta-1,4-dien-3-ylidene)but-2-en-2-amine oxide (1v, *E/Z* = 1:1**)**, yellow liquid, 0.050 g, 50% yield. *one isomer*: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, J = 7.2 Hz, 1H), 7.54−7.31 (m, 8H), 6.93−6.89 (m, 3H), 6.82 (d, J = 16.0 Hz, 1H), 5.70−5.67 (m, 1H), 3.82 (s, 3H), 2.11 (s, 3H), 1.79 (d, J = 14.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.4, 144.9, 141.7, 139.2, 136.6, 134.1, 129.3, 128.9, 128.8, 128.6, 127.3, 123.1, 120.4, 118.1, 114.3, 55.3, 14.4, 12.6; *another iosmer*: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, J = 7.2 Hz, 1H), 7.54−7.31 (m, 8H), 6.93−6.89 (m, 4H), 5.70−5.67 (m, 1H), 3.82 (s, 3H), 2.11 (s, 3H), 1.79 (d, J = 14.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.2, 144.9, 141.7, 139.0, 136.3, 133.9, 128.9, 128.9, 128.8, 128.1, 126.7, 123.0, 119.2, 116.8, 114.1, 55.2, 14.4, 12.6; IR (thin film) 2932, 2839, 1681, 1512, 1451, 1252, 1175, 966, 731 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{24}NO_2$ $(M+H)^+$ 334.1802, found 334.1795.



1w (*E/Z* = 1:1)

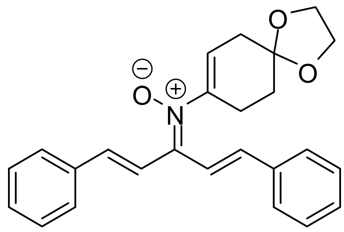
(2*E*,*NE*)-*N*-((1*E*,4*E*)-1-Phenyl-5-(4-(trifluoromethyl)phenyl)penta-1,4-dien-3-ylidene)but-2-en-2-amine oxide (1w, *E*/*Z* = 1:1**)**, yellow liquid. 0.083 g, 74% yield. *one iosmer*: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68−7.34 (m, 11H), 7.07 (d, J = 8.0 Hz, 1H), 6.99 (d, J = 14.8 Hz, 1H), 5.72−5.69 (m, 1H), 2.12 (s, 3H), 1.81 (d, J = 2.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.1, 141.9, 140.1, 139.3, 136.5, 134.0, 130.6 (q, J = 16.8 Hz), 129.1, 128.8, 127.4, 126.9, 125.9 (q, J = 36.0 Hz), 125.3, 123.5, 122.7, 122.7 (q, J = 260.3 Hz), 14.5, 12.8; *another iosme*r: ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.68−7.34 (m, 11H), 6.99 (d, J = 14.8 Hz, 2H), 5.72−5.69 (m, 1H), 2.12 (s, 3H), 1.81 (d, J = 2.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 141.8, 139.8, 137.2, 136.2, 131.9, 130.3 (q, J = 16.8 Hz), 128.9, 128.7, 127.4, 126.8, 125.7 (q, J = 37.0 Hz), 125.3, 123.4, 122.6, 121.4 (q, J = 263.9 Hz), 14.5, 12.8; ^{19}F NMR (100 MHz, $CDCl_3$): δ −62.6; IR (thin film) 2926, 2723, 1633, 1453, 1377, 1325, 1165, 972, 694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{22}H_{21}F_3NO$ $(M+H)^+$ 372.1570, found 372.1566.



1ah

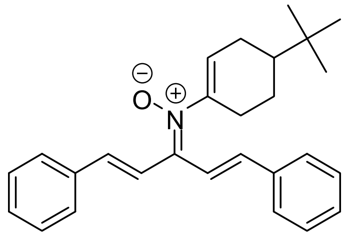
***N*-((1*E*,4*E*)-1,5-Diphenylpenta-1,4-dien-3-ylidene)-3,6-dihydro-2H-pyran-4-amin**

e oxide (1ah), light yellow solid, 0.050 g, 50% yield. Mp: 124−125 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50−7.48 (m, 2H), 7.45 (d, J = 11.2 Hz, 1H), 7.38−7.36 (m, 2H), 7.31−7.24 (m, 6H), 6.91−6.90 (m, 2H), 5.88−5.82 (m, 1H), 4.24−4.18 (m, 2H), 3.90−3.82 (m, 2H), 2.60−2.54 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 141.0, 139.8, 136.3, 135.9, 134.6, 129.1, 128.9, 128.7, 128.2, 127.3, 126.8, 123.6, 119.8, 118.9, 64.1, 63.9, 26.3; IR (thin film) 3054, 2926, 2851, 1609, 1574, 1443, 1223, 1187, 965, 759 cm^{-1}; HRMS (ESI) m/z calcd for $C_{22}H_{22}NO_2$ $(M+H)^+$ 332.1645, found 332.1641.



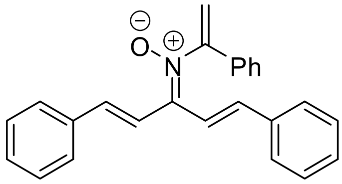
1ai

***N*-((1*E*,4*E*)-1,5-Diphenylpenta-1,4-dien-3-ylidene)-1,4-dioxaspiro[4.5]dec-7-en-8-amine oxide (1ai)**, light yellow solid, 0.074 g, 64% yield. Mp: 124−125 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54−7.38 (m, 5H), 7.30−7.22 (m, 6H), 7.07 (d, J = 16.0 Hz, 1H), 6.88 (d, J = 16.4 Hz, 1H), 5.72−5.68 (m, 1H), 3.98−3.92 (m, 4H), 2.66−2.60 (m, 2H), 2.38−2.32 (m, 2H), 1.90−1.88 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.4, 143.5, 139.4, 136.4, 136.2, 133.8, 128.9, 128.7, 128.6, 127.3, 126.8, 122.7, 120.3, 118.3, 110.0, 64.5, 34.4, 30.6, 29.5, 25.6; IR (thin film) 2925, 1637, 1451, 1376, 1165, 997, 758, 693 cm^{-1} HRMS (ESI) m/z calcd for $C_{25}H_{26}NO_3$ $(M+H)^+$ 388.1907, found 388.1909.



1aj

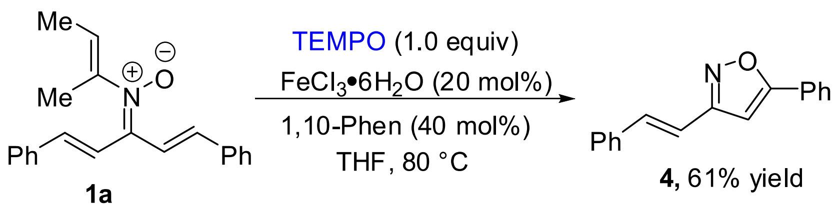
4-tert-Butyl-*N*-((1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-ylidene)cyclohex-1-enamine oxide (1aj), yellow liquid. 0.077 g, 67% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50−7.48 (m, 2H), 7.41−7.40 (m, 2H), 7.37−7.22 (m, 7H), 6.90−6.86 (m, 2H), 5.86−5.82 (m, 1H), 2.49−2.32 (m, 2H), 2.20−2.16 (m, 1H), 1.94−1.91 (m, 2H), 1.33−1.32 (m, 2H), 0.83 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.3, 144.0, 139.4, 136.4, 136.2, 134.3, 128.9, 128.8, 128.6, 127.3, 126.7, 125.7, 124.0, 120.1, 119.1, 56.9, 53.3, 43.3, 32.1, 27.4, 27.1, 25.7, 23.4; IR (thin film) 2959, 2874, 1720, 1627, 1450, 1216, 1183, 968, 694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{27}H_{32}NO$ $(M+H)^+$ 386.2478, found 386.2477.



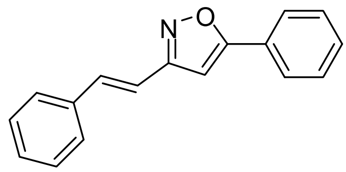
1al

***N*-((1*E*,4*E*)-1,5-Diphenylpenta-1,4-dien-3-ylidene)-1-phenylethenamine oxide (1al)**, yellow liquid. 0.074 g, 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, J = 6.8 Hz, 2H), 7.44 (d, J = 6.4 Hz, 2H), 7.26−7.14 (m, 13H), 6.88−6.84 (m, 2H), 5.72 (s, 1H), 5.35 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.2, 146.3, 139.9, 136.3, 135.8, 134.8, 132.6, 129.4, 129.1, 128.8, 128.7, 128.7, 128.6, 127.3, 126.8, 125.4, 119.7, 118.0, 110.5; IR (thin film) 2924, 2852, 1629, 1447, 1371, 1259, 1176, 962, 691 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{25}H_{22}NO$ $(M+H)^+$ 352.1696, found 352.1692.

6. Synthesis of isoxazole 4



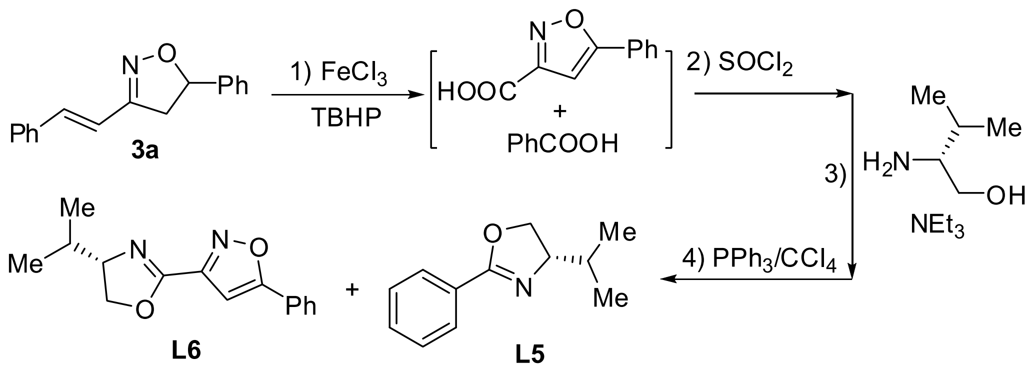
General procedure D: A Teflon-sealed flask was charged with *N*-vinyl nitrone **1a** (0.061 g, 0.2 mmol), $FeCl_3$•$6H_2O$ (11 mg, 20 mol%), 1,10-Phen (15 mg, 40 mol%), TEMPO (31 mg, 1.0 equiv) under N_2 atmosphere. THF (2 mL) was then added via syringe and the reaction vessel was once again sealed with a Teflon cap. The reaction mixture was stirred at 25 °C for 5 min and then heated at 80 °C for 7 h until nitrone **1a** was consumed completely (monitored by TLC). At this time, the solvent was removed under reduced pressure and the crude product was purified by flash column chromatography (the crude residue was dry loaded on silica gel, 1/50 to 1/20, ethyl acetate/petroleum ether) to afford isoxazole **4**.



4

(*E*)-5-Phenyl-3-styrylisoxazole (4)[9], light yellow solid, 0.030 g, 61% yield. Mp: 112−113 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.74−7.72 (m, 2H), 7.47 −7.45 (m, 2H), 7.41 −7.35 (m, 3H), 7.33 −7.23 (m, 3H), 7.18 (d, J = 16.4 Hz, 1H), 6.63 (d, J = 16.8 Hz, 1H), 6.67 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.7, 162.2, 135.8, 135.8, 130.2, 129.0, 128.9, 128.8, 127.4, 127.0, 125.8, 116.1, 96.4.

7. Synthesis of ligand L5 and L6

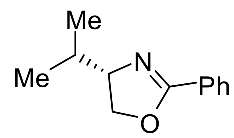


General procedure E: A Teflon-sealed reaction flask was charged with isoxazoline **3a** (1 mmol), $FeCl_3{\bullet}6H_2O$ (5 mol%), TBHP (6 mmol, aq. 70%) and 1 mL of water. The reaction was stirred for 1 h and then NaOH (4 mmol) was added. The reaction mixture was then heated at 80 °C (using oil bath) for 10 h. Progress of the reaction was monitored by TLC. The reaction mixture was then cooled to room temperature and extracted with ethyl acetate (2 × 15 mL), and the aqueous layer was treated with diluted HCl and crushed ice. This mixture was then extracted with ethyl acetate (1 × 10 mL) and combined organic phase was washed with saturated brine solution, dried with anhydrous Na_2SO_4, and concentrated under reduced pressure to afford the mixture of isoxazole and benzoic acids.

In a 25 mL flask was charged with the above mixture of isoxazole and benzoic acids. Thionyl chloride (2 mL) was added. The reaction mixture was refluxed for 2 h and then concentrated in vacuo. Dry CH_2Cl_2 (2 mL) was then added, and the mixture was concentrated in vacuo to remove any remaining thionyl chloride.

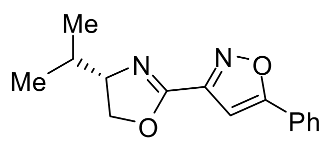
In a 25 mL flask, L-valinol (1.2 equiv) and Et_3N (1.5 equiv) were dissolved in dry CH_2Cl_2 (2 mL) and cooled to 0 °C. The resulted solution of the above chloride in DCM (2 mL) was added dropwise over a period of 10 min. The reaction mixture was stirred at 0 °C for 0.2 h, allowed to warm to room temperature, and stirred for 8 h. The result solution was washed with water (10 mL) and dried over anhydrous Na_2SO_4.The solvent was removed in vacuo to give amide as an oil, which was used directly in next step.

To a solution of the above resulted amide in MeCN (2 mL) were added PPh_3 (3 equiv), Et_3N (3 equiv), and CCl_4 (10 equiv). The reaction mixture was stirred for overnight at room temperature. After completion of the reaction (monitored by TLC), water (10 mL) was added and the resulting mixture wasextracted with CH_2Cl_2 (10 mL). The combined organic phase was dried over anhydrous Na_2SO_4. The solvent was removed in vacuo, and the crude product was purified by flash column chromatography (EtOAc / petroleum ether) 1/20−1/10) to give ligand **L5** and **L6**.



L5

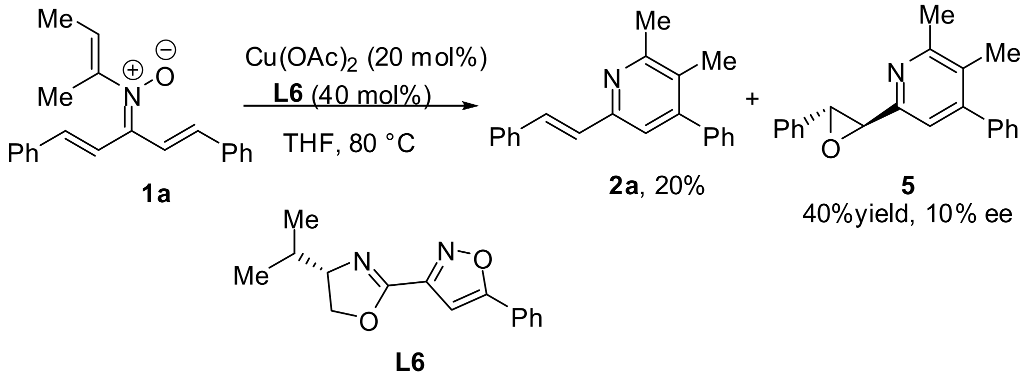
(S)-4-Isopropyl-2-phenyl-4,5-dihydrooxazole (L5)[10], yellow oil, 0.100 g, 53% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 7.2Hz, 2H), 7.46−7.36 (m, 3H), 4.40−4.35 (m, 1H), 4.14−4.06 (m, 2H), 1.87−1.81 (m, 1H), 1.03 (d, J = 6.8 Hz, 3H), 0.92 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.2, 131.0, 128.1, 128.1, 127.8, 72.5, 69.9, 32.7, 18.8, 18.0.



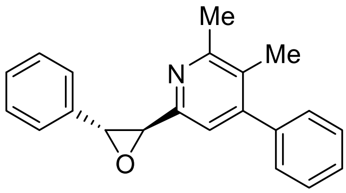
L6

(S)-3-(4-Isopropyl-4,5-dihydrooxazol-2-yl)-5-phenylisoxazole (L6), yellow oil, 0.095 g, 37% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49−7.47 (m, 2H), 7.38−7.32 (m, 3H), 6.69 (d, J = 16 Hz, 1H), 4.37−4.32 (m, 1H), 4.07−4.02 (m, 2H), 1.84−1.79 (m, 1H), 1.03 (d, J = 6.4 Hz, 3H), 0.94 (d, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.2, 139.7, 135.3, 129.3, 128.8, 128.7, 127.4, 115.3, 72.6, 69.9, 32.8, 18.9, 18.3; IR (thin film) 2959 , 2927, 1654, 1609, 1453, 1362, 1256, 1174, 988, 759, 694 cm^{-1}; HRMS (ESI) *m/z* calcd for $C_{15}H_{17}N_2O_2$ $(M+H)^+$ 257.1285, found 257.1281; $[\alpha]^{20}_D$ = −6.8° (c = 0.1, MeOH).

8. Synthesis of epoxypyridine 5



General procedure F: A Teflon-sealed flask was charged with *N*-vinyl nitrone **1a** (61 mg, 0.2 mmol), $Cu(OAc)_2$ (7 mg, 20 mol%), **L6** (21 mg, 40 mol%) under N_2 atmosphere. THF (2 mL) was then added via syringe and the reaction vessel was once again sealed with a Teflon cap. The reaction mixture was stirred at 25 °C for 5 min and then heated at 80 °C for 10 h until nitrone **1a** was consumed completely (monitored by TLC). At this time, the solvent was removed under reduced pressure and the crude product was purified by flash column chromatography (the crude residue was dry loaded on silica gel, 1/50 to 1/20, ethyl acetate/petroleum ether) to afford pyridine **2a** (20% yield) and epoxypyridine **5** (40% yield).



5

2,3-Dimethyl-4-phenyl-6-(3-phenyloxiran-2-yl)pyridine (5), light yellow oil, 0.024 g, 40% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47−7.40 (m, 3H), 7.37−7.29 (m, 7H), 7.07 (s, 1H), 4.05 (d, J = 3.2 Hz, 2H), 2.60 (s, 3H), 2.21 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.7, 152.4, 150.3, 139.7, 136.9, 128.7, 128.6, 128.4, 128.3, 128.3, 127.8, 125.7, 118.5, 62.9, 61.8, 23.4, 16.1; IR (thin film) 3032, 2967, 2931, 1614, 1551, 1495, 1452, 1260, 1071, 967, 752 cm^{-1}, HRMS (ESI) *m/z* calcd for $C_{21}H_{20}NO$ $(M+H)^+$ 302.1539, found 302.1536. Ee = 10%, conditions: AD-H; hexane/i-PrOH = 95/5; flow rate: 0.3 mL/min; λ = 254 nm; t (minor) = 50.0 min; t (major) = 73.3 min.

9. References

[1] D. Kontokosta, D. S. Muller, D.-L. Mo, W. H. Pace, R. A. Simpon, and L. L. Anderson, *Beilstein J. Org. Chem.* 2015, **11**, 2097.

[2] C. K. Banerjee, J. D. Umarye, and P. R. Kanjilal, *Synth. Commun.* 2013, **43**, 2208.

[3] Liu, J.; Wei, W.; Zhao, T.; Liu, X. Y.; Wu, J.; Yu, W. Q.; Chang, Junbiao;*J. Org. Chem.* **2016**, *81*, 9326.

[4] D. Muri, J. W. Bode, and E. M. Carreira, *Org. Lett.* 2000, **2**, 539.

[5] D. Shi, H.-T. Qin, C. Zhu, and F. Liu, *Eur. J. Org. Chem.* 2015, 5084.

[6] A. Yoshimura, C. Zhu, K. R. Middleton, A. D. Todora, B. J. Kastern, A. V. Maskaev, and V. V. Zhdankin, *Chem. Commun.* 2013, **49**, 4800.

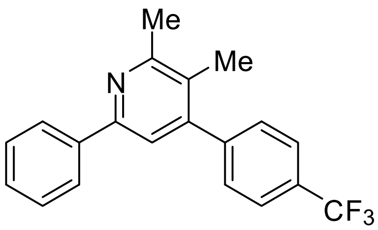
[7] H. Huang, F. Li, Z. Xu, J. Cai, X. Ji, and G.-J. Deng, *Adv. Synth. Catal.* 2017, **359**, 3102.

[8] N. Zou, J.-W. Jiao, Y. Feng, C.-H. Chen, C. Liang, G.-F. Su, and D.-L. Mo, *Adv. Synth. Catal.* 2017, **359**, 3545.

[9] S. Tang, J. He, Y. Sun, L. He, and X. She, *Org. Lett.* 2009, **11**, 3982.

[10] P. Wipf, and X. Wang, *J. Comb. Chem.* 2002, **4**, 656.

10. X-ray structure of compounds 2n and 3m



2n

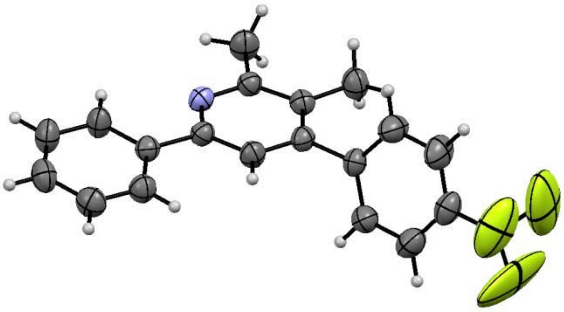
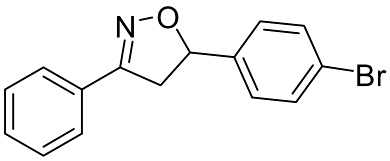


Figure S1: ORTEP diagram of **2n** at 50% ellipsoid probability.



3m

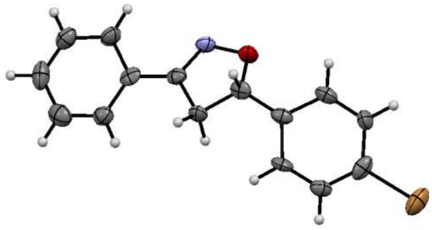
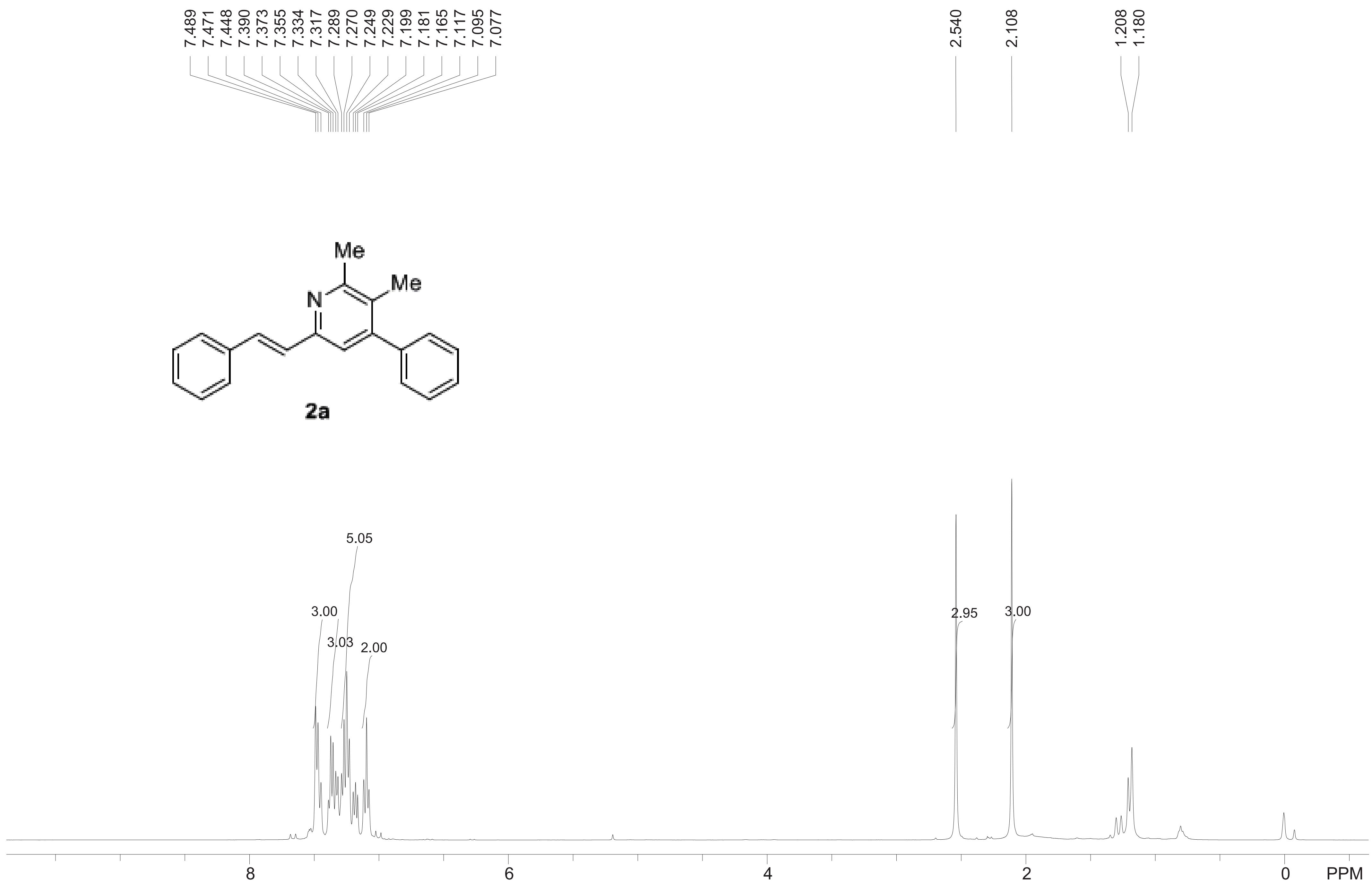
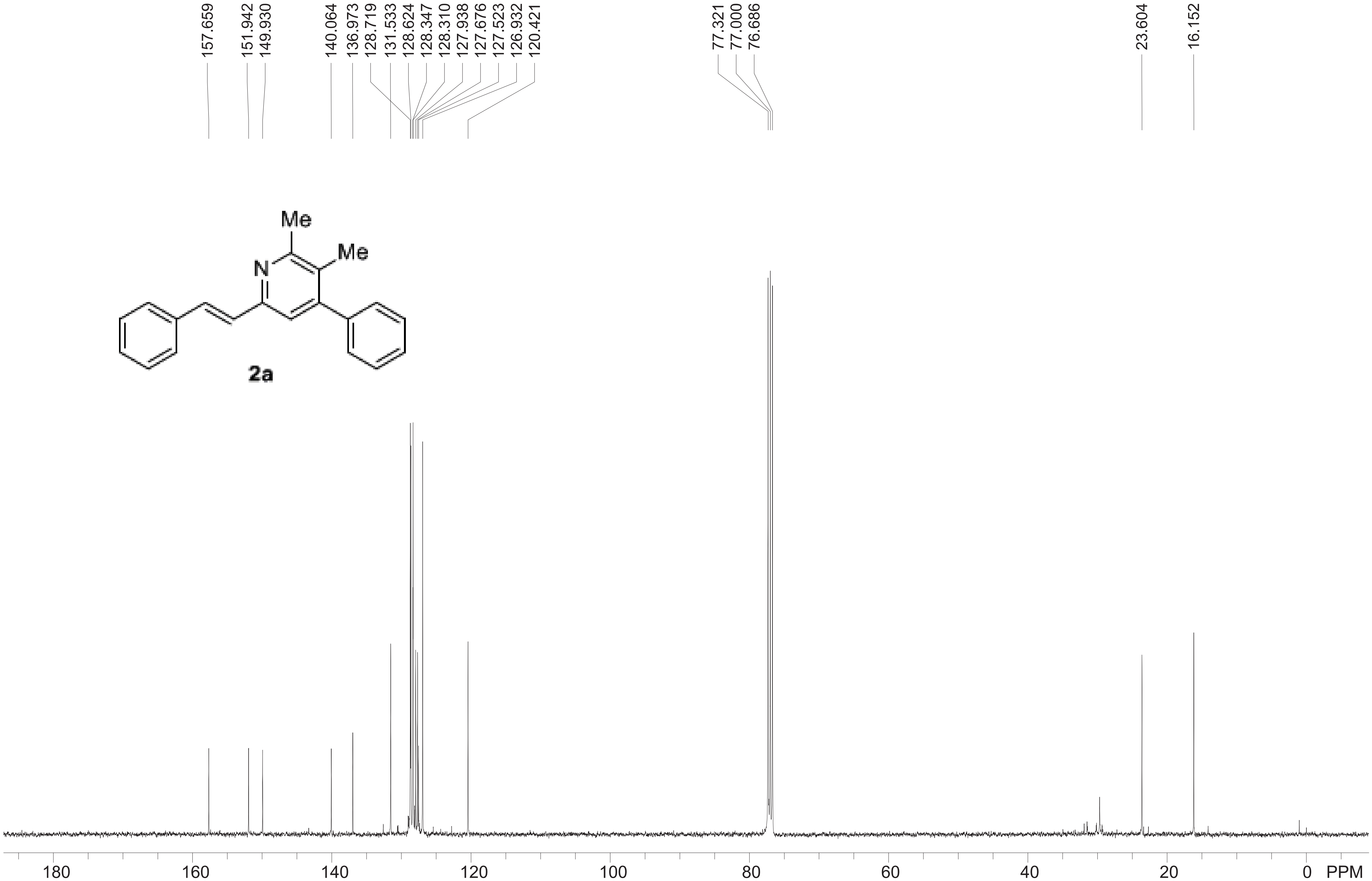


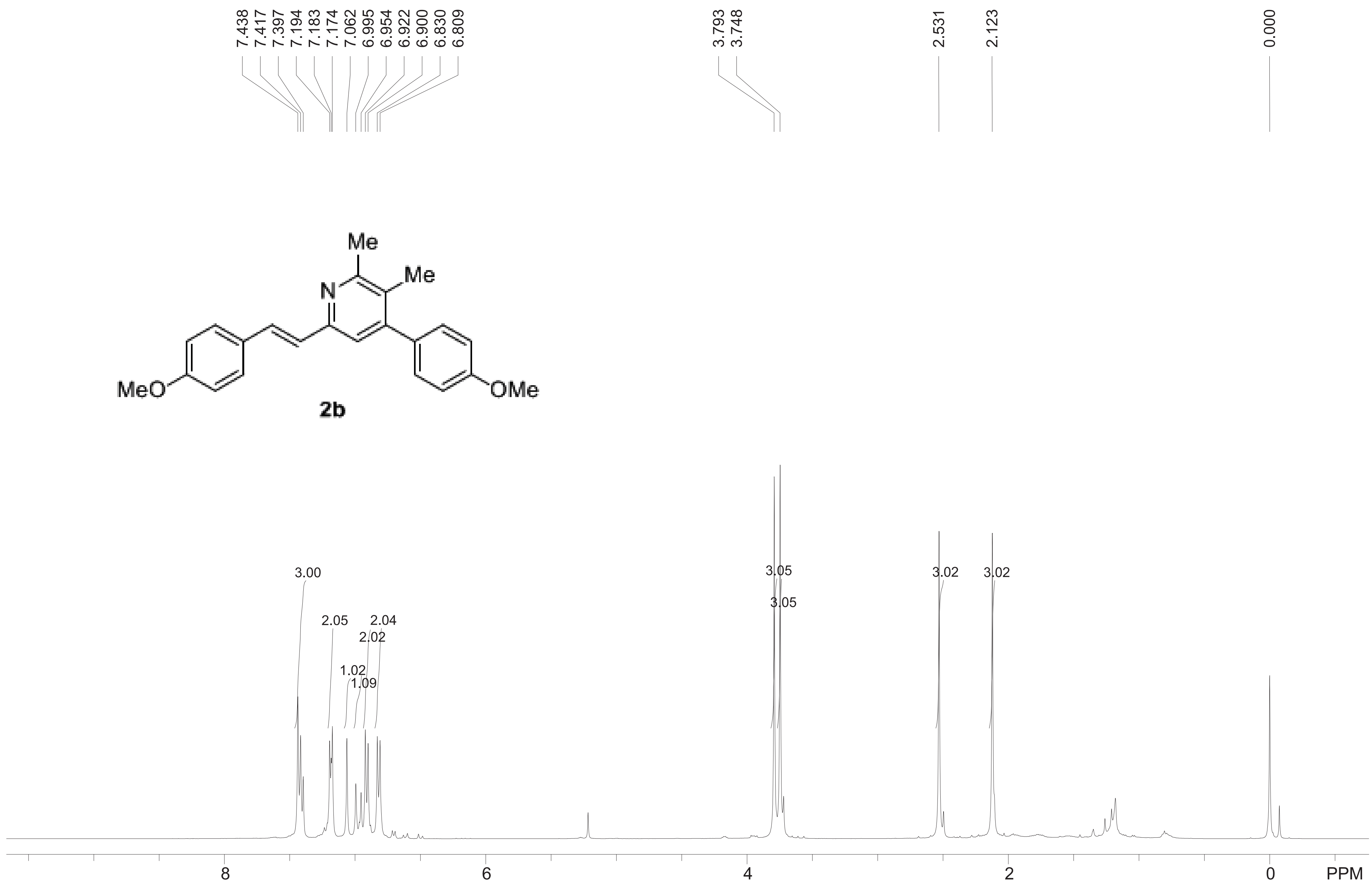
Figure S2: ORTEP diagram of **3m** at 50% ellipsoid probability.

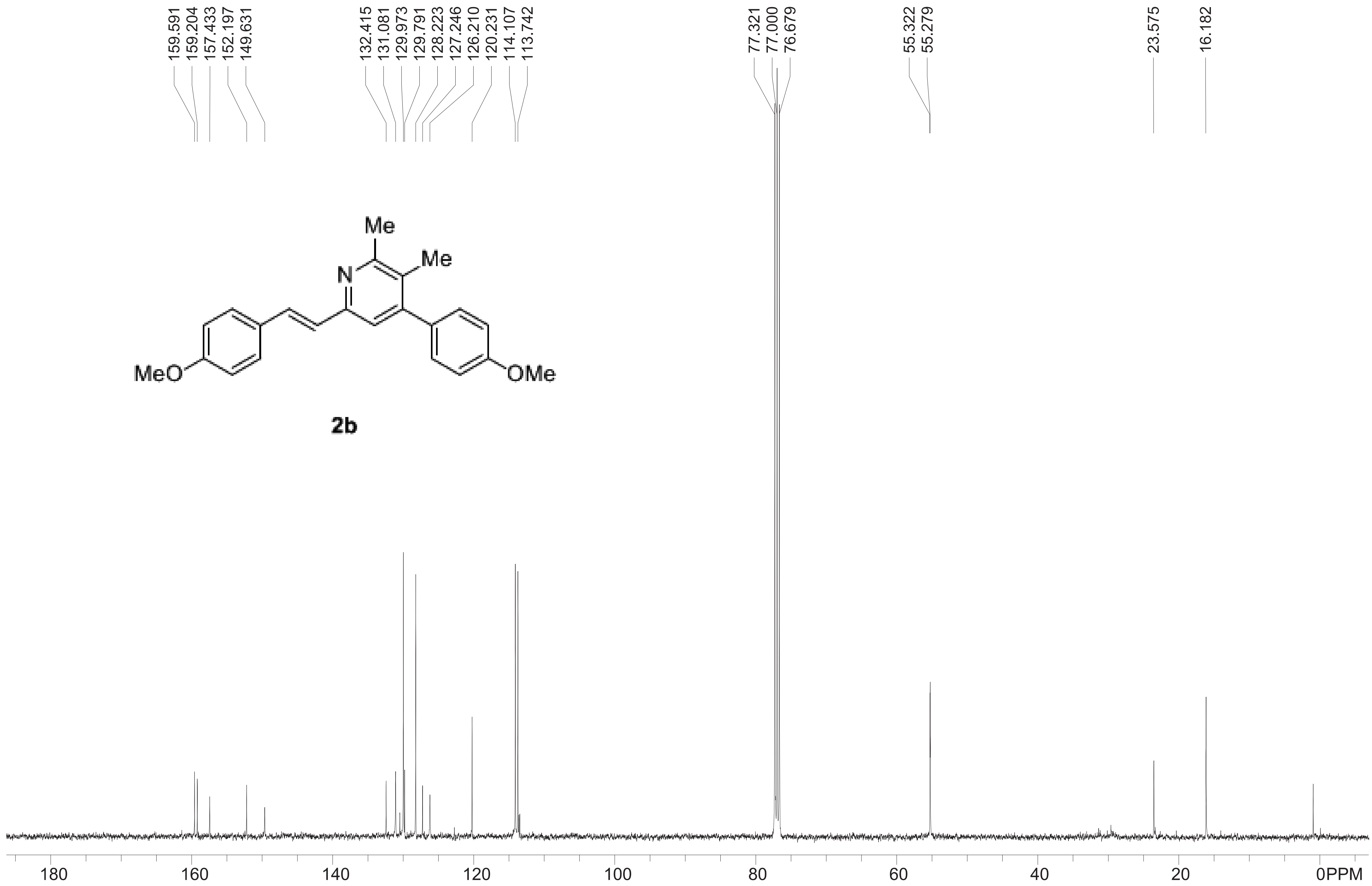
11. NMR spectra for 2, 3, 1, 4, L5, L6, 5, and HPLC for 5

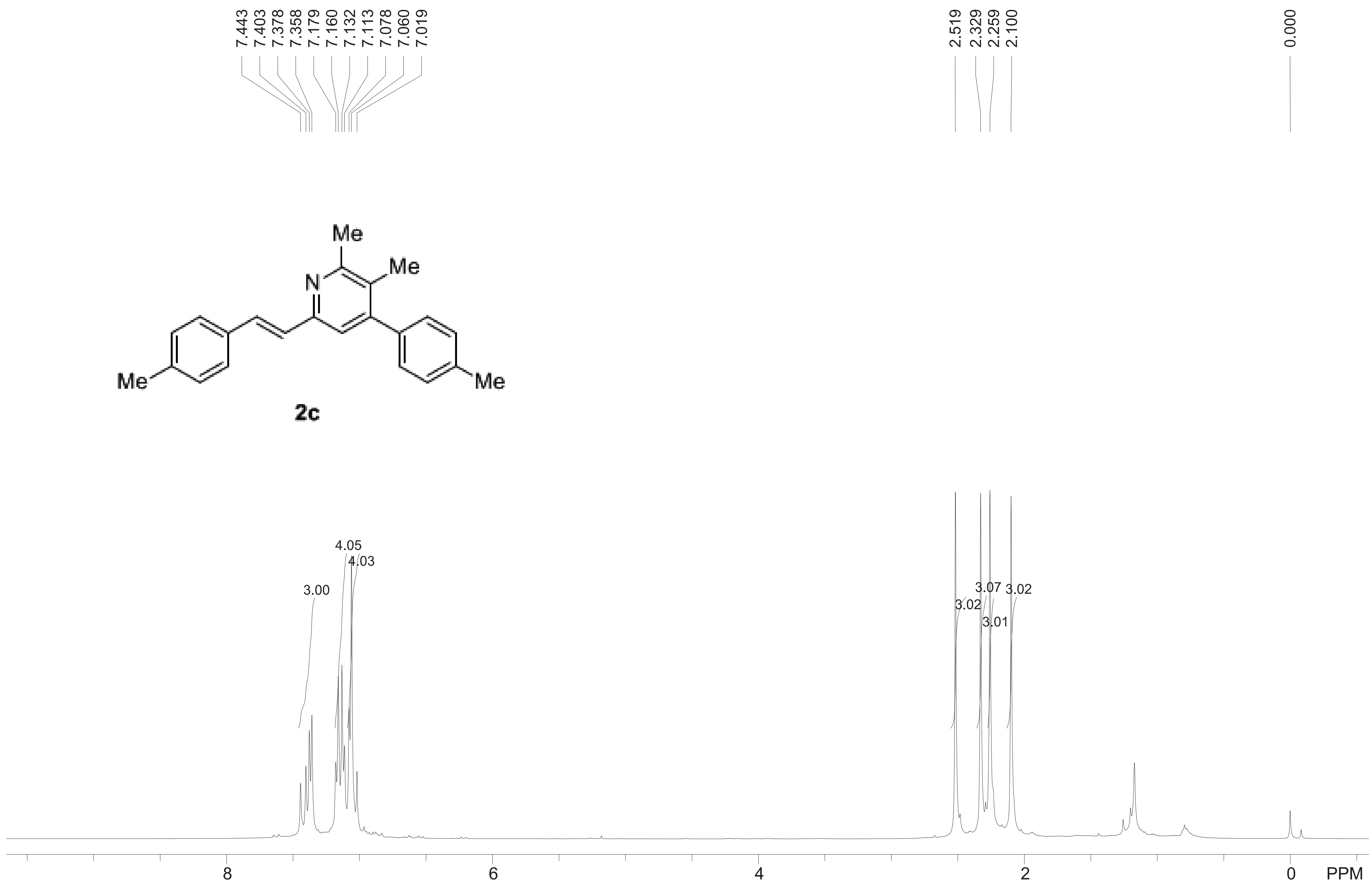

7.489
7.471
7.448
7.390
7.373
7.355
7.334
7.317
7.289
7.270
7.249
7.229
7.199
7.181
7.165
7.117
7.095
7.077
2.540
2.108
1.208
1.180
Me
Me
N
2a
5.05
3.00
3.03
2.00
2.95
3.00
8
6
4
2
0
PPM

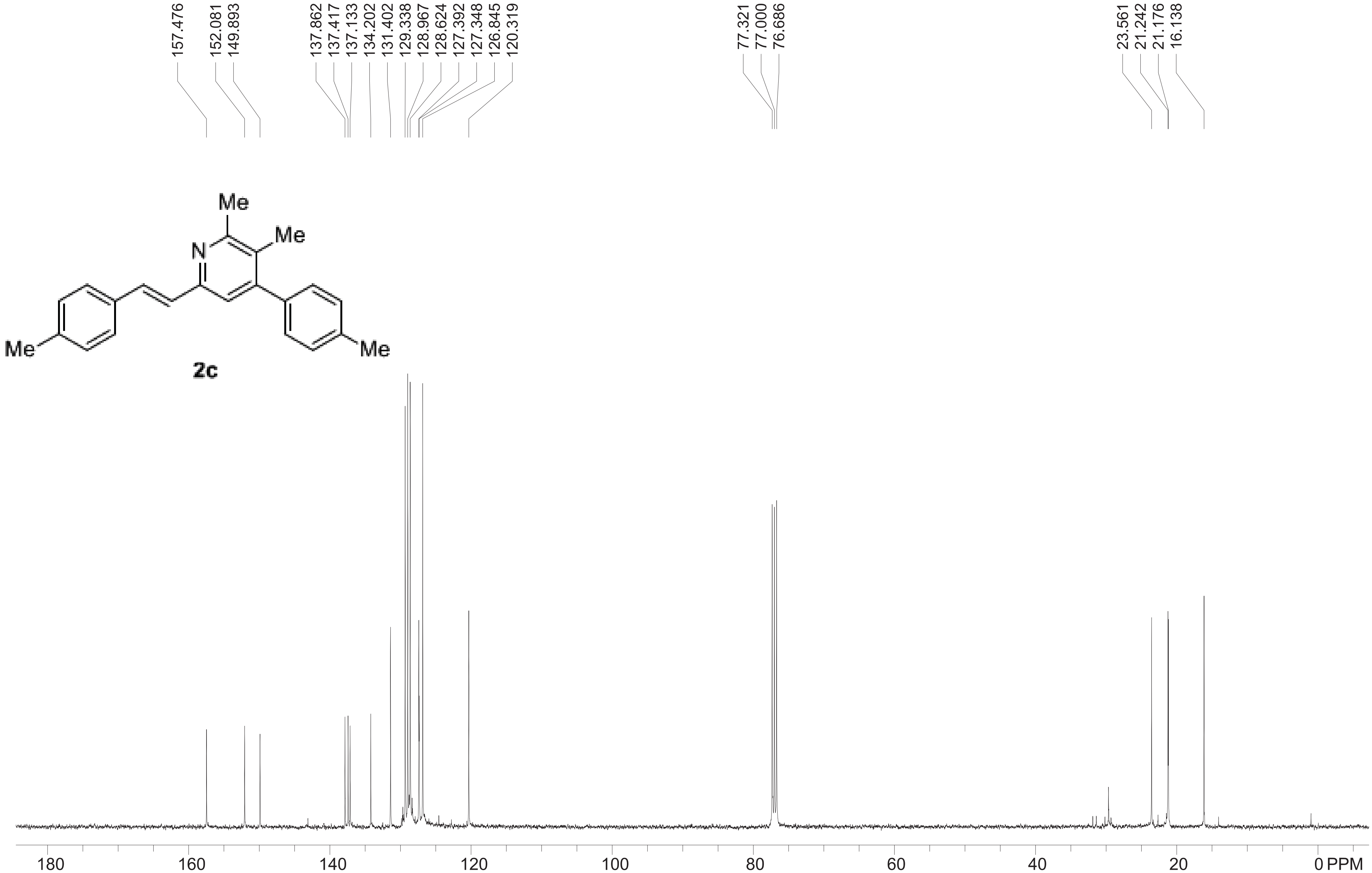

157.659
151.942
149.930
140.064
136.973
128.719
131.533
128.624
128.347
128.310
127.938
127.676
127.523
126.932
120.421
77.321
77.000
76.686
23.604
16.152
180
160
140
120
100
80
60
40
20
0
PPM
Me
Me
N

2a


7.438
7.417
7.397
7.194
7.183
7.174
7.062
6.995
6.954
6.922
6.900
6.830
6.809
3.793
3.748
2.531
2.123
0.000
Me
Me
N
MeO
OMe
2b
3.00
2.05
2.04
2.02
1.02
1.09
3.05
3.05
3.02
3.02
8
6
4
2
0
PPM

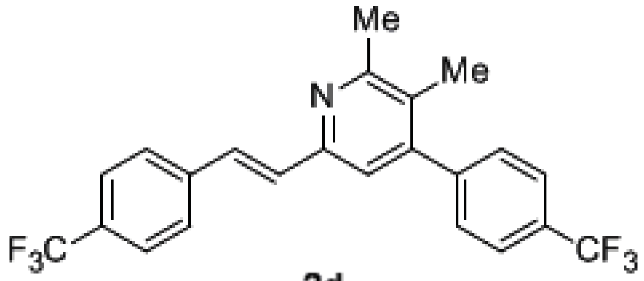

159.591
159.204
157.433
152.197
149.631
132.415
131.081
129.973
129.791
128.223
127.246
126.210
120.231
114.107
113.742
77.321
77.000
76.679
55.322
55.279
23.575
16.182
Me
Me
N
MeO
OMe
2b
180
160
140
120
100
80
60
40
20
0PPM


7.443
7.403
7.378
7.358
7.179
7.160
7.132
7.113
7.078
7.060
7.019
2.519
2.329
2.259
2.100
0.000
Me
Me
N
Me
Me
2c
4.05
4.03
3.00
3.07
3.02
3.02
3.01
8
6
4
2
0
PPM

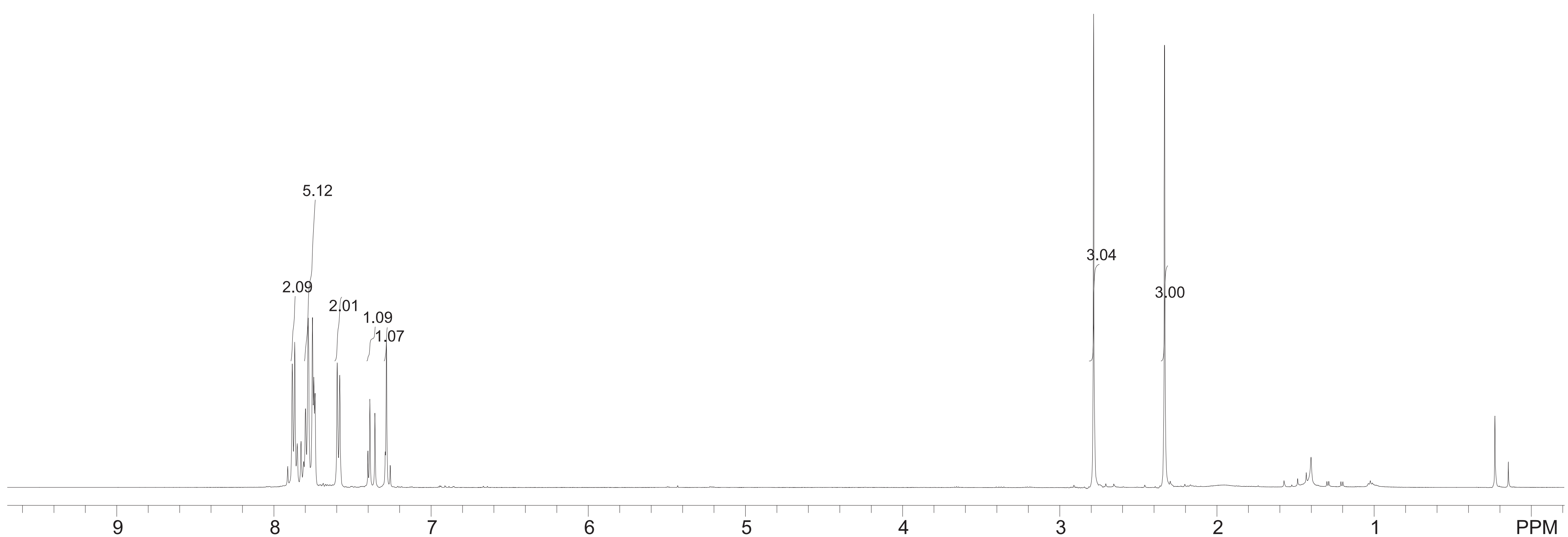


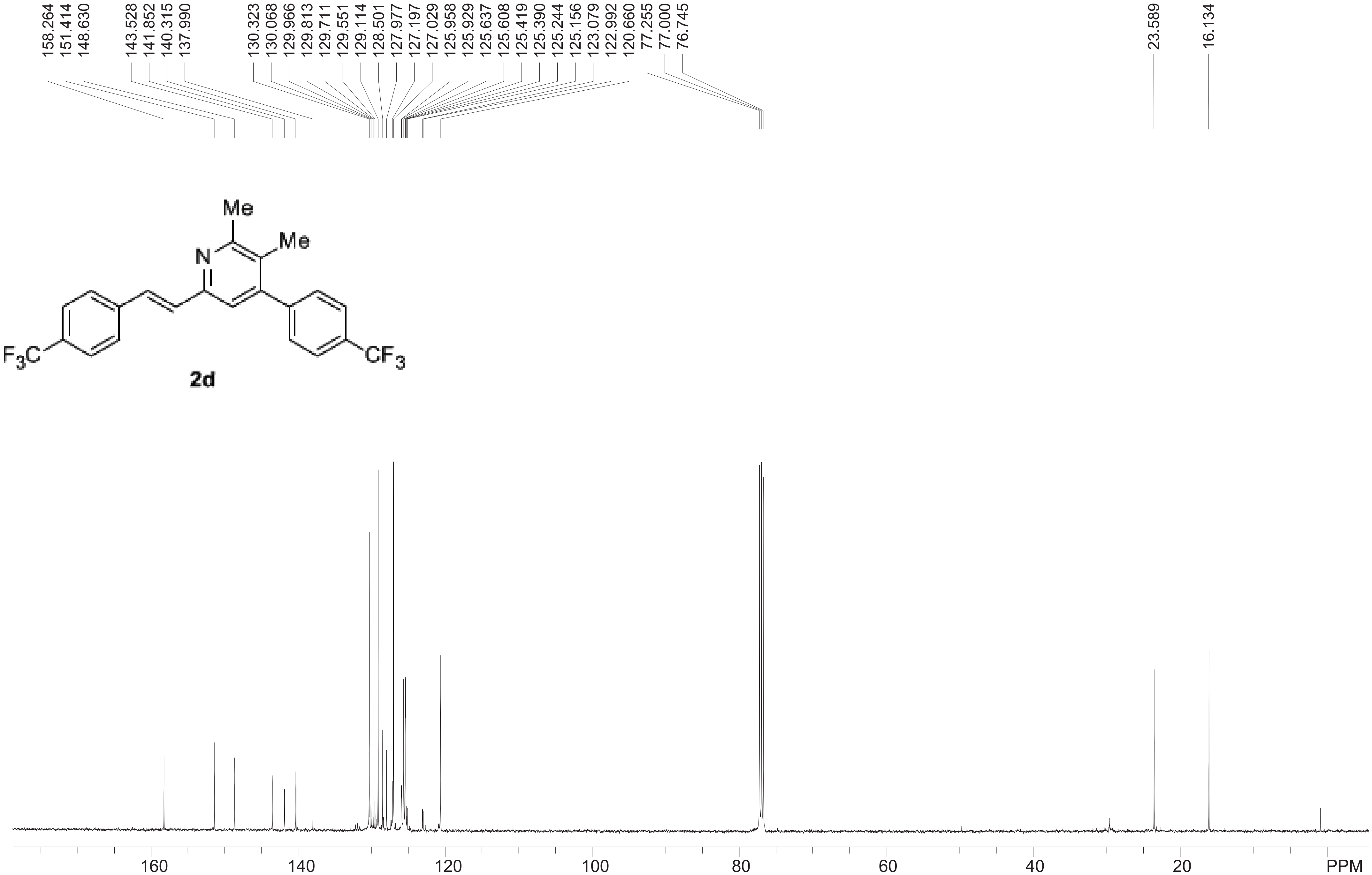
157.476
152.081
149.893
137.862
137.417
137.133
134.202
131.402
129.338
128.967
128.624
127.392
127.348
126.845
120.319
77.321
77.000
76.686
23.561
21.242
21.176
16.138
Me
Me
N
Me
Me
180
160
140
120
100
80
60
40
20
0 PPM

2c

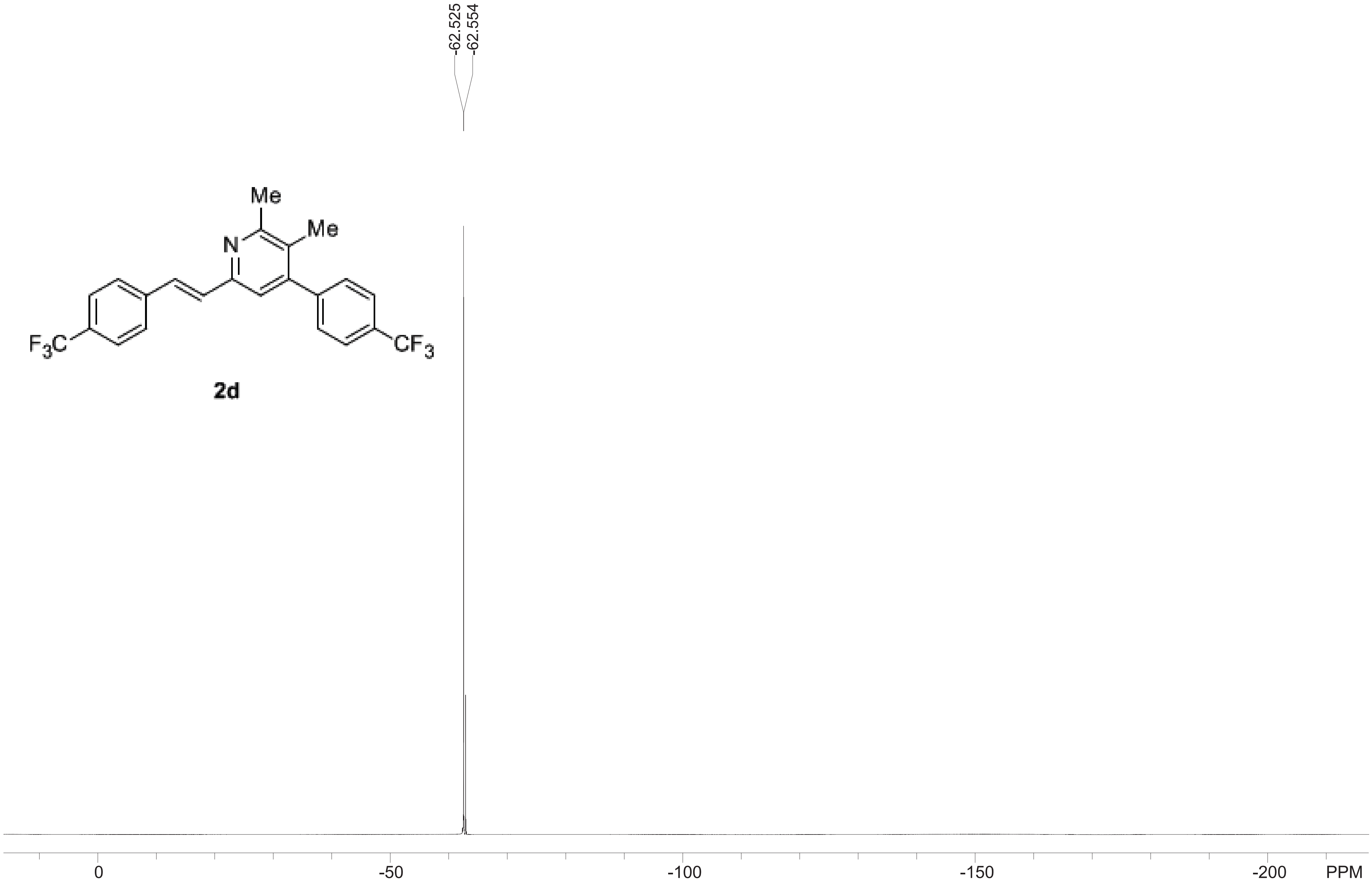

Me
Me
N
F3C
CF3

2d

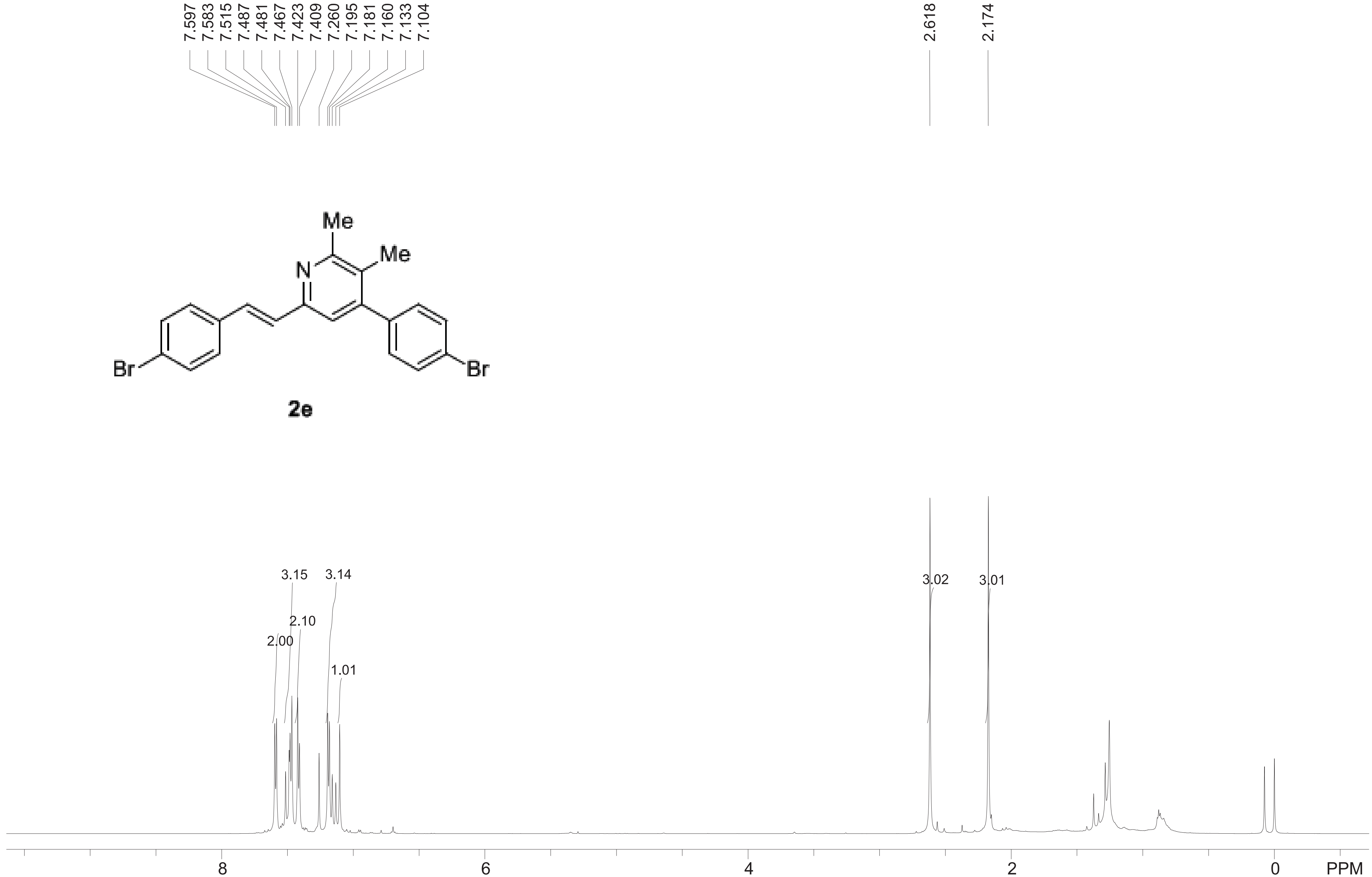

7.912
7.884
7.867
7.851
7.827
7.810
7.799
7.782
7.754
7.746
7.738
7.597
7.581
7.402
7.389
7.357
7.292
7.284
7.260
2.784
2.333
5.12
2.09
2.01
1.09
1.07
3.04
3.00
9
8
7
6
5
4
3
2
1
PPM


158.264
151.414
148.630
143.528
141.852
140.315
137.990
130.323
130.068
129.966
129.813
129.711
129.551
129.114
128.501
127.977
127.197
127.029
125.958
125.929
125.637
125.608
125.419
125.390
125.244
125.156
123.079
122.992
120.660
77.255
77.000
76.745
23.589
16.134
Me
Me
N
F3C
CF3
160
140
120
100
80
60
40
20
PPM

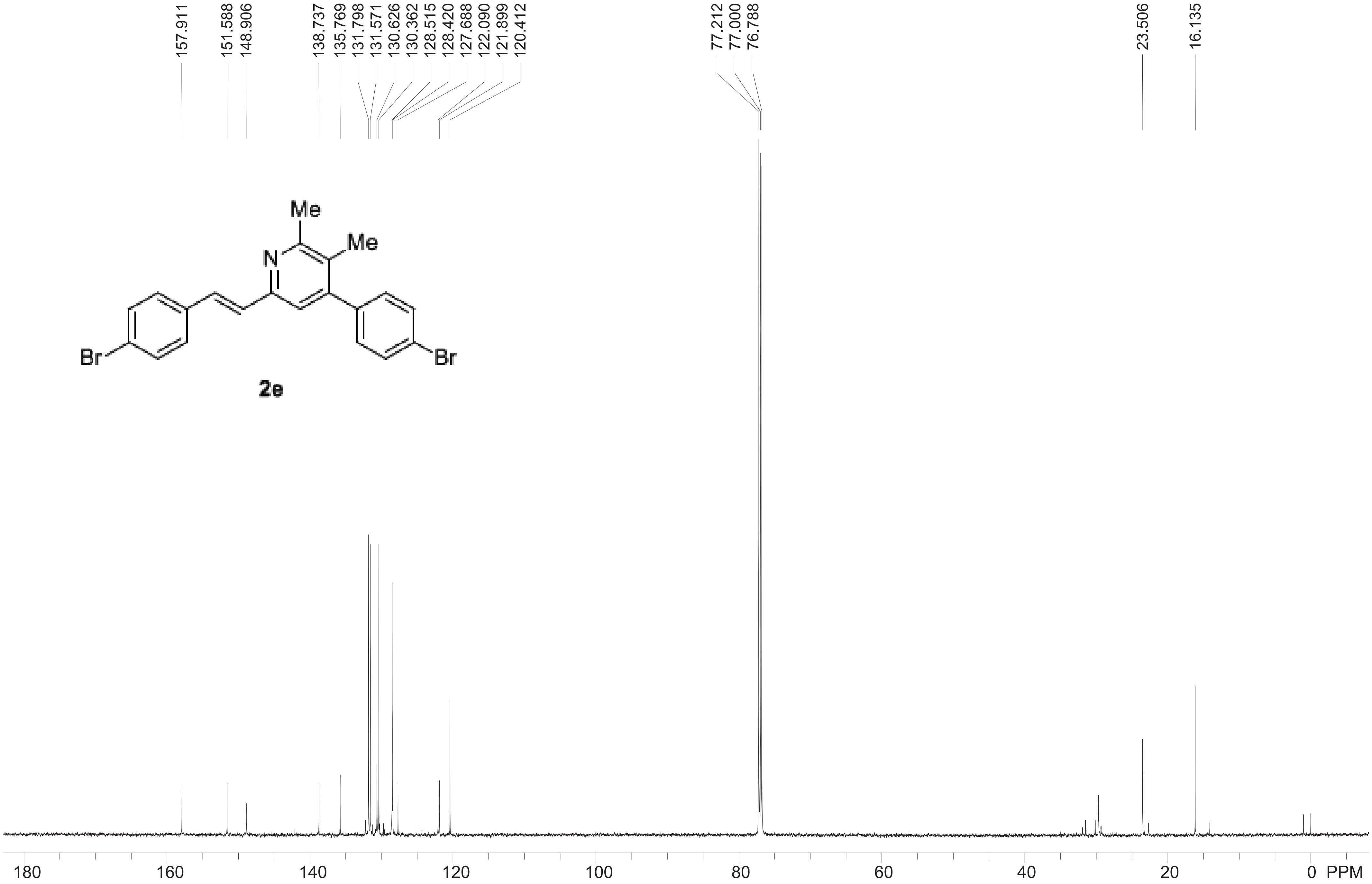
2d

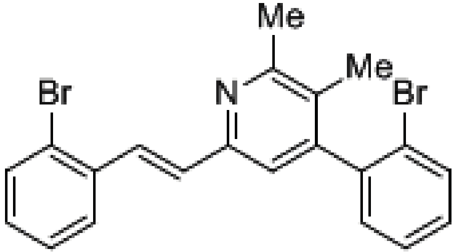


2d

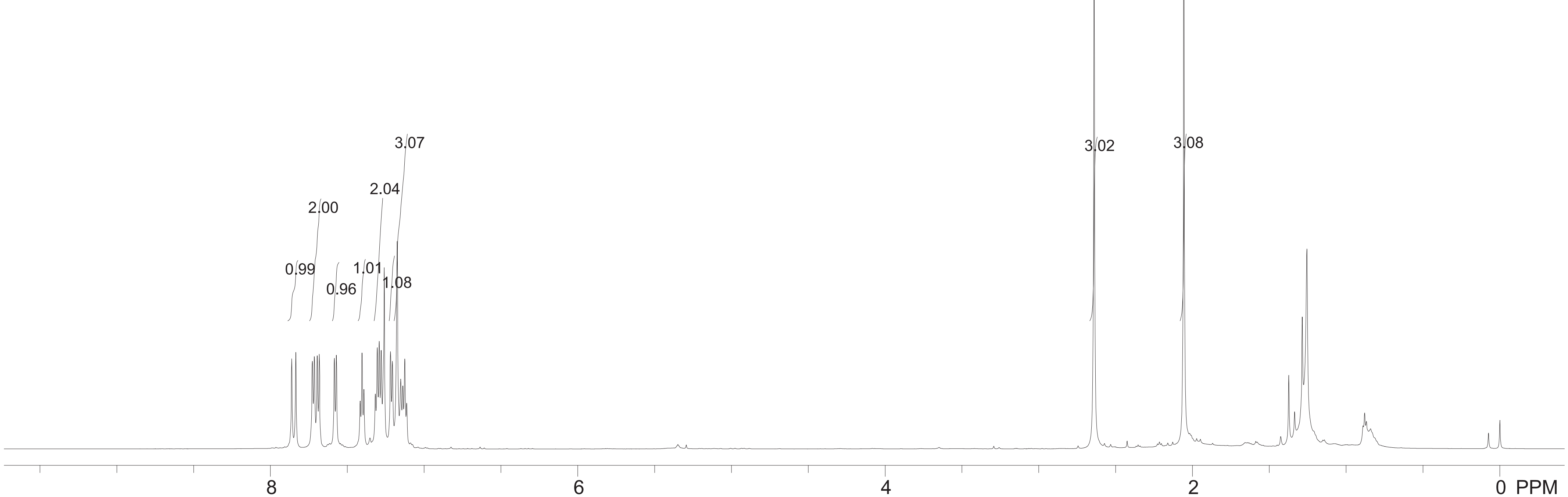


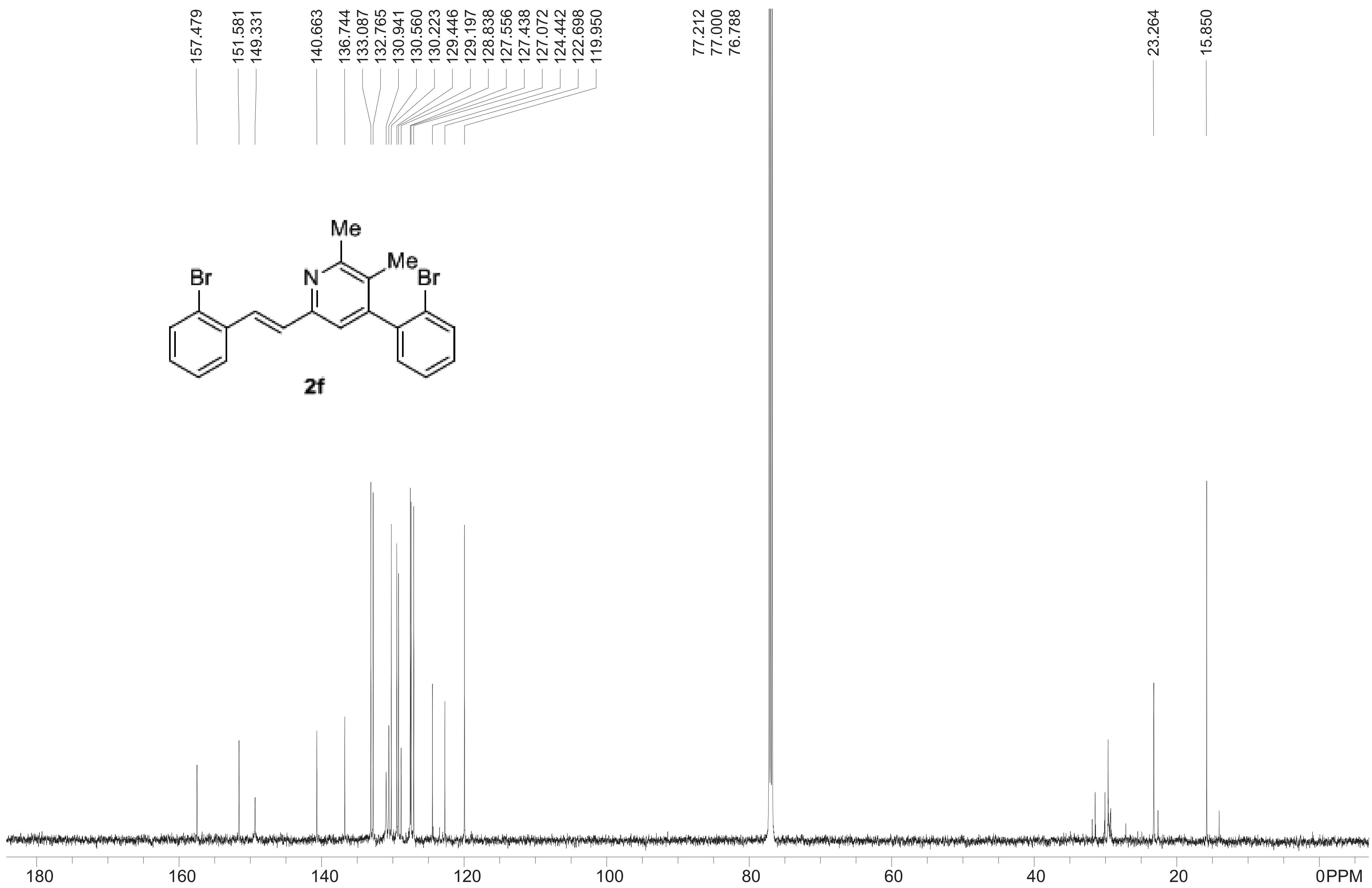
7.597
7.583
7.515
7.487
7.481
7.467
7.423
7.409
7.260
7.195
7.181
7.160
7.133
7.104
2.618
2.174
Me
Me
N
Br
Br
2e
3.15
3.14
2.10
2.00
1.01
3.02
3.01
8
6
4
2
0
PPM


157.911
151.588
148.906
138.737
135.769
131.798
131.571
130.626
130.362
128.515
128.420
127.688
122.090
121.899
120.412
77.212
77.000
76.788
23.506
16.135
Me
Me
N
Br
Br
2e
180
160
140
120
100
80
60
40
20
0 PPM

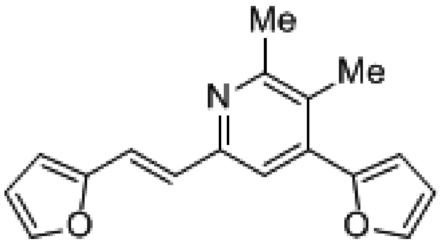

Me
Me
N
Br
Br

2f

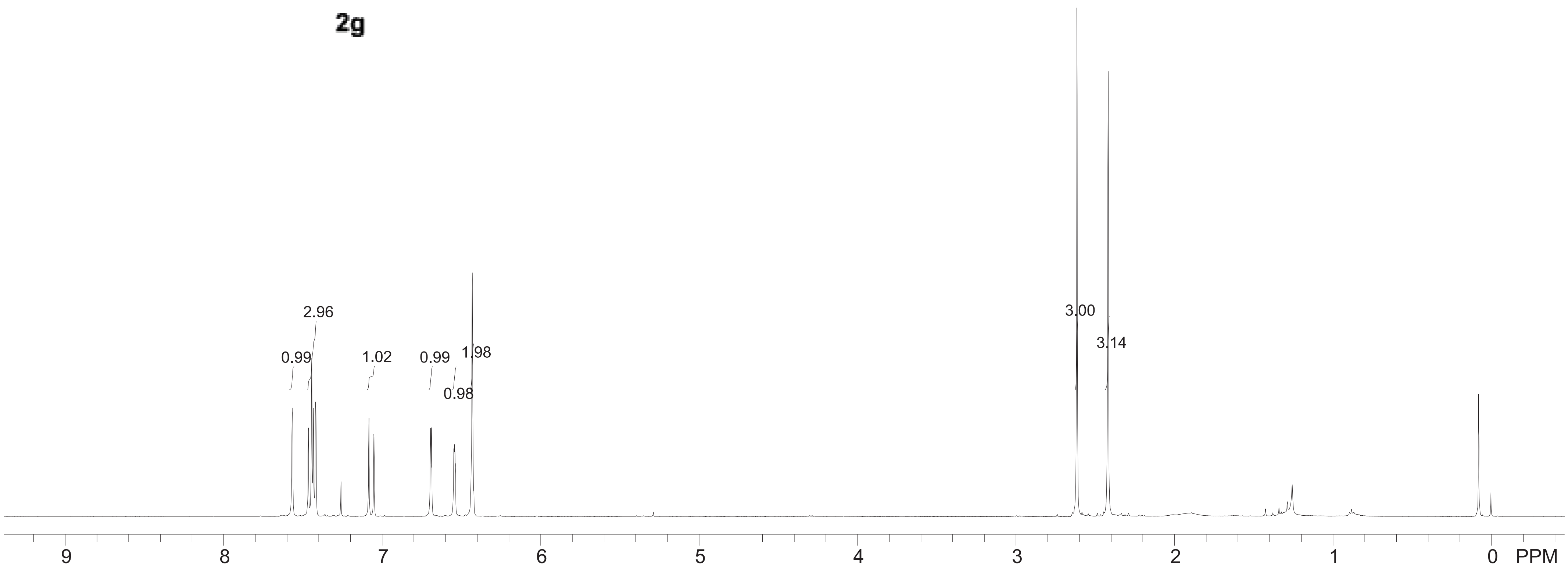

7.861
7.835
7.727
7.714
7.696
7.682
7.584
7.571
7.416
7.404
7.391
7.317
7.305
7.292
7.280
7.259
7.218
7.206
7.175
7.152
7.138
7.126
7.113
2.640
2.056
-0.000
0.99
2.00
0.96
1.01
2.04
1.08
3.07
3.02
3.08
8
6
4
2
0 PPM

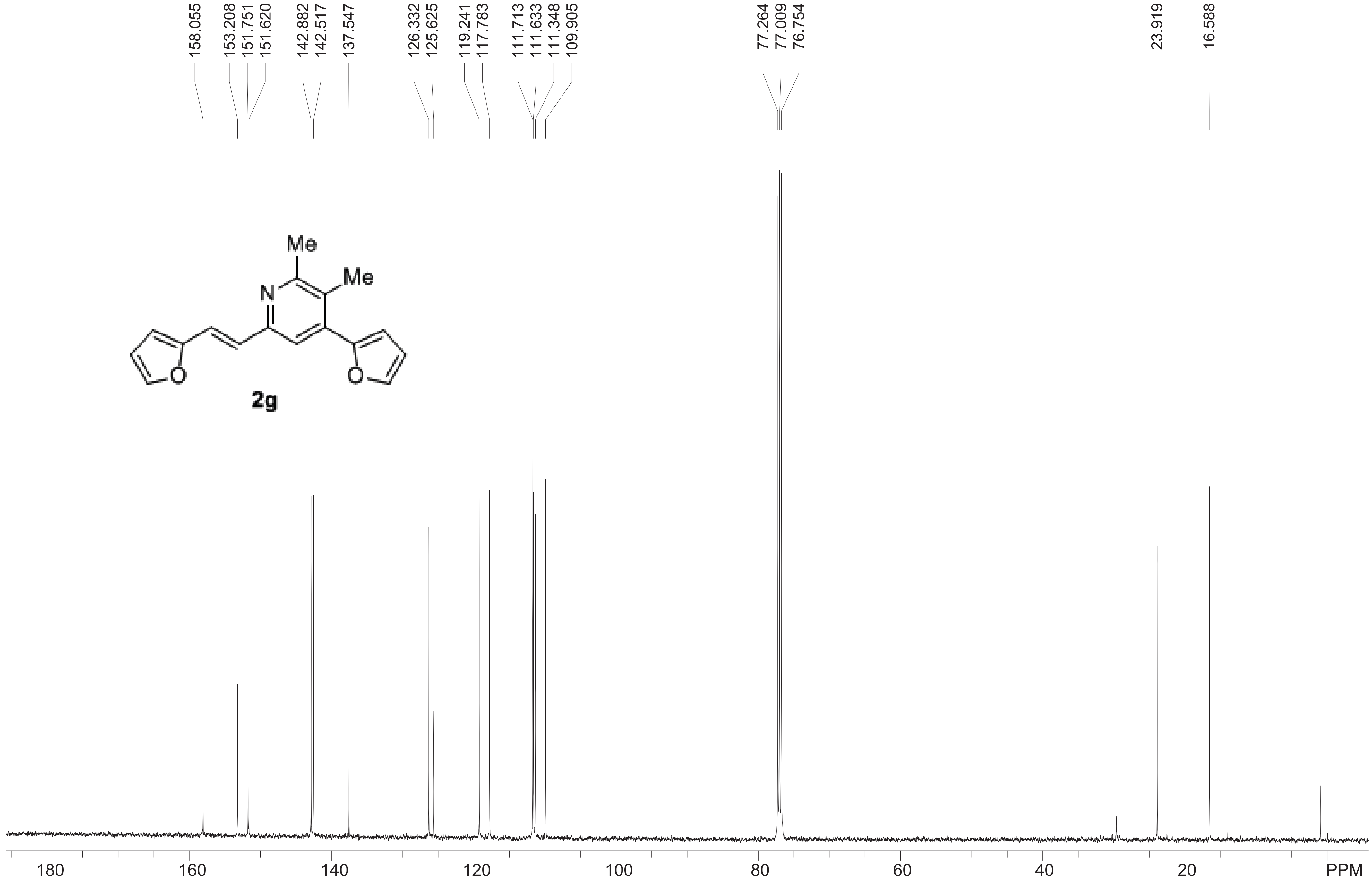


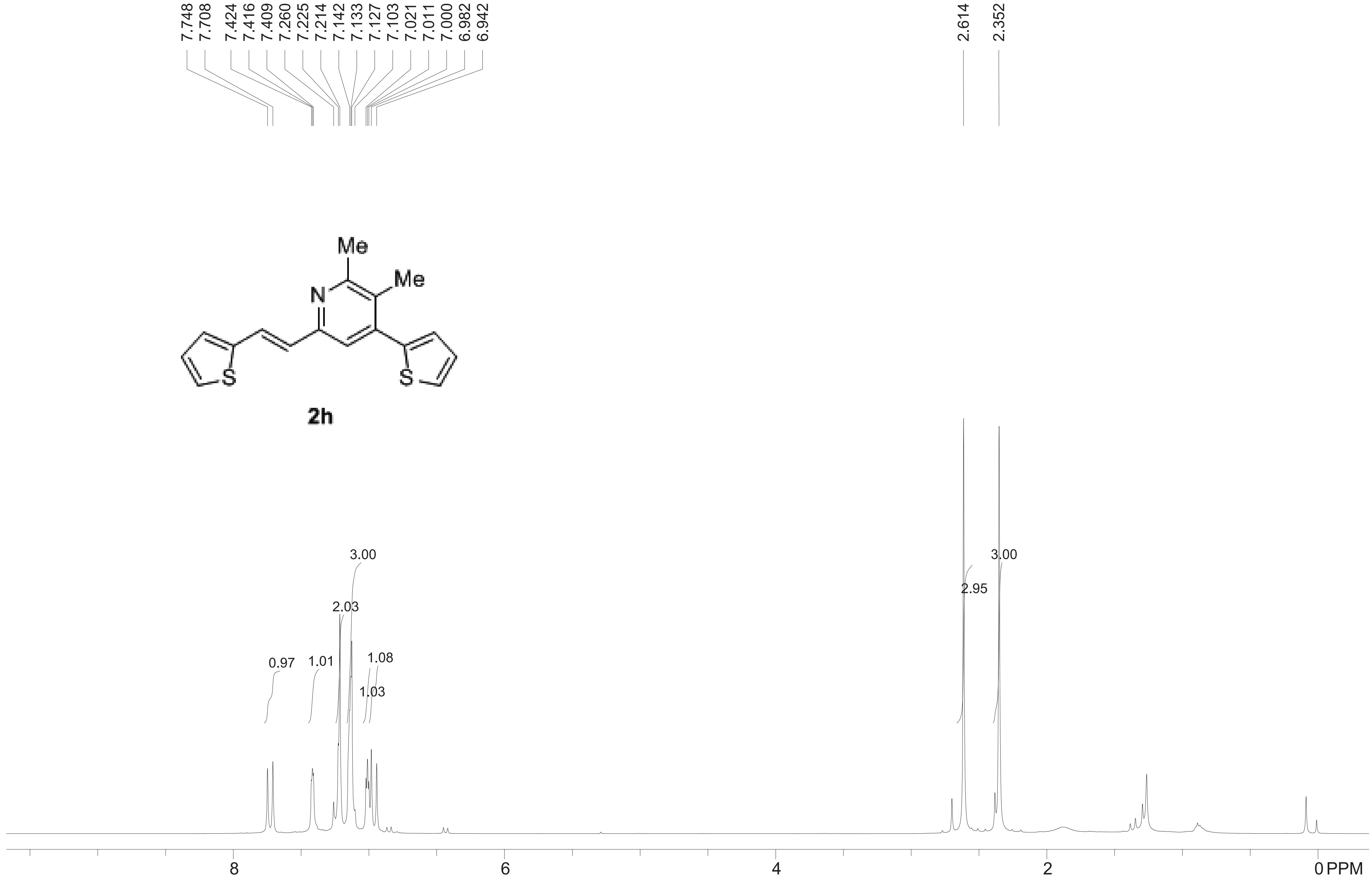
157.479
151.581
149.331
140.663
136.744
133.087
132.765
130.941
130.560
130.223
129.446
129.197
128.838
127.556
127.438
127.072
124.442
122.698
119.950
77.212
77.000
76.788
23.264
15.850
Me
Me
Br
N
Br
2f
180
160
140
120
100
80
60
40
20
0PPM

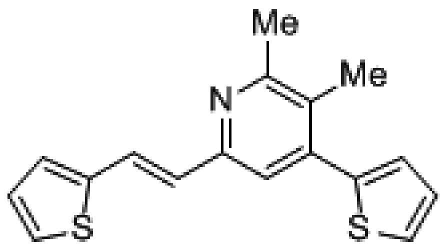

Me
Me
N
O
O

2g


7.568
7.466
7.444
7.434
7.419
7.260
7.083
7.052
6.695
6.688
6.547
6.544
6.541
6.538
6.432
2.616
2.419
2.96
0.99
1.02
0.99
1.98
0.98
3.00
3.14
9
8
7
6
5
4
3
2
1
0
PPM

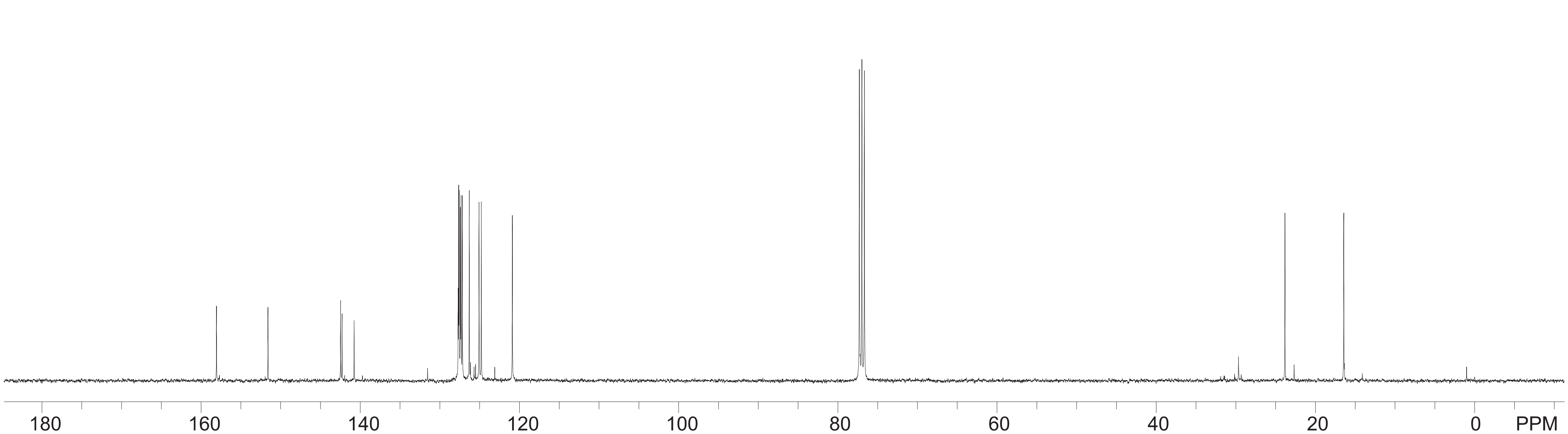

158.055
153.208
151.751
151.620
142.882
142.517
137.547
126.332
125.625
119.241
117.783
111.713
111.633
111.348
109.905
77.264
77.009
76.754
23.919
16.588
Me
Me
N
O
O
2g
180
160
140
120
100
80
60
40
20
PPM


7.748
7.708
7.424
7.416
7.409
7.260
7.225
7.214
7.142
7.133
7.127
7.103
7.021
7.011
7.000
6.982
6.942
2.614
2.352
Me
Me
N
S
S
2h
0.97
1.01
2.03
3.00
1.08
1.03
2.95
3.00
8
6
4
2
0 PPM

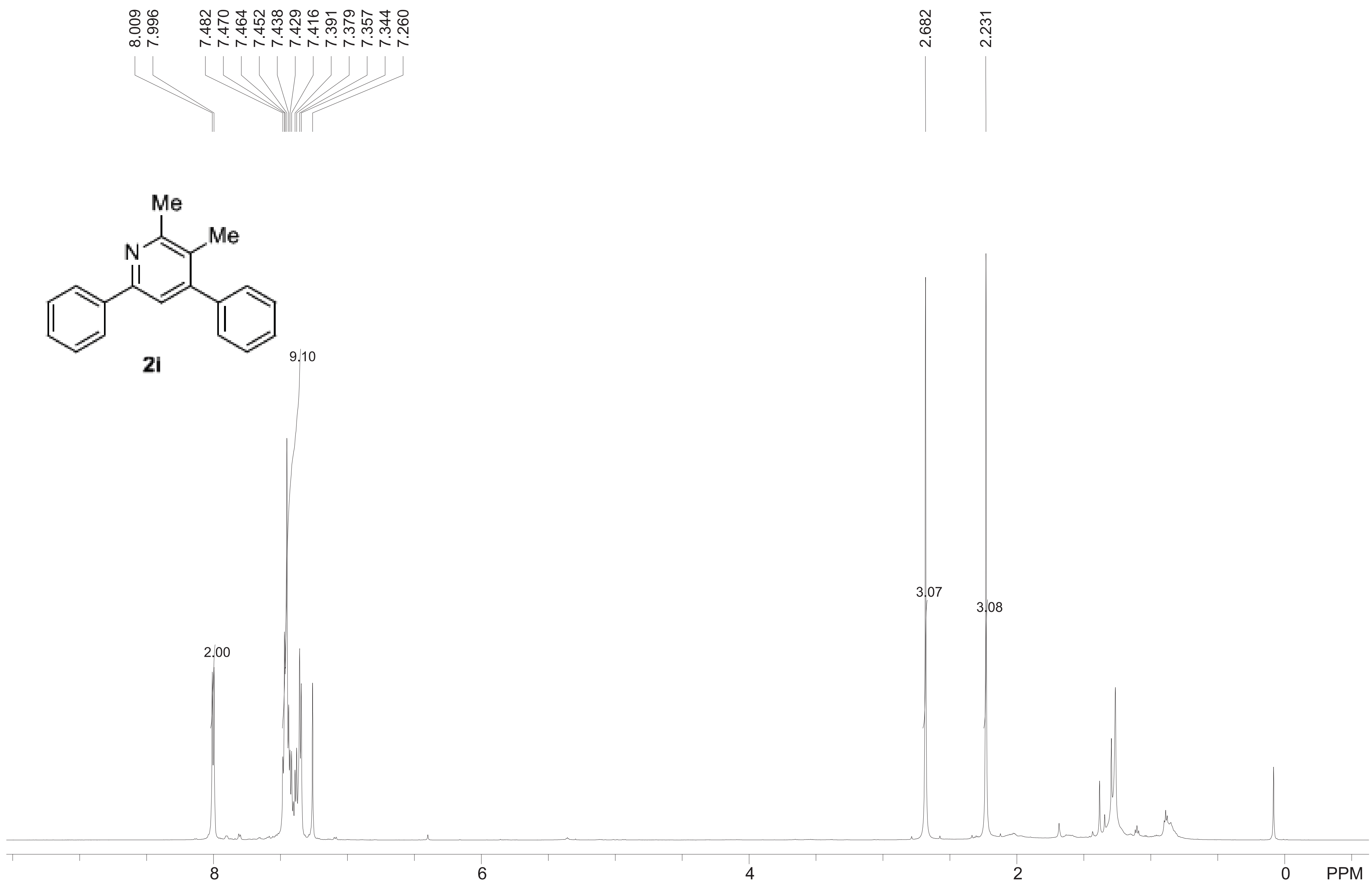


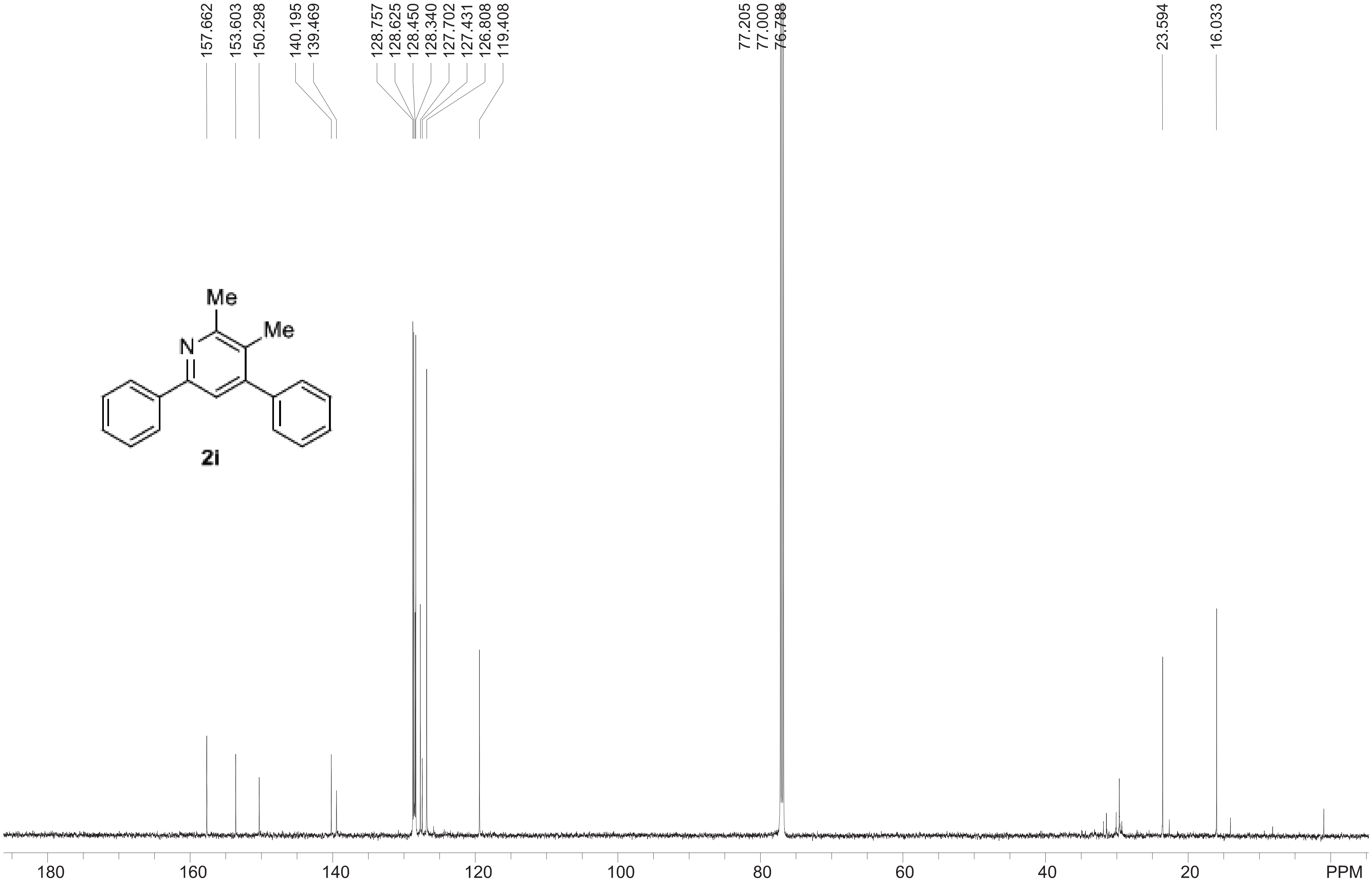
Me
Me
N
S
S

2h

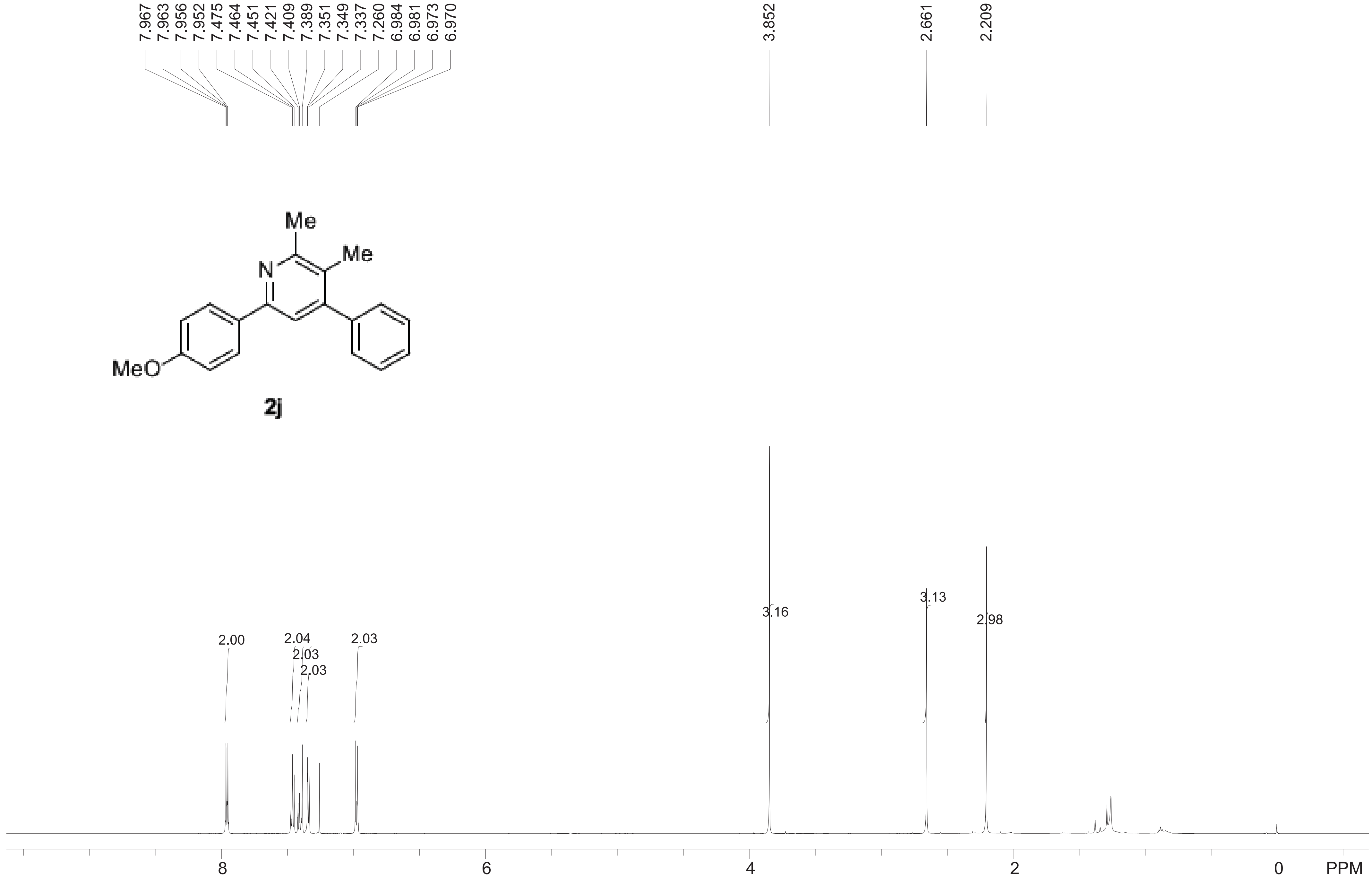


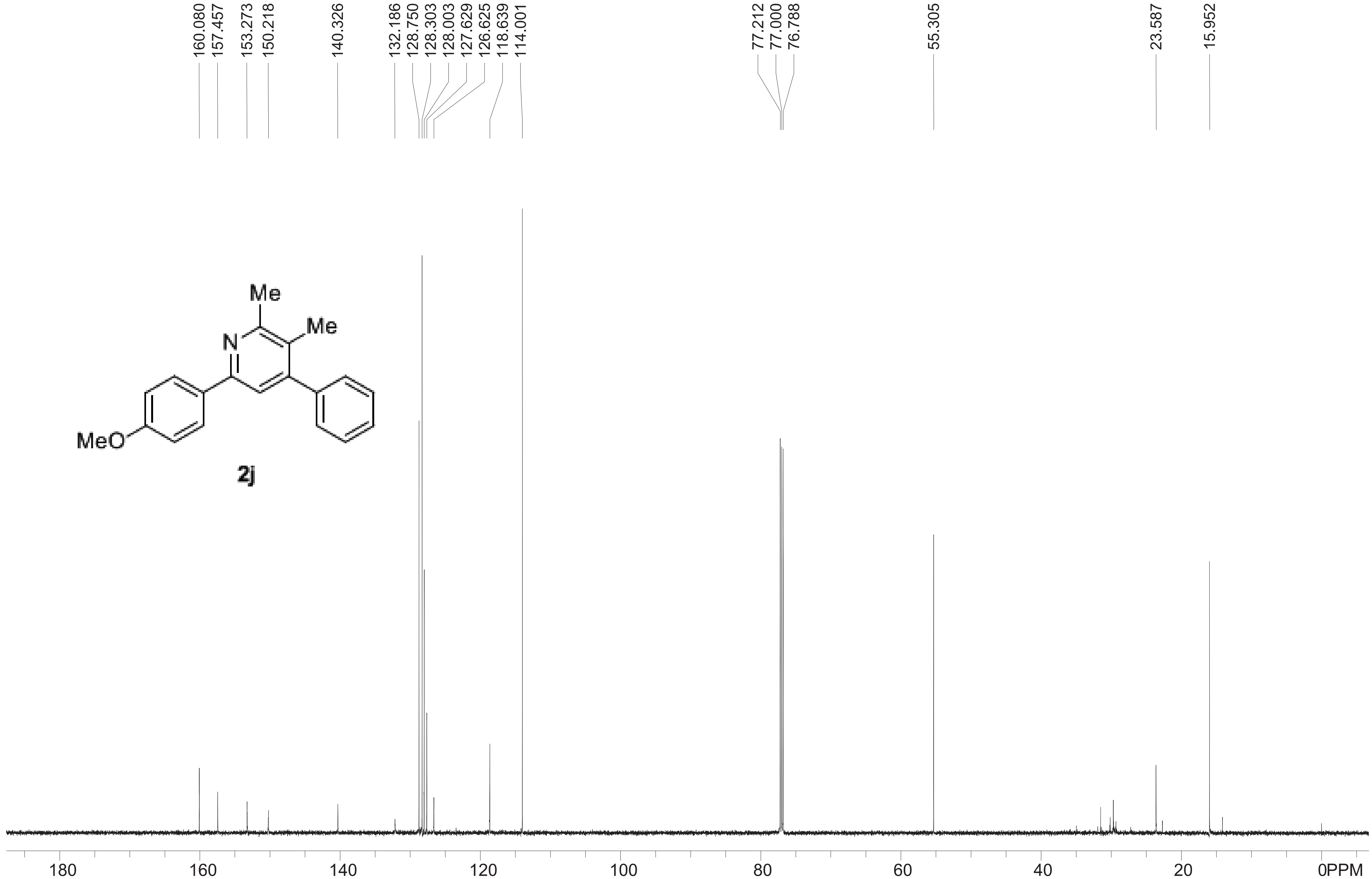
158.060
151.585
142.470
142.274
140.772
127.698
127.632
127.559
127.414
127.326
127.173
126.305
125.066
124.789
120.895
77.321
77.000
76.687
23.823
16.422
180
160
140
120
100
80
60
40
20
0
PPM


8.009
7.996
7.482
7.470
7.464
7.452
7.438
7.429
7.416
7.391
7.379
7.357
7.344
7.260
2.682
2.231
Me
Me
N
2l
9.10
2.00
3.07
3.08
8
6
4
2
0
PPM



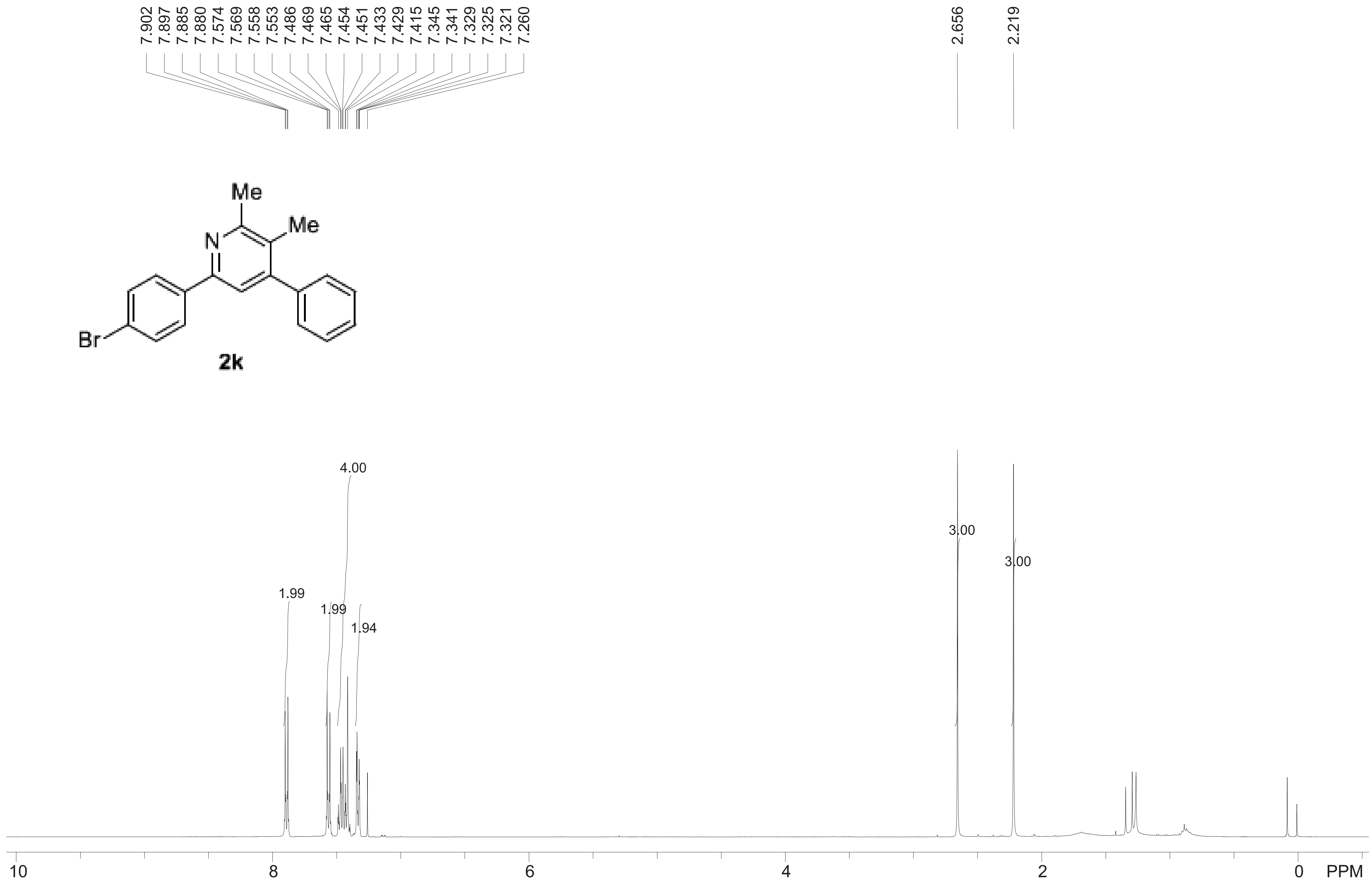
157.662
153.603
150.298
140.195
139.469
128.757
128.625
128.450
128.340
127.702
127.431
126.808
119.408
77.205
77.000
76.788
23.594
16.033
Me
Me
N
2i
180
160
140
120
100
80
60
40
20
PPM

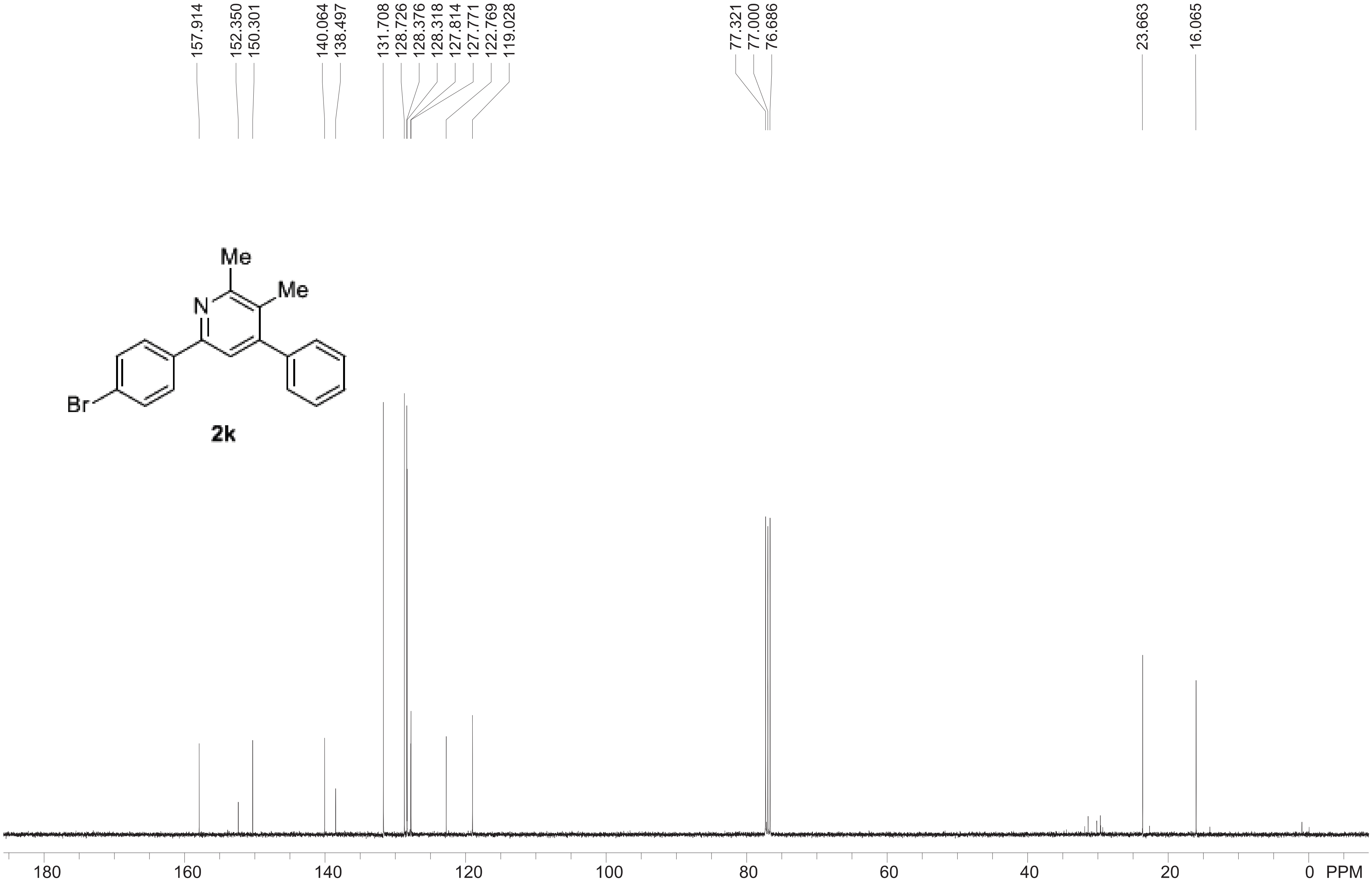

7.967 7.963 7.956 7.952 7.475 7.464 7.451 7.421 7.409 7.389 7.351 7.349 7.337 7.260 6.984 6.981 6.973 6.970
3.852
2.661
2.209
Me
Me
N
MeO
2j
2.00
2.04
2.03
2.03
2.03
3.16
3.13
2.98
8
6
4
2
0
PPM

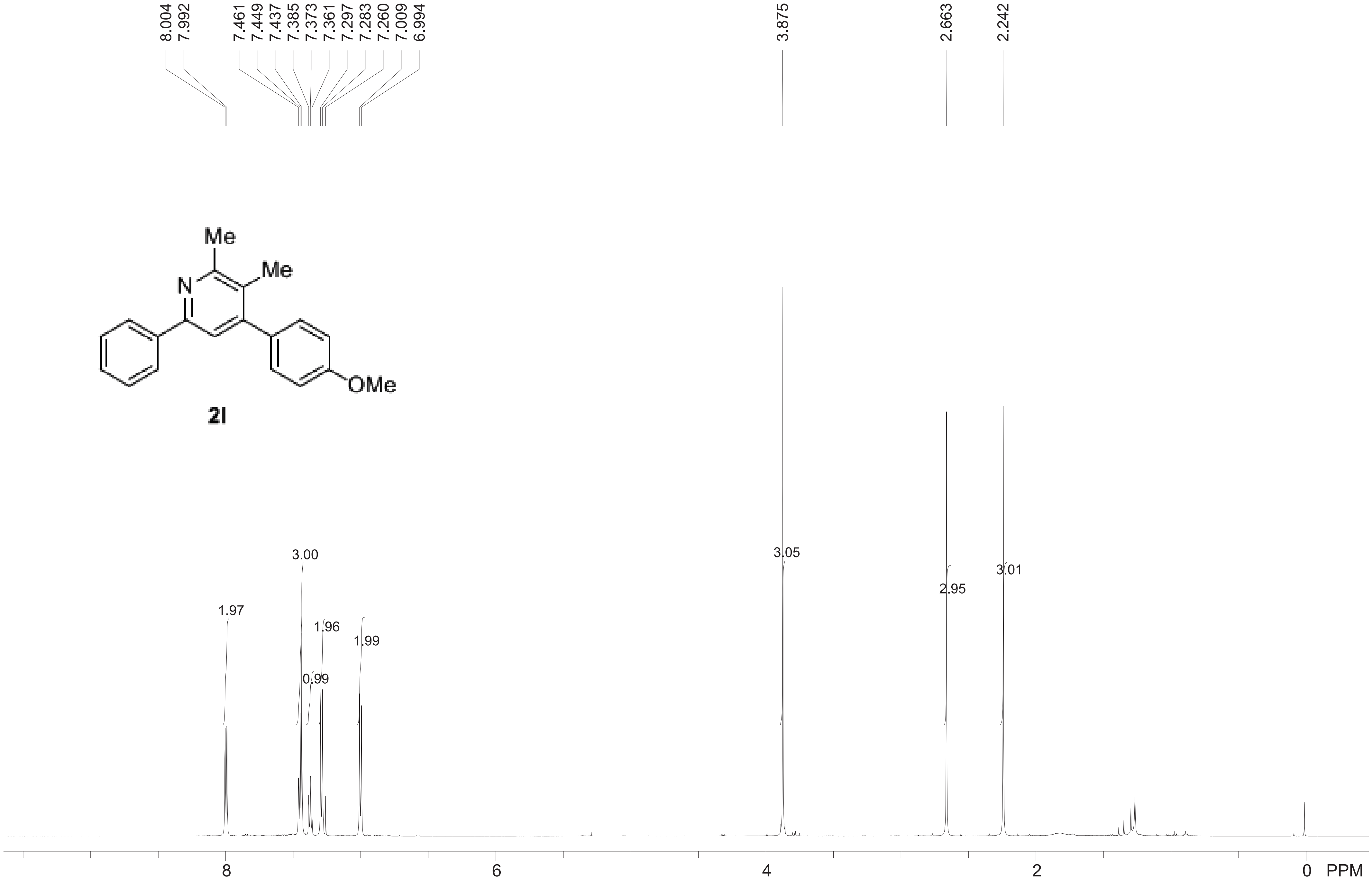


160.080
157.457
153.273
150.218
140.326
132.186
128.750
128.303
128.003
127.629
126.625
118.639
114.001
77.212
77.000
76.788
55.305
23.587
15.952
Me
Me
N
MeO
180
160
140
120
100
80
60
40
20
0PPM

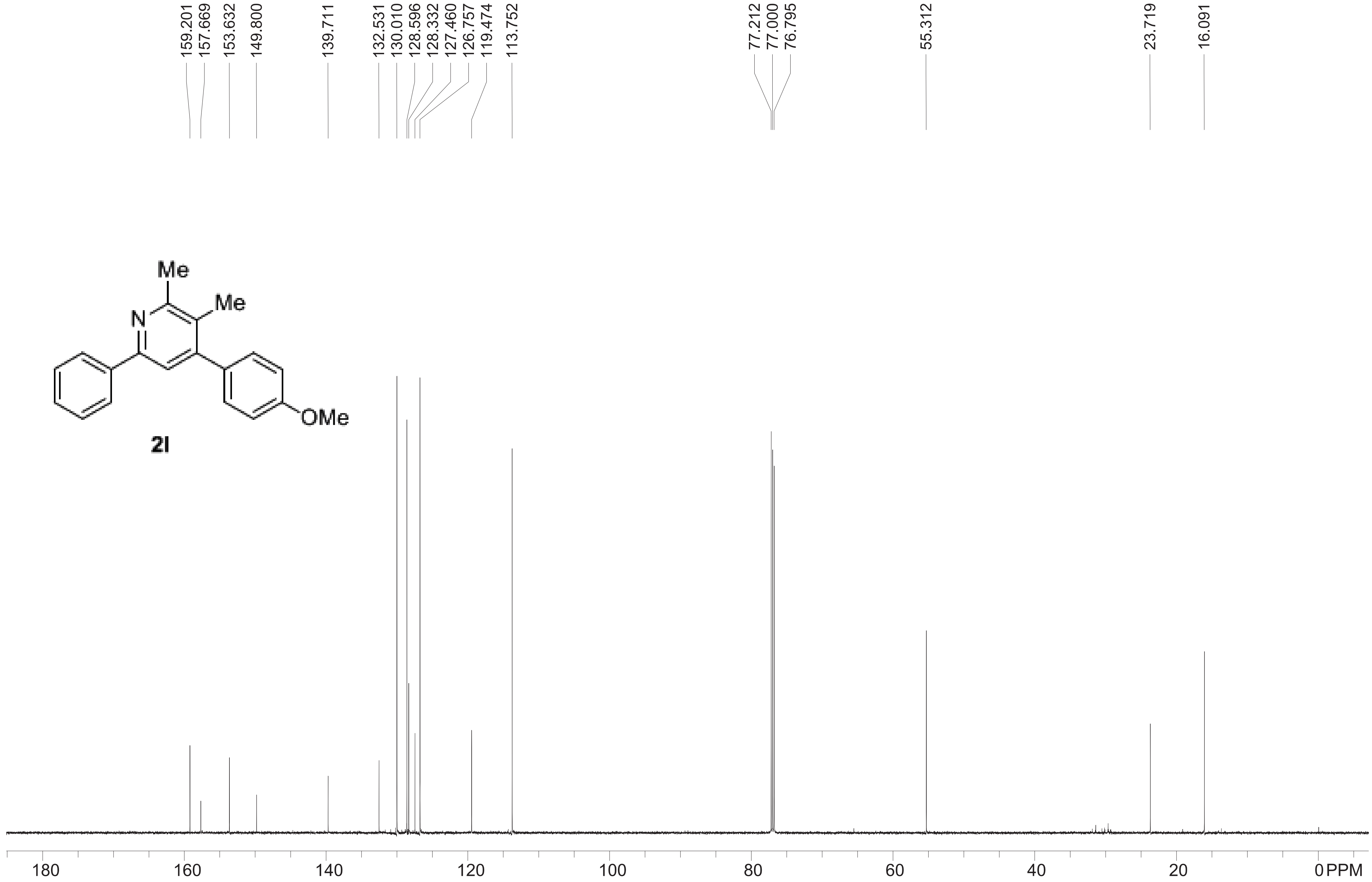
2j

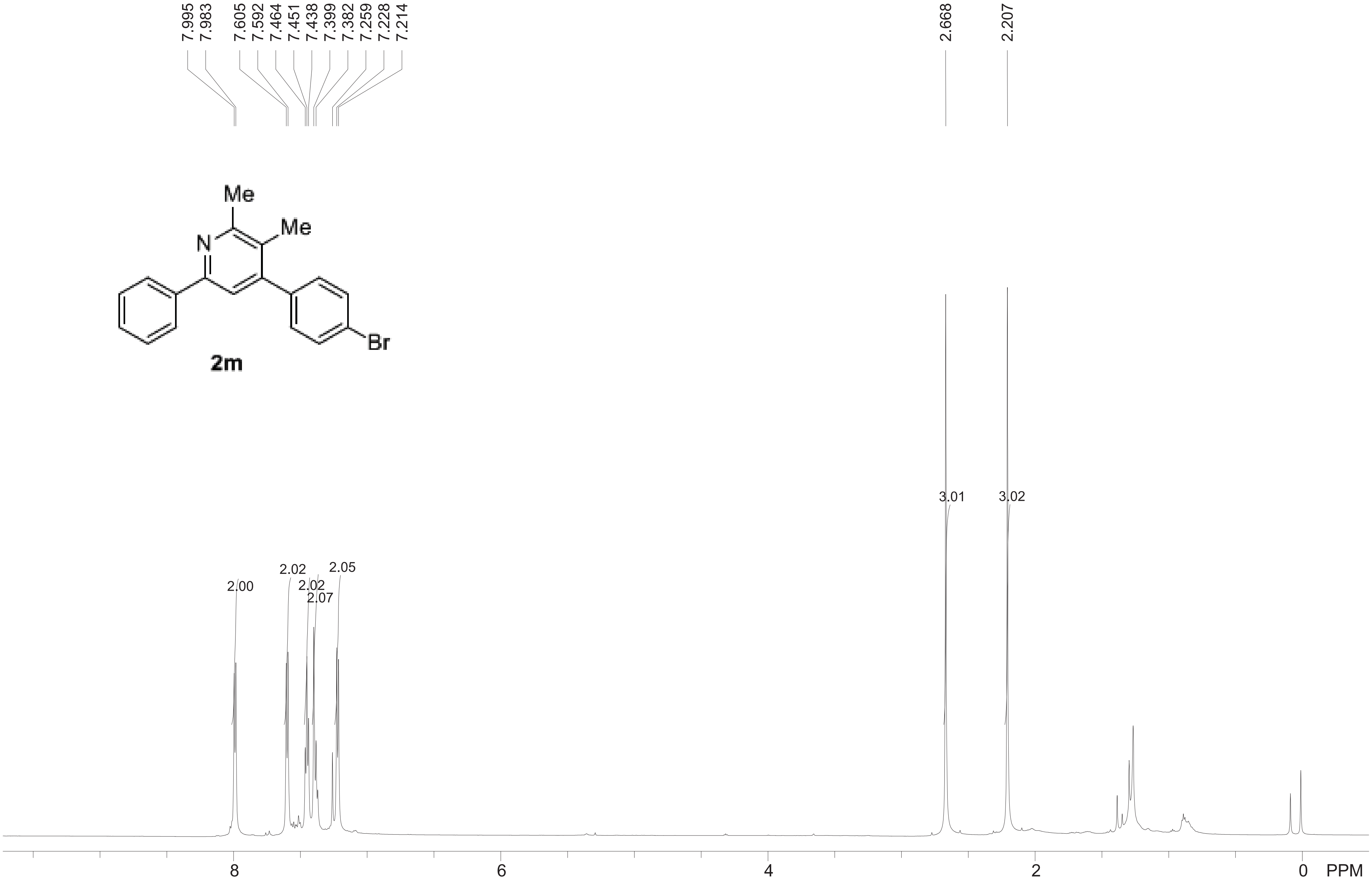

7.902
7.897
7.885
7.880
7.574
7.569
7.558
7.553
7.486
7.469
7.465
7.454
7.451
7.433
7.429
7.415
7.345
7.341
7.329
7.325
7.321
7.260
2.656
2.219
Me
Me
N
Br
2k
4.00
1.99
1.99
1.94
3.00
3.00
10
8
6
4
2
0
PPM


157.914
152.350
150.301
140.064
138.497
131.708
128.726
128.376
128.318
127.814
127.771
122.769
119.028
77.321
77.000
76.686
23.663
16.065
Me
Me
N
Br
2k
180
160
140
120
100
80
60
40
20
0 PPM


8.004
7.992
7.461
7.449
7.437
7.385
7.373
7.361
7.297
7.283
7.260
7.009
6.994
3.875
2.663
2.242
Me
Me
N
OMe
1.97
3.00
0.99
1.96
1.99
3.05
2.95
3.01
8
6
4
2
0 PPM

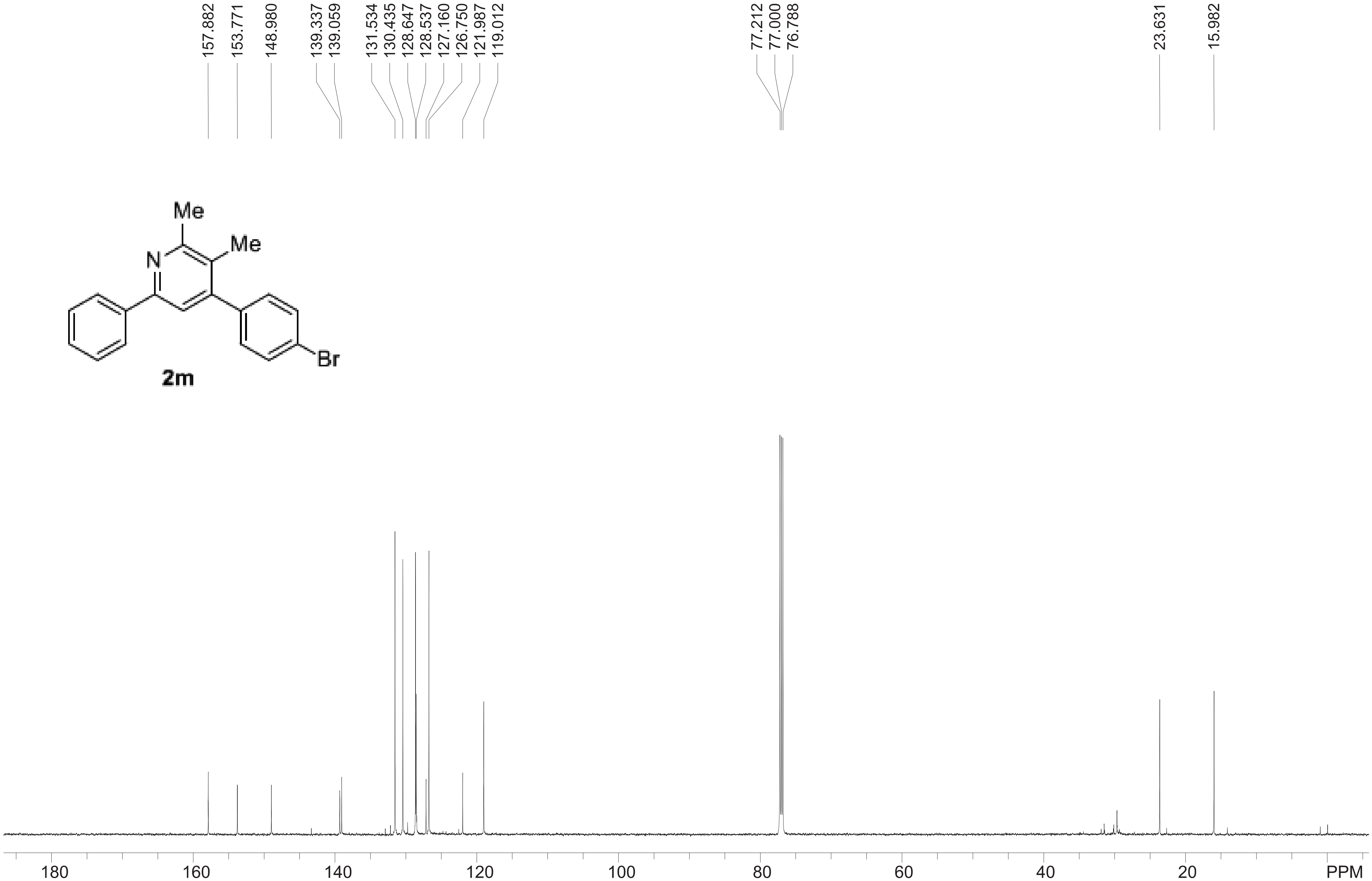
2l


159.201
157.669
153.632
149.800
139.711
132.531
130.010
128.596
128.332
127.460
126.757
119.474
113.752
77.212
77.000
76.795
55.312
23.719
16.091
Me
Me
N
OMe
2l
180
160
140
120
100
80
60
40
20
0 PPM

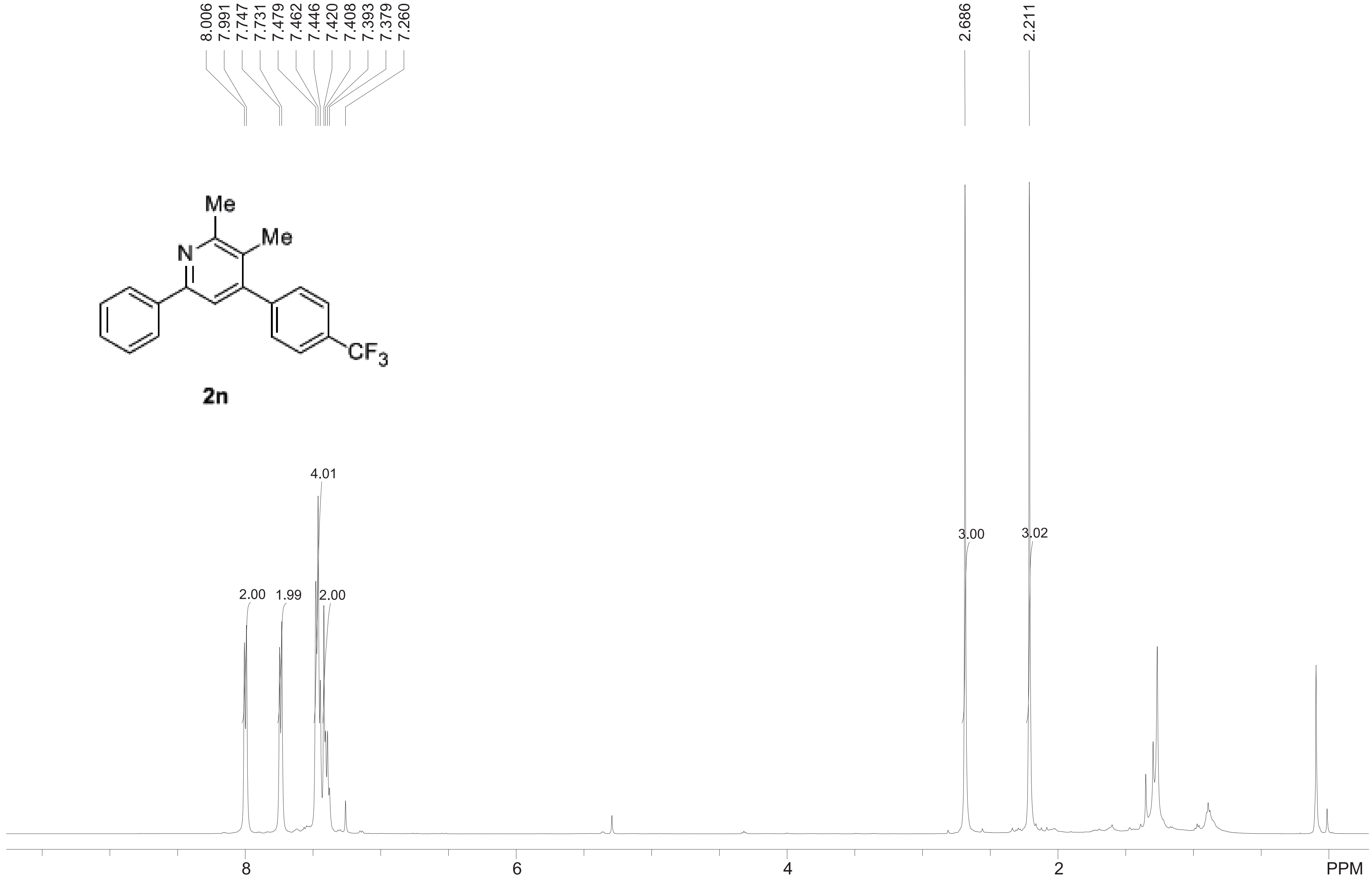

Me
Me
N
Br

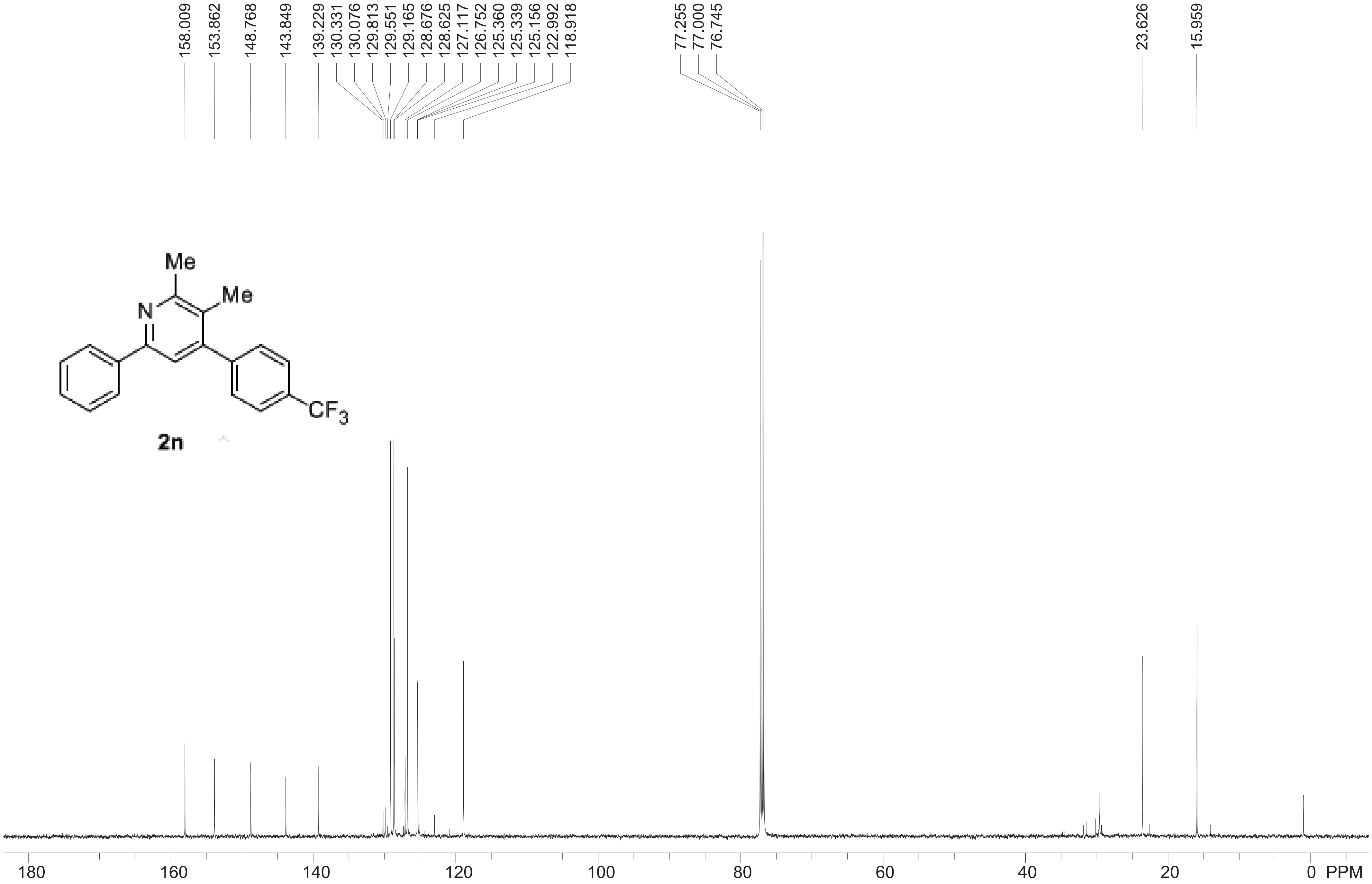
2m

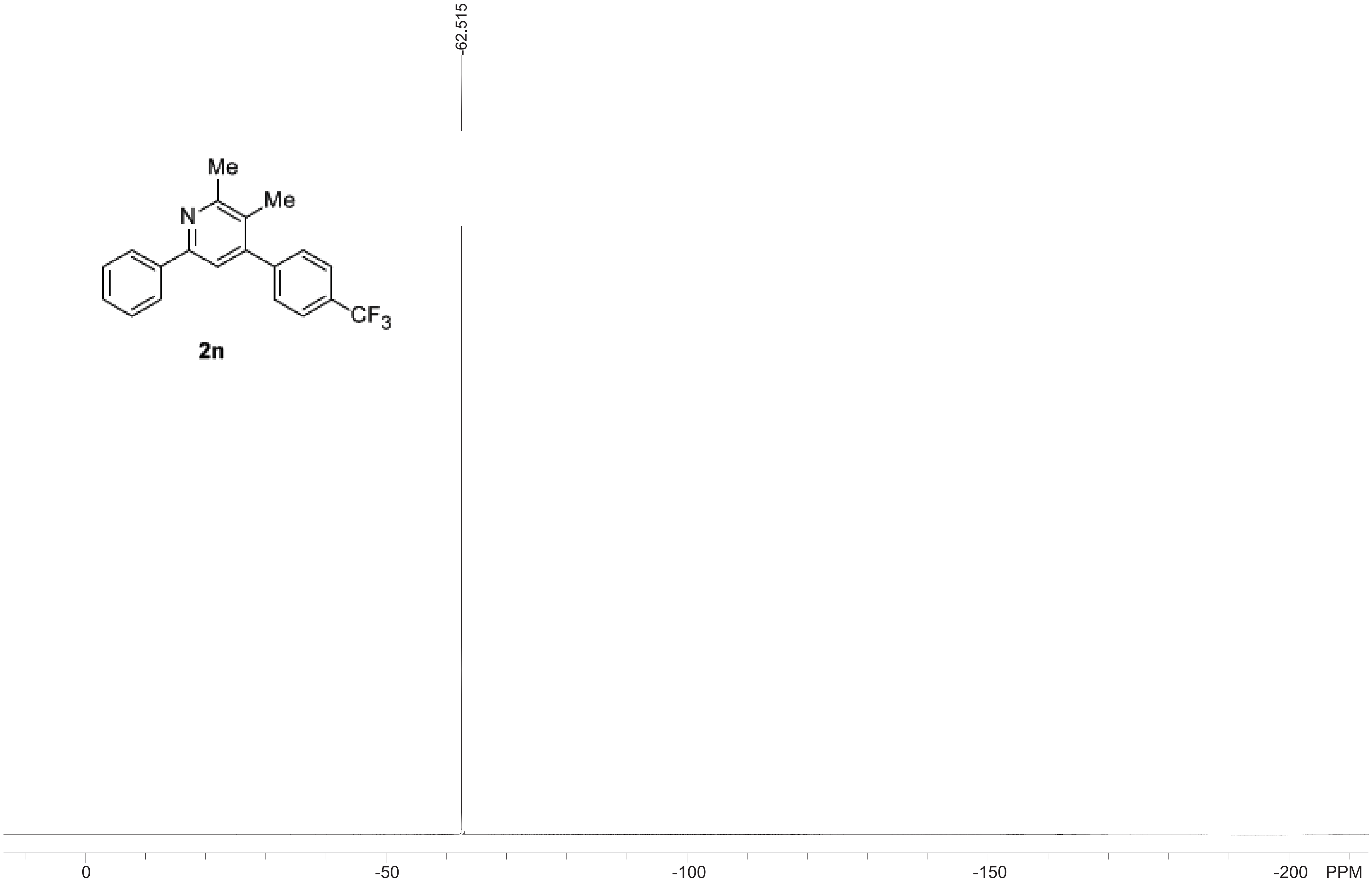
7.995
7.983
7.605
7.592
7.464
7.451
7.438
7.399
7.382
7.259
7.228
7.214
2.668
2.207
3.01
3.02
2.00
2.02
2.02
2.07
2.05
8
6
4
2
0
PPM

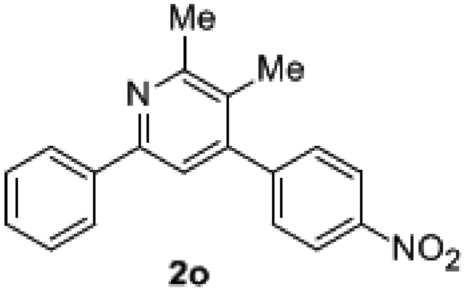

157.882
153.771
148.980
139.337
139.059
131.534
130.435
128.647
128.537
127.160
126.750
121.987
119.012
77.212
77.000
76.788
23.631
15.982
Me
Me
N
Br
180
160
140
120
100
80
60
40
20
PPM

2m


8.006
7.991
7.747
7.731
7.479
7.462
7.446
7.420
7.408
7.393
7.379
7.260
2.686
2.211
Me
Me
N
CF3
2n
4.01
2.00
1.99
2.00
3.00
3.02
8
6
4
2
PPM

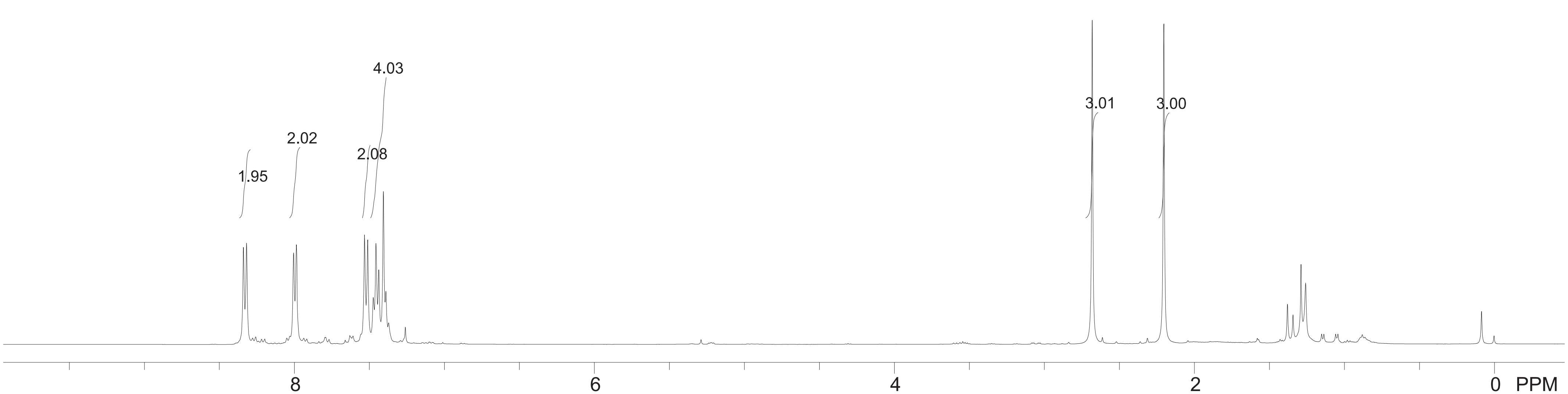

158.009
153.862
148.768
143.849
139.229
130.331
130.076
129.813
129.551
129.165
128.676
128.625
127.117
126.752
125.360
125.339
125.156
122.992
118.918
77.255
77.000
76.745
23.626
15.959
Me
Me
N
CF3
2n
180
160
140
120
100
80
60
40
20
0 PPM


-62.515
Me
Me
N
CF3
2n
0
-50
-100
-150
-200
PPM



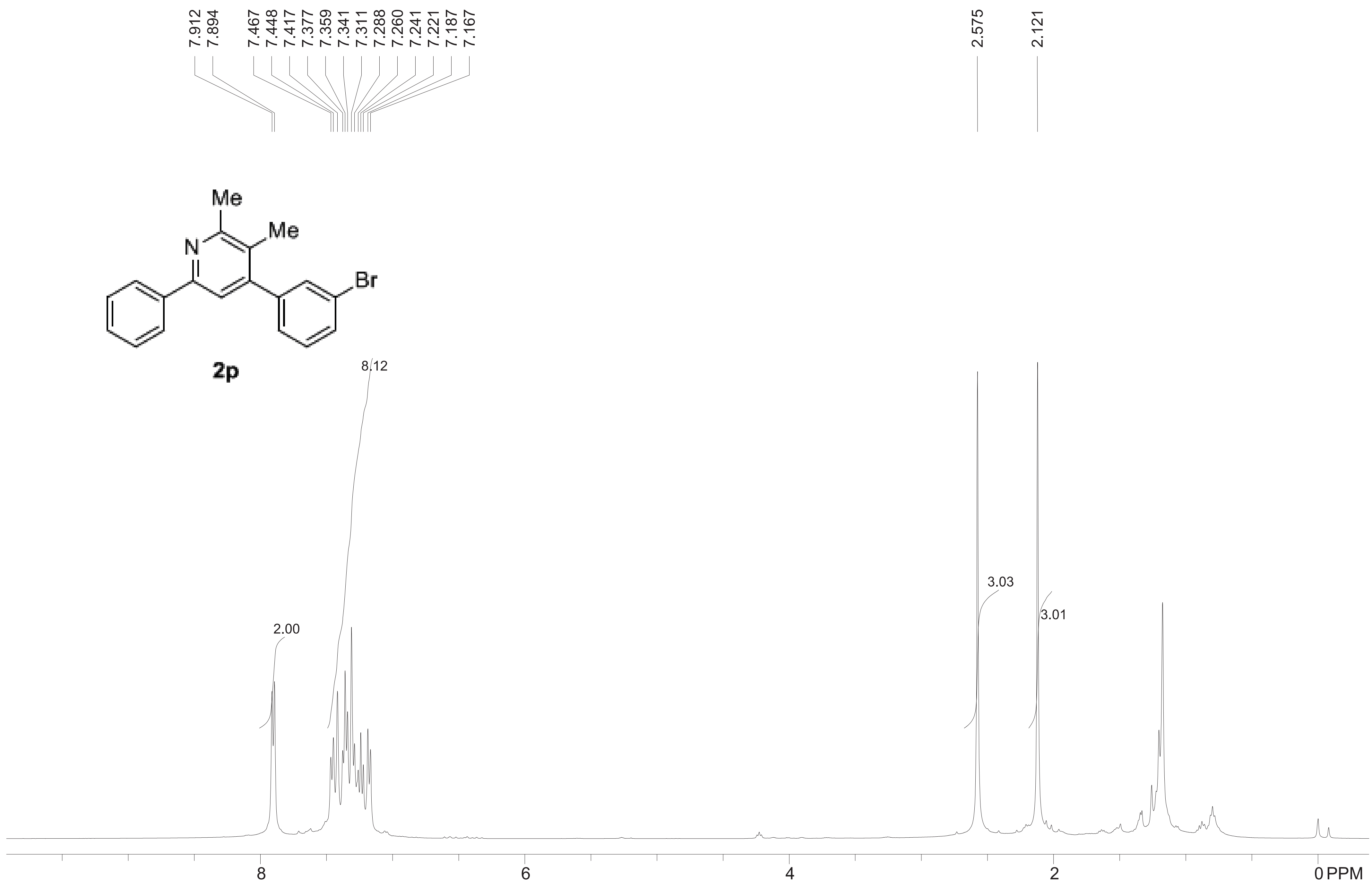
Me
Me
N
NO2

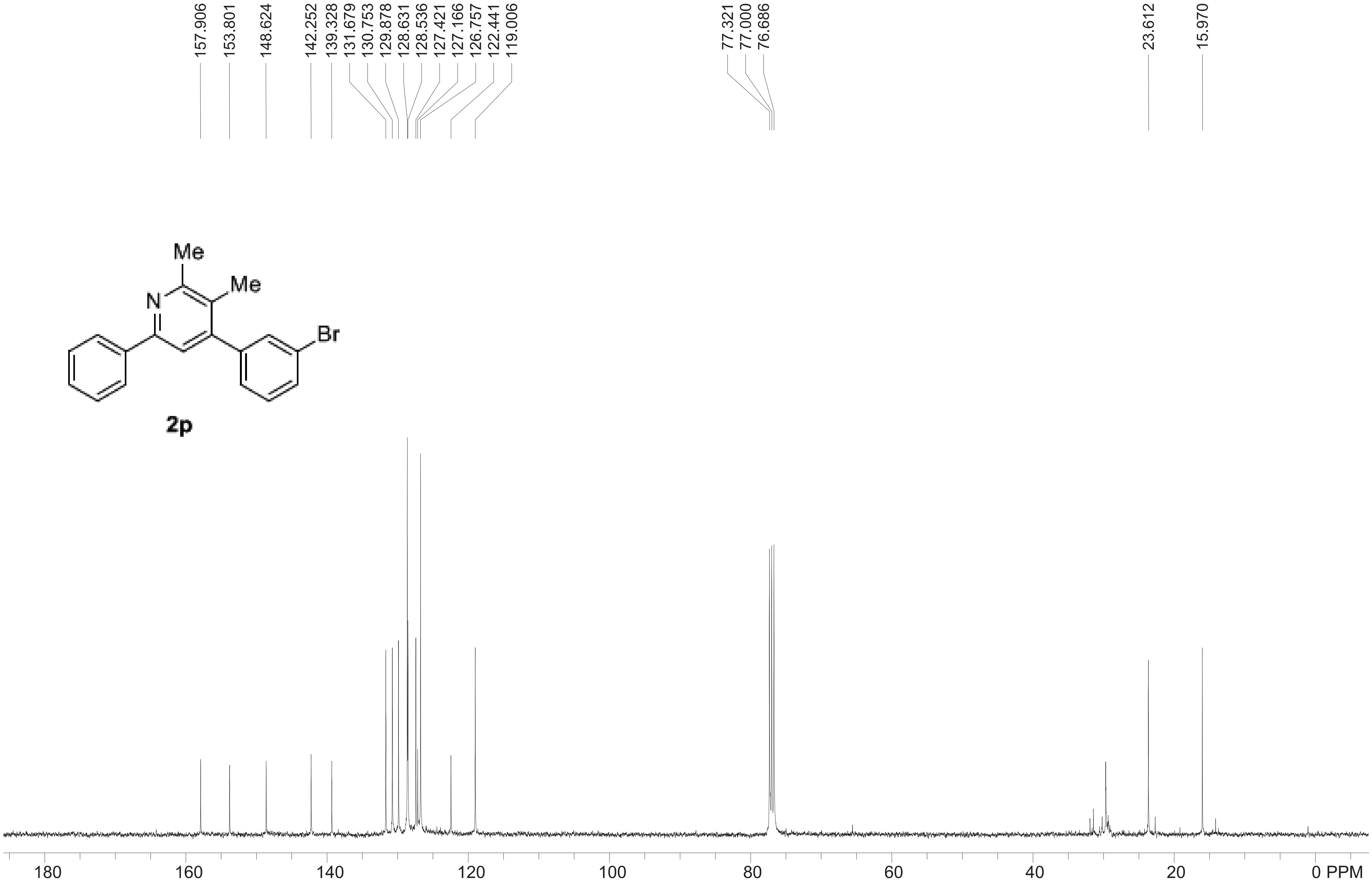
2o



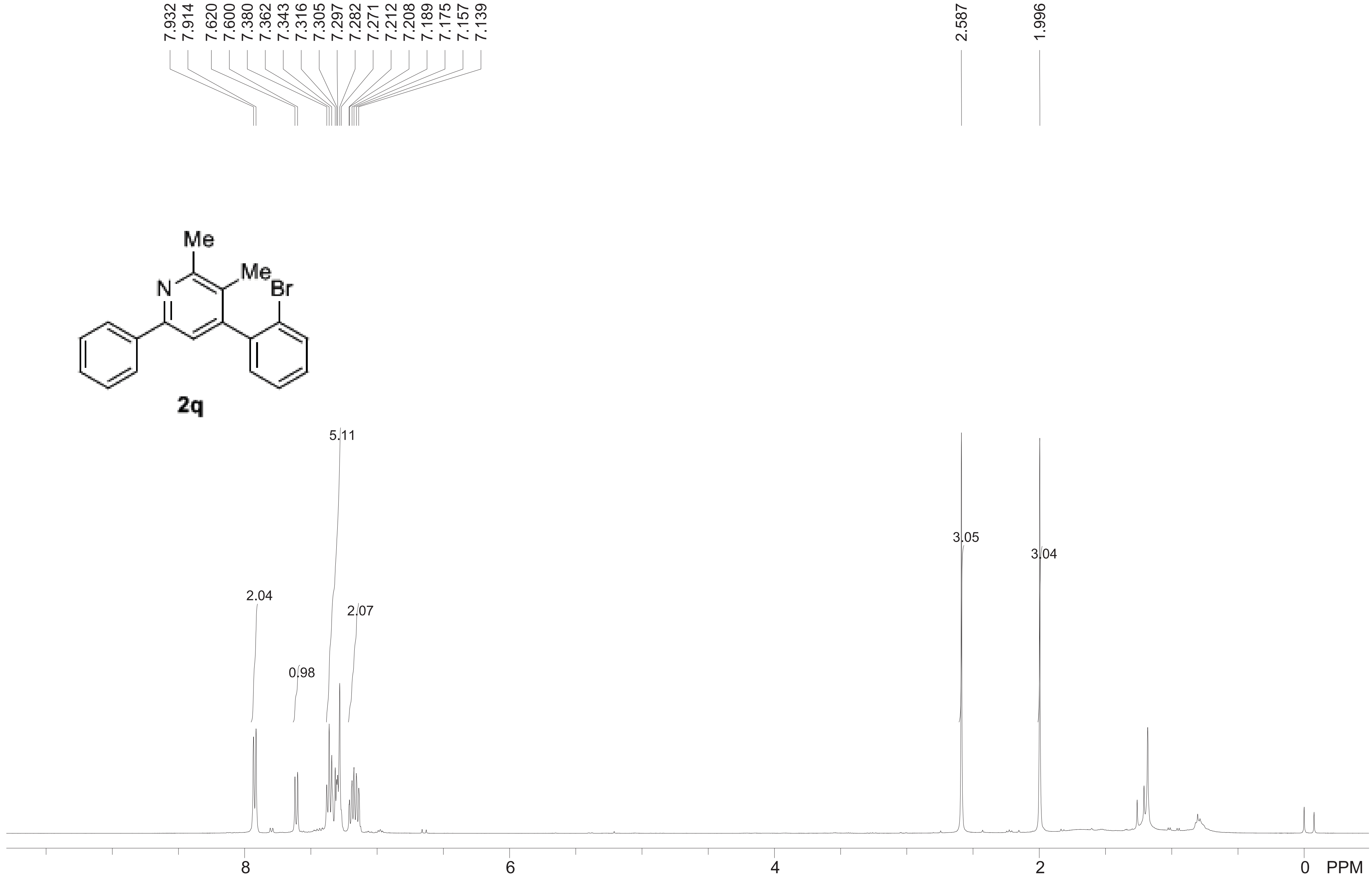
8.339
8.318
8.004
7.986
7.532
7.511
7.474
7.456
7.437
7.407
7.390
7.260
2.682
2.205
1.381
1.290
1.260
4.03
2.02
1.95
2.08
3.01
3.00
8
6
4
2
0 PPM

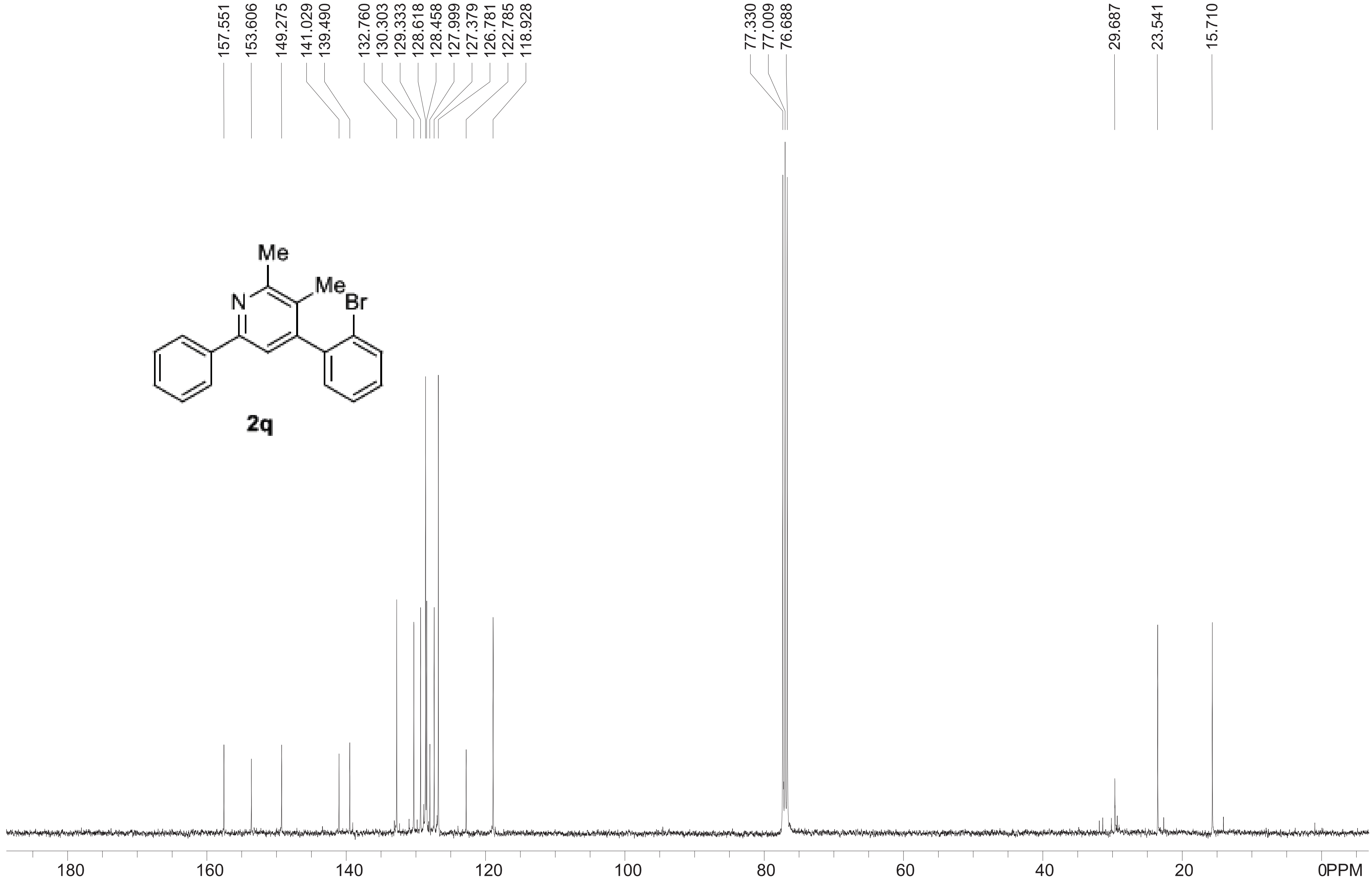

158.220
153.998
147.859
147.407
146.875
139.058
129.791
128.726
128.690
126.808
126.714
123.637
118.481
77.321
77.000
76.679
23.634
15.970
Me
Me
N
NO2
2o
180
160
140
120
100
80
60
40
20
0
PPM

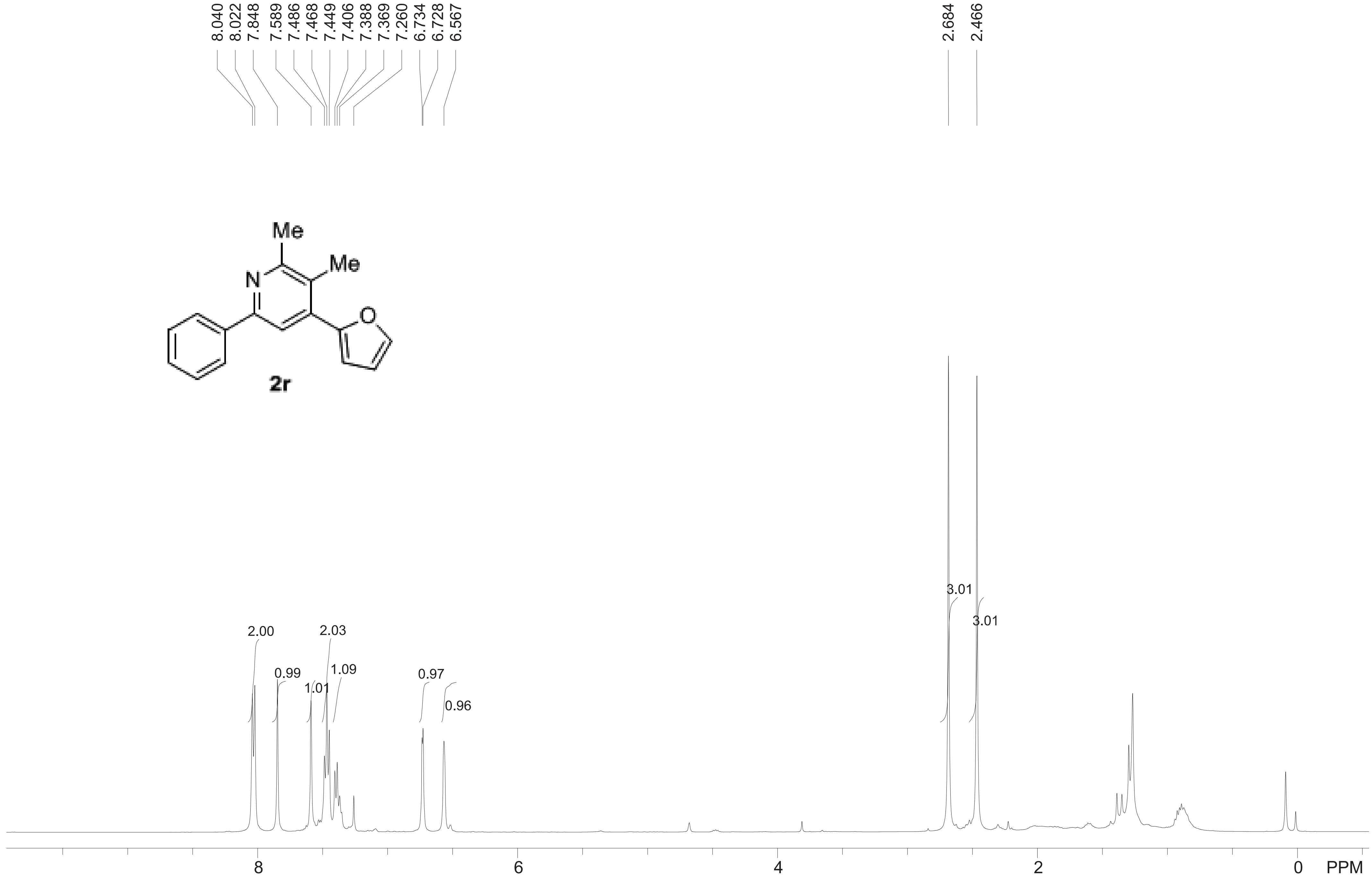

7.912
7.894
7.467
7.448
7.417
7.377
7.359
7.341
7.311
7.288
7.260
7.241
7.221
7.187
7.167
2.575
2.121
Me
Me
N
Br
2p
8.12
2.00
3.03
3.01
8
6
4
2
0 PPM

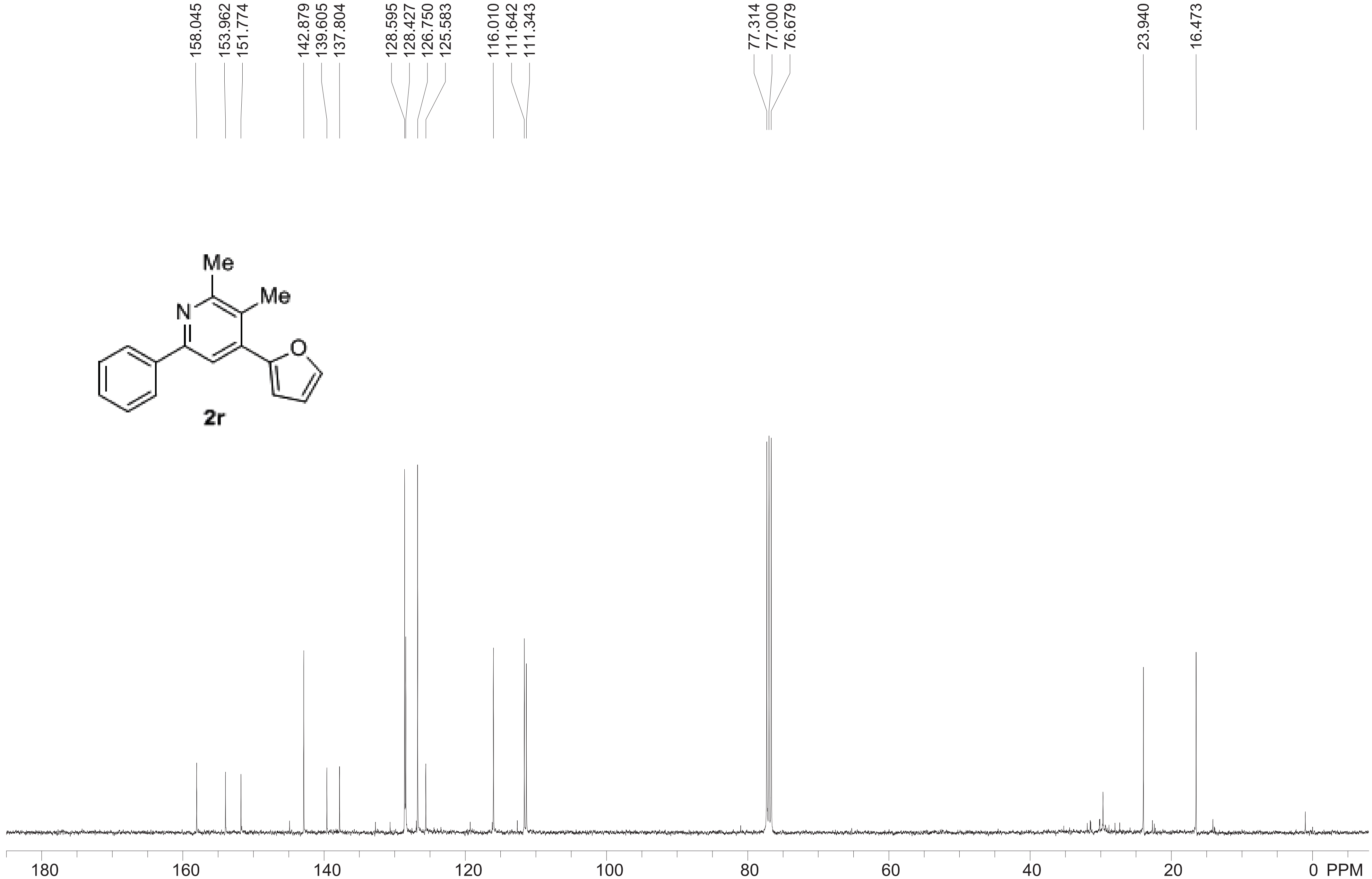


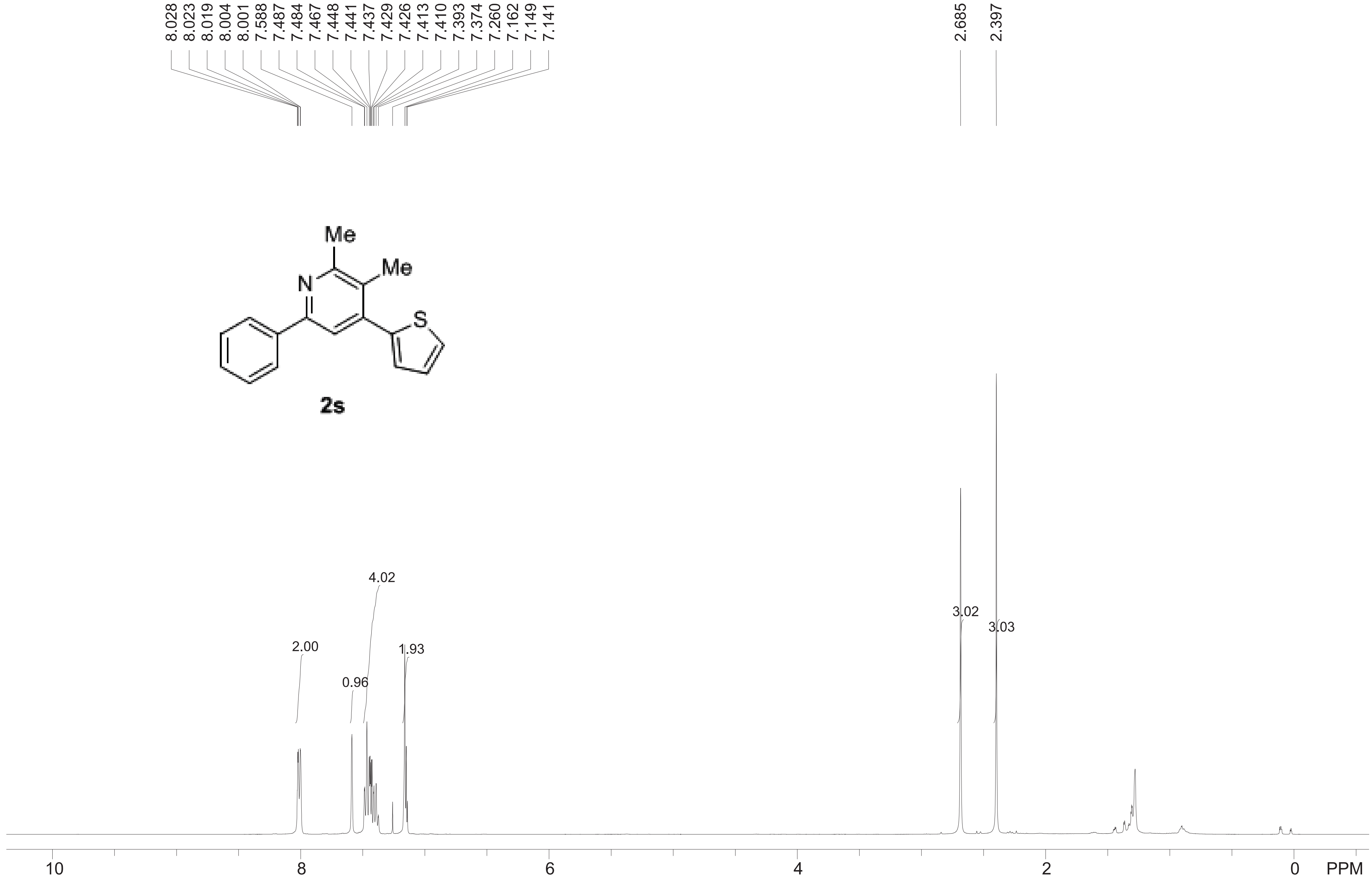
157.906
153.801
148.624
142.252
139.328
131.679
130.753
129.878
128.631
128.536
127.421
127.166
126.757
122.441
119.006
77.321
77.000
76.686
23.612
15.970
Me
Me
N
Br
2p
180
160
140
120
100
80
60
40
20
0 PPM

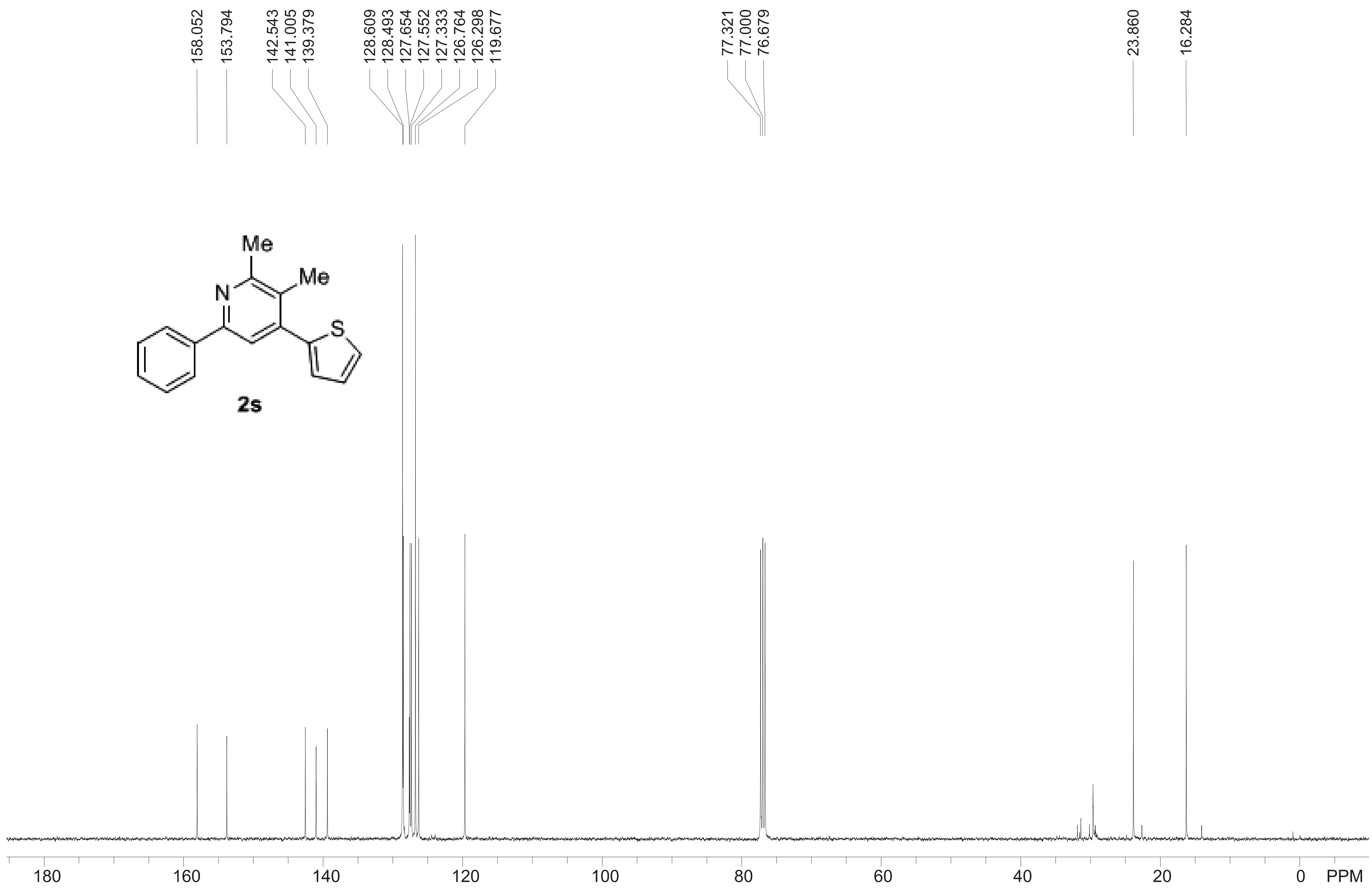

7.932
7.914
7.620
7.600
7.380
7.362
7.343
7.316
7.305
7.297
7.282
7.271
7.212
7.208
7.189
7.175
7.157
7.139
2.587
1.996
Me
Me
Br
N
2q
5.11
3.05
3.04
2.04
2.07
0.98
8
6
4
2
0
PPM


157.551
153.606
149.275
141.029
139.490
132.760
130.303
129.333
128.618
128.458
127.999
127.379
126.781
122.785
118.928
77.330
77.009
76.688
29.687
23.541
15.710
Me
Me
N
Br
2q
180
160
140
120
100
80
60
40
20
0PPM

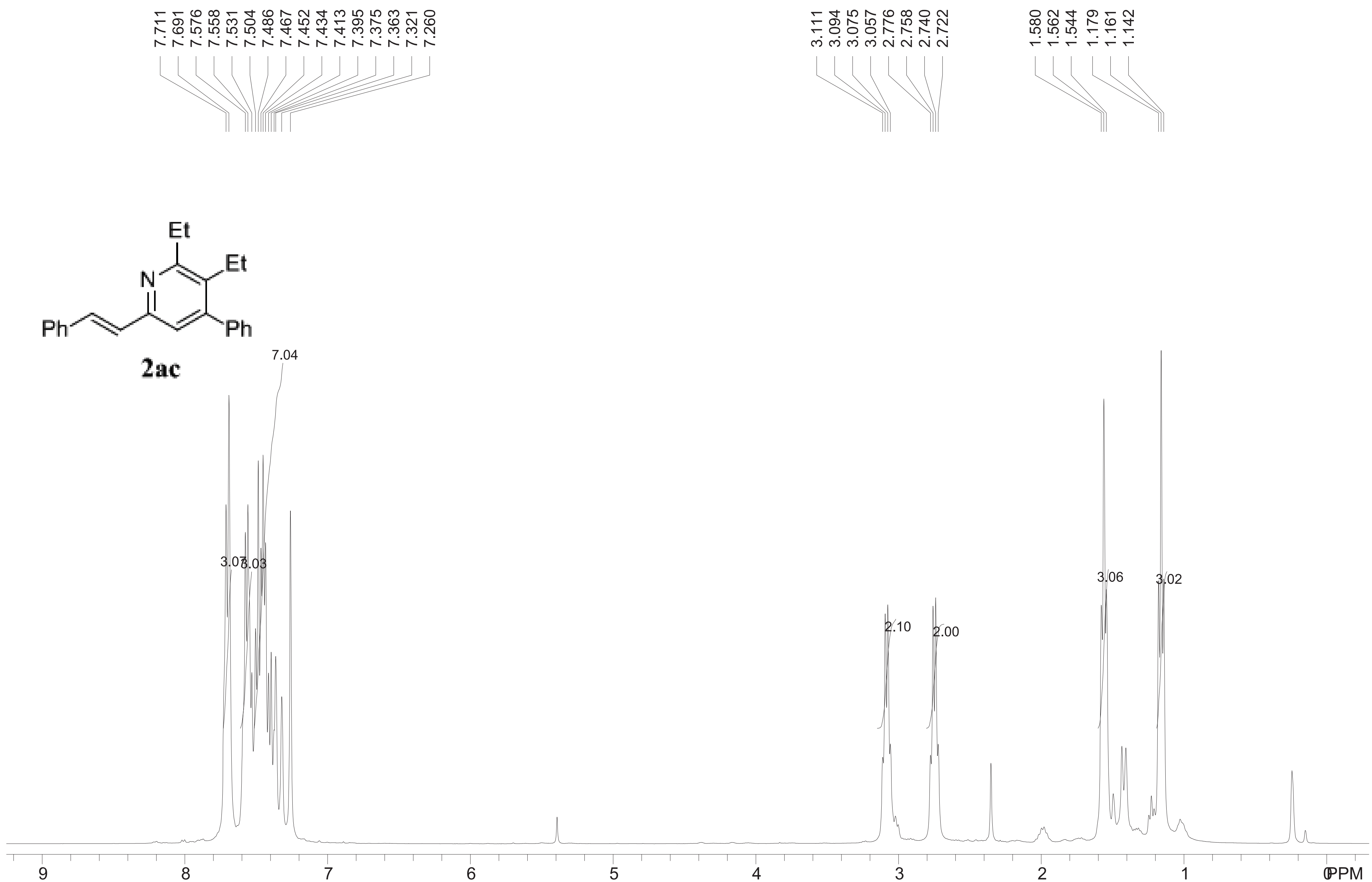

8.040
8.022
7.848
7.589
7.486
7.468
7.449
7.406
7.388
7.369
7.260
6.734
6.728
6.567
2.684
2.466
Me
Me
N
O
2r
2.00
0.99
1.01
2.03
1.09
0.97
0.96
3.01
3.01
8
6
4
2
0
PPM


158.045
153.962
151.774
142.879
139.605
137.804
128.595
128.427
126.750
125.583
116.010
111.642
111.343
77.314
77.000
76.679
23.940
16.473
Me
Me
N
O
2r
180
160
140
120
100
80
60
40
20
0 PPM

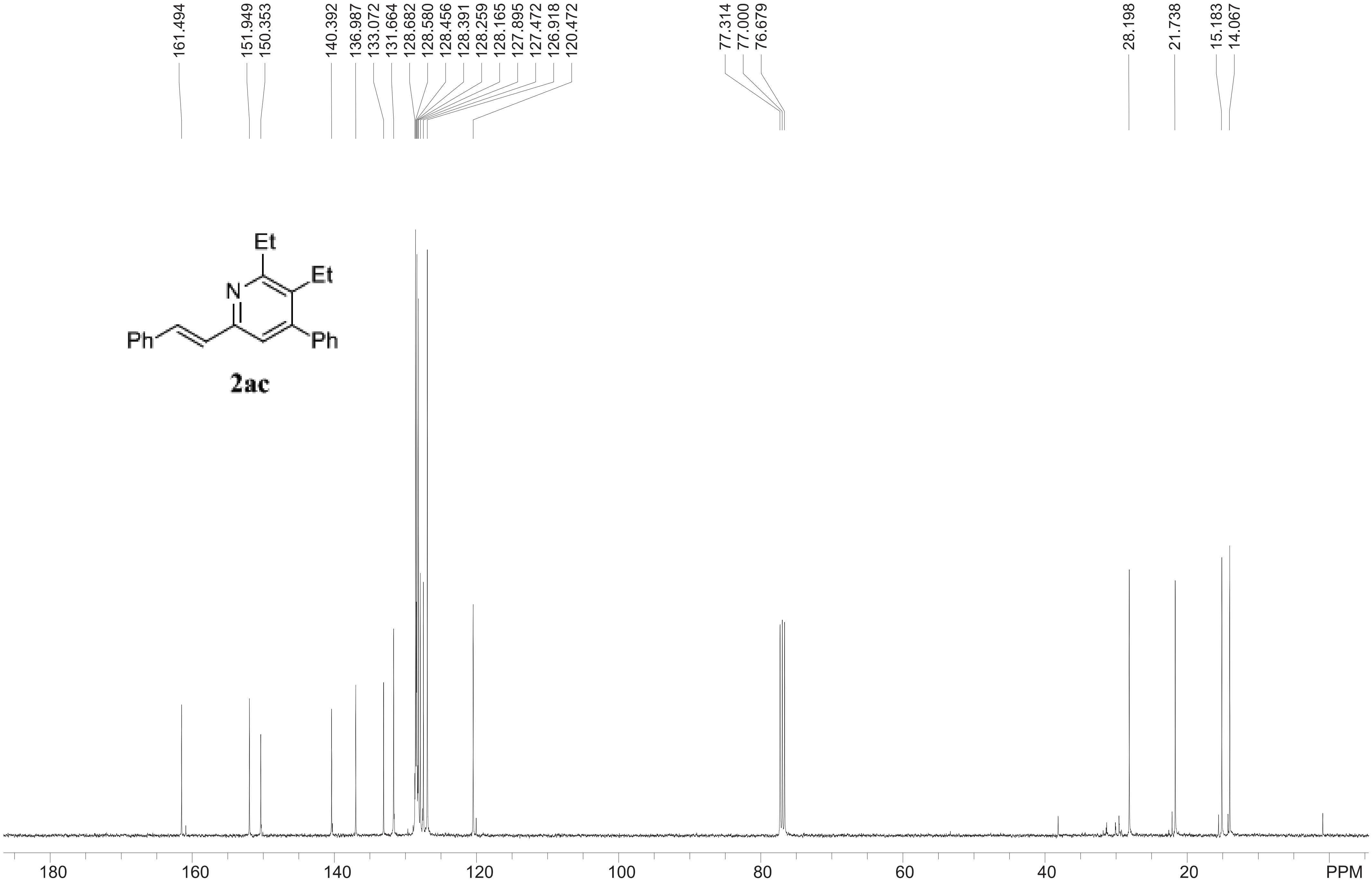

8.028
8.023
8.019
8.004
8.001
7.588
7.487
7.484
7.467
7.448
7.441
7.437
7.429
7.426
7.413
7.410
7.393
7.374
7.260
7.162
7.149
7.141
2.685
2.397
Me
Me
N
S
2s
2.00
0.96
4.02
1.93
3.02
3.03
10
8
6
4
2
0
PPM



158.052
153.794
142.543
141.005
139.379
128.609
128.493
127.654
127.552
127.333
126.764
126.298
119.677
77.321
77.000
76.679
23.860
16.284
Me
Me
N
S
2s
180
160
140
120
100
80
60
40
20
0
PPM


7.711
7.691
7.576
7.558
7.531
7.504
7.486
7.467
7.452
7.434
7.413
7.395
7.375
7.363
7.321
7.260
3.111
3.094
3.075
3.057
2.776
2.758
2.740
2.722
1.580
1.562
1.544
1.179
1.161
1.142
Et
Et
N
Ph
Ph
7.04
3.07
3.03
2.10
2.00
3.06
3.02
9
8
7
6
5
4
3
2
1
0
PPM

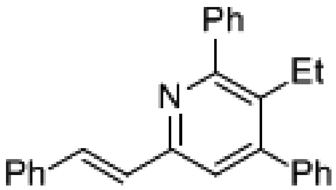
2ac



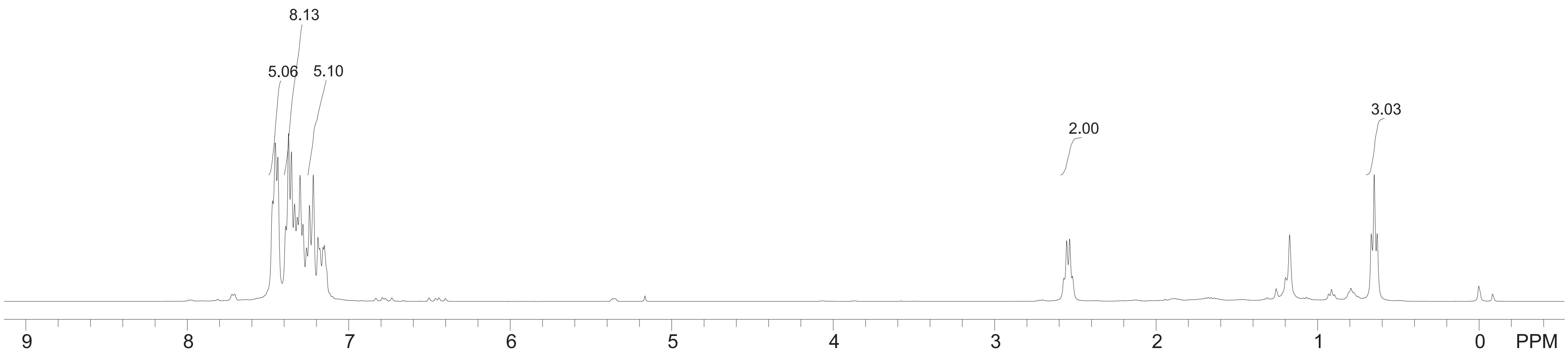
161.494
151.949
150.353
140.392
136.987
133.072
131.664
128.682
128.580
128.456
128.391
128.259
128.165
127.895
127.472
126.918
120.472
77.314
77.000
76.679
28.198
21.738
15.183
14.067
Et
Et
N
Ph
Ph
180
160
140
120
100
80
60
40
20
PPM

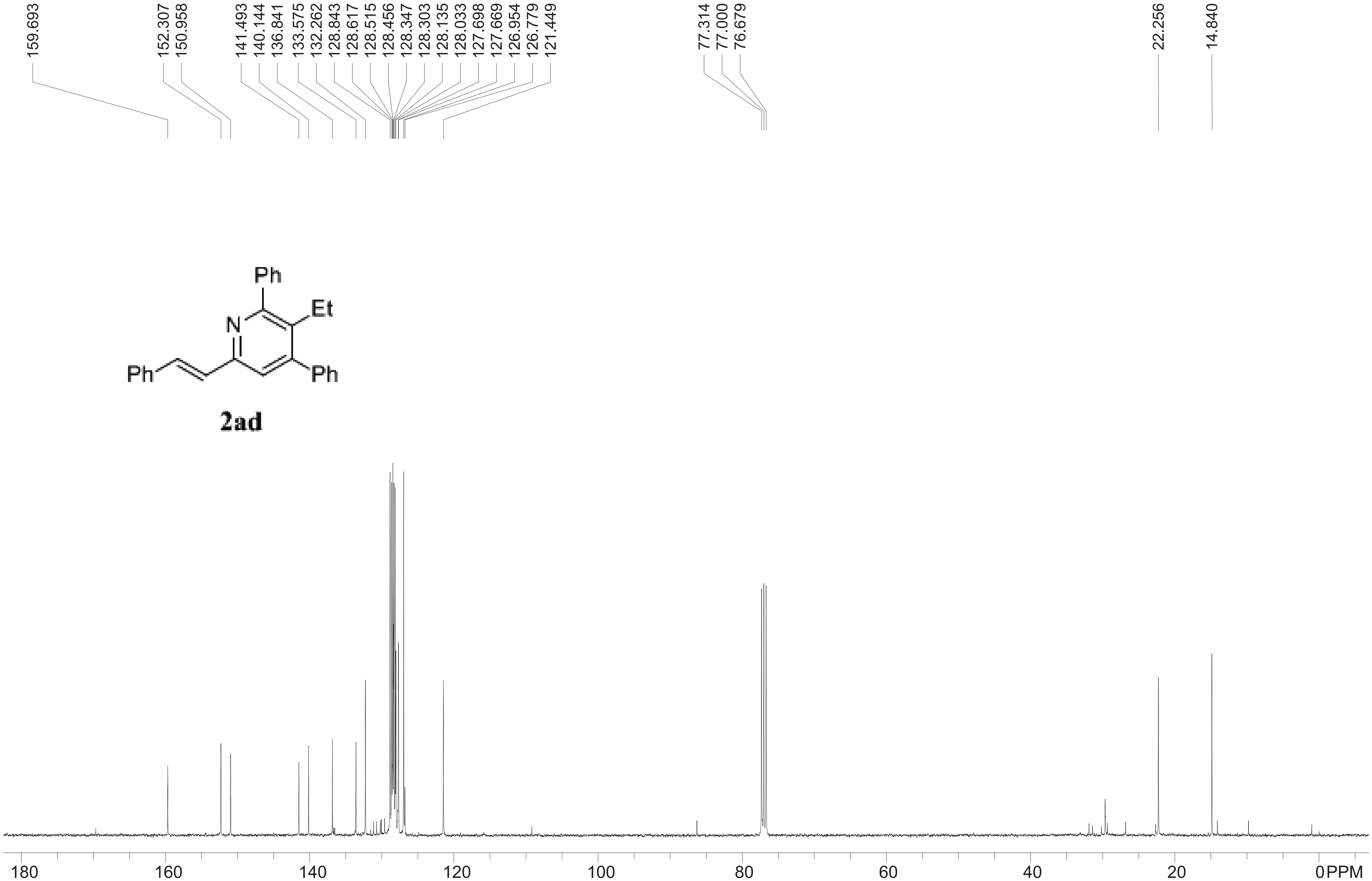
2ac


7.471 7.455 7.439 7.391 7.373 7.355 7.336 7.319 7.301 7.284 7.262 7.243 7.220 7.191 7.179 7.160 7.152
2.570 2.553 2.535 2.517
0.666 0.648 0.630

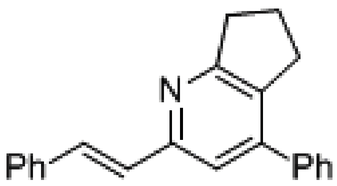


2ad

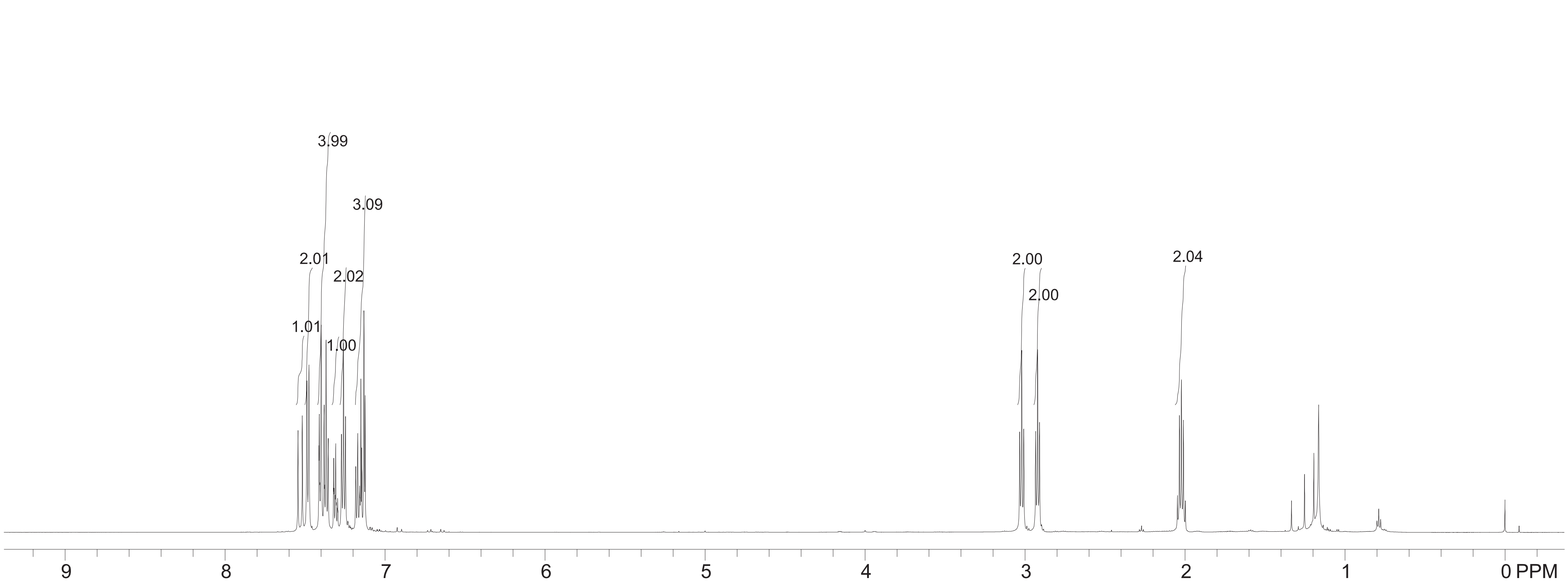

8.13
5.06
5.10
2.00
3.03
9 8 7 6 5 4 3 2 1 0 PPM


159.693
152.307
150.958
141.493
140.144
136.841
133.575
132.262
128.843
128.617
128.515
128.456
128.347
128.303
128.135
128.033
127.698
127.669
126.954
126.779
121.449
77.314
77.000
76.679
22.256
14.840
180
160
140
120
100
80
60
40
20
0PPM
Ph
N
Et
Ph
Ph

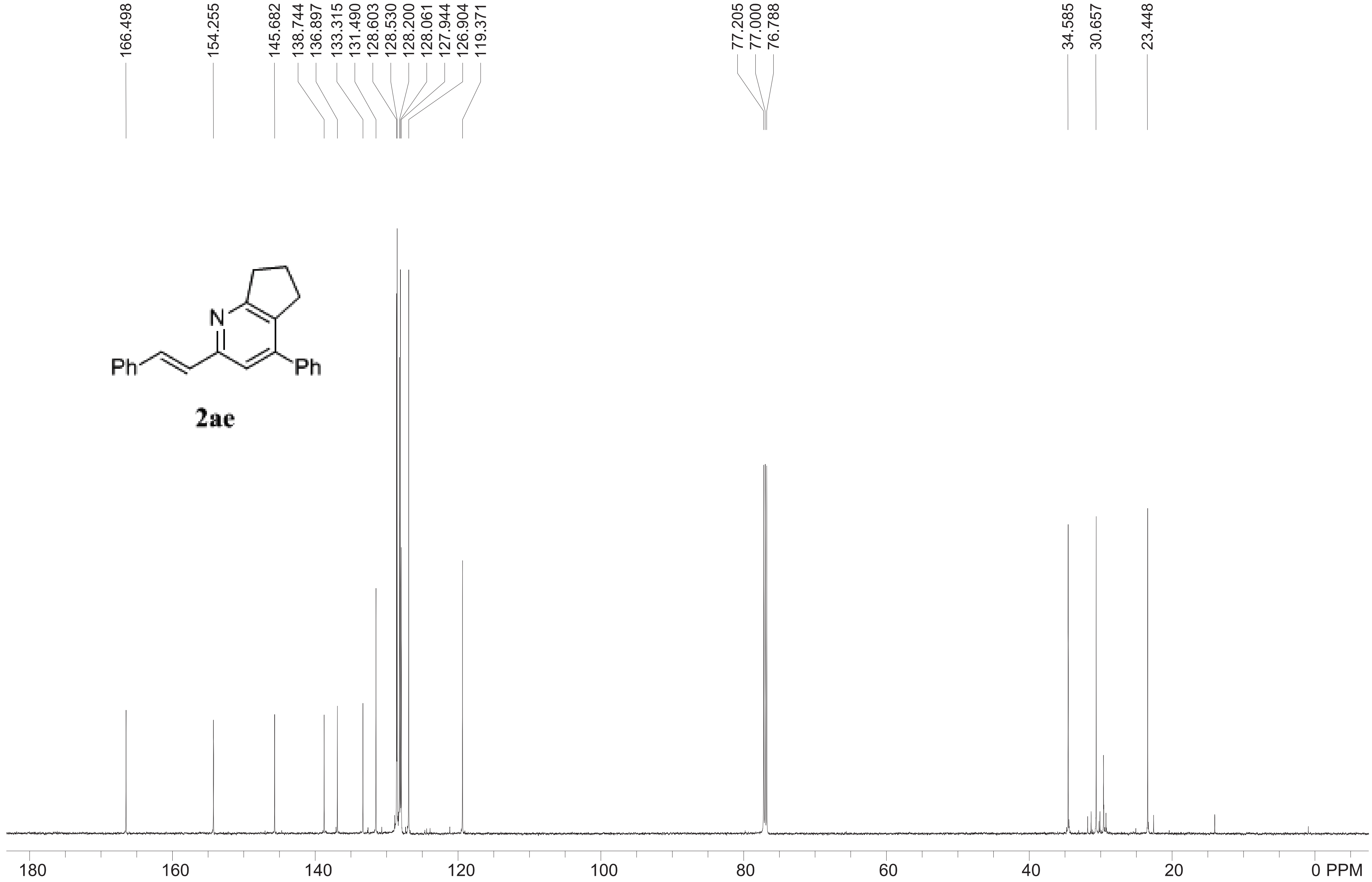
2ad

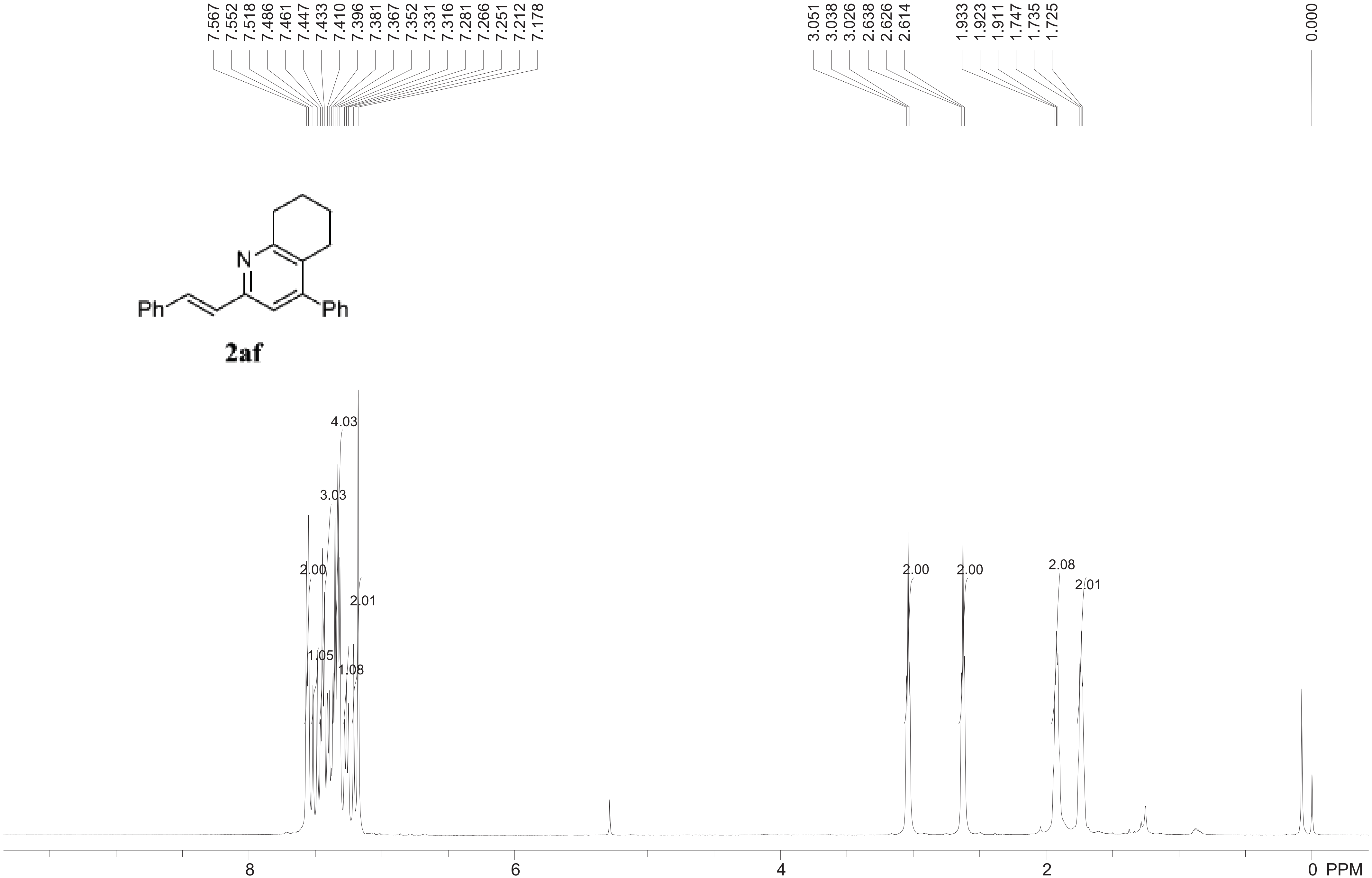


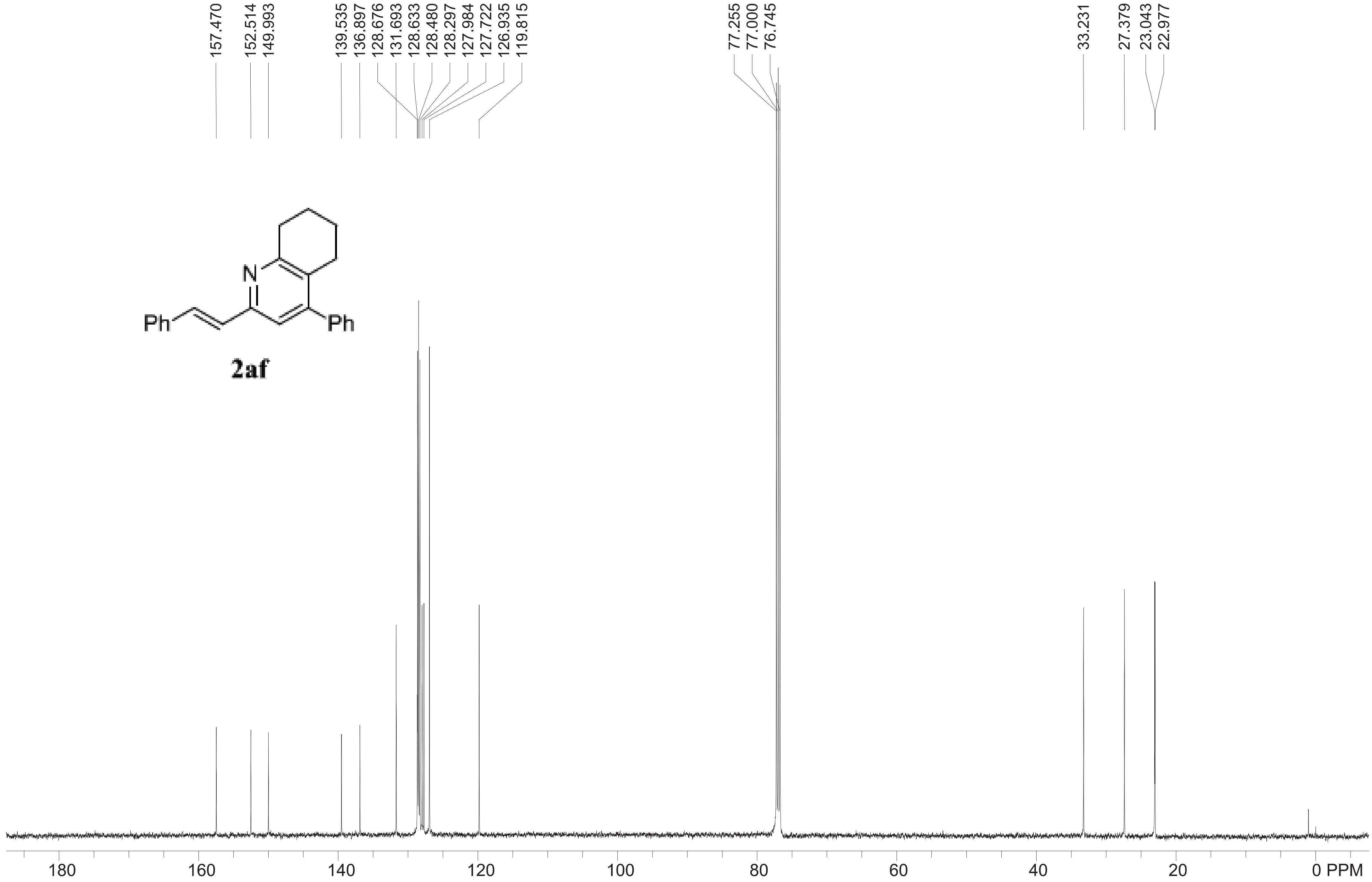
2ae

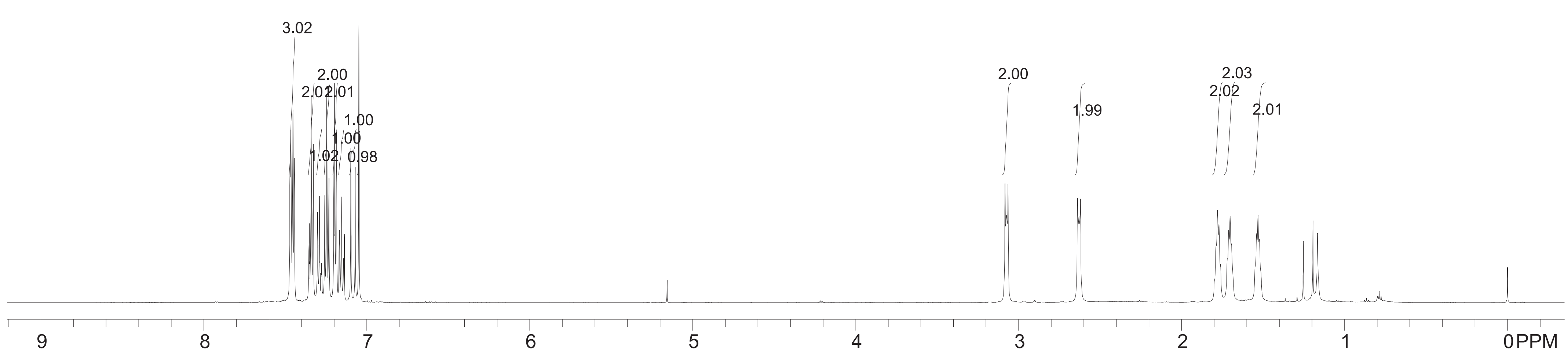


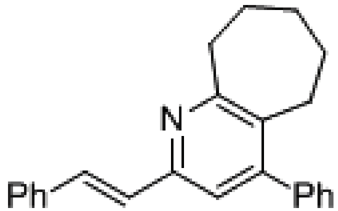
7.544
7.517
7.488
7.476
7.414
7.411
7.408
7.399
7.380
7.377
7.368
7.355
7.323
7.321
7.318
7.312
7.308
7.297
7.272
7.260
7.247
7.183
7.171
7.159
7.157
7.151
7.146
7.132
7.124
3.034
3.021
3.008
2.934
2.922
2.910
2.047
2.035
2.022
2.010
1.998
3.99
3.09
2.01
2.02
1.01
1.00
2.00
2.00
2.04
9
8
7
6
5
4
3
2
1
0 PPM


166.498
154.255
145.682
138.744
136.897
133.315
131.490
128.603
128.530
128.200
128.061
127.944
126.904
119.371
77.205
77.000
76.788
34.585
30.657
23.448
N
Ph
Ph
2ae
180
160
140
120
100
80
60
40
20
0 PPM

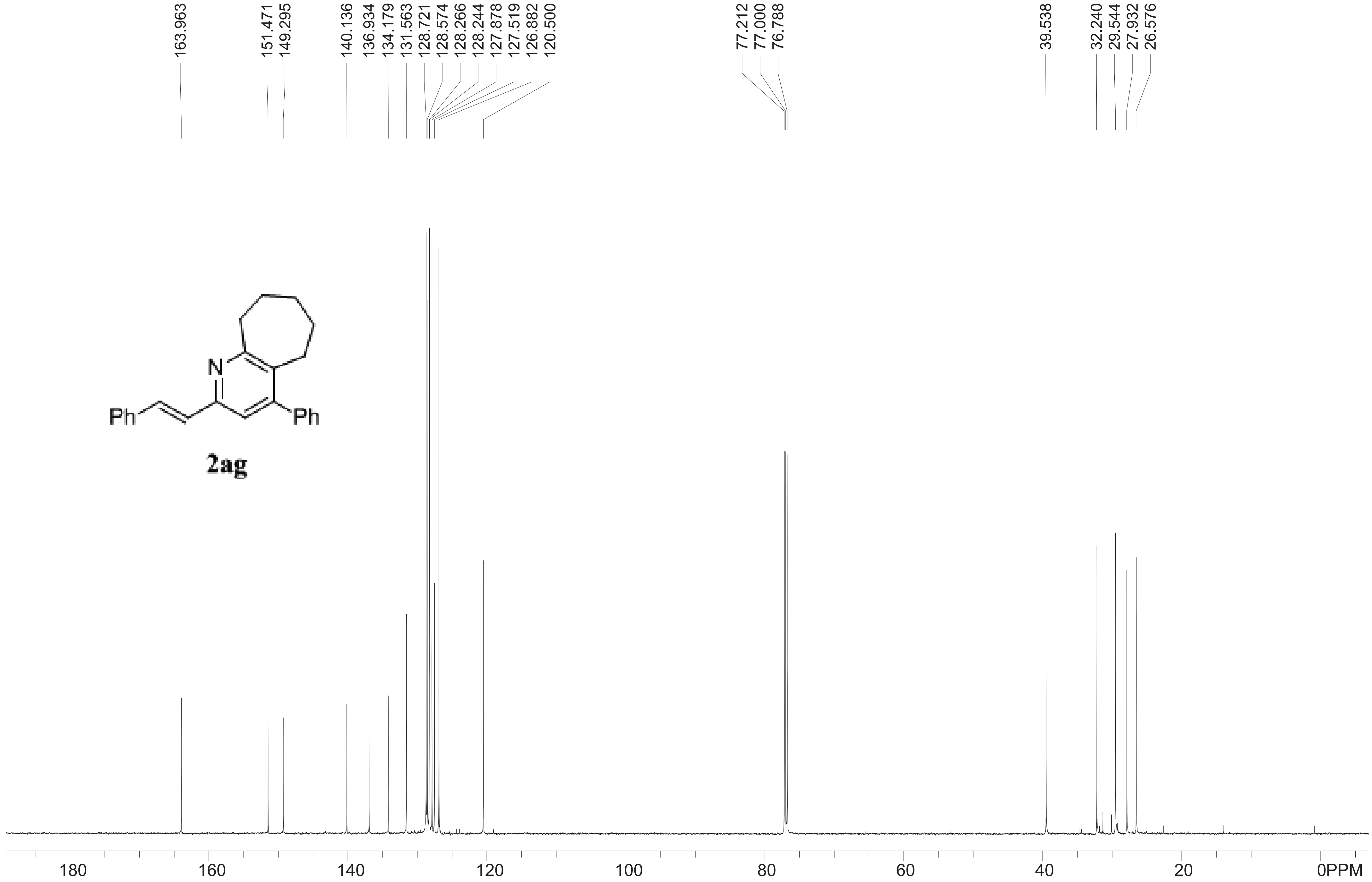

7.567
7.552
7.518
7.486
7.461
7.447
7.433
7.410
7.396
7.381
7.367
7.352
7.331
7.316
7.281
7.266
7.251
7.212
7.178
3.051
3.038
3.026
2.638
2.626
2.614
1.933
1.923
1.911
1.747
1.735
1.725
0.000
N
Ph
Ph
2af
4.03
3.03
2.00
1.05
1.08
2.01
2.00
2.00
2.08
2.01
8
6
4
2
0 PPM

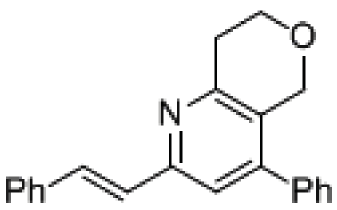

157.470
152.514
149.993
139.535
136.897
128.676
131.693
128.633
128.480
128.297
127.984
127.722
126.935
119.815
77.255
77.000
76.745
33.231
27.379
23.043
22.977
N
Ph
Ph
2af
180
160
140
120
100
80
60
40
20
0 PPM


7.470
7.465
7.453
7.444
7.352
7.350
7.341
7.328
7.302
7.299
7.293
7.289
7.277
7.258
7.245
7.232
7.201
7.198
7.195
7.187
7.168
7.156
7.144
7.137
7.097
7.071
7.048
3.083
3.074
3.064
2.638
2.629
2.620
1.788
1.779
1.771
1.761
1.719
1.711
1.702
1.694
1.540
1.531
1.522
3.02
2.00
2.01
2.01
1.00
1.02
1.00
0.98
2.00
1.99
2.03
2.02
2.01
9
8
7
6
5
4
3
2
1
0 PPM

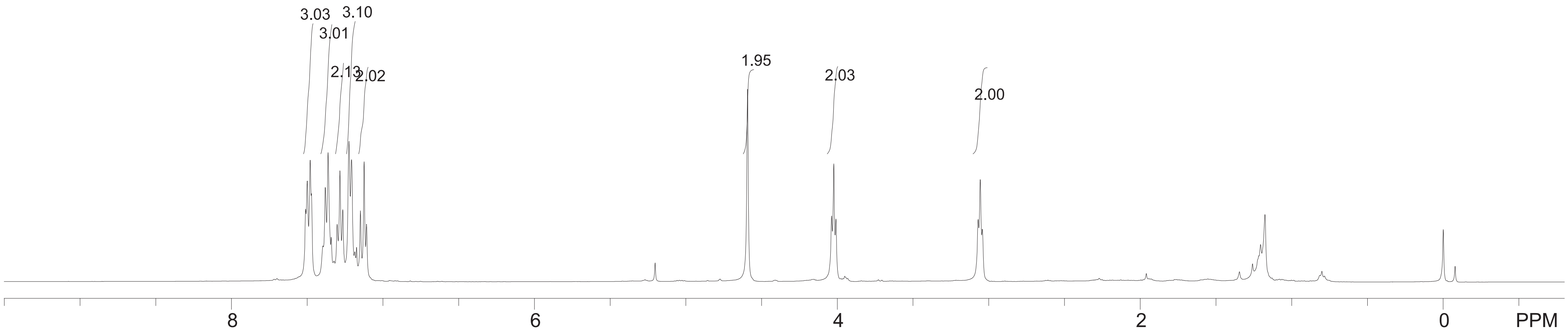

N
Ph
Ph

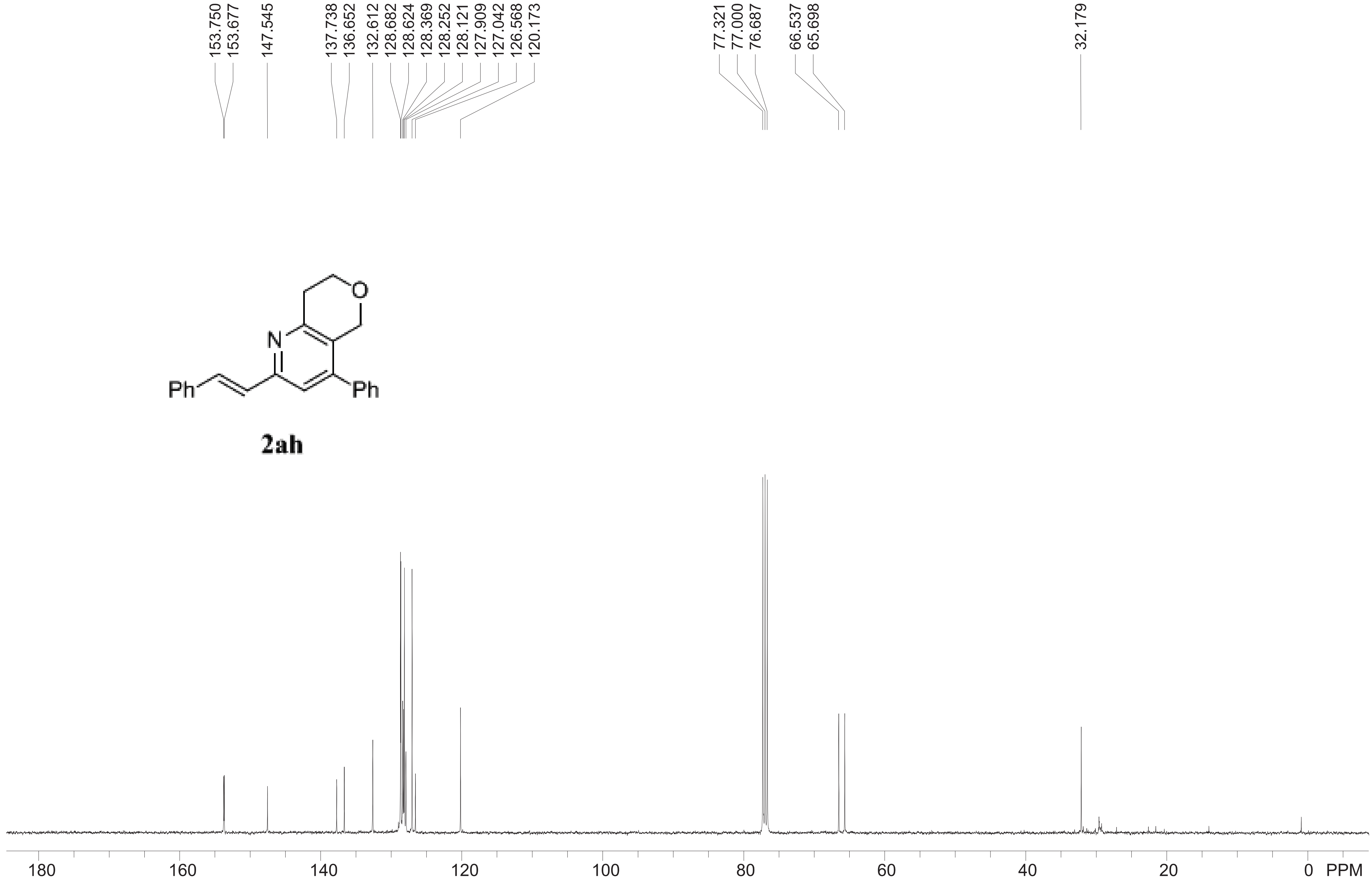
2ag

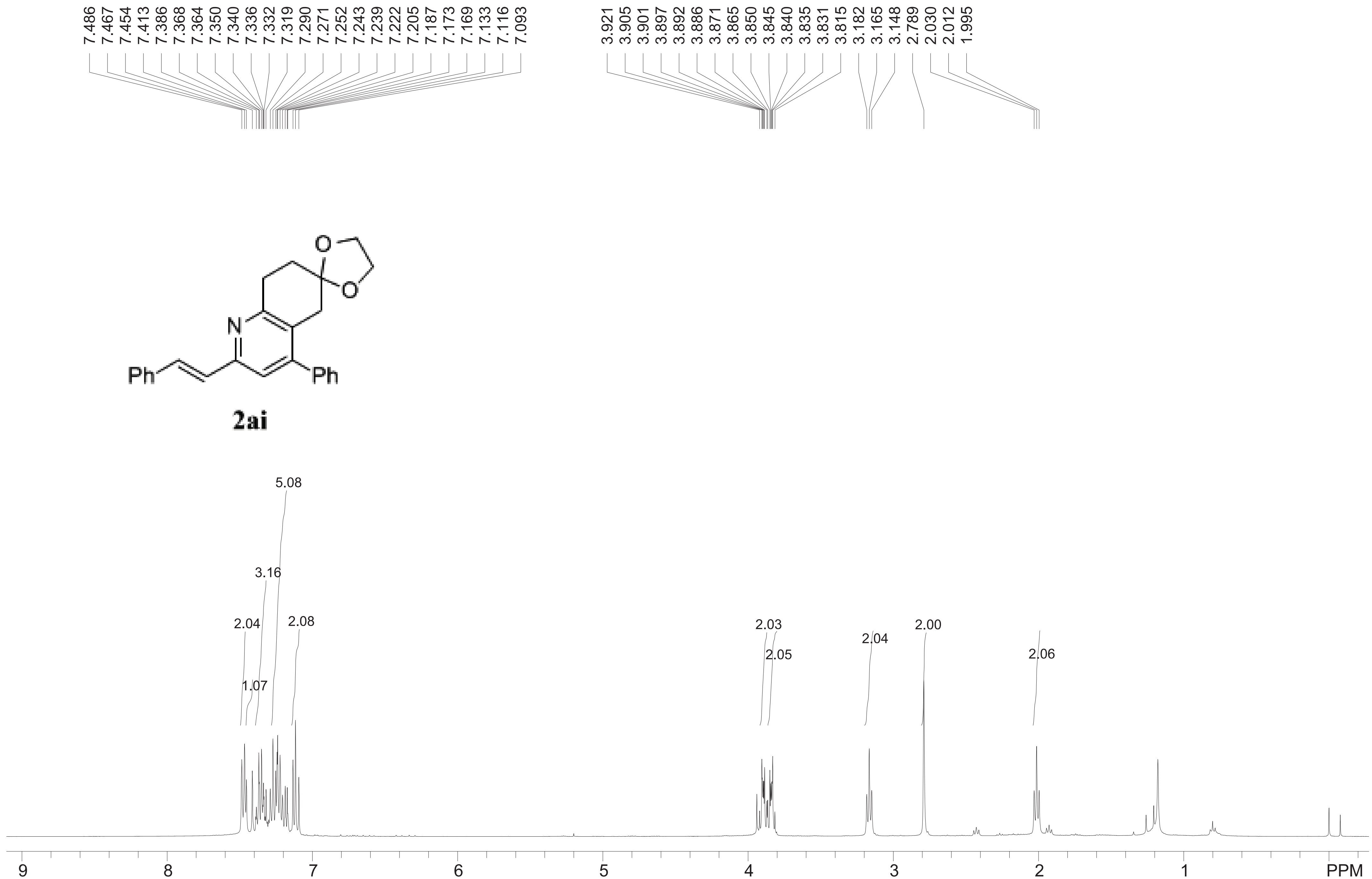

163.963
151.471
149.295
140.136
136.934
134.179
131.563
128.721
128.574
128.266
128.244
127.878
127.519
126.882
120.500
77.212
77.000
76.788
39.538
32.240
29.544
27.932
26.576
N
Ph
Ph
2ag
180
160
140
120
100
80
60
40
20
0PPM

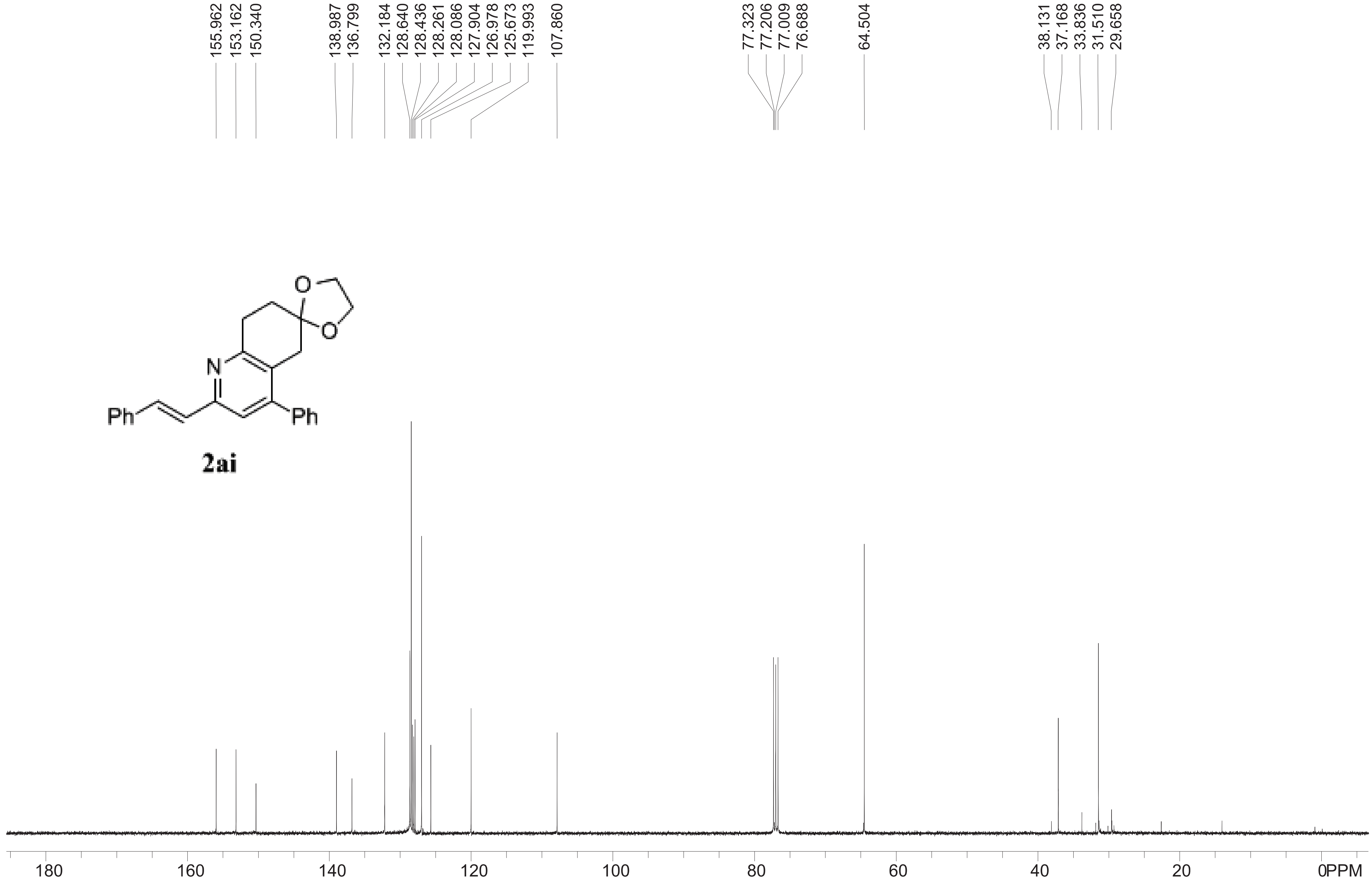


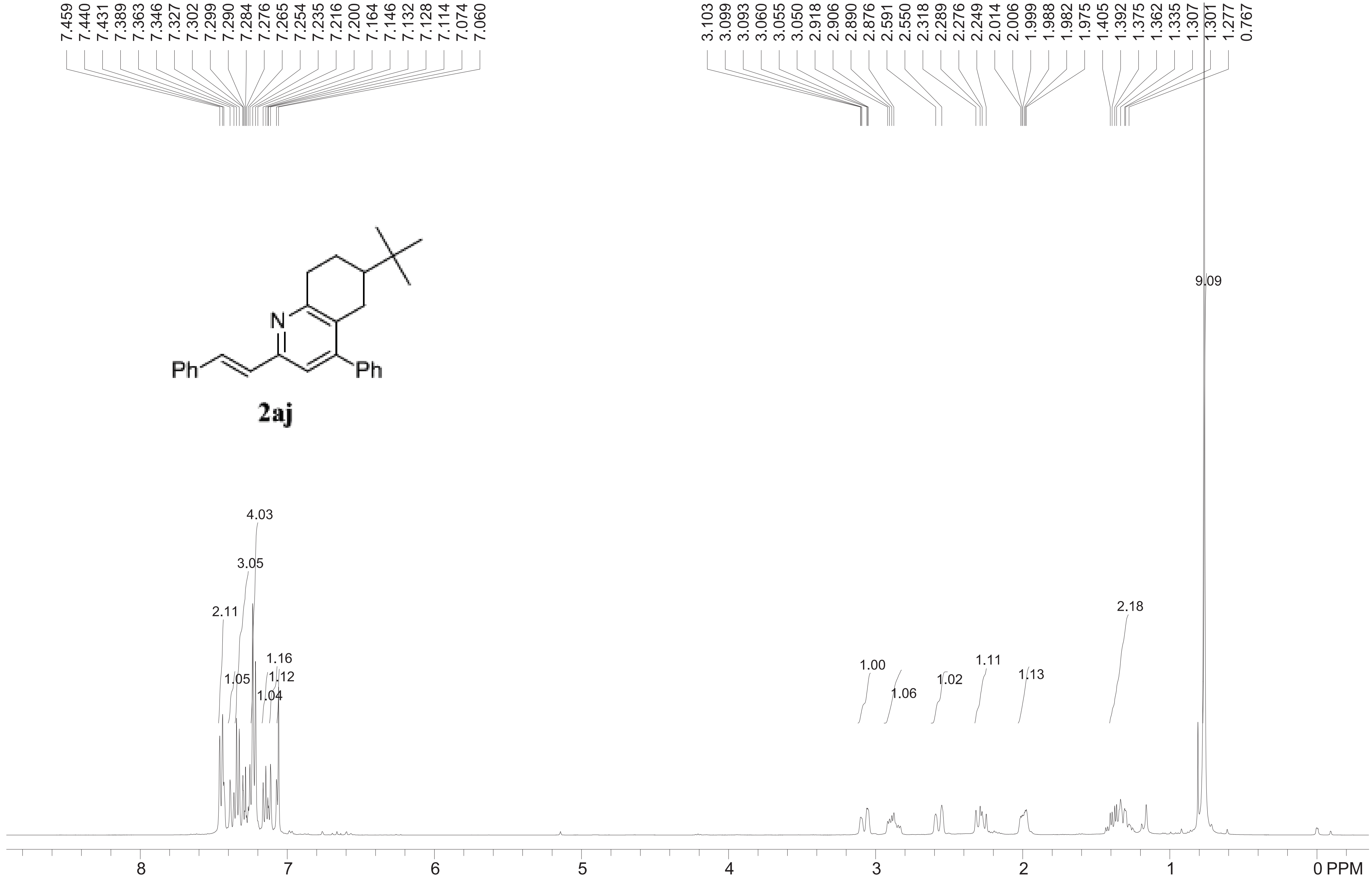
2ah

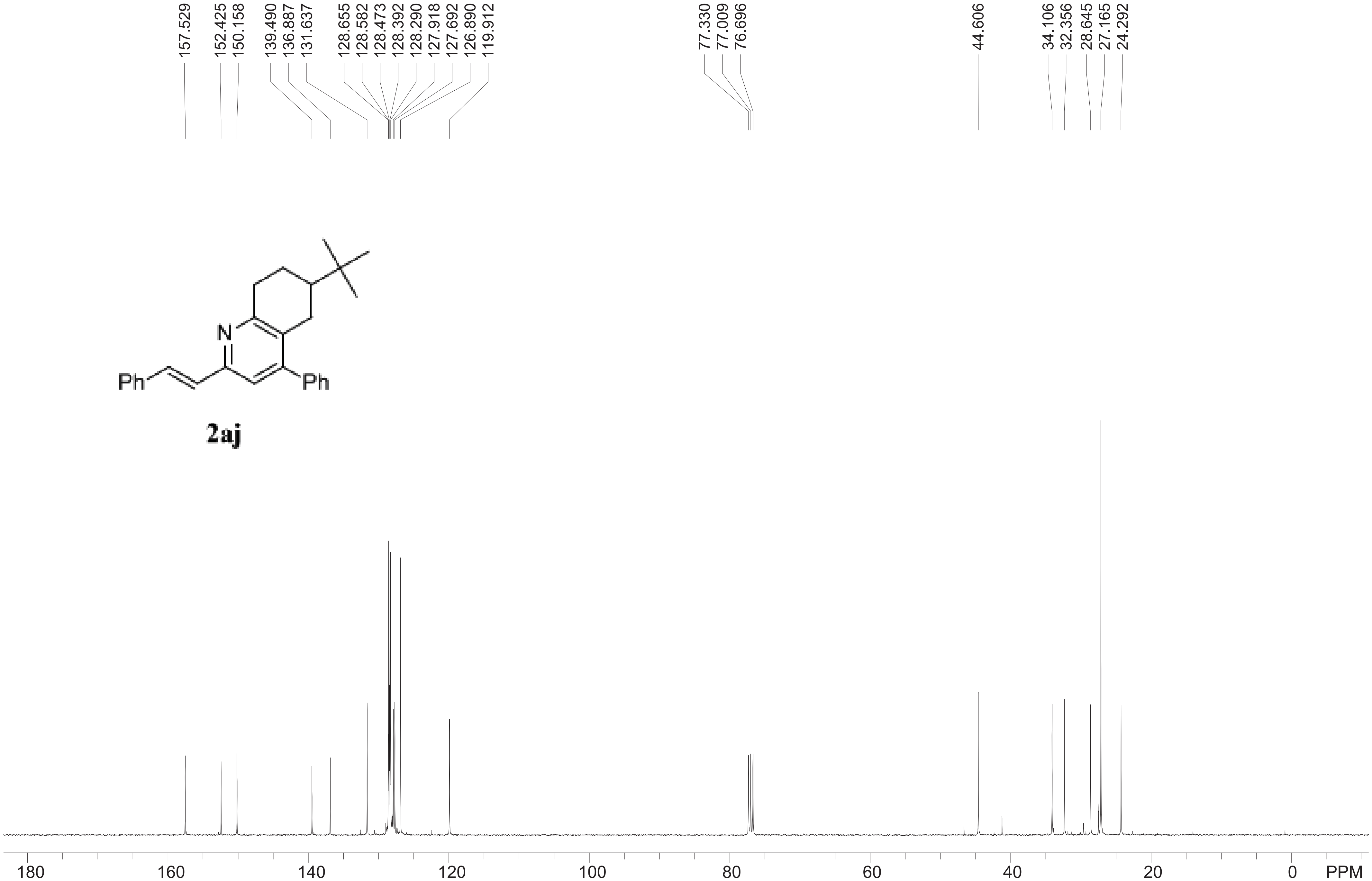

7.510
7.499
7.480
7.472
7.396
7.381
7.363
7.341
7.302
7.284
7.265
7.224
7.207
7.174
7.149
7.124
7.108
4.593
4.038
4.024
4.010
3.070
3.056
3.042
-0.000
3.03
3.01
3.10
2.13
2.02
1.95
2.03
2.00
8
6
4
2
0
PPM

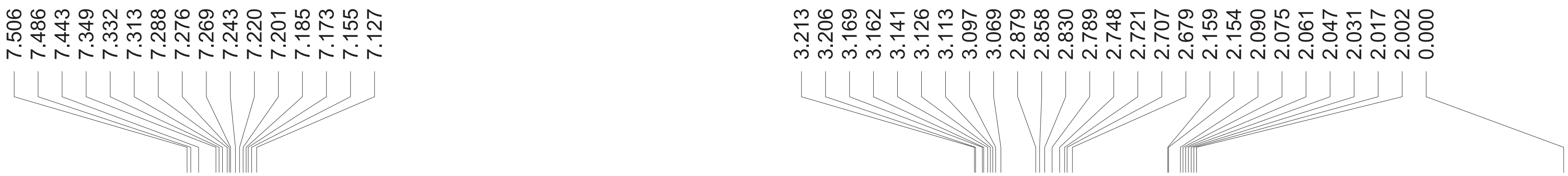

153.750
153.677
147.545
137.738
136.652
132.612
128.682
128.624
128.369
128.252
128.121
127.909
127.042
126.568
120.173
77.321
77.000
76.687
66.537
65.698
32.179
N
O
Ph
Ph
2ah
180
160
140
120
100
80
60
40
20
0 PPM

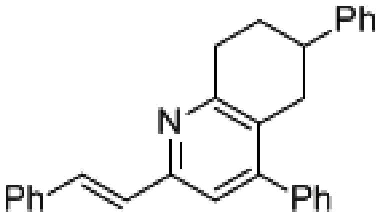

7.486
7.467
7.454
7.413
7.386
7.368
7.364
7.350
7.340
7.336
7.332
7.319
7.290
7.271
7.252
7.243
7.239
7.222
7.205
7.187
7.173
7.169
7.133
7.116
7.093
3.921
3.905
3.901
3.897
3.892
3.886
3.871
3.865
3.850
3.845
3.840
3.835
3.831
3.815
3.182
3.165
3.148
2.789
2.030
2.012
1.995
O
O
N
Ph
Ph
2ai
5.08
3.16
2.04
2.08
1.07
2.03
2.05
2.04
2.00
2.06
9
8
7
6
5
4
3
2
1
PPM


155.962
153.162
150.340
138.987
136.799
132.184
128.640
128.436
128.261
128.086
127.904
126.978
125.673
119.993
107.860
77.323
77.206
77.009
76.688
64.504
38.131
37.168
33.836
31.510
29.658
O
O
N
Ph
Ph
2ai
180
160
140
120
100
80
60
40
20
0PPM

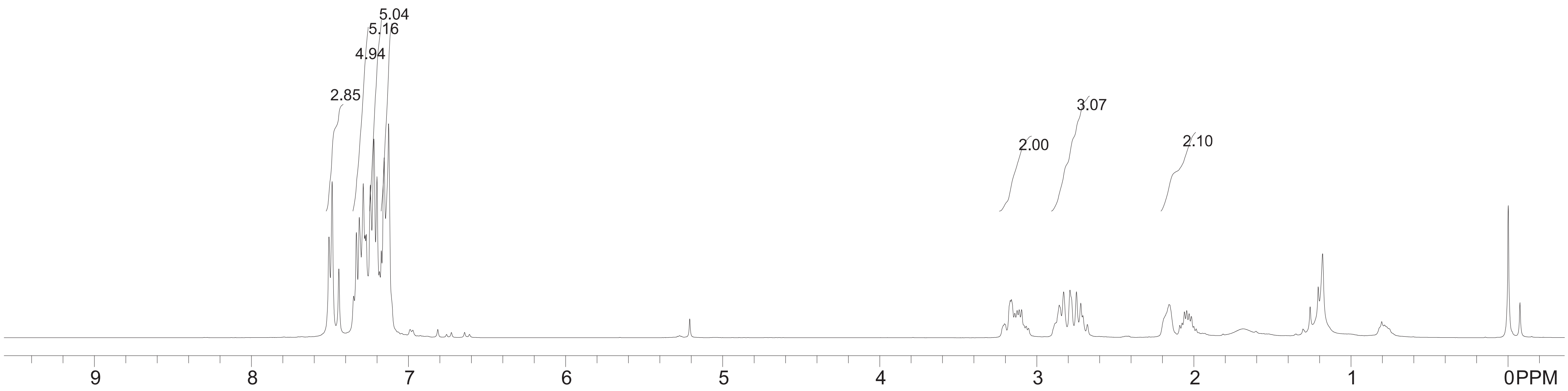

7.459 7.440 7.431 7.389 7.363 7.346 7.327 7.302 7.299 7.290 7.284 7.276 7.265 7.254 7.235 7.216 7.200 7.164 7.146 7.132 7.128 7.114 7.074 7.060
3.103 3.099 3.093 3.060 3.055 3.050 2.918 2.906 2.890 2.876 2.591 2.550 2.318 2.289 2.276 2.249 2.014 2.006 1.999 1.988 1.982 1.975 1.405 1.392 1.375 1.362 1.335 1.307 1.301 1.277 0.767
N
Ph
Ph
2aj
9.09
4.03
3.05
2.11
1.05
1.16
1.12
1.04
1.00
1.06
1.02
1.11
1.13
2.18
8
7
6
5
4
3
2
1
0 PPM

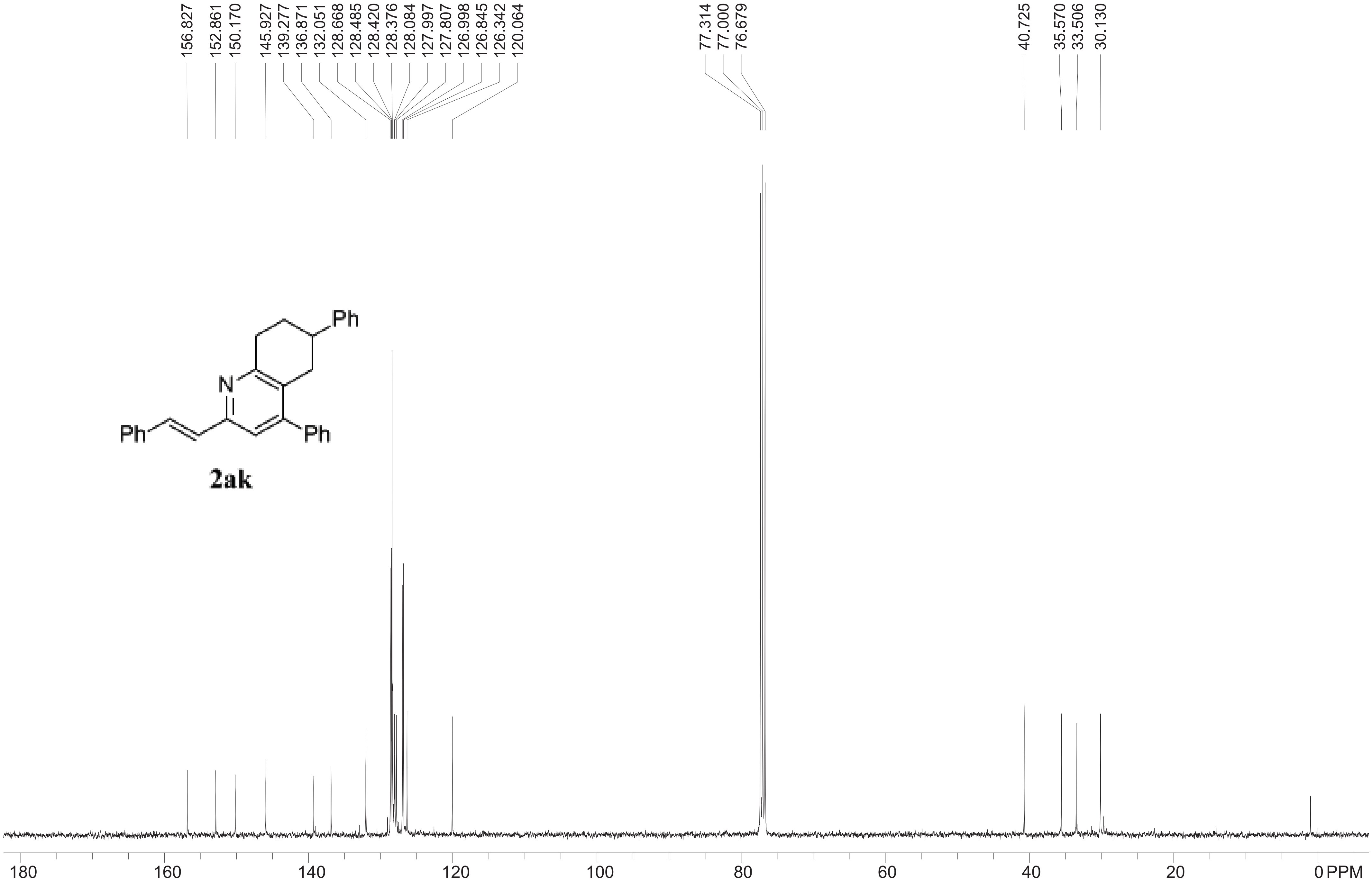

157.529
152.425
150.158
139.490
136.887
131.637
128.655
128.582
128.473
128.392
128.290
127.918
127.692
126.890
119.912
77.330
77.009
76.696
44.606
34.106
32.356
28.645
27.165
24.292
N
Ph
Ph
2aj
180
160
140
120
100
80
60
40
20
0
PPM

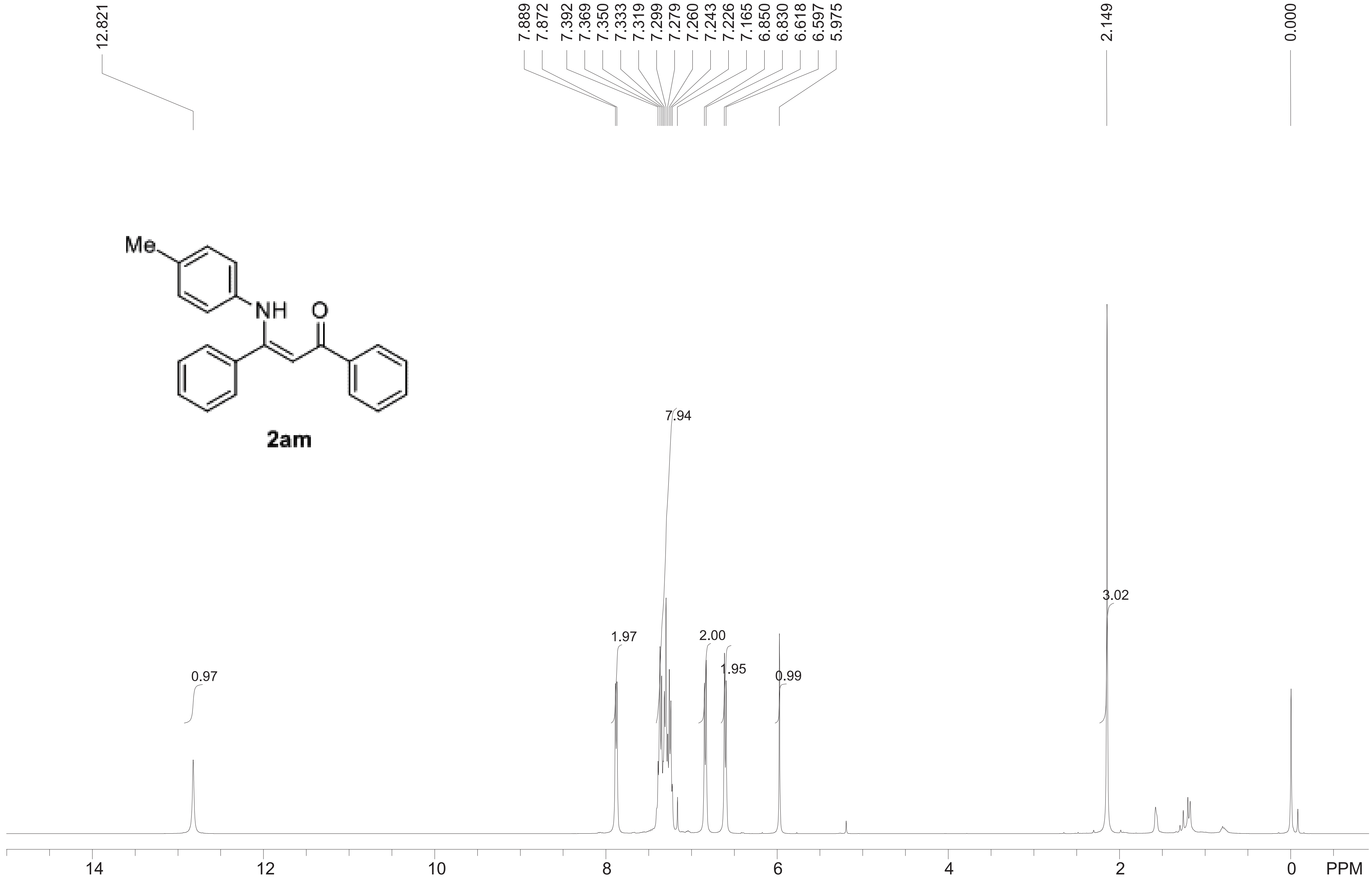

7.506
7.486
7.443
7.349
7.332
7.313
7.288
7.276
7.269
7.243
7.220
7.201
7.185
7.173
7.155
7.127
3.213
3.206
3.169
3.162
3.141
3.126
3.113
3.097
3.069
2.879
2.858
2.830
2.789
2.748
2.721
2.707
2.679
2.159
2.154
2.090
2.075
2.061
2.047
2.031
2.017
2.002
0.000

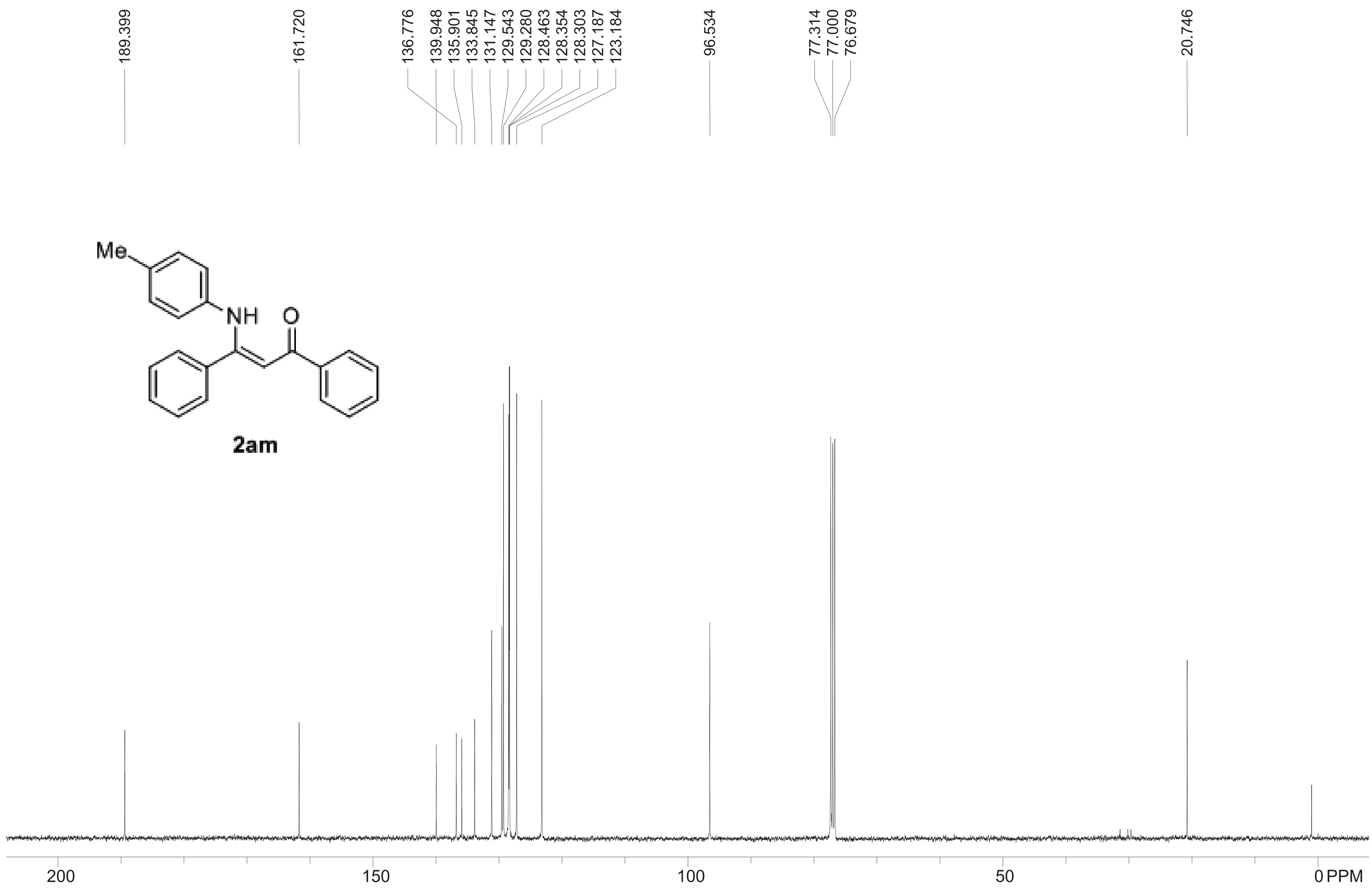

Ph
N
Ph
Ph

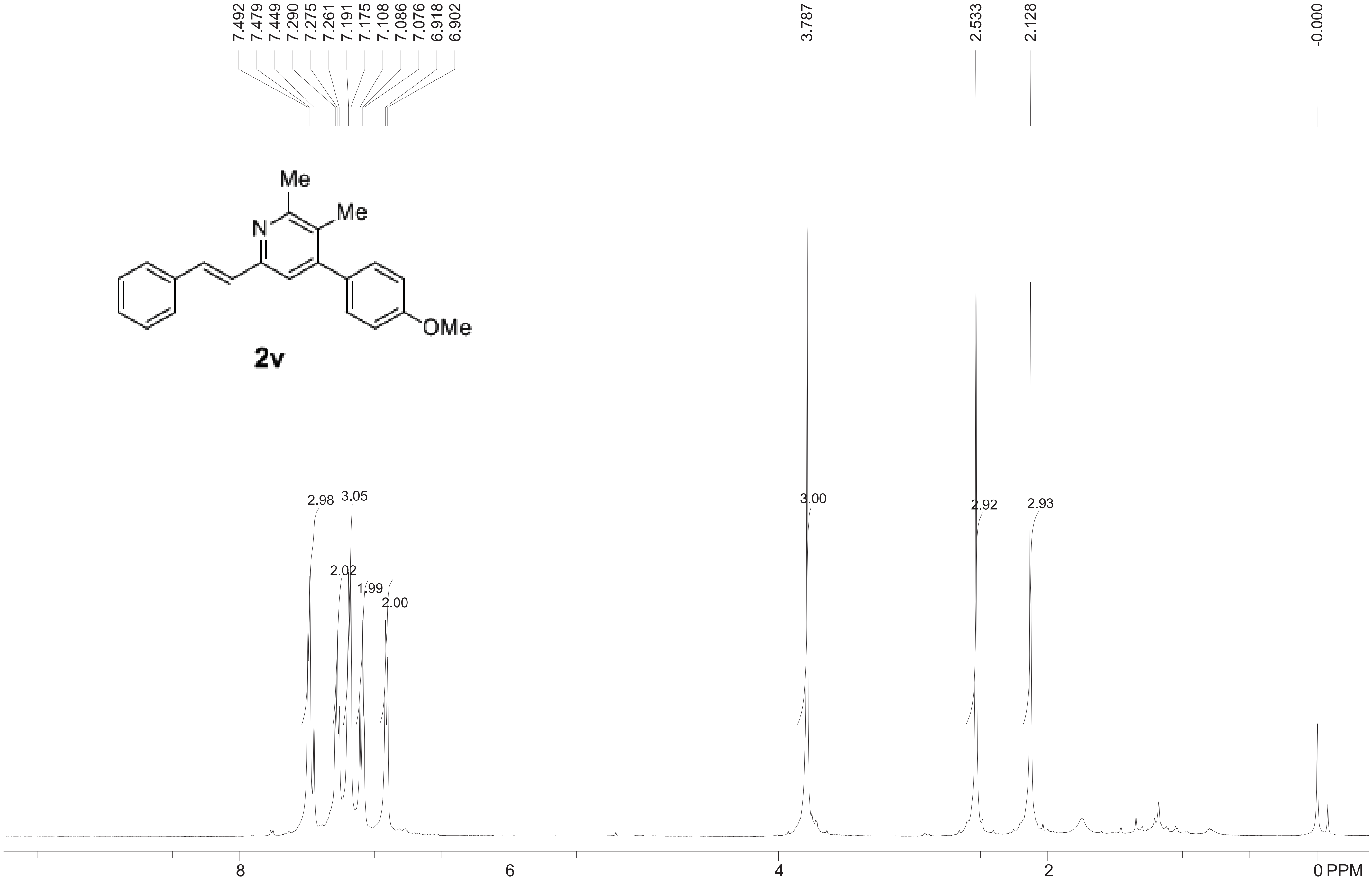
2ak

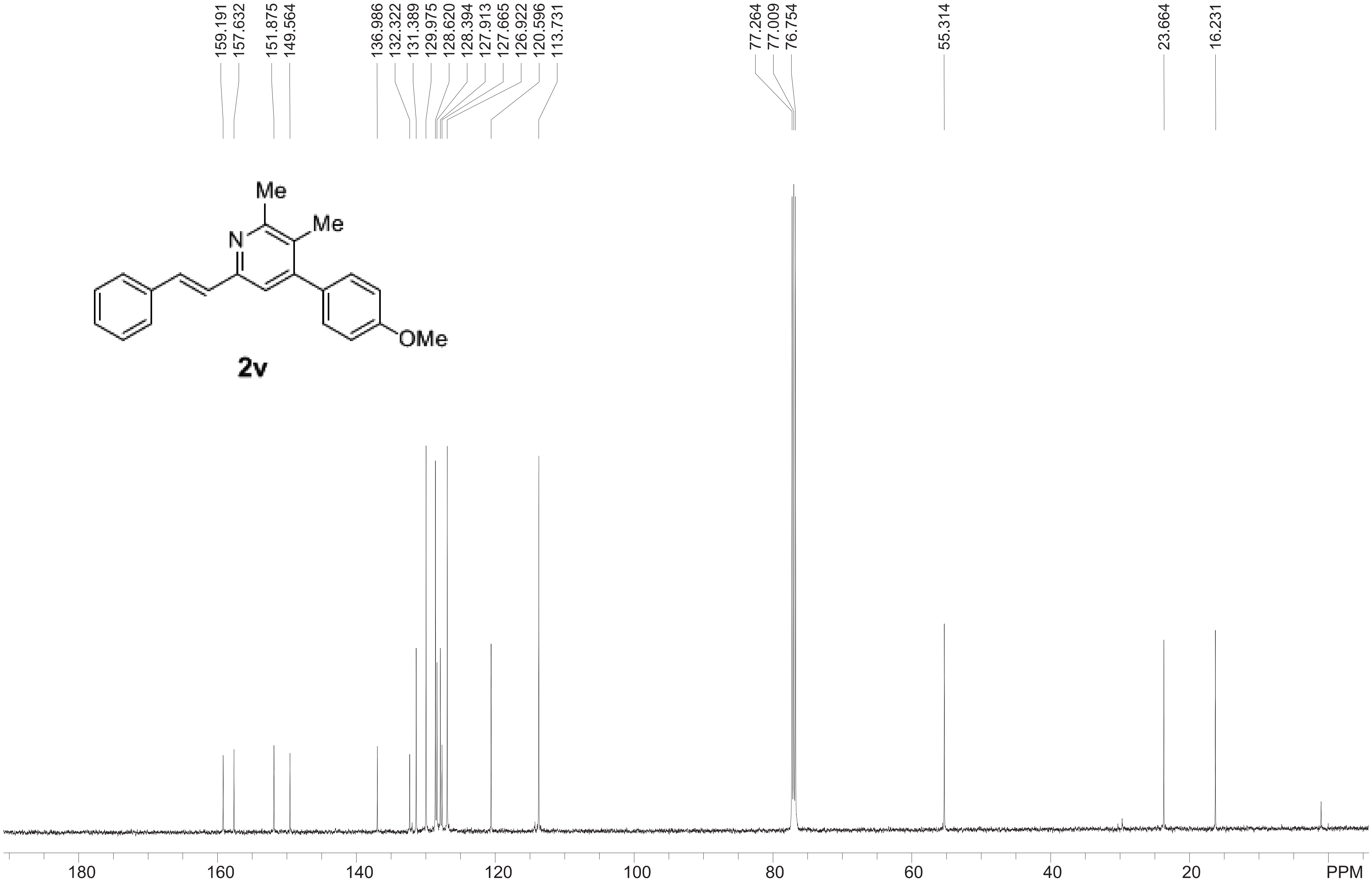

2.85
4.94
5.16
5.04
2.00
3.07
2.10
9
8
7
6
5
4
3
2
1
0PPM

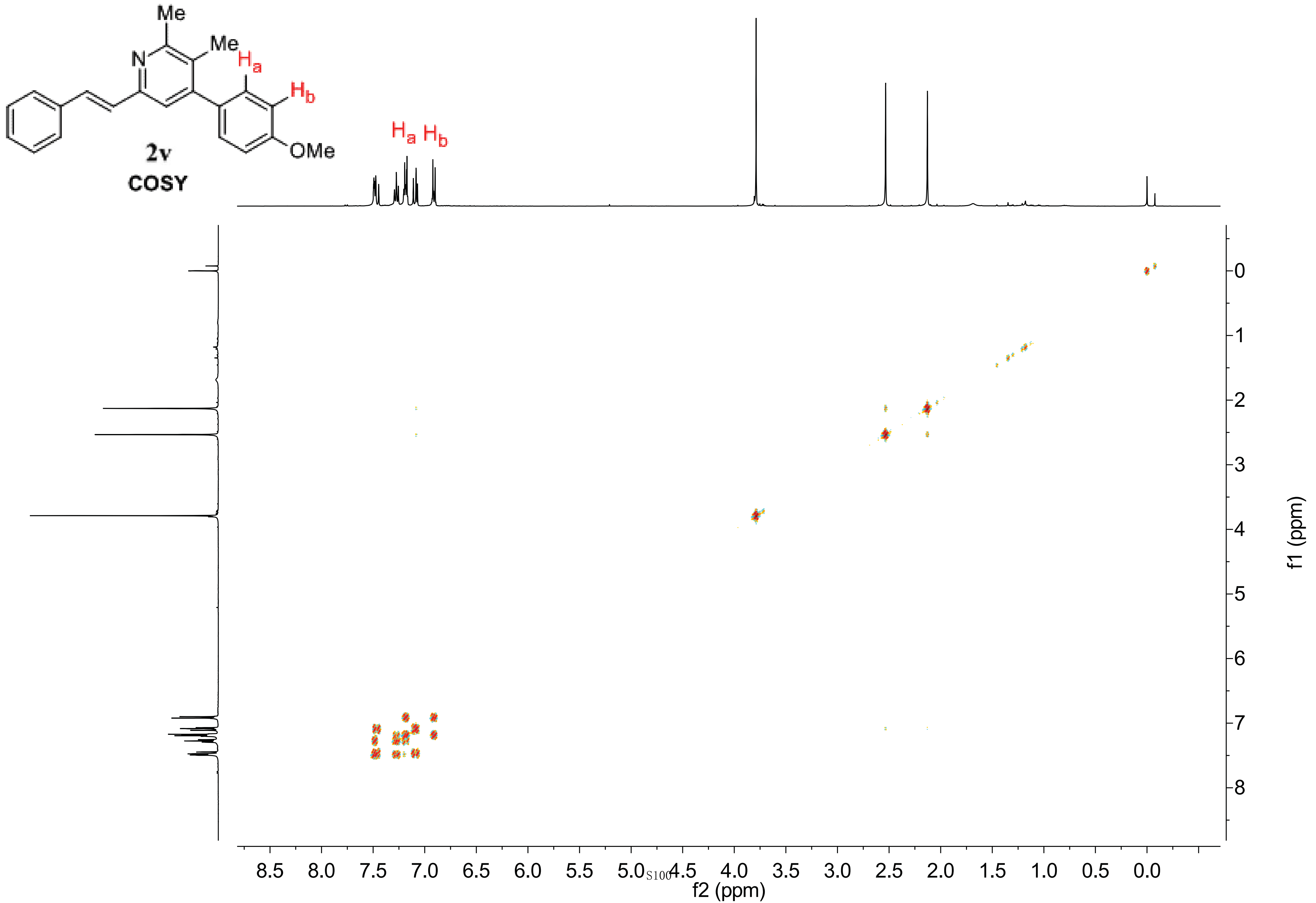

156.827
152.861
150.170
145.927
139.277
136.871
132.051
128.668
128.485
128.420
128.376
128.084
127.997
127.807
126.998
126.845
126.342
120.064
77.314
77.000
76.679
40.725
35.570
33.506
30.130
Ph
N
Ph
Ph
2ak
180
160
140
120
100
80
60
40
20
0 PPM

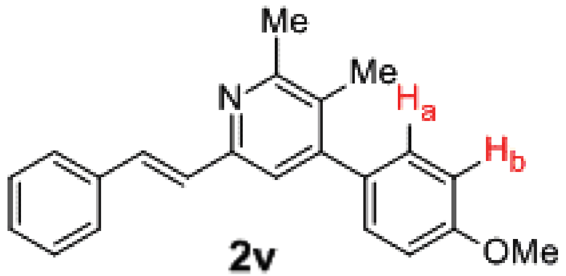

12.821
7.889
7.872
7.392
7.369
7.350
7.333
7.319
7.299
7.279
7.260
7.243
7.226
7.165
6.850
6.830
6.618
6.597
5.975
2.149
0.000
Me
NH
O
2am
0.97
1.97
7.94
2.00
1.95
0.99
3.02
14
12
10
8
6
4
2
0
PPM


189.399
161.720
136.776
139.948
135.901
133.845
131.147
129.543
129.280
128.463
128.354
128.303
127.187
123.184
96.534
77.314
77.000
76.679
20.746
Me
NH
O
2am
200
150
100
50
0 PPM

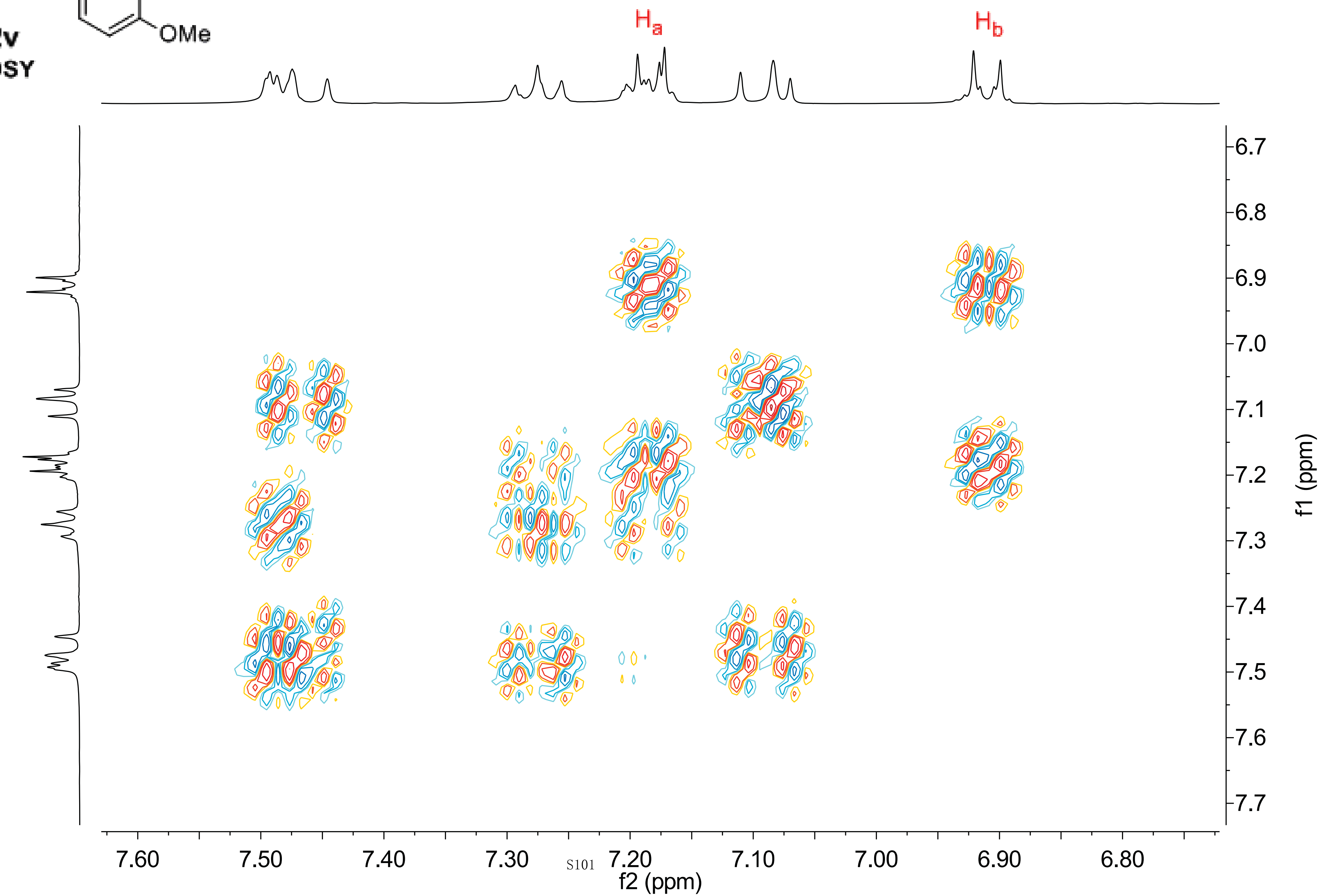

7.492
7.479
7.449
7.290
7.275
7.261
7.191
7.175
7.108
7.086
7.076
6.918
6.902
3.787
2.533
2.128
-0.000
Me
Me
N
OMe
2v
2.98
3.05
2.02
1.99
2.00
3.00
2.92
2.93
8
6
4
2
0 PPM

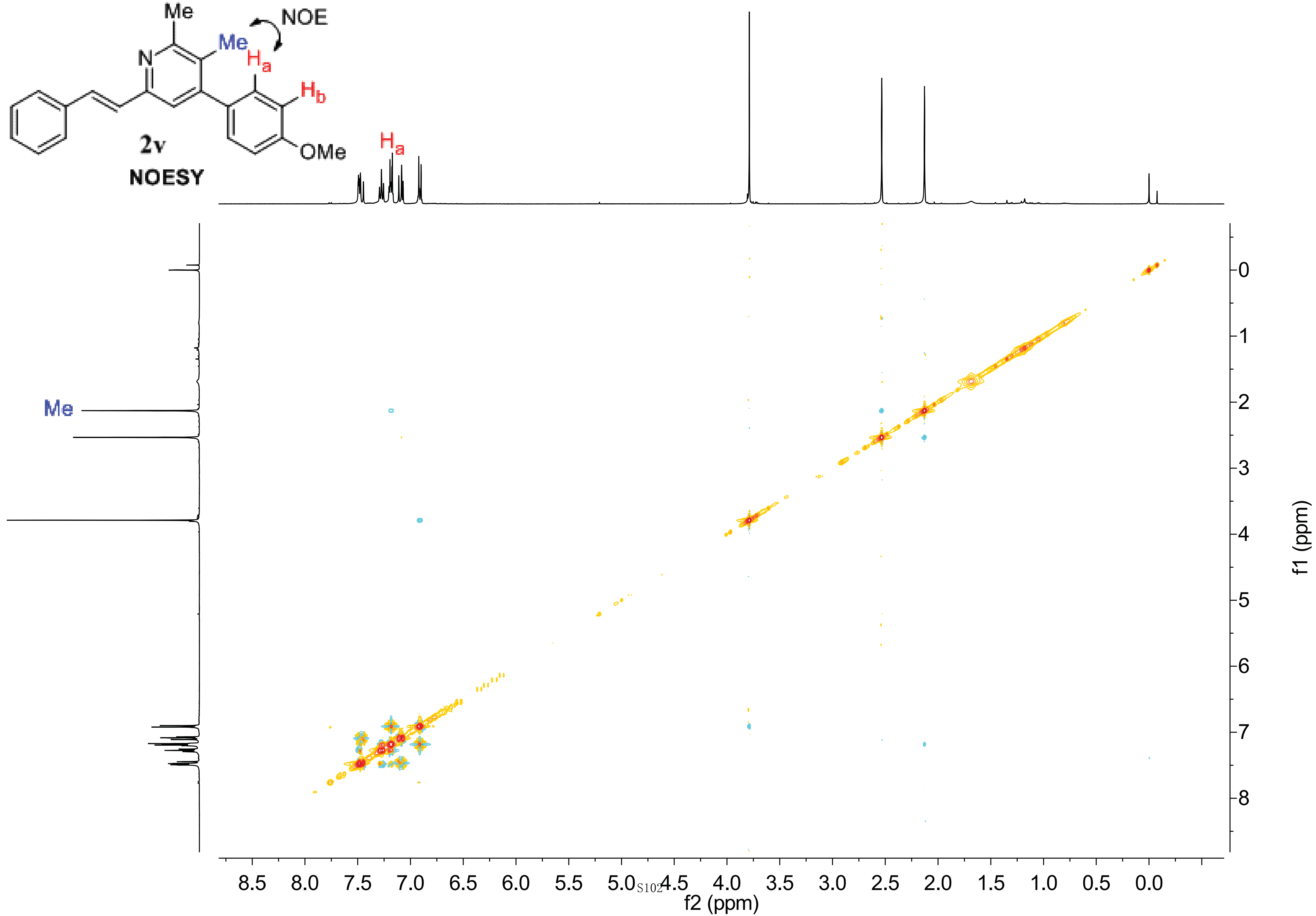

159.191
157.632
151.875
149.564
136.986
132.322
131.389
129.975
128.620
128.394
127.913
127.665
126.922
120.596
113.731
77.264
77.009
76.754
55.314
23.664
16.231
Me
Me
N
OMe
2v
180
160
140
120
100
80
60
40
20
PPM

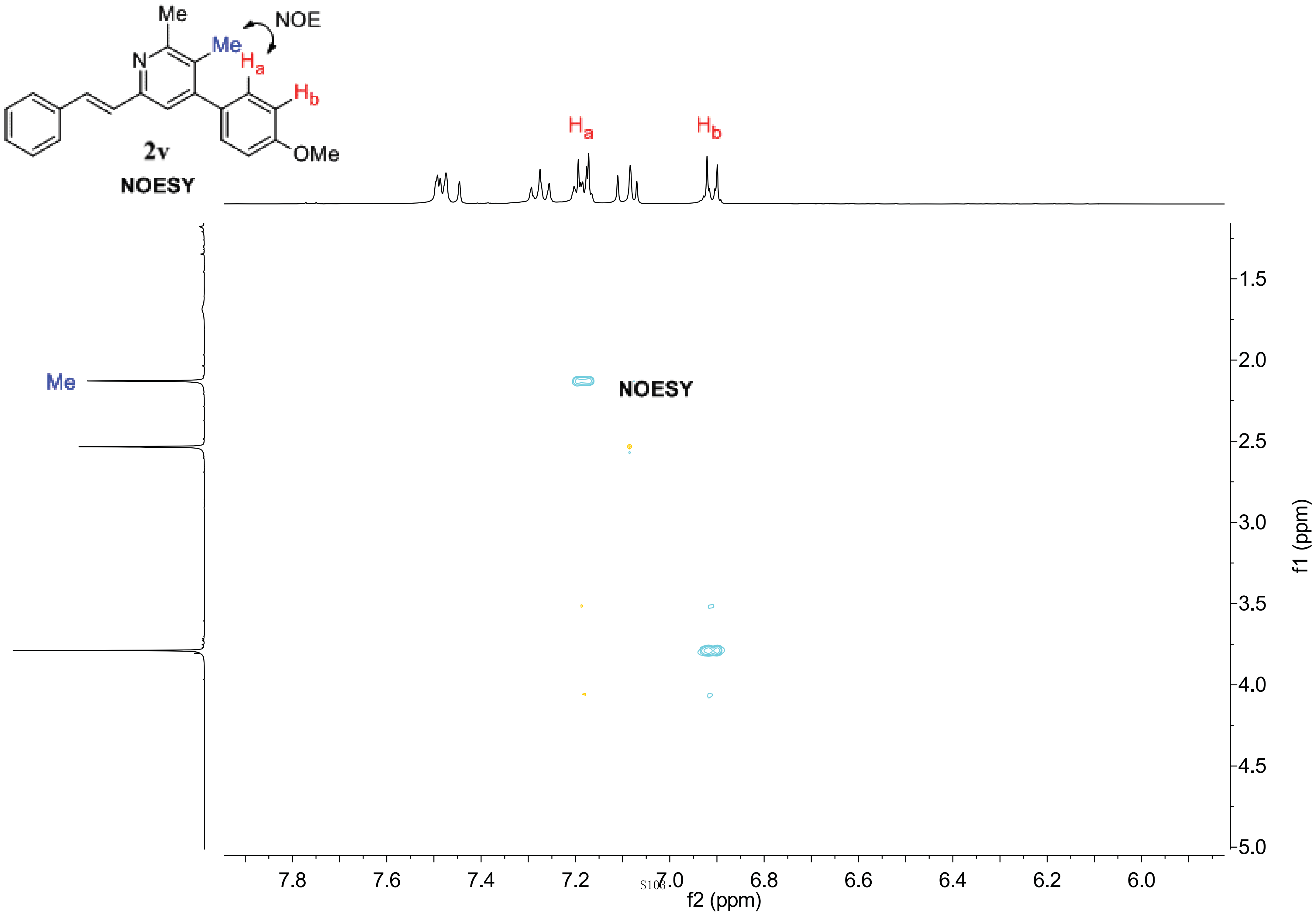

Me
Me
N
Ha
Hb
OMe
2v
COSY
Ha Hb
f1 (ppm)
f2 (ppm)

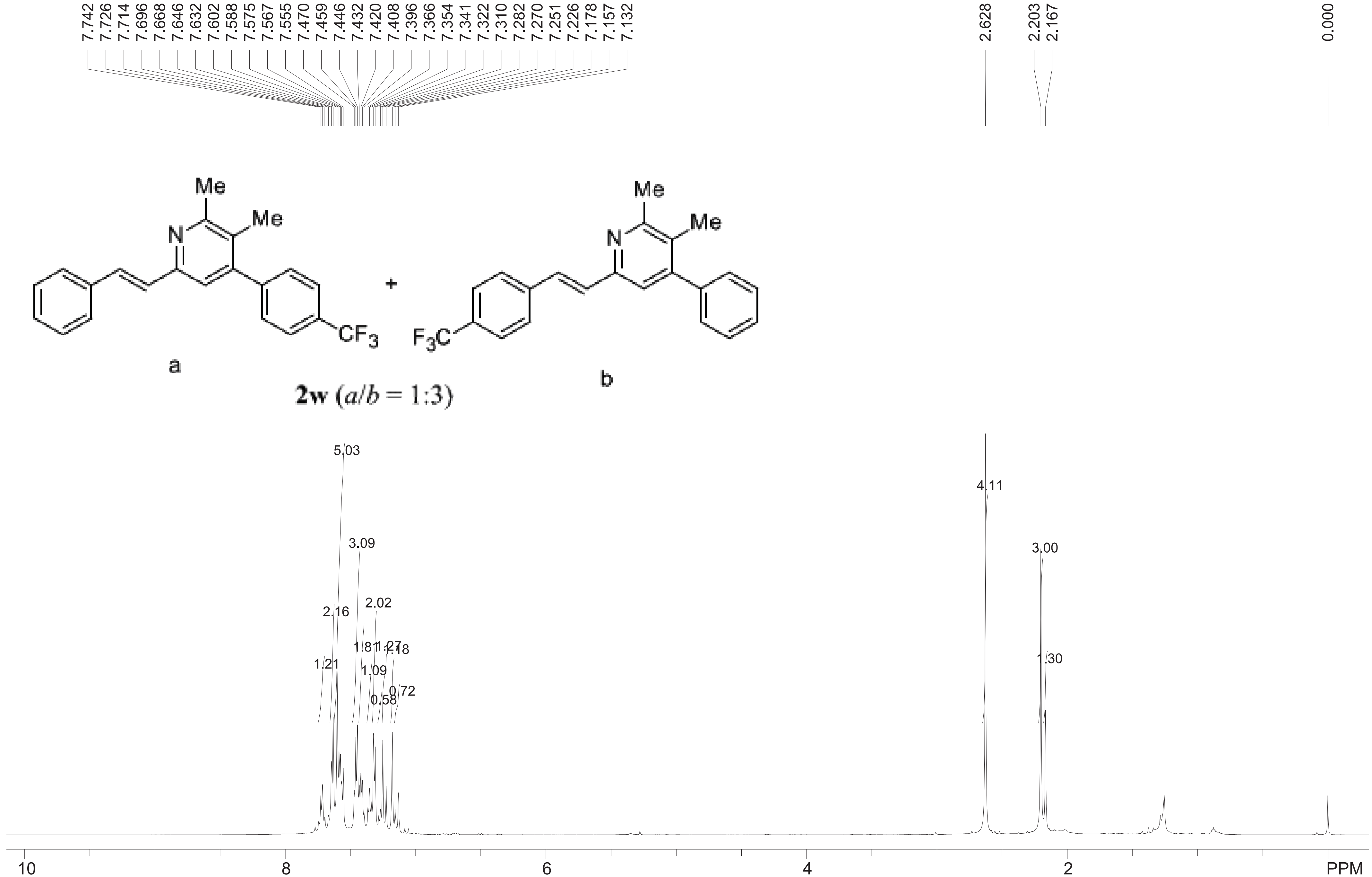

Me
Me
N
Ha
Hb
OMe

2v
COSY

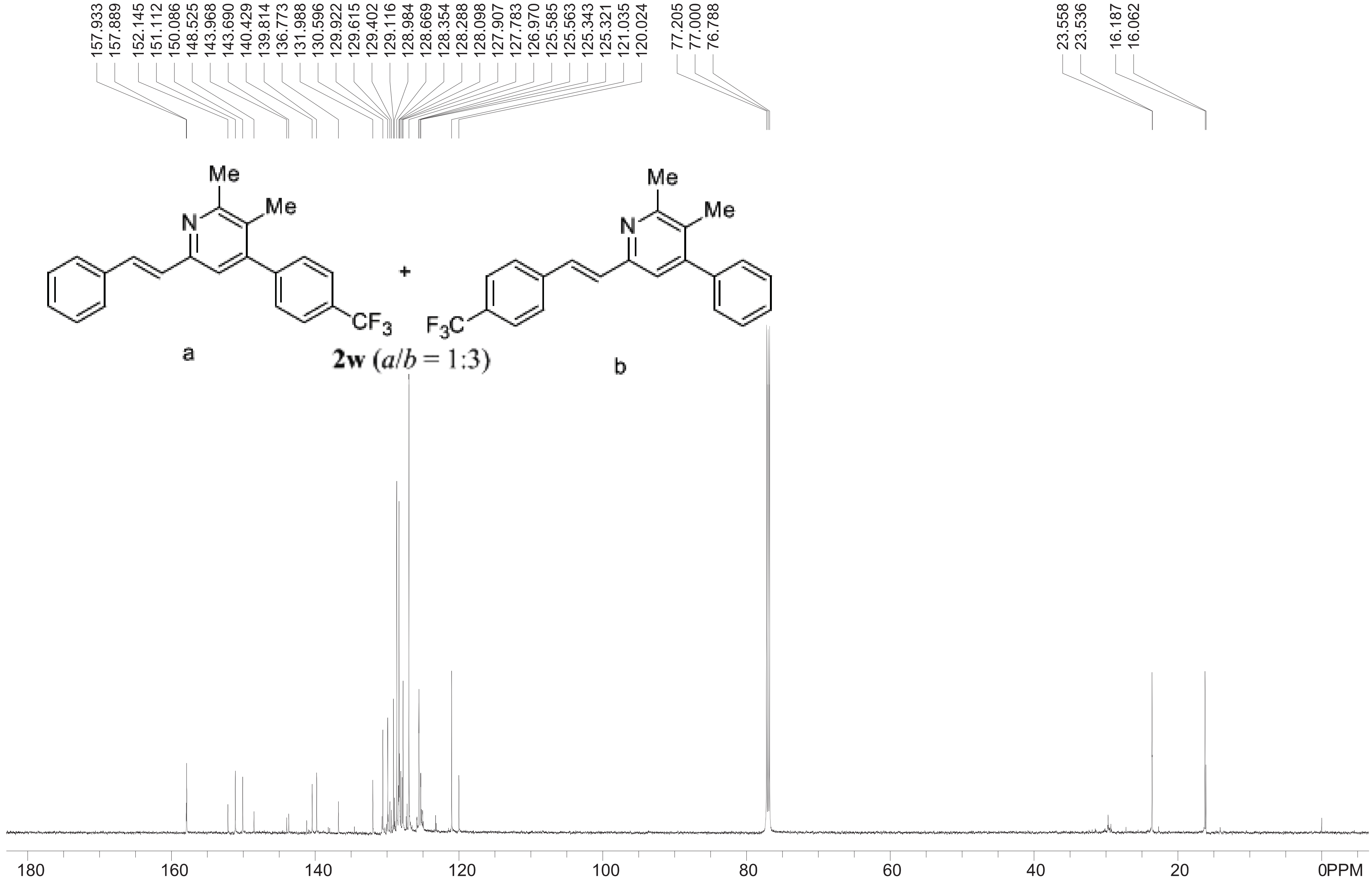

Ha
Hb
7.60
7.50
7.40
7.30
7.20
7.10
7.00
6.90
6.80
f2 (ppm)
6.7
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
f1 (ppm)

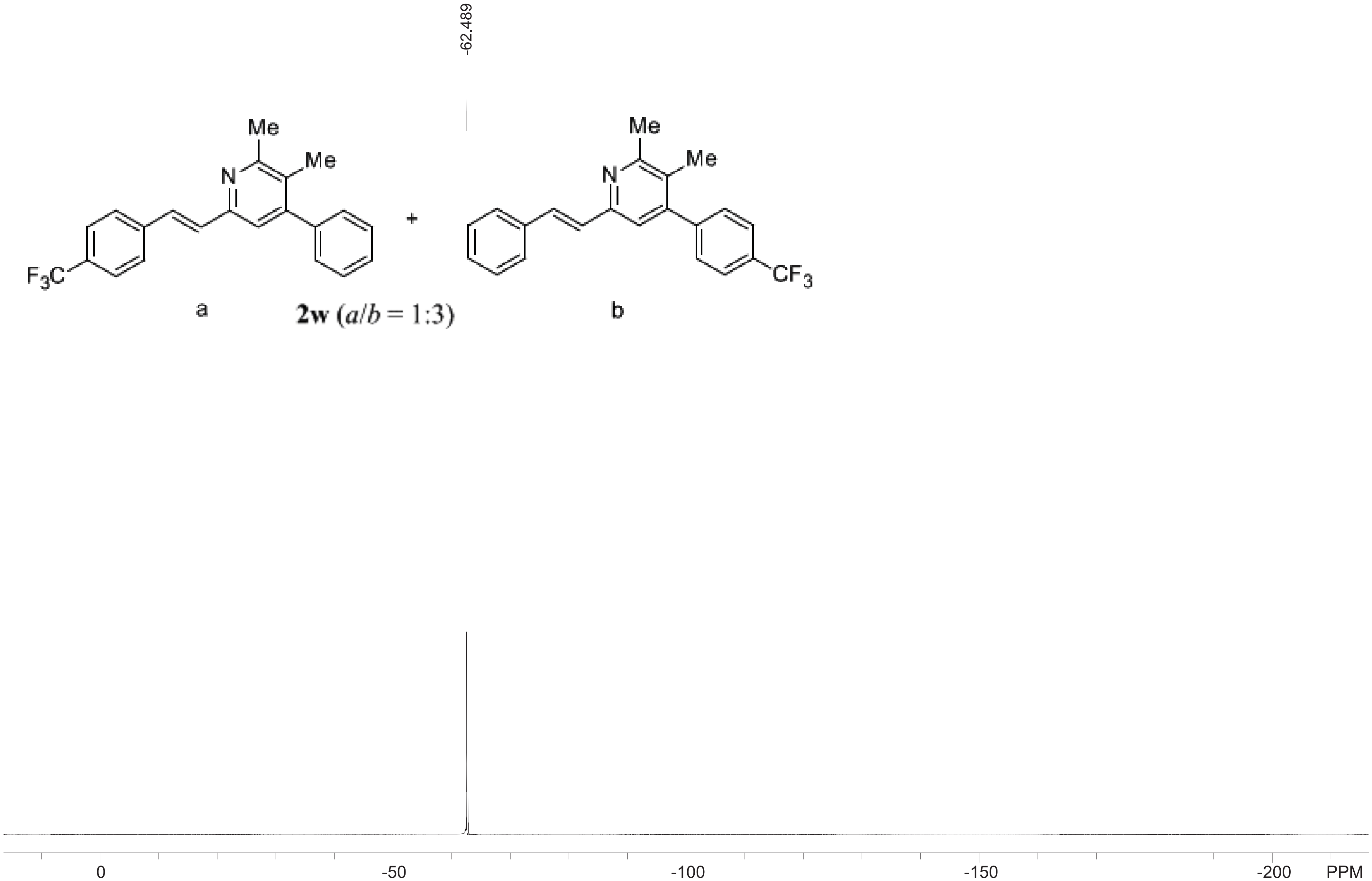

Me
NOE
N
Me
Ha
Hb
OMe
2v
NOESY
Ha
Me
f1 (ppm)
f2 (ppm)

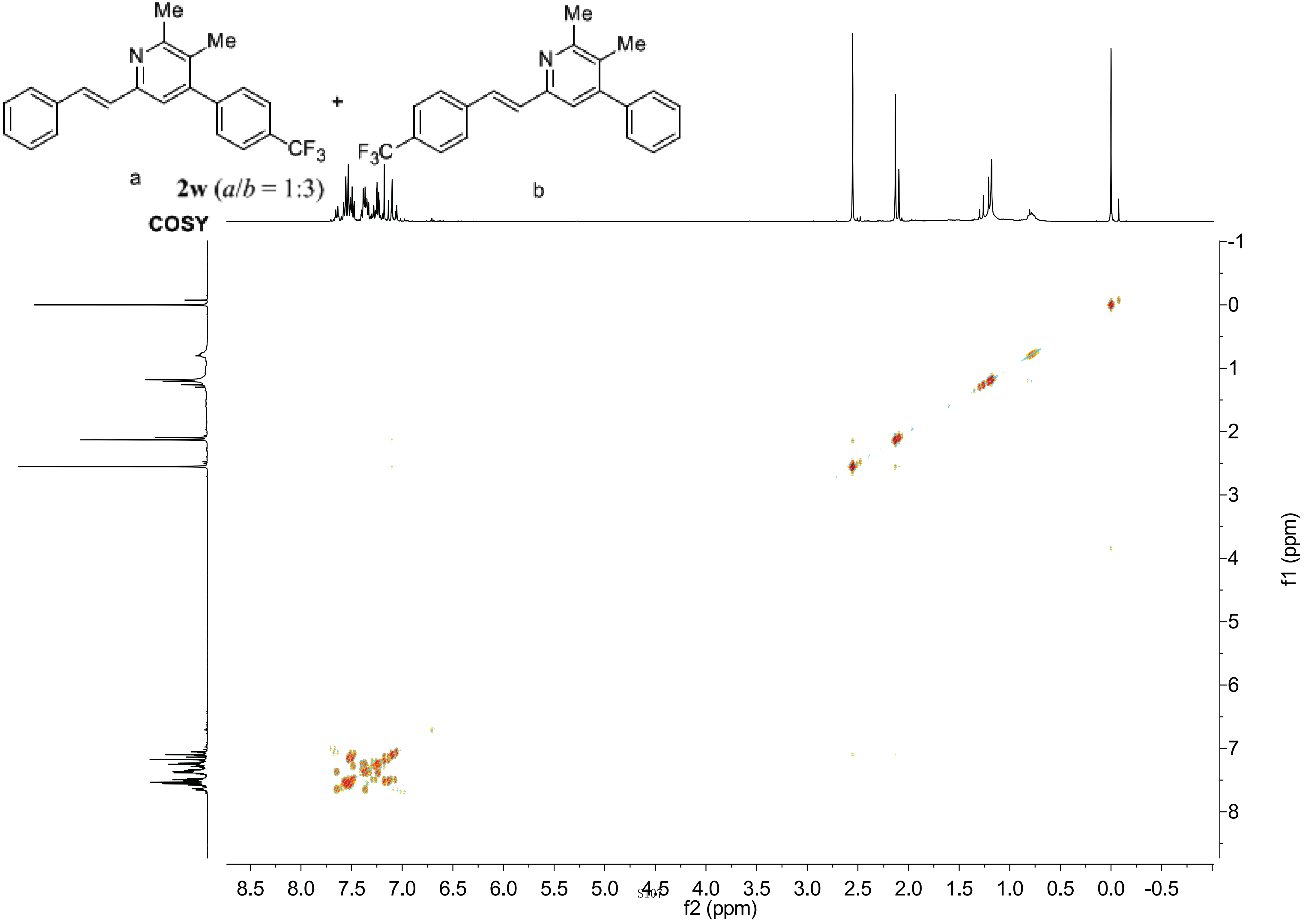

Me
N
Me
NOE
Ha
Hb
OMe
2v
NOESY
Ha
Hb
Me
NOESY
f1 (ppm)
f2 (ppm)

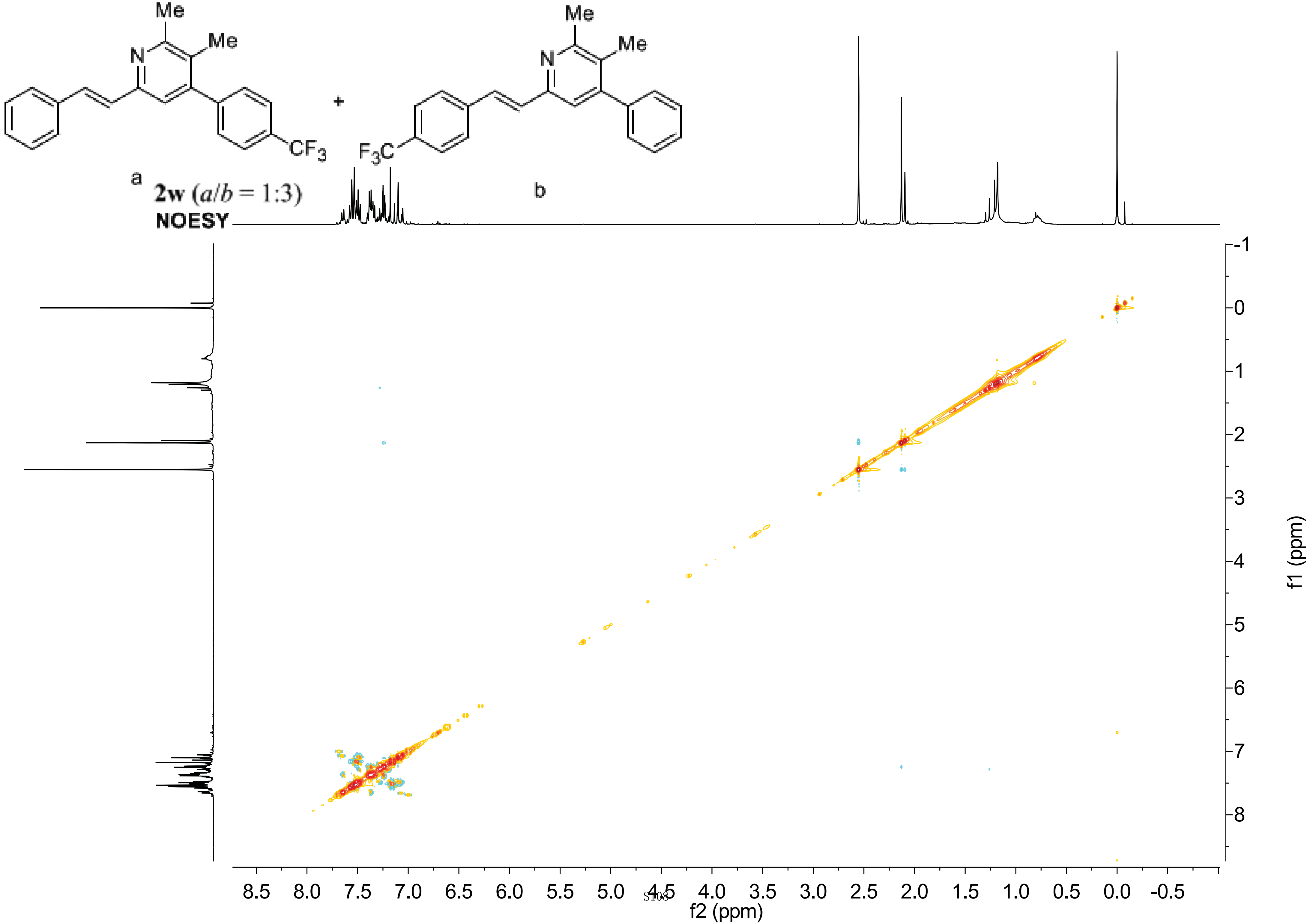

7.742
7.726
7.714
7.696
7.668
7.646
7.632
7.602
7.588
7.575
7.567
7.555
7.470
7.459
7.446
7.432
7.420
7.408
7.396
7.366
7.354
7.341
7.322
7.310
7.282
7.270
7.251
7.226
7.178
7.157
7.132
2.628
2.203
2.167
0.000
Me
Me
N
CF3
F3C
a
b
+
5.03
3.09
2.16
2.02
1.81
1.27
1.18
1.21
1.09
0.58
0.72
4.11
3.00
1.30
10
8
6
4
2
PPM

2w (*a*/*b* = 1:3)

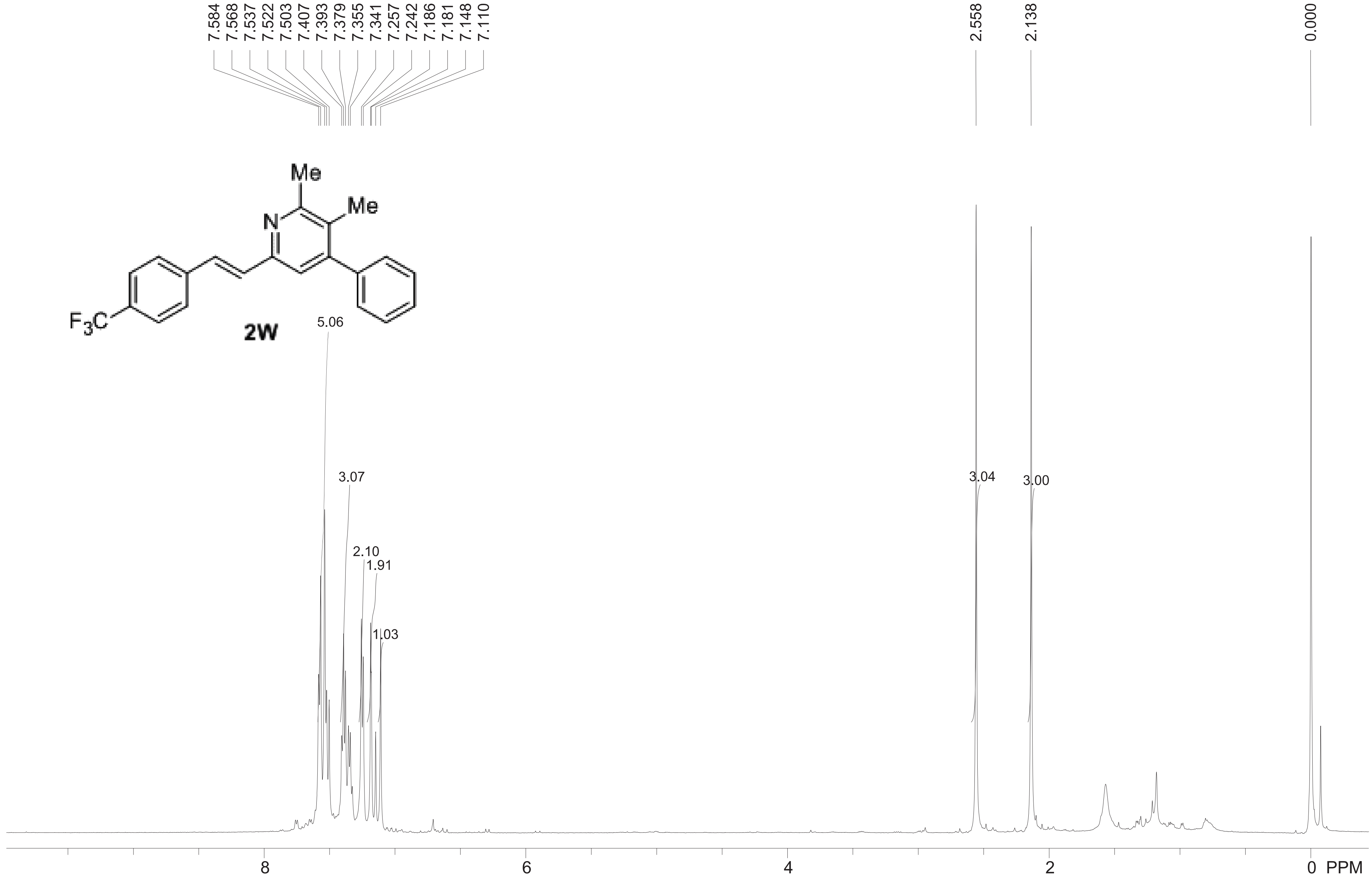

157.933
157.889
152.145
151.112
150.086
148.525
143.968
143.690
140.429
139.814
136.773
131.988
130.596
129.922
129.615
129.402
129.116
128.984
128.669
128.354
128.288
128.098
127.907
127.783
126.970
125.585
125.563
125.343
125.321
121.035
120.024
77.205
77.000
76.788
23.558
23.536
16.187
16.062
Me
Me
N
CF_3
a
+
Me
Me
N
F_3C
b
2w (a/b = 1:3)
180
160
140
120
100
80
60
40
20
0PPM

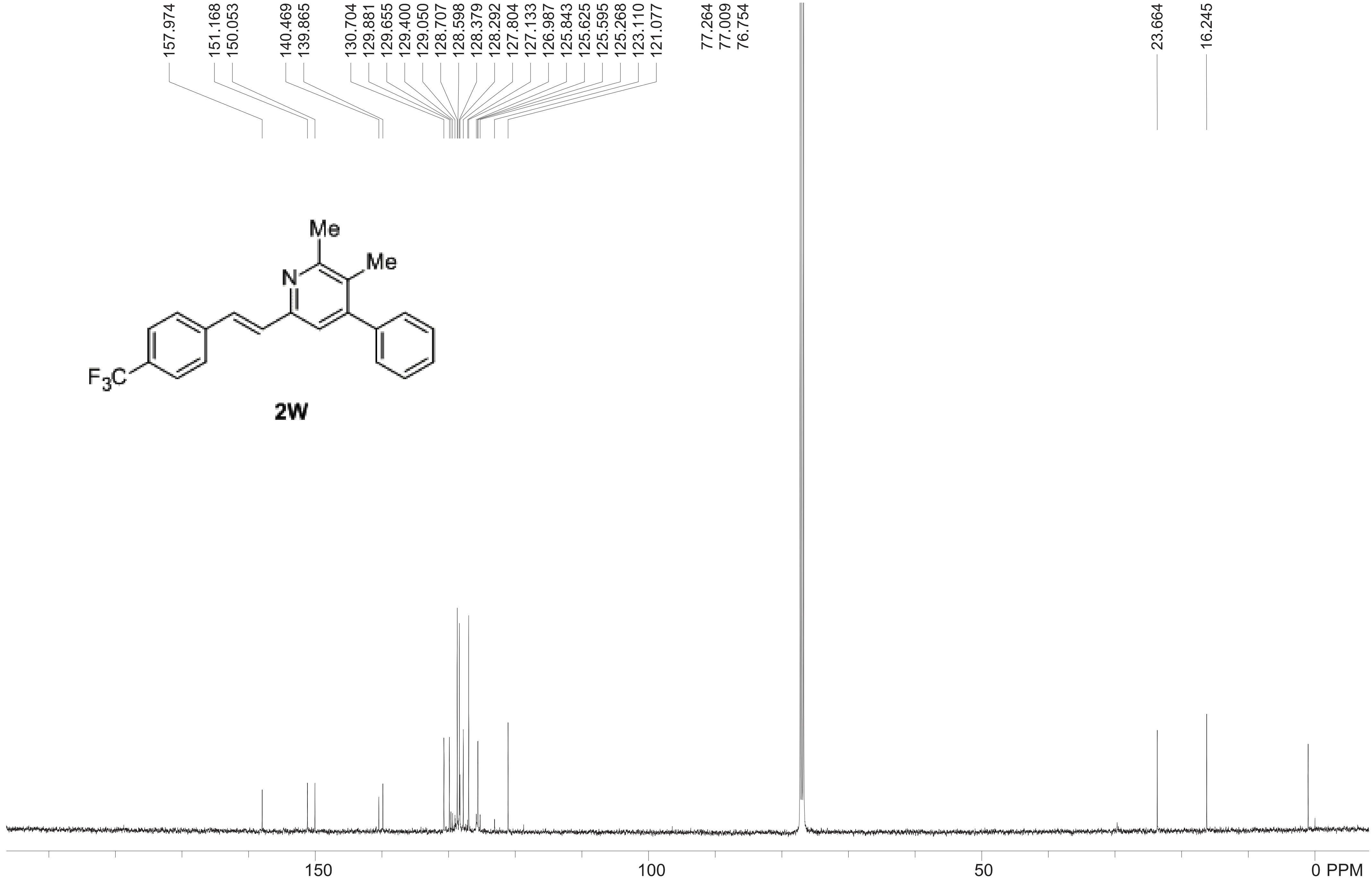

-62.489
Me
Me
N
F3C
a
+
b
CF3
2w (a/b = 1:3)
0
-50
-100
-150
-200
PPM


Me
Me
N
CF3
a
+
F3C
b
2w (a/b = 1:3)
COSY
f2 (ppm)
f1 (ppm)

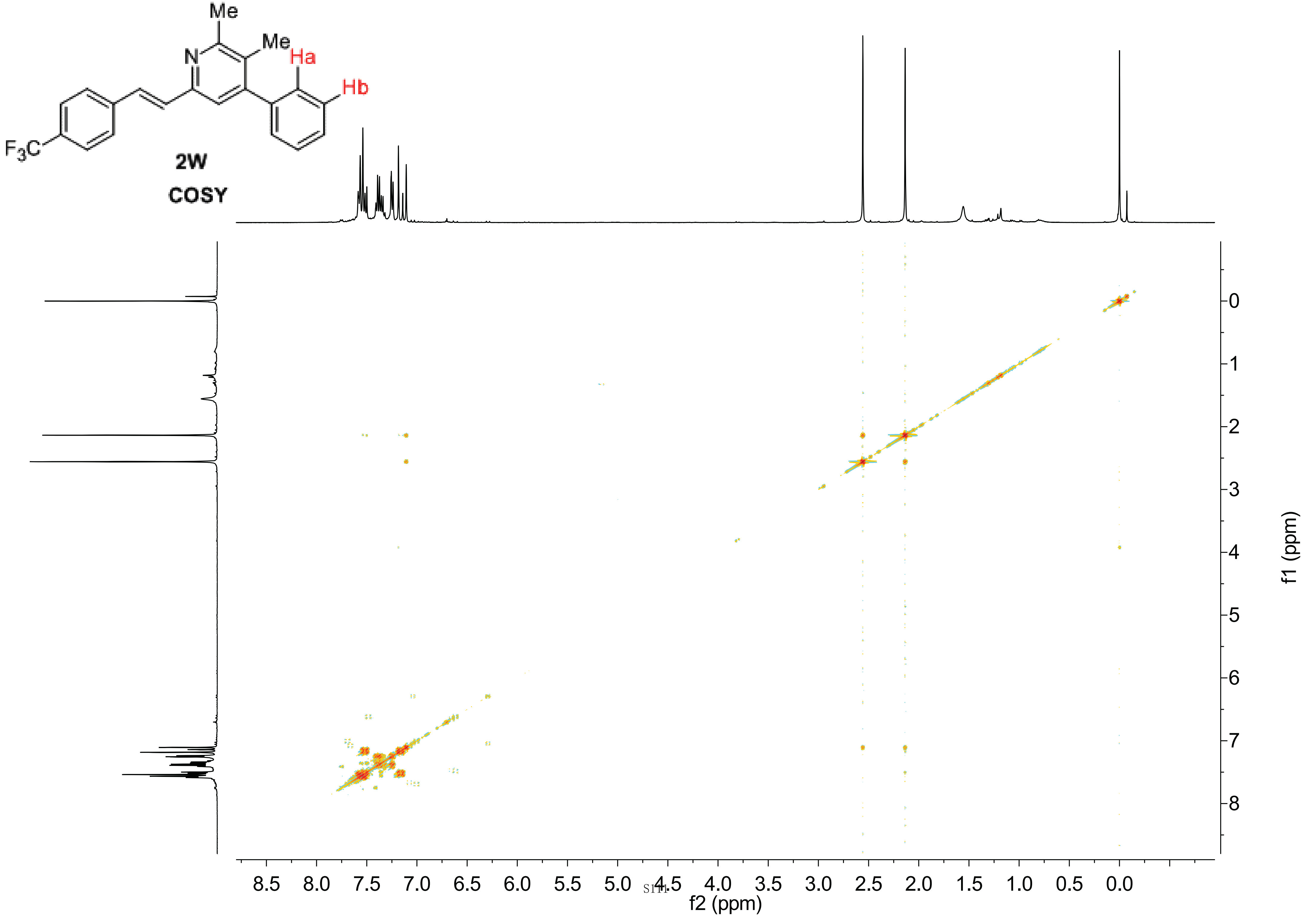


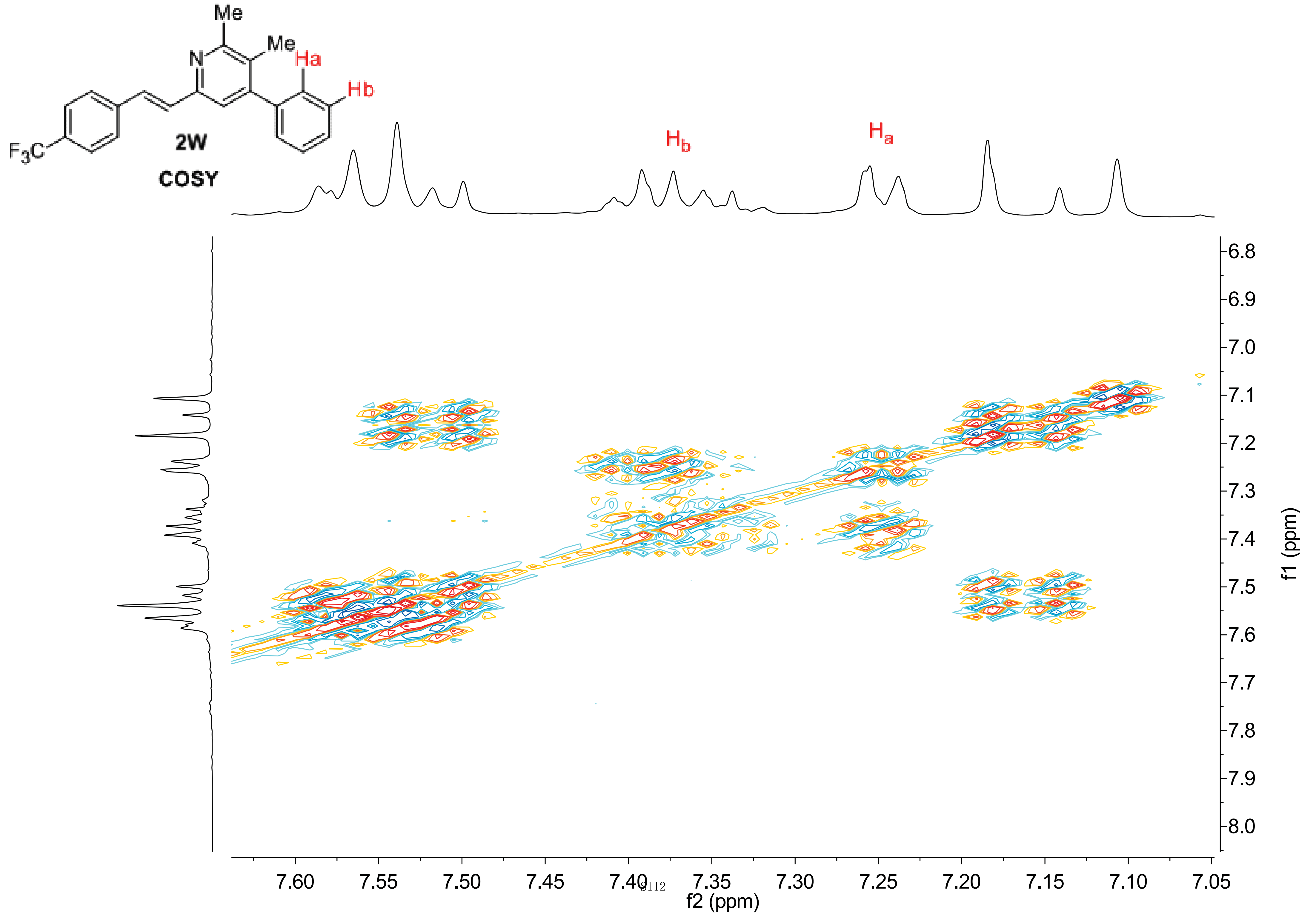
Me
Me
N
CF3
F3C
a
b
2w (a/b = 1:3)
NOESY
f1 (ppm)
f2 (ppm)

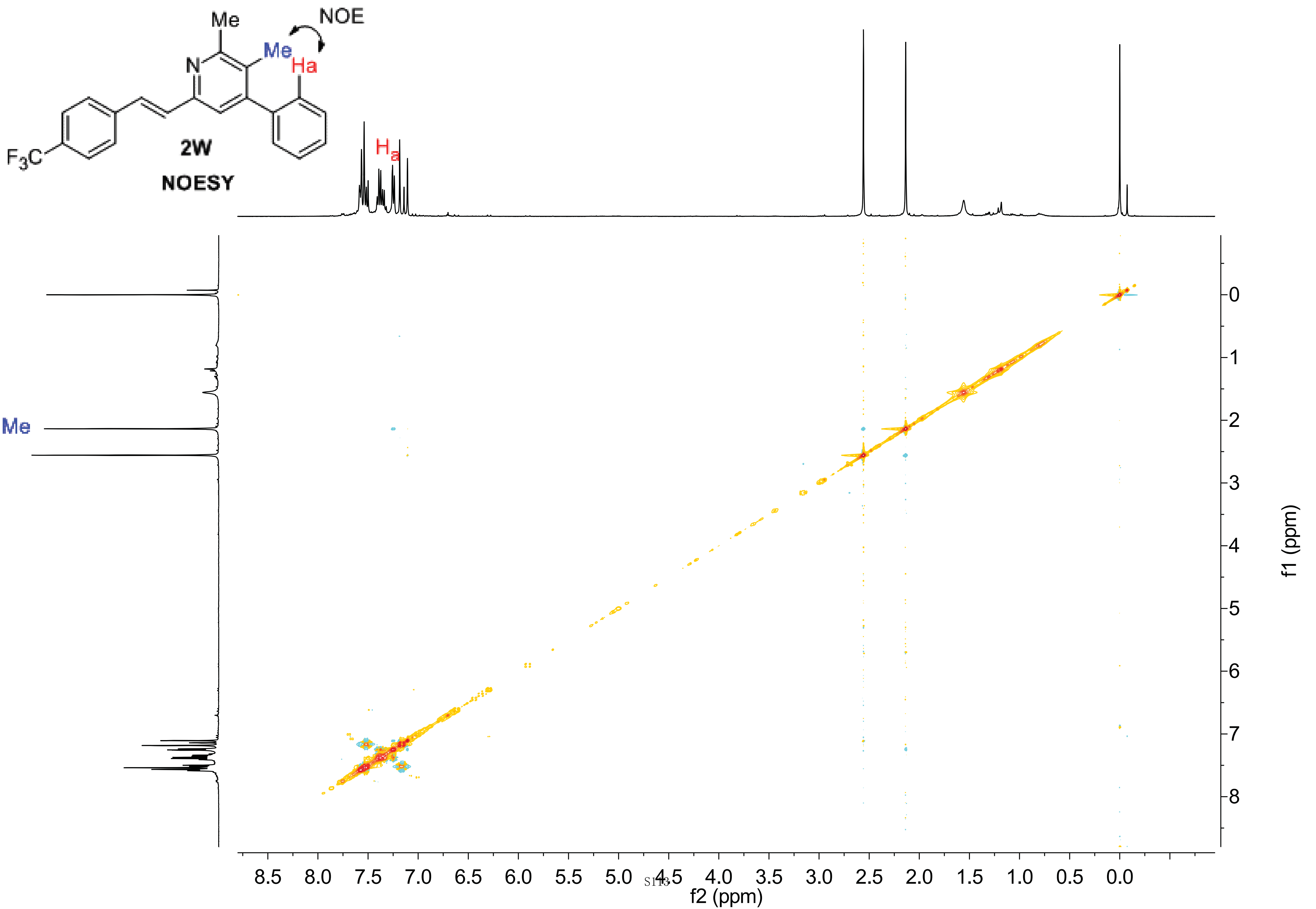

7.584
7.568
7.537
7.522
7.503
7.407
7.393
7.379
7.355
7.341
7.257
7.242
7.186
7.181
7.148
7.110
2.558
2.138
0.000
Me
Me
N
F3C
2W
5.06
3.07
2.10
1.91
1.03
3.04
3.00
8
6
4
2
0 PPM



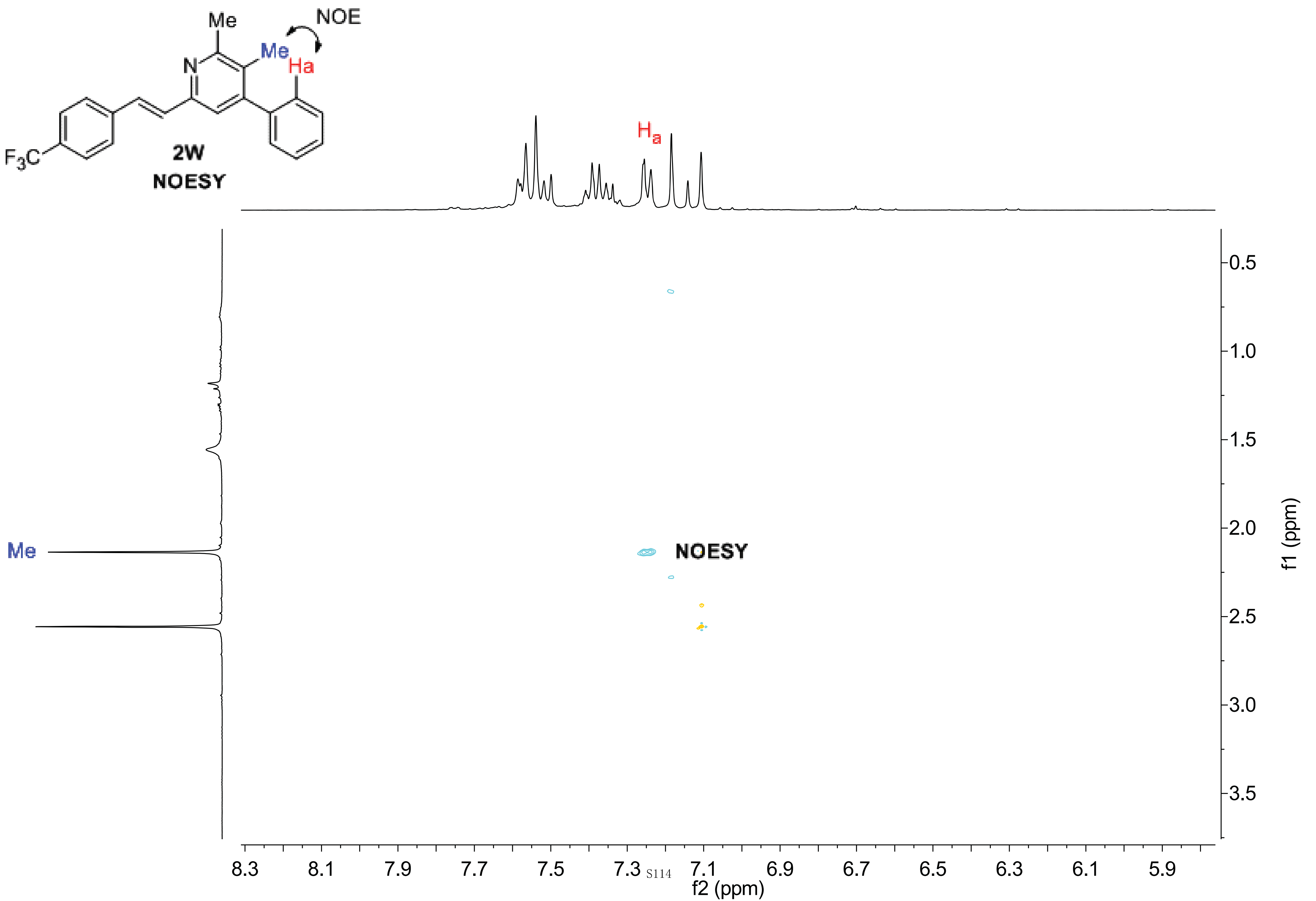
157.974
151.168
150.053
140.469
139.865
130.704
129.881
129.655
129.400
129.050
128.707
128.598
128.379
128.292
127.804
127.133
126.987
125.843
125.625
125.595
125.268
123.110
121.077
77.264
77.009
76.754
23.664
16.245
Me
Me
N
F_3C
2W
150
100
50
0 PPM


Me
Me
N
Ha
Hb
F3C
2W
COSY
f1 (ppm)
f2 (ppm)

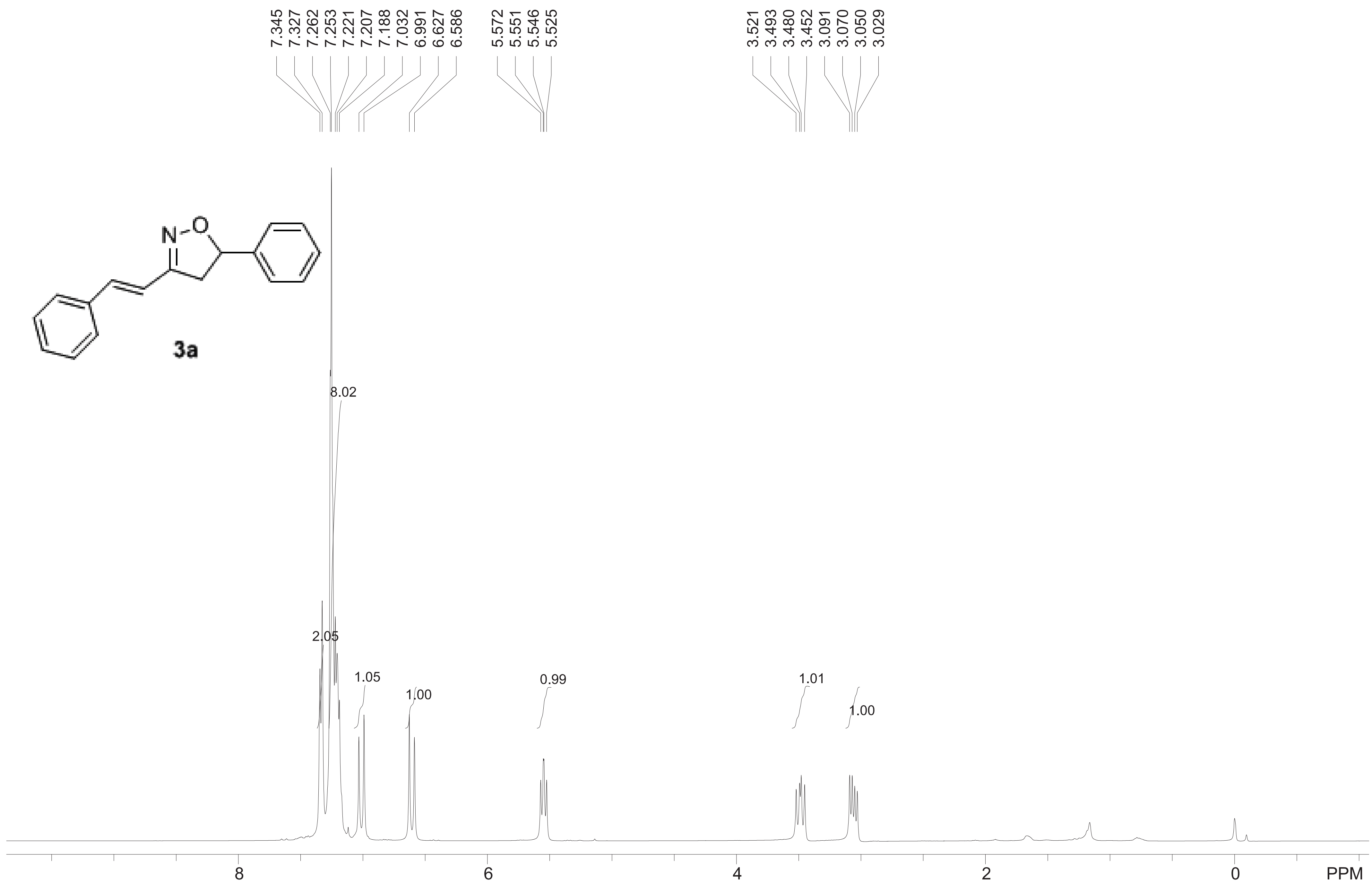

Me
Me
N
Ha
Hb
F3C
2W
COSY
Hb
Ha
f1 (ppm)
f2 (ppm)

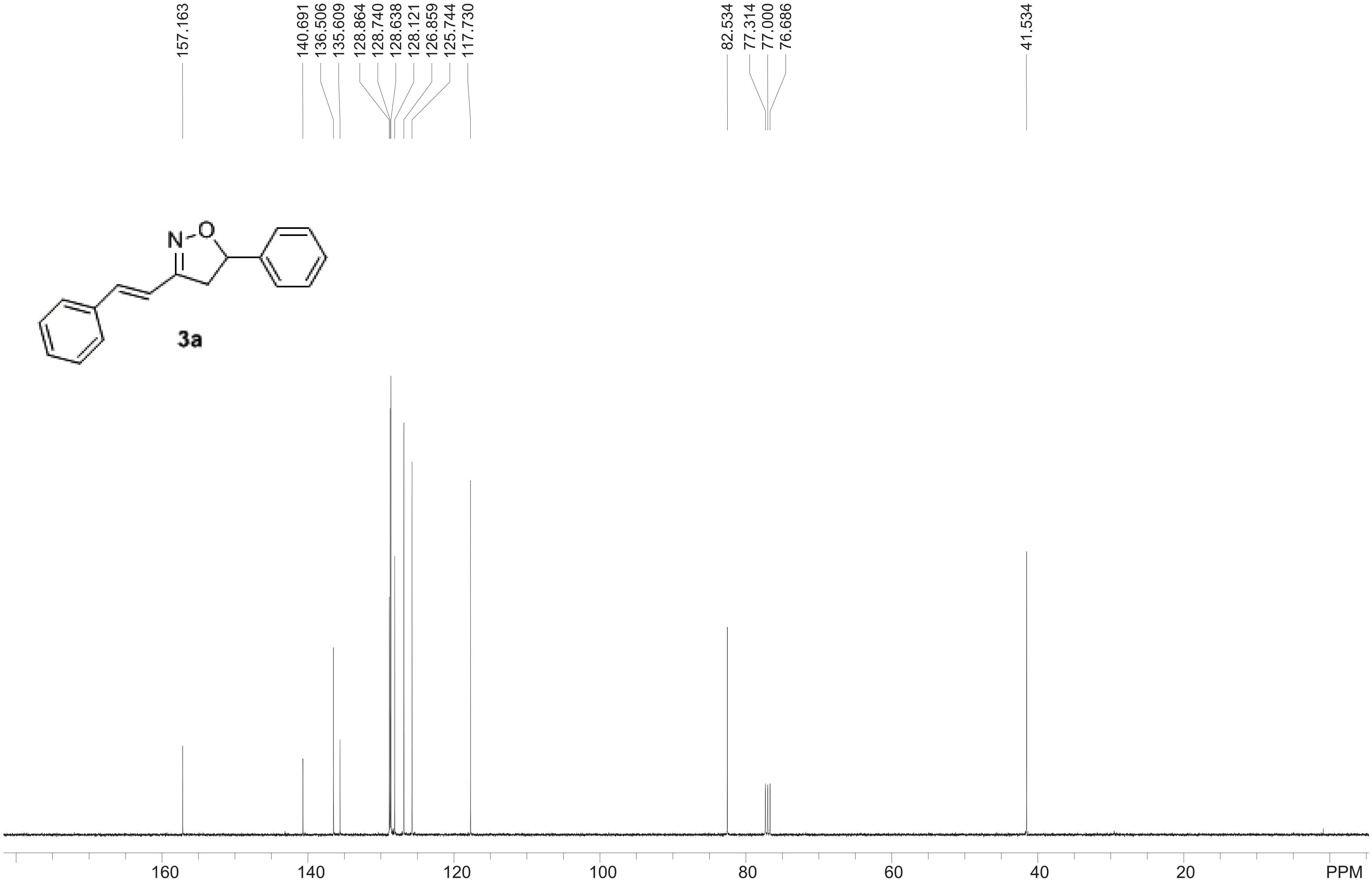


Me
NOE
Me
Ha
N
F3C
2W
NOESY
Ha
Me
f1 (ppm)
f2 (ppm)

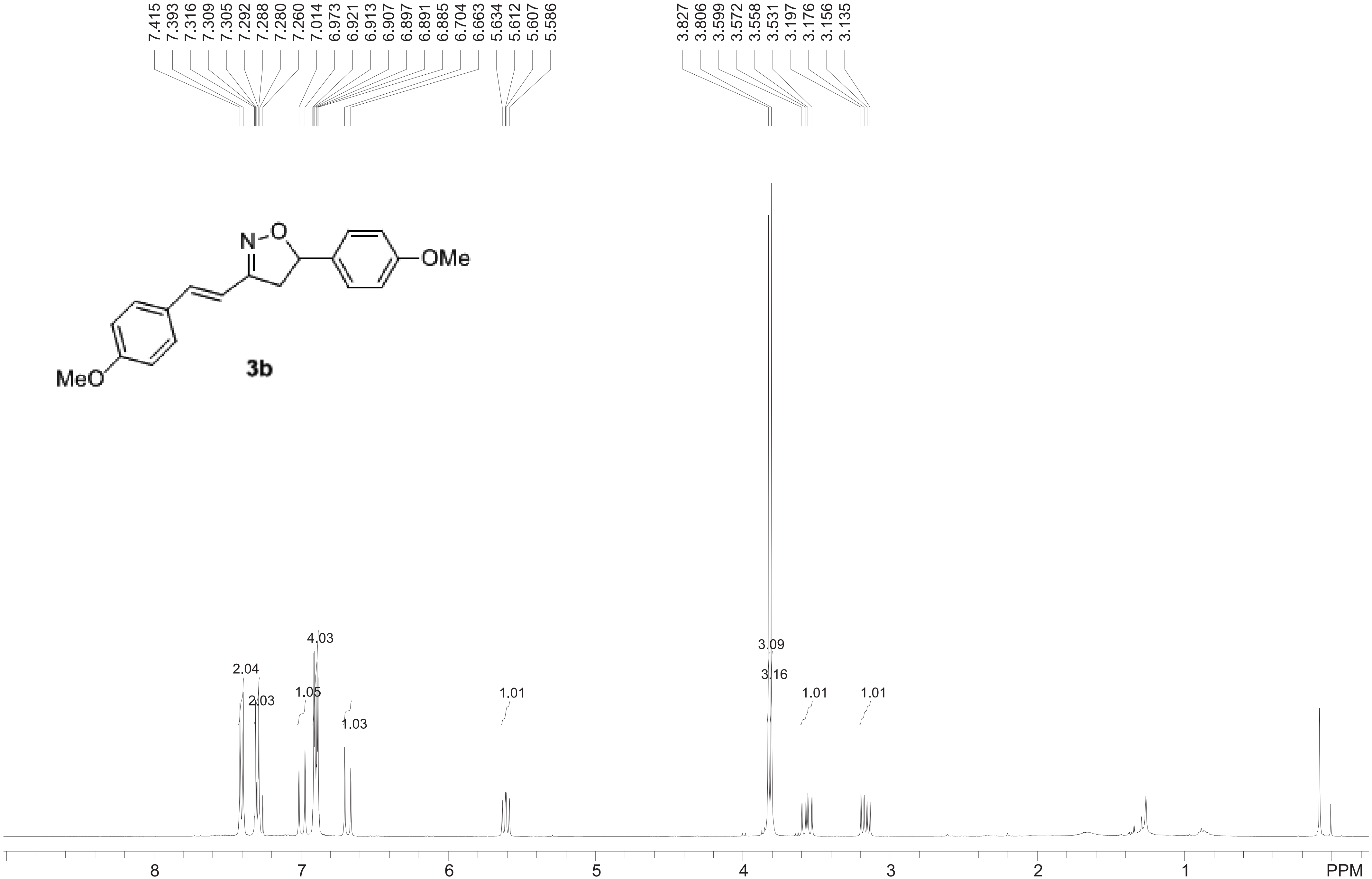


Me
NOE
Me
Ha
N
F_3C
2W
NOESY
H_a
Me
NOESY
f1 (ppm)
f2 (ppm)

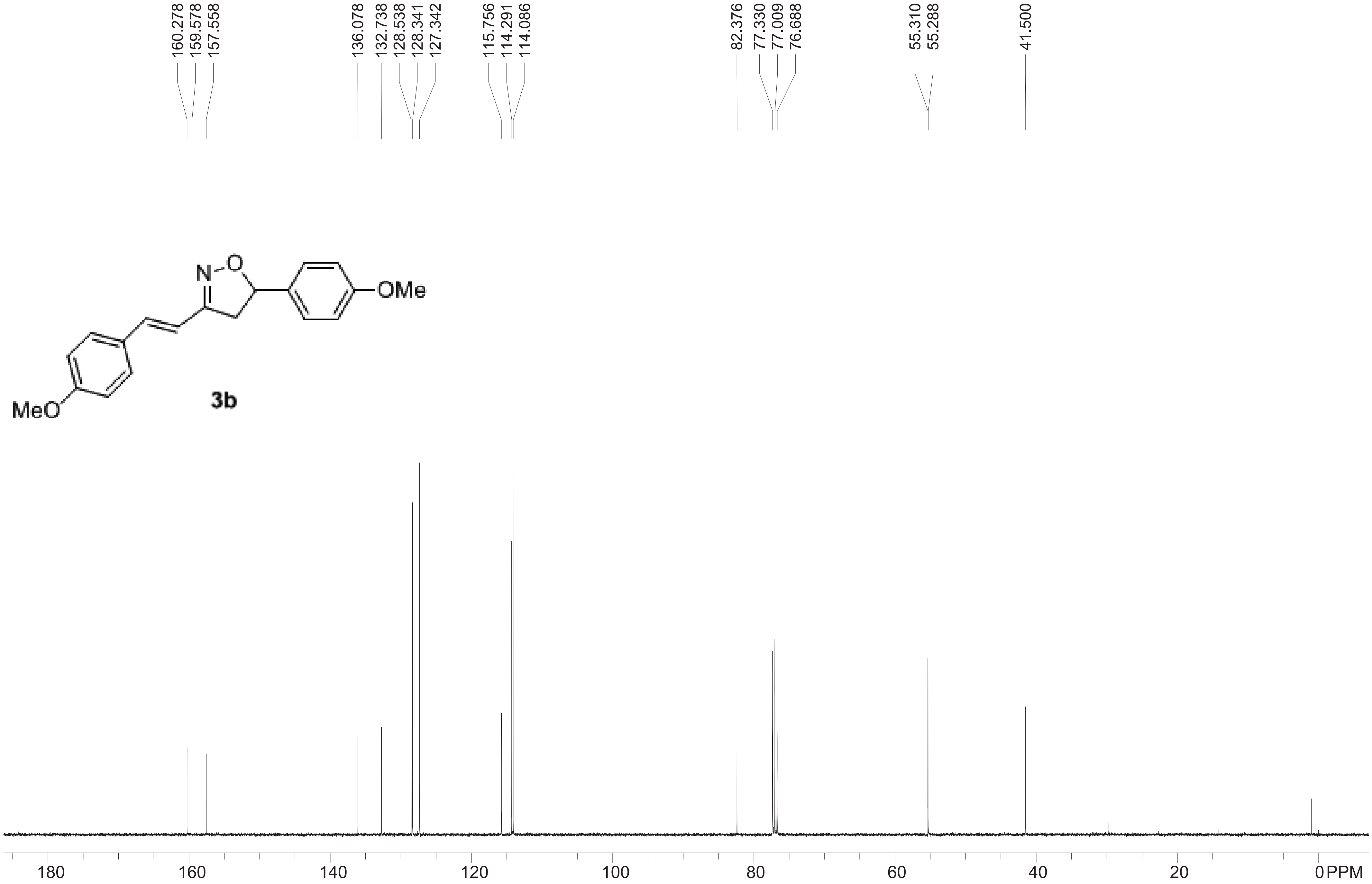

7.345
7.327
7.262
7.253
7.221
7.207
7.188
7.032
6.991
6.627
6.586
5.572
5.551
5.546
5.525
3.521
3.493
3.480
3.452
3.091
3.070
3.050
3.029
N
O
3a
8.02
2.05
1.05
1.00
0.99
1.01
1.00
8
6
4
2
0
PPM

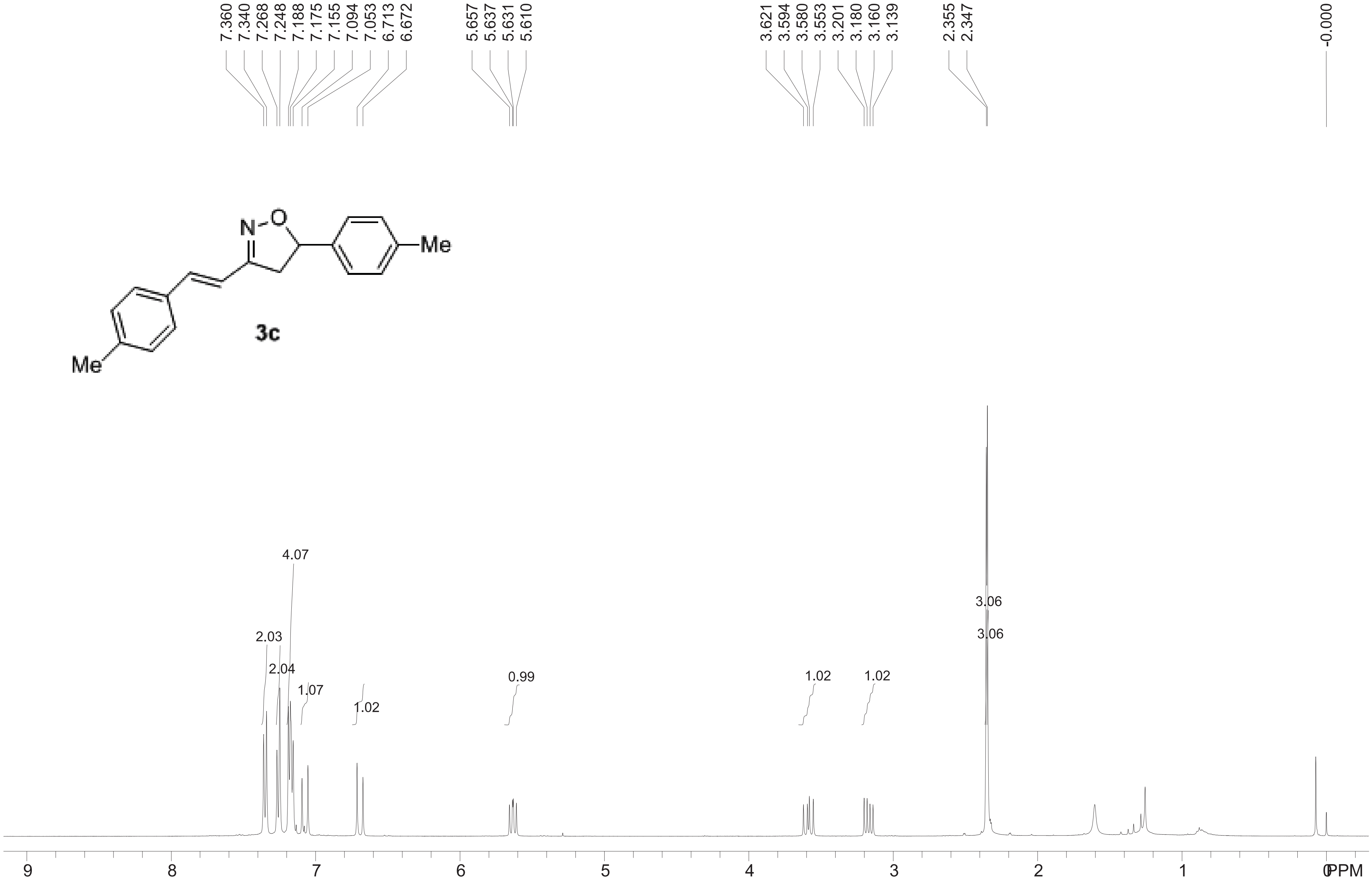

157.163
140.691
136.506
135.609
128.864
128.740
128.638
128.121
126.859
125.744
117.730
82.534
77.314
77.000
76.686
41.534
160
140
120
100
80
60
40
20
PPM
N
O

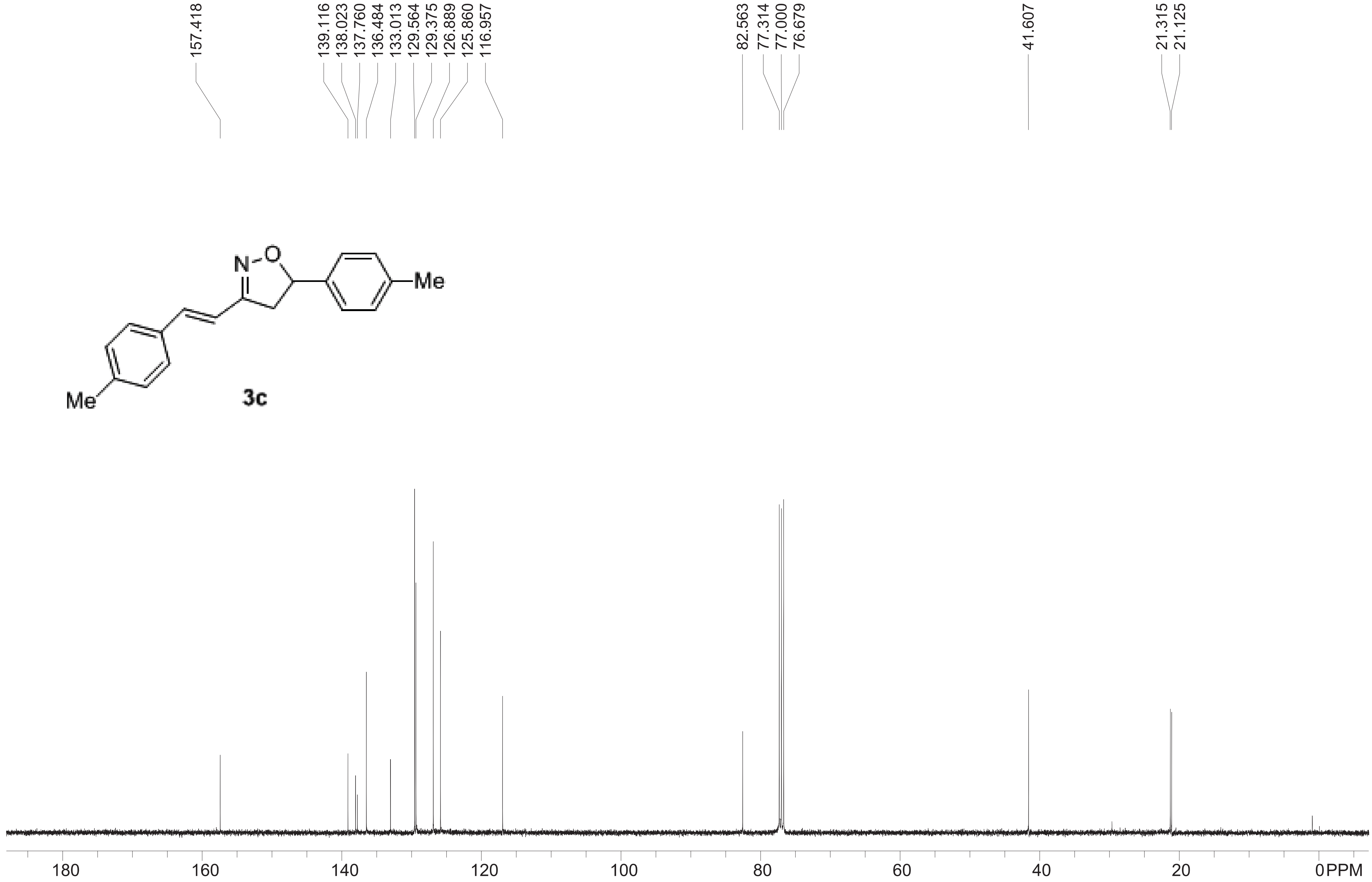
3a

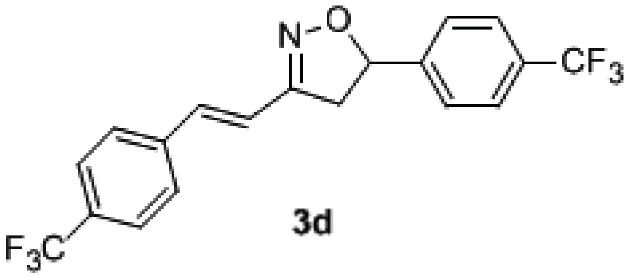


3b
7.415
7.393
7.316
7.309
7.305
7.292
7.288
7.280
7.260
7.014
6.973
6.921
6.913
6.907
6.897
6.891
6.885
6.704
6.663
5.634
5.612
5.607
5.586
3.827
3.806
3.599
3.572
3.558
3.531
3.197
3.176
3.156
3.135
2.04
2.03
4.03
1.05
1.03
1.01
3.09
3.16
1.01
1.01
8
7
6
5
4
3
2
1
PPM


160.278
159.578
157.558
136.078
132.738
128.538
128.341
127.342
115.756
114.291
114.086
82.376
77.330
77.009
76.688
55.310
55.288
41.500
N
O
OMe
MeO
3b
180
160
140
120
100
80
60
40
20
0 PPM

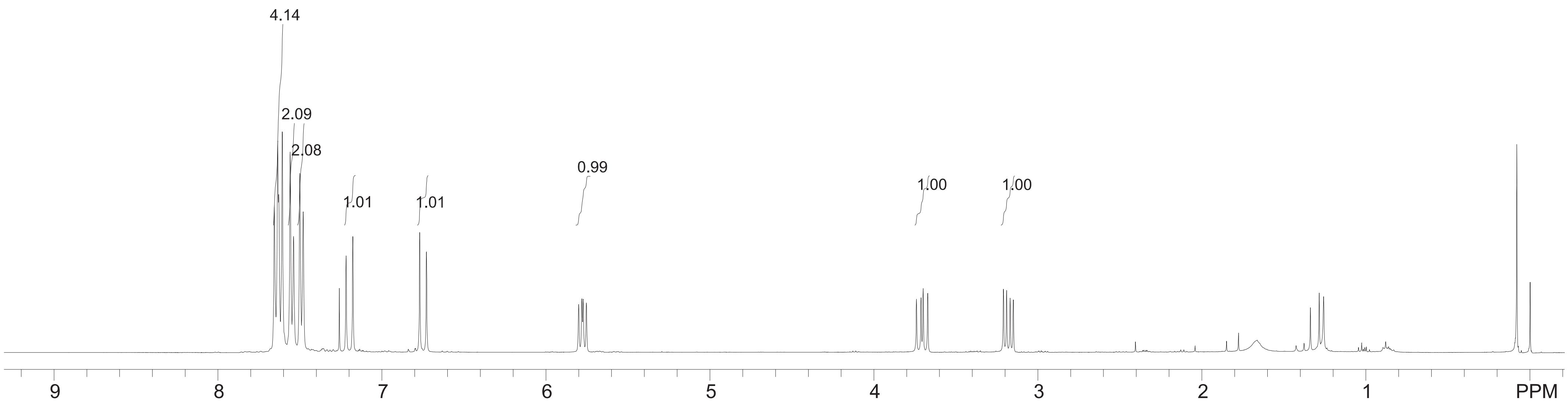

7.360
7.340
7.268
7.248
7.188
7.175
7.155
7.094
7.053
6.713
6.672
5.657
5.637
5.631
5.610
3.621
3.594
3.580
3.553
3.201
3.180
3.160
3.139
2.355
2.347
-0.000
N
O
Me
Me
3c
4.07
2.03
2.04
1.07
1.02
0.99
1.02
1.02
3.06
3.06
9
8
7
6
5
4
3
2
1
0
PPM


157.418
139.116
138.023
137.760
136.484
133.013
129.564
129.375
126.889
125.860
116.957
82.563
77.314
77.000
76.679
41.607
21.315
21.125
N
O
Me
Me
3c
180
160
140
120
100
80
60
40
20
0 PPM

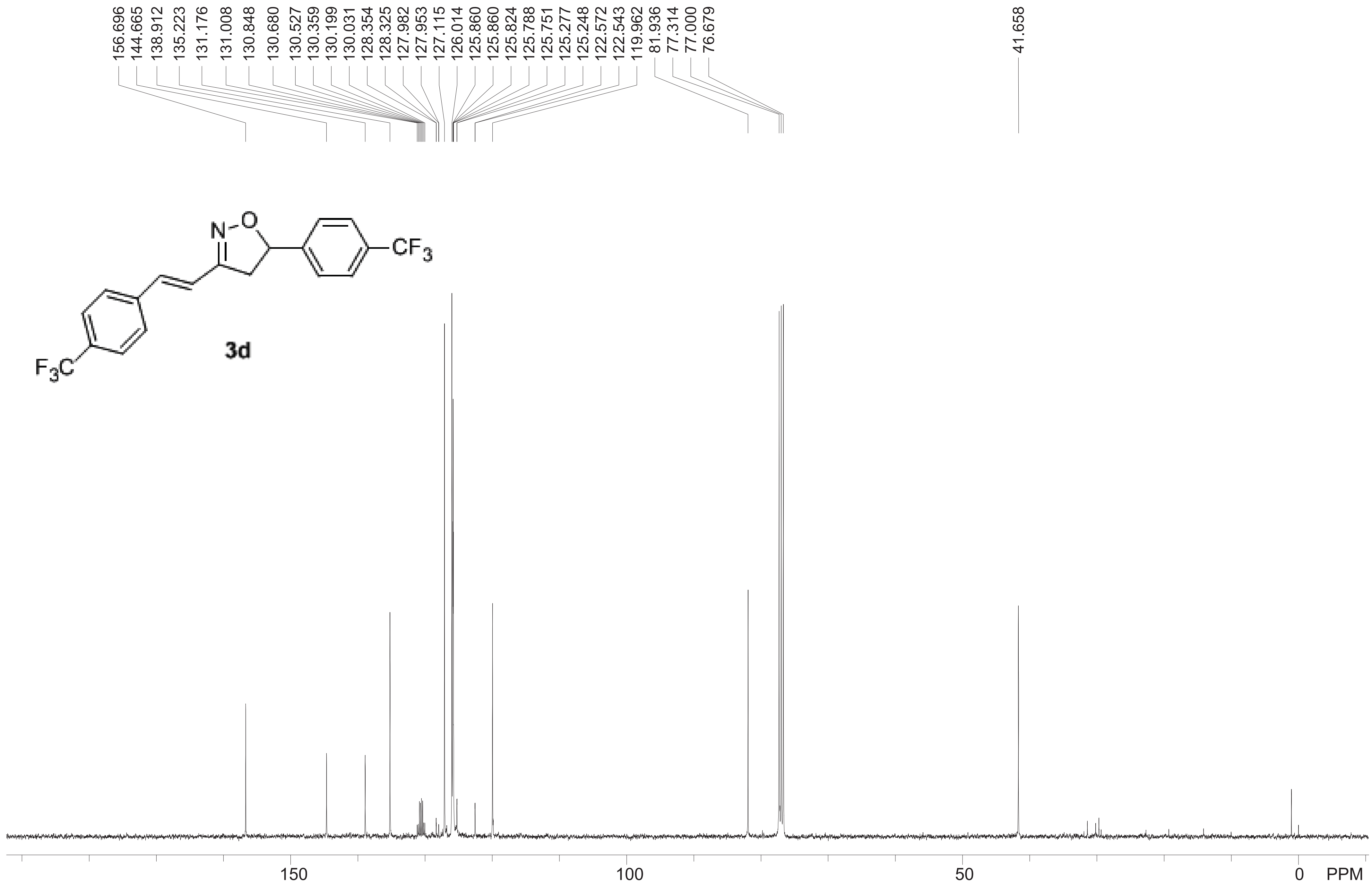


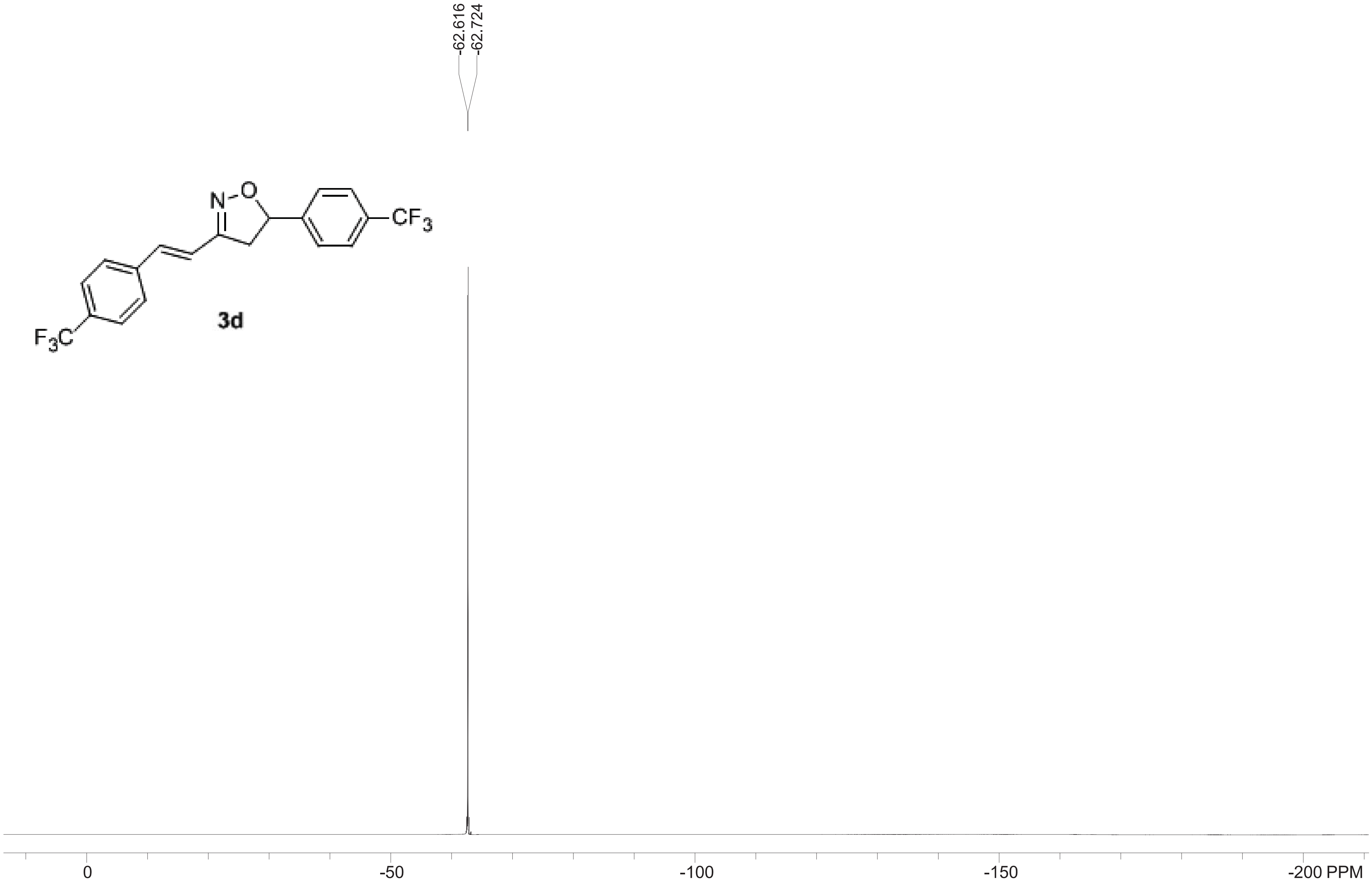
N
O
CF3
F3C

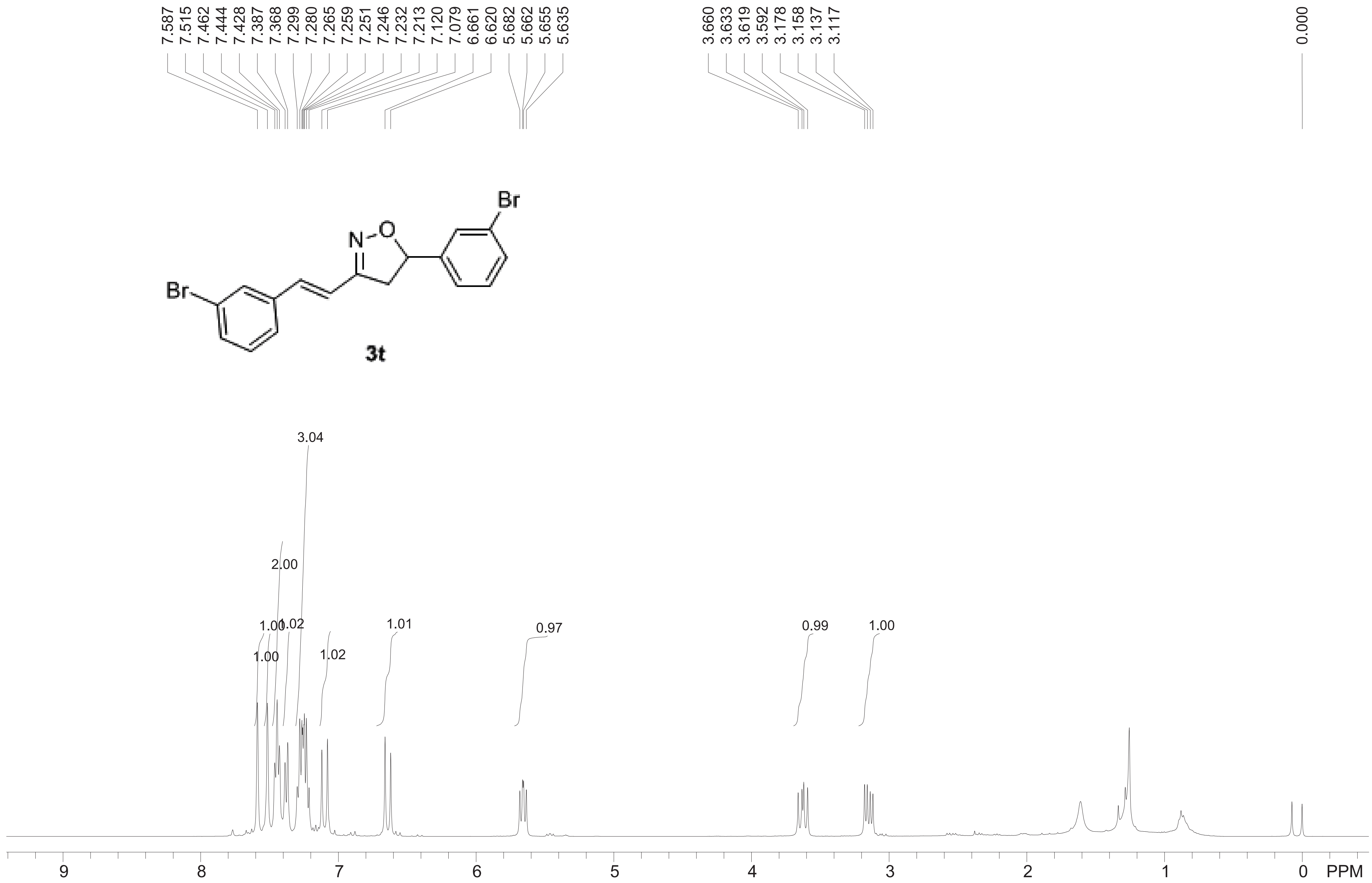
3d

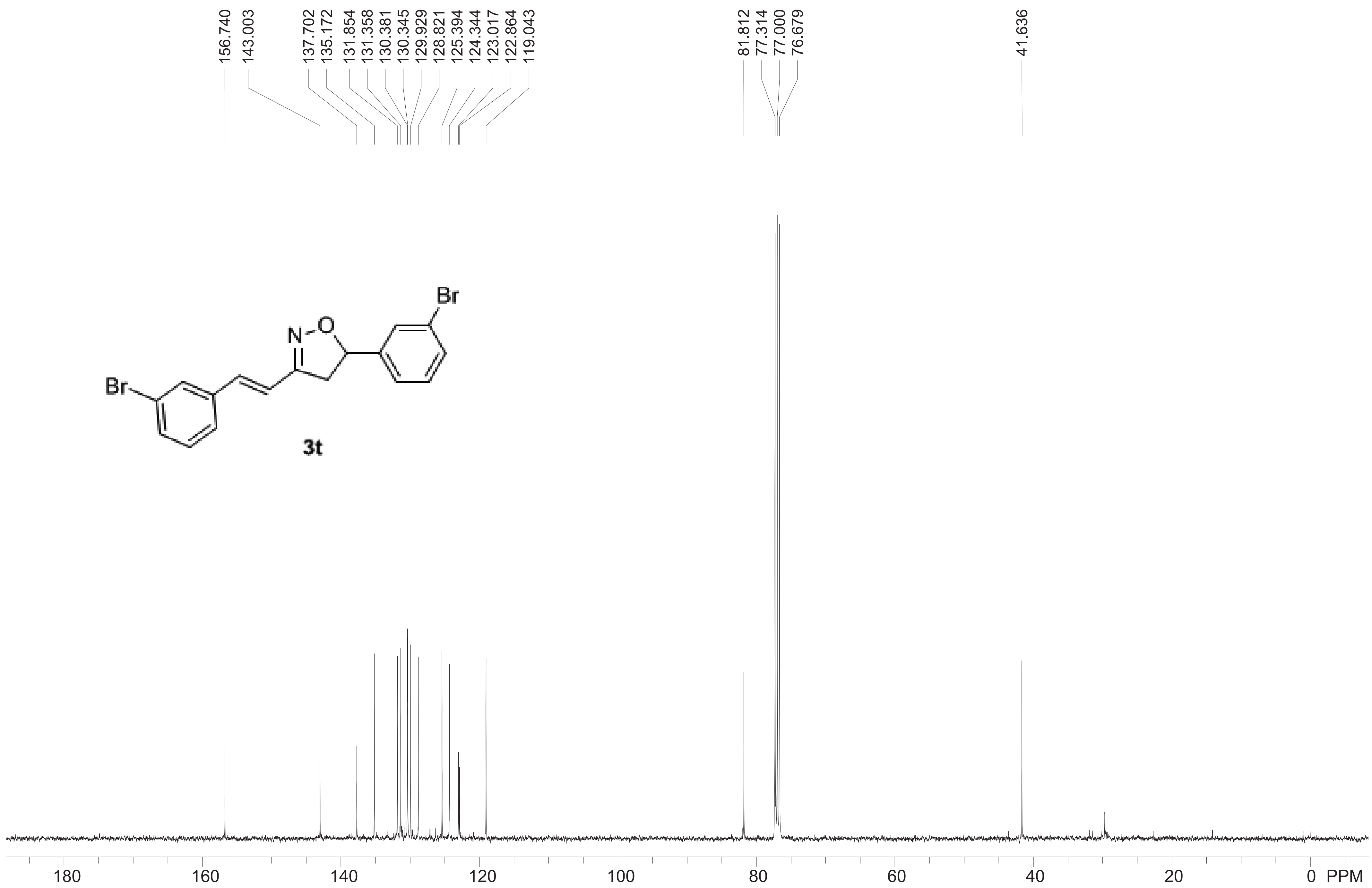


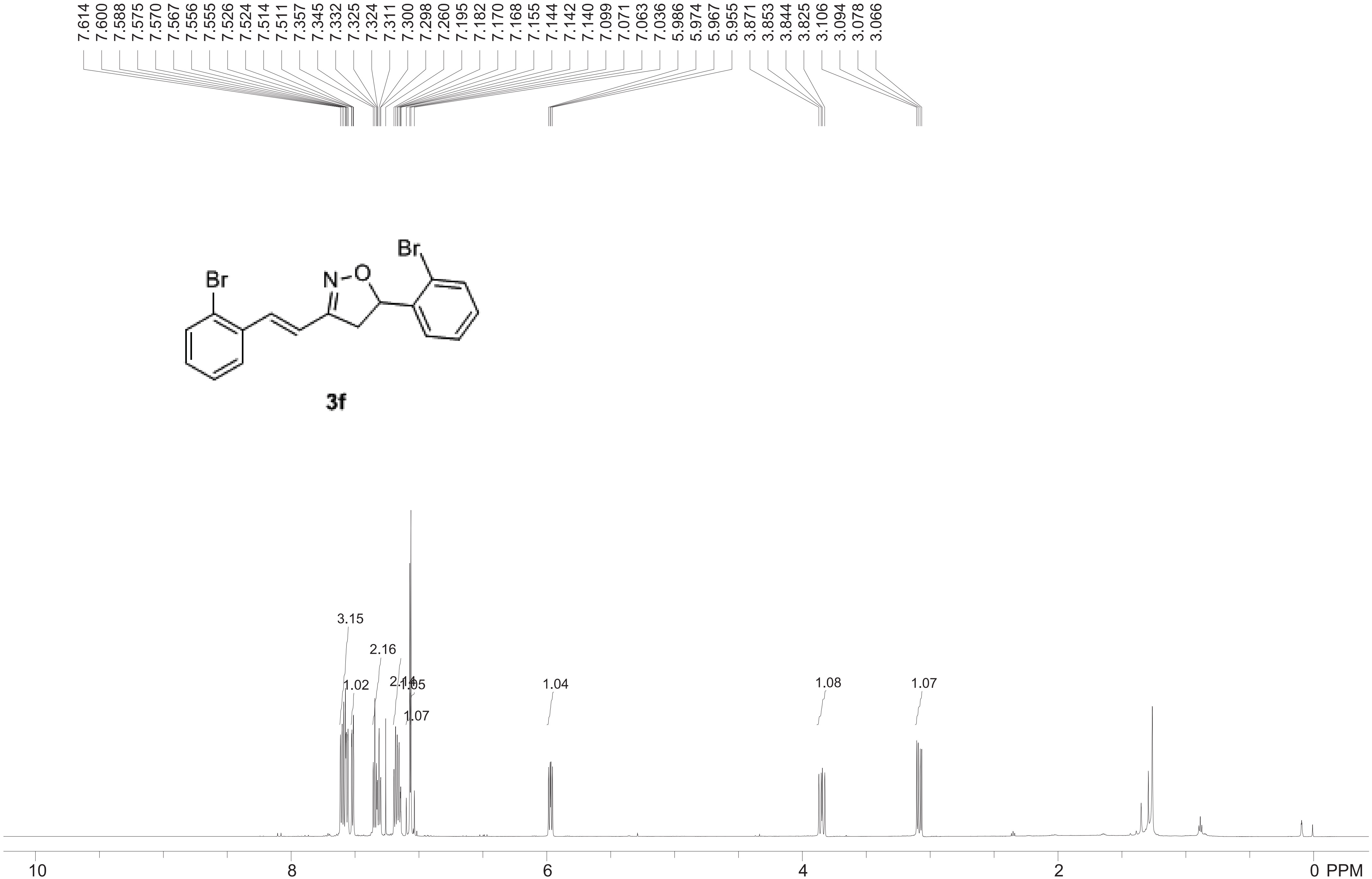
7.656
7.635
7.629
7.608
7.559
7.539
7.501
7.480
7.260
7.218
7.177
6.770
6.728
5.800
5.781
5.772
5.753
3.740
3.712
3.699
3.671
3.209
3.190
3.168
3.149
-0.000
4.14
2.09
2.08
1.01
1.01
0.99
1.00
1.00
9
8
7
6
5
4
3
2
1
PPM

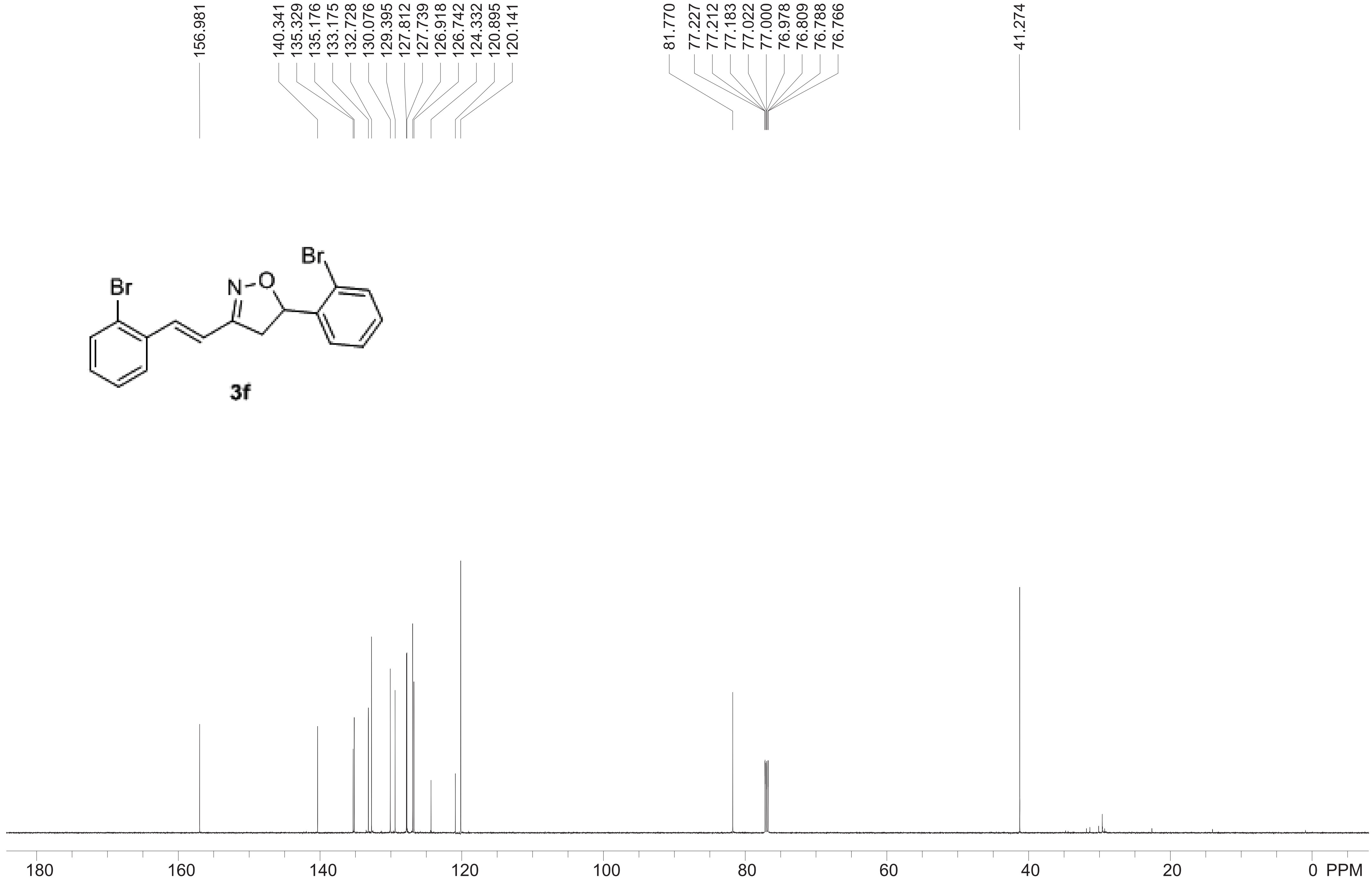

156.696
144.665
138.912
135.223
131.176
131.008
130.848
130.680
130.527
130.359
130.199
130.031
128.354
128.325
127.982
127.953
127.115
126.014
125.860
125.860
125.824
125.788
125.751
125.277
125.248
122.572
122.543
119.962
81.936
77.314
77.000
76.679
41.658
N-O
CF3
F3C
3d
150
100
50
0
PPM

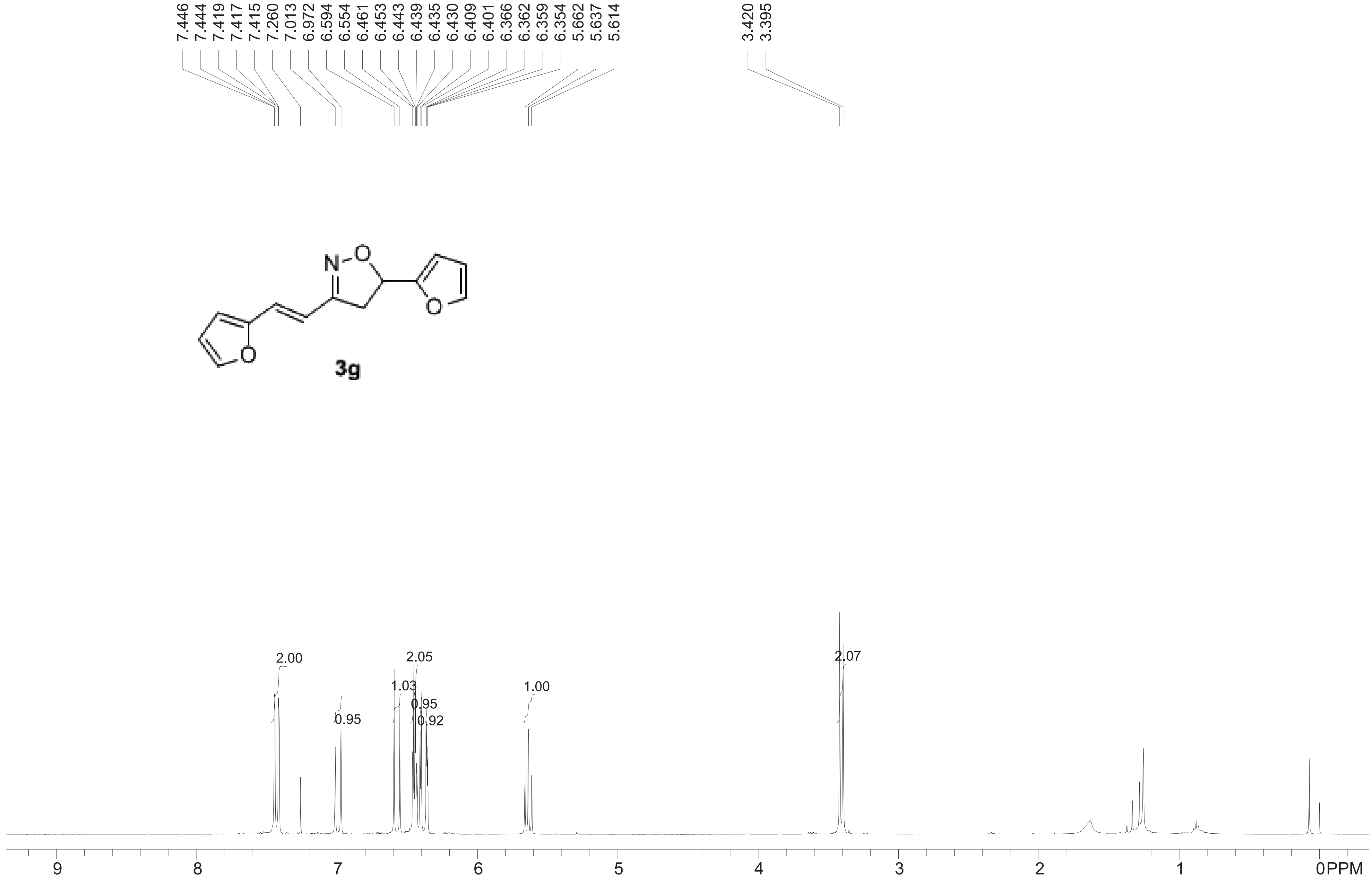

-62.616
-62.724
N
O
CF3
F3C
3d
0
-50
-100
-150
-200 PPM

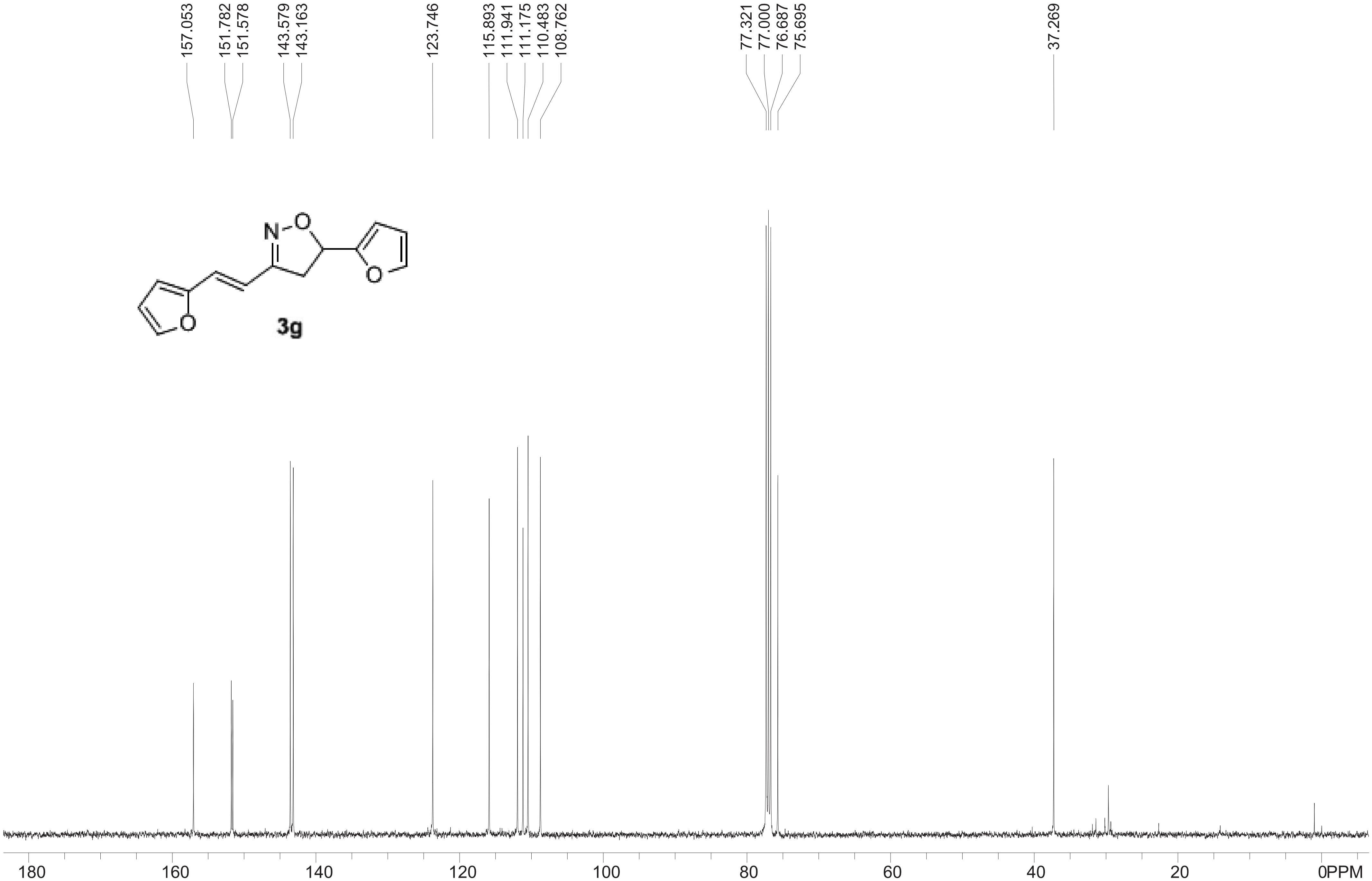

7.587 7.515 7.462 7.444 7.428 7.387 7.368 7.299 7.280 7.265 7.259 7.251 7.246 7.232 7.213 7.120 7.079 6.661 6.620 5.682 5.662 5.655 5.635
3.660 3.633 3.619 3.592 3.178 3.158 3.137 3.117
0.000
Br
N
O
Br
3t
3.04
2.00
1.00
1.02
1.00
1.02
1.01
0.97
0.99
1.00
9
8
7
6
5
4
3
2
1
0
PPM

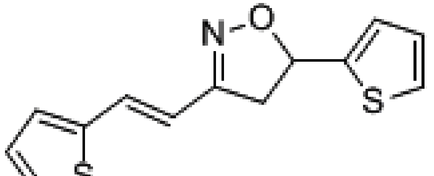

156.740
143.003
137.702
135.172
131.854
131.358
130.381
130.345
129.929
128.821
125.394
124.344
123.017
122.864
119.043
81.812
77.314
77.000
76.679
41.636
Br
N
O
Br
3t
180
160
140
120
100
80
60
40
20
0 PPM


7.614 7.600 7.588 7.575 7.570 7.567 7.556 7.555 7.526 7.524 7.514 7.511 7.357 7.345 7.332 7.325 7.324 7.311 7.300 7.298 7.260 7.195 7.182 7.170 7.168 7.155 7.144 7.142 7.140 7.099 7.071 7.063 7.036 5.986 5.974 5.967 5.955 3.871 3.853 3.844 3.825 3.106 3.094 3.078 3.066
Br
N
O
Br
3f
3.15
2.16
1.02
2.14
1.05
1.07
1.04
1.08
1.07
10
8
6
4
2
0 PPM

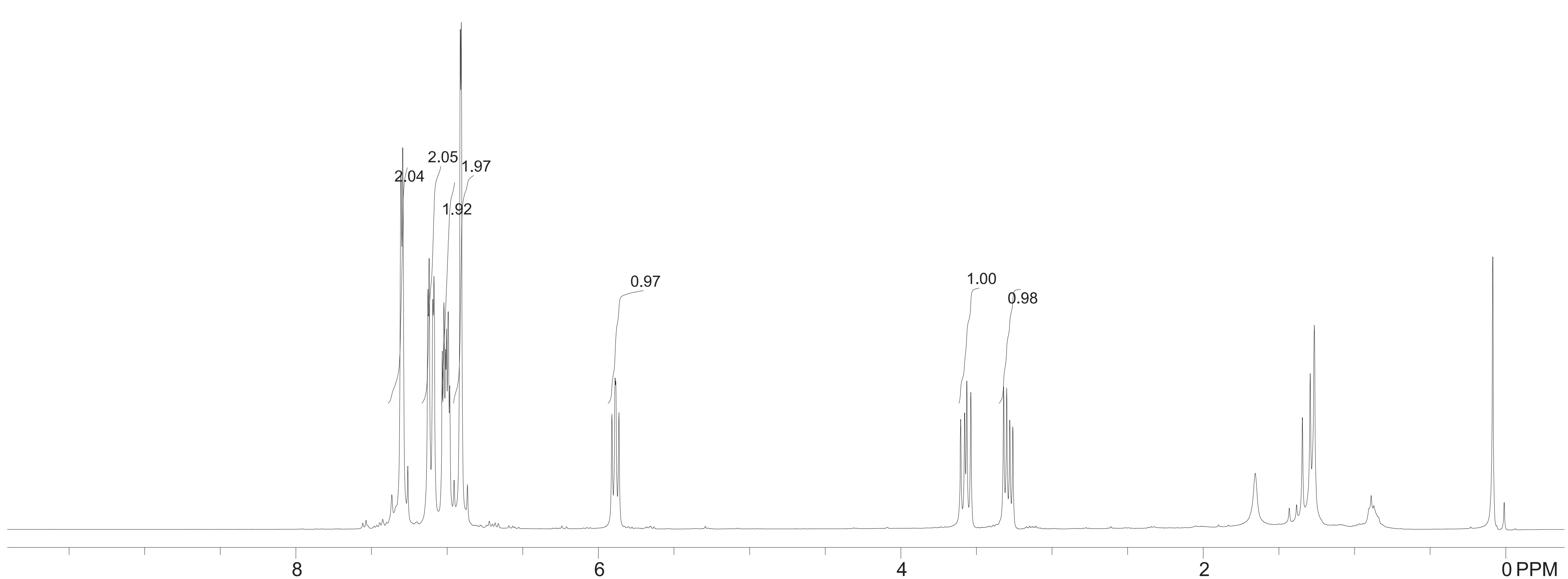

156.981
140.341
135.329
135.176
133.175
132.728
130.076
129.395
127.812
127.739
126.918
126.742
124.332
120.895
120.141
81.770
77.227
77.212
77.183
77.022
77.000
76.978
76.809
76.788
76.766
41.274
Br
N
O
Br
3f
180
160
140
120
100
80
60
40
20
0 PPM


7.446
7.444
7.419
7.417
7.415
7.260
7.013
6.972
6.594
6.554
6.461
6.453
6.443
6.439
6.435
6.430
6.409
6.401
6.366
6.362
6.359
6.354
5.662
5.637
5.614
3.420
3.395
N
O
O
O
3g
2.00
0.95
1.03
2.05
0.95
0.92
1.00
2.07
9
8
7
6
5
4
3
2
1
0PPM

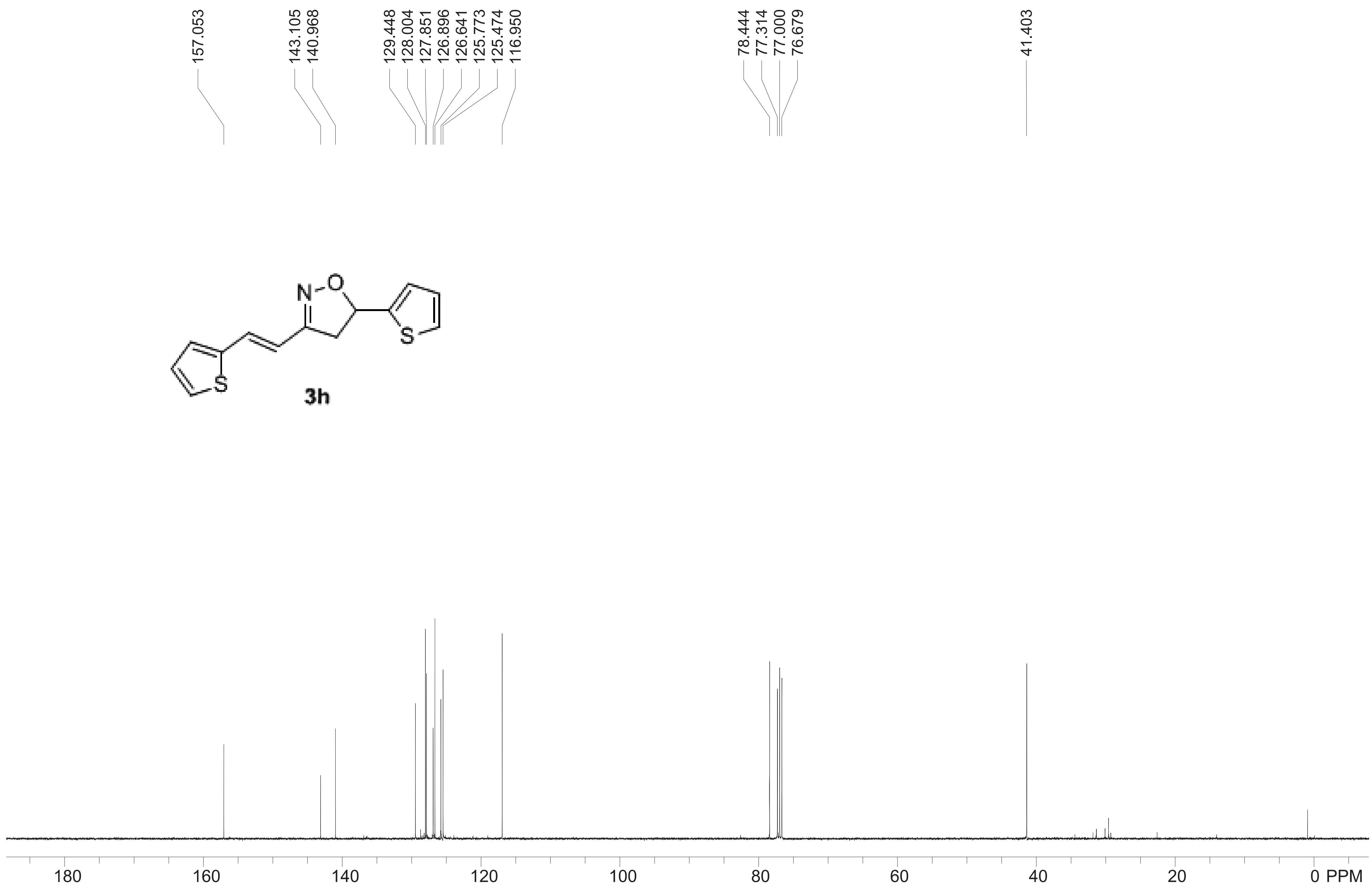

157.053
151.782
151.578
143.579
143.163
123.746
115.893
111.941
111.175
110.483
108.762
77.321
77.000
76.687
75.695
37.269
3g
180
160
140
120
100
80
60
40
20
0PPM

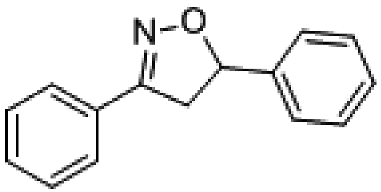


3h

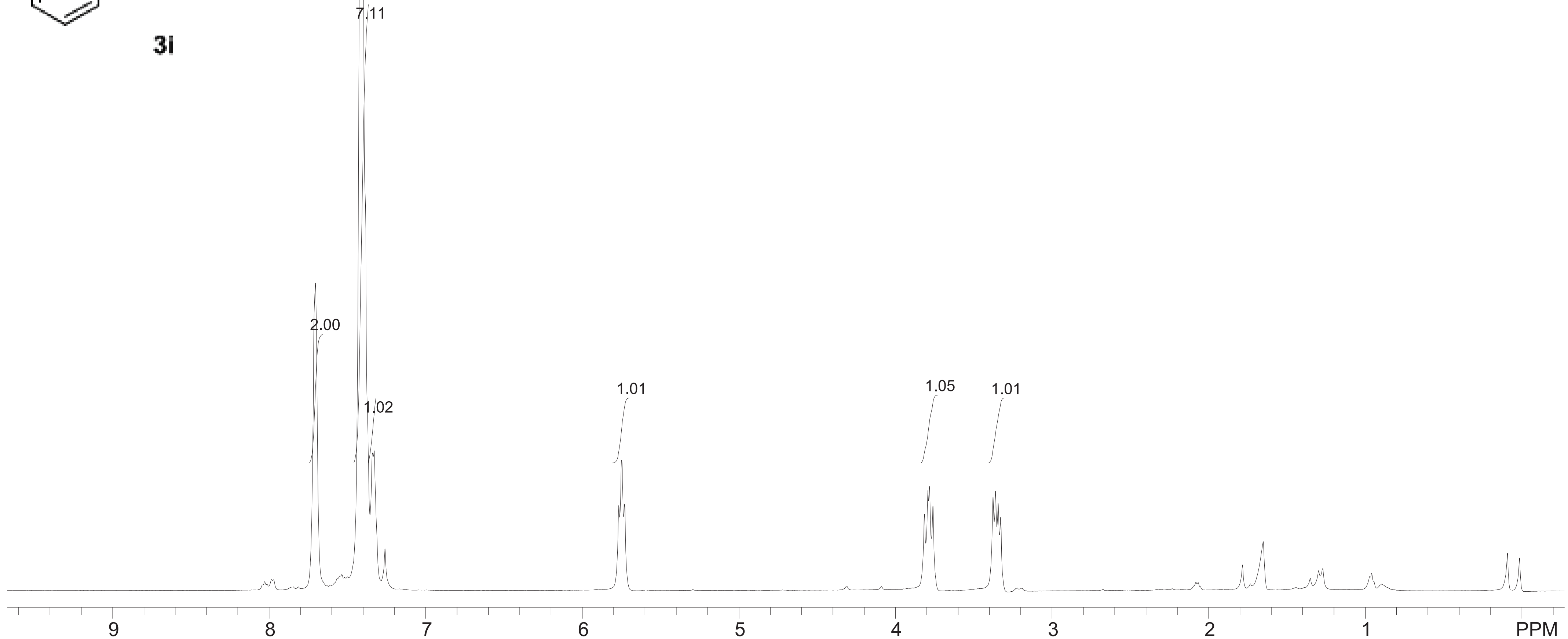


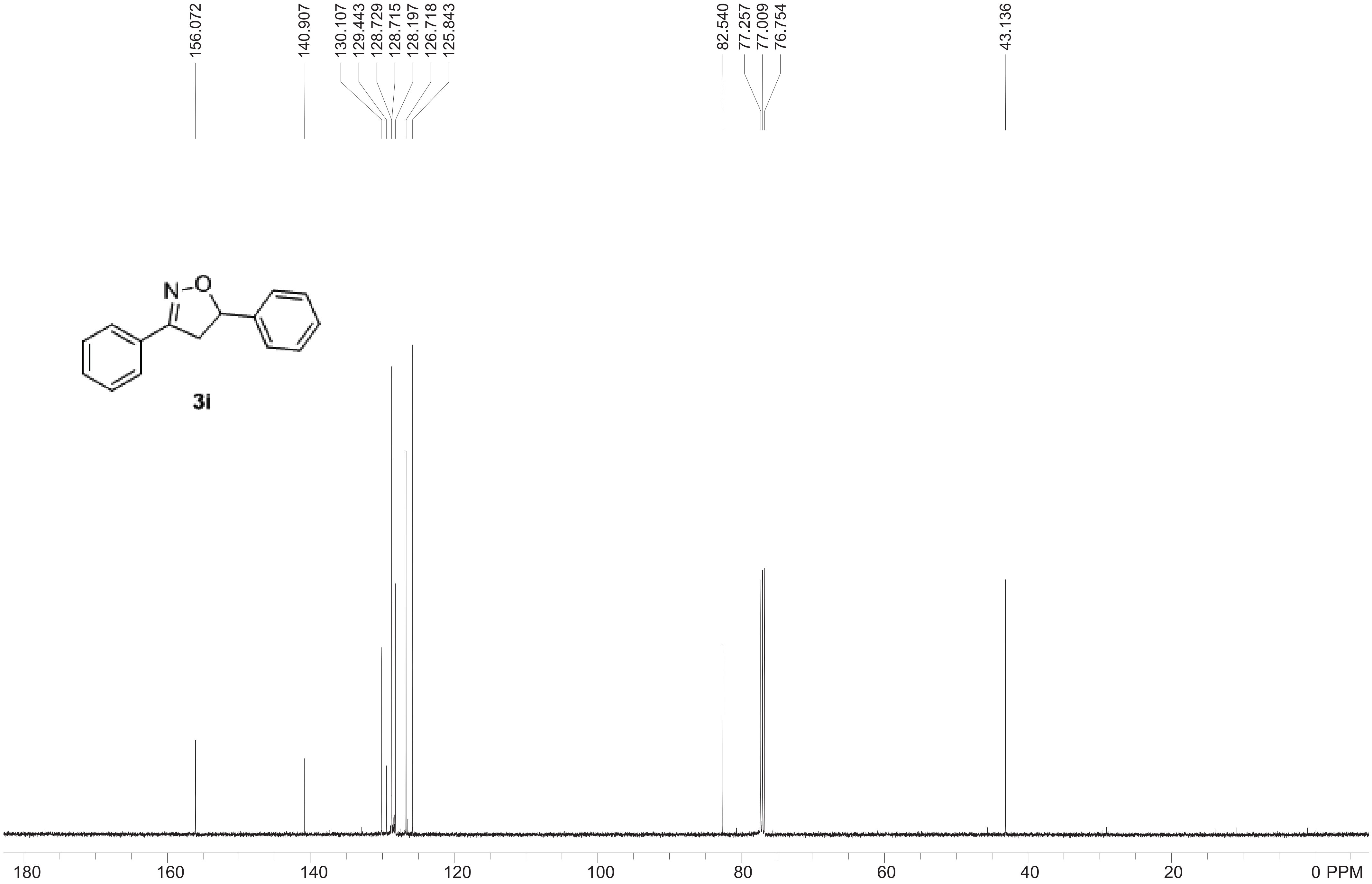
7.306 7.294 7.260 7.127 7.119 7.094 7.087 7.033 7.021 7.011 7.004 6.993 6.983 6.913 6.907 5.911 5.890 5.886 5.864
3.605 3.578 3.564 3.537 3.321 3.300 3.280 3.259
2.04
2.05
1.92
1.97
0.97
1.00
0.98
8
6
4
2
0 PPM

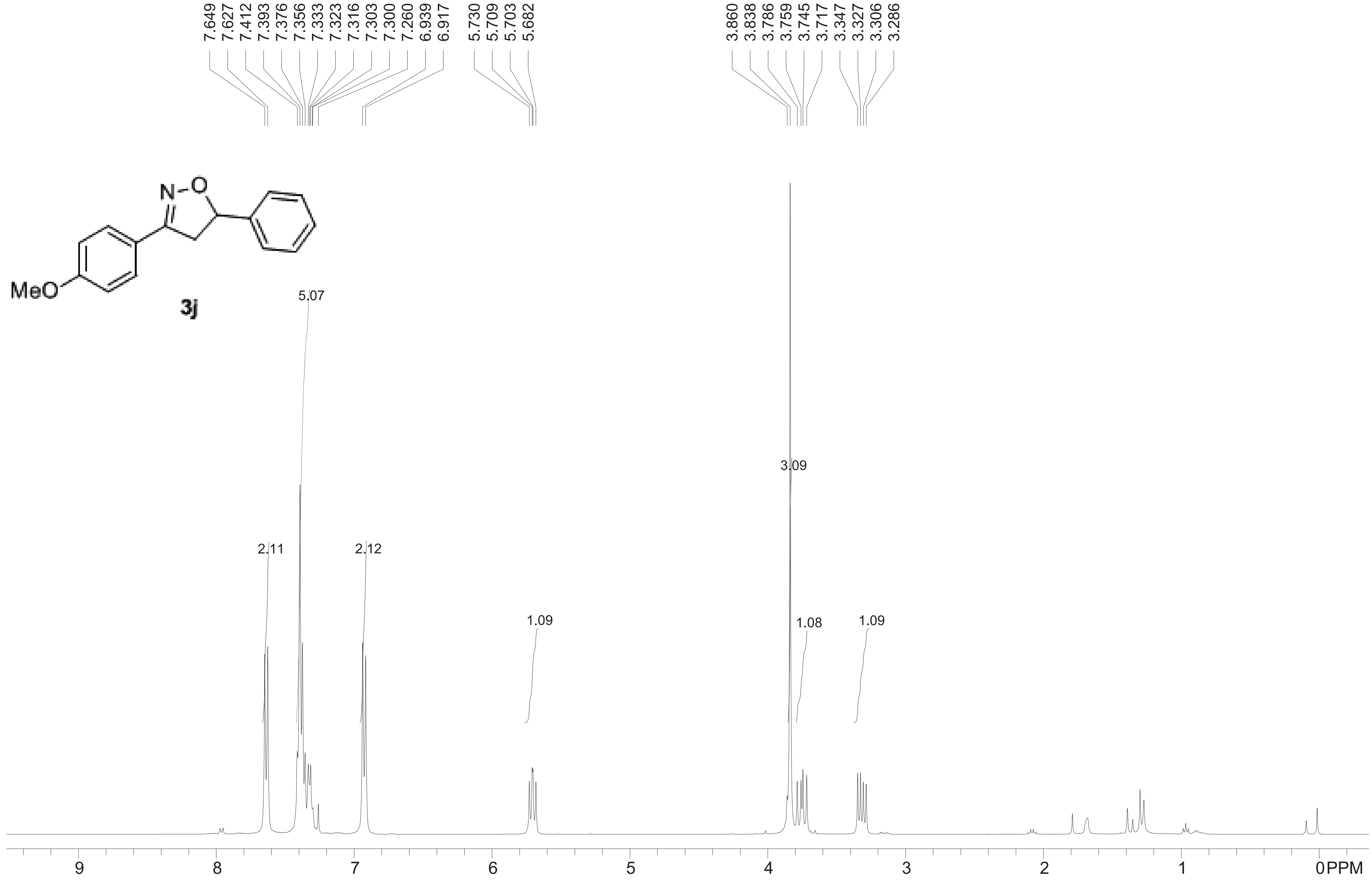

157.053
143.105
140.968
129.448
128.004
127.851
126.896
126.641
125.773
125.474
116.950
78.444
77.314
77.000
76.679
41.403
N
O
S
S
3h
180
160
140
120
100
80
60
40
20
0 PPM

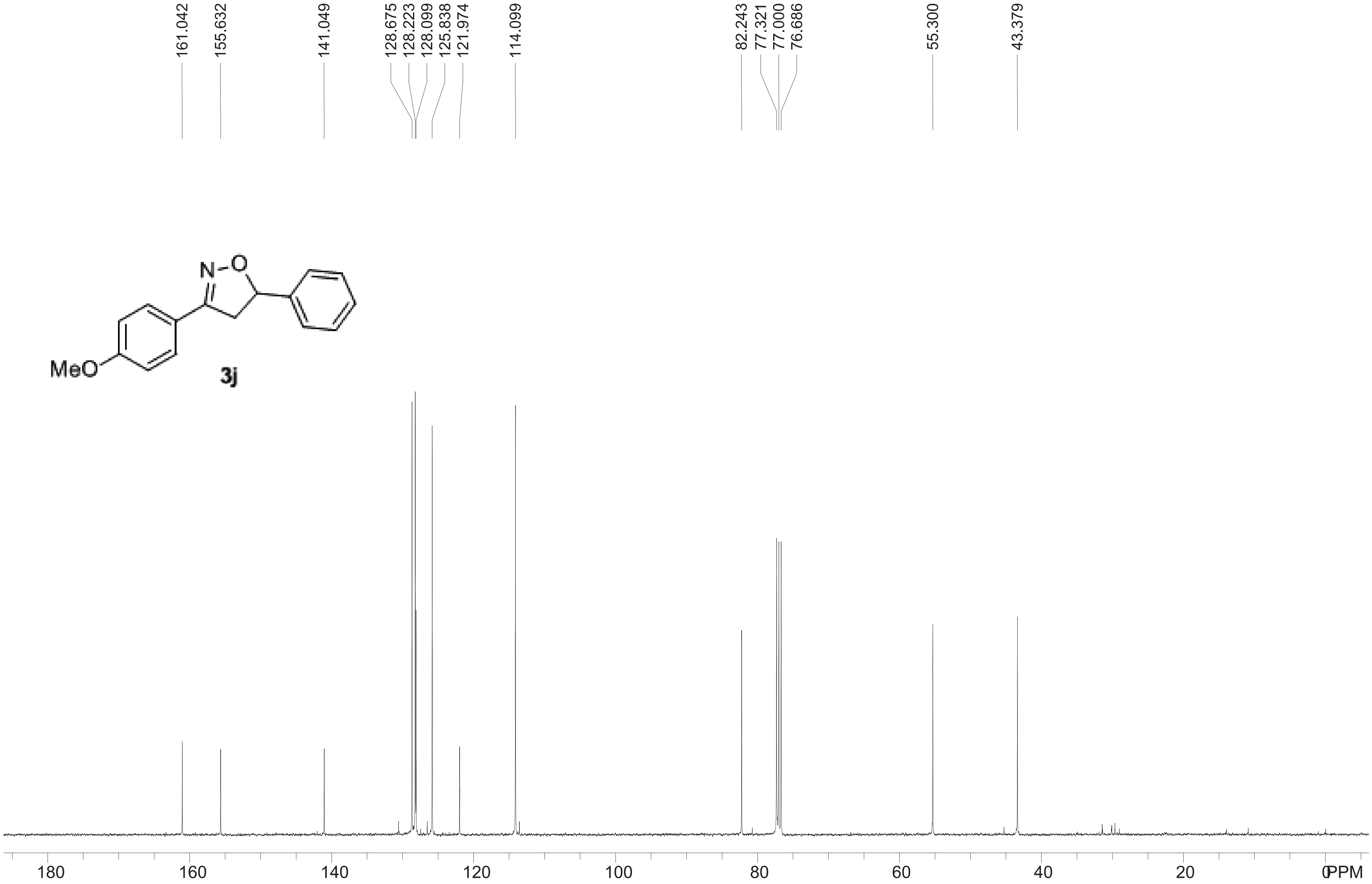


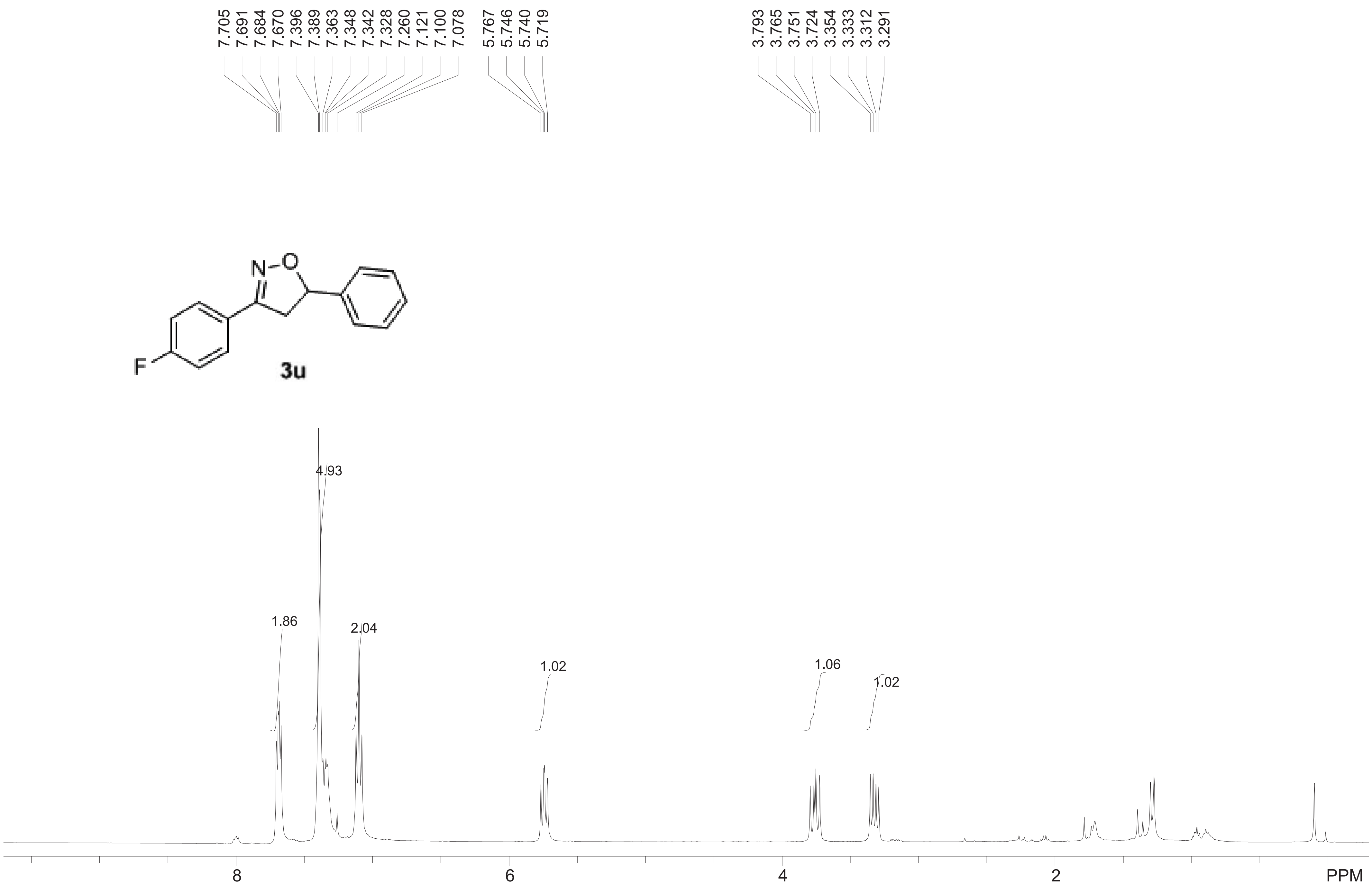
3i


7.704
7.704
7.419
7.402
7.341
7.329
7.260
5.767
5.750
5.750
5.730
3.816
3.794
3.783
3.761
3.378
3.362
3.345
3.329
2.00
7.11
1.02
1.01
1.05
1.01
9
8
7
6
5
4
3
2
1
PPM

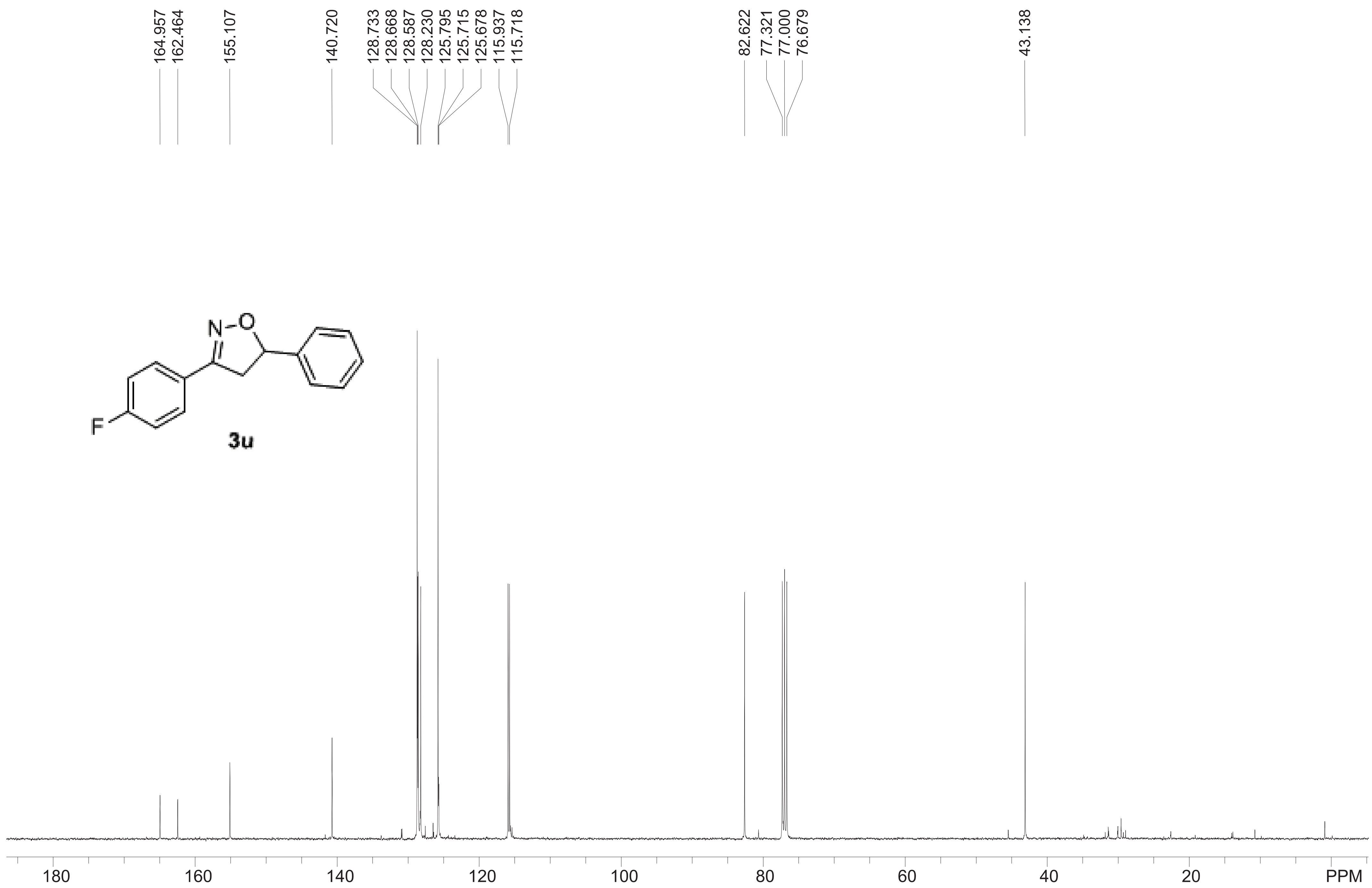

156.072
140.907
130.107
129.443
128.729
128.715
128.197
126.718
125.843
82.540
77.257
77.009
76.754
43.136
N-O
3i
180
160
140
120
100
80
60
40
20
0 PPM


7.649
7.627
7.412
7.393
7.376
7.356
7.333
7.323
7.316
7.303
7.300
7.260
6.939
6.917
5.730
5.709
5.703
5.682
3.860
3.838
3.786
3.759
3.745
3.717
3.347
3.327
3.306
3.286
N
O
MeO
3j
5.07
3.09
2.11
2.12
1.09
1.08
1.09
9
8
7
6
5
4
3
2
1
0PPM

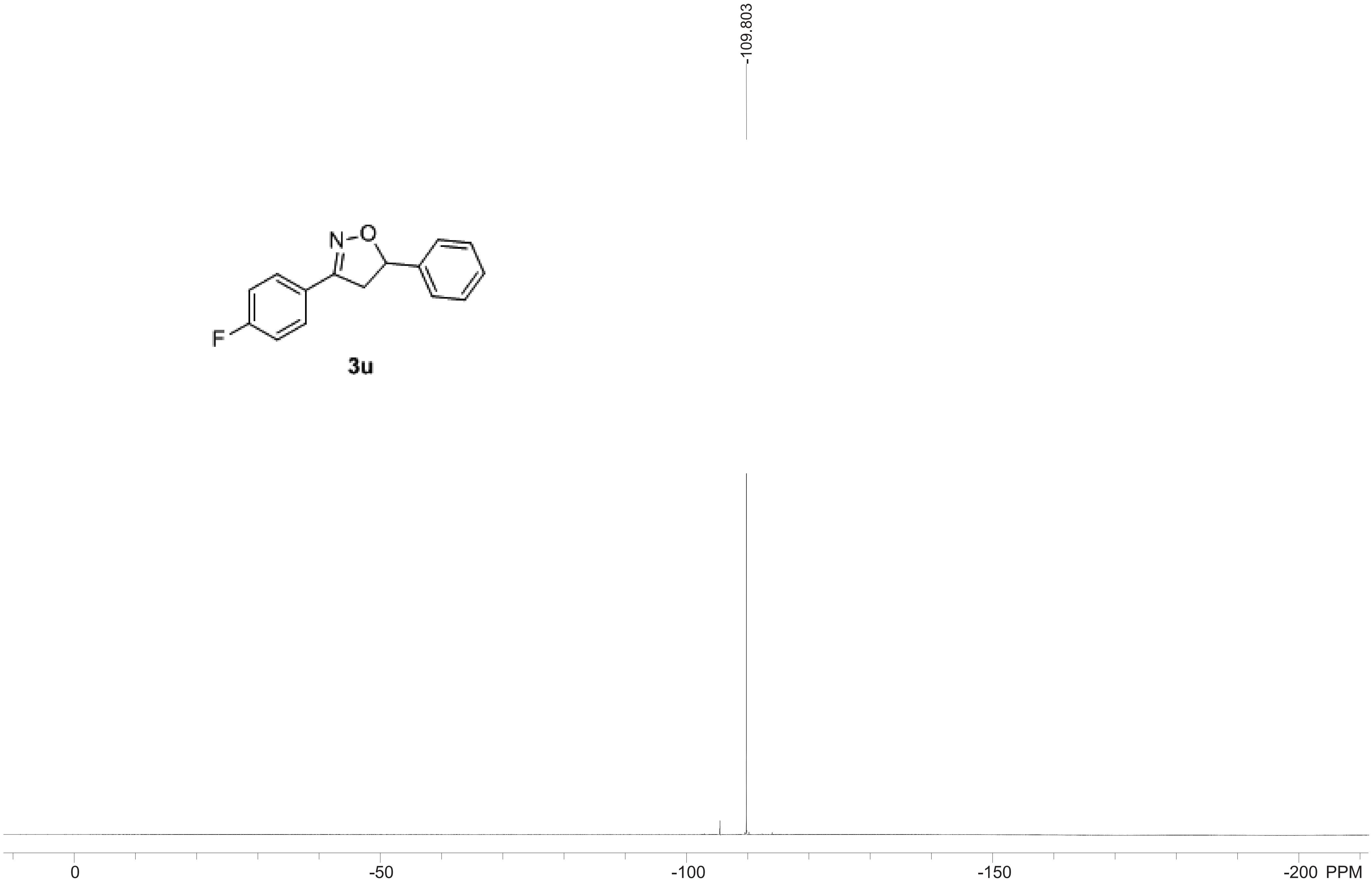

161.042
155.632
141.049
128.675
128.223
128.099
125.838
121.974
114.099
82.243
77.321
77.000
76.686
55.300
43.379
N
O
MeO
3j
180
160
140
120
100
80
60
40
20
0
PPM


N
O
F
7.705
7.691
7.684
7.670
7.396
7.389
7.363
7.348
7.342
7.328
7.260
7.121
7.100
7.078
5.767
5.746
5.740
5.719
3.793
3.765
3.751
3.724
3.354
3.333
3.312
3.291
4.93
1.86
2.04
1.02
1.06
1.02
8
6
4
2
PPM

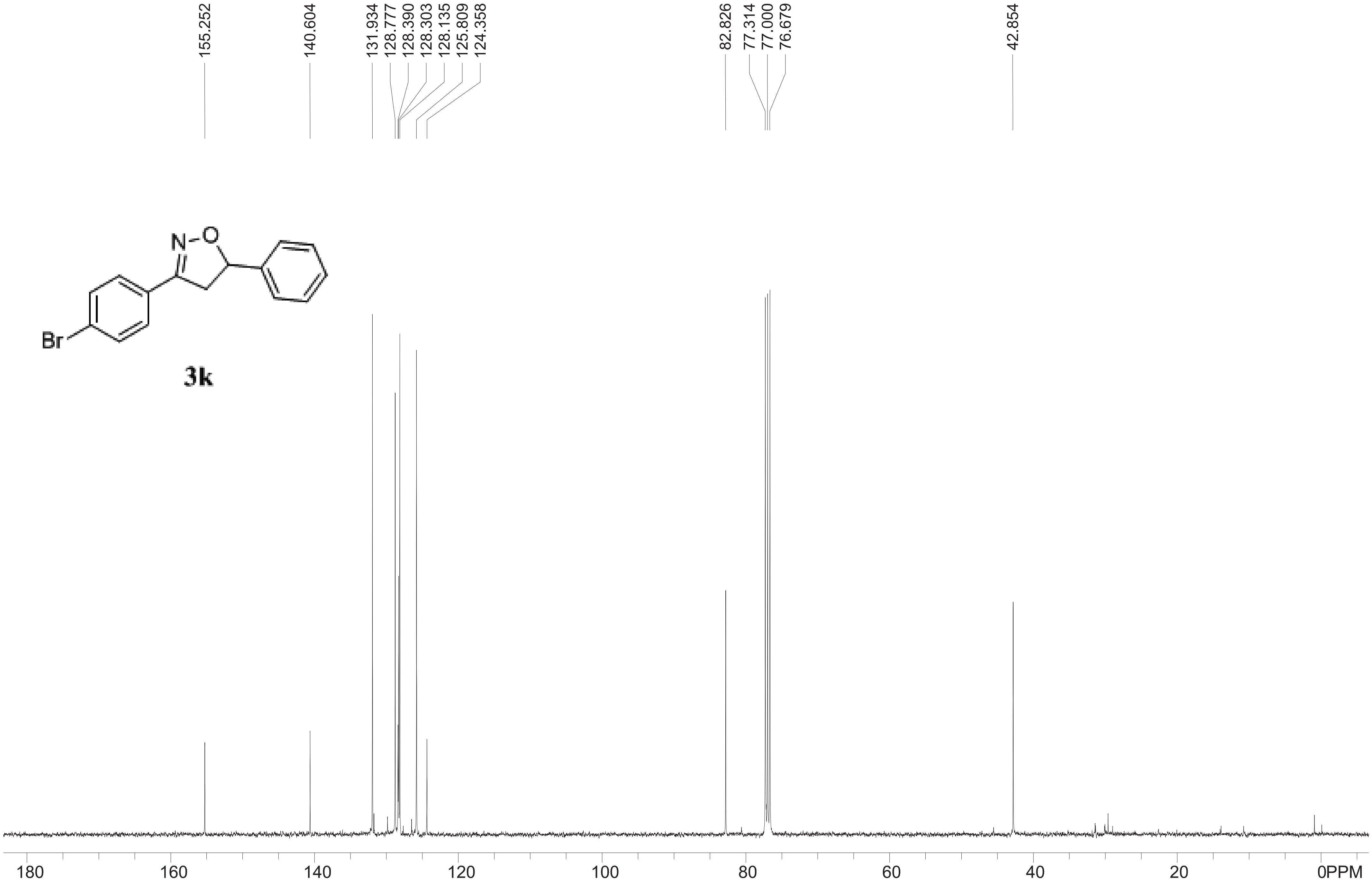
3u


164.957
162.464
155.107
140.720
128.733
128.668
128.587
128.230
125.795
125.715
125.678
115.937
115.718
82.622
77.321
77.000
76.679
43.138
N
O
F
180
160
140
120
100
80
60
40
20
PPM

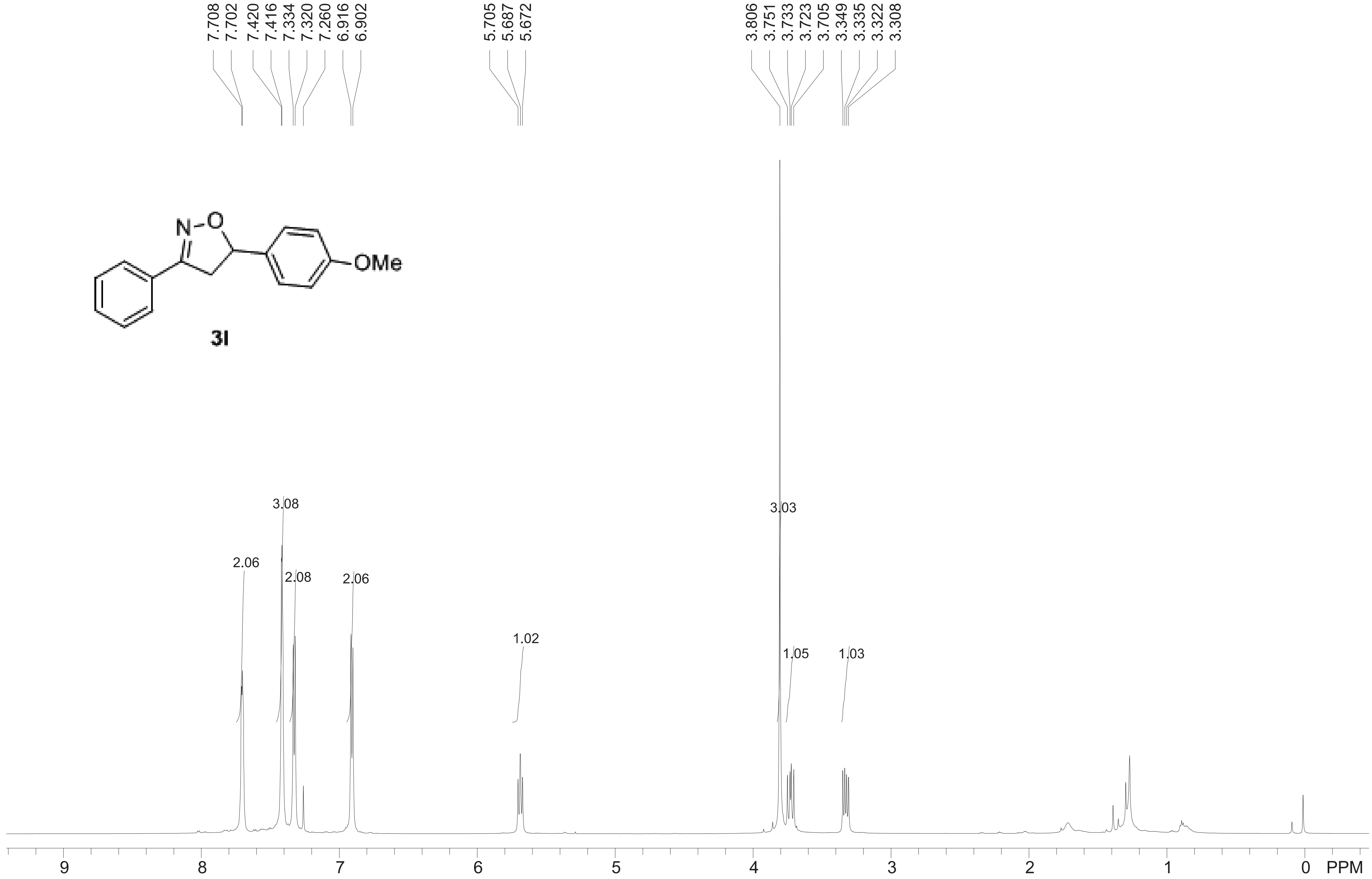
3u

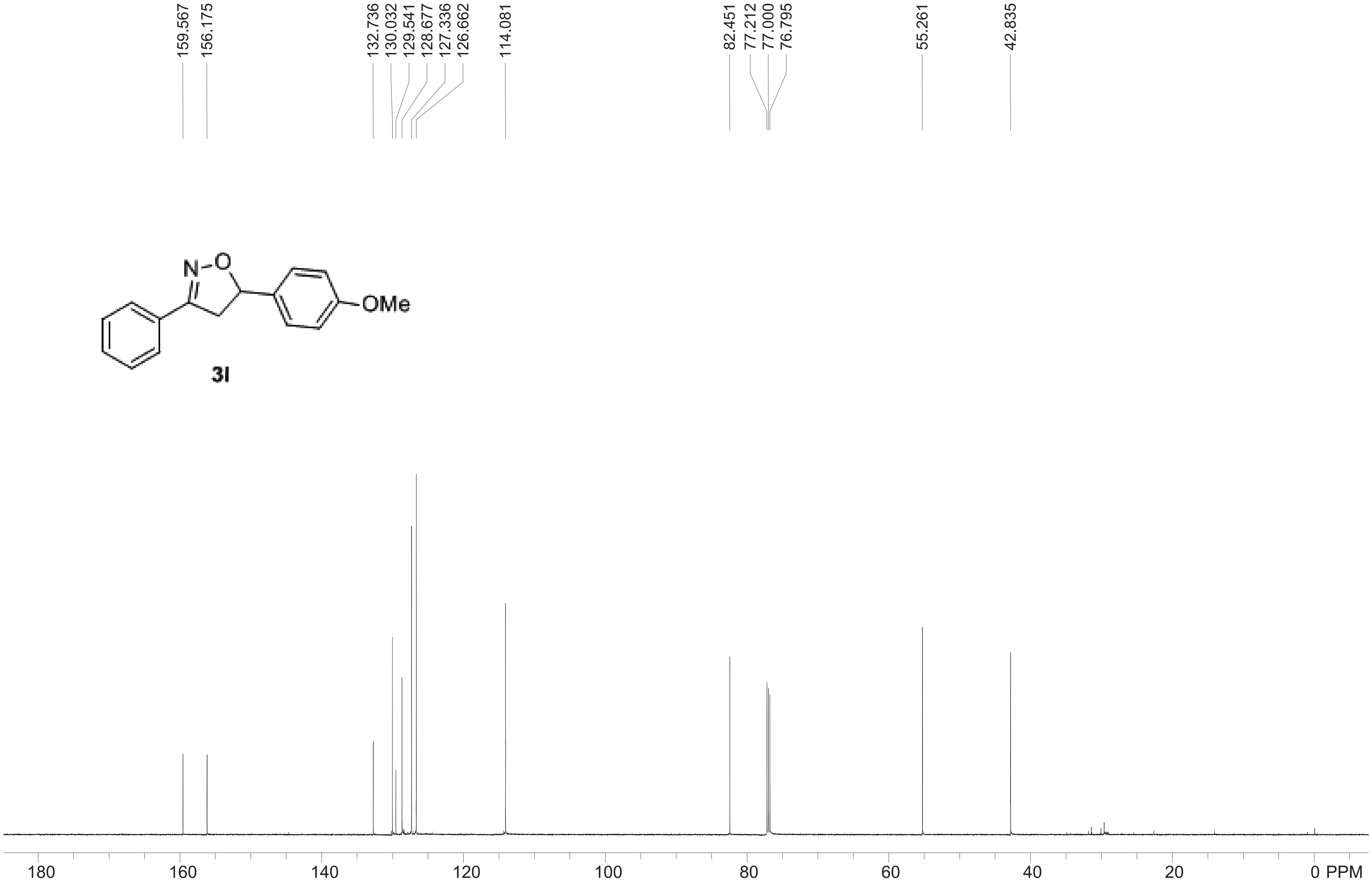

-109.803
N
O
F
3u
0
-50
-100
-150
-200 PPM

7.574
7.550
7.526
7.391
7.381
7.360
7.350
7.340
7.329
7.320
7.260
5.776
5.755
5.749
5.727
3.785
3.757
3.743
3.715
3.346
3.325
3.304
3.283
N
O
Br
3k
5.08
4.05
1.04
1.07
1.04
8
6
4
2
0
PPM

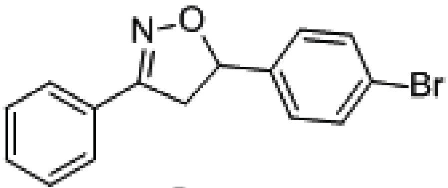


155.252
140.604
131.934
128.777
128.390
128.303
128.135
125.809
124.358
82.826
77.314
77.000
76.679
42.854
Br
N
O
3k
180
160
140
120
100
80
60
40
20
0PPM

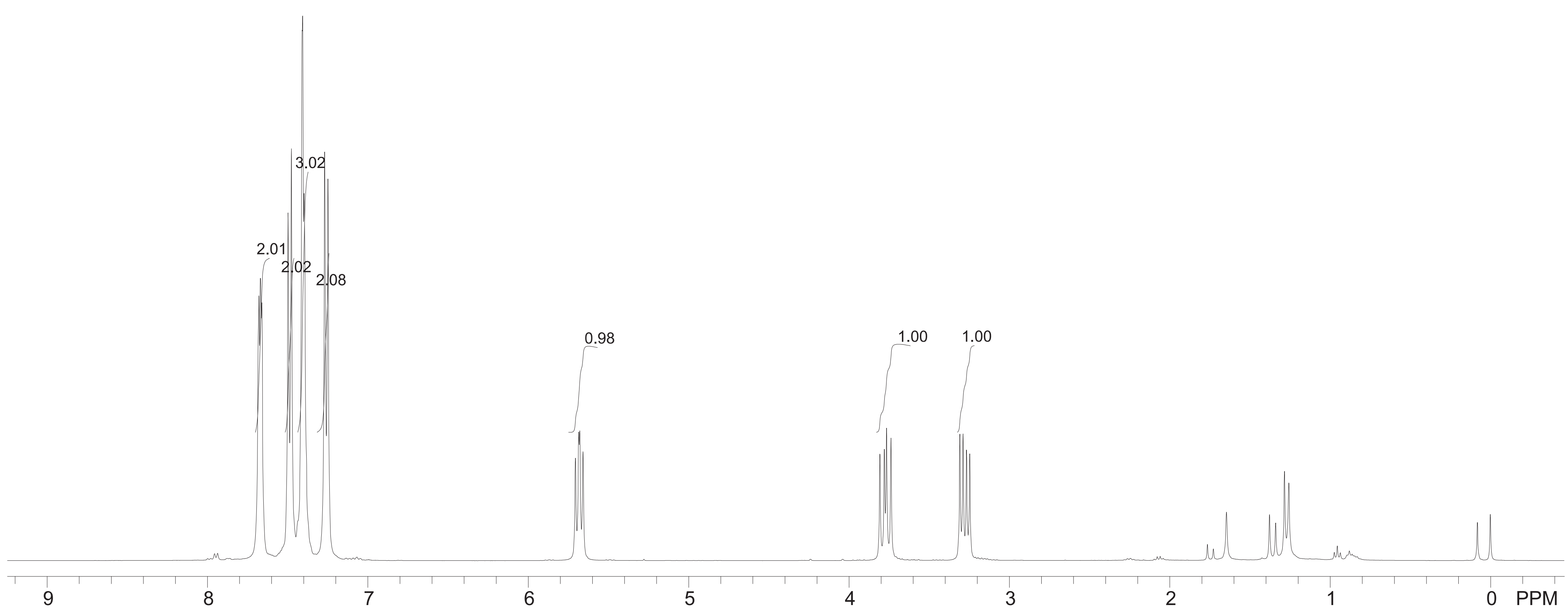

7.708
7.702
7.420
7.416
7.334
7.320
7.260
6.916
6.902
5.705
5.687
5.672
3.806
3.751
3.733
3.723
3.705
3.349
3.335
3.322
3.308
N-O
OMe
3l
3.08
2.06
2.08
2.06
1.02
3.03
1.05
1.03
9
8
7
6
5
4
3
2
1
0
PPM

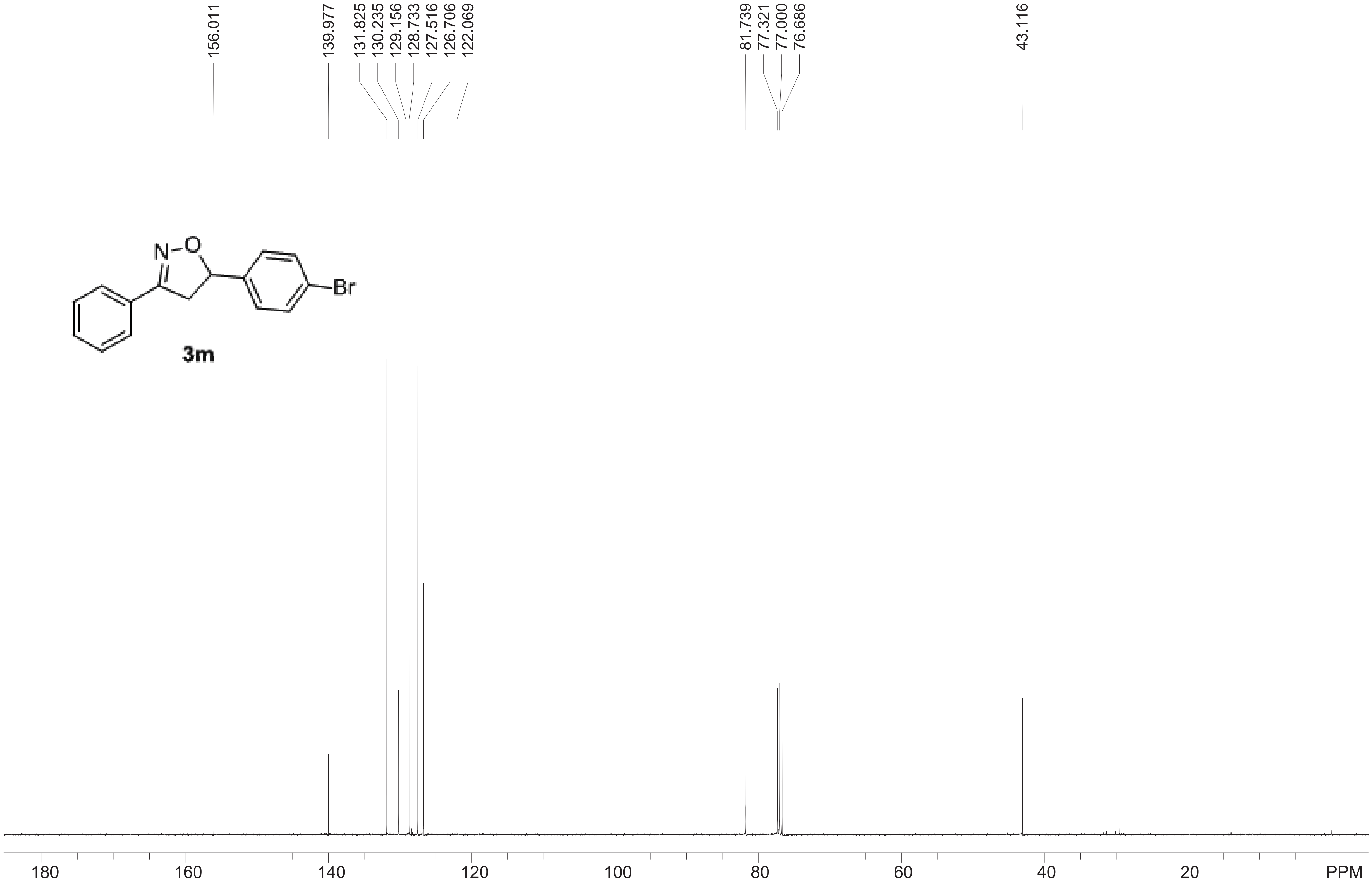

159.567
156.175
132.736
130.032
129.541
128.677
127.336
126.662
114.081
82.451
77.212
77.000
76.795
55.261
42.835
180
160
140
120
100
80
60
40
20
0 PPM
N
O
OMe

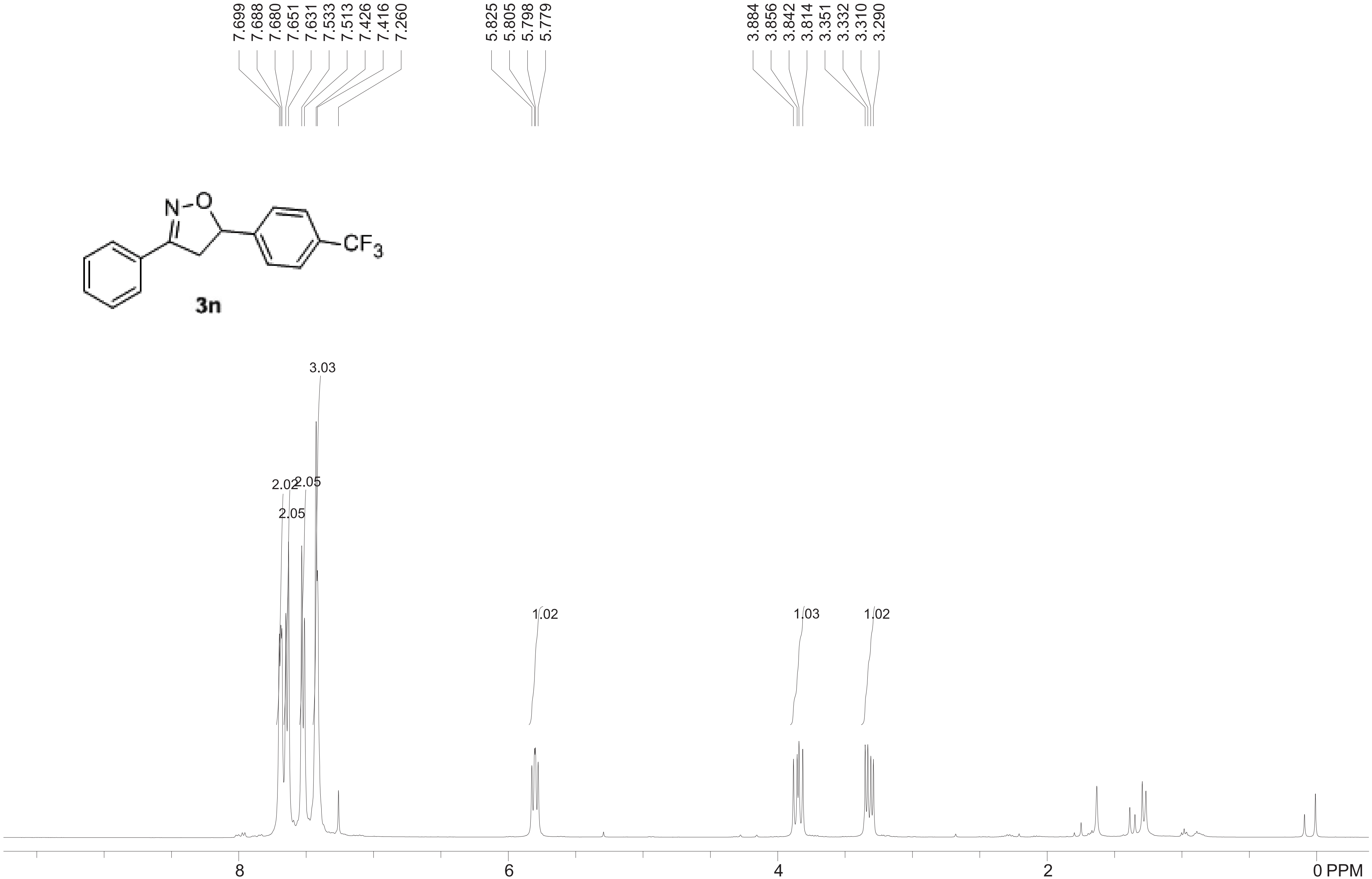
3l

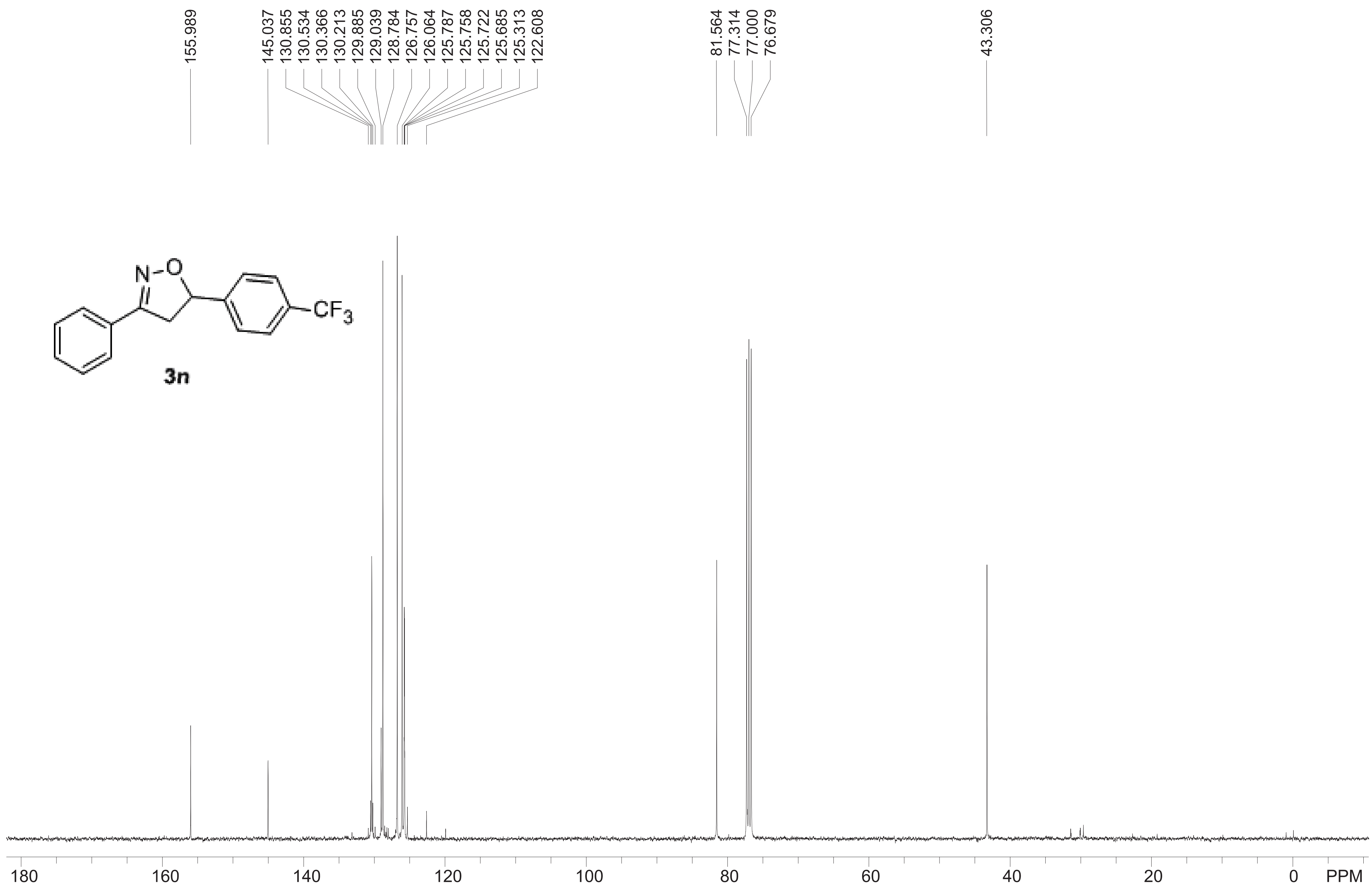


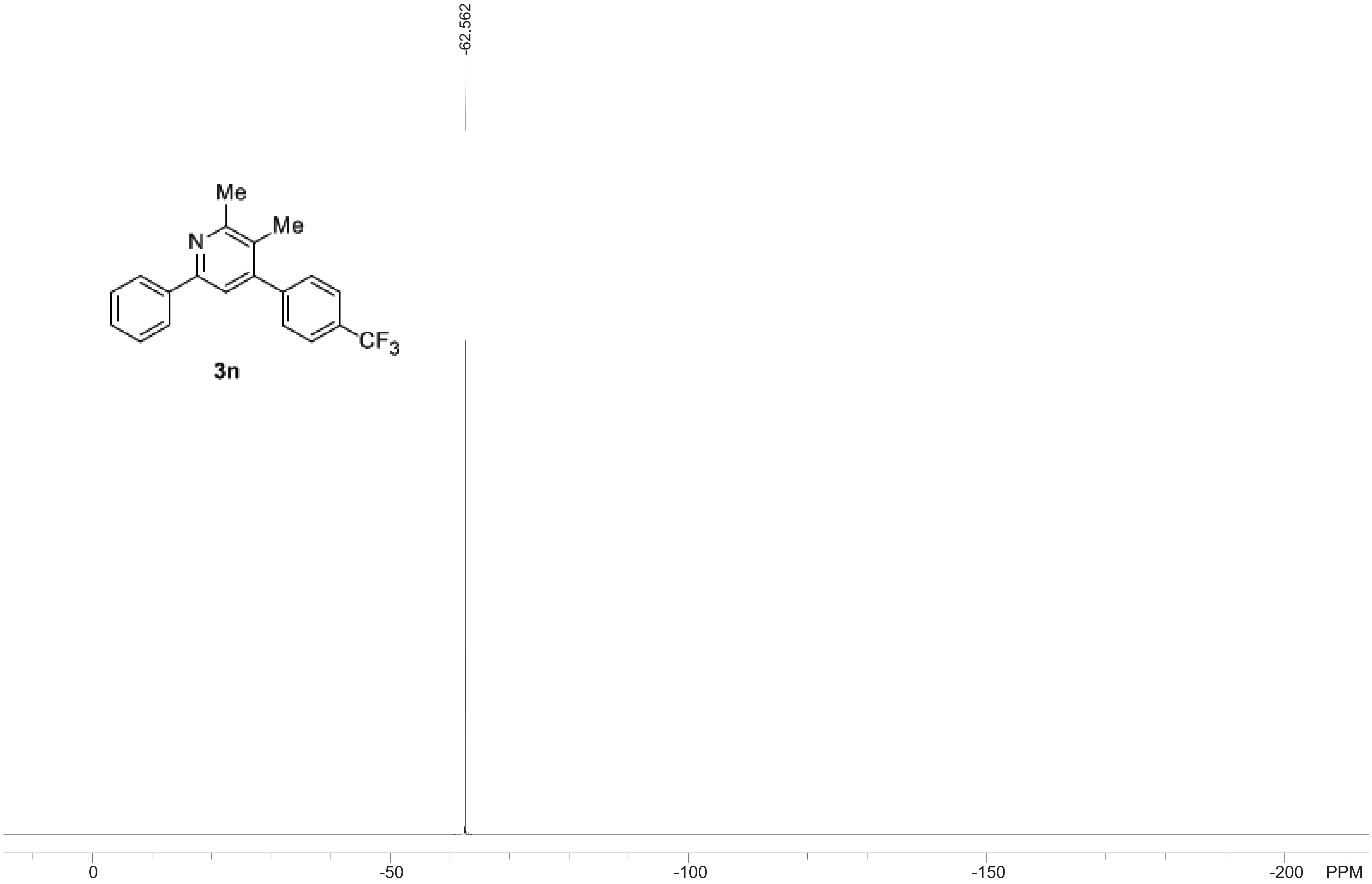
3m

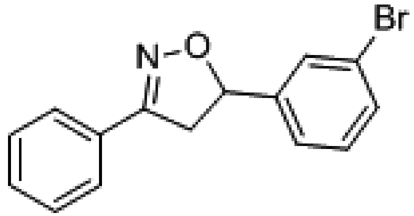

7.680
7.670
7.661
7.498
7.477
7.436
7.410
7.407
7.397
7.270
7.249
5.706
5.686
5.679
5.659
3.807
3.780
3.766
3.738
3.308
3.288
3.267
3.246
-0.000
2.01
2.02
3.02
2.08
0.98
1.00
1.00
9
8
7
6
5
4
3
2
1
0
PPM


156.011
139.977
131.825
130.235
129.156
128.733
127.516
126.706
122.069
81.739
77.321
77.000
76.686
43.116
Br
N
O
3m
180
160
140
120
100
80
60
40
20
PPM

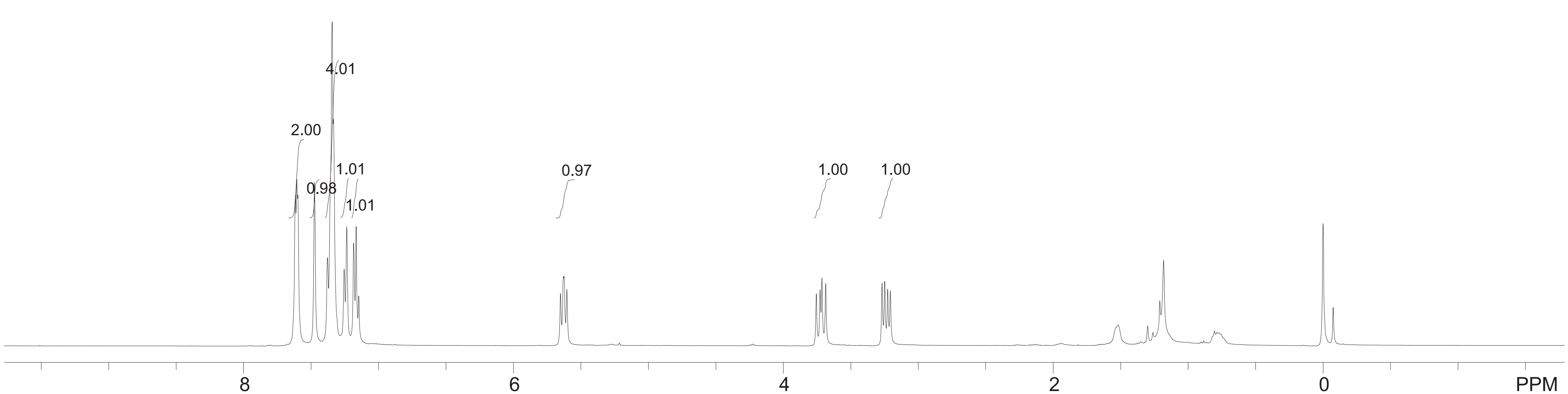

7.699
7.688
7.680
7.651
7.631
7.533
7.513
7.426
7.416
7.260
5.825
5.805
5.798
5.779
3.884
3.856
3.842
3.814
3.351
3.332
3.310
3.290
3n
3.03
2.02
2.05
2.05
1.02
1.03
1.02
8
6
4
2
0 PPM

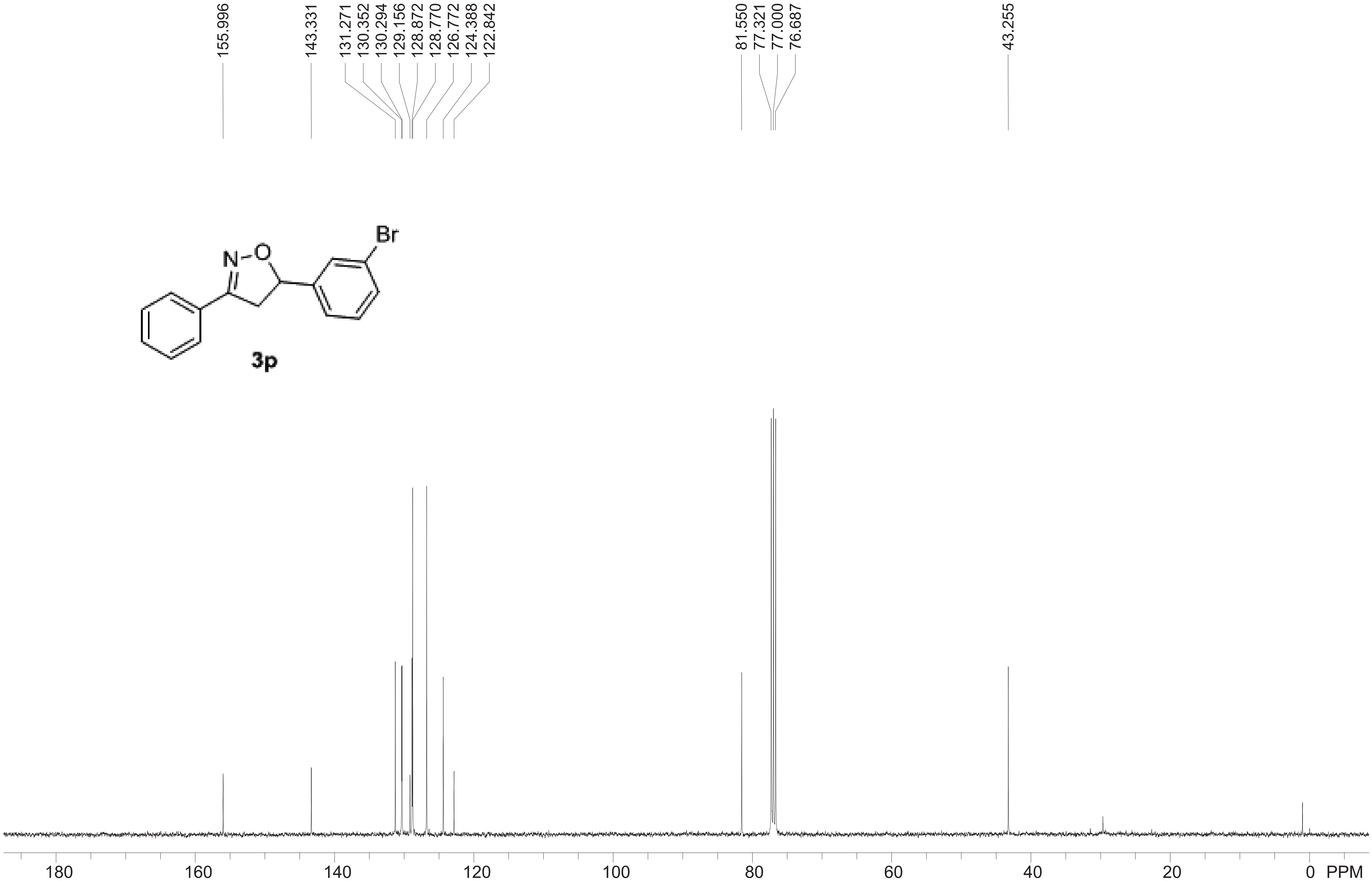

155.989
145.037
130.855
130.534
130.366
130.213
129.885
129.039
128.784
126.757
126.064
125.787
125.758
125.722
125.685
125.313
122.608
81.564
77.314
77.000
76.679
43.306
N-O
CF3
3n
180
160
140
120
100
80
60
40
20
0
PPM

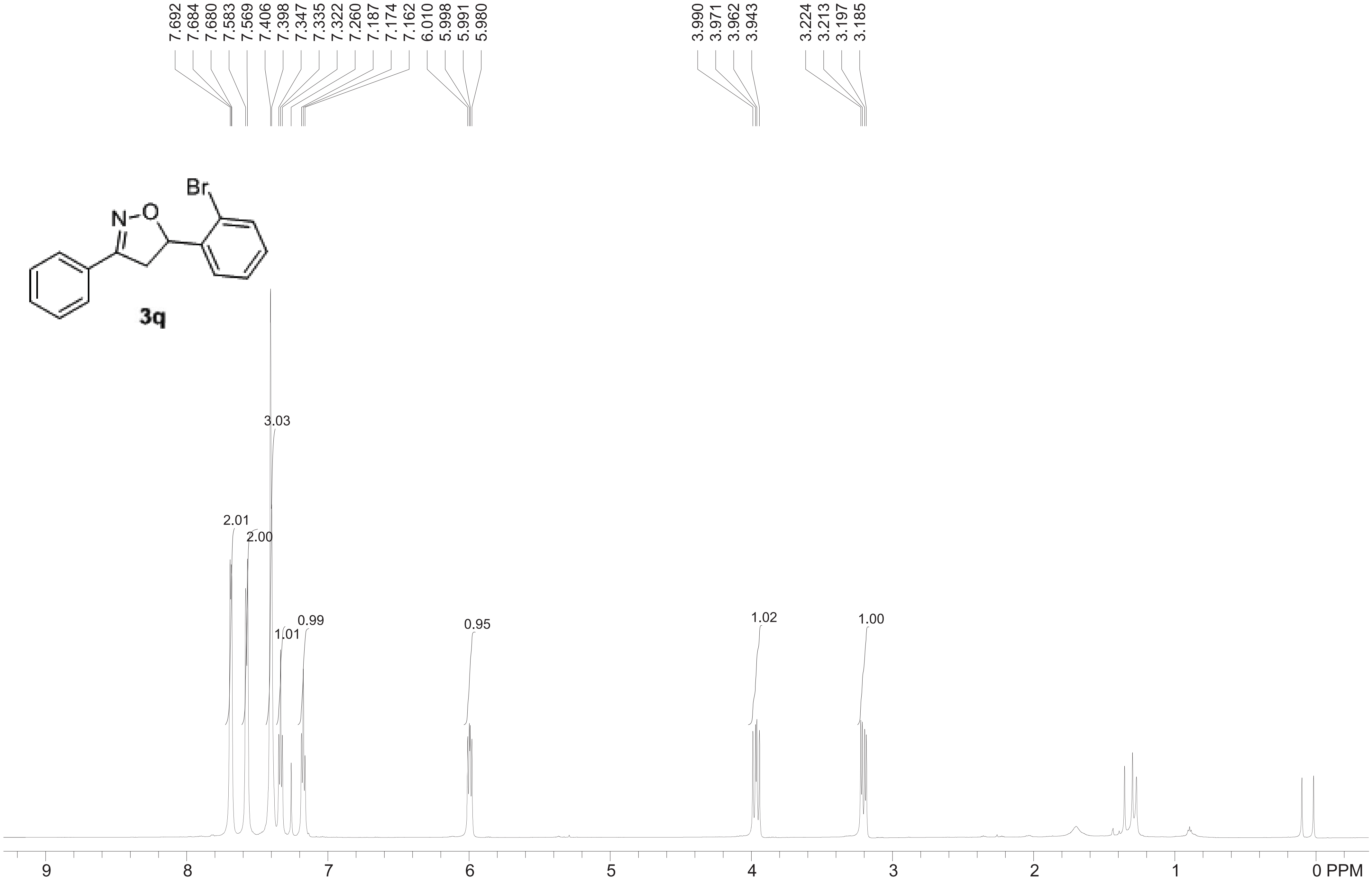

-62.562
3n
0
-50
-100
-150
-200
PPM

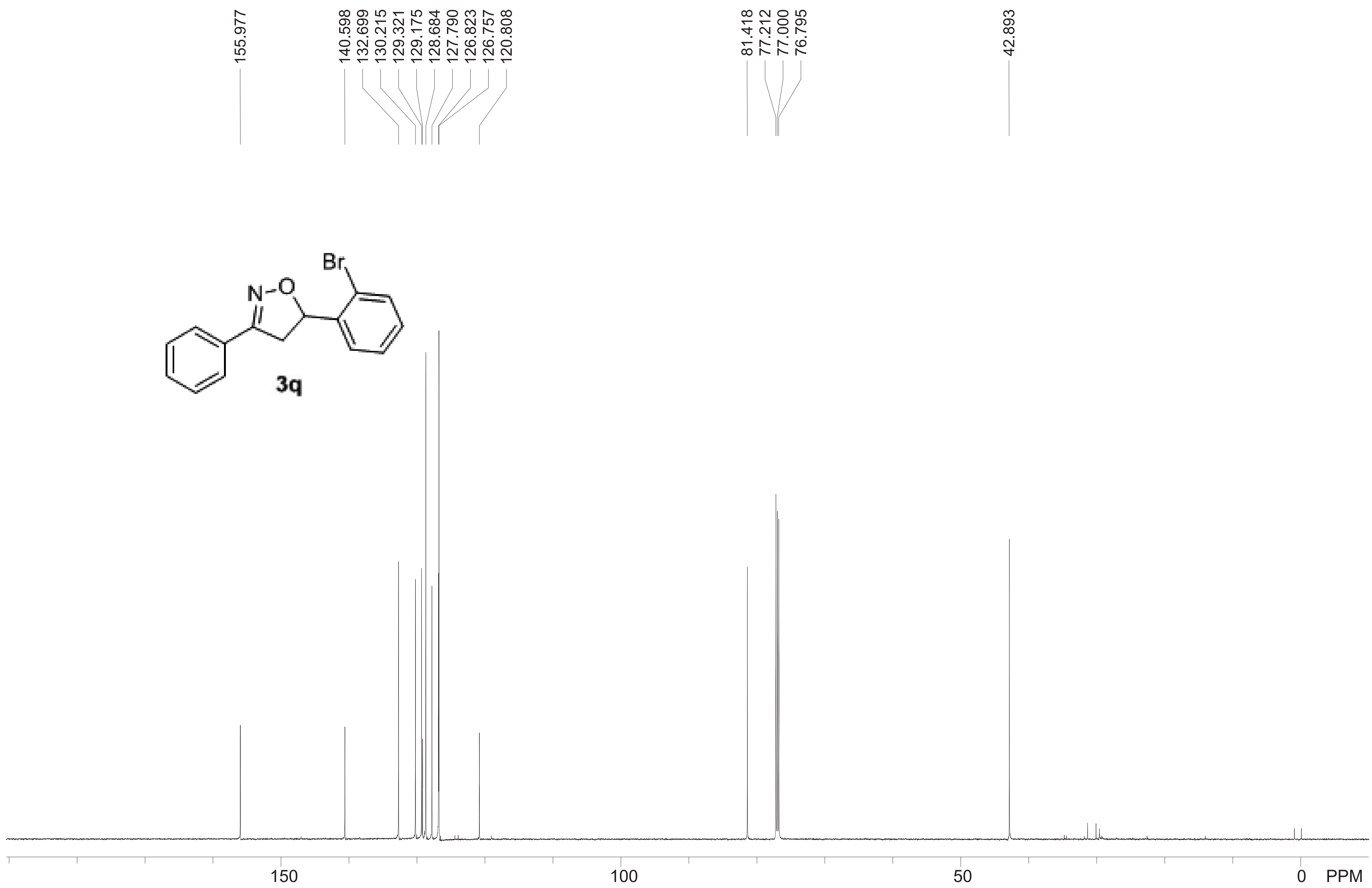


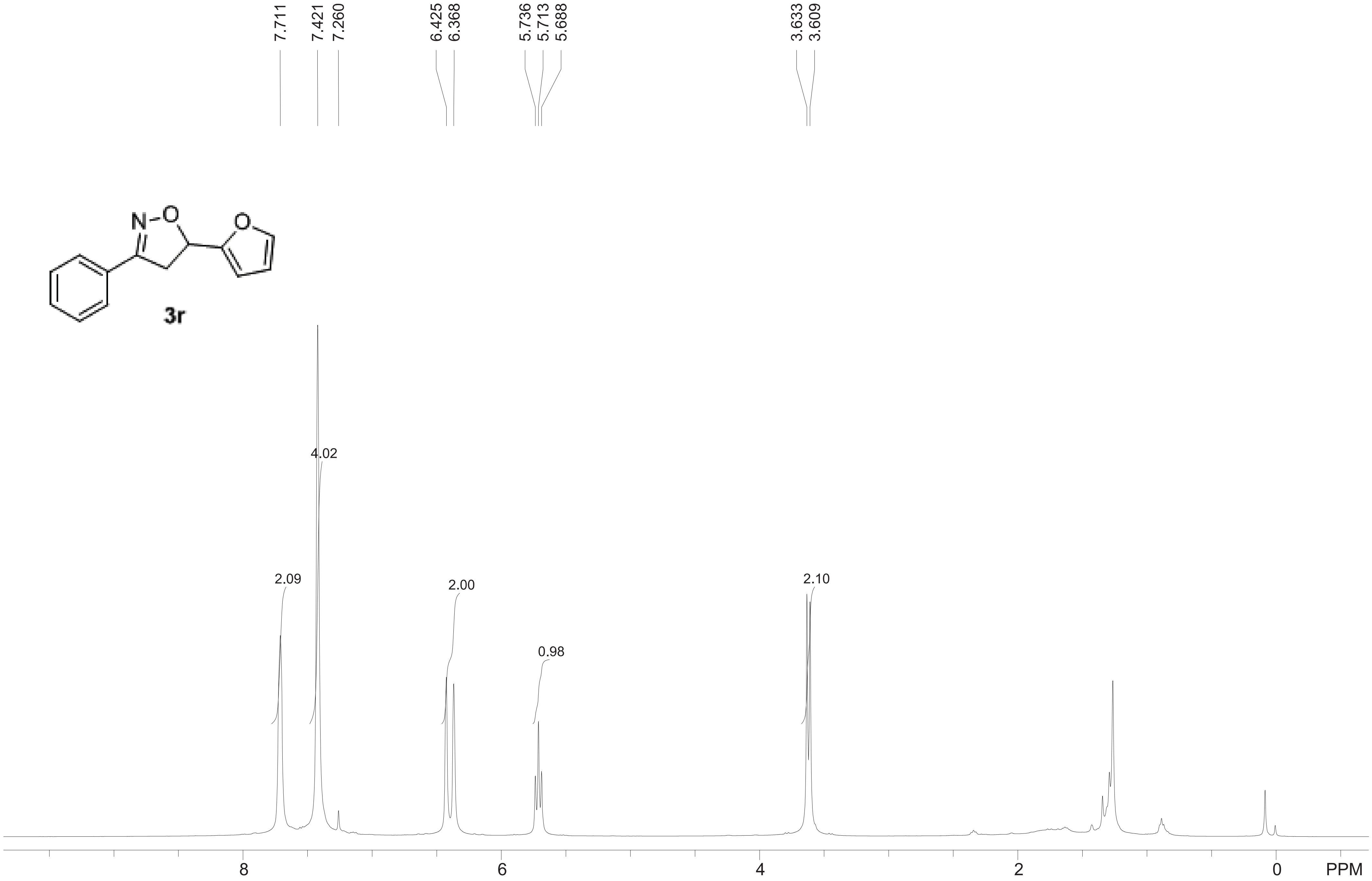
3p

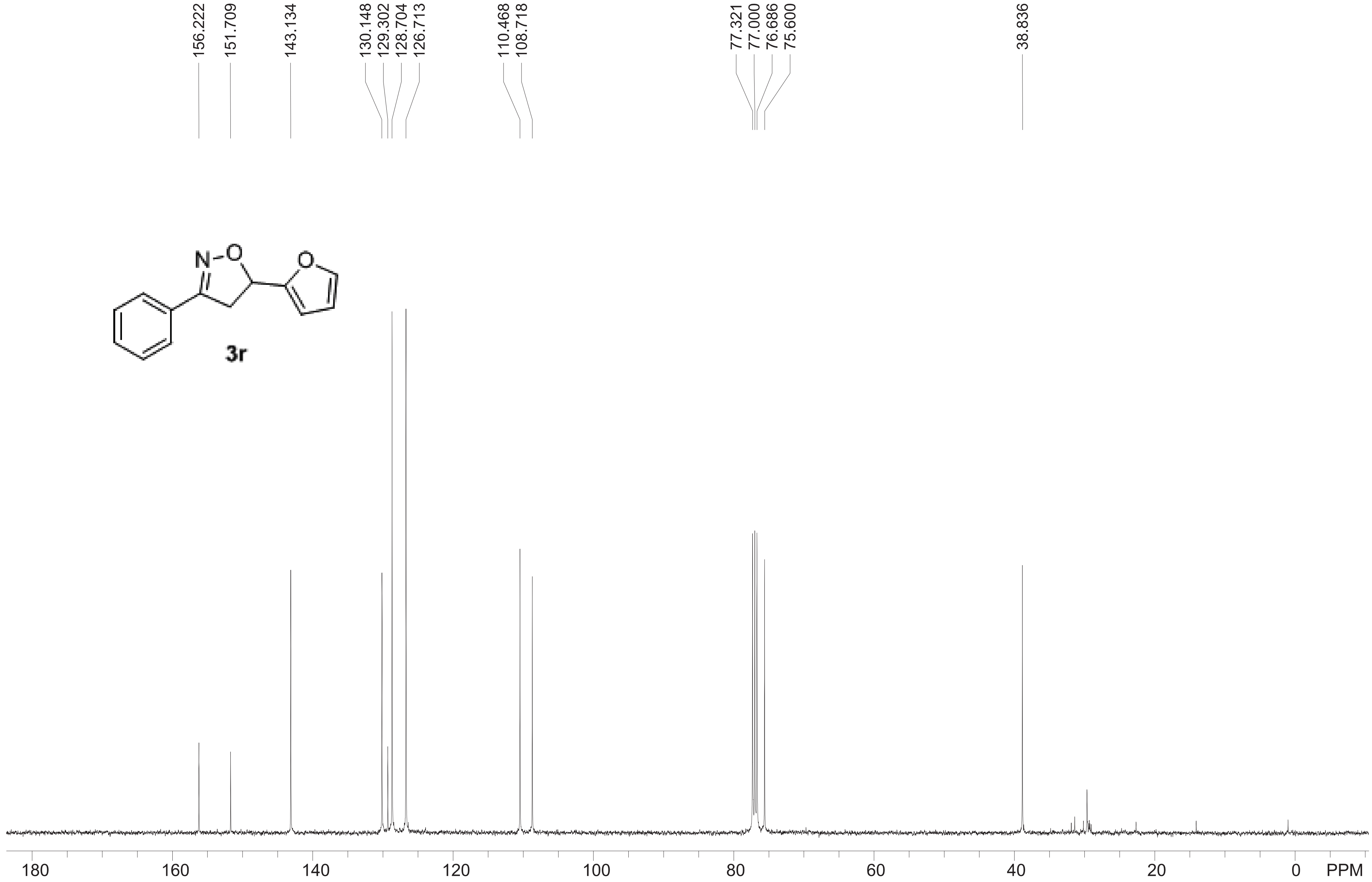

7.616
7.606
7.597
7.474
7.378
7.343
7.334
7.254
7.235
7.185
7.166
7.147
5.651
5.631
5.625
5.604
3.756
3.728
3.714
3.686
3.268
3.249
3.227
3.207
-0.000
2.00
0.98
4.01
1.01
1.01
0.97
1.00
1.00
8
6
4
2
0
PPM

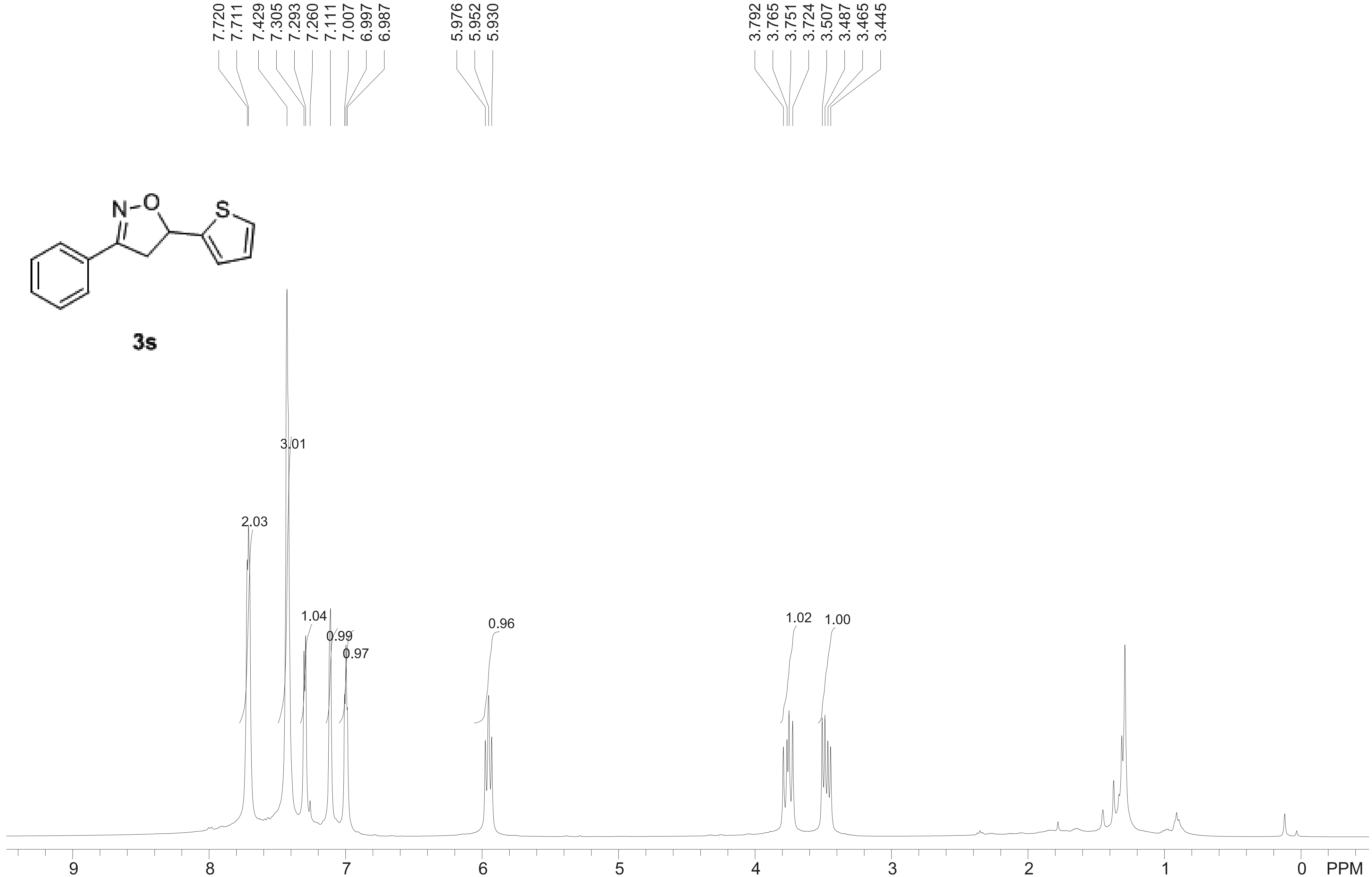

155.996
143.331
131.271
130.352
130.294
129.156
128.872
128.770
126.772
124.388
122.842
81.550
77.321
77.000
76.687
43.255
Br
N
O
3p
180
160
140
120
100
80
60
40
20
0 PPM


7.692
7.684
7.680
7.583
7.569
7.406
7.398
7.347
7.335
7.322
7.260
7.187
7.174
7.162
6.010
5.998
5.991
5.980
3.990
3.971
3.962
3.943
3.224
3.213
3.197
3.185
Br
N-O
3q
3.03
2.01
2.00
1.01
0.99
0.95
1.02
1.00
9
8
7
6
5
4
3
2
1
0 PPM

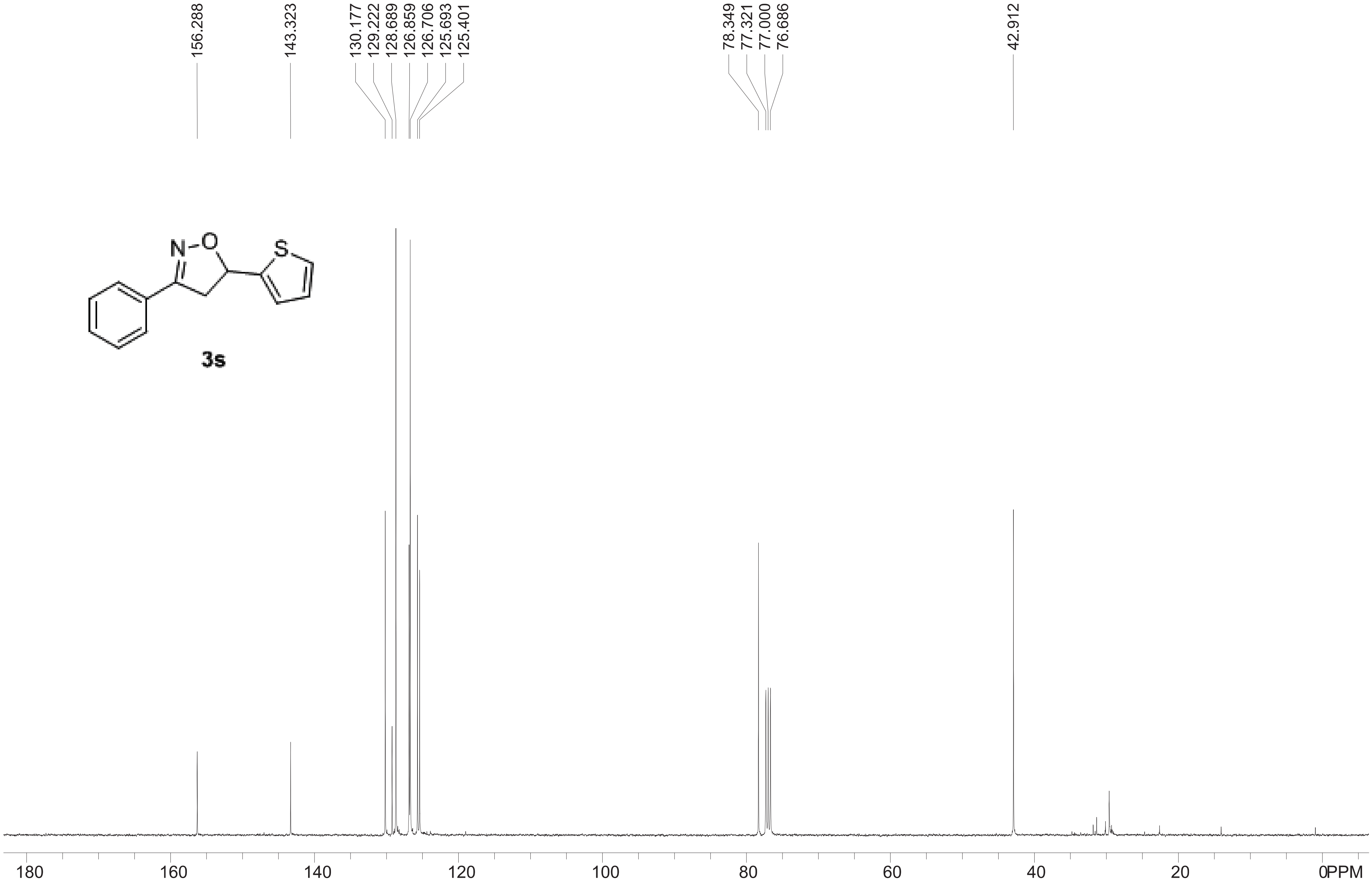

155.977
140.598
132.699
130.215
129.321
129.175
128.684
127.790
126.823
126.757
120.808
81.418
77.212
77.000
76.795
42.893
Br
N-O
3q
150
100
50
0
PPM

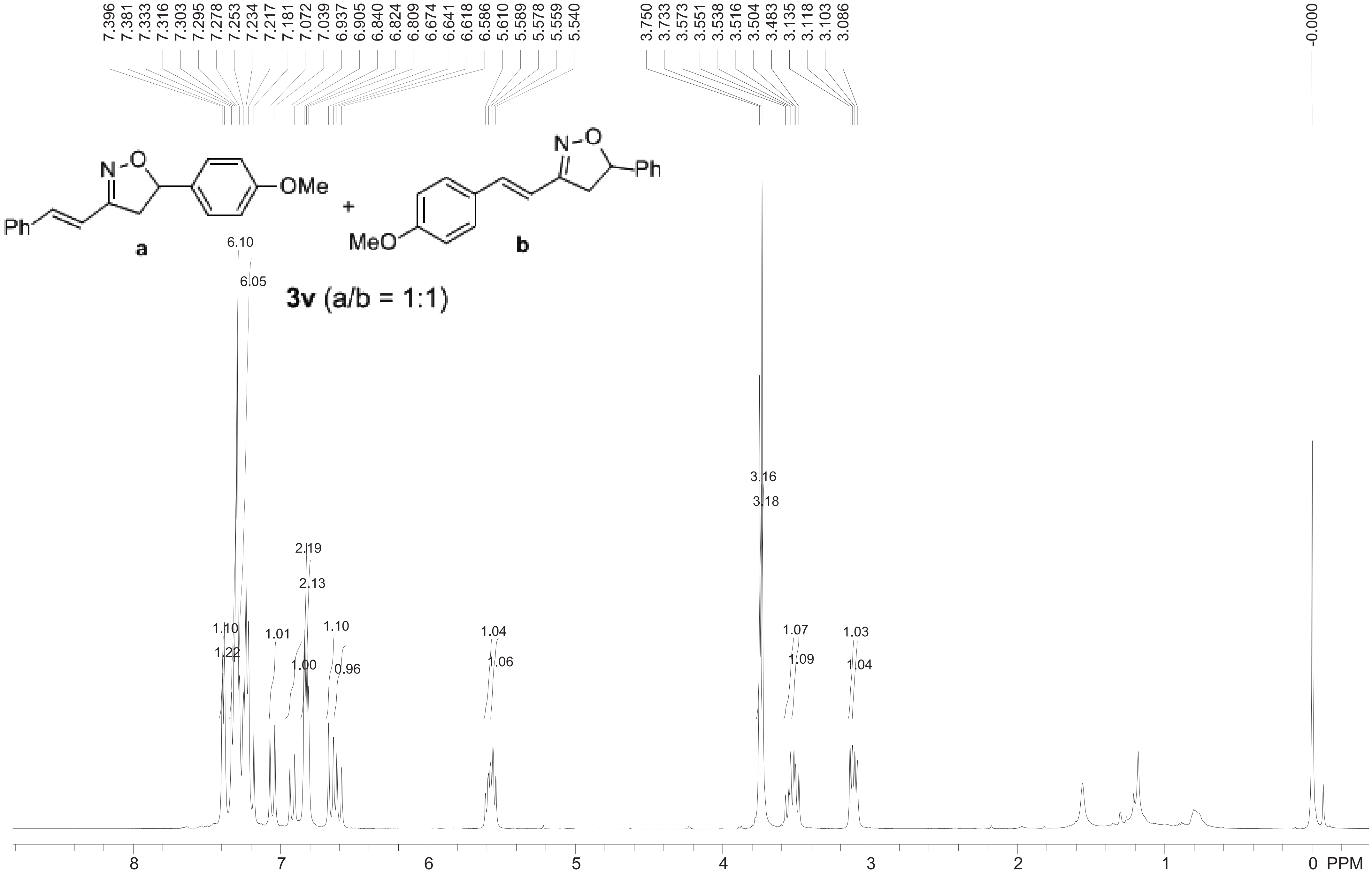

7.711
7.421
7.260
6.425
6.368
5.736
5.713
5.688
3.633
3.609
3r
2.09
4.02
2.00
0.98
2.10
8
6
4
2
0
PPM

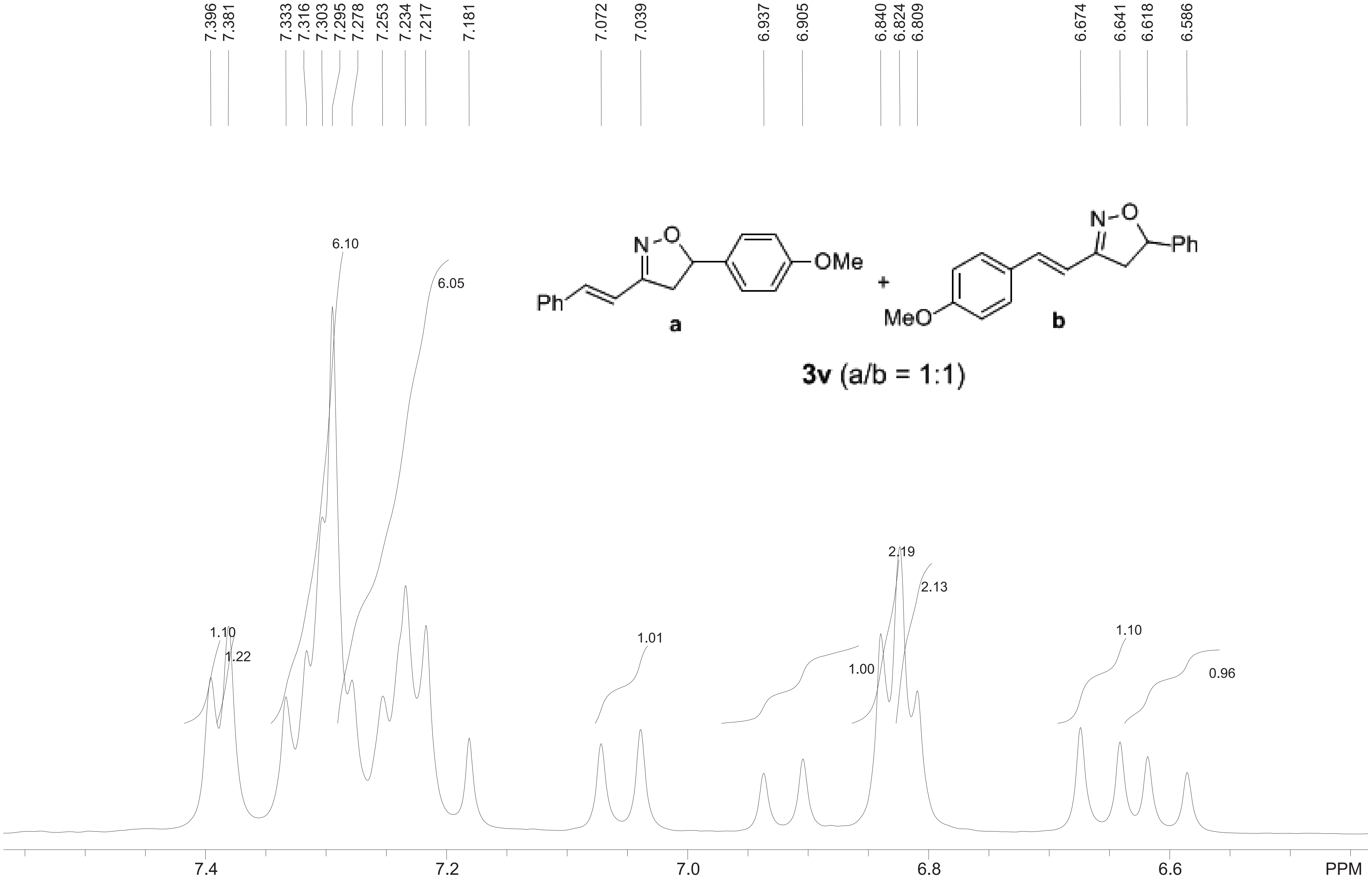

156.222
151.709
143.134
130.148
129.302
128.704
126.713
110.468
108.718
77.321
77.000
76.686
75.600
38.836
3r
180
160
140
120
100
80
60
40
20
0
PPM

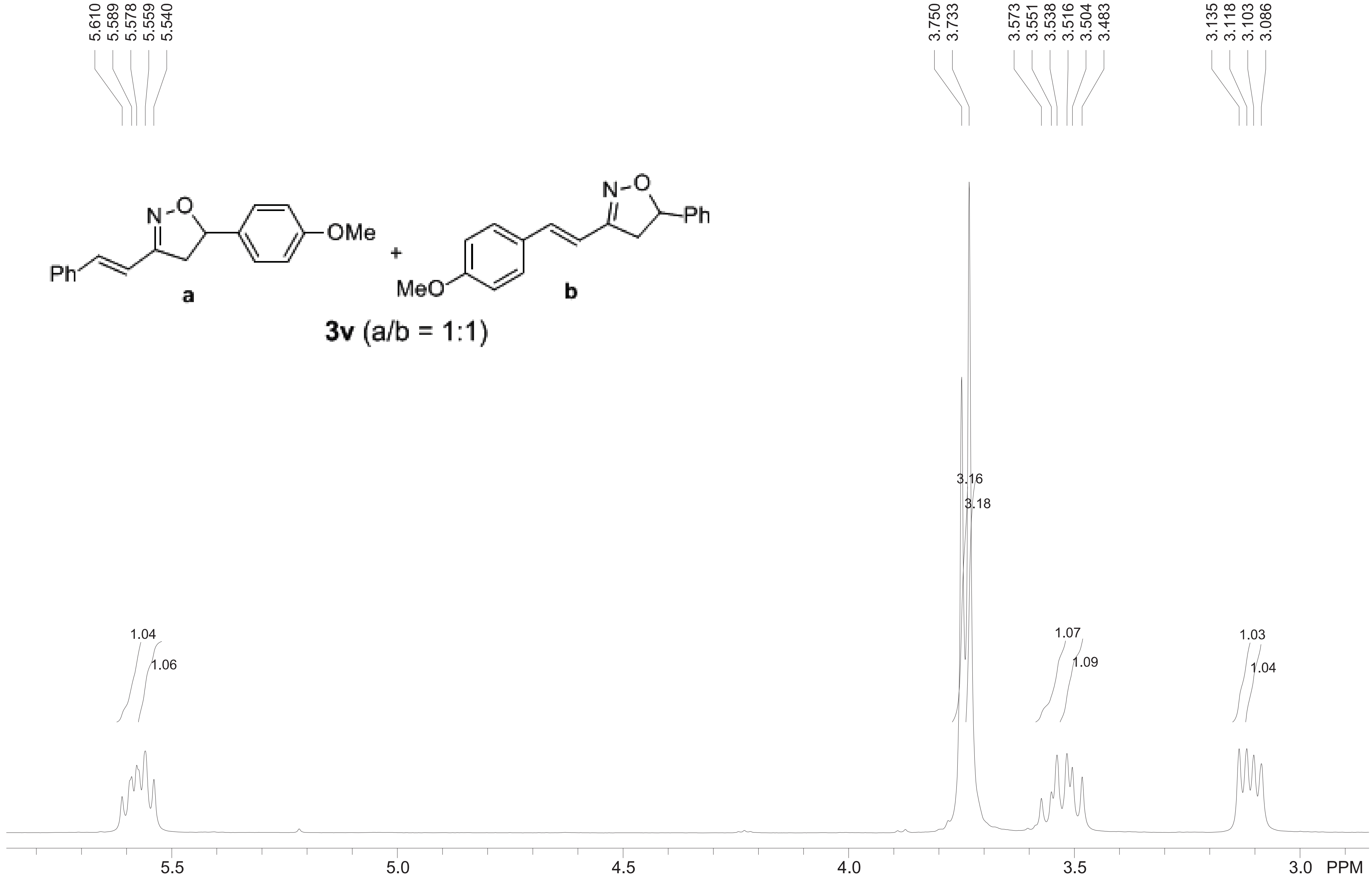


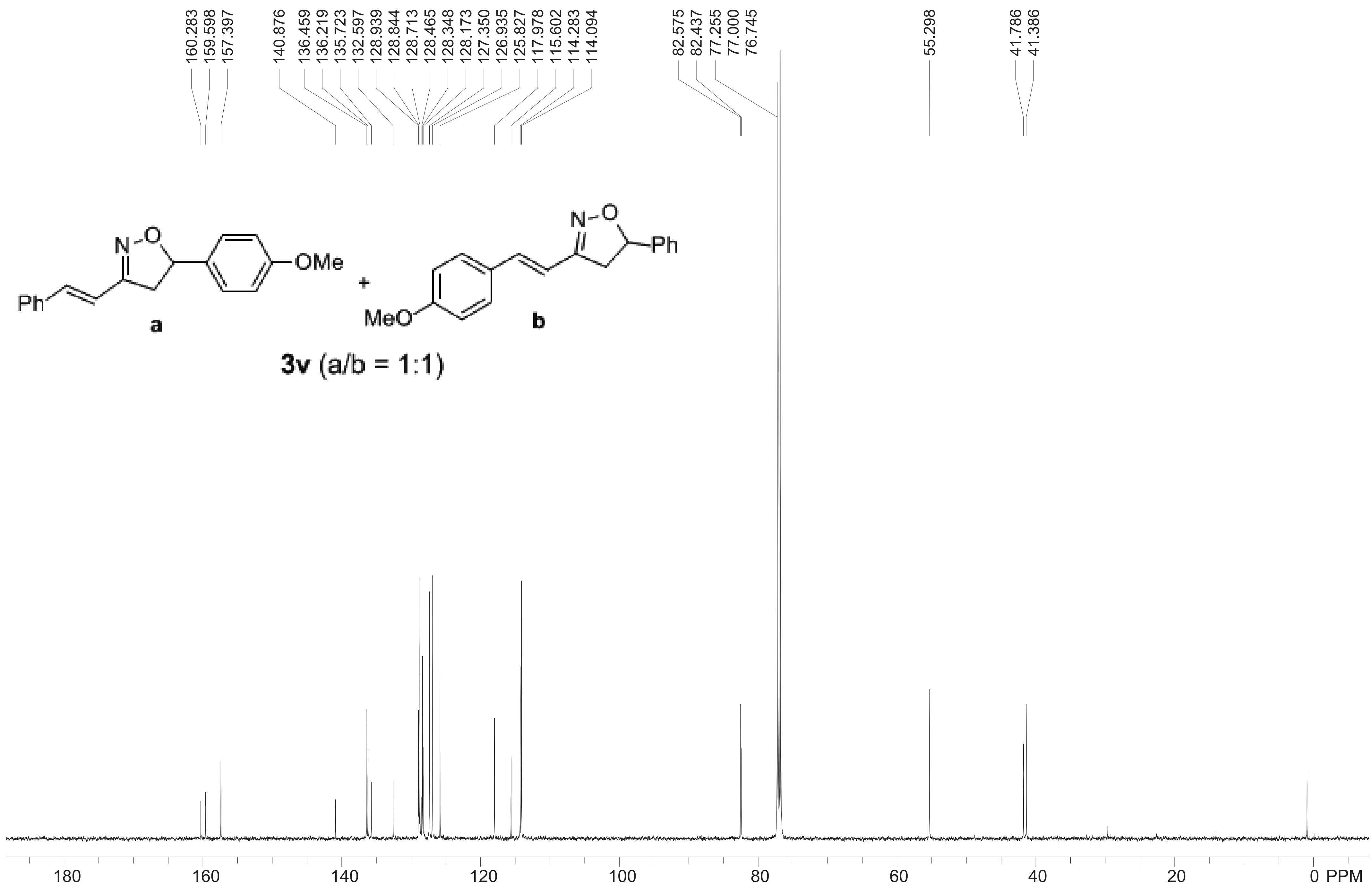
7.720
7.711
7.429
7.305
7.293
7.260
7.111
7.007
6.997
6.987
5.976
5.952
5.930
3.792
3.765
3.751
3.724
3.507
3.487
3.465
3.445
N
O
S
3s
3.01
2.03
1.04
0.99
0.97
0.96
1.02
1.00
9
8
7
6
5
4
3
2
1
0
PPM

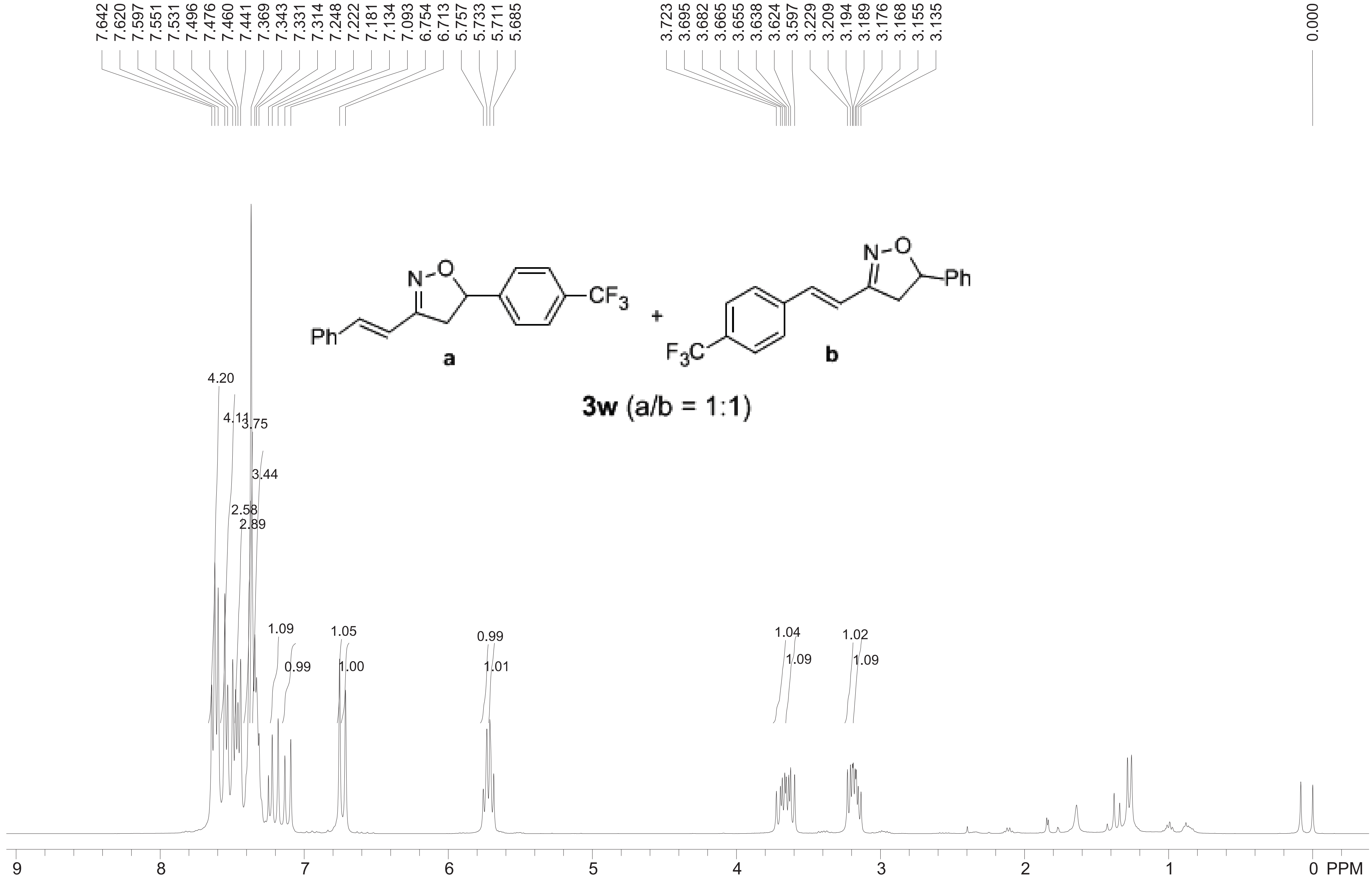

156.288
143.323
130.177
129.222
128.689
126.859
126.706
125.693
125.401
78.349
77.321
77.000
76.686
42.912
N-O
S
3s
180
160
140
120
100
80
60
40
20
0PPM


7.396 7.381 7.333 7.316 7.303 7.295 7.278 7.253 7.234 7.217 7.181 7.072 7.039 6.937 6.905 6.840 6.824 6.809 6.674 6.641 6.618 6.586 5.610 5.589 5.578 5.559 5.540
3.750 3.733 3.573 3.551 3.538 3.516 3.504 3.483 3.135 3.118 3.103 3.086
-0.000
OMe
Ph
a
MeO
Ph
b
3v (a/b = 1:1)
6.10 6.05 1.10 1.22 1.01 1.00 2.19 2.13 1.10 0.96 1.04 1.06 3.16 3.18 1.07 1.09 1.03 1.04
8 7 6 5 4 3 2 1 0 PPM

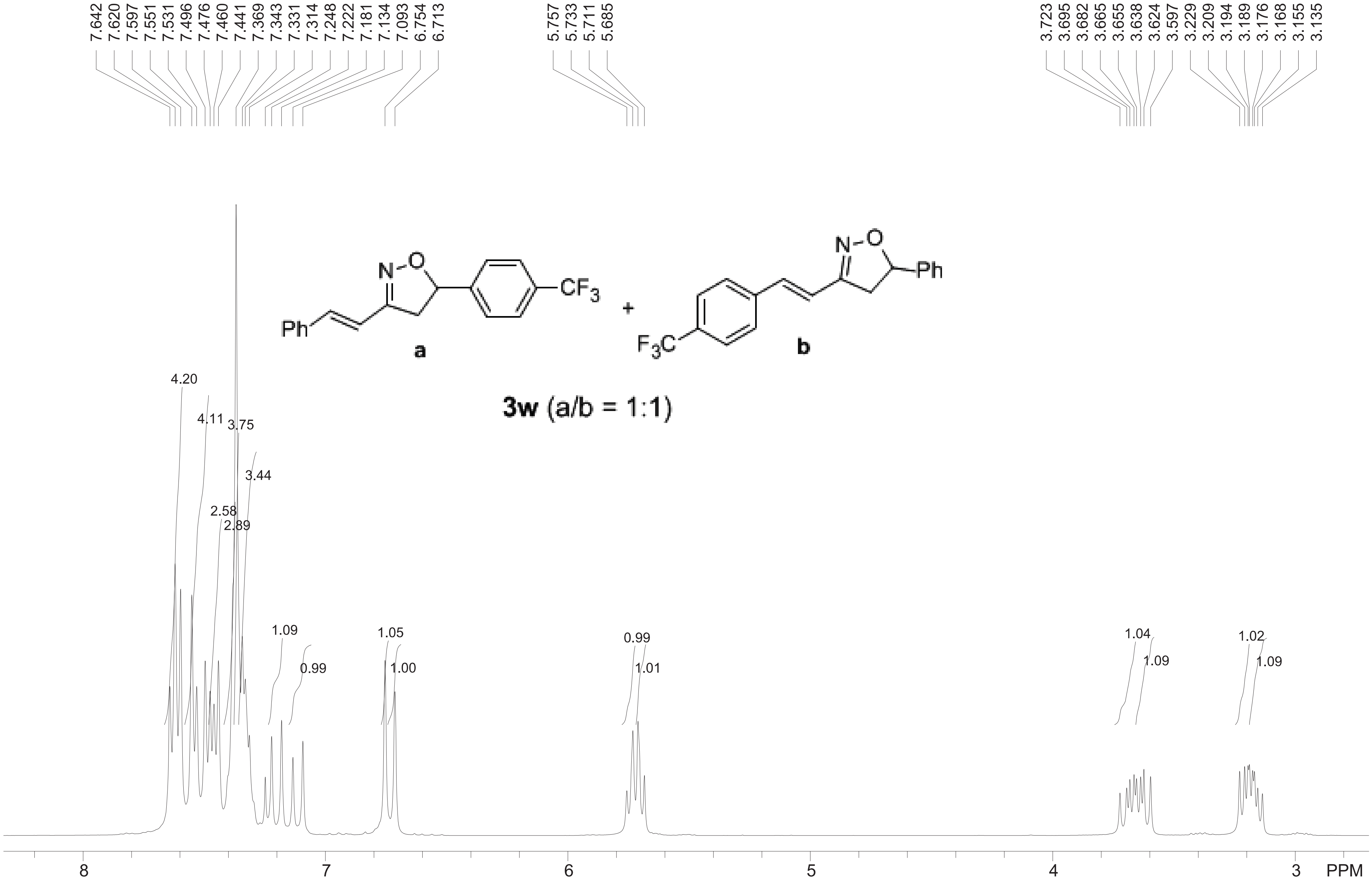

7.396
7.381
7.333
7.316
7.303
7.295
7.278
7.253
7.234
7.217
7.181
7.072
7.039
6.937
6.905
6.840
6.824
6.809
6.674
6.641
6.618
6.586
N-O
OMe
Ph
a
MeO
Ph
b
3v (a/b = 1:1)
6.10
6.05
1.10
1.22
1.01
1.00
2.19
2.13
1.10
0.96
7.4
7.2
7.0
6.8
6.6
PPM

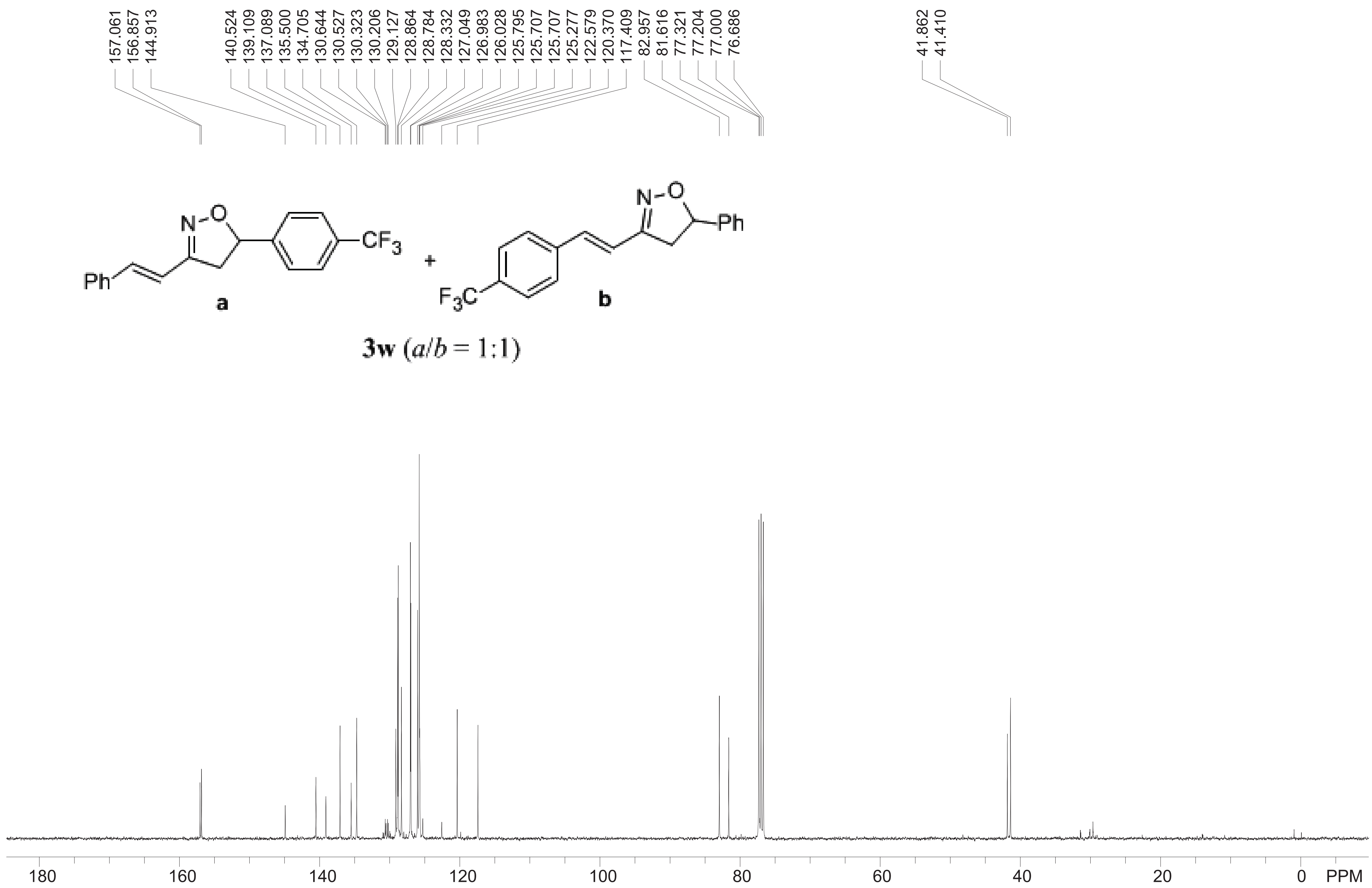

5.610
5.589
5.578
5.559
5.540
3.750
3.733
3.573
3.551
3.538
3.516
3.504
3.483
3.135
3.118
3.103
3.086
N-O
OMe
Ph
a
+
MeO
b
Ph
3v (a/b = 1:1)
3.16
3.18
1.04
1.06
1.07
1.09
1.03
1.04
5.5
5.0
4.5
4.0
3.5
3.0 PPM

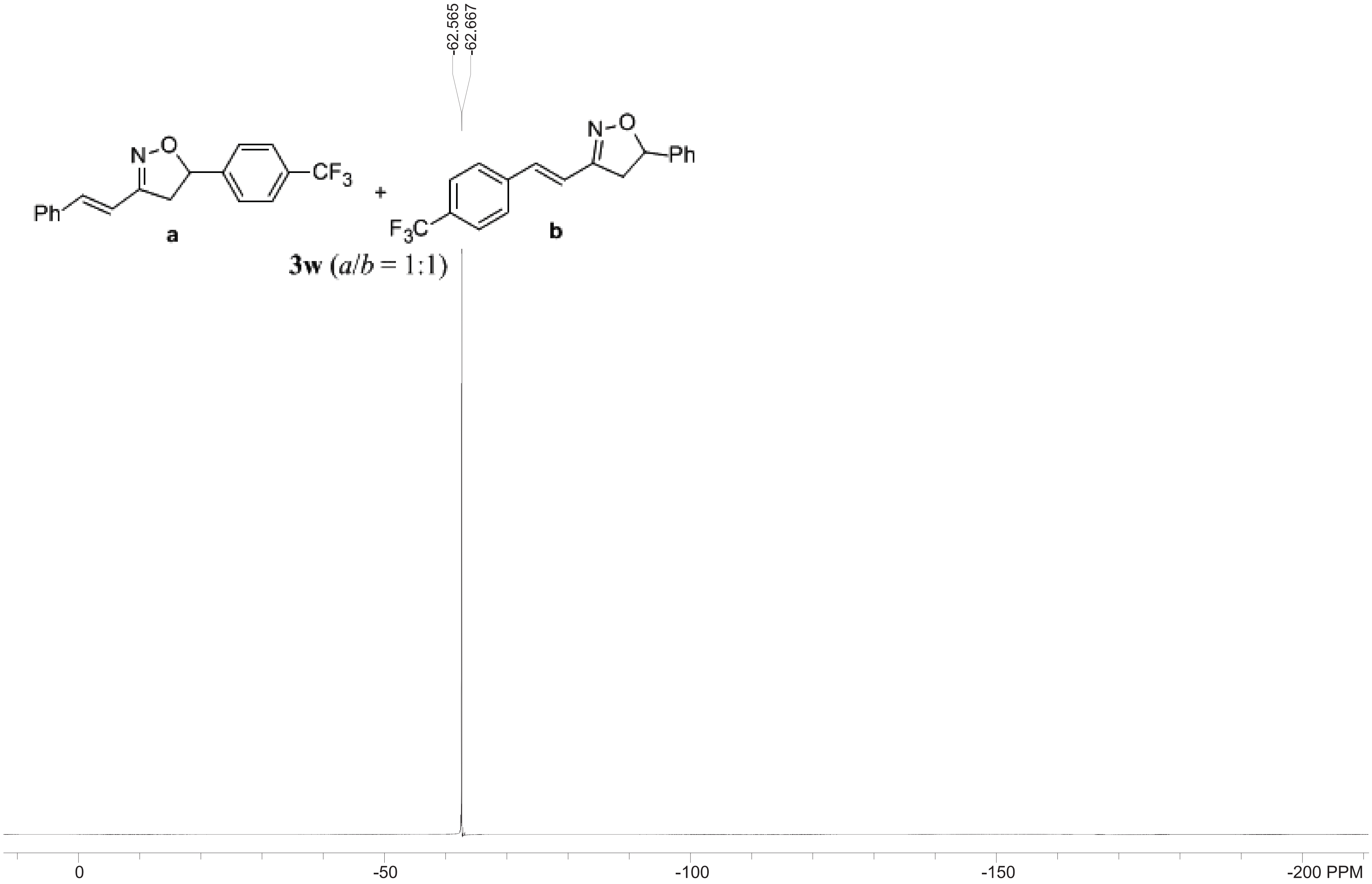

160.283
159.598
157.397
140.876
136.459
136.219
135.723
132.597
128.939
128.844
128.713
128.465
128.348
128.173
127.350
126.935
125.827
117.978
115.602
114.283
114.094
82.575
82.437
77.255
77.000
76.745
55.298
41.786
41.386
N-O
OMe
Ph
a
+
N-O
Ph
MeO
b
3v (a/b = 1:1)
180
160
140
120
100
80
60
40
20
0 PPM

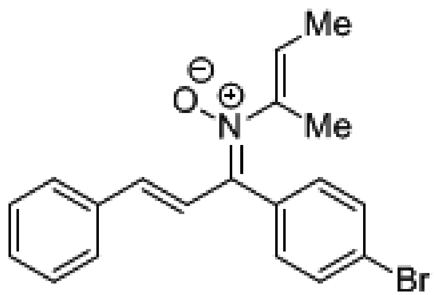


7.642 7.620 7.597 7.551 7.531 7.496 7.476 7.460 7.441 7.369 7.343 7.331 7.314 7.248 7.222 7.181 7.134 7.093 6.754 6.713 5.757 5.733 5.711 5.685
3.723 3.695 3.682 3.665 3.655 3.638 3.624 3.597 3.229 3.209 3.194 3.189 3.176 3.168 3.155 3.135
0.000
N-O
CF3
Ph
a
+
b
F3C
3w (a/b = 1:1)
4.20
4.11
3.75
3.44
2.58
2.89
1.09
0.99
1.05
1.00
0.99
1.01
1.04
1.09
1.02
1.09
9 8 7 6 5 4 3 2 1 0 PPM

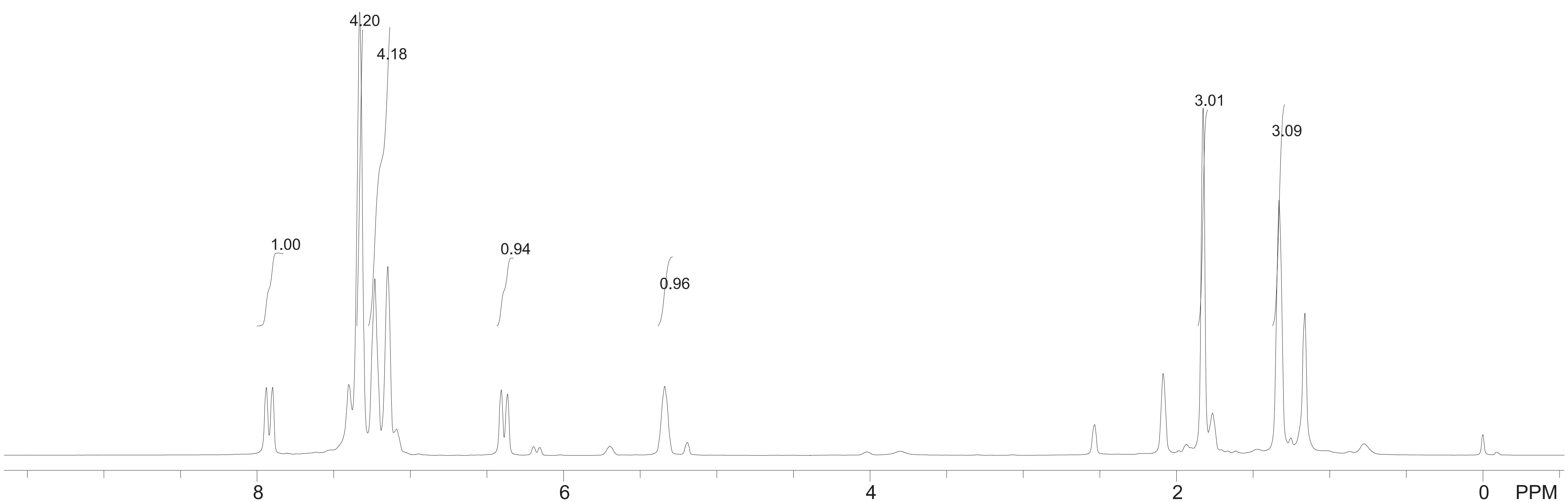

7.642 7.620 7.597 7.551 7.531 7.496 7.476 7.460 7.441 7.369 7.343 7.331 7.314 7.248 7.222 7.181 7.134 7.093 6.754 6.713
5.757 5.733 5.711 5.685
3.723 3.695 3.682 3.665 3.655 3.638 3.624 3.597 3.229 3.209 3.194 3.189 3.176 3.168 3.155 3.135
N-O
CF3
Ph
a
+
N-O
Ph
F3C
b
3w (a/b = 1:1)
4.20
4.11
3.75
3.44
2.58
2.89
1.09
0.99
1.05
1.00
0.99
1.01
1.04
1.09
1.02
1.09
8
7
6
5
4
3
PPM

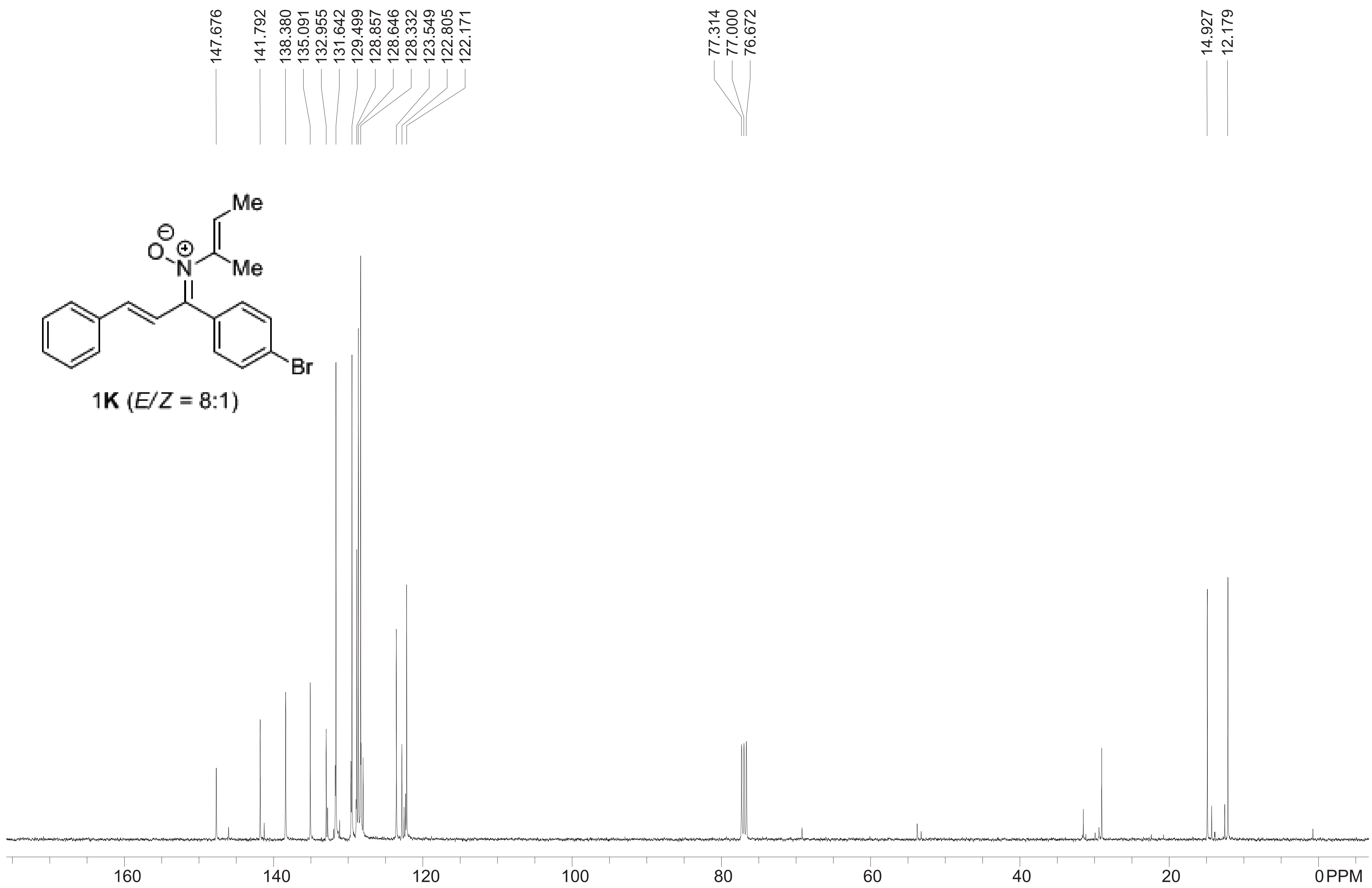

157.061
156.857
144.913
140.524
139.109
137.089
135.500
134.705
130.644
130.527
130.323
130.206
129.127
128.864
128.784
128.332
127.049
126.983
126.028
125.795
125.707
125.707
125.277
122.579
120.370
117.409
82.957
81.616
77.321
77.204
77.000
76.686
41.862
41.410
a
b
3w (a/b = 1:1)
180
160
140
120
100
80
60
40
20
0
PPM


-62.565
-62.667
N-O
CF3
Ph
a
+
F3C
Ph
b
3w (a/b = 1:1)
0
-50
-100
-150
-200 PPM

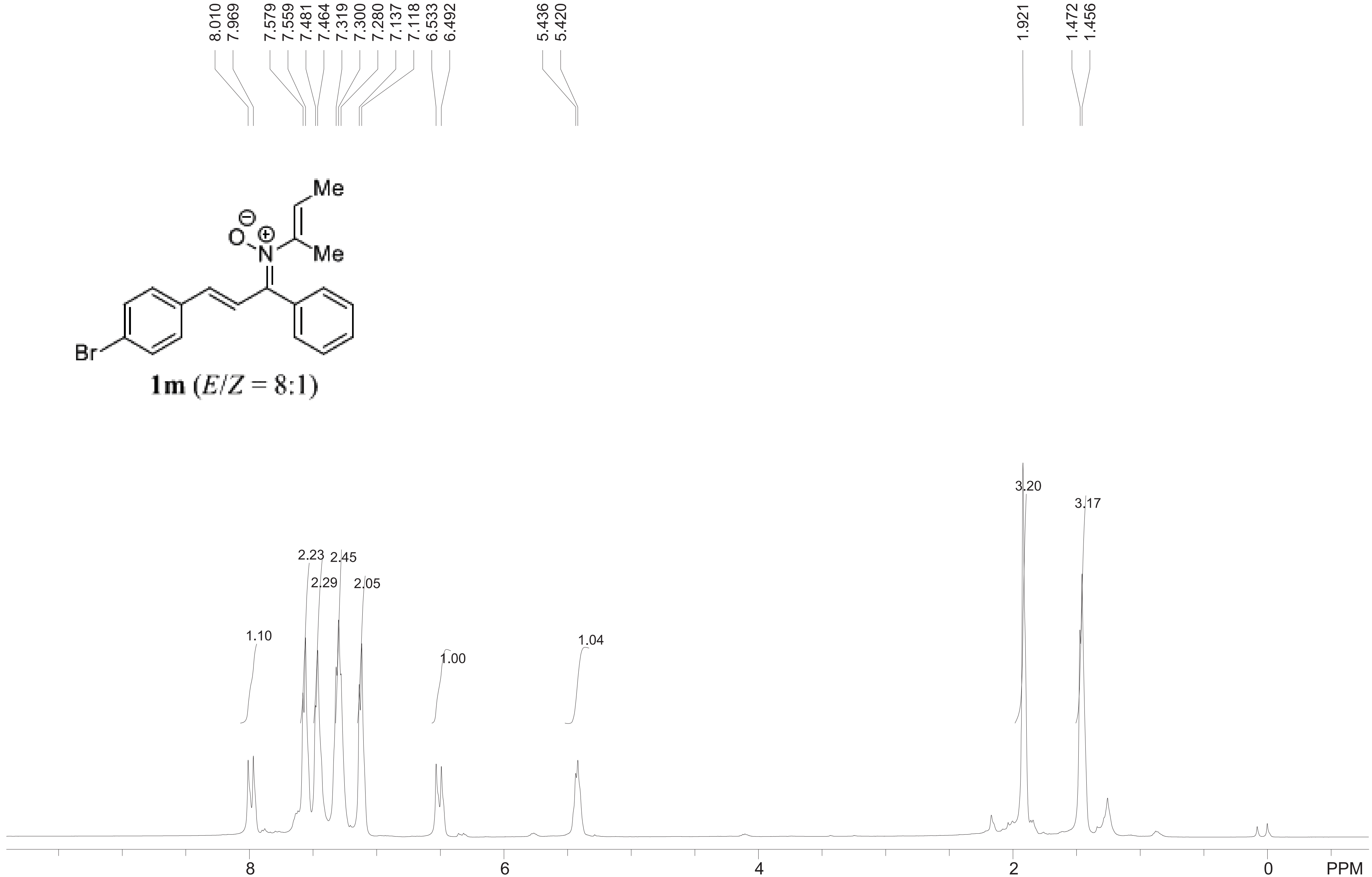

Me
O
N
Me
Br

1**K** (*E*/*Z* = 8:1)

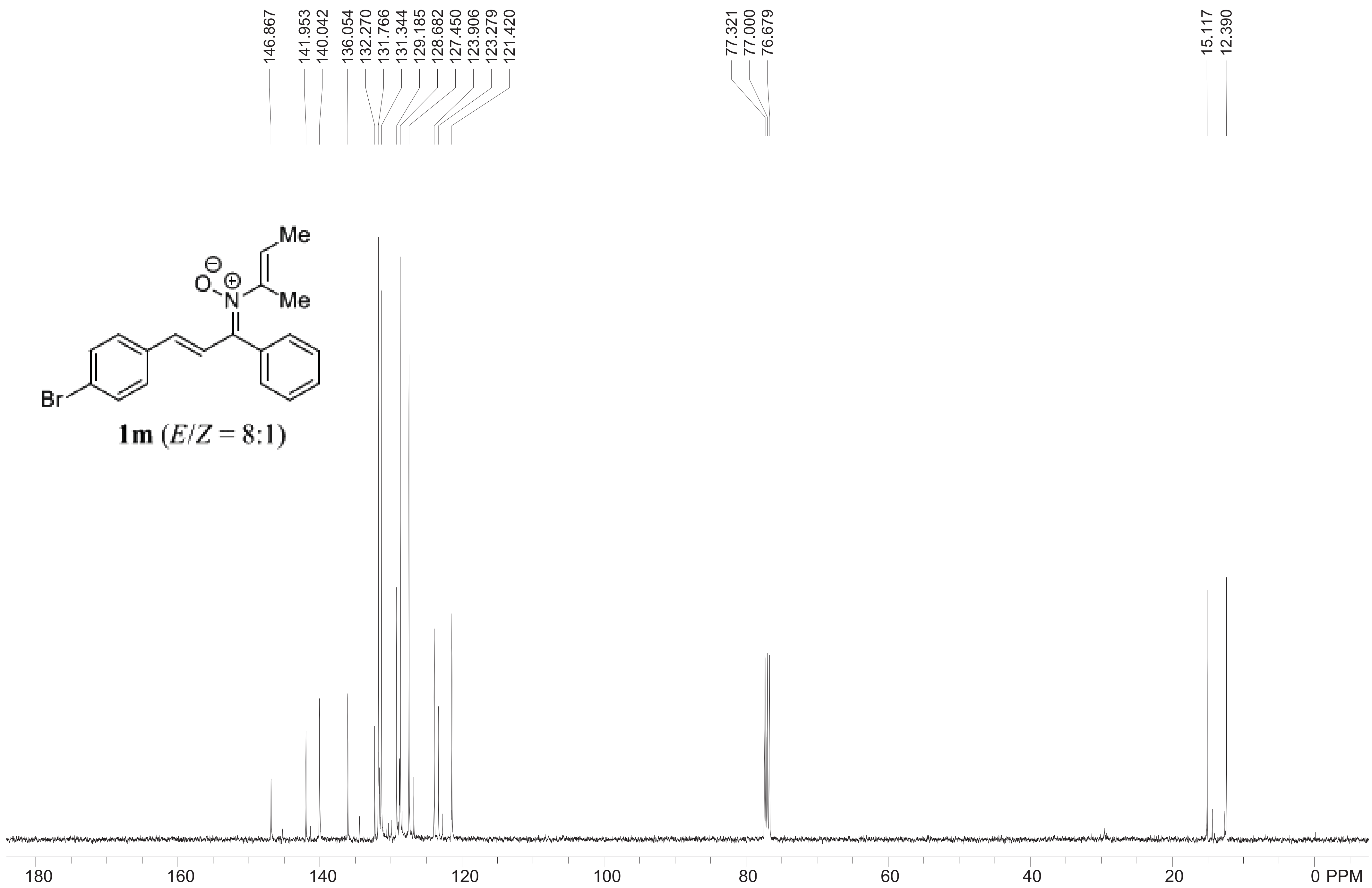

7.939
7.899
7.402
7.330
7.231
7.147
6.406
6.365
5.340
1.826
1.331
1.00
4.20
4.18
0.94
0.96
3.01
3.09
8
6
4
2
0
PPM

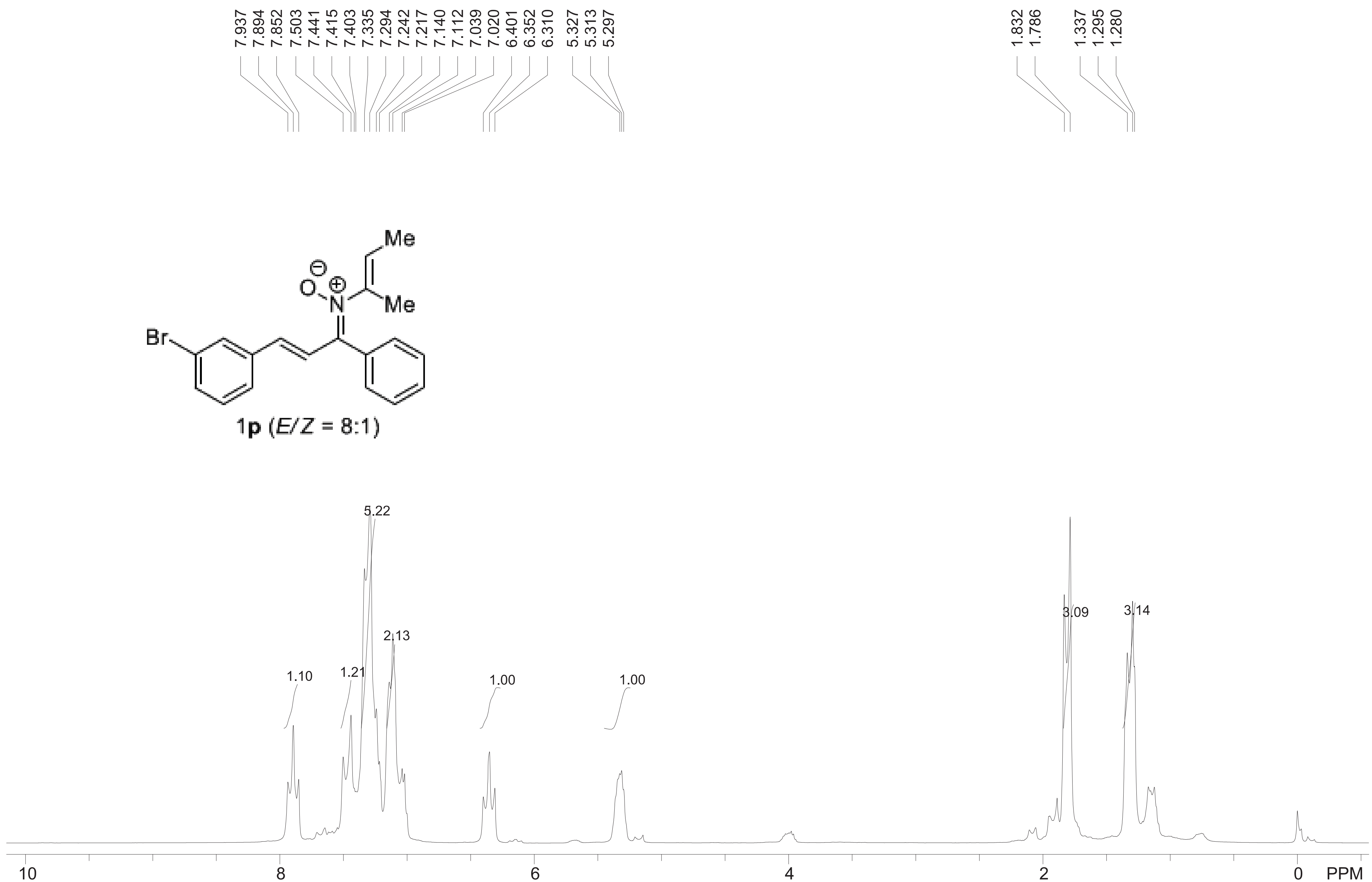

147.676
141.792
138.380
135.091
132.955
131.642
129.499
128.857
128.646
128.332
123.549
122.805
122.171
77.314
77.000
76.672
14.927
12.179
Me
Me
O
N
Br
160
140
120
100
80
60
40
20
0 PPM

1**K** (*E*/*Z* = 8:1)

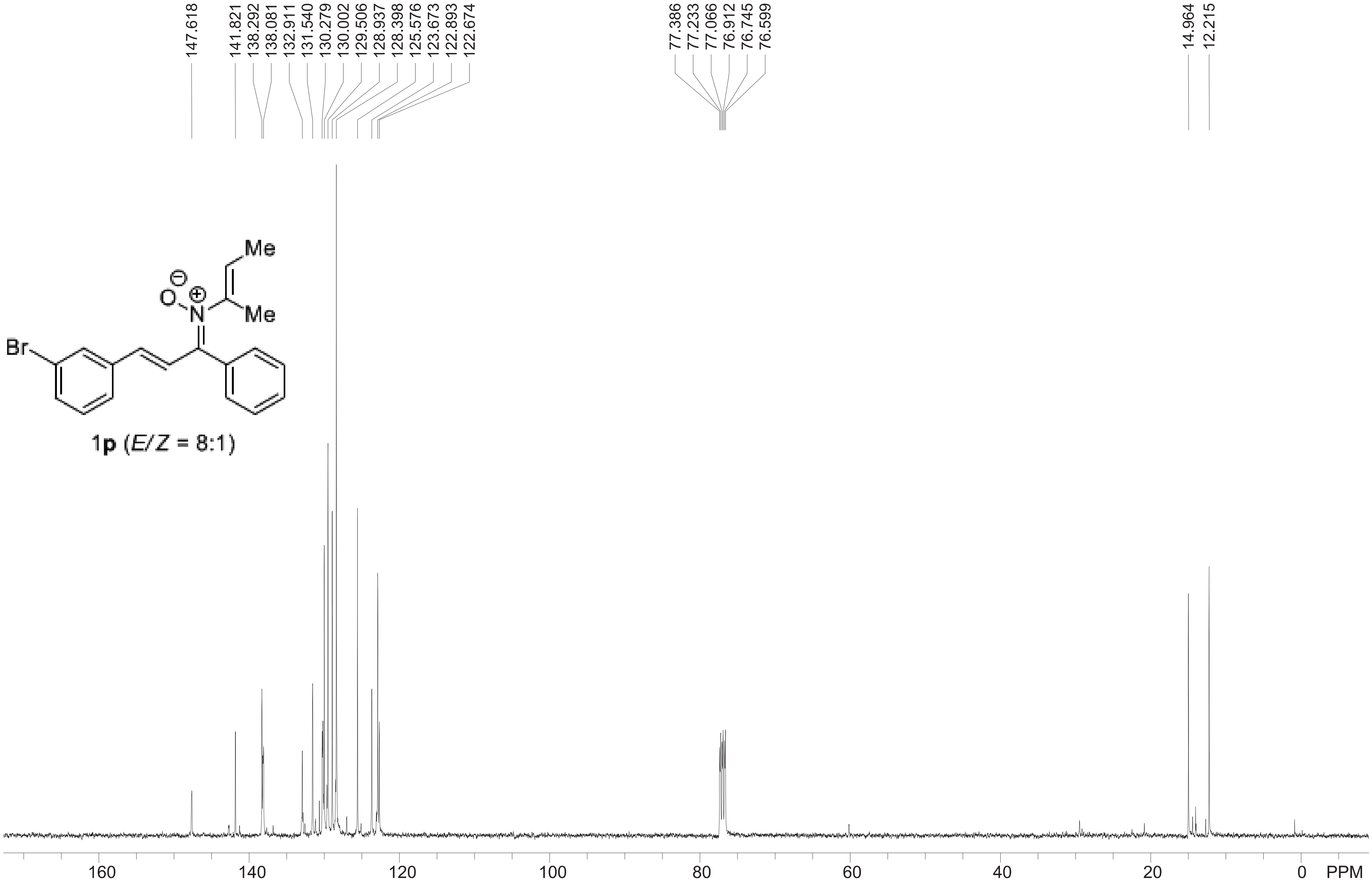

8.010
7.969
7.579
7.559
7.481
7.464
7.319
7.300
7.280
7.137
7.118
6.533
6.492
5.436
5.420
1.921
1.472
1.456
Me
O
N
Me
Br
1.10
2.23
2.29
2.45
2.05
1.00
1.04
3.20
3.17
8
6
4
2
0
PPM

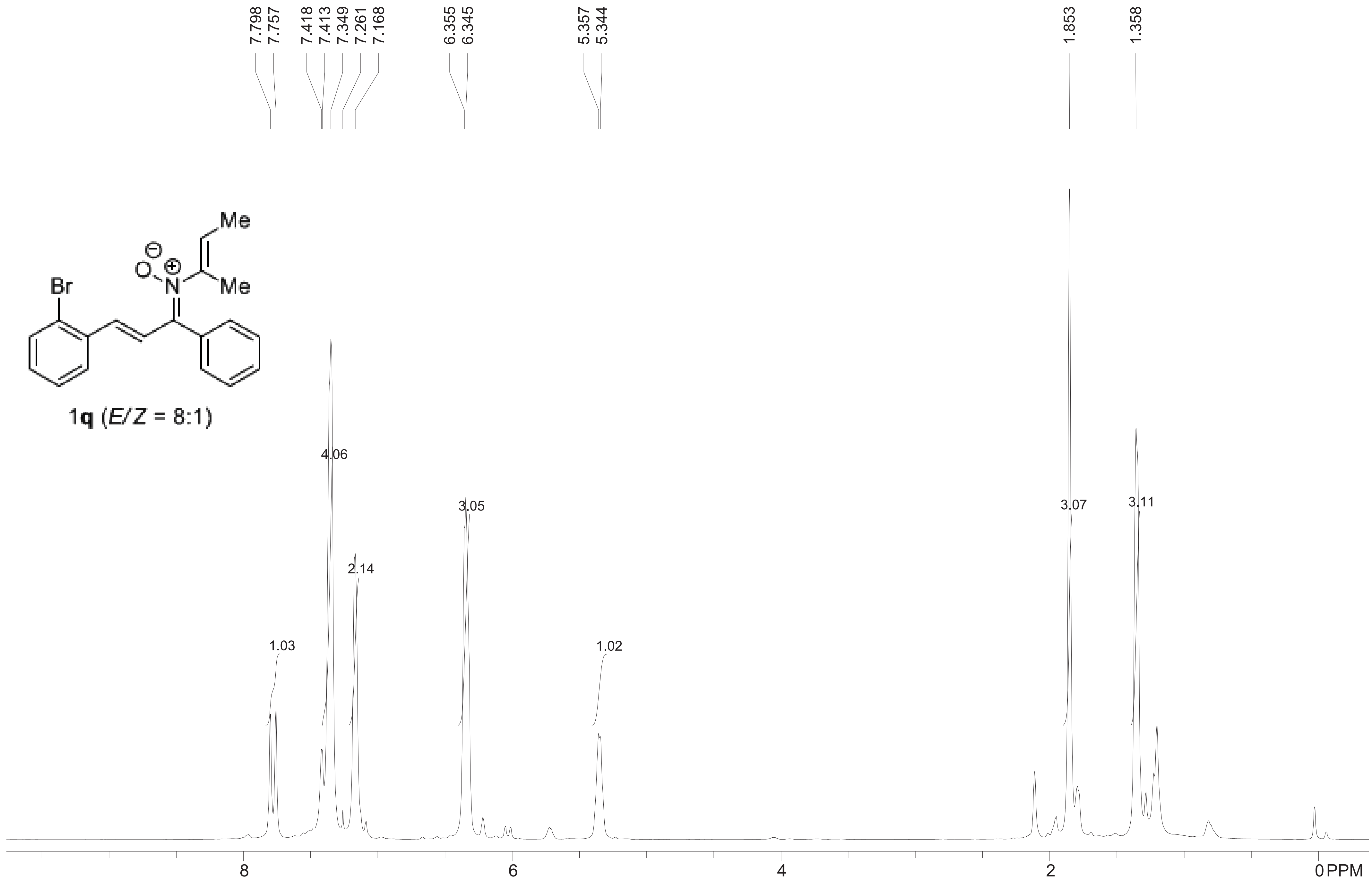
1m (*E*/Z = 8:1)

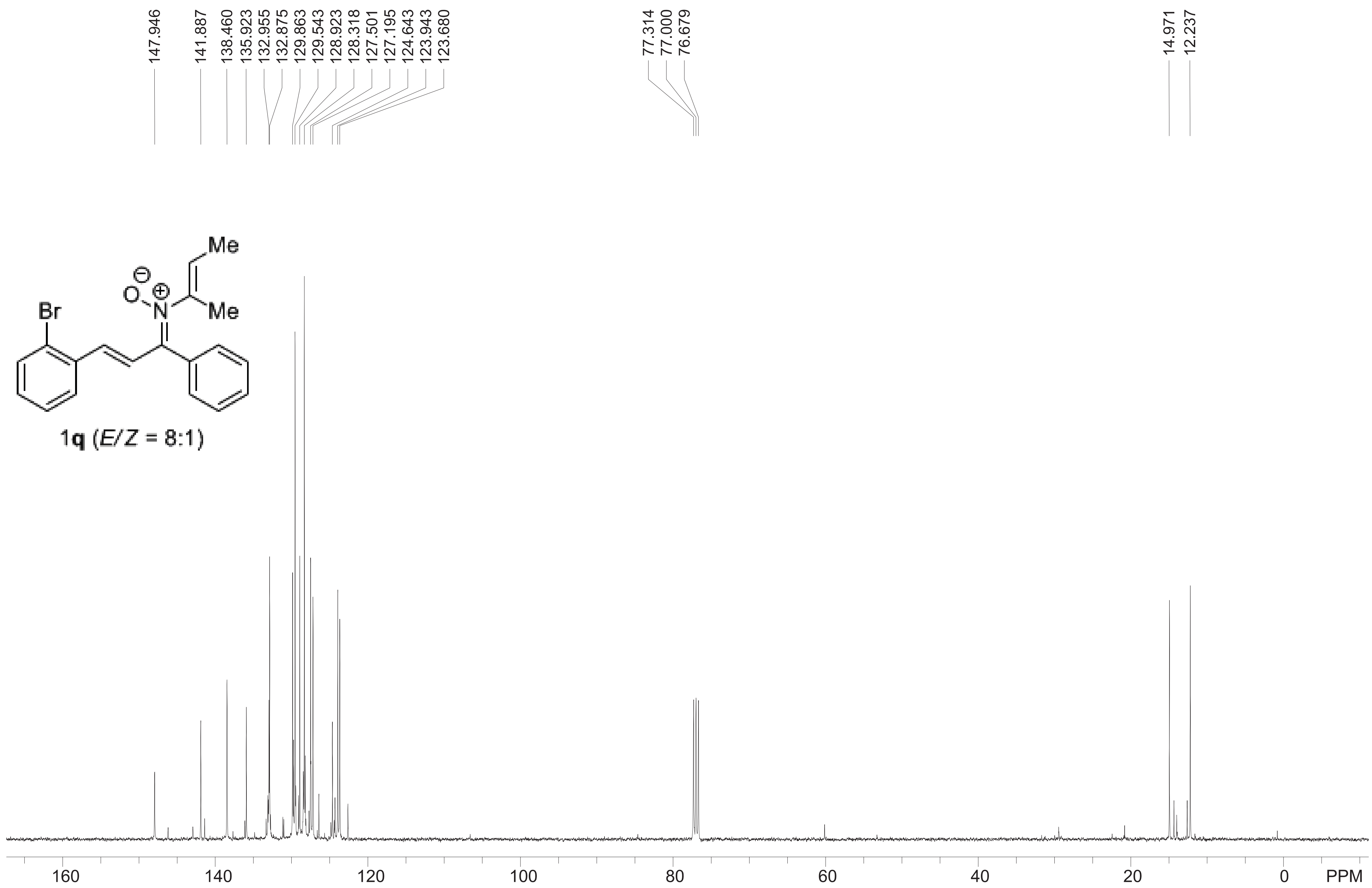

146.867
141.953
140.042
136.054
132.270
131.766
131.344
129.185
128.682
127.450
123.906
123.279
121.420
77.321
77.000
76.679
15.117
12.390
Me
O
N
Me
Br
1m (E/Z = 8:1)
180
160
140
120
100
80
60
40
20
0 PPM


7.937
7.894
7.852
7.503
7.441
7.415
7.403
7.335
7.294
7.242
7.217
7.140
7.112
7.039
7.020
6.401
6.352
6.310
5.327
5.313
5.297
1.832
1.786
1.337
1.295
1.280
Me
O
N
Me
Br
1.10
1.21
5.22
2.13
1.00
1.00
3.09
3.14
10
8
6
4
2
0
PPM

1p (*E*/*Z* = 8:1)

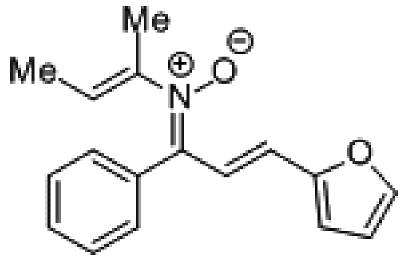

147.618
141.821
138.292
138.081
132.911
131.540
130.279
130.002
129.506
128.937
128.398
125.576
123.673
122.893
122.674
77.386
77.233
77.066
76.912
76.745
76.599
14.964
12.215
Me
Me
Br
1p (E/Z = 8:1)
160
140
120
100
80
60
40
20
0
PPM


7.798
7.757
7.418
7.413
7.349
7.261
7.168
6.355
6.345
5.357
5.344
1.853
1.358
Me
Br
Me
1q (E/Z = 8:1)
4.06
3.05
2.14
1.03
1.02
3.07
3.11
8
6
4
2
0 PPM

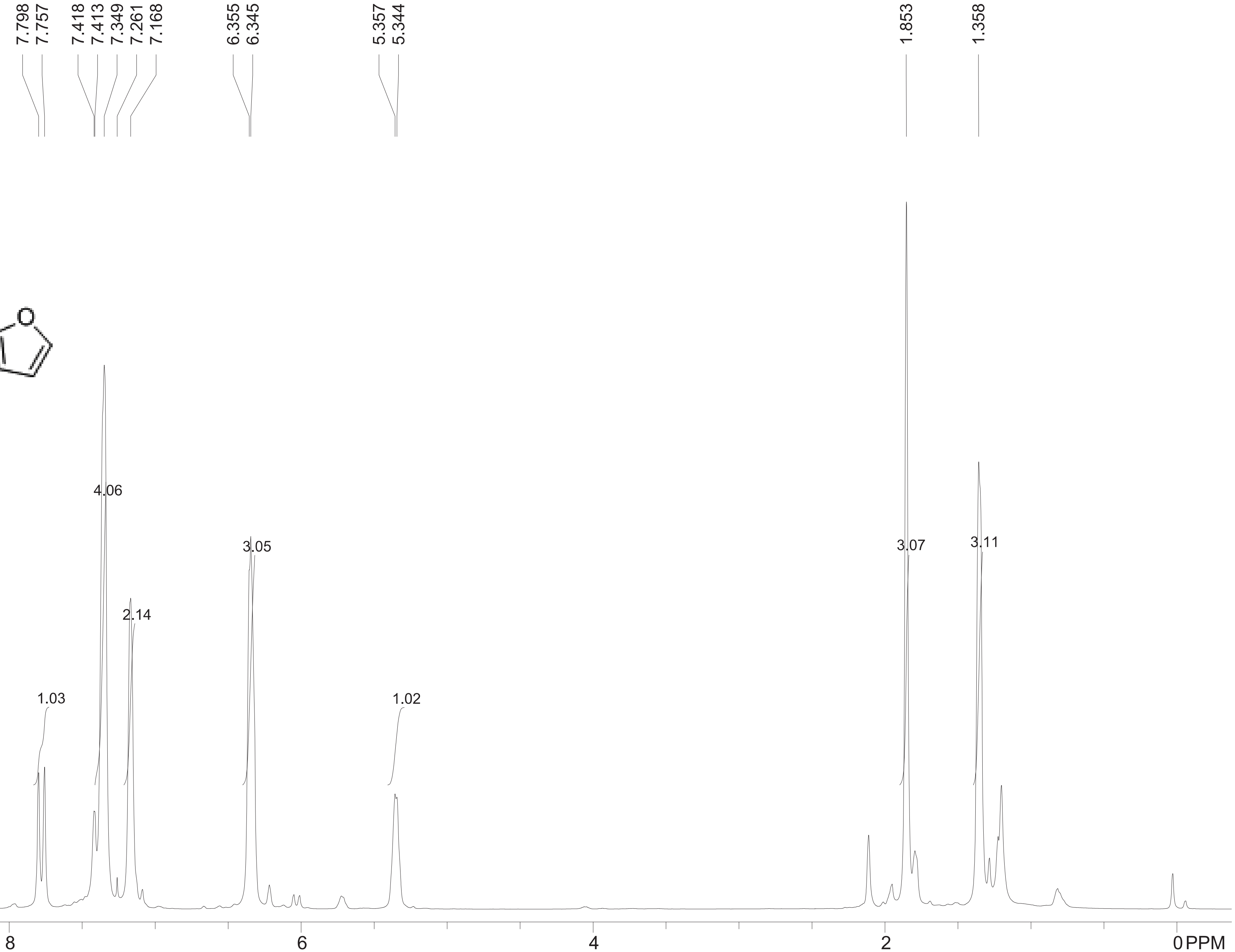


147.946
141.887
138.460
135.923
132.955
132.875
129.863
129.543
128.923
128.318
127.501
127.195
124.643
123.943
123.680
77.314
77.000
76.679
14.971
12.237
160
140
120
100
80
60
40
20
0
PPM
Me
Br
O
N
Me

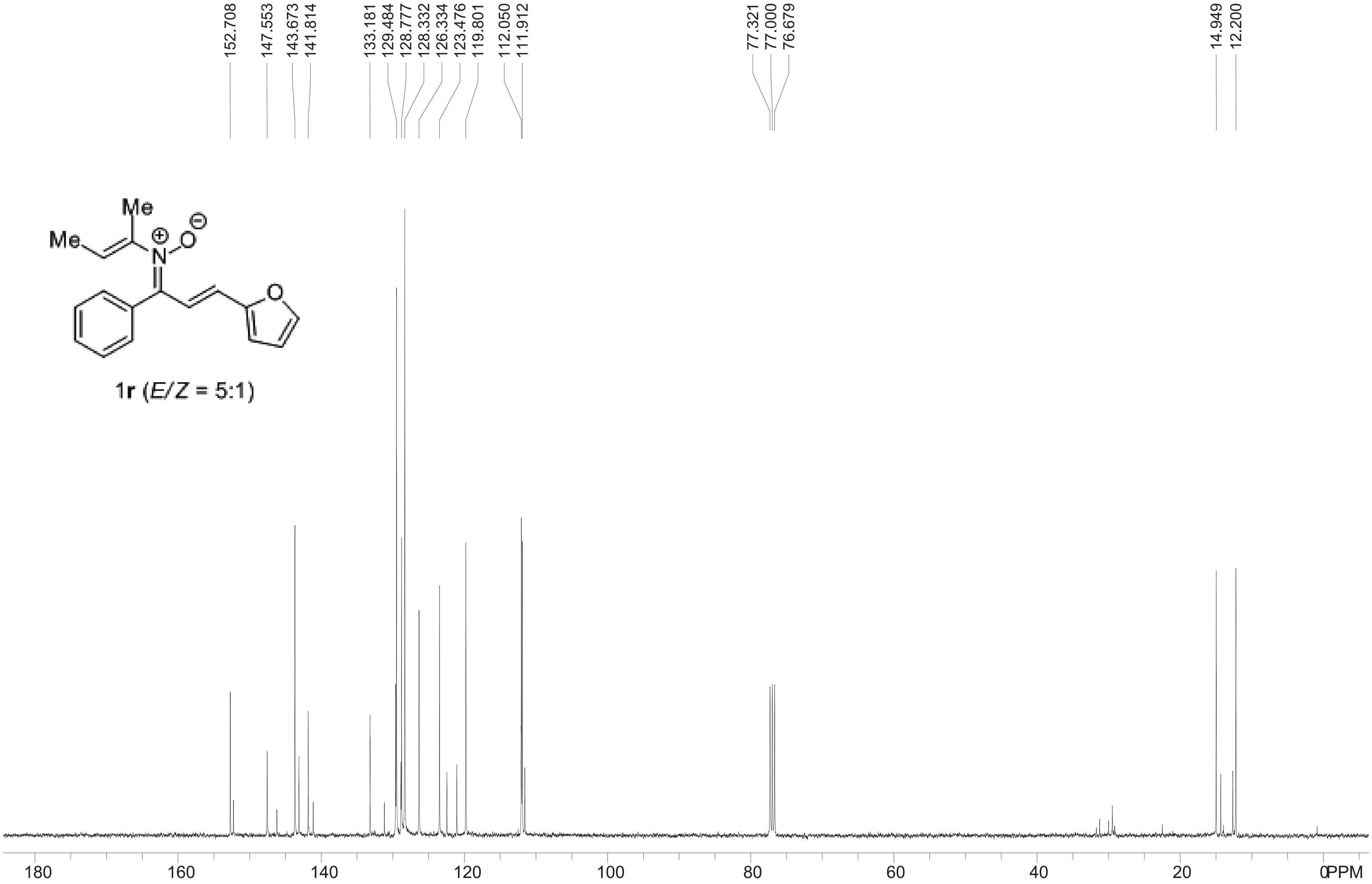
1q (*E*/*Z* = 8:1)



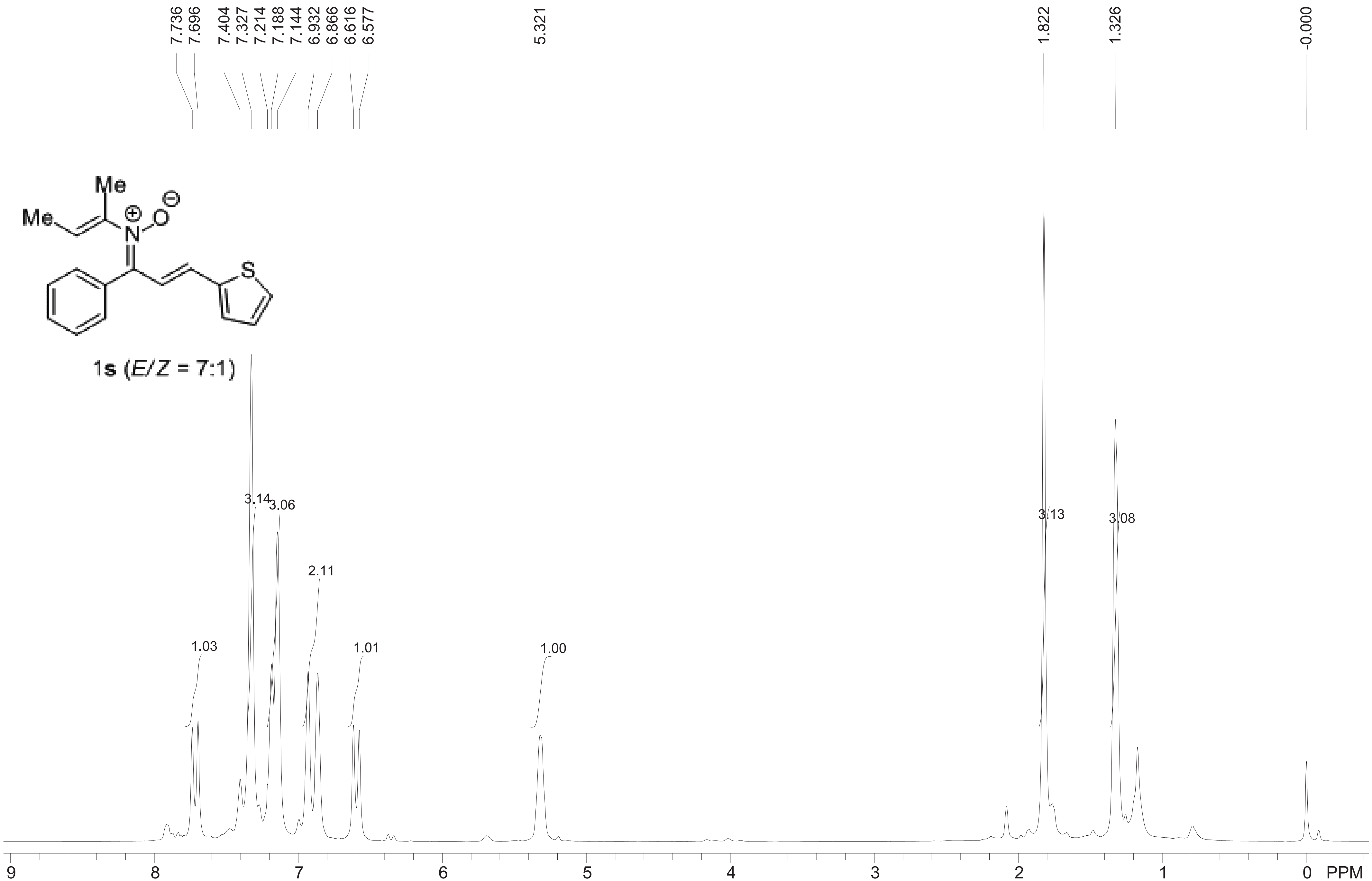
1r (*E*/*Z* = 5:1)



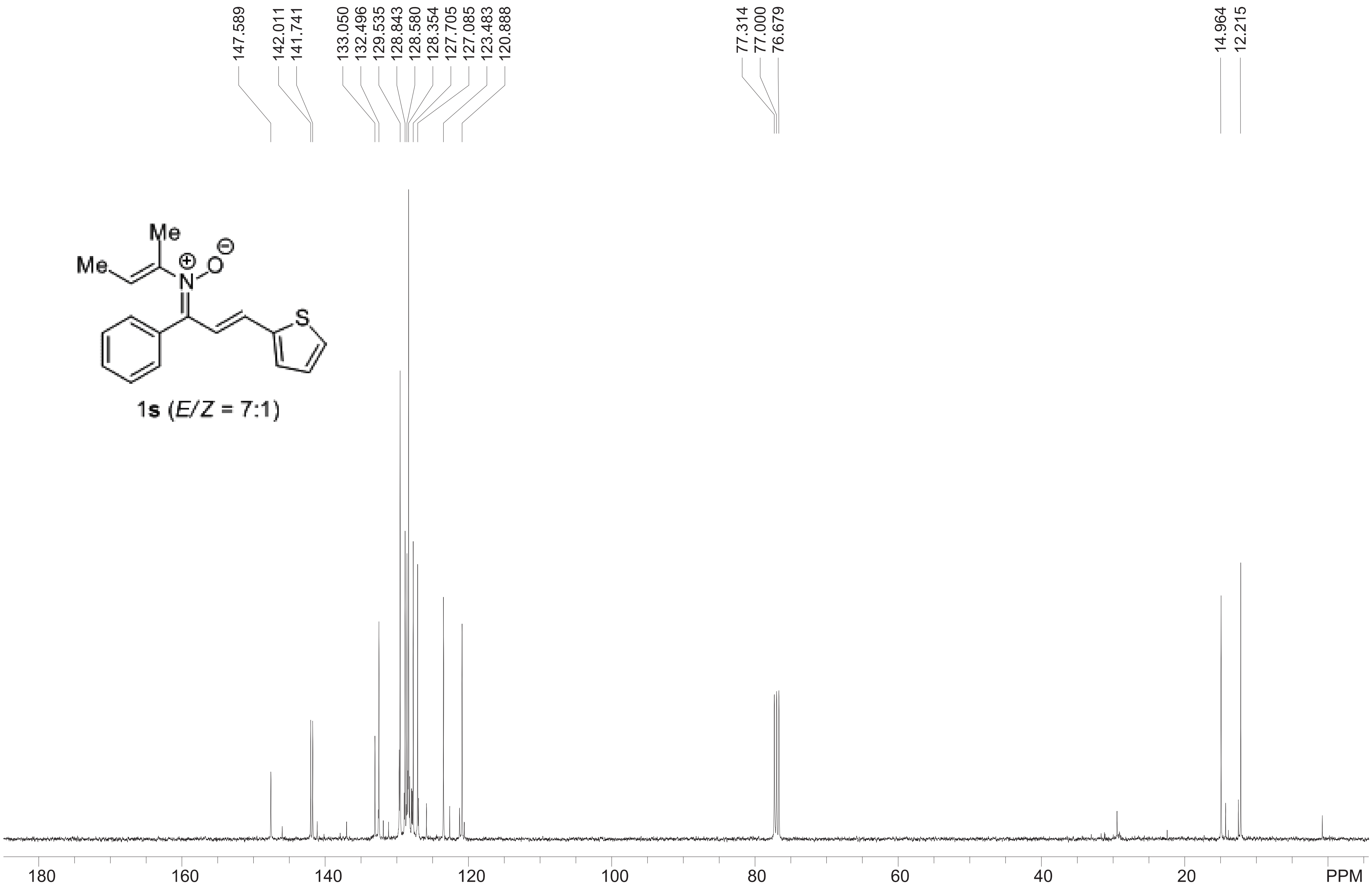
7.798
7.757
7.418
7.413
7.349
7.261
7.168
6.355
6.345
5.357
5.344
1.853
1.358
4.06
3.05
2.14
1.03
1.02
3.07
3.11
8
6
4
2
0 PPM



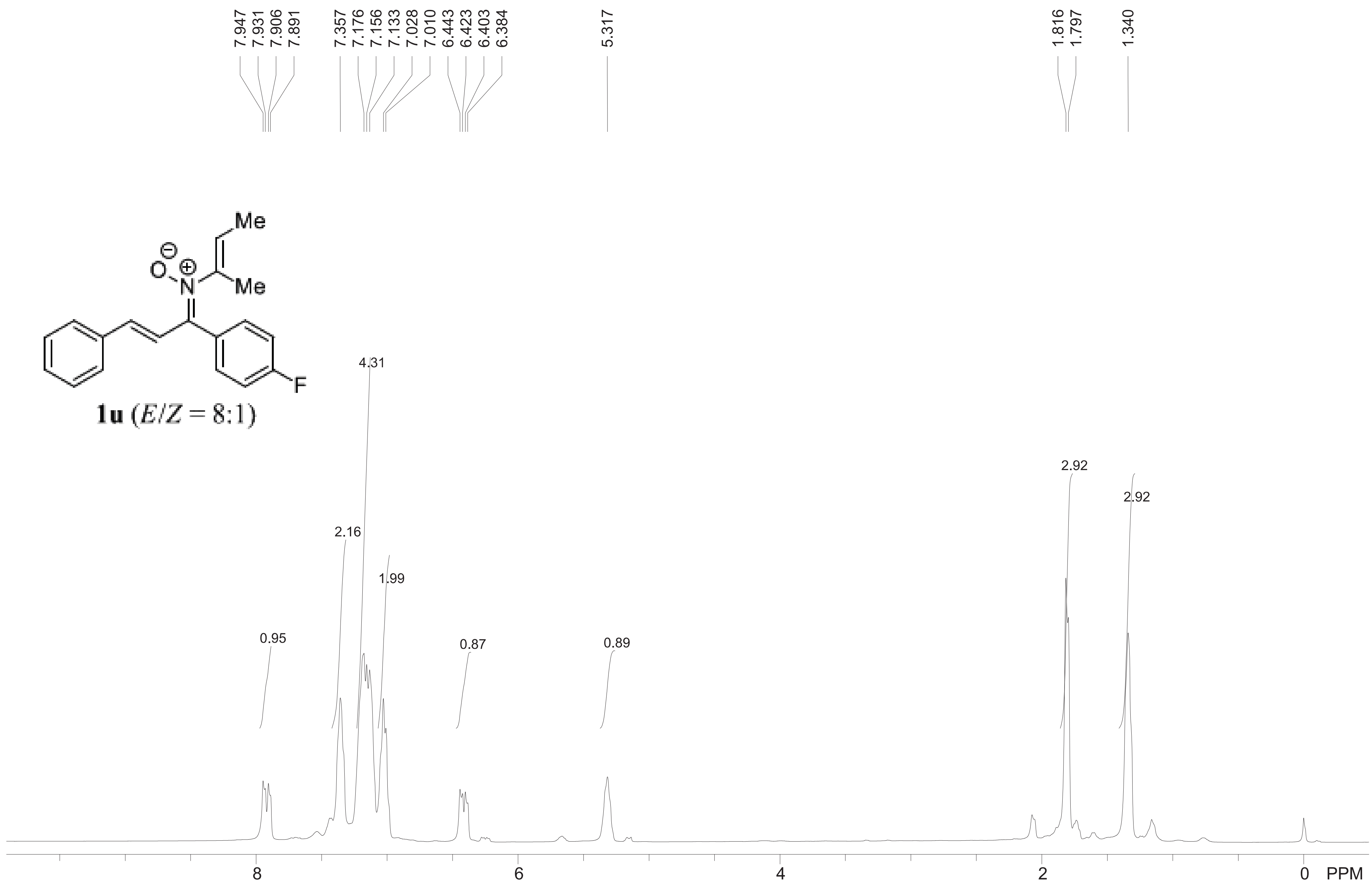
152.708
147.553
143.673
141.814
133.181
129.484
128.777
128.332
126.334
123.476
119.801
112.050
111.912
77.321
77.000
76.679
14.949
12.200
Me
Me
N
O
O
1r (E/Z = 5:1)
180
160
140
120
100
80
60
40
20
0
PPM


7.736
7.696
7.404
7.327
7.214
7.188
7.144
6.932
6.866
6.616
6.577
5.321
1.822
1.326
-0.000
1.03
3.14
3.06
2.11
1.01
1.00
3.13
3.08
9
8
7
6
5
4
3
2
1
0
PPM

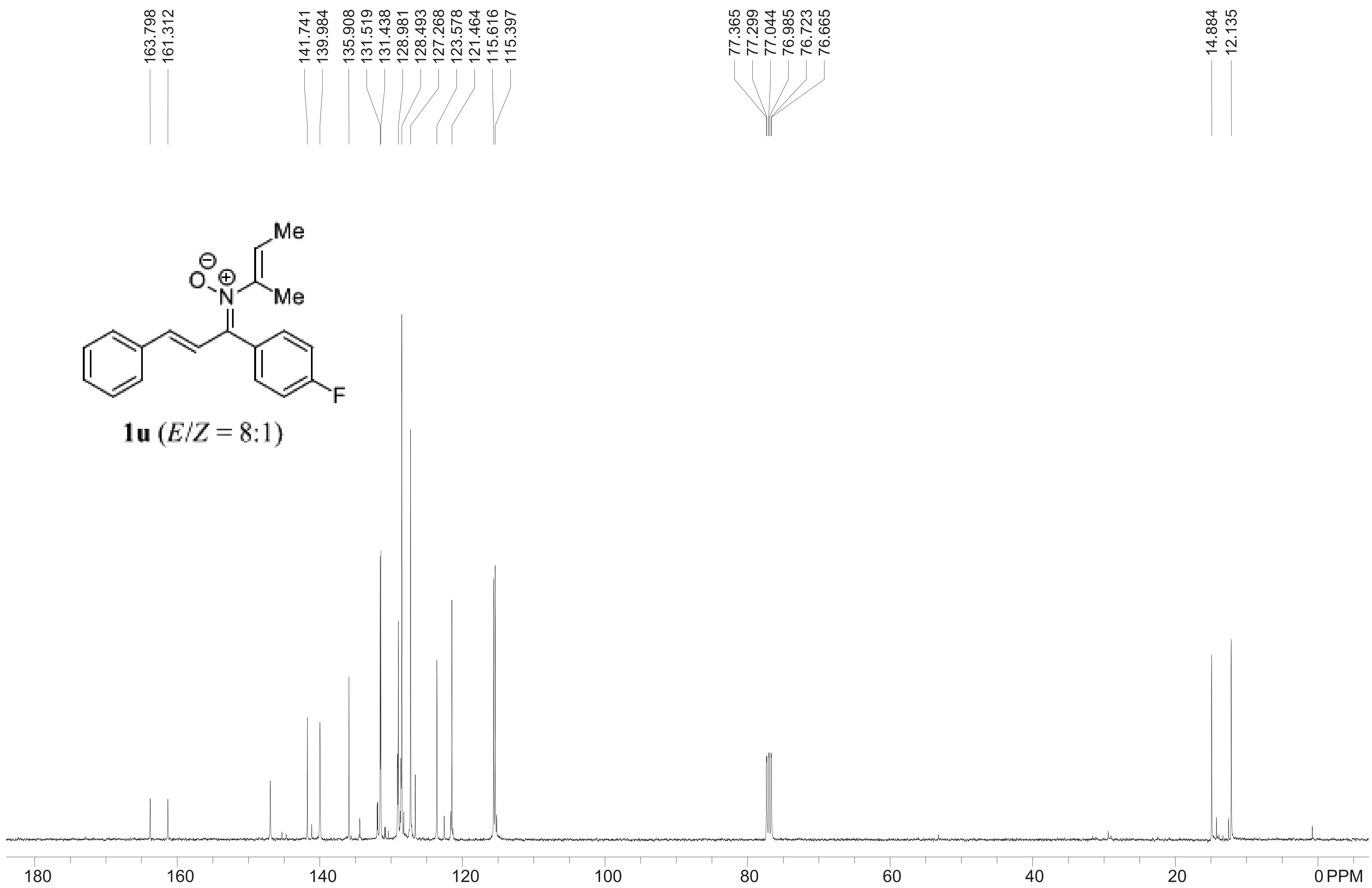
1s (*E*/*Z* = 7:1)


147.589
142.011
141.741
133.050
132.496
129.535
128.843
128.580
128.354
127.705
127.085
123.483
120.888
77.314
77.000
76.679
14.964
12.215
Me
Me
N
O
S
180
160
140
120
100
80
60
40
20
PPM

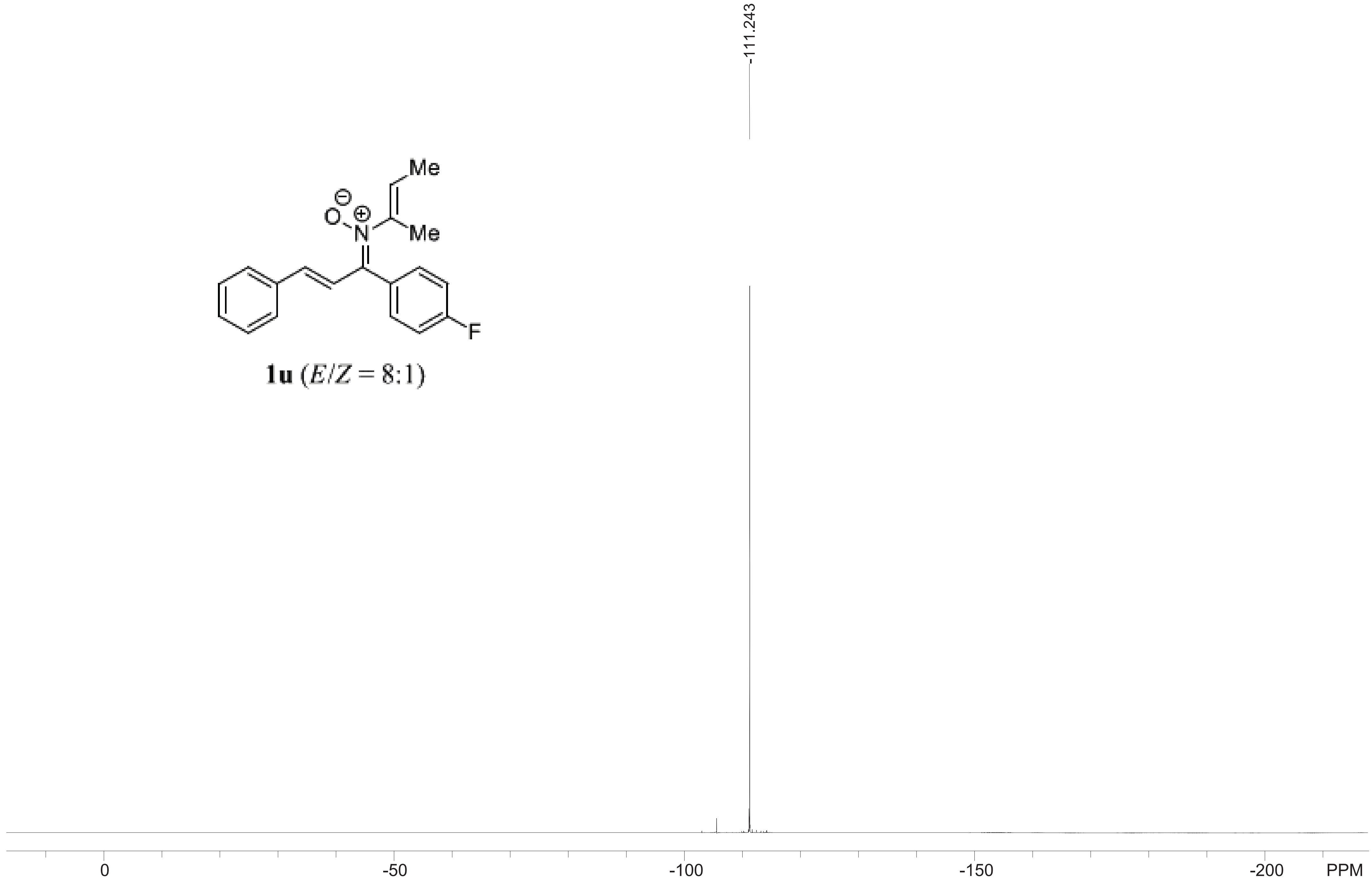
1s (*E*/*Z* = 7:1)

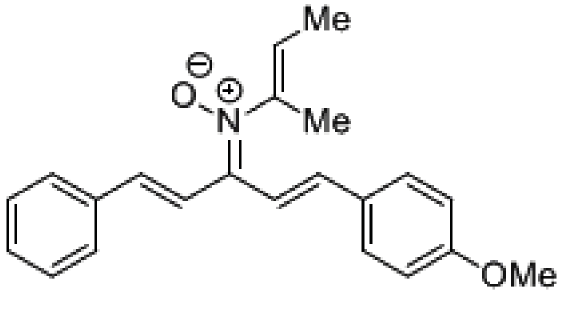

Me
Me
O
N
F
7.947
7.931
7.906
7.891
7.357
7.176
7.156
7.133
7.028
7.010
6.443
6.423
6.403
6.384
5.317
1.816
1.797
1.340
0.95
2.16
4.31
1.99
0.87
0.89
2.92
2.92
8
6
4
2
0
PPM

1u (*E*/*Z* = 8:1)

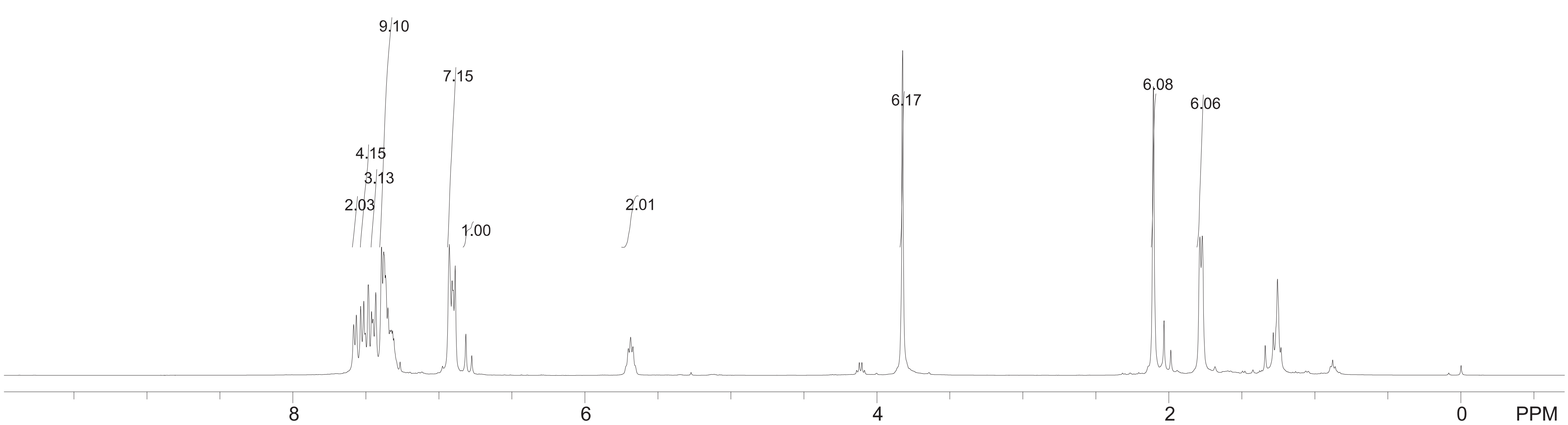


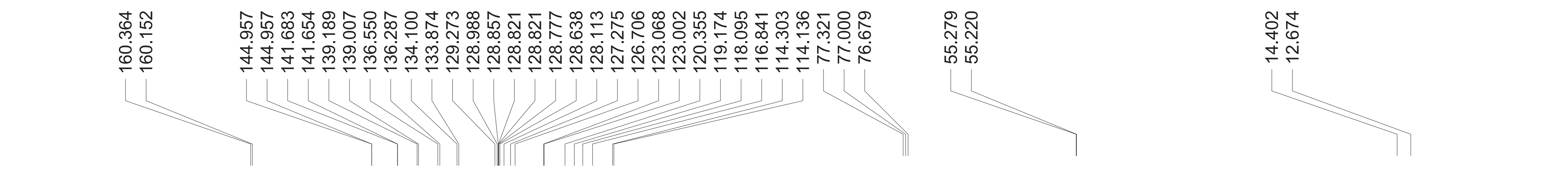
163.798
161.312
141.741
139.984
135.908
131.519
131.438
128.981
128.493
127.268
123.578
121.464
115.616
115.397
77.365
77.299
77.044
76.985
76.723
76.665
14.884
12.135
Me
Me
F
1u (E/Z = 8:1)
180
160
140
120
100
80
60
40
20
0 PPM


-111.243
Me
Me
F
1u (*E*/*Z* = 8:1)
0
-50
-100
-150
-200
PPM

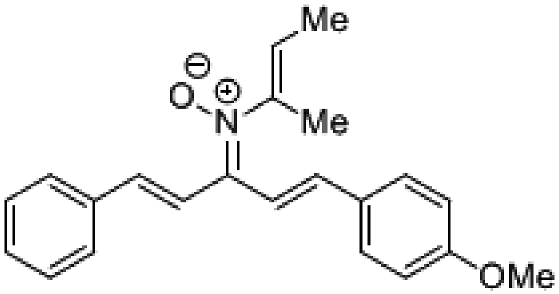

Me
Me
O
N
OMe

1v (*E*/*Z* = 1:1)


7.583
7.565
7.536
7.514
7.504
7.483
7.461
7.451
7.432
7.393
7.377
7.374
7.365
7.349
7.334
7.325
7.317
7.308
6.928
6.909
6.902
6.888
6.815
6.775
5.702
5.687
5.671
3.824
2.107
1.788
1.772
9.10
7.15
4.15
3.13
2.03
1.00
2.01
6.17
6.08
6.06
8
6
4
2
0
PPM

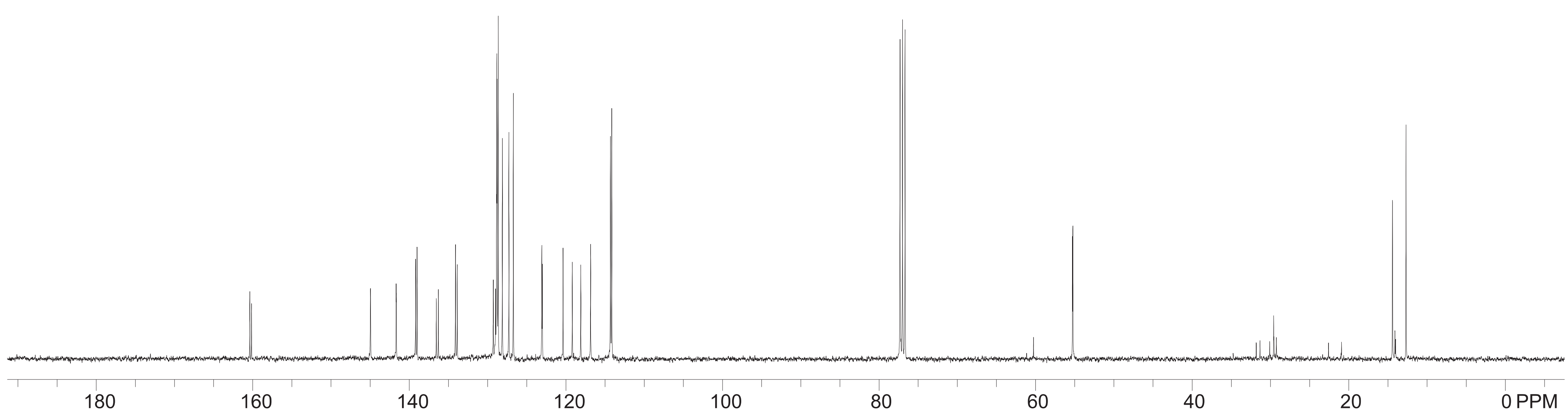


160.364
160.152
144.957
144.957
141.683
141.654
139.189
139.007
136.550
136.287
134.100
133.874
129.273
128.988
128.857
128.821
128.821
128.777
128.638
128.113
127.275
126.706
123.068
123.002
120.355
119.174
118.095
116.841
114.303
114.136
77.321
77.000
76.679
55.279
55.220
14.402
12.674

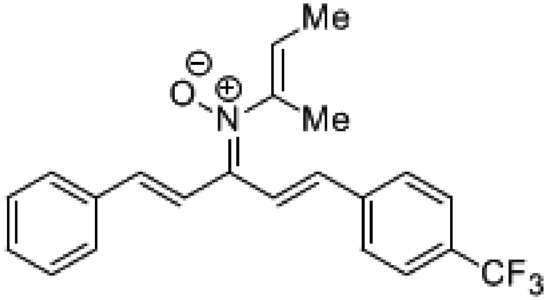


Me
O
N
Me
OMe

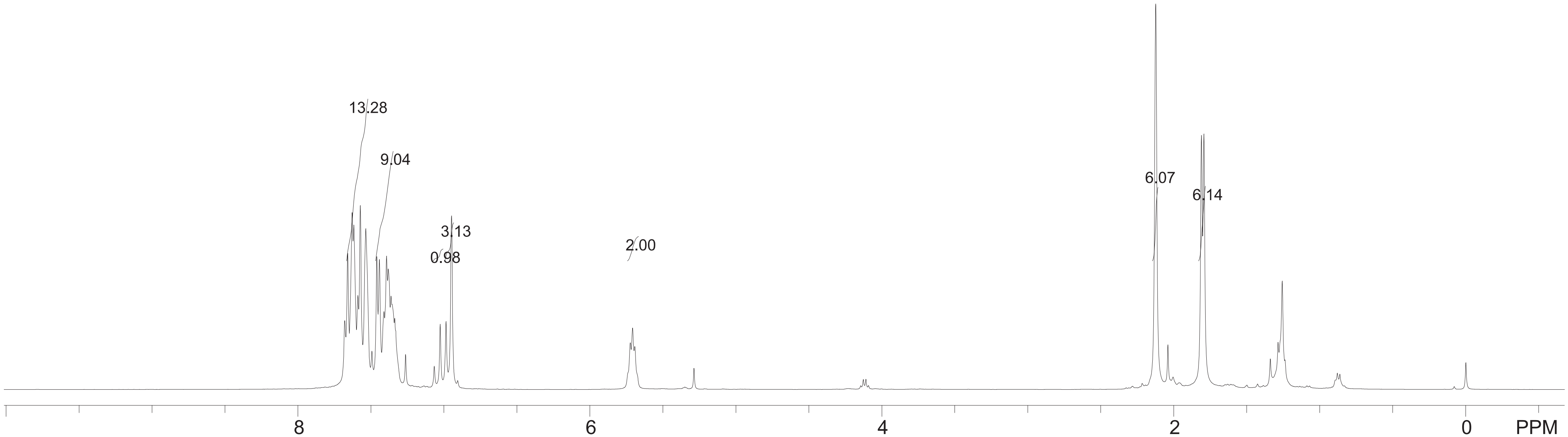
1v (*E*/*Z* = 1:1)

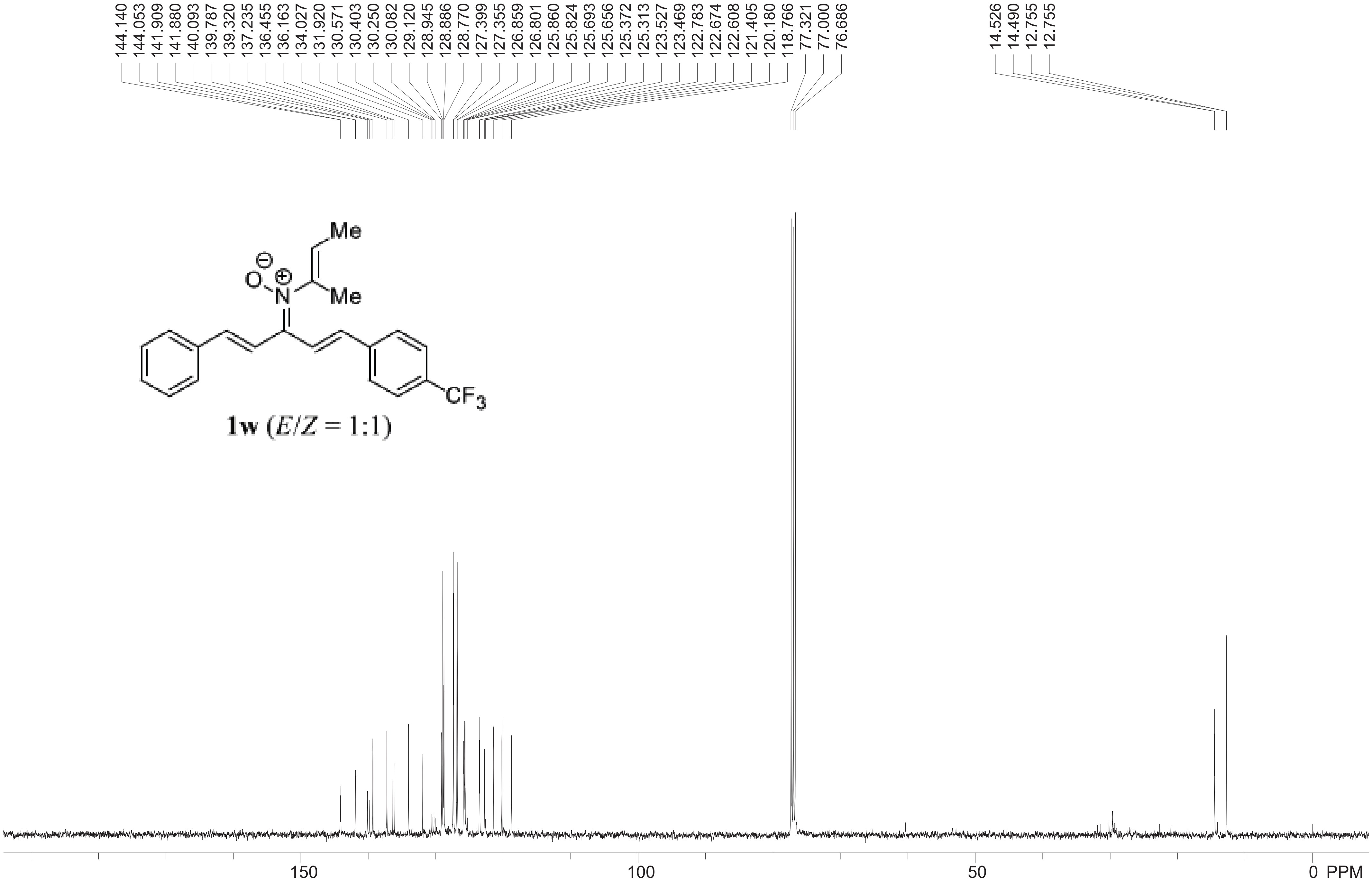


180
160
140
120
100
80
60
40
20
0 PPM

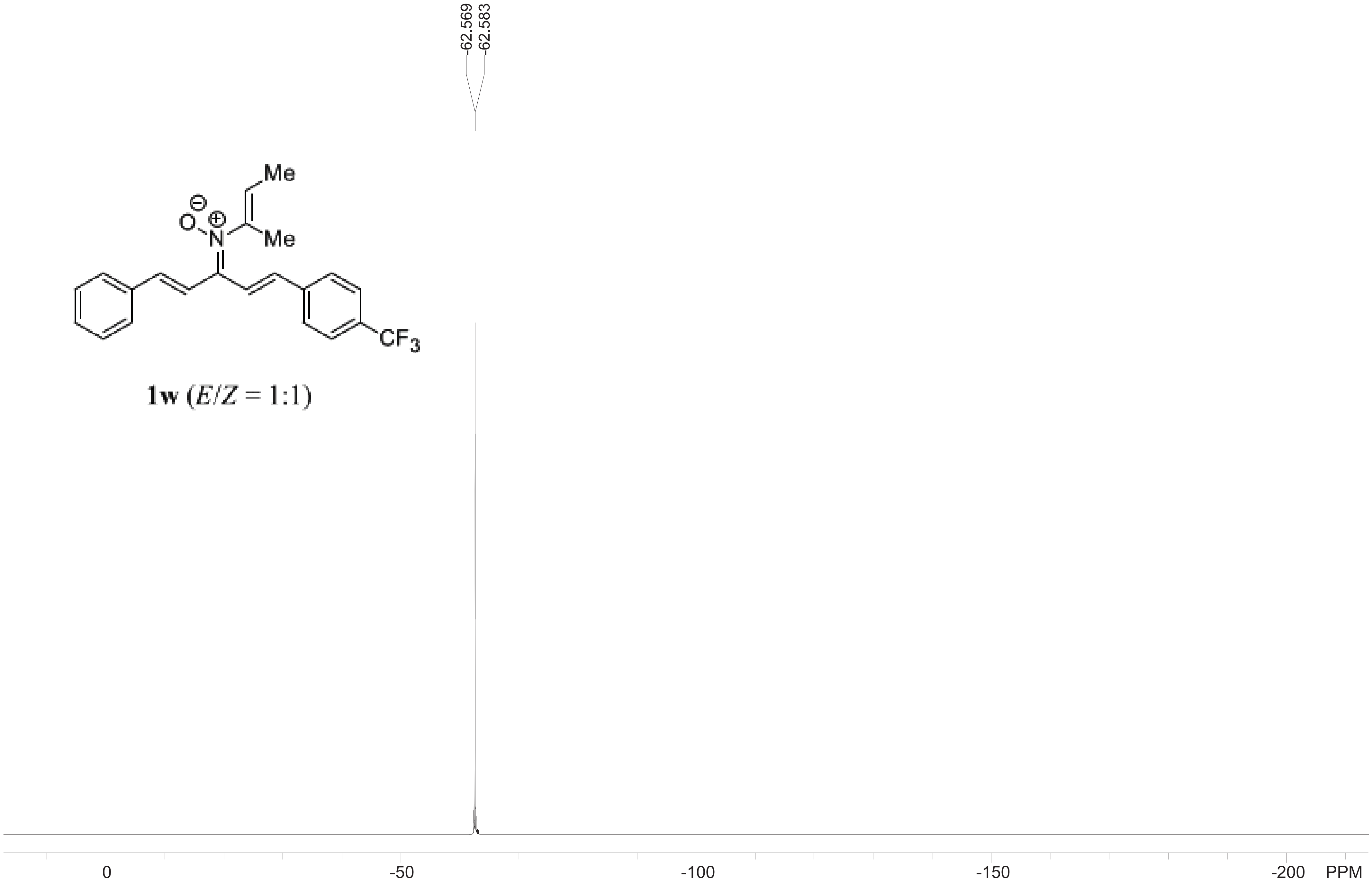

Me
O
N
Me
CF3

1w (*E*/*Z* = 1:1)


7.680 7.660 7.629 7.617 7.590 7.573 7.535 7.493 7.460 7.442 7.411 7.393 7.381 7.363 7.355 7.337 7.263 7.066 7.026 6.985 6.948 5.723 5.708 5.692
2.123 2.123 1.810 1.793
13.28
9.04
3.13
0.98
2.00
6.07
6.14
8
6
4
2
0
PPM

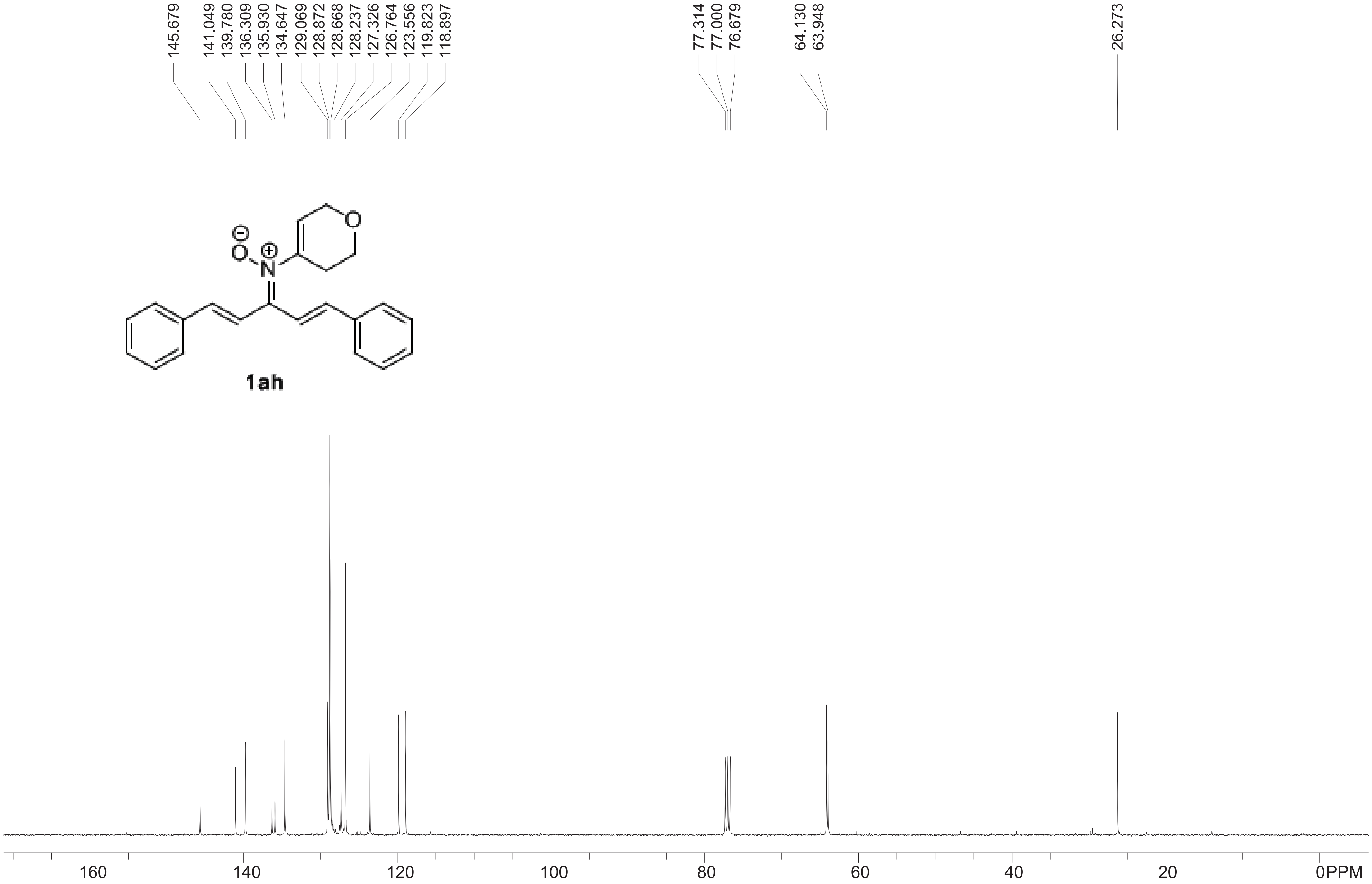

144.140
144.053
141.909
141.880
140.093
139.787
139.320
137.235
136.455
136.163
134.027
131.920
130.571
130.403
130.250
130.082
129.120
128.945
128.886
128.770
127.399
127.355
126.859
126.801
125.860
125.824
125.693
125.656
125.372
125.313
123.527
123.469
122.783
122.674
122.608
121.405
120.180
118.766
77.321
77.000
76.686
14.526
14.490
12.755
12.755
150
100
50
0 PPM
Me
O
N
Me
CF3

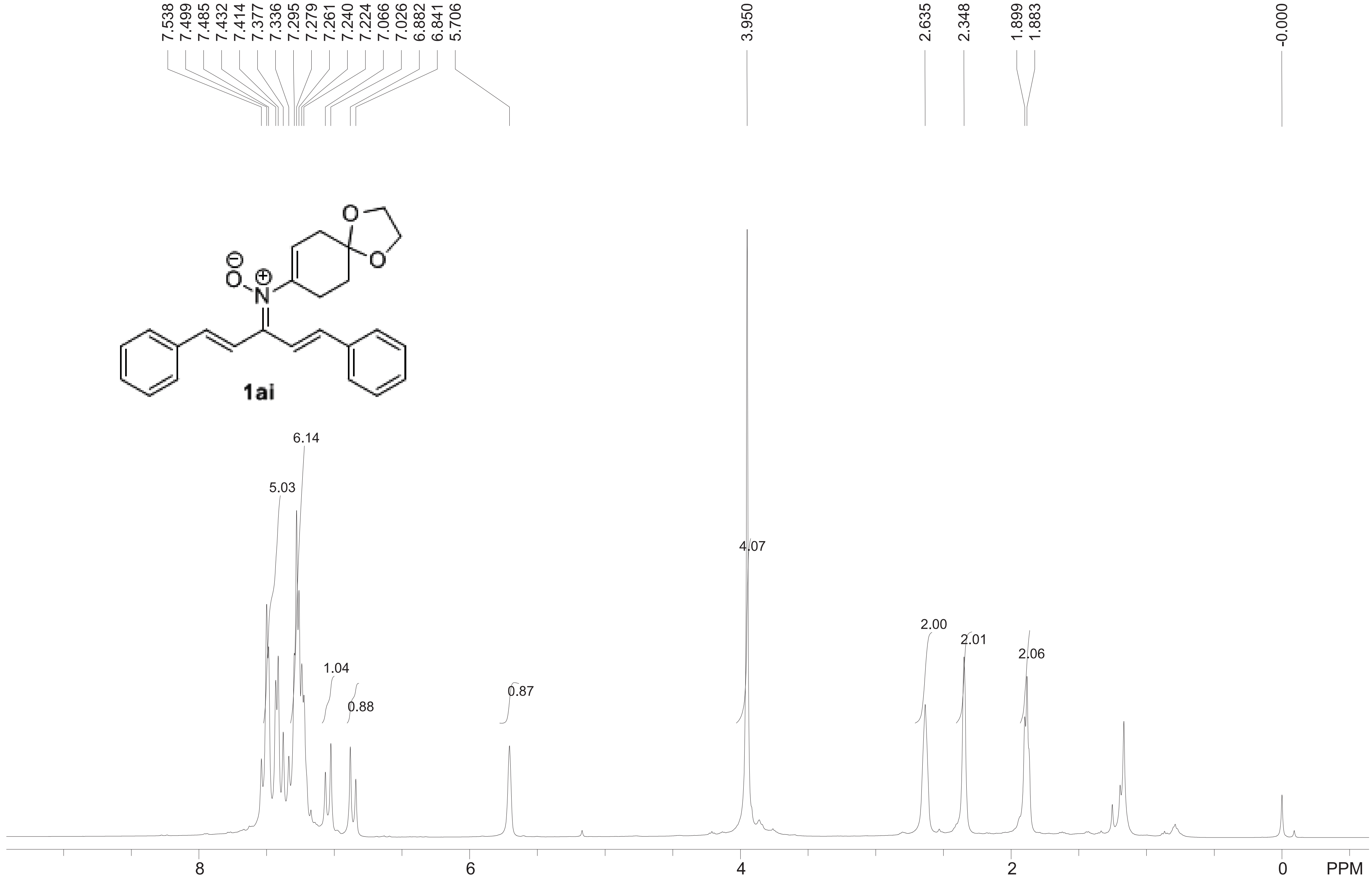
1w (*E*/*Z* = 1:1)

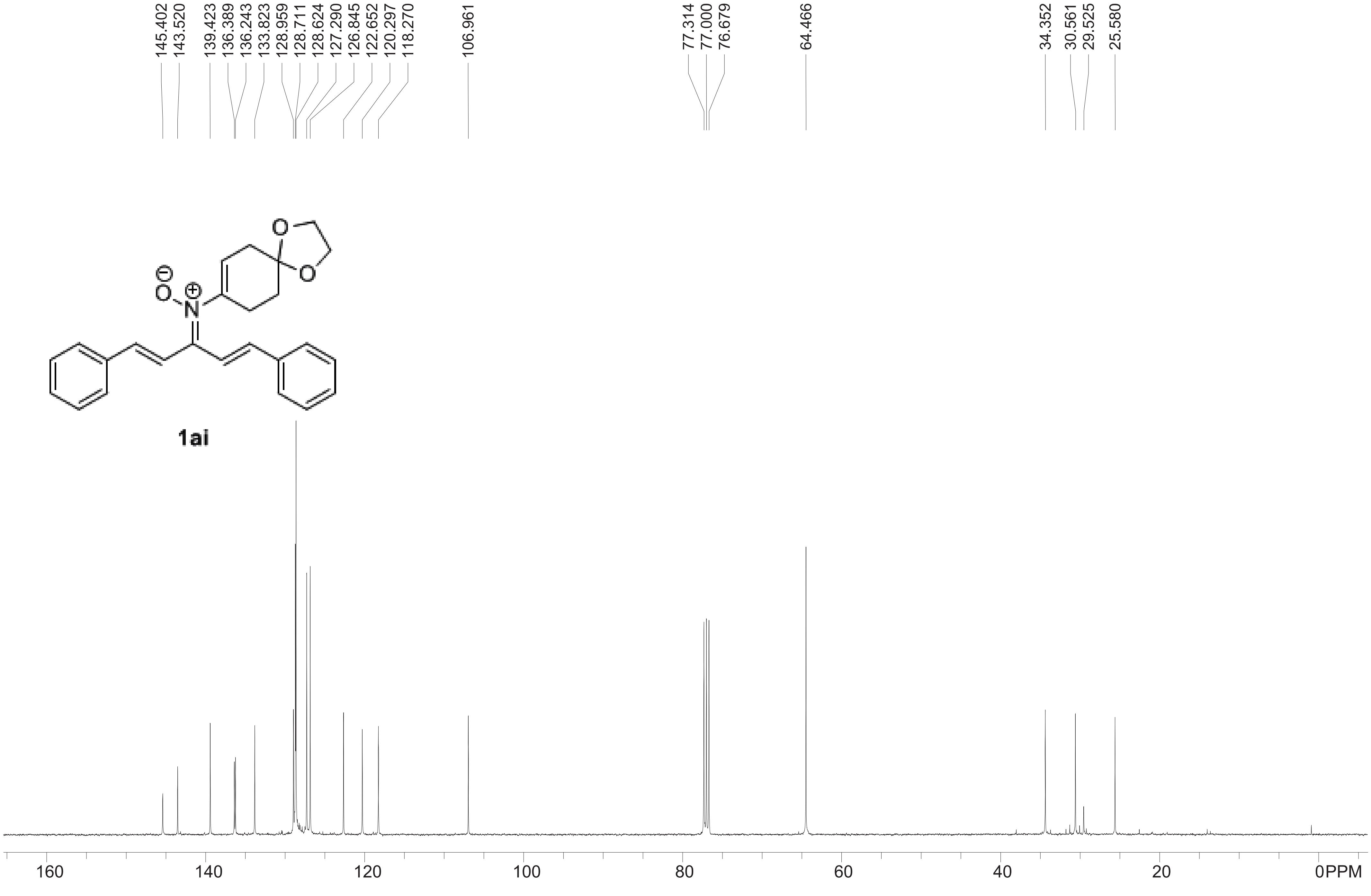


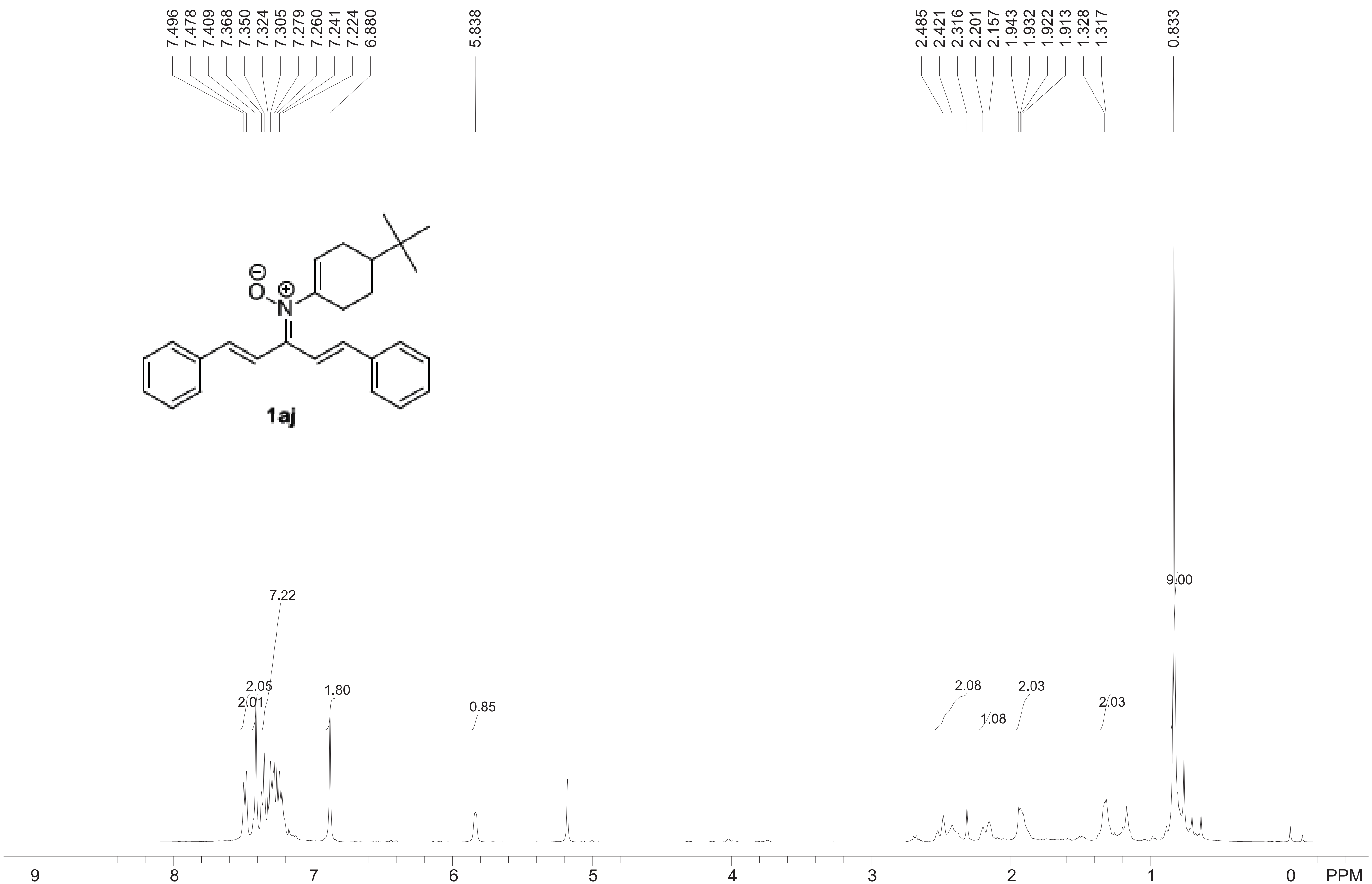
-62.569
-62.583
Me
O
N
Me
CF3
1w (E/Z = 1:1)
0
-50
-100
-150
-200
PPM

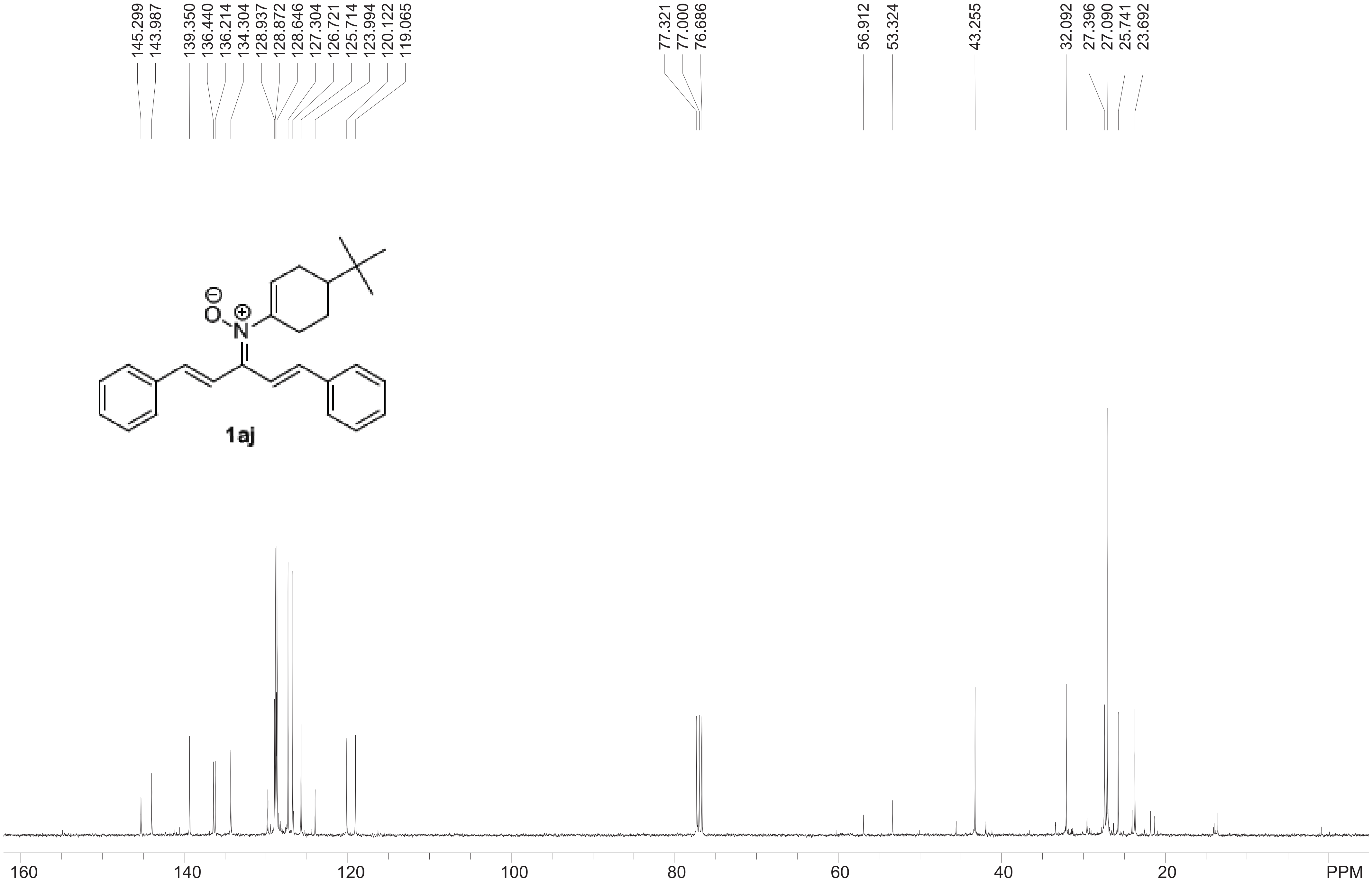

7.498
7.481
7.450
7.422
7.378
7.359
7.312
7.296
7.281
7.263
7.240
6.912
6.902
5.851
4.217
3.869
2.571
1ah
6.04
2.11
2.08
1.24
1.95
0.92
1.97
2.10
2.00
8
6
4
2
0
PPM

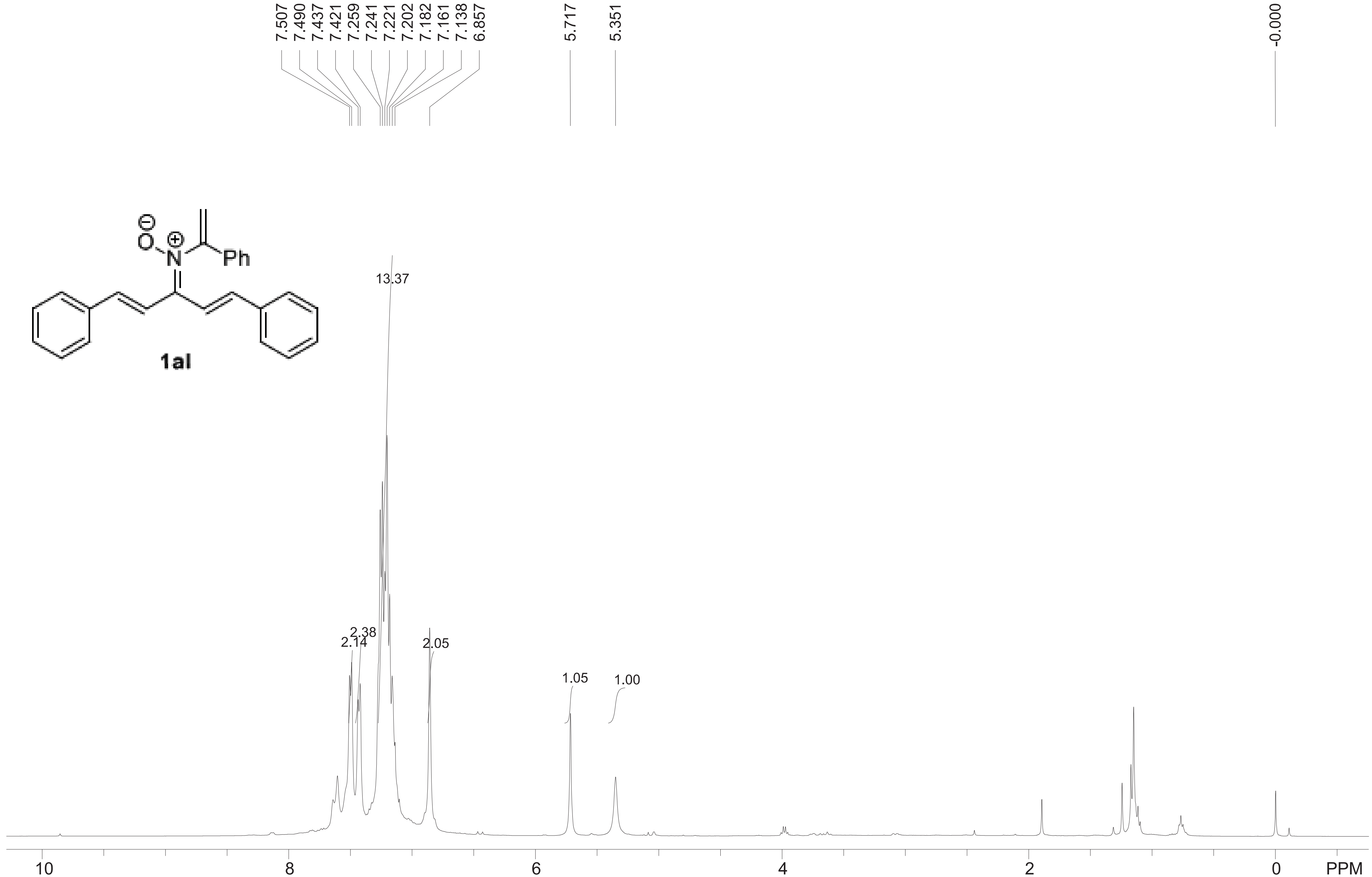

145.679
141.049
139.780
136.309
135.930
134.647
129.069
128.872
128.668
128.237
127.326
126.764
123.556
119.823
118.897
77.314
77.000
76.679
64.130
63.948
26.273
1ah
160
140
120
100
80
60
40
20
0PPM

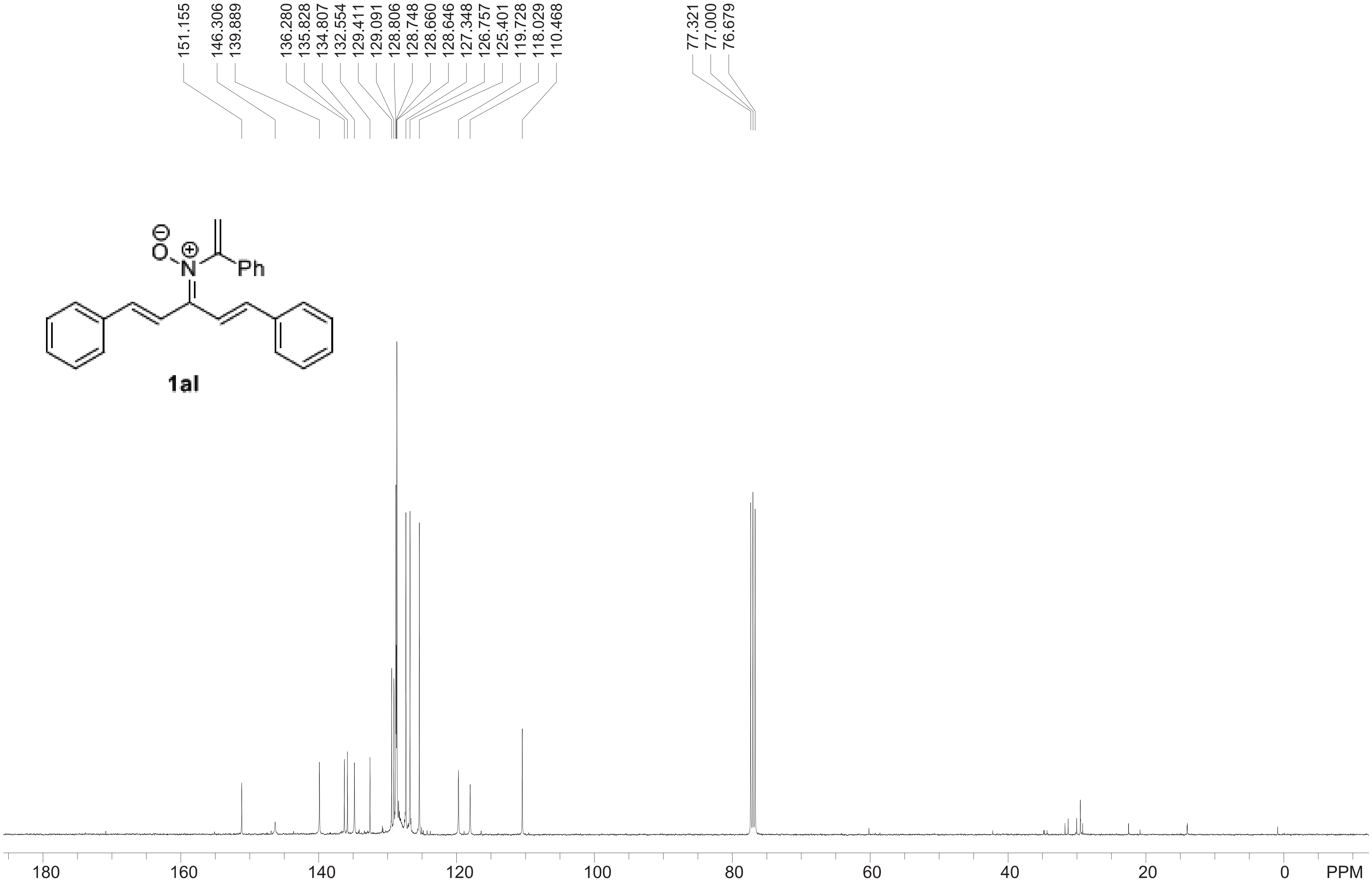

7.538
7.499
7.485
7.432
7.414
7.377
7.336
7.295
7.279
7.261
7.240
7.224
7.066
7.026
6.882
6.841
5.706
3.950
2.635
2.348
1.899
1.883
-0.000
1ai
6.14
5.03
1.04
0.88
0.87
4.07
2.00
2.01
2.06
8
6
4
2
0
PPM

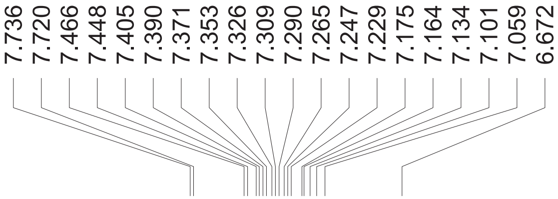

145.402
143.520
139.423
136.389
136.243
133.823
128.959
128.711
128.624
127.290
126.845
122.652
120.297
118.270
106.961
77.314
77.000
76.679
64.466
34.352
30.561
29.525
25.580
1ai
160
140
120
100
80
60
40
20
0PPM

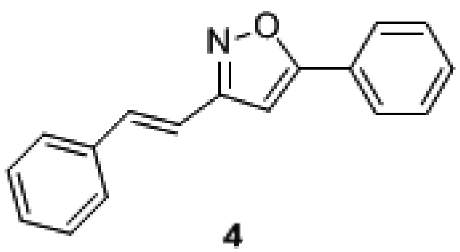

7.496
7.478
7.409
7.368
7.350
7.324
7.305
7.279
7.260
7.241
7.224
6.880
5.838
2.485
2.421
2.316
2.201
2.157
1.943
1.932
1.922
1.913
1.328
1.317
0.833
1aj
2.01
2.05
7.22
1.80
0.85
2.08
1.08
2.03
2.03
9.00
9
8
7
6
5
4
3
2
1
0
PPM


145.299
143.987
139.350
136.440
136.214
134.304
128.937
128.872
128.646
127.304
126.721
125.714
123.994
120.122
119.065
77.321
77.000
76.686
56.912
53.324
43.255
32.092
27.396
27.090
25.741
23.692
1aj
160
140
120
100
80
60
40
20
PPM

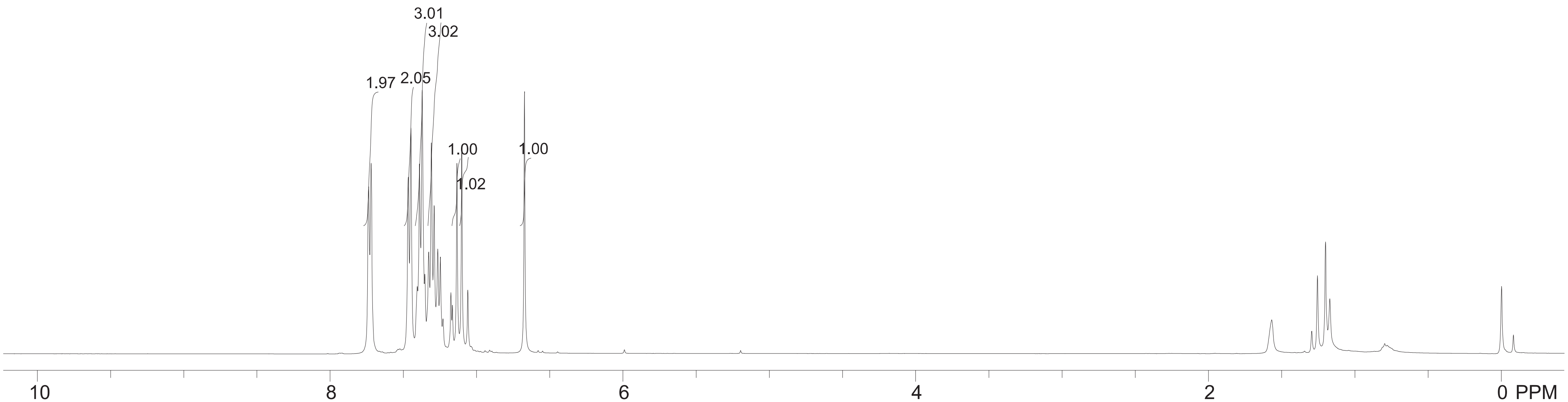

7.507
7.490
7.437
7.421
7.259
7.241
7.221
7.202
7.182
7.161
7.138
6.857
5.717
5.351
-0.000
Ph
1al
13.37
2.38
2.14
2.05
1.05
1.00
10
8
6
4
2
0
PPM

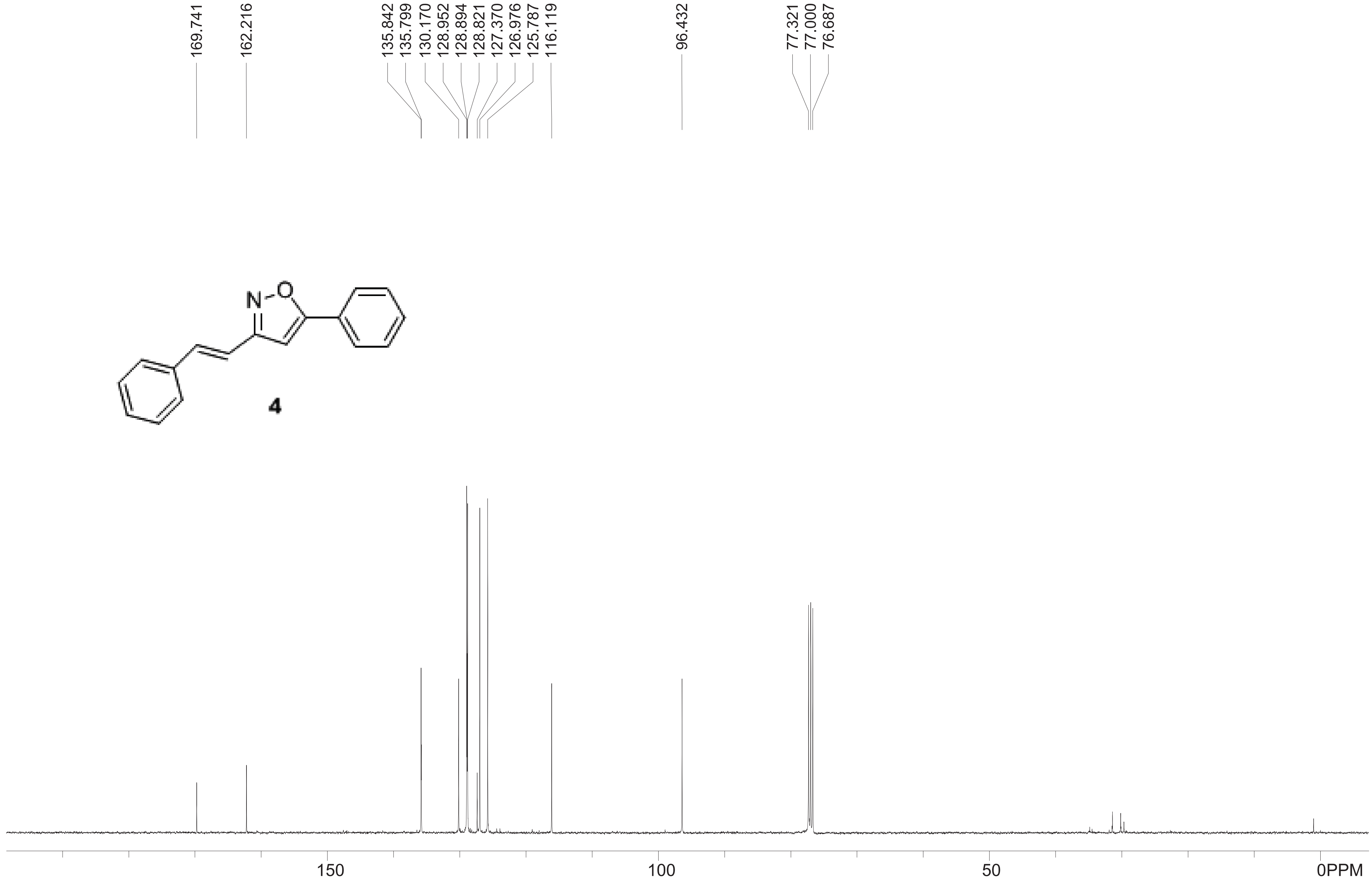

151.155
146.306
139.889
136.280
135.828
134.807
132.554
129.411
129.091
128.806
128.748
128.660
128.646
127.348
126.757
125.401
119.728
118.029
110.468
77.321
77.000
76.679
O
N
Ph
1al
180
160
140
120
100
80
60
40
20
0
PPM

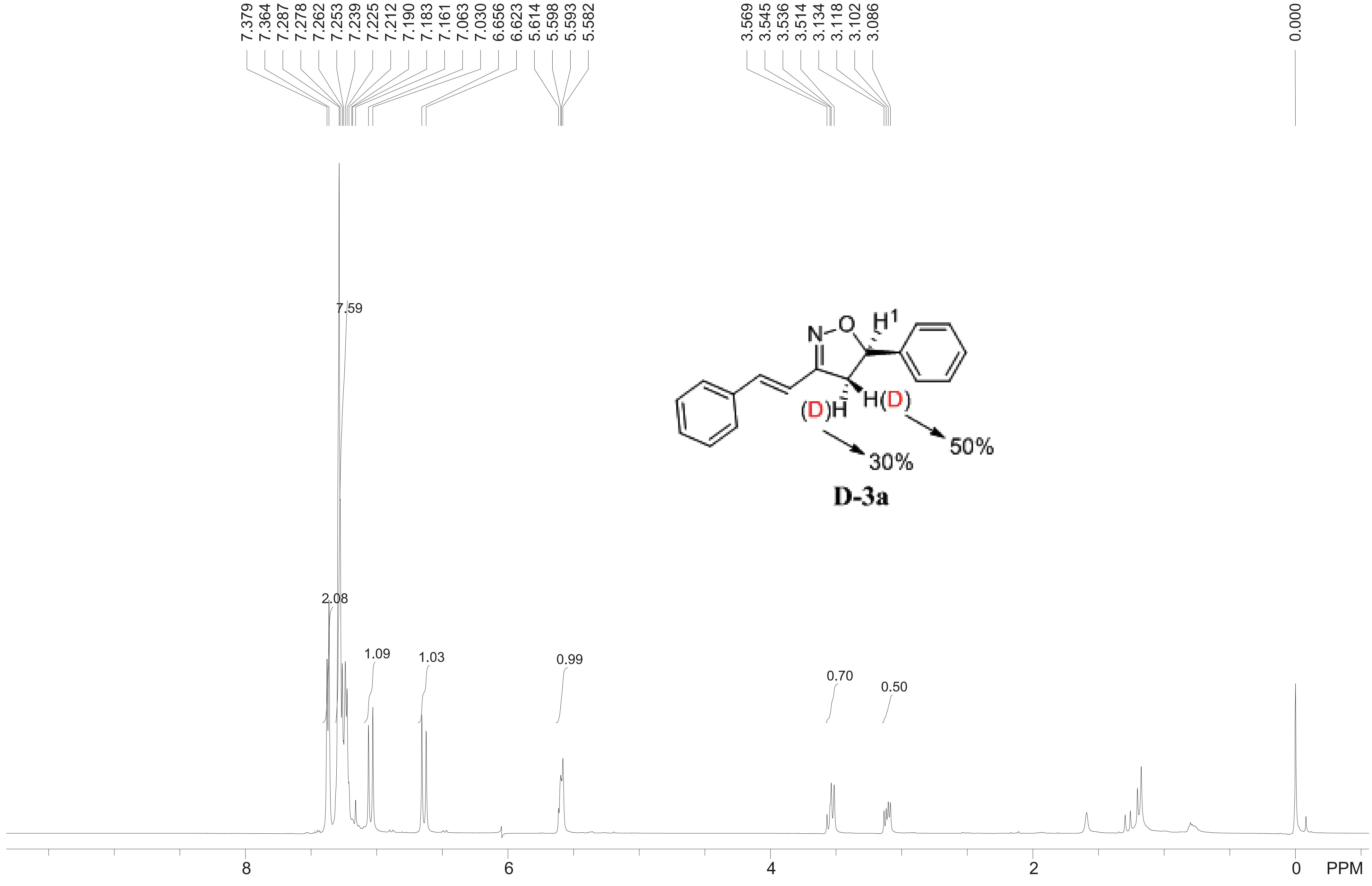

7.736
7.720
7.466
7.448
7.405
7.390
7.371
7.353
7.326
7.309
7.290
7.265
7.247
7.229
7.175
7.164
7.134
7.101
7.059
6.672

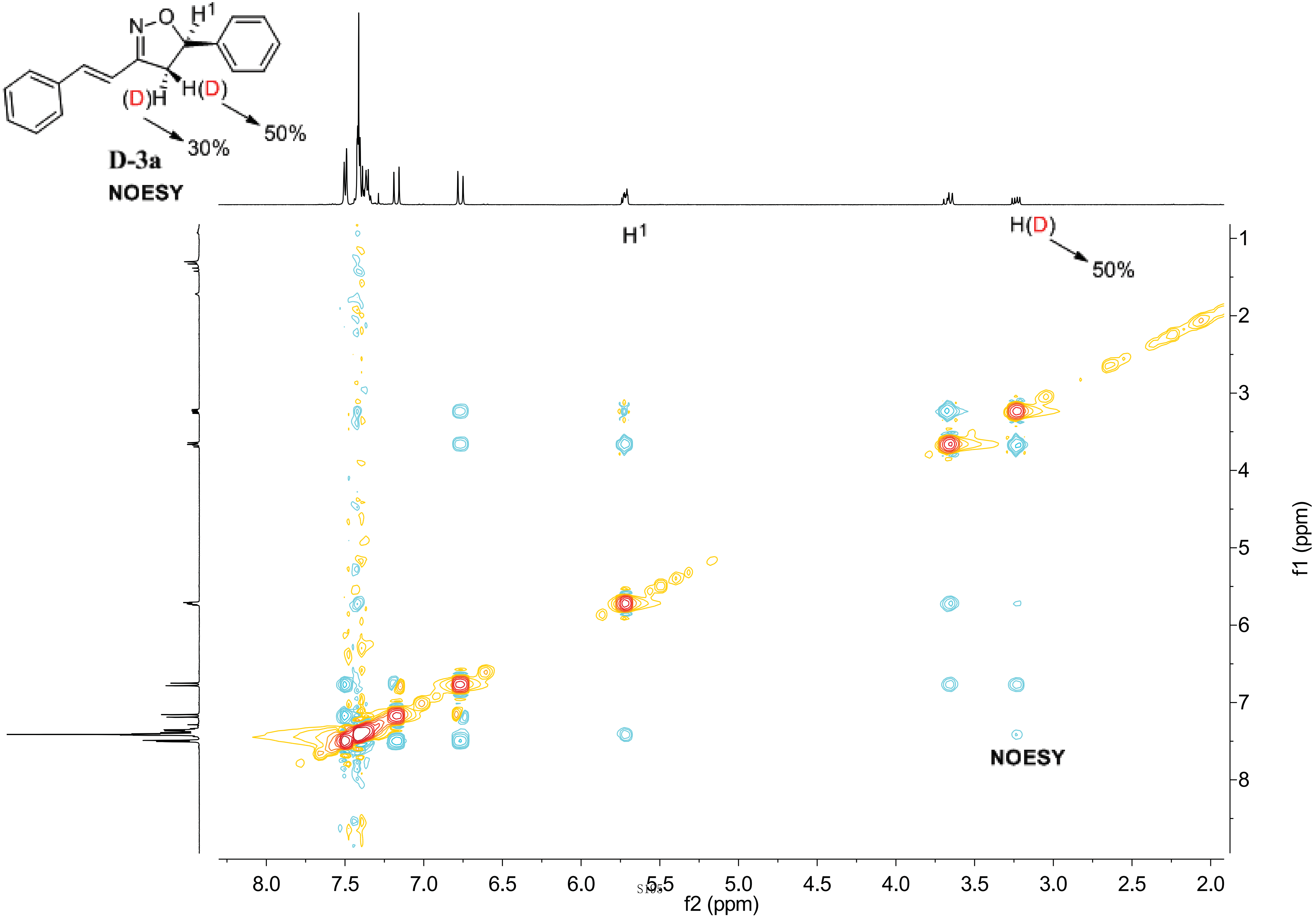


4

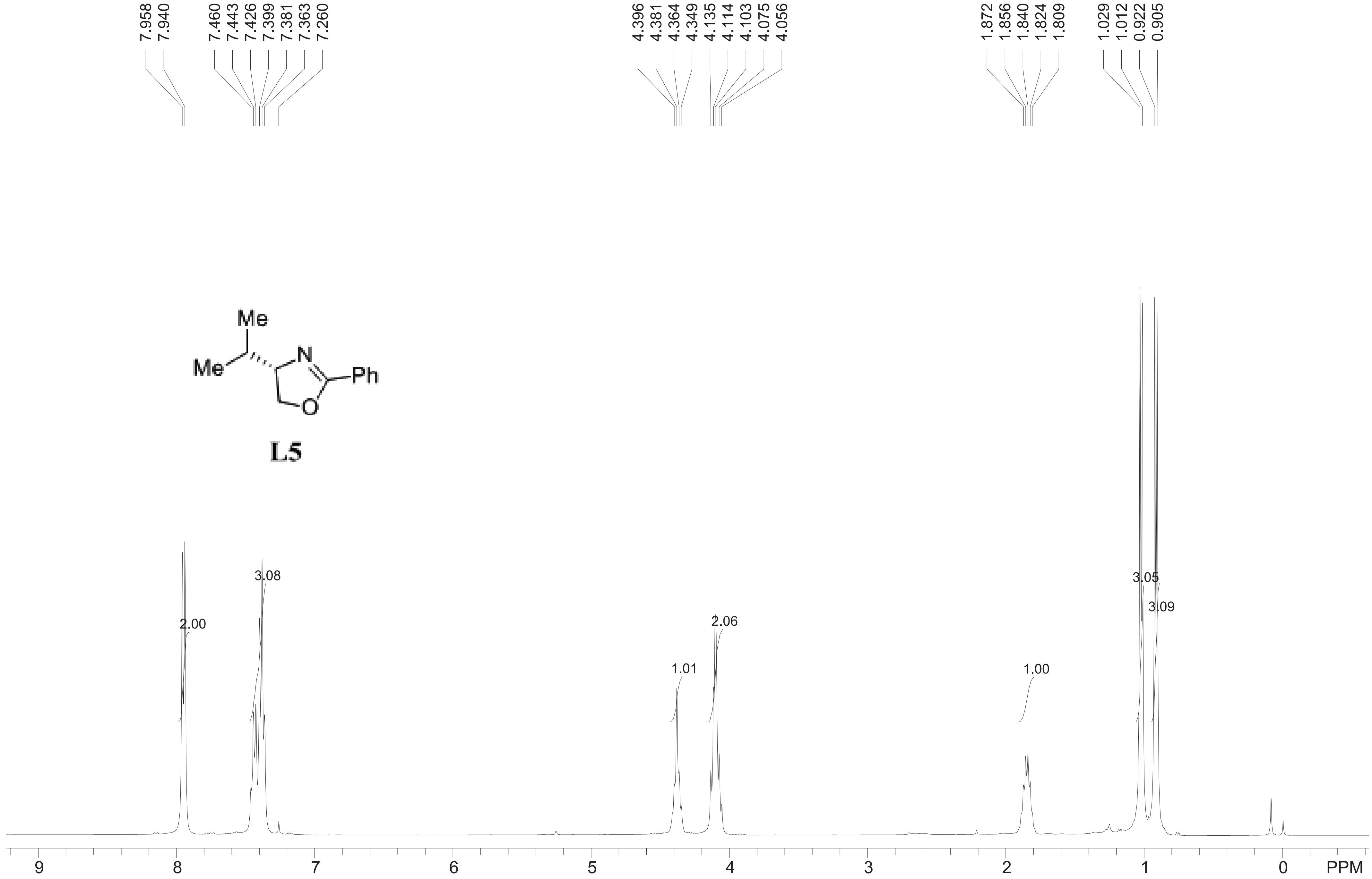

3.01
3.02
1.97
2.05
1.00
1.02
1.00
10
8
6
4
2
0 PPM

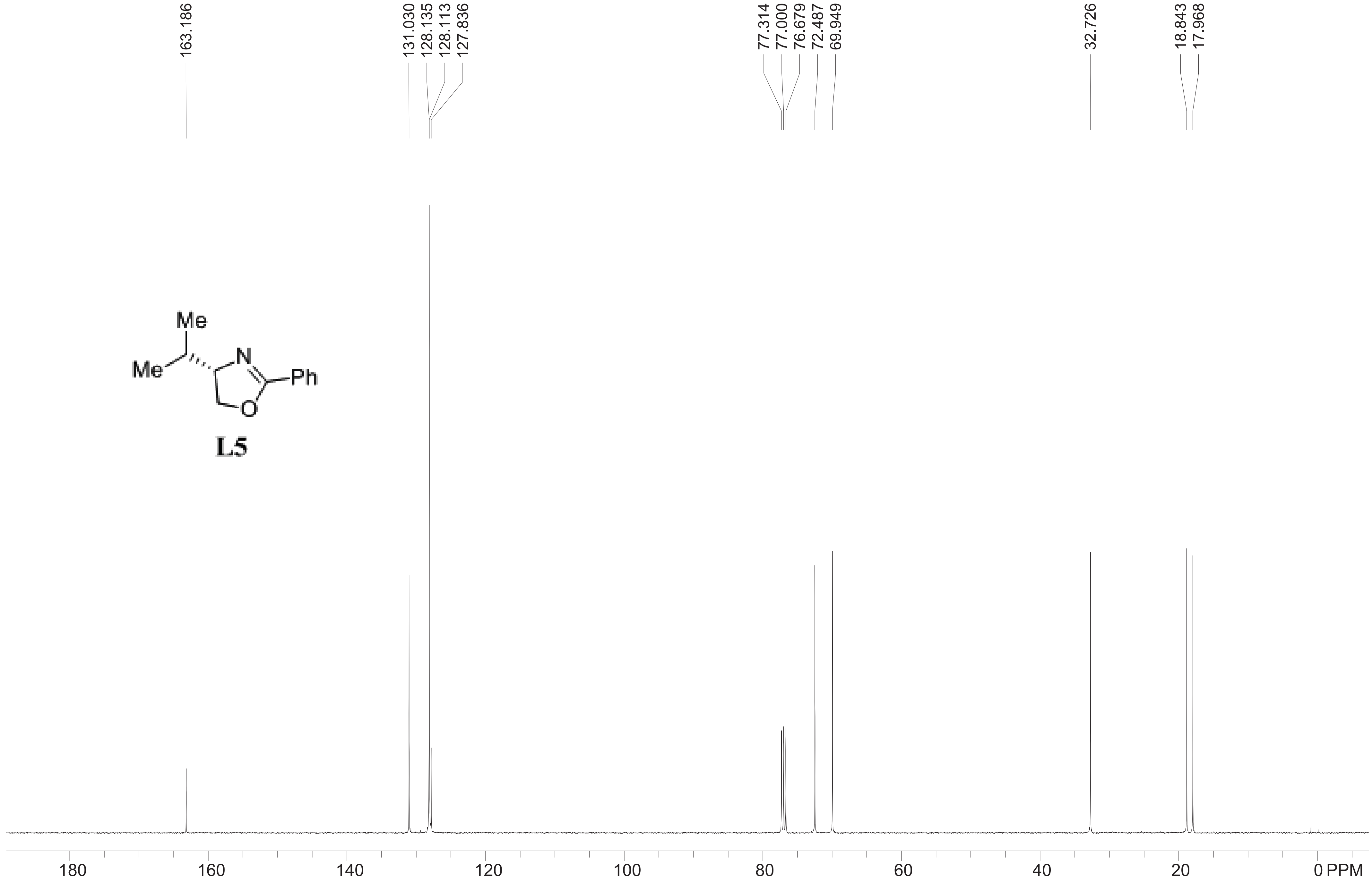

169.741
162.216
135.842
135.799
130.170
128.952
128.894
128.821
127.370
126.976
125.787
116.119
96.432
77.321
77.000
76.687
N
O
4
150
100
50
0PPM

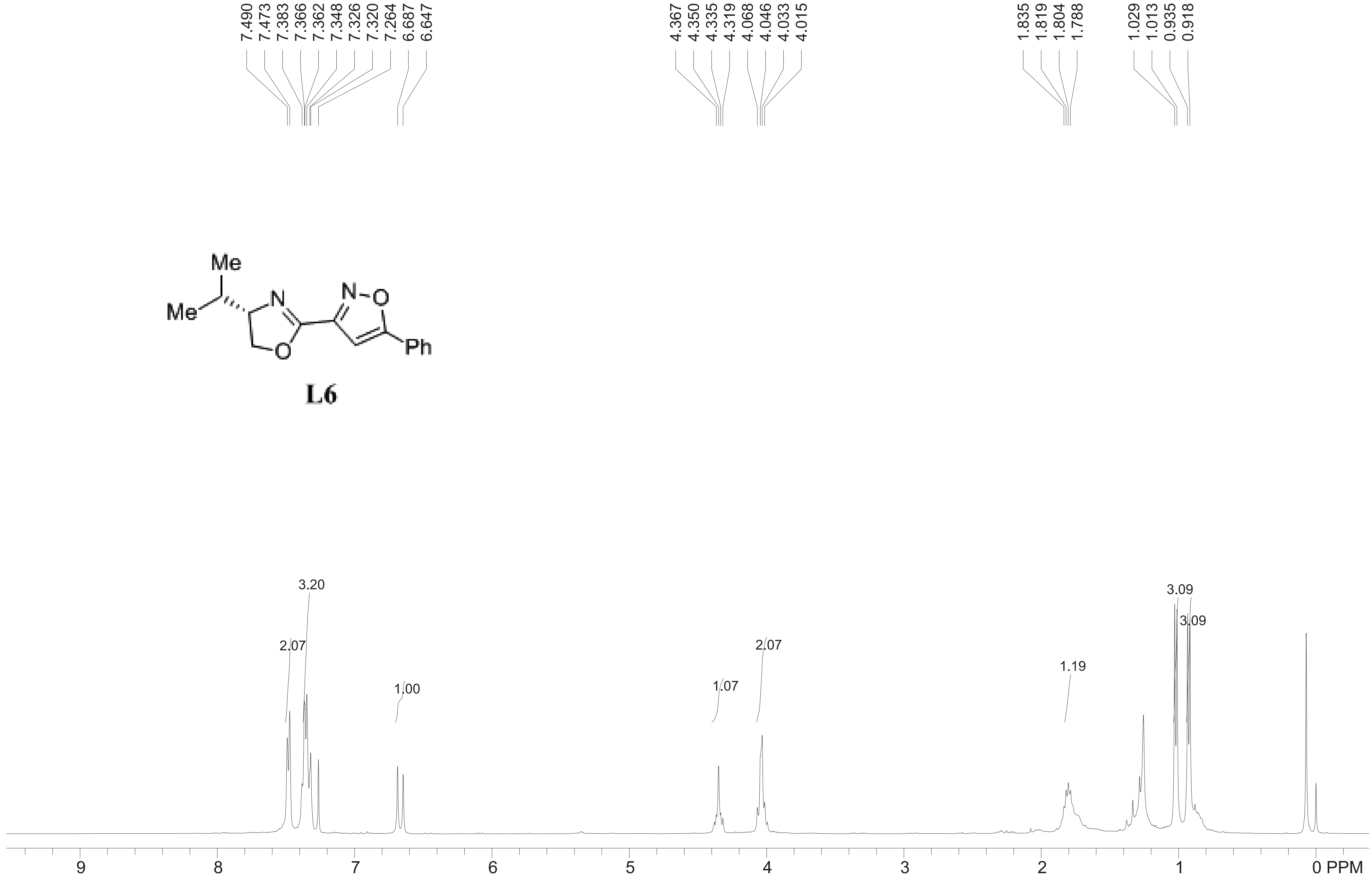

7.379 7.364 7.287 7.278 7.262 7.253 7.239 7.225 7.212 7.190 7.183 7.161 7.063 7.030 6.656 6.623 5.614 5.598 5.593 5.582
3.569 3.545 3.536 3.514 3.134 3.118 3.102 3.086
0.000
7.59
2.08
1.09
1.03
0.99
0.70
0.50
N
O
H1
(D)H
H(D)
30%
50%
D-3a
8
6
4
2
0
PPM

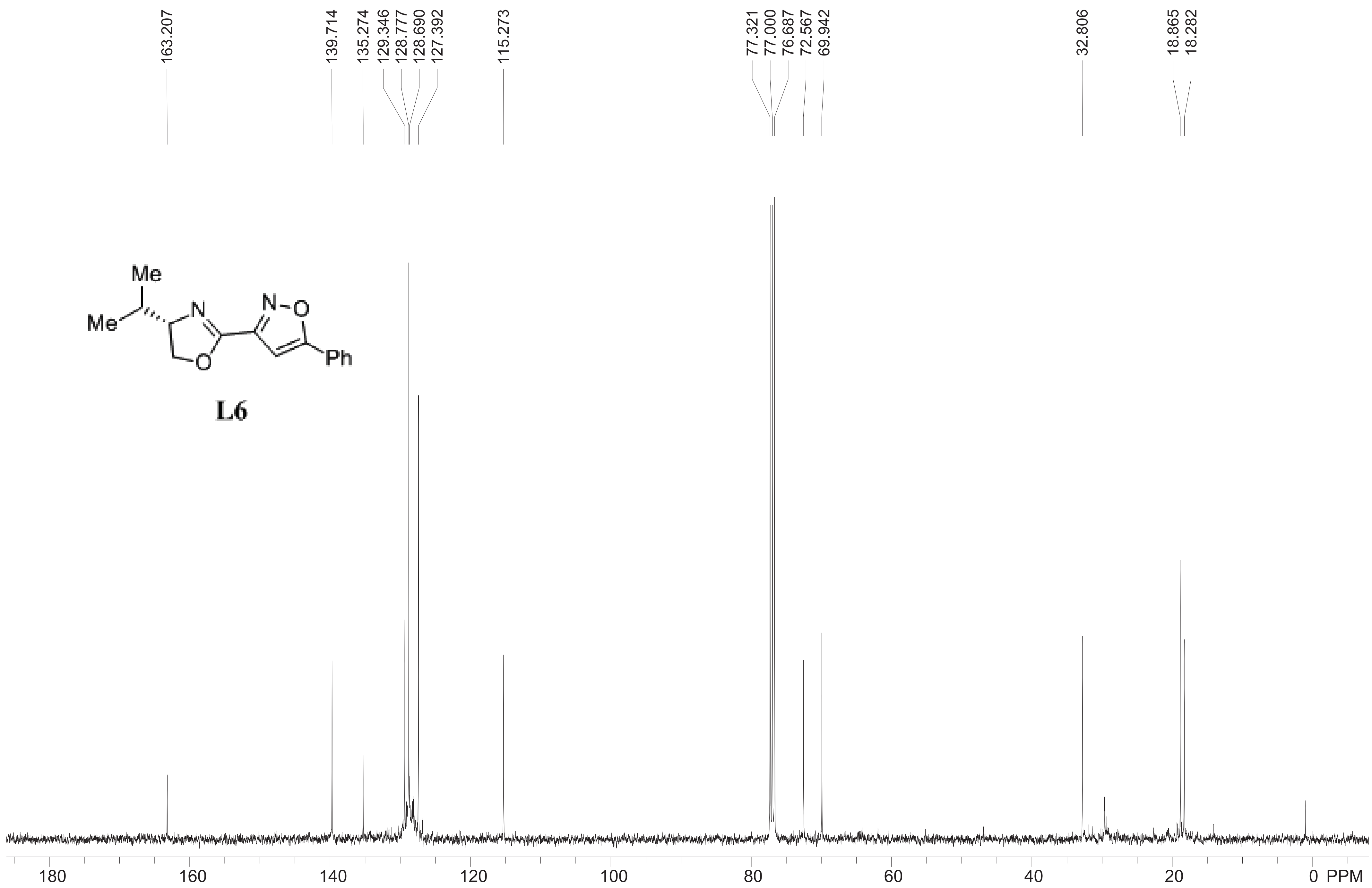


N-O
H1
(D)H
H(D)
30%
50%
D-3a
NOESY
H1
H(D)
50%
NOESY
f1 (ppm)
f2 (ppm)

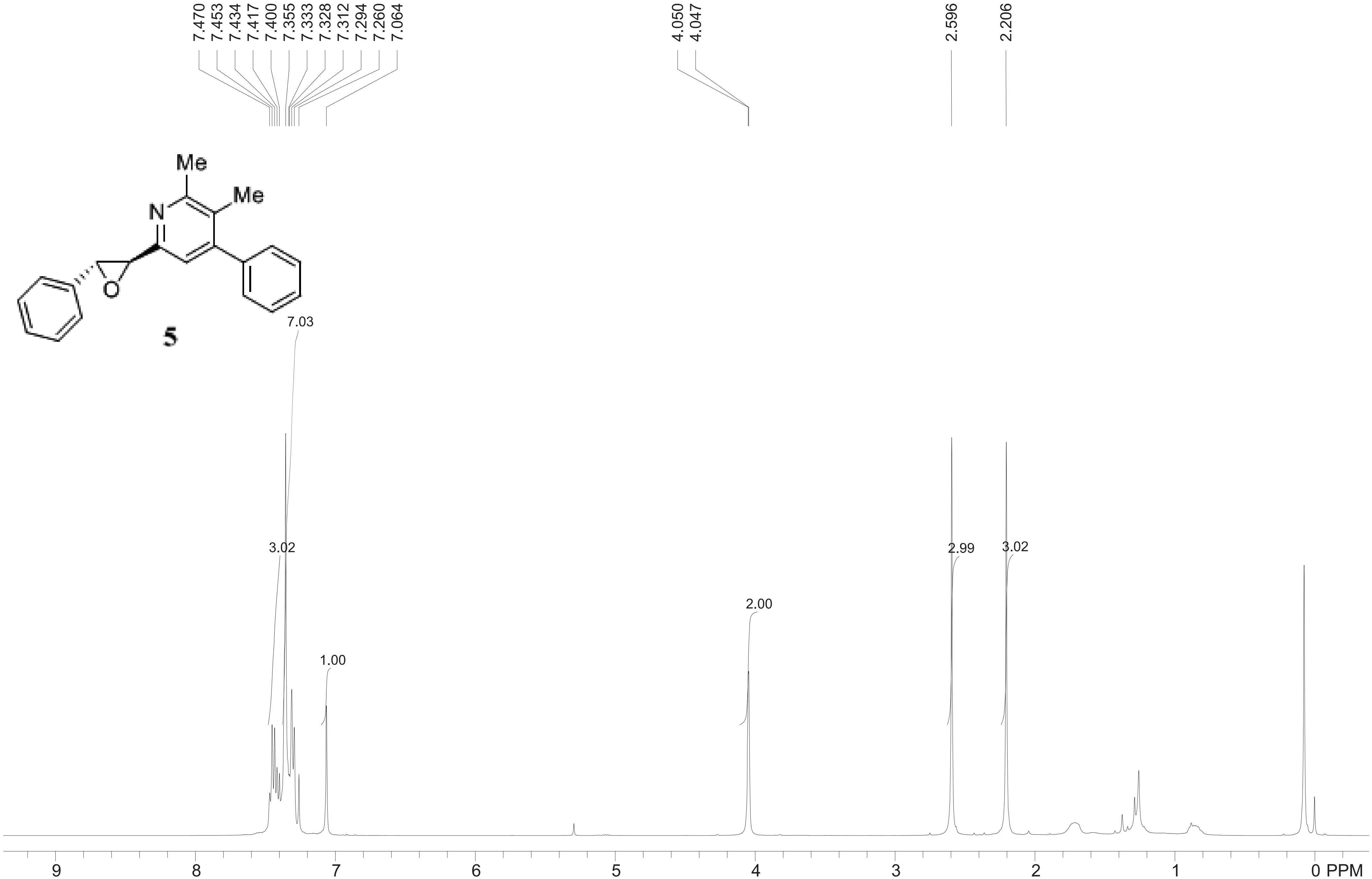

7.958
7.940
7.460
7.443
7.426
7.399
7.381
7.363
7.260
4.396
4.381
4.364
4.349
4.135
4.114
4.103
4.075
4.056
1.872
1.856
1.840
1.824
1.809
1.029
1.012
0.922
0.905
Me
Me
N
Ph
O
L5
2.00
3.08
1.01
2.06
1.00
3.05
3.09
9
8
7
6
5
4
3
2
1
0
PPM


163.186
131.030
128.135
128.113
127.836
77.314
77.000
76.679
72.487
69.949
32.726
18.843
17.968
Me
Me
N
Ph
O
L5
180
160
140
120
100
80
60
40
20
0 PPM

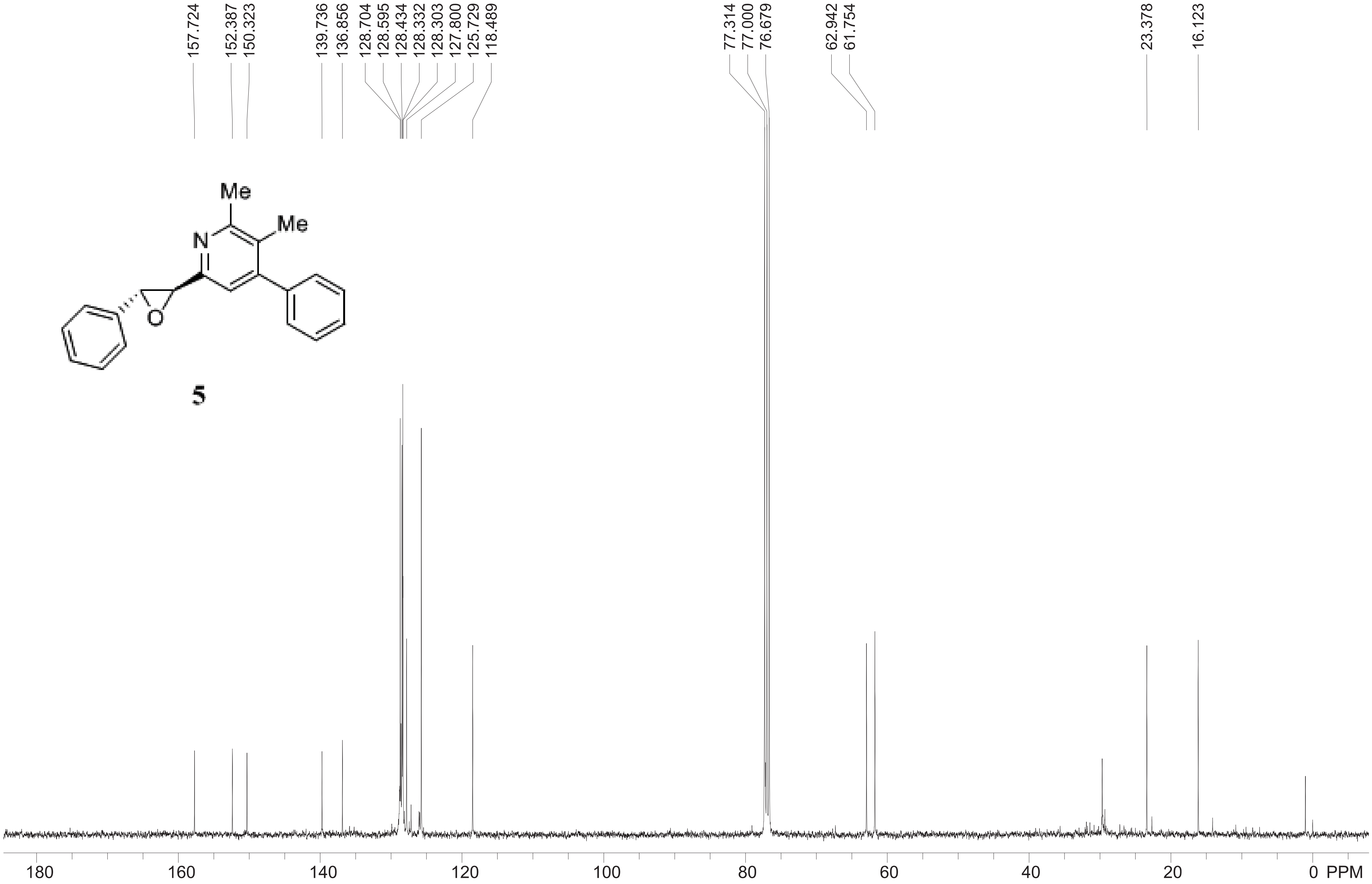

Me
Me
N
N-O
O
Ph
L6
7.490
7.473
7.383
7.366
7.362
7.348
7.326
7.320
7.264
6.687
6.647
4.367
4.350
4.335
4.319
4.068
4.046
4.033
4.015
1.835
1.819
1.804
1.788
1.029
1.013
0.935
0.918
2.07
3.20
1.00
1.07
2.07
1.19
3.09
3.09
9
8
7
6
5
4
3
2
1
0 PPM

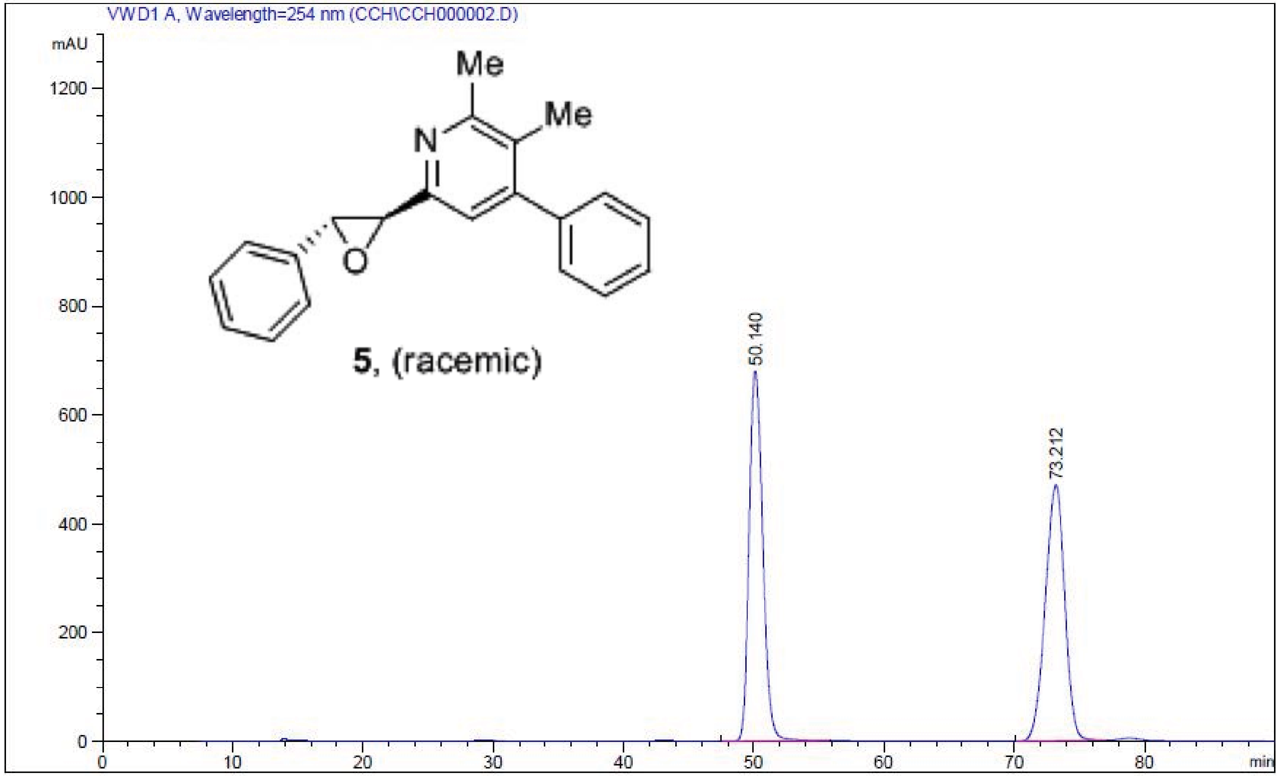

163.207
139.714
135.274
129.346
128.777
128.690
127.392
115.273
77.321
77.000
76.687
72.567
69.942
32.806
18.865
18.282
Me
Me
N
N
O
O
Ph
180
160
140
120
100
80
60
40
20
0 PPM

L6

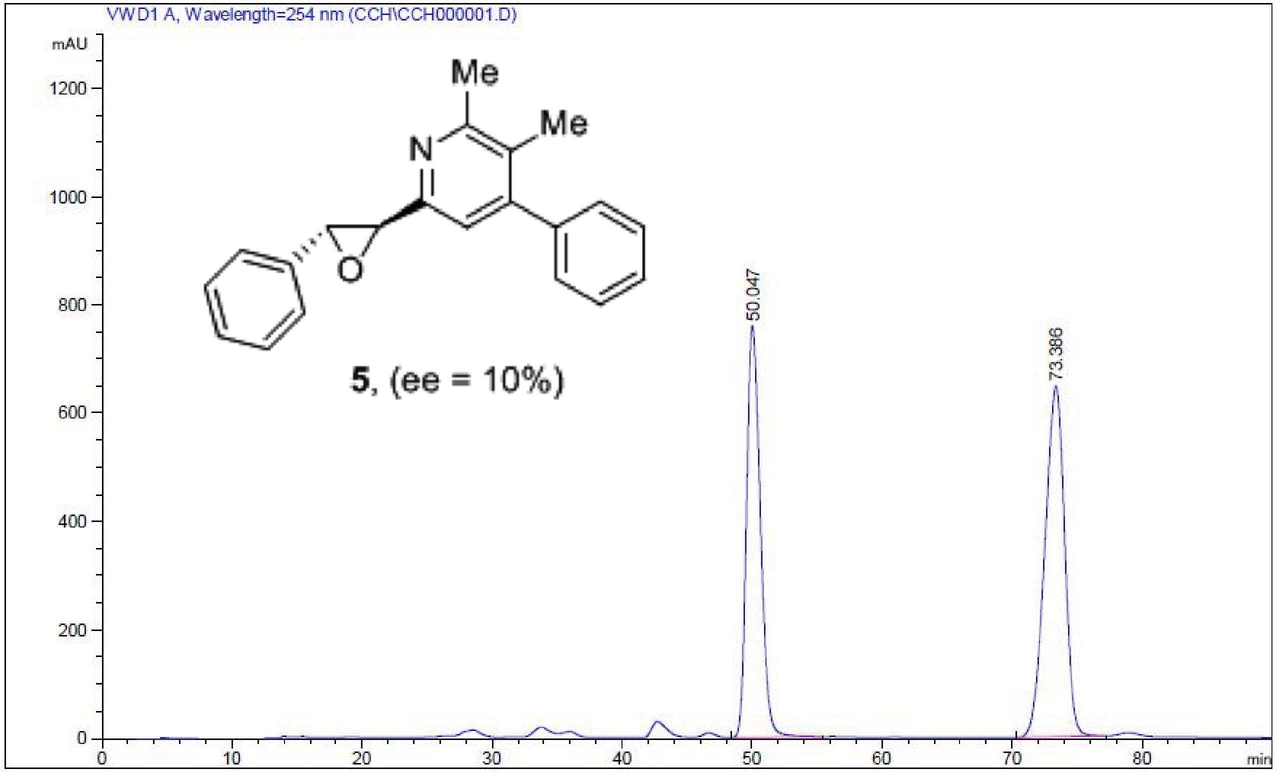


Me
Me
N
O
5
7.470
7.453
7.434
7.417
7.400
7.355
7.333
7.328
7.312
7.294
7.260
7.064
4.050
4.047
2.596
2.206
7.03
3.02
1.00
2.00
2.99
3.02
9
8
7
6
5
4
3
2
1
0 PPM

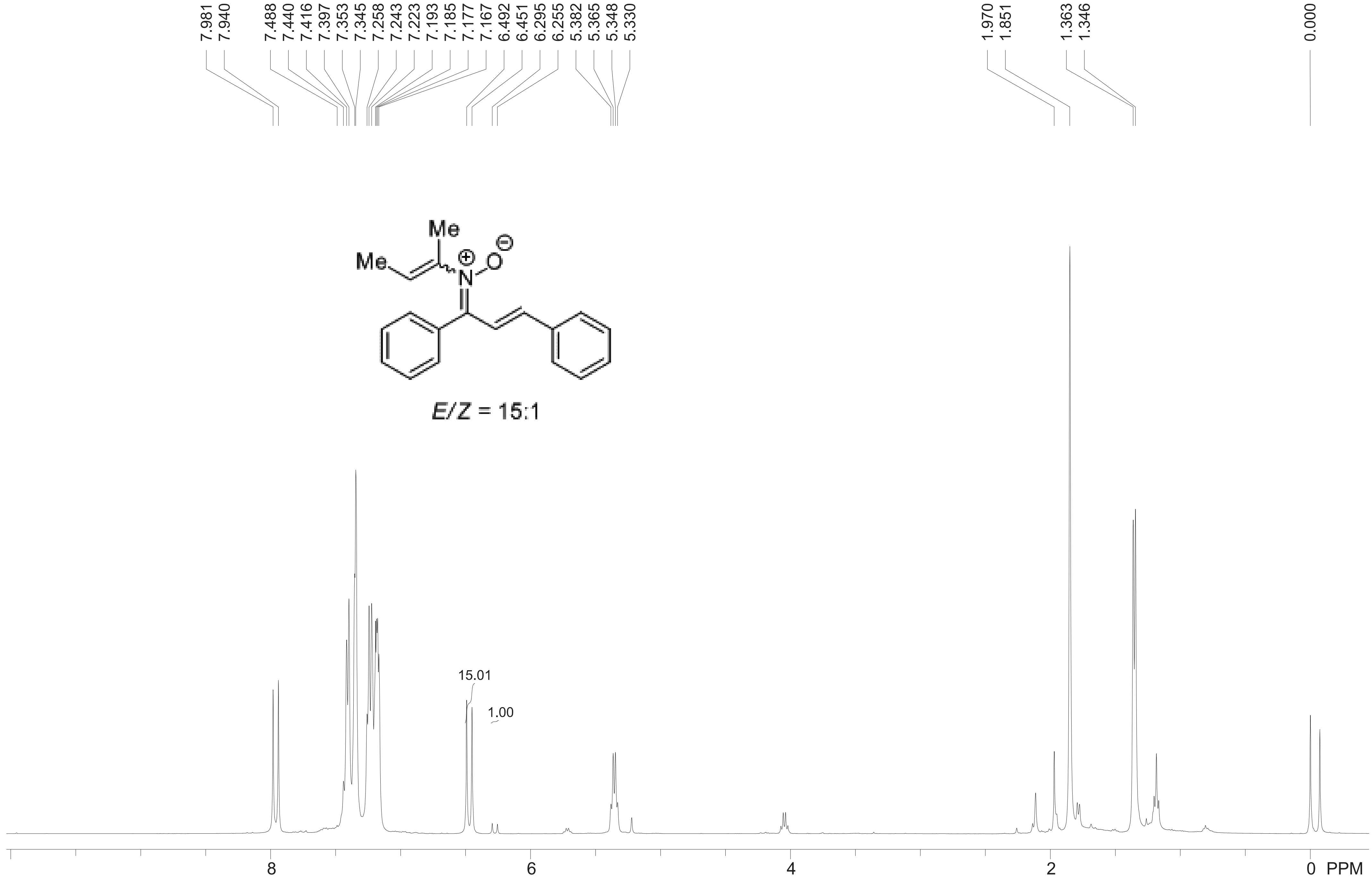

157.724
152.387
150.323
139.736
136.856
128.704
128.595
128.434
128.332
128.303
127.800
125.729
118.489
77.314
77.000
76.679
62.942
61.754
23.378
16.123
Me
Me
N
O
5
180
160
140
120
100
80
60
40
20
0 PPM

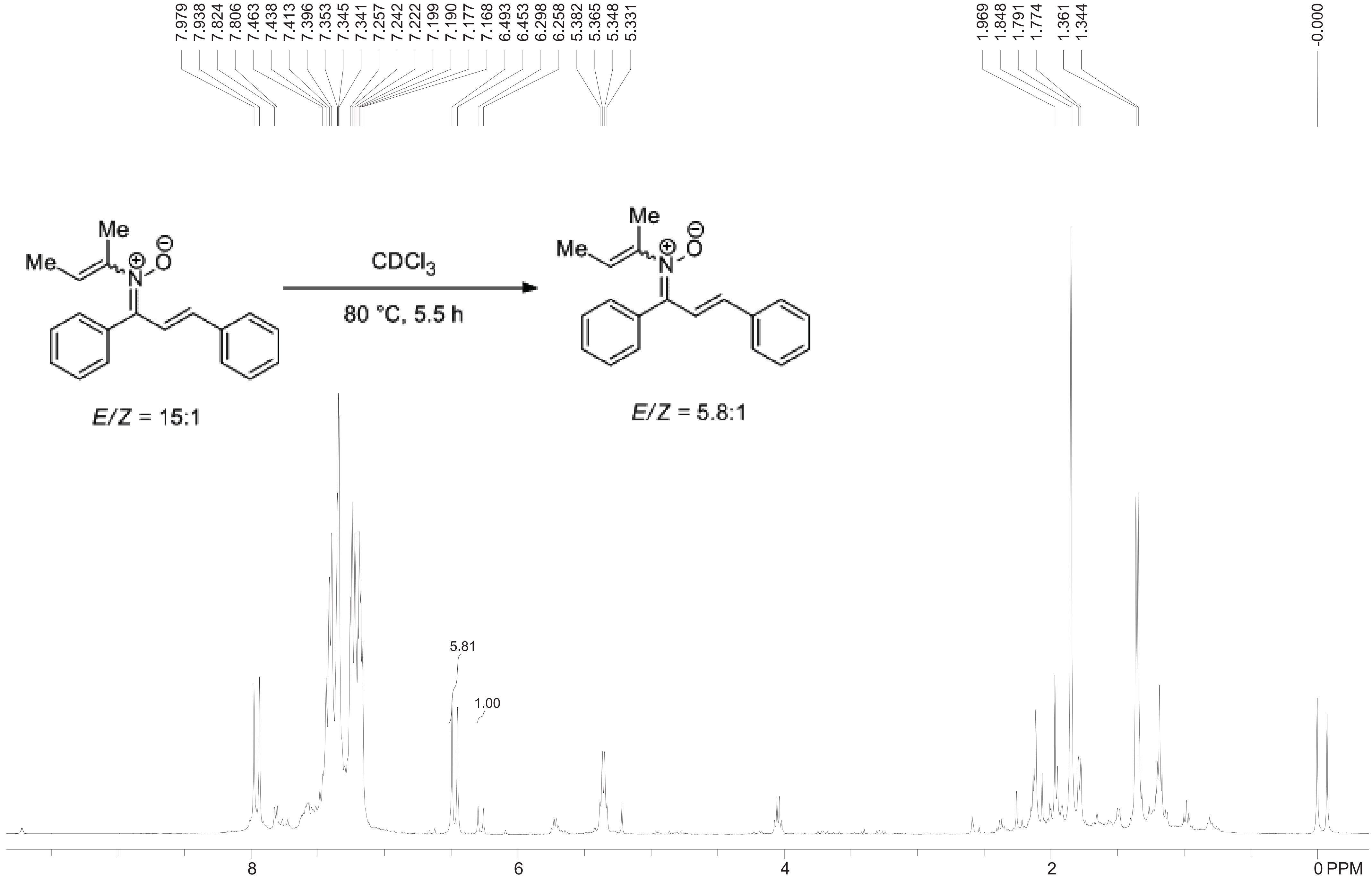


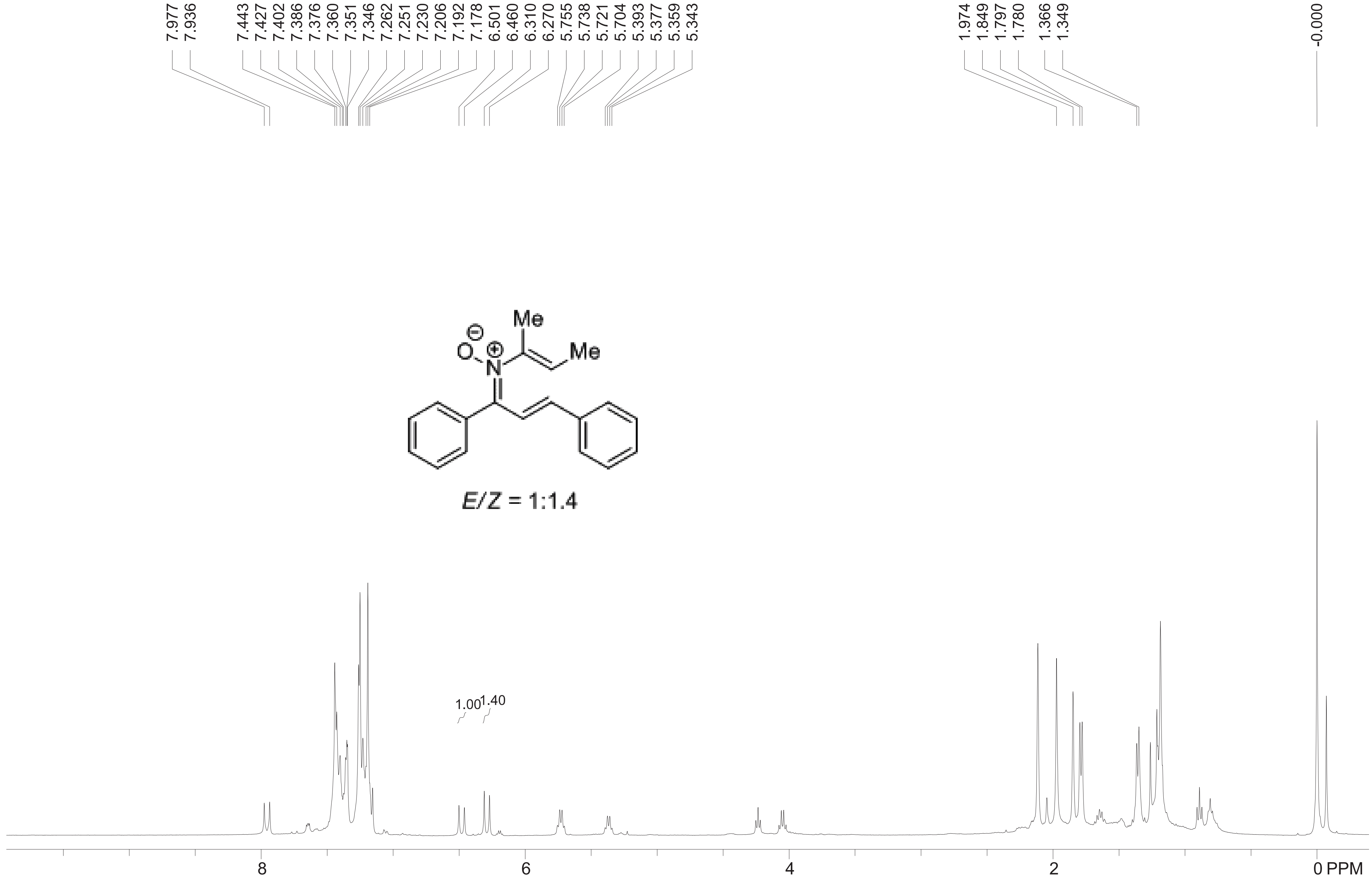
Peak	ReTime [min]	Type	Width [min]	Area [MAU*s]	Height [MAU]	Area %
1	50.140	BB	1.1286	4.92342e4	681.17688	50.1966
2	73.212	BB	1.6100	4.88486e4	470.02066	49.8034



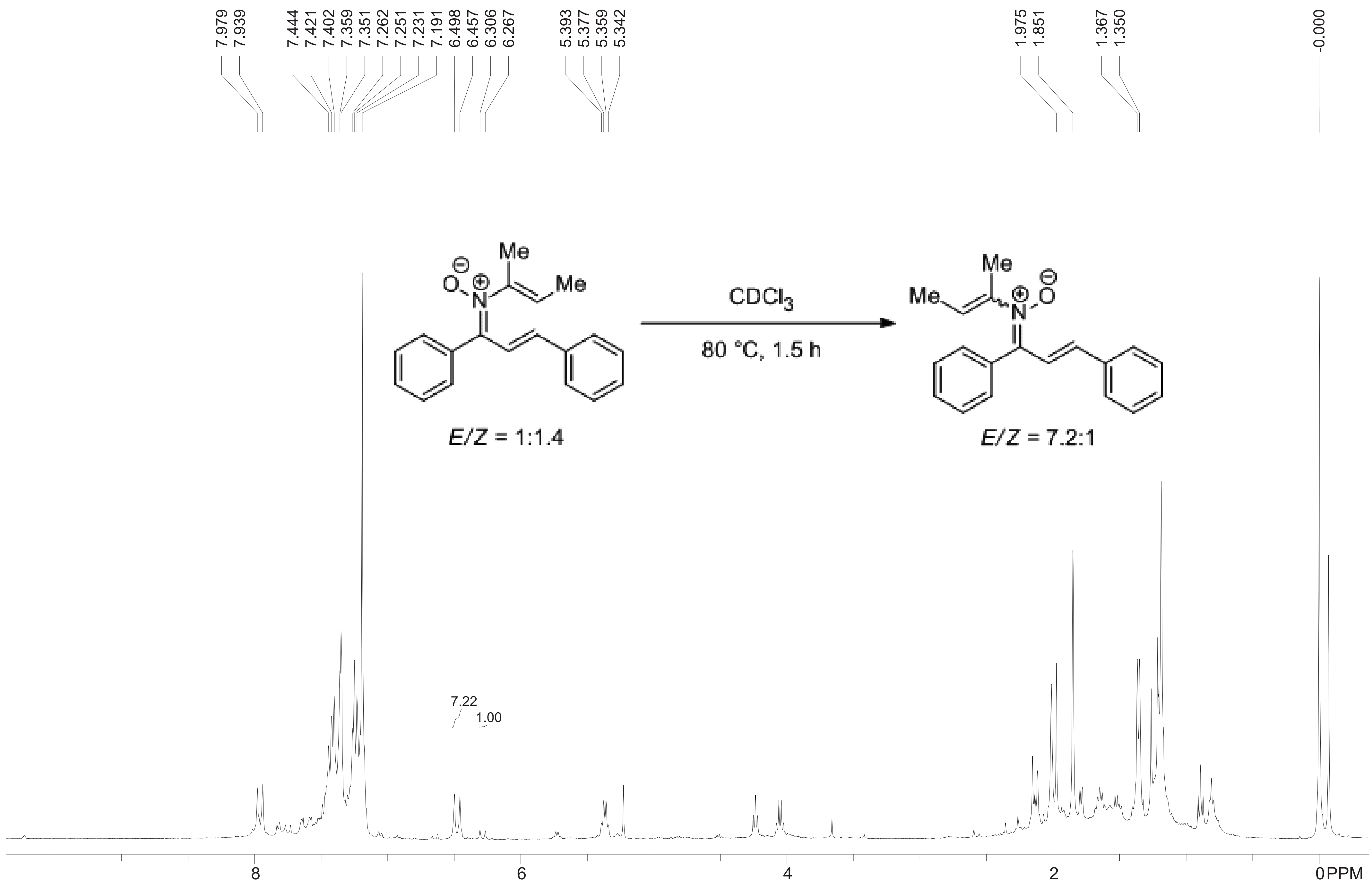
Peak	ReTime [min]	Type	Width [min]	Area [MAU*s]	Height [MAU]	Area %
1	50.047	BB	1.1539	5.60500e4	759.30621	45.0492
2	73.386	BB	1.6182	6.83695e4	647.99658	54.9508


7.981 7.940 7.488 7.440 7.416 7.397 7.353 7.345 7.258 7.243 7.223 7.193 7.185 7.177 7.167 6.492 6.451 6.295 6.255 5.382 5.365 5.348 5.330
1.970 1.851 1.363 1.346
0.000
Me
Me
E/Z = 15:1
15.01
1.00
8
6
4
2
0 PPM


7.979
7.938
7.824
7.806
7.463
7.438
7.413
7.396
7.353
7.345
7.341
7.257
7.242
7.222
7.199
7.190
7.177
7.168
6.493
6.453
6.298
6.258
5.382
5.365
5.348
5.331
1.969
1.848
1.791
1.774
1.361
1.344
-0.000
Me
Me
CDCl3
80 °C, 5.5 h
E/Z = 15:1
E/Z = 5.8:1
5.81
1.00
8
6
4
2
0 PPM


7.977
7.936
7.443
7.427
7.402
7.386
7.376
7.360
7.351
7.346
7.262
7.251
7.230
7.206
7.192
7.178
6.501
6.460
6.310
6.270
5.755
5.738
5.721
5.704
5.393
5.377
5.359
5.343
1.974
1.849
1.797
1.780
1.366
1.349
-0.000
1.00
1.40
8
6
4
2
0 PPM
Me
Me
O
N

E/Z = 1:1.4


7.979
7.939
7.444
7.421
7.402
7.359
7.351
7.262
7.251
7.231
7.191
6.498
6.457
6.306
6.267
5.393
5.377
5.359
5.342
1.975
1.851
1.367
1.350
-0.000
CDCl3
80 °C, 1.5 h
E/Z = 1:1.4
E/Z = 7.2:1
7.22
1.00
8
6
4
2
0 PPM