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Supporting Information

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Fluorescent Naphthyl- and Anthrylazoles Through Catalytic Coupling of Phenylazoles with Internal Alkynes via Multiple C-H Bond Cleavages

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Abbreviations

Cp* = η^5-pentamethylcyclopentadienyl
cod = 1,5-cyclooctadiene

Experimental Section

General. ^{1}H and ^{13}C NMR spectra were recorded at 400 and 100 MHz for $CDCl_3$ solutions. MS data were obtained by EI. GC analysis was carried out using a silicon OV-17 column (i. d. 2.6 mm x 1.5 m). GC-MS analysis was carried out using a CBP-1 capillary column (i. d. 0.25 mm x 25 m). The structures of all products listed below were unambiguously determined by ^{1}H and ^{13}C NMR with the aid of NOE, COSY, HMQC, and HMBC experiments.

Diarylacetylenes **2b**-**d**[13], $C_5H_3Ph_3$ (1,2,4-triphenyl-1,3-cyclopentadiene)[14], and 1-Methyl-2-phenylbenzimidazole (**4a**)[15] were prepared according to published procedures. Other reagents were commercially available.

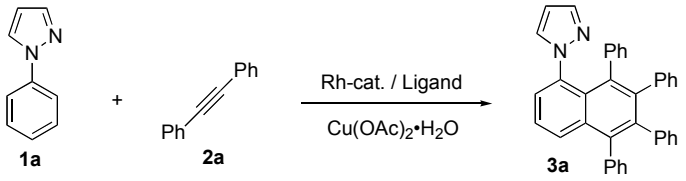
The fluorescence analysis of some products was carried out with the samples recrystallized from hexane-toluene or hexane-dichloromethane and then crashed.[16] The absolute fluorescence quantum efficiency of the crashed crystal of **8**, encapsulated in a quartz cell (30 x 30 x 0.3 mm) under deoxygenated conditions, was measured by using an integrating sphere unit (the excitation wavelength: 350 nm).

The following experimental procedures may be regarded as typical in methodology and scale.

Rh-Catalyzed Reaction of 1-Phenylpyrazole (1a) with Diphenylacetylene (2a) (entry 1 in Table 1): A mixture of 1-phenylpyrazole (**1a**) (1 mmol, 144 mg), diphenylacetylene (**2a**) (1 mmol, 178 mg), $[Cp^*RhCl_2]_2$ (0.01 mmol, 6.2 mg), 1,2,3,4-tetraphenyl-1,3-cyclopentadiene (0.04 mmol, 14.8 mg), $Cu(OAc)_2$-H_2O (1 mmol, 200 mg), and dibenzyl (ca. 50 mg) as internal standard was stirred in DMF (5 mL) under nitrogen at 80 ºC. After 6 h, the reaction mixture was cooled to room temperature, CH_2Cl_2 (100 mL), water (100 mL) and ethylenediamine (2 mL) were added. Then the organic layer was washed by water (100 mL, three times) and dried over $CaCl_2$. GC and GC-MS analyses confirmed the formation of 1-(1,2,3,4-tetraphenylnaphthalen-5-yl)pyrazole (**3a**) (0.47 mmol, 93%). The product **3a** (232 mg, 93%) was also isolated by column chromatography on silica gel using hexane-ethyl acetate (95:5, v/v) as eluant.

Selected results of the reaction of 1-phenylpyrazole (**1a**) with diphenylacetylene (**2a**) for the optimization of reaction conditions are summarized in Table S1.

Table S1. Reaction of 1-phenylpyrazole (**1a**) with Diphenylacetylene (**2a**)[a]



1a + **2a** → **3a**

Entry	Rh-cat.	Ligand	Temp. [°C]	% Yield of **3a**[b]
1[c]	$[Cp^*RhCl_2]_2$	-	80	63
2	$[Cp^*RhCl_2]_2$	-	80	87
3	$[Cp^*RhCl_2]_2$	-	60	41
4	$[Cp^*RhCl_2]_2$	$C_5H_2Ph_4$	80	93 (93)
5	$[Cp^*RhCl_2]_2$	$C_5H_3Ph_3$	80	73
6	$[Cp^*RhCl_2]_2$	C_5HPh_5	80	78
7	$[RhCl(cod)]_2$	$C_5H_2Ph_4$	80	37
8	$[Cp^*IrCl_2]_2$	$C_5H_2Ph_4$	80	0

[a] Reaction conditions: **1a** (1 mmol), **2a** (1 mmol), Rh-cat. (0.01 mmol), ligand (0.04 mmol), $Cu(OAc)_2•H_2O$ (1 mmol), DMF (5 mL) under N_2 for 6 h. [b] GC yield based on the amount of **2a** used. Value in parentheses indicates yield after isolation. [c] **1a** (0.5 mmol) was used.

Characterization Data of Products

1-(1,2,3,4-Tetraphenylnaphthalen-5-yl)pyrazole (3a) (entry 1 in Table 1): mp 173-174 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.72 (dd, *J* = 1.8, 2.2 Hz, 1H), 6.63-6.81 (m, 15H), 7.15 (d, *J* = 2.2 Hz, 1H), 7.22-7.26 (m, 6H), 7.41-7.42 (m, 2H), 7.74-7.77 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ105.97, 124.79, 125.04, 125.09, 125.37, 126.22 (overlapped), 126.52, 126.61, 127.52, 127.60 (overlapped), 128.91, 130.95 (overlapped), 131.11, 131.23, 132.28, 134.27, 136.15, 137.93, 138.69, 139.42, 139.56, 139.74, 139.89, 140.17, 140.28, 142.16; HRMS *m/z* calcd for $C_{37}H_{26}N_2$ (M^+) 498.2096, found 498.2091. Anal. Calcd for $C_{37}H_{26}N_2$: C, 89.13; H, 5.26; N, 5.62. Found: C, 88.91; H, 5.52; N, 5.43.

1-[1,2,3,4-Tetrakis(4-methylphenyl)naphthalen-5-yl]pyrazole (3b) (entry 2 in Table 1): mp 130-132 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ2.06 (s, 3H), 2.08 (s, 3H), 2.09 (s, 3H), 2.31 (s, 3H), 5.71(t, *J* = 2.2 Hz, 1H), 6.48-6.65 (m, 12H), 7.05-7.08 (m, 5H), 7.21-7.22 (m, 1H), 7.35-7.36 (m, 2H), 7.69-7.73 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ20.96, 21.00, 21.04, 21.22, 105.55, 124.38, 126.69, 126.89, 127.19, 127.32, 127.67, 128.25, 128.84, 130.77 (overlapped), 130.95, 131.11, 132.34, 134.06, 134.10, 134.35, 134.48, 135.82, 136.05, 136.67, 136.82, 137.31, 137.53, 137.91, 138.54, 139.63, 139.94, 142.32; HRMS *m/z* calcd for $C_{41}H_{34}N_2$ (M^+) 554.2722, found 554.2719.

1-[1,2,3,4-Tetrakis(4-methoxyphenyl)naphthalen-5-yl]pyrazole (3c) (entry 3 in Table 1): mp 107-110 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ3.58 (s, 3H), 3.61 (s, 3H), 3.65 (s, 3H), 3.79 (s, 3H), 5.77 (dd, *J* = 1.8, 2.2 Hz, 1H), 6.32 (d, *J* = 8.8 Hz, 2H), 6.34 (d, *J* = 8.8 Hz, 2H), 6.40 (d, *J* = 8.8 Hz, 2H), 6.50 (d, *J* = 8.8 Hz, 2H), 6.63-6.67 (m, 2H), 6.68 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.8 Hz, 2H), 7.08-7.10 (m, 2H), 7.12 (d, *J* = 2.2 Hz, 1H), 7.24 (d, *J* = 1.8 Hz, 1H), 7.37-7.39 (m, 2H), 7.73-7.76 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ54.84, 54.85, 55.09, 55.12, 105.83, 111,85, 111.91, 112.12 (overlapped), 113.07, 124.45, 127.28, 127.59, 128.78, 131.91, 132.06, 132.14, 132.26, 132.30, 132.41, 132.87, 133.12, 134.66, 135.82, 137.83, 138.47, 139.83, 139.99, 142.33, 156.75, 156.87 (overlapped), 158.00; HRMS *m/z* calcd for $C_{41}H_{34}N_2O_4$ (M^+) 618.2519, found 618.2516.

1-[1,2,3,4-Tetrakis(4-chlorophenyl)naphthalen-5-yl]pyrazole (3d) (entry 4 in Table 1): mp 282-283 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ5.86 (dd, *J* = 1.8, 2.2 Hz, 1H), 6.49-6.56 (m, 2H), 6.65-6.71 (m, 4H), 6.76-6.83 (m, 4H), 6.88-6.90 (m, 2H), 7.09-7.11 (m, 2H), 7.15 (d, *J* = 1.8 Hz, 1H), 7.25-7.28 (m, 3H), 7.41-7.48 (m, 2H), 7.67-7.69 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ106.31, 125.53, 126.20, 126.64, 127.03, 127.33, 127.77, 128.11, 128.20, 128.67, 131.30, 131.40, 131.74, 131.90 (overlapped), 131.99, 132.05, 132.18, 133.09, 134.16, 135.55, 137.35, 137.79, 137.88, 138.00, 138.03, 138.31, 140.30, 140.55; HRMS *m/z* calcd for $C_{37}H_{22}Cl_4N_2$ (M^+) 634.0537, found 634.0544. Anal. Calcd for $C_{37}H_{22}Cl_4N_2$: C, 69.83; H, 3.48; N, 4.40. Found: C, 69.87; H, 3.57; N, 4.39.

1-(1,4-Dibutyl-2,3-diphenylnaphthalen-5-yl)pyrazole (3e) (entry 5 in Table 1): oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ0.48 (t, *J* =7.3 Hz, 3H), 0.69-0.79 (m, 5H), 1.02-1.10 (m, 2H), 1.20-1.29 (m, 2H), 1.54-1.59 (m, 3H), 1.90 (s, br, 1H), 2.84 (t, *J* =8.1 Hz, 2H), 6.44 (t, *J* =2.2 Hz, 1H), 6.92-7.12 (m, 10H), 7.40 (d, *J* =7.1 Hz, 1H), 7.53 (d, *J* =7.3 Hz,1H), 7.72(d, *J* =2.2 Hz, 1H), 7.73

(d, J =2.2 Hz, 1H), 8.28 (d, J =8.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 13.48, 13.60, 22.63, 23.05, 30.22, 30.42, 33.33, 35.38, 106.37, 124.20, 125.73, 125.85, 126.86, 127.10 (overlapped), 127.21, 128.15, 130.04 (overlapped), 131.94, 133.82, 134.57, 134.73, 137.91, 140.05, 140.20, 141.26, 141.47, 142.29; HRMS m/z calcd for $C_{33}H_{34}N_2$ (M^+) 458.2722, found 458.2718.

1-(1,4-Dimethyl-2,3-diphenylnaphthalen-5-yl)pyrazole (3f) (entry 6 in Table 1): oil; ^{1}H NMR (400 MHz, $CDCl_3$) 1.60 (s, 3H), 2.49 (s, 3H), 6.45 (dd, J = 1.8, 2.2 Hz, 1H), 6.91-7.15 (m, 10H), 7.51 (d, J = 7.4, 1H), 7.58 (dd, J =7.4, 8.4 Hz, 1H), 7.66 (d, J =2.2 Hz, 1H), 7.72 (d, J = 1.8 Hz, 1H), 8.29 (d, J =8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 16.71, 17.44, 106.54, 124.50, 125.89, 125.97, 126.92, 127.28 (overlapped), 129.09, 129.63, 130.26 (overlapped), 132.52, 134.11, 138.04, 140.05, 140.22, 141.41, 141.65, 142.57; HRMS m/z calcd for $C_{27}H_{22}N_2$ (M^+) 374.1783, found 374.1775.

3-Methyl-1-(1,2,3,4-tetraphenylnaphthalen-5-yl)pyrazole (3g) (entry 7 in Table 1): mp 169-170 ºC; ^{1}H NMR (400MHz, $CDCl_3$) 2.05 (s, 3H), 5.46 (d, J = 2.2 Hz, 1H), 6.56-6.81 (m, 15H), 7.09 (d, J = 2.6 Hz, 1H), 7.18-7.24 (m, 5H), 7.38-7.40(m, 2H), 7.70-7.73 (m, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) 13.32, 105.64, 124.72, 124.97, 124.99, 125.32, 125.47, 126.18, 126.50, 126.56, 127.07, 127.55 (overlapped), 127.79, 128.68, 130.95 (overlapped), 131.23, 132.46, 134.38, 136.30, 137.99, 138.57, 139.49, 139.61, 139.67, 140.24, 140.35, 141.97, 148,85; HRMS m/z calcd for $C_{38}H_{28}N_2$ (M^+) 512.2252, found 512.2244. Anal. Calcd for $C_{38}H_{28}N_2$: C, 89.03; H, 5.51; N, 5.46. Found: C, 89.07; H, 5.65; N, 5.37.

3,5-Dimethyl-1-(1,2,3,4-tetraphenylnaphthalen-5-yl)pyrazole (3h) (entry 8 in Table 1): mp 201 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) 2.01 (s, 3H), 2.02 (s, 3H), 5.18 (s, 1H), 6.50-6.52 (m, 1H), 6.70-6.86 (m, 14H), 7.20-7.29 (m, 6H), 7.39-7.43 (m, 1H), 7.72-7.74 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 12.1, 13.3, 105.24, 124.71, 124.95, 125.07, 125.10, 125.28, 125.30, 126.06, 126.19, 126.40, 126.54, 126.55, 127.53, 127.55, 128.02, 128.83, 129.53, 130.82, 130.91, 130.98, 131.00, 131.25, 131.38, 131.50, 134.41, 136.39, 138.60, 139.11, 139.60, 139.64, 139.98, 140.25, 140.36, 142.08, 148.14; HRMS m/z calcd for $C_{39}H_{30}N_2$ (M^+) 526.2409, found 526.2406. Anal. Calcd for $C_{39}H_{30}N_2$: C, 88.94; H, 5.74; N, 5.32. Found: C, 88.87; H, 5.98; N, 4.93.

1-Methyl-2-(1,2,3,4-tetraphenylnaphthalen-5-yl)benzimidazole (5) (eq 2): mp198-199 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) 3.40 (s, 3H), 6.24-6.28 (m, 1H), 6.30-6.34 (m,1H), 6.59-6.63 (m, 2H), 6.68-6.94 (m, 12H), 7.10-7.13 (m, 2H), 7.18-7.22 (m, 2H), 7.24-7.27 (m, 3H), 7.44-7.53 (m, 3H), 7.80-7.83 (m, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) 30.87, 108.57, 119.18, 121.47, 121.82, 124.46, 124.66, 124.76, 125.08, 125.35, 125.73, 126.12, 126.35, 126.42, 126.57, 126.61, 127.49, 127.67, 128.37, 128.97, 129.75, 130.82, 130.95, 131.08, 131.11, 131.15, 131.25, 131.33, 131.52, 132.46, 133.44, 134.89, 137.67, 138.74, 138.89, 139.55, 139.59, 140.28, 140.35, 141.71, 142.84, 154.22; HRMS m/z calcd for $C_{42}H_{30}N_2$ (M^+) 562.2409, found 562.2407.

5,6-Diphenylbenzimidazo[2,1-*a*]isoquinoline (6) (eq 3): mp 281-283 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) 6.01 (d, J = 8.4 Hz, 1H), 6.93 (dd, J = 7.3, 8.1 Hz, 1H), 7.21-7.30 (m, 5H), 7.33-7.42 (m, 7H), 7.57 (dd, J = 7.0, 8.0 Hz, 1H), 7.68 (dd, J = 7.0, 8.1 Hz,1H), 7.98 (d, J = 8.1 Hz, 1H), 8.99 (d, J = 8.1 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) 116.04, 121.49, 123.14, 124.85, 125.44, 126.02, 126.97, 128.29, 129.17, 129.67, 129.93, 130.65, 131.13, 131.79, 132.55, 133.12, 133.42, 134.56, 135.65, 137.04, 137.58, 146.19, 149.68; HRMS m/z calcd for $C_{27}H_{18}N_2$ (M^+) 370.1470, found 370.1467. Anal. Calcd for $C_{27}H_{18}N_2$: C, 87.54; H, 4.90; N, 7.56. Found: C, 87.39; H, 4.95; N, 7.57.

5,6-Diphenylimidazo[2,1-*a*]isoquinoline (7) (eq 4):[17] mp 236-237 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) 7.18-7.21 (m, 3H), 7.25-7.33 (m, 8H), 7.39 (d, J = 8.2 Hz, 1H), 7.47 (dd, J = 7.3, 8.0 Hz, 1H), 7.54 (s, 1H), 7.64 (t, J = 7.6 Hz, 1H), 8.76 (d, J = 8.0 Hz, 1H); ^{13}C-NMR (100 MHz, $CDCl_3$) 123.15, 123.25, 124.42, 126.42, 127.27, 127.81, 127.99, 128.03, 128.06, 128.55, 128.63, 128.82, 130.20, 130.60, 130.89, 131.41, 133.39, 133.50, 135.93; HRMS m/z calcd for $C_{23}H_{16}N_2$ (M^+) 320.1313, found 320.1307. Anal. Calcd for $C_{23}H_{16}N_2$: C, 86.22; H, 5.03; N, 8.74. Found: C, 86.07; H, 5.16; N, 8.49.

2-(1,2,3,4,5,6,7,8-Octaphenylanthracen-9-yl)benzoxazole (8) (eq 5): mp >290 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) 6.11 (t, J = 7.7 Hz, 2H), 6.28 (dd, J = 7.7, 8.0 Hz, 4H), 6.54-6.56 (m, 4H), 6.63 (d, J = 8.4 Hz, 4H), 6.67-6.68 (m, 6H), 6.73-6.82 (m, 11H), 6.89-6.94 (m, 2H), 6.97-7.06 (m, 11H), 8.11 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) 110.59, 119.71, 121.80, 122.89, 123.69, 124.13, 124.95, 125.11, 125.40, 126.18, 126.30, 126.55, 127.26, 130.60, 130.95, 131.05, 131.20, 131.25, 131.74, 131.76, 136.89, 138.51, 138.53, 138.58, 139.01, 140.39, 140.58, 141.38, 142.87, 149.79, 161.34; HRMS m/z calcd for $C_{69}H_{45}NO$ (M^+) 903.3501, found 903.3499.

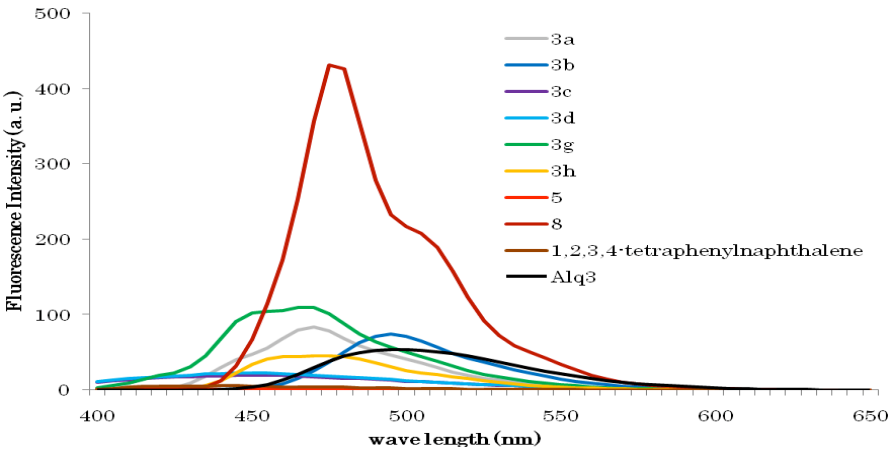


Figure S1. Fluoescence Spectra of **3a**-**d**, **3g**-**h**, **5**, **8**, 1,2,3,4,-tetraphenylnaphthalene, and Alq_3 in the solid-state[16]

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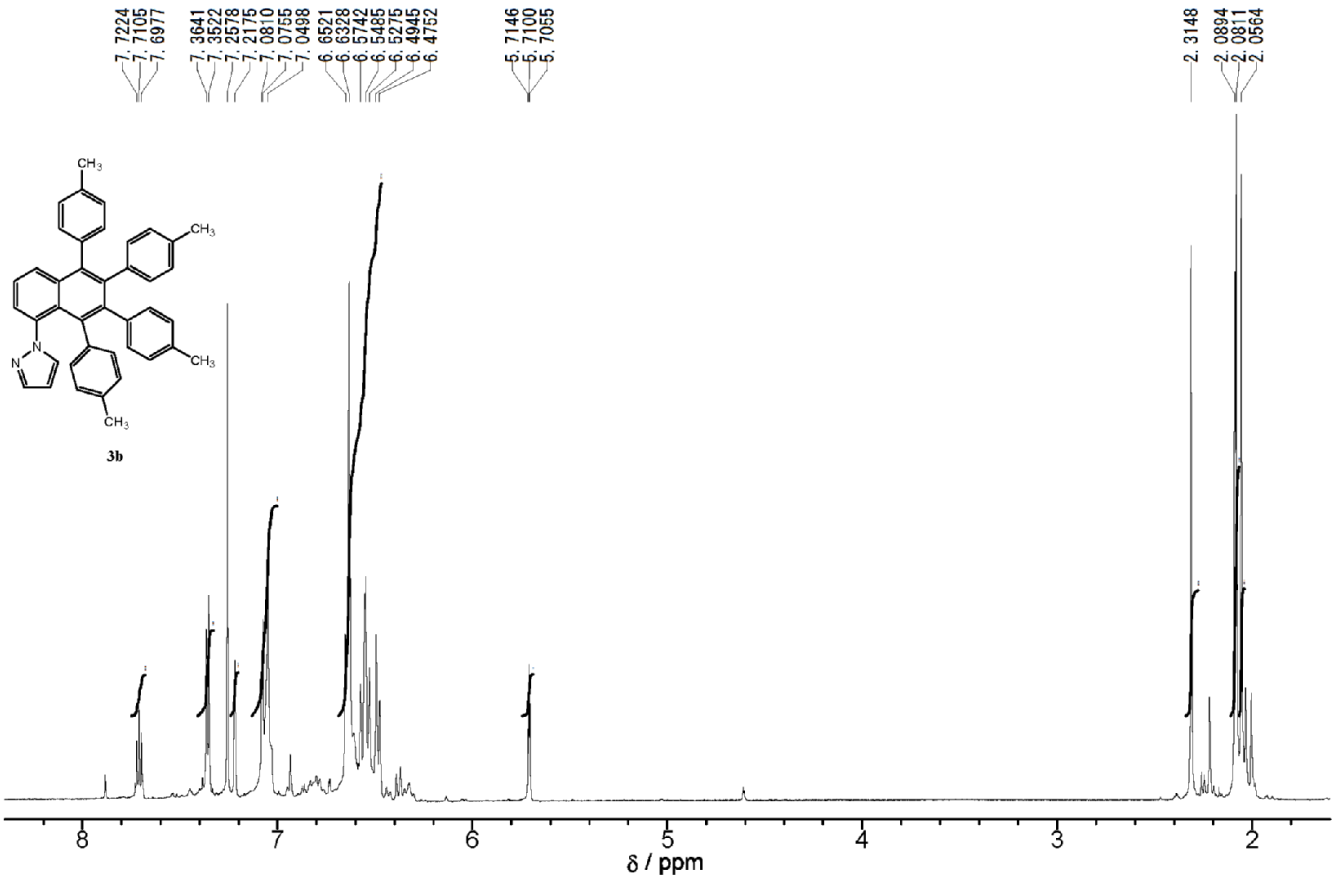
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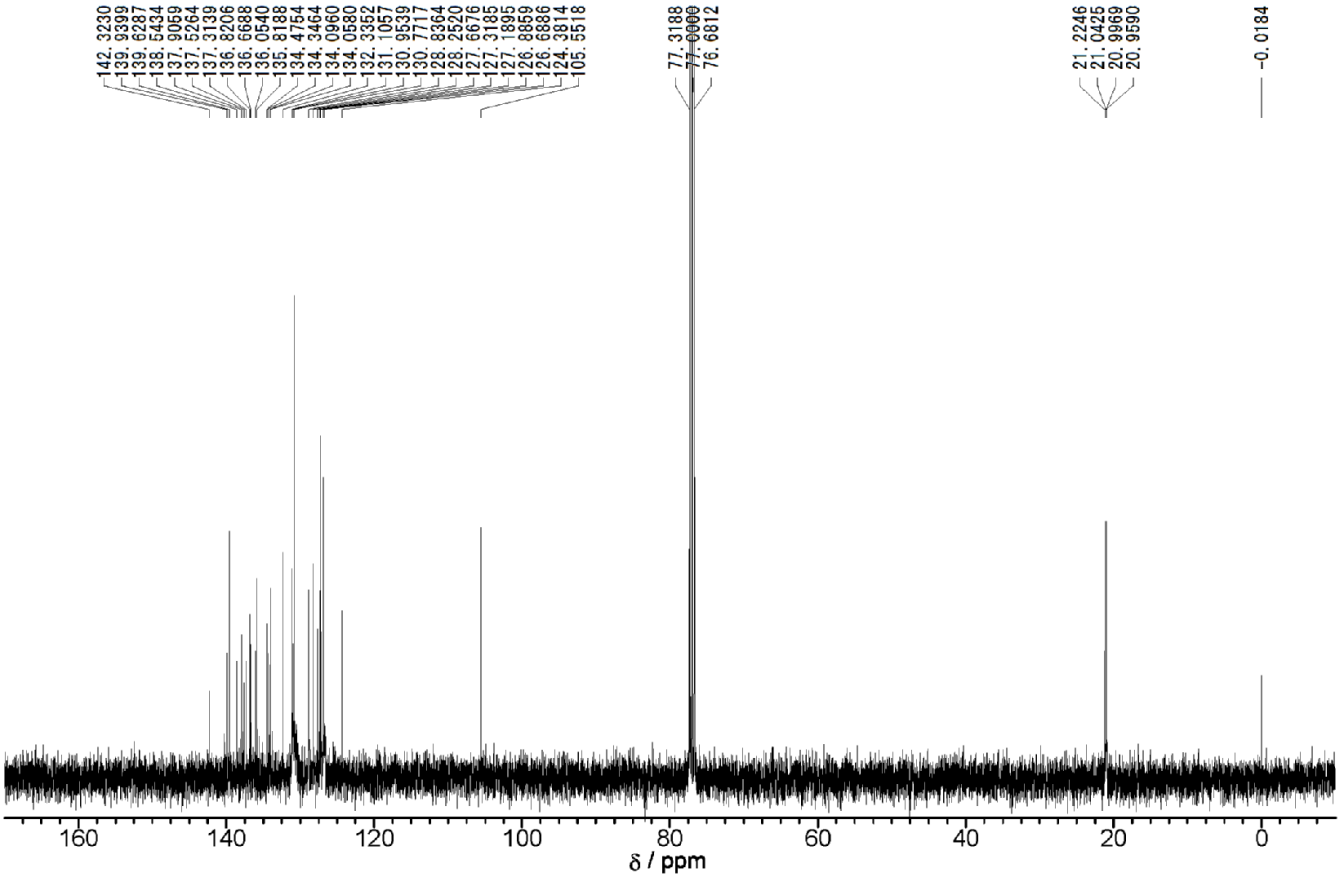
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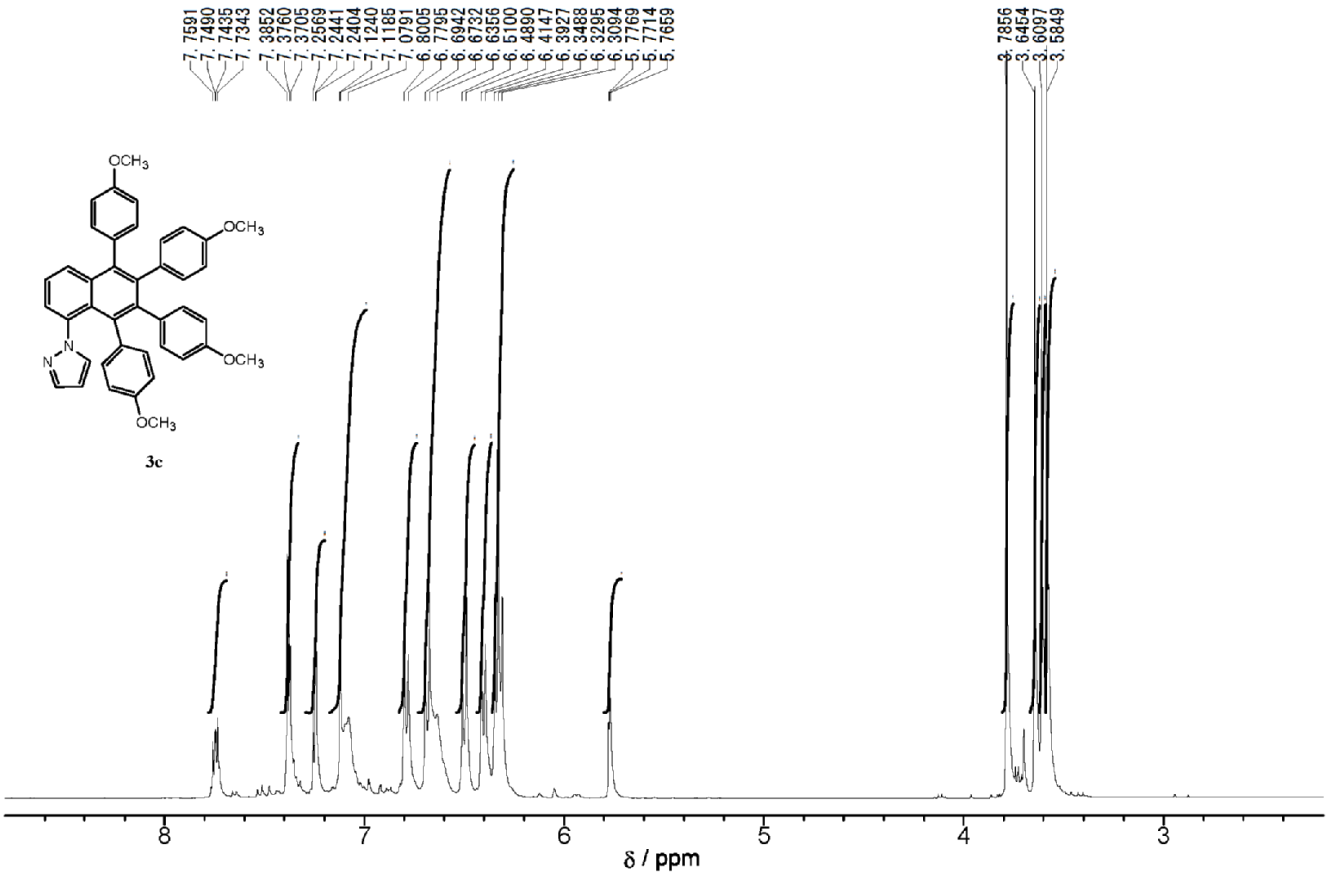
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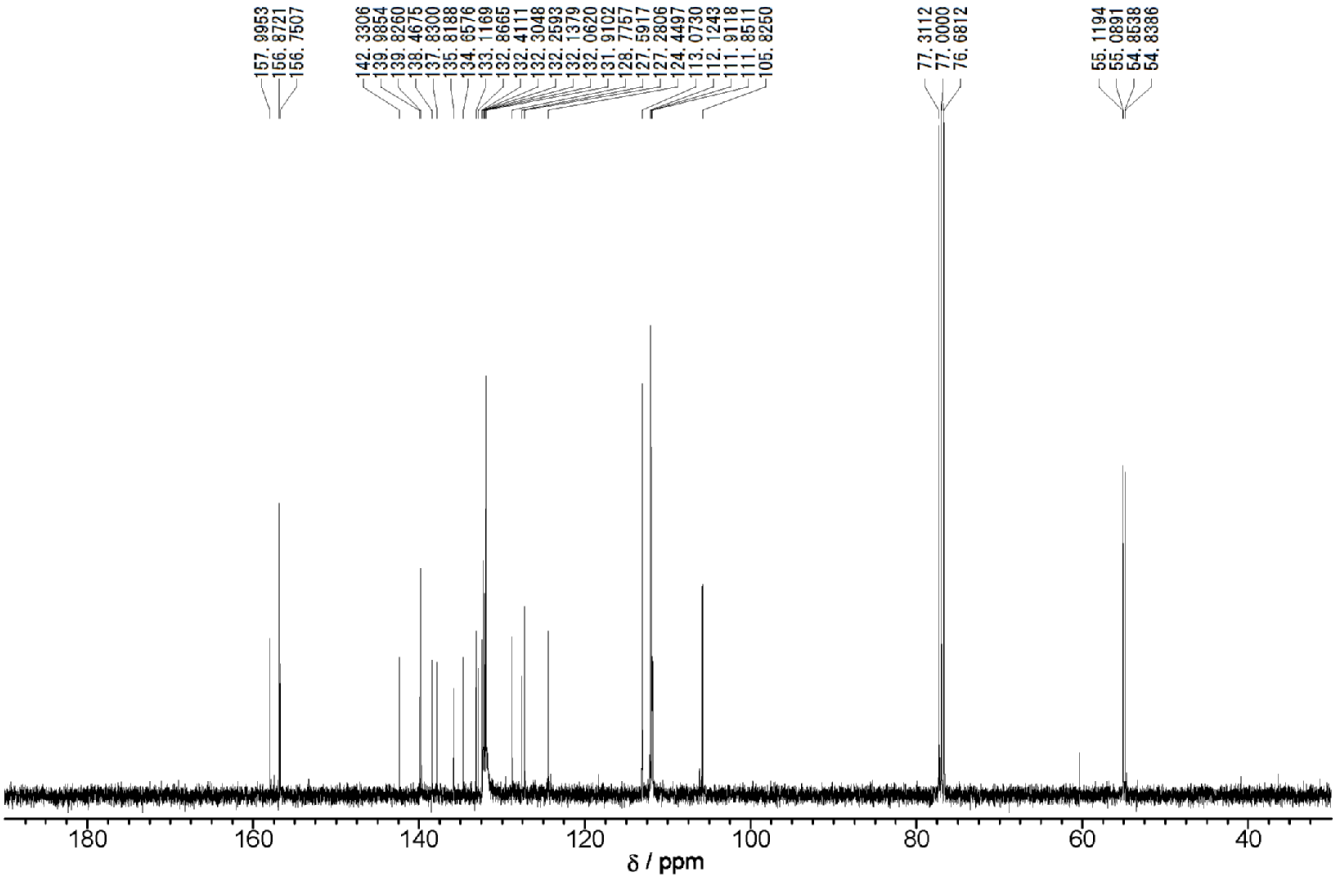
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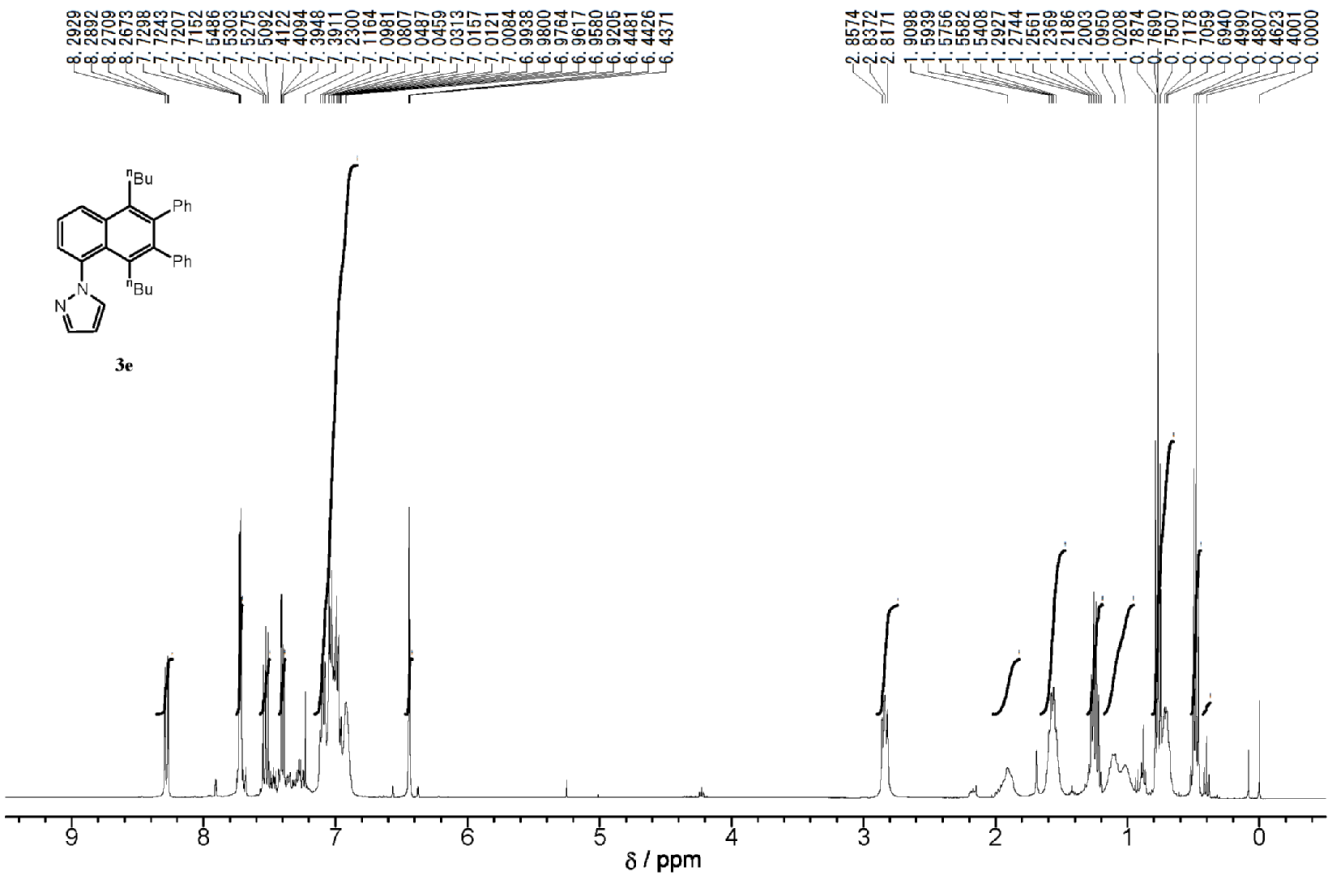
δ / ppm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7.3948
7.3911
7.2300
7.1164
7.0981
7.0807
7.0487
7.0459
7.0313
7.0157
7.0121
7.0084
6.9938
6.9800
6.9764
6.9617
6.9580
6.9205
6.4481
6.4426
6.4371
2.8574
2.8372
2.8171
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1.5939
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1.5582
1.5408
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1.2744
1.2561
1.2369
1.2186
1.2003
1.0950
1.0208
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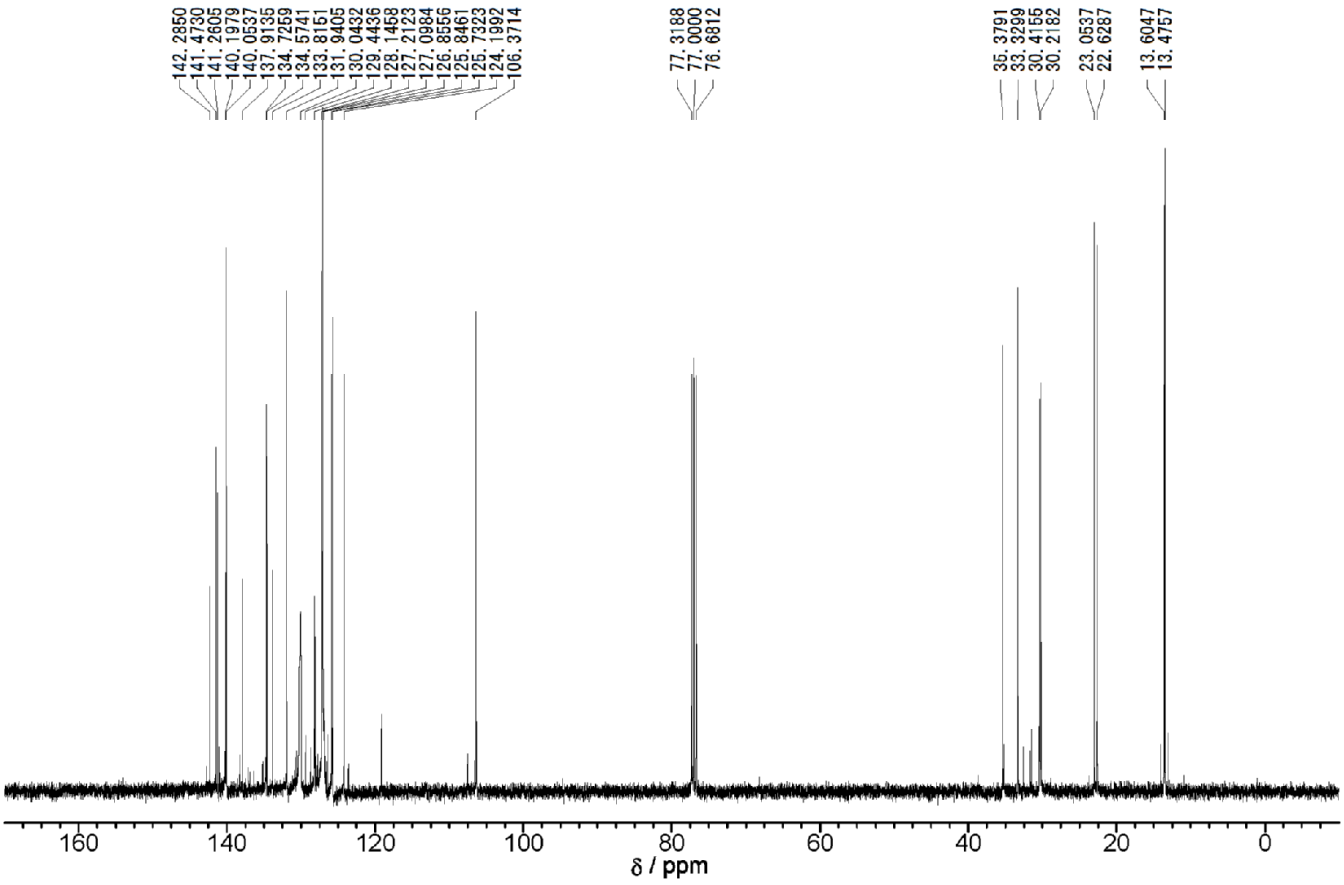
0.7690
0.7507
0.7178
0.7059
0.6940
0.4990
0.4807
0.4623
0.4001
0.0000
nBu
Ph
Ph
nBu
N
N
3e
9
8
7
6
5
4
3
2
1
0
δ / ppm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129.4436
128.1458
127.2123
127.0984
126.8556
125.8461
125.7323
124.1992
106.3714
77.3188
77.0000
76.6812
35.3791
33.3299
30.4155
30.2182
23.0537
22.6287
13.6047
13.4757
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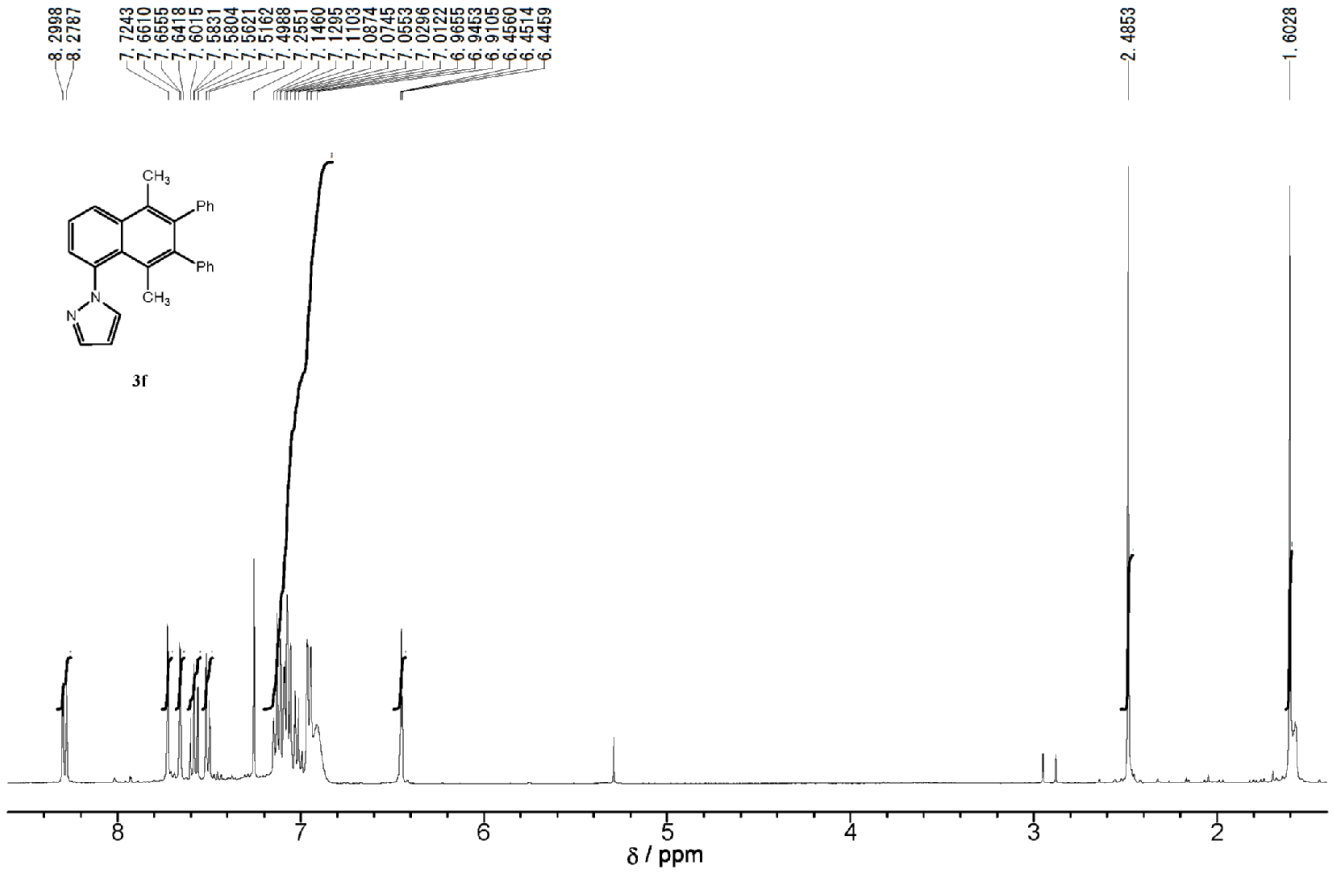
140
120
100
80
60
40
20
0
δ / ppm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7.4988
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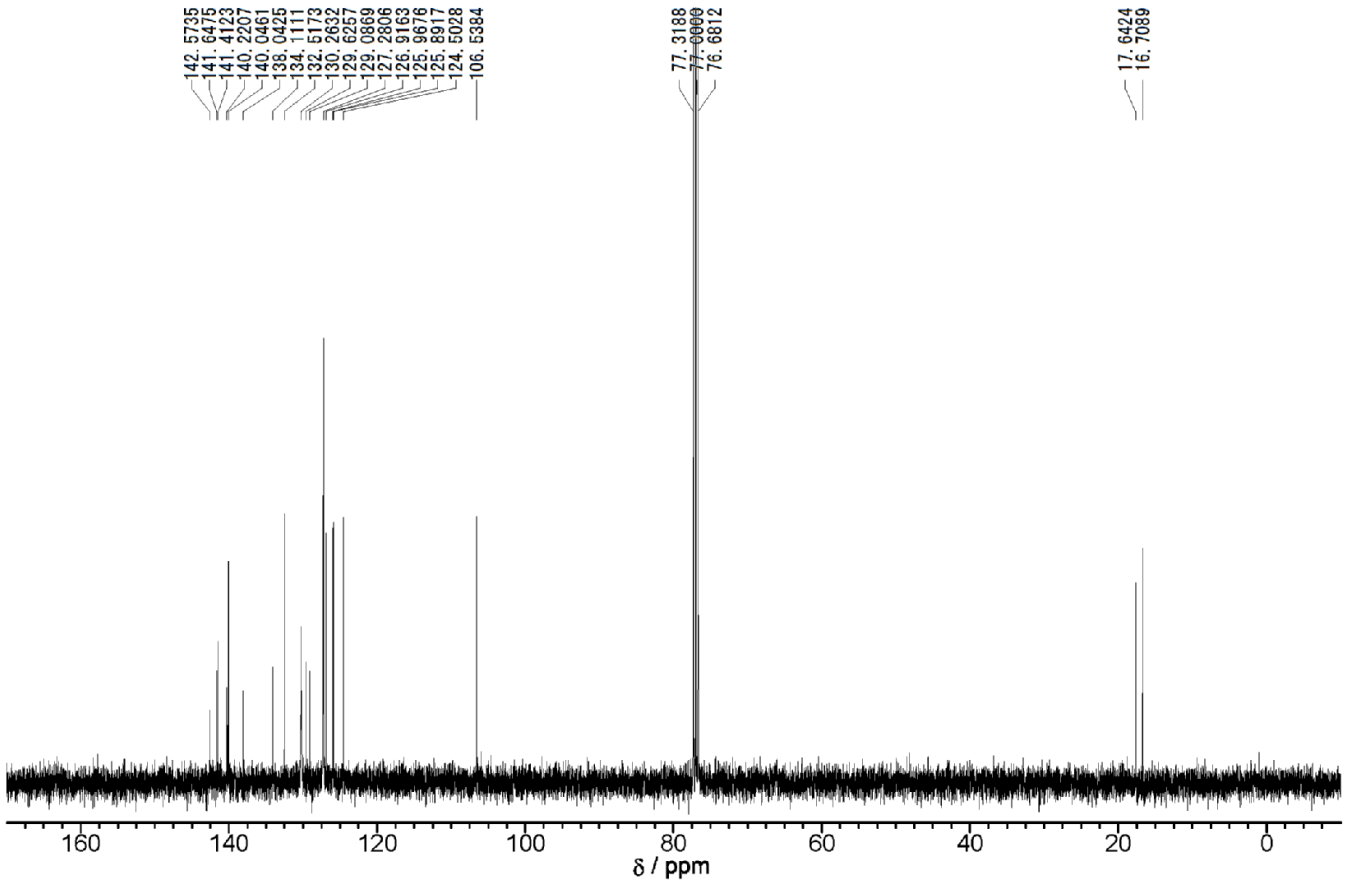
7.1460
7.1295
7.1103
7.0874
7.0745
7.0553
7.0296
7.0122
6.9655
6.9463
6.9105
6.4560
6.4514
6.4469
2.4853
1.6028
CH3
Ph
Ph
CH3
N
N
3f
8
7
6
5
4
3
2
δ / ppm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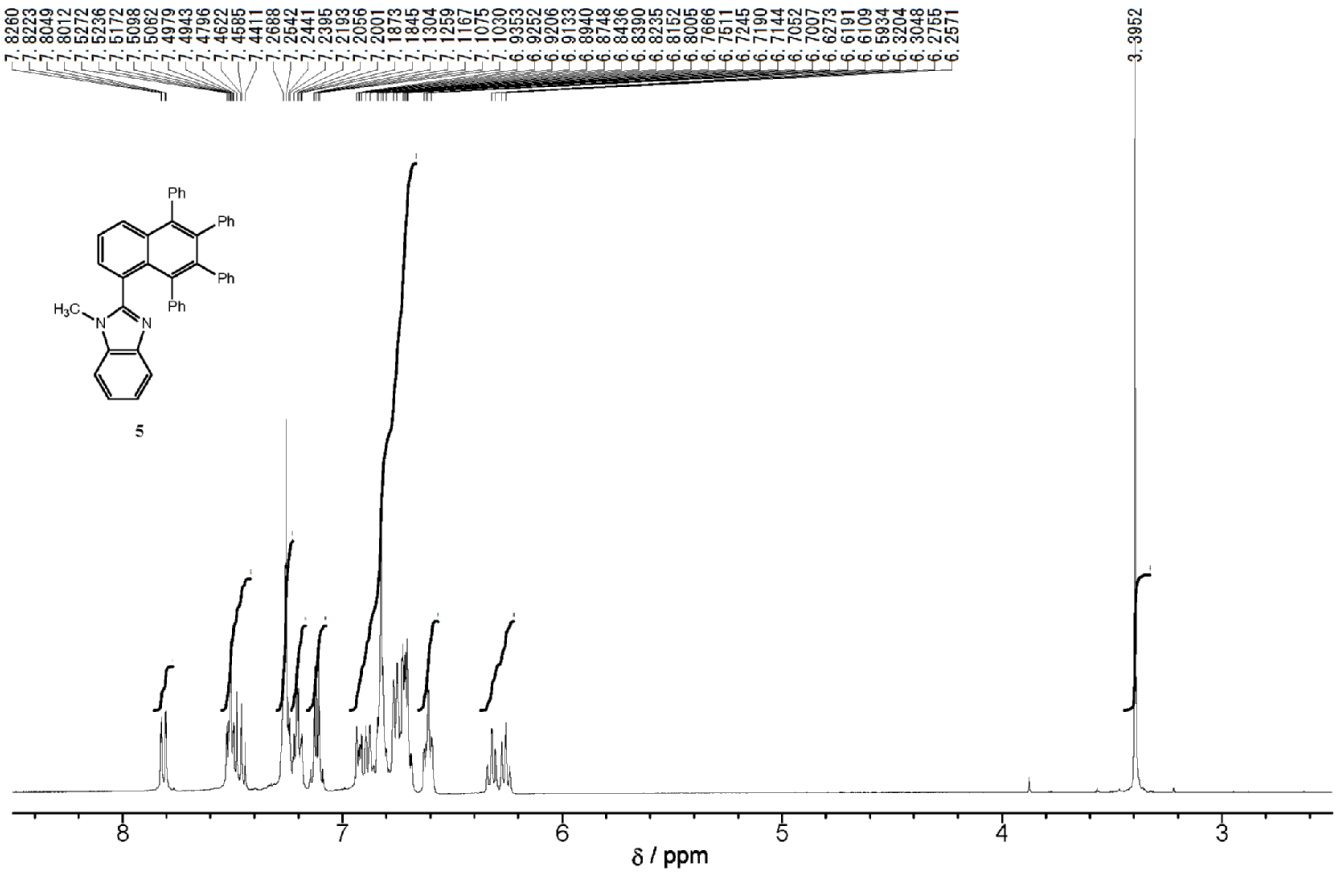
16.7089
160
140
120
100
80
60
40
20
0
δ / ppm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7.4622
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7.4411
7.2688
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6.9206
6.9133
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6.8748
6.8436
6.8390
6.8235
6.8152
6.8005
6.7666
6.7511
6.7245
6.7190
6.7144
6.7052
6.7007
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6.6191
6.6109
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6.3204
6.3048
6.2755
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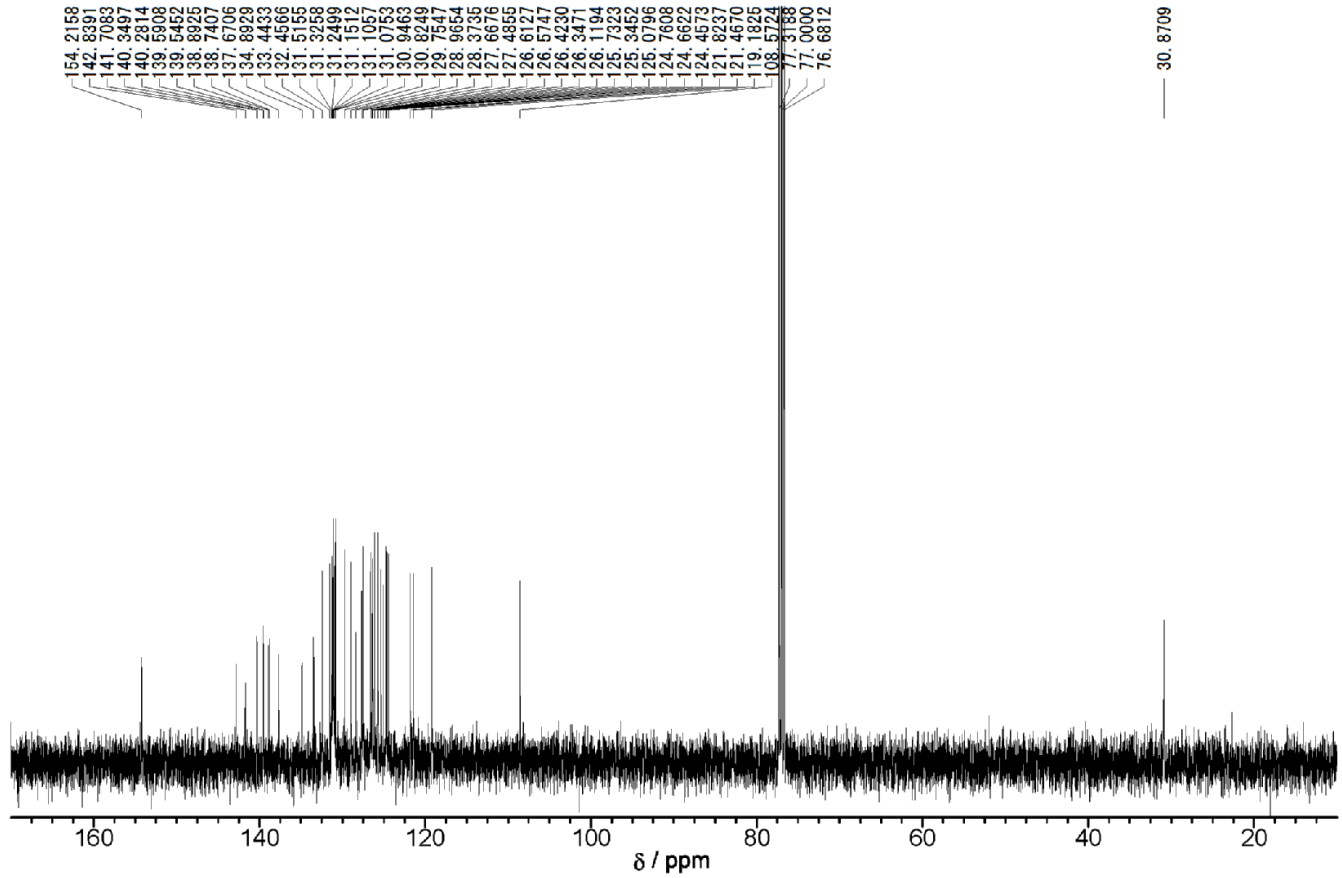
Ph
Ph
Ph
Ph
H3C
N
N
5
8
7
6
5
4
3
δ / ppm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127.4855
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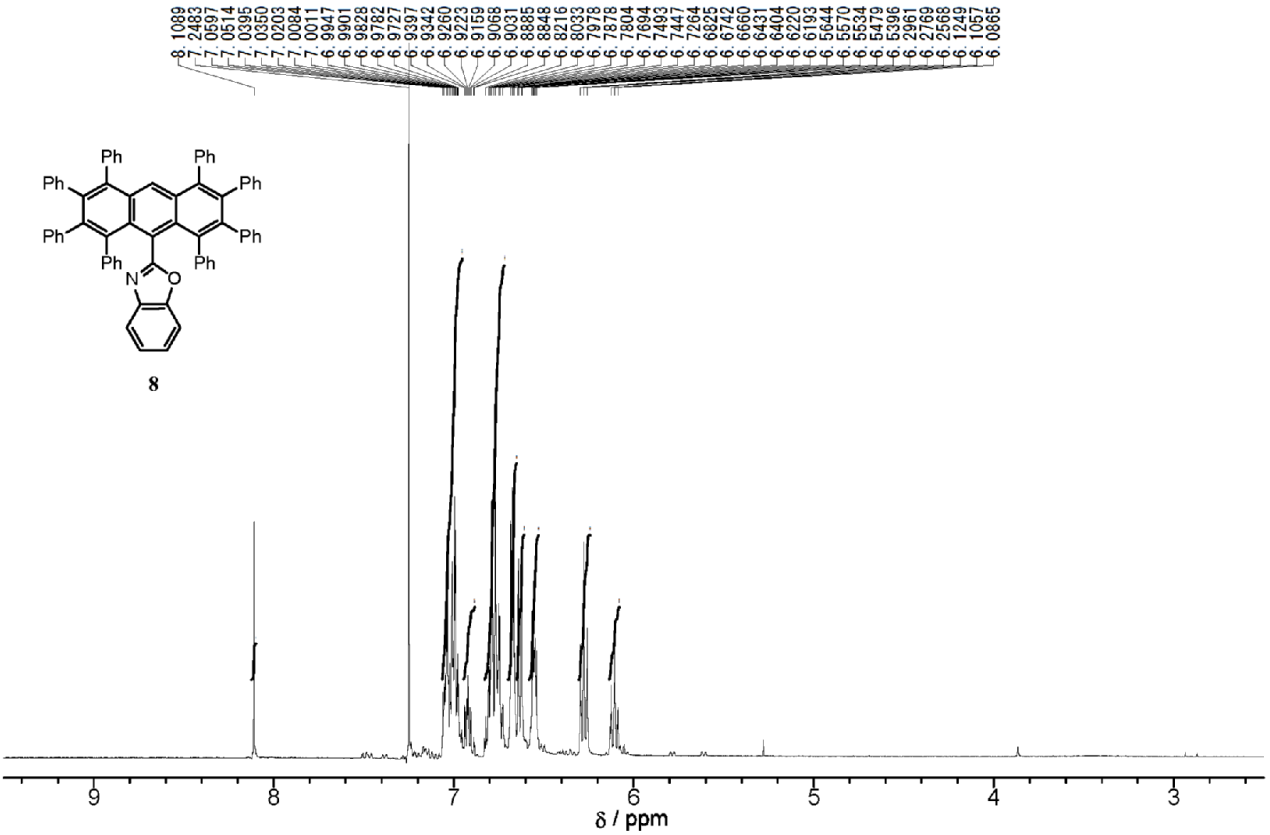
77.0000
76.6812
30.8709
160
140
120
100
80
60
40
20
δ / ppm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7.0011
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6.9828
6.9782
6.9727
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6.9342
6.9260
6.9223
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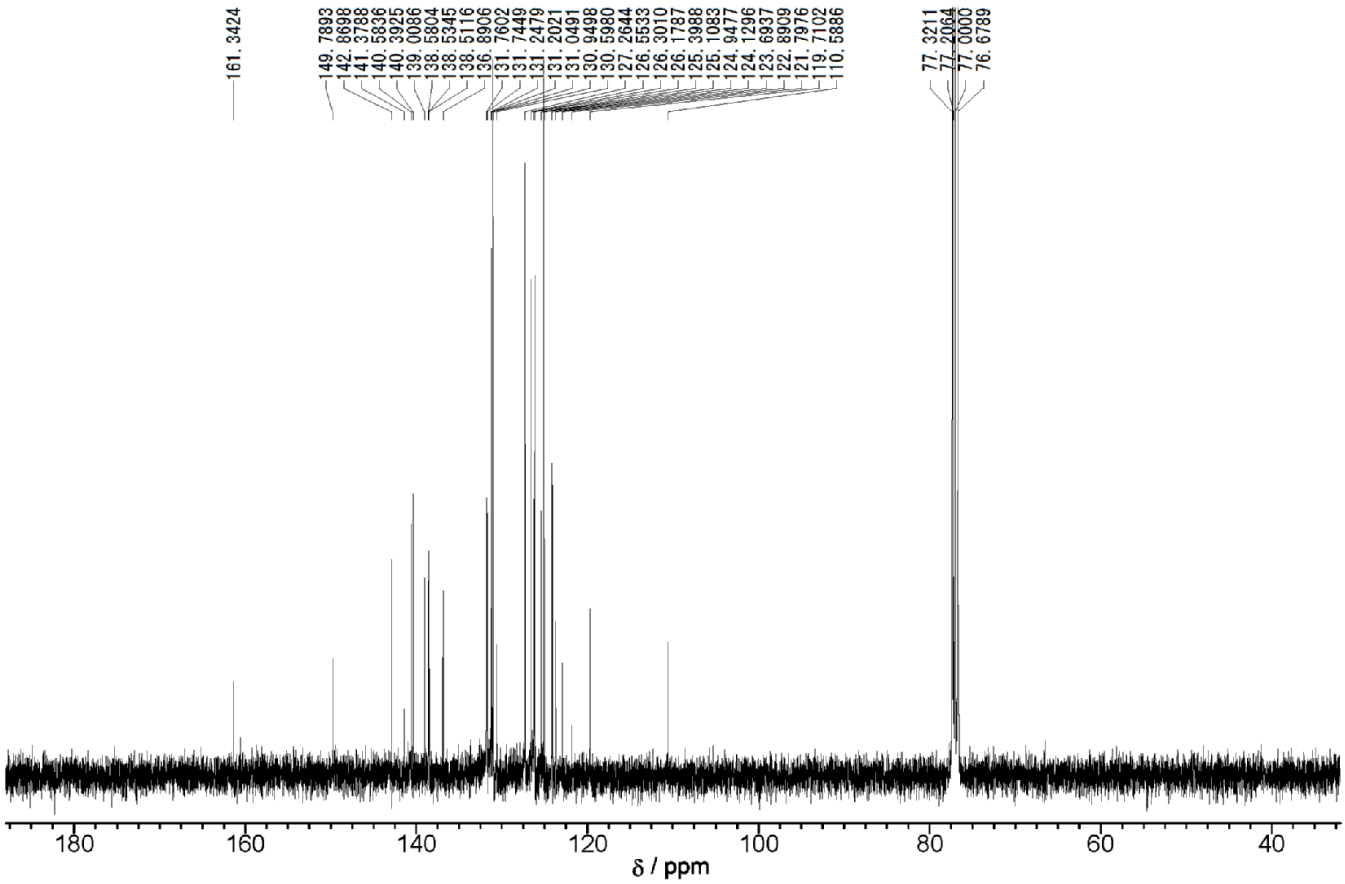
6.9068
6.9031
6.8885
6.8848
6.8216
6.8033
6.7978
6.7878
6.7804
6.7694
6.7493
6.7447
6.7264
6.6825
6.6742
6.6660
6.6431
6.6404
6.6220
6.6193
6.5644
6.5570
6.5534
6.5479
6.5396
6.2961
6.2769
6.2568
6.1249
6.1057
6.0865
Ph
Ph
Ph
Ph
Ph
Ph
Ph
Ph
N
O
8
9
8
7
6
5
4
3
δ / ppm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130.9498
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123.6937
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119.7102
110.5886
77.3211
77.2064
77.0000
76.6789
180
160
140
120
100
80
60
40
δ / ppm