Lewis Acid-Catalyzed Double Addition of Indoles to Ketones: Synthesis of Bis(indolyl)methanes with All-Carbon Quaternary Centers

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Supporting Information

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I. General Information

Unless otherwise specified, all reactions were conducted with stirring under an atmosphere of nitrogen. All reagents including anhydrous solvents were purchased from Sigma Aldrich, TCI or Alfa Aesar and used as received. N-ethylindole and N-allylindole were prepared by the reaction between indole and alkyl halides.[1] Flash column chromatography was performed on silica gel 60 (40—63 μm) as a stationary phase.

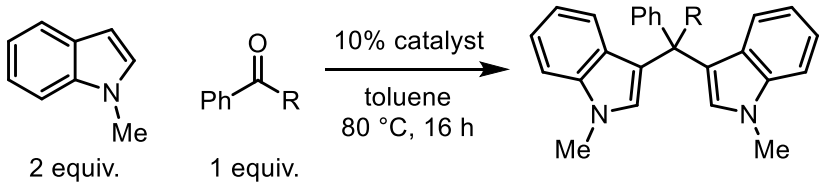
NMR spectra were recorded with a Bruker AVANCE III HD 300 (300 MHz) or a Bruker AVANCE III HD 400 (400 MHz) at Yonsei University, using $CDCl_3$ as the solvent. Chemical shifts were expressed in parts per million (ppm, δ), referenced to the residual signal of $CDCl_3$ (7.26 ppm for ^1H, 77.16 ppm for ^{13}C). All coupling constants (*J*) were expressed in Hertz (Hz). The following abbreviations were used for the descriptions of splitting patterns: s = singlet, d = doublet, t = triplet, m = multiplet.

High resolution mass spectra were obtained using an Agilent 6530 Accurate-Mass Q-TOF.

[1] (a) G. Raina, P. Kannaboina, N. Mupparapu, S. Raina, Q. N. Ahmed and P. Das, *Org. Biomol. Chem.*, 2019, **17**, 2134-2147. (b) K. J. Hock, A. Knorrscheidt, R. Hommelsheim, J. Ho, M. J. Weissenborn and R. M. Koenigs, *Angew. Chem. Int. Ed.*, 2019, **58**, 3630-3634.

II. Development of LA-Catalyzed Additions of Indoles to Ketones

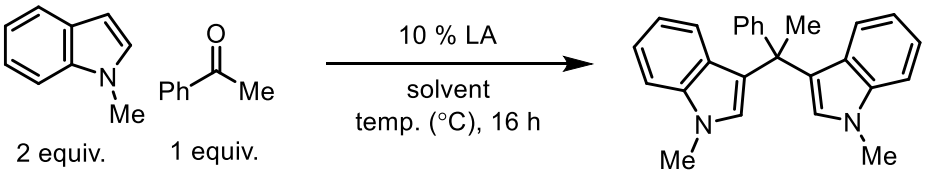
General procedure for Table 1 in the main paper and other experiments in Section II.



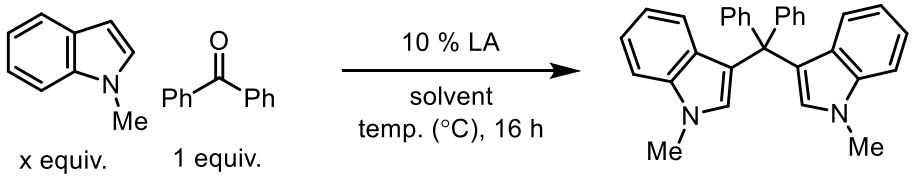
2a, R = Me
2b, R = Ph

In a nitrogen-filled glovebox, ketone (**2a** 6.1 mg/ **2b** 9.1 mg, 0.050 mmol, 1.0 equiv.), 1-methylindole (24.9-49.8 μL, 2.00–4.00 equiv.), a Lewis acid catalyst (10 mol% of $B(C_6F_5)_3$ (2.5 mg) or $PhSiCl_3$ (0.80 μL)) and toluene (1.0 M, 0.050 mL) were combined in a 4-mL vial equipped with a stir bar. The resulting mixture was stirred at 80 °C overnight. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The yield of the product was determined by ^1H NMR spectroscopy with 1,1,2,2-tetrachloroethane as an internal standard.

***Table S1**. Effect of temperatures and solvents.*[a]



entry	cat.	cat. mol%	T (°C)	solvent	yield (%)[b]
1	$B(C_6F_5)_3$	10	r.t.	chloroform	79
2	$B(C_6F_5)_3$	10	r.t.	toluene	78
3	$B(C_6F_5)_3$	10	r.t.	THF	6
4	$B(C_6F_5)_3$	10	80	chloroform	77
5	$B(C_6F_5)_3$	10	80	toluene	**96** (85[c])
6	$B(C_6F_5)_3$	10	80	THF	27
7	$B(C_6F_5)_3$	10	80	neat	71
8	$B(C_6F_5)_3$	5	80	toluene	52 (>95[d])
9	$PhSiCl_3$	10	r.t.	chloroform	99
10	$PhSiCl_3$	10	r.t.	toluene	80
11	$PhSiCl_3$	10	r.t.	THF	60
12	$PhSiCl_3$	10	80	chloroform	90
13	$PhSiCl_3$	10	80	toluene	**99** (93[c])
14	$PhSiCl_3$	10	80	THF	83
15	$PhSiCl_3$	10	80	neat	78



entry	cat.	x equiv.	T (°C)	solvent	yield (%)[b]
1	$B(C_6F_5)_3$	2	r.t.	chloroform	<5
2	$B(C_6F_5)_3$	2	r.t.	toluene	<5
3	$B(C_6F_5)_3$	2	80	chloroform	7
4	$B(C_6F_5)_3$	2	80	toluene	22
5	$B(C_6F_5)_3$	4	80	chloroform	36
6	$B(C_6F_5)_3$	4	80	toluene	**56**

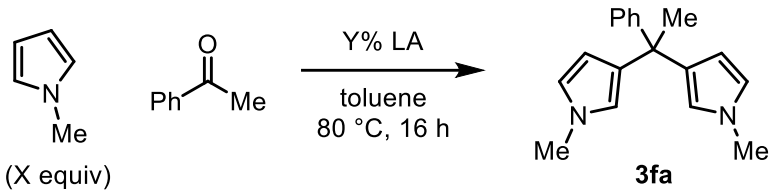
[a] Reaction conditions: acetophenone (0.050 mmol), 1-methylindole (0.10 mmol), and catalyst (10 mol%) in solvent for 16 h. [b] Determined by ^1H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard. [c] Yield after 3 h. [d] Yield after 72 h.

***Table S2**. Effect of boron and silicon catalysts for each substrate.*[a]

product	cat.	yield (%)[b]	cat.	yield (%)[c]
3aa	$PhSiCl_3$	99	$B(C_6F_5)_3$	97
3ab	$B(C_6F_5)_3$	56	$PhSiCl_3$	41
3ac	$PhSiCl_3$	72	$B(C_6F_5)_3$	57
3ad	$B(C_6F_5)_3$	80	$PhSiCl_3$	48
3ae	$PhSiCl_3$	96	$B(C_6F_5)_3$	95
3af	$PhSiCl_3$	98	$B(C_6F_5)_3$	57
3ag	$B(C_6F_5)_3$	55	$PhSiCl_3$	30
3ah	$B(C_6F_5)_3$	72	$PhSiCl_3$	95
3ai	$B(C_6F_5)_3$	64	$PhSiCl_3$	42
3aj	$B(C_6F_5)_3$	45	$PhSiCl_3$	11
3ak	$PhSiCl_3$	94	$B(C_6F_5)_3$	74
3al	$PhSiCl_3$	98	$B(C_6F_5)_3$	63
3am	$B(C_6F_5)_3$	57	$PhSiCl_3$	<5
3an	$B(C_6F_5)_3$	63	$PhSiCl_3$	40
3ao	$B(C_6F_5)_3$	94	$PhSiCl_3$	75
3ap	$PhSiCl_3$	92	$B(C_6F_5)_3$	43
3aq	$B(C_6F_5)_3$	63	$PhSiCl_3$	<5
3ar	$B(C_6F_5)_3$	78	$PhSiCl_3$	15
3as	$PhSiCl_3$	46	$B(C_6F_5)_3$	30
3at	$PhSiCl_3$	83	$B(C_6F_5)_3$	46
3ba	$PhSiCl_3$	30	$B(C_6F_5)_3$	23
3ca	$PhSiCl_3$	76	$B(C_6F_5)_3$	93
3da	$PhSiCl_3$	88	$B(C_6F_5)_3$	57
3ea	$PhSiCl_3$	79	$B(C_6F_5)_3$	80
3fa	$B(C_6F_5)_3$	97	$PhSiCl_3$	70

[a] Reaction conditions: standard condition. [b] Isolated yield under optimized condition. [c] Determined by ^1H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

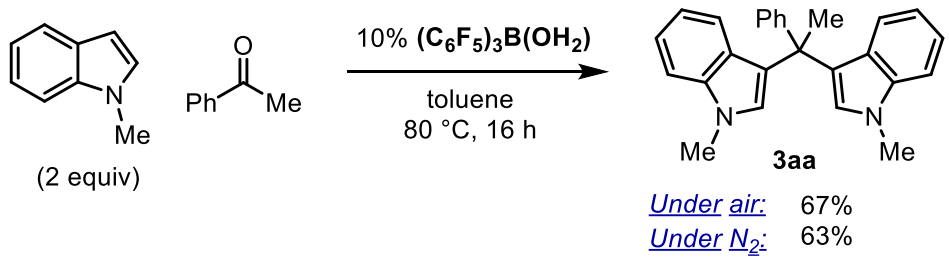
Table S3. *LA-catalyzed double-additions of 1-methylpyrrole to acetophenone: Effect of reaction parameters.*[a]



entry	cat.	Y (%)	X (equiv)	yield (%)[b]
1	$B(C_6F_5)_3$	10	2	5
2	$B(C_6F_5)_3$	20	2	12
3	$B(C_6F_5)_3$	20	3	62
4	$B(C_6F_5)_3$	20	4	97[c]
5	$PhSiCl_3$	10	2	4
6	$PhSiCl_3$	20	4	70

[a] Reaction conditions: acetophenone (0.050 mmol), 1-methylpyrrole (0.10-0.20 mmol), and catalyst (10-20%) in toluene for 16 h. [b] Determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard. [c] Isolated yield

Scheme S1. *Reaction with $(C_6F_5)_3B(OH_2)$ as a catalyst.*[a]



[a] Reaction conditions: acetophenone (0.050 mmol), 1-methylindole (0.10 mmol), and catalyst (10%) in toluene for 16 h. [b] Yields were determined by ^{1}H NMR spectroscopic analysis with 1,1,2,2-tetrachloroethane as an internal standard.

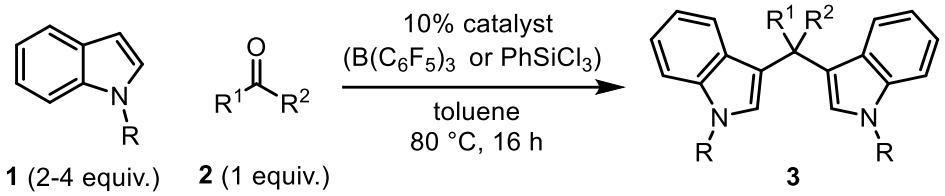
: Because water was generated as a side-product in our reaction, the hydrated form of a catalyst, $(C_6F_5)_3B(OH_2)$[2] was tried as a catalyst. The product was produced in 63-67% yield (lower than the product yield obtained from the reaction with $B(C_6F_5)_3$); once $B(C_6F_5)_3$ is hydrated, the reaction atmosphere (air or N_2) does not seem to affect the reaction yield.

[2] Rabanzo-Castillo, K. M.; Kumar, V. B.; Söhnel, T.; Leitao, E. M. Catalytic Synthesis of Oligosiloxanes Mediated by an Air Stable Catalyst, $(C_6F_5)_3B(OH_2)$. *Front. Chem.* **2020**, *8*, 477.

III. LA-Catalyzed Additions of *N*-Heterocycles to Ketones

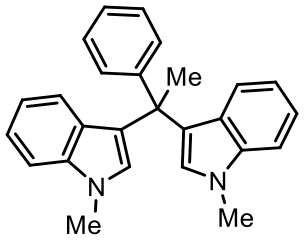
General Procedure

Table 2, Scheme 2, Scheme 3



In a nitrogen-filled glovebox, ketone (0.20–0.25 mmol, 1.0 equiv.), indole (2.0–4.0 equiv.), a Lewis acid catalyst (10 mol% of $B(C_6F_5)_3$ or $PhSiCl_3$) and toluene (1.0 M, 0.20–0.25 mL) were combined in 20-mL vial equipped with a stir bar. The resulting mixture was stirred at 80 ℃ overnight. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography. The yield of the product was calculated based on the amount of ketone. The yield of the product was calculated based on the amount of ketone.

The following compounds have been reported previously: **3aa**, **3ad**, **3ah** in Table 2, **3ar** in Scheme 2, and **3ba**, **3ca**, **3da**, **3ea**, **3fa** in Scheme 3.



3,3'-(1-Phenylethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3aa) [96005-17-3]. The title compound was prepared according to General Procedure, using acetophenone [98-86-2] (29.1 μL, 0.250 mmol), 1-methylindole [603-76-9] (62.5 μL, 0.500 mmol), and toluene (0.25 mL), with 10 mol% $PhSiCl_3$ (4.0 μL, 0.025 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (90.2 mg, 99% yield).

Spectral data matched those of the previous report.[3]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 – 7.46 (m, 2H), 7.40 (s, 1H), 7.38 (s, 1H), 7.36 (s, 1H), 7.35 – 7.30 (m, 3H), 7.30 – 7.27 (m, 1H), 7.26 – 7.20 (m, 2H), 7.03 – 6.96 (m, 2H), 6.56 (s, 2H), 3.71 (s, 6H), 2.44 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5, 137.9, 128.2, 127.9, 127.0, 125.8, 123.4, 122.3, 121.1, 118.5, 109.3, 43.8, 32.7, 29.3.

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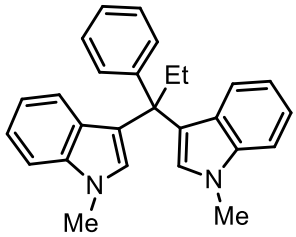
[3] F. Ling, L. Xiao, L. Fang, C. Feng, Z. Xie, Y. Lv and W. Zhong, *Org. Biomol. Chem.*, 2018, **16**, 9274-9278.

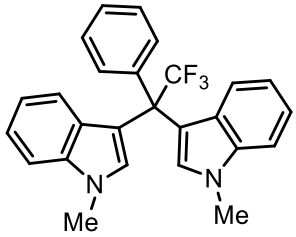


3,3'-(1-Phenylpropane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ac). The title compound was prepared according to General Procedure, using propiophenone [93-55-0] (26.5 μL, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). Reaction temperature 40 °C. After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (54.3 mg, 72% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dt, *J* = 3.3, 1.9 Hz, 2H), 7.35 – 7.27 (m, 4H), 7.25 – 7.10 (m, 5H), 6.94 – 6.88 (m, 2H), 6.81 (s, 2H), 3.72 (s, 6H), 2.83 (q, *J* = 7.3 Hz, 2H), 0.85 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 147.7, 137.7, 128.6, 128.6, 127.7, 127.4, 125.5, 122.6, 121.0, 120.7, 118.3, 109.1, 48.4, 33.4, 32.8, 11.2.

HRMS (ESI) *m*/*z* calcd for **$C_{27}H_{26}N_2$** $[M + H]^+$: 379.2174, found: 379.2052.



3,3'-(2,2,2-Trifluoro-1-phenylethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ad) [1640116-58-0]. The title compound was prepared according to General Procedure, using 2,2,2-trifluoroacetophenone [434-45-7] (28.0 μL, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (66.9 mg, 80% yield).

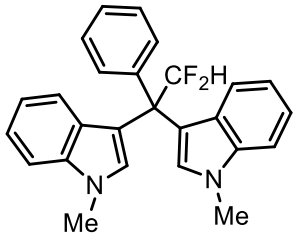
Spectral data matched those of the previous report.[4]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.58 – 7.54 (m, 2H), 7.34 – 7.27 (m, 5H), 7.23 – 7.16 (m, 4H), 6.94 (ddd, *J* = 8.1, 7.0, 1.0 Hz, 2H), 6.75 (s, 2H), 3.73 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 139.74, 137.57, 131.06, 129.73, 128.05, 127.56, 126.97, 122.62, 122.59, 121.61, 119.34, 113.97, 109.33, 33.01.

^{19}F NMR (377 MHz, $CDCl_3$) δ -62.6.

[4] V. K. Pandey and P. Anbarasan, *J. Org. Chem.*, 2017, **82**, 12328-12336.



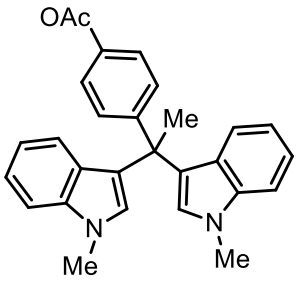
3,3'-(2,2-Difluoro-1-phenylethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ae). The title compound was prepared according to General Procedure, using 2,2-difluoroacetophenone [395-01-7] (26.4 μL, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (77.1 mg, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.50 (m, 2H), 7.35 – 7.28 (m, 5H), 7.23 – 7.14 (m, 4H), 6.94 (t, *J* = 7.6 Hz, 2H), 6.89 (t, *J* = 55.9 Hz, 1H), 6.79 (s, 2H), 3.73 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 141.0, 137.6, 130.3, 129.6, 128.1, 127.2, 122.3, 121.6, 121.4, 119.2, 118.9, 115.0 (t, *J* = 2.7 Hz), 109.4, 53.7, 33.0.

^{19}F NMR (377 MHz, $CDCl_3$) δ -116.5.

HRMS (ESI) *m*/*z* calcd for **$C_{26}H_{22}F_2N_2$** $[M + H]^+$: 401.1829, found: 401.1708.

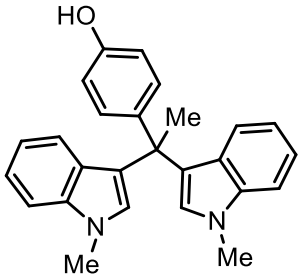


4-(1,1-Bis(1-methyl-1H-indol-3-yl)ethyl)phenyl acetate (Table 2, 3af). The title compound was prepared according to General Procedure, using 4'-acetoxyacetophenone [13031-43-1] (35.6 mg, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (82.6 mg, 98% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 2H), 7.39 – 7.31 (m, 4H), 7.22 (t, *J* = 7.5 Hz, 2H), 7.06 – 6.95 (m, 4H), 6.55 (s, 2H), 3.70 (s, 6H), 2.40 (s, 3H), 2.32 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 148.7, 146.0, 137.9, 129.2, 128.2, 126.8, 123.2, 122.2, 121.2, 120.7, 118.6, 109.3, 43.5, 32.7, 29.4, 21.3.

HRMS (ESI) *m*/*z* calcd for **$C_{28}H_{26}N_2O_2$** $[M + H]^+$: 423.2072, found: 423.1940.

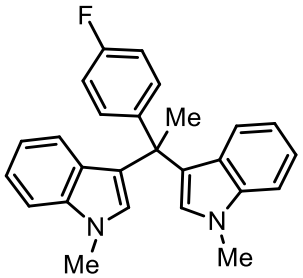


4-(1,1-Bis(1-methyl-1H-indol-3-yl)ethyl)phenol (Table 2, 3ag). The title compound was prepared according to General Procedure, using 4′-hydroxyacetophenone [99-93-4] (27.2 mg, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (1 mg, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (42.0 mg, 55% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 – 7.23 (m, 8H), 7.02 (t, *J* = 7.2 Hz, 2H), 6.73 (d, *J* = 8.3 Hz, 2H), 6.57 (s, 2H), 5.02 (s, 1H), 3.71 (s, 6H), 2.41 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 153.8, 140.6, 137.9, 129.3, 128.2, 126.9, 123.7, 122.3, 121.1, 118.4, 114.7, 109.3, 43.1, 32.7, 29.3.

HRMS (ESI) *m*/*z* calcd for **$C_{26}H_{24}N_2O$** $[M + Na]^+$: 403.1786, found: 403.1657.



3,3'-(1-(4-Fluorophenyl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ah) [1352821-96-5]. The title compound was prepared according to General Procedure, using 4′-fluoroacetophenone [403-42-9] (24.3 μL, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (55.4 mg, 72% yield). Spectral data matched those of the previous report.[5]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 – 7.35 (m, 6H), 7.26 (t, *J* = 7.5 Hz, 2H), 7.07 – 6.97 (m, 4H), 6.57 (s, 2H), 3.74 (s, 6H), 2.43 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 162.8 (s), 144.3 (d, *J* = 3.1 Hz), 137.9 (s), 129.7 (d, *J* = 7.7 Hz), 128.1 (s), 126.8 (s), 123.3 (s), 122.2 (s), 121.3(s), 118.6 (s), 114.5 (d, *J* = 20.8 Hz), 109.3 (s), 43.4 (s), 32.7 (s), 29.4 (s).

^{19}F NMR (282 MHz, $CDCl_3$) δ -118.00 (s).

HRMS (ESI) *m*/*z* calcd for **$C_{26}H_{23}FN_2$** $[M + H]^+$: 383.1923, found: 383.1808.

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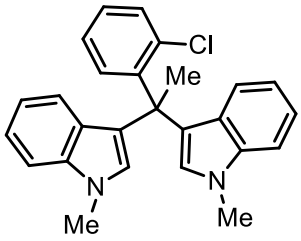
[5] D. Xia, Y. Wang, Z. Du, Q.-Y. Zheng and C. Wang, *Org. Lett.*, 2012, **14**, 588-591.

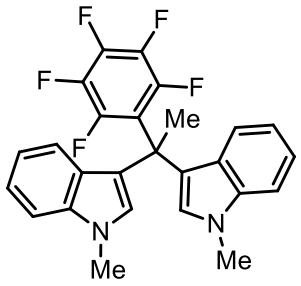


3,3'-(1-(2-Chlorophenyl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ai). The title compound was prepared according to General Procedure, using 2′-chloroacetophenone [2142-68-9] (26.0 μL, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as light green solid (51.3 mg, 64% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 7.6, 1.2 Hz, 1H), 7.35 (d, *J* = 8.2 Hz, 2H), 7.32 – 7.26 (m, 3H), 7.22 (t, *J* = 7.6 Hz, 2H), 7.20 – 7.14 (m, 2H), 6.98 (t, *J* = 7.5 Hz, 2H), 6.61 (s, 2H), 3.73 (s, 6H), 2.59 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 144.8, 137.8, 134.7, 132.1, 131.4, 128.6, 127.8, 127.1, 126.2, 122.02, 121.98, 121.1, 118.6, 109.4, 44.9, 32.8, 27.7.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{26}H_{23}ClN_2}$ $[M + H]^+$: 399.1628, found: 399.1629.



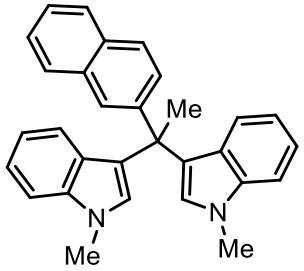
3,3'-(1-(Perfluorophenyl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3aj). The title compound was prepared according to General Procedure, using 2′,3′,4′,5′,6′-pentafluoroacetophenone [652-29-9] (28.5 μL, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (40.8 mg, 45% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.1 Hz, 2H), 7.39 (d, *J* = 8.2 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 2H), 6.71 (s, 2H), 3.75 (s, 6H), 2.57 (t, *J* = 2.7 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 147.4, 144.8, 139.2, 137.9, 136.7, 127.2, 126.3, 121.5, 120.9, 120.5, 118.9, 109.7, 42.7, 32.8 (d, *J* = 3.0 Hz), 29.7.

^{19}F NMR (377 MHz, $CDCl_3$) δ -135.1, -136.6 (m), -157.4 (t, *J* = 21.3 Hz), -162.5 (dt, *J* = 21.8, 6.5 Hz).

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{26}H_{19}F_5N_2}$ $[M + H]^+$: 455.1546, found: 455.1405.

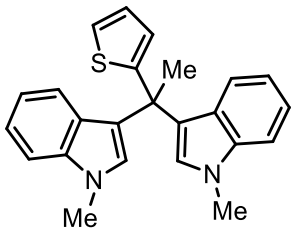


3,3'-(1-(Naphthalen-2-yl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ak). The title compound was prepared according to General Procedure, using 2-acetonaphthone [93-08-3] (34.0 mg, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (78.0 mg, 94% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.07 – 8.05 (m, 1H), 7.92 – 7.79 (m, 3H), 7.68 – 7.63 (m, 1H), 7.55 – 7.48 (m, 4H), 7.41 (d, *J* = 8.2 Hz, 2H), 7.32 – 7.26 (m, 2H), 7.07 – 7.00 (m, 2H), 6.64 (s, 2H), 3.74 (s, 6H), 2.60 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 146.1, 137.9, 133.4, 132.1, 128.5, 128.3, 127.7, 127.5, 127.3, 126.9, 125.9, 125.6, 125.4, 123.2, 122.3, 121.2, 118.6, 109.3, 44.0, 32.7, 29.1.

HRMS (ESI) *m*/*z* calcd for **$C_{30}H_{26}N_2$** $[M + H]^+$: 415.2174, found: 415.2037.

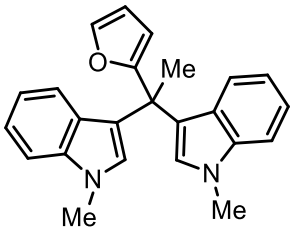


3,3'-(1-(Thiophen-2-yl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3al). The title compound was prepared according to General Procedure, using 2-acetylthiophene [88-15-3] (21.6 μL, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (72.6 mg, 98% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.2 Hz, 2H), 7.29 – 7.23 (m, 3H), 7.07 – 7.02 (m, 2H), 7.00 (d, *J* = 3.2 Hz, 2H), 6.70 (s, 2H), 3.75 (s, 6H), 2.51 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 155.0, 137.8, 127.8, 126.6, 126.1, 125.0, 123.6, 123.2, 122.0, 121.2, 118.6, 109.3, 41.9, 32.8, 30.8.

HRMS (ESI) *m*/*z* calcd for **$C_{24}H_{22}N_2S$** $[M + H]^+$: 371.1582, found: 371.1463.

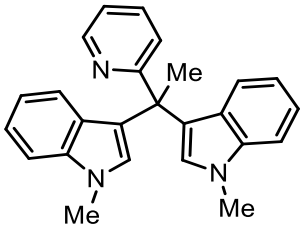


3,3'-(1-(Furan-2-yl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3am). The title compound was prepared according to General Procedure, using 2-acetylfuran [1192-62-7] (22.0 mg, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: diethyl ether in hexane), the title compound was isolated as an off-white solid (40.0 mg, 57% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 – 7.39 (m, 3H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.24 (t, *J* = 7.2 Hz, 2H), 7.02 (t, *J* = 7.0 Hz, 2H), 6.73 (s, 2H), 6.39 – 6.34 (m, 1H), 6.18 (d, *J* = 2.4 Hz, 1H), 3.75 (s, 6H), 2.35 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 161.1, 141.1, 137.7, 127.4, 126.7, 121.8, 121.2, 120.9, 118.6, 110.0, 109.3, 106.1, 40.4, 32.8, 27.5.

HRMS (ESI) *m*/*z* calcd for **$C_{24}H_{22}N_2O$** $[M + Na]^+$: 377.1630, found: 377.1608.

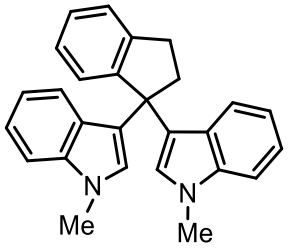


3,3'-(1-(Pyridin-2-yl)ethane-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3an). The title compound was prepared according to General Procedure, using 2-acetylpyridine [1122-62-9] (22.4 μL, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (45.9 mg, 63% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69 – 8.65 (m, 1H), 7.52 (td, *J* = 7.8, 1.9 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.31 (d, *J* = 7.7 Hz, 4H), 7.22 – 7.16 (m, 2H), 7.14 – 7.09 (m, 1H), 6.98 – 6.92 (m, 2H), 6.64 (s, 2H), 3.70 (s, 6H), 2.44 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 166.5, 148.9, 137.8, 135.9, 128.1, 126.8, 123.3, 122.1, 121.9, 121.1, 121.0, 118.5, 109.3, 46.3, 32.8, 28.1.

HRMS (ESI) *m*/*z* calcd for **$C_{25}H_{23}N_3$** $[M + H]^+$: 366.1970, found: 366.1863.

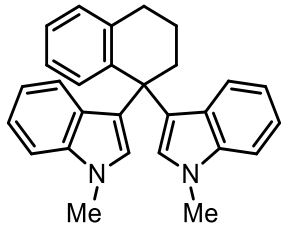


3,3'-(2,3-Dihydro-1H-indene-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ao). The title compound was prepared according to General Procedure, using 1-indanone [83-33-0] (26.4 mg, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). Reaction temperature 40 °C. After purification by flash column chromatography (eluent: diethyl ether in hexane), the title compound was isolated as a white solid (70.9 mg, 94% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 – 7.30 (m, 6H), 7.30 – 7.17 (m, 3H), 7.14 (t, *J* = 7.5 Hz, 1H), 7.02 (t, *J* = 7.4 Hz, 2H), 6.55 (s, 2H), 3.69 (s, 6H), 3.17 – 2.98 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 150.0, 143.5, 138.1, 128.5, 126.9, 126.6, 126.3, 125.5, 124.8, 121.8, 121.2, 120.9, 118.4, 109.3, 51.7, 40.8, 32.7, 30.9.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{27}H_{24}N_2}$ $[M + H]^+$: 377.2017, found: 377.1907.

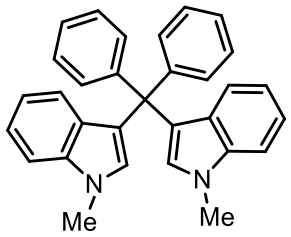


3,3'-(1,2,3,4-Tetrahydronaphthalene-1,1-diyl)bis(1-methyl-1H-indole) (Table 2, 3ap). The title compound was prepared according to General Procedure, using α-tetralone [629-34-0] (26.6 μL, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (71.5 mg, 92% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 7.8 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.29 – 7.16 (m, 4H), 7.10 – 6.96 (m, 4H), 6.47 (s, 2H), 3.70 (s, 6H), 3.05 (t, *J* = 6.0 Hz, 2H), 2.95 – 2.84 (m, 2H), 1.72 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 143.1, 138.0, 136.8, 130.6, 130.1, 129.0, 126.4, 125.9, 125.4, 122.8, 122.1, 121.1, 118.3, 109.3, 44.3, 35.3, 32.6, 30.0, 20.1.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{28}H_{26}N_2}$ $[M + H]^+$: 391.2174, found: 391.2058.

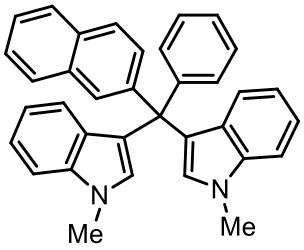


Bis(1-methyl-1H-indol-3-yl)diphenylmethane (Scheme 2, 3ab). The title compound was prepared according to General Procedure, using benzophenone [119-61-9] (38.8 mg, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: diethyl ether in hexane), the title compound was isolated as a white solid (48.0 mg, 56% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.34 (m, 4H), 7.30 (d, *J* = 8.3 Hz, 3H), 7.27 – 7.23 (m, 5H), 7.17 – 7.13 (m, 2H), 6.81 (d, *J* = 3.9 Hz, 4H), 6.73 (s, 2H), 3.72 (s, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 146.4, 137.7, 130.3, 129.7, 128.3, 127.5, 126.0, 122.8, 122.6, 121.1, 118.6, 109.0, 53.9, 32.8.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{31}H_{26}N_2}$ $[M + H]^+$: 427.2174, found: 427.2043.

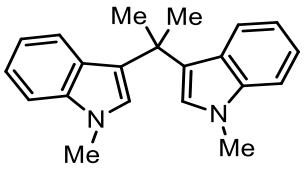


3,3'-(Naphthalen-2-yl(phenyl)methylene)bis(1-methyl-1H-indole) (Scheme 2, 3aq). The title compound was prepared according to General Procedure, using 2-benzoylnaphthalene [644-13-3] (46.4 mg, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). After purification by flash column chromatography (eluent: diethyl ether in hexane), the title compound was isolated as red to yellow solid (59.8 mg, 63% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (s, 1H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.50 – 7.38 (m, 5H), 7.33 – 7.22 (m, 5H), 7.14 (t, *J* = 7.0 Hz, 2H), 6.90 – 6.73 (m, 6H), 3.73 (s, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 146.1, 144.1, 137.6, 133.1, 132.0, 130.3, 129.8, 129.7, 128.6, 128.2, 127.7, 127.5, 127.3, 126.6, 126.0, 125.6, 125.5, 122.6, 122.1, 121.1, 118.6, 109.0, 54.0, 32.8.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{35}H_{28}N_2}$ $[M + H]^+$: 477.2330, found: 477.2183.

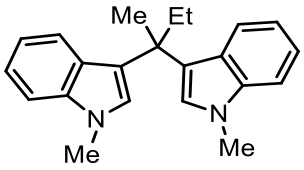


3,3'-(Propane-2,2-diyl)bis(1-methyl-1H-indole) (Scheme 2, 3ar) [94861-81-1]. The title compound was prepared according to General Procedure, using 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), with 10 mol% $B(C_6F_5)_3$ (10 mg, 0.020 mmol). Acetone (0.20 mL) instead of toluene. Reaction temperature 40 °C. After purification by flash column chromatography (eluent: EtOAc in hexane) and washed by hexane, the title compound was isolated as a white solid (47.1 mg, 78% yield).

Spectral data matched those of the previous report.[6]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.0 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.17 – 7.10 (m, 2H), 6.91 (ddd, *J* = 8.0, 7.0, 1.0 Hz, 2H), 6.89 (s, 2H), 3.76 (s, 6H), 1.92 (s, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 137.9, 126.8, 125.6, 124.2, 121.6, 121.0, 118.2, 109.2, 35.1, 32.8, 30.5.



3,3'-(Butane-2,2-diyl)bis(1-methyl-1H-indole) (Scheme 2, 3as). The title compound was prepared according to General Procedure, using 2-butanone [78-93-3] (27.2 mg, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). Reaction temperature 40 ℃. After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (28.9 mg, 46% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 7.16 (t, *J* = 7.5 Hz, 2H), 6.95 (s, 2H), 6.91 (t, *J* = 7.5 Hz, 2H), 3.79 (s, 6H), 2.46 (q, *J* = 7.4 Hz, 2H), 1.86 (s, 3H), 0.82 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.8, 127.0, 126.4, 122.9, 121.6, 120.9, 118.1, 109.1, 38.9, 33.1, 32.8, 26.8, 9.3.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{22}H_{24}N_2}$ $[M + H]^+$: 317.2017, found: 317.1918.

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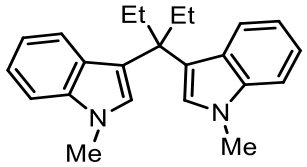
[6] C. Huo, L. Kang, X. Xu, X. Jia, X. Wang, H. Xie and Y. Yuan, *Tetrahedron Lett.*, 2014, **55**, 954-958.

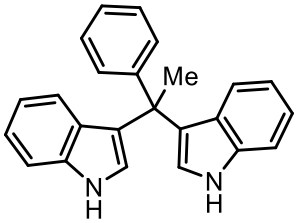


3,3'-(Pentane-3,3-diyl)bis(1-methyl-1H-indole) (Scheme 2, 3at) [1042375-24-5]. The title compound was prepared according to General Procedure, using 3-pentanone [96-22-0] (21.1 μL, 0.200 mmol), 1-methylindole [603-76-9] (99.8 μL, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol) at 40 °C. After purification by flash column chromatography (eluent: diethyl ether in hexane), the title compound was isolated as a white solid (54.8 mg, 83% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.23 (m, 4H), 7.08 (dt, *J* = 13.2, 3.0 Hz, 2H), 7.04 (s, 2H), 6.83 – 6.76 (m, 2H), 3.80 (s, 6H), 2.33 (q, *J* = 7.3 Hz, 4H), 0.72 (t, *J* = 7.3 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 137.8, 127.3, 127.0, 121.5, 121.4, 120.8, 117.9, 108.9, 42.2, 32.9, 28.5, 8.6.

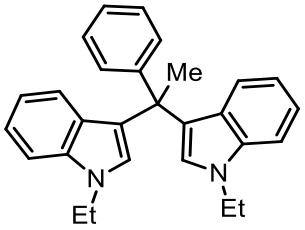
HRMS (ESI) *m*/*z* calcd for **$C_{23}H_{26}N_2$** $[M + H]^+$: 331.2174, found: 331.2075.



3,3'-(1-phenylethane-1,1-diyl)bis(1H-indole) (Scheme 3, 3ba) [96413-90-0]. The title compound was prepared according to General Procedure, using acetophenone [98-86-2] (23.3 μL, 0.200 mmol), indole [120-72-9] (93.7mg, 0.800 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). 36% of the title compound was produced, as determined by ^{1}H NMR analysis of the crude mixture with 1,1,2,2-tetrachloroehtane as an internal standard. After purification by flash column chromatography (eluent: ethyl acetate in hexane), the title compound was isolated as an off-white solid (20.1 mg, 30% yield). Spectral data matched those of the previous report.[7]

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (s, 2H), 7.44 (d, *J* = 6.8 Hz, 2H), 7.37 (d, *J* = 8.3 Hz, 4H), 7.28 (dd, *J* = 15.4, 8.0 Hz, 3H), 7.18 (t, *J* = 7.4 Hz, 2H), 6.98 (t, *J* = 7.6 Hz, 2H), 6.63 (s, 2H), 2.41 (s, 3H).

7 J. Nie, G.-W. Zhang, L. Wang, A. Fu, Y. Zheng and J.-A. Ma, *Chem. Commun.*, 2009, 2356-2358.

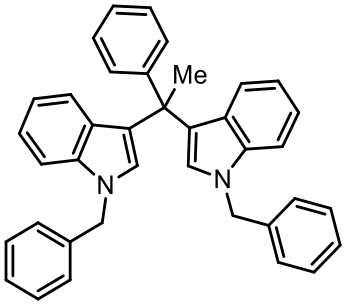


3,3'-(1-Phenylethane-1,1-diyl)bis(1-ethyl-1H-indole) (Scheme 3, 3ca) [791836-02-7]. The title compound was prepared according to General Procedure, using acetophenone [98-86-2] (23.3 μL, 0.200 mmol), 1-ethylindole [10604-59-8] (58.0mg, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). 76% of the title compound was produced, as determined by ^1H NMR analysis of the crude mixture with 1,1,2,2-tetrachloroehtane as an internal standard. After purification by flash column chromatography (eluent: diethyl ether in hexane), the title compound was isolated as an off-white solid (30.0 mg, 38% yield).

^1H NMR (400 MHz, $CDCl_3$) δ 7.49 (d, *J* = 7.5 Hz, 2H), 7.39 (d, *J* = 8.5 Hz, 4H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 (d, *J* = 7.7 Hz, 1H), 7.22 (t, *J* = 7.8 Hz, 2H), 6.99 (t, *J* = 7.5 Hz, 2H), 6.63 (s, 2H), 4.12 (q, *J* = 7.2 Hz, 4H), 2.44 (s, 3H), 1.44 (t, *J* = 7.2 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 148.5, 136.9, 128.2, 127.8, 127.2, 126.7, 125.8, 123.5, 122.4, 121.0, 118.3, 109.3, 43.9, 40.9, 29.2, 15.6.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{28}H_{28}N_2}$ $[M + H]^+$: 393.2330, found: 393.2217.



3,3'-(1-Phenylethane-1,1-diyl)bis(1-benzyl-1H-indole) (Scheme 3, 3da) [2166321-60-2]. The title compound was prepared according to General Procedure, using acetophenone [98-86-2] (23.3 μL, 0.200 mmol), 1-benzylindole [3377-71-7] (82.9 mg, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a white solid (91.2 mg, 88% yield). Spectral data matched those of the previous report.[8]

^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, *J* = 7.2 Hz, 2H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.54 – 7.49 (m, 6H), 7.48 – 7.43 (m, 5H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.29 (d, *J* = 7.2 Hz, 4H), 7.19 – 7.13 (m, 2H), 6.97 (s, 2H), 5.44 (s, 4H), 2.66 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 148.1, 138.0, 137.5, 128.7, 128.2, 127.9, 127.8, 127.5, 127.3, 126.5, 125.9, 123.8, 122.4, 121.4, 118.7, 109.9, 49.9, 43.9, 29.4.

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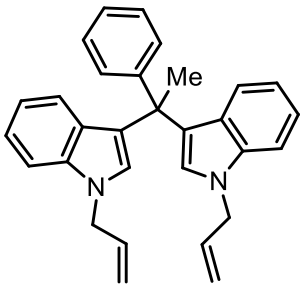
[8] F. Ling, L. Xiao, L. Fang, C. Feng, Z. Xie, Y. Lv and W. Zhong, *Org. Biomol. Chem.*, 2018, **16**, 9274-9278.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{38}H_{32}N_2}$ [M + H]$^+$: 517.2643, found: 517.2469.

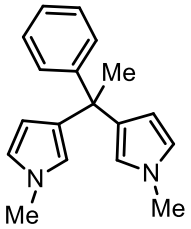


3,3'-(1-Phenylethane-1,1-diyl)bis(1-allyl-1H-indole) (Scheme 3, 3ea) [2254296-56-3]. The title compound was prepared according to General Procedure, using acetophenone [98-86-2] (23.3 μL, 0.200 mmol), 1-allylindole [16886-08-1] (62.8 mg, 0.400 mmol), and toluene (0.20 mL), with 10 mol% $PhSiCl_3$ (3.2 μL, 0.020 mmol). Reaction temperature 40 ℃. After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (65.8 mg, 79% yield). Spectral data matched those of the previous report.[9]

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.4 Hz, 2H), 7.42 (d, *J* = 8.1 Hz, 2H), 7.39 – 7.34 (m, 4H), 7.32 – 7.27 (m, 1H), 7.24 (t, *J* = 7.6 Hz, 2H), 7.02 (t, *J* = 7.5 Hz, 2H), 6.68 (s, 2H), 6.08 – 5.97 (m, 2H), 5.27 – 5.21 (m, 2H), 5.14 – 5.07 (m, 2H), 4.70 (d, *J* = 5.1 Hz, 4H), 2.48 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 148.3, 137.3, 133.8 (d, *J* = 4.8 Hz), 128.2, 127.9, 127.3, 127.2, 125.8, 123.6, 122.4, 121.2, 118.6, 116.8, 109.7, 48.7, 43.8, 29.3.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{30}H_{28}N_2}$ [M + H]$^+$: 417.2330, found: 417.2205.



3,3'-(1-Phenylethane-1,1-diyl)bis(1-methyl-1H-pyrrole) (Scheme 3, 3fa) [1167441-87-3]. The title compound was prepared according to General Procedure, using acetophenone [98-86-2] (23.3 μL, 0.200 mmol), 1-methylpyrrole [96-54-8] (71.0 μL, 0.800 mmol), and toluene (0.20 mL), with 20 mol% $B(C_6F_5)_3$ (20.4 mg, 0.040 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as an off-white solid (51.4 mg, 97% yield).

Spectral data matched those of the previous report.[10]

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[9] F. Ling, L. Xiao, L. Fang, C. Feng, Z. Xie, Y. Lv and W. Zhong, *Org. Biomol. Chem.*, 2018, **16**, 9274-9278.

[10] T. Tsuchimoto, T. Ainoya, K. Aoki, T. Wagatsuma and E. Shirakawa, *Eur. J. Org. Chem.*, 2009, **2009**, 2437-2440.

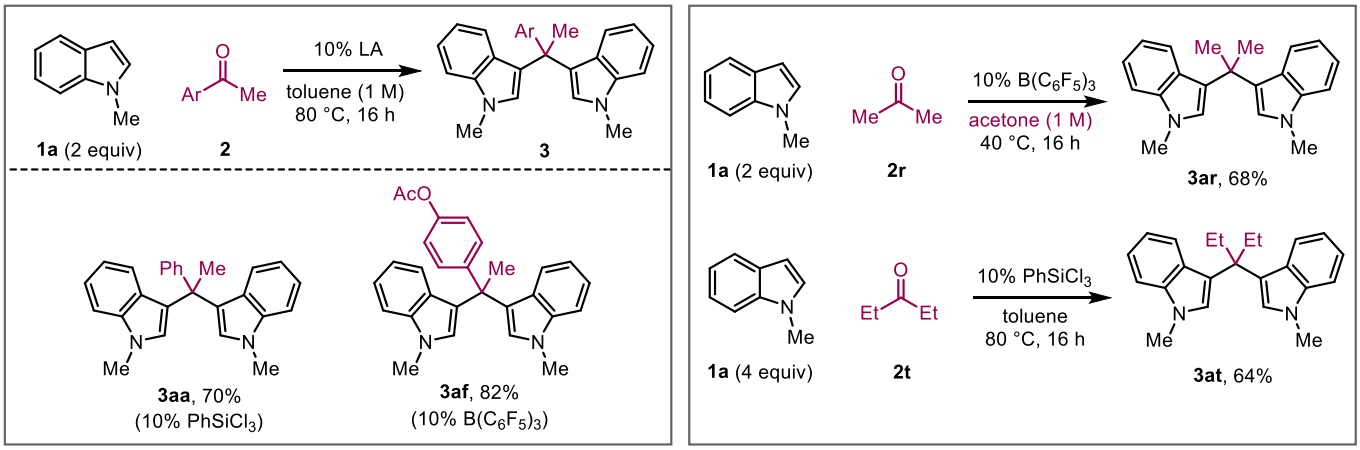
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.38 (m, 2H), 7.35 – 7.30 (m, 2H), 7.26 – 7.21 (m, 1H), 6.61 (s, 2H), 6.27 (s, 2H), 6.05 (s, 2H), 3.65 (s, 6H), 2.05 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 151.3, 134.3, 127.8, 127.5, 125.3, 121.3, 120.1, 108.4, 42.9, 36.1, 30.8.

HRMS (ESI) *m*/*z* calcd for **$C_{18}H_{20}N_2$** $[M + H]^+$: 265.1704, found: 265.1633.

Glovebox-Free Procedure.

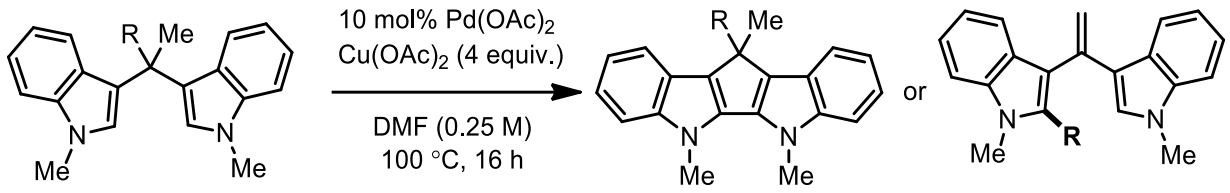
A Lewis acid catalyst (10 mol% of $B(C_6F_5)_3$ (2.5 mg) or $PhSiCl_3$ (0.80 μL)) was placed in a 20-mL vial equipped with a stir bar. This vial was capped with a septum-lined cap, and then it was evacuated and backfilled with nitrogen (three cycles). Toluene (1 M, 0.2 mL) (acetone 0.2 mL instead of toluene for the synthesis of **3ar**) was added via syringe; next, ketone (0.2 mmol, 1 equiv) , 1-methylindole (0.4–0.8 mmol, 2–4 equiv) were added in turn via syringe. The resulting mixture was stirred at 80 ℃ overnight. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography. The yield of the product was calculated based on the amount of ketone. The yield of the product was calculated based on the amount of ketone.



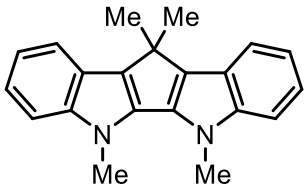
IV. Pd-Catalyzed Coupling Reactions of Bis(indolyl)methanes

General Procedure for Pd-Catalyzed Coupling Reactions of Bis(indolyl)methanes

Scheme 5 of the main paper



In a nitrogen-filled glovebox, bis(indolyl)methane product (0.200 mmol, 1.0 equiv.), palladium(II) acetate (0.020 mmol, 0.1 equiv.), copper(II) acetate (0.800 mmol, 4.0 equiv.) and DMF (0.25 M, 0.8 mL) were combined in a 20-mL vial equipped with a stir bar. The resulting mixture was stirred at 100 °C for 16 h. The mixture then went through Celite filter and work-up step. The solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography.

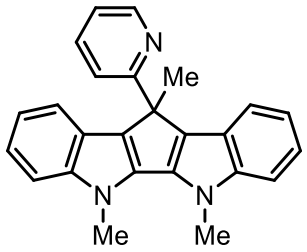


5,6,11,11-Tetramethyl-6,11-dihydro-5H-cyclopenta[1,2-b:5,4-b']diindole (Scheme 5, 6ar). The title compound was prepared according to General Procedure, using **3ar** (60.4 mg, 0.200 mmol) and palladium(II) acetate [3375-31-3] (4.4 mg, 0.020 mmol), with copper(II) acetate [142-71-2] (145 mg, 0.800 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a yellow solid (24.0 mg, 40% yield.)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (s, 2H), 7.41 (d, *J* = 4.6 Hz, 2H), 7.23 (s, 4H), 4.12 (s, 6H), 1.75 (s, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 136.2, 120.3, 119.9, 117.7, 110.1, 53.5, 32.9, 26.1.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{21}H_{20}N_2}$ $[M]^+$: 300.1626, found: 300.1650.

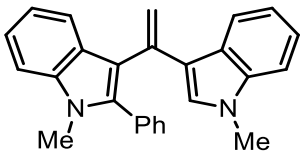


5,6,11-Trimethyl-11-(pyridin-2-yl)-6,11-dihydro-5H-cyclopenta[1,2-b:5,4-b']diindole (Scheme 5, 6an). The title compound was prepared according to General Procedure, using **3an** (73.2 mg, 0.200 mmol) and palladium(II) acetate [3375-31-3] (4.4 mg, 0.020 mmol), with copper(II) acetate [142-71-2] (145 mg, 0.800 mmol). After purification by flash column chromatography (eluent: Hex, EtOAc), the title compound was isolated as a yellow solid (14.5 mg, 20% yield.)

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.90 (d, *J* = 5.3 Hz, 1H), 7.68 (d, *J* = 7.2 Hz, 1H), 7.40 (d, *J* = 7.9 Hz, 1H), 7.25 – 7.11 (m, 2H), 4.18 (s, 2H), 2.34 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 162.2, 159.3, 141.1, 137.3, 132.2, 126.4, 123.4, 122.7, 122.2, 121.3, 120.8, 118.1, 110.1, 33.0, 29.6, 22.7.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{21}H_{20}N_2}$ $[M + H]^+$: 364.1813, found: 364.1828.

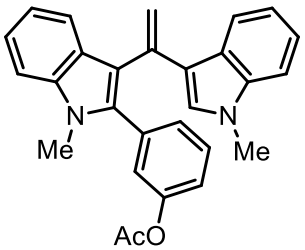


1-methyl-3-(1-(1-methyl-1H-indol-3-yl)vinyl)-2-phenyl-1H-indole (Scheme 5, 7aa). the title compound was prepared according to General Procedure, using **3aa** (72.8 mg, 0.200 mmol) and palladium(II) acetate [3375-31-3] (4.4 mg, 0.020 mmol), with copper(II) acetate [142-71-2] (145 mg, 0.800 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a yellow sticky liquid (17.4 mg, 24% yield.)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 – 7.66 (m, 1H), 7.47 – 7.41 (m, 3H), 7.36 – 7.29 (m, 3H), 7.27 – 7.23 (m, 3H), 7.21 – 7.16 (m, 2H), 7.12 – 7.06 (m, 1H), 6.64 (d, *J* = 0.6 Hz, 1H), 5.51 (d, *J* = 1.6 Hz, 1H), 5.14 (d, *J* = 1.6 Hz, 1H), 3.72 (s, 3H), 3.61 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 142.0, 141.3, 137.6, 137.5, 130.5, 129.9, 128.0, 127.7, 127.5, 127.3, 127.0, 122.6, 122.0, 121.1, 120.7, 120.0, 119.8, 117.9, 115.8, 109.6, 109.6, 105.6, 30.8, 30.6.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{26}H_{22}N_2}$ $[M + H]^+$: 363.1861, found: 363.1875.



3-(1-Methyl-3-(1-(1-methyl-1H-indol-3-yl)vinyl)-1H-indol-2-yl)phenyl acetate (Scheme 5, 7af). The title compound was prepared according to General Procedure, using **3af** (84.9 mg, 0.200 mmol) and palladium(II) acetate [3375-31-3] (4.4 mg, 0.020 mmol), with copper(II) acetate

[142-71-2] (145 mg, 0.800 mmol). After purification by flash column chromatography (eluent: EtOAc in hexane), the title compound was isolated as a yellow solid (33.6 mg, 40% yield.)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, *J* = 7.8 Hz, 1H), 7.42 (t, *J* = 8.4 Hz, 3H), 7.36 – 7.27 (m, 3H), 7.23 – 7.14 (m, 2H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 8.5 Hz, 2H), 6.64 (s, 1H), 5.49 (d, *J* = 6.2 Hz, 1H), 5.12 (d, *J* = 5.6 Hz, 1H), 3.72 (s, 3H), 3.60 (s, 3H), 2.31 (s, 3H).

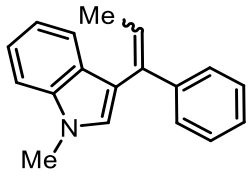
^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 150.2, 141.1, 139.1, 137.7, 137.6, 130.4, 130.1, 128.3, 127.8, 127.0, 122.7, 122.2, 121.1, 120.9, 120.2, 119.9, 117.7, 116.1, 109.7, 105.7, 30.9, 30.7, 21.3.

HRMS (ESI) *m*/*z* calcd for **$C_{28}H_{24}N_2O_2$** $[M + H]^+$: 421.1916, found: 421.1922.

V. Reactivity Studies: Isolation of an Alkene Side Product

Procedure for Scheme 4 (a) of the Main Paper. *Formation of an Alkene*

In a nitrogen-filled glovebox, propiophenone [93-55-0] (26.6 μL, 0.200 mmol), 1-methylindole [603-76-9] (49.9 μL, 0.400 mmol), Lewis acid catalyst (0.200 mmol, 10 mol% of $B(C_6F_5)_3$) and toluene (1 M) were combined in 4-mL vial equipped with a stir bar. The resulting mixture was stirred at 80 ℃ for 16 h. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography (eluent: Hex, EtOAc), the title compound was isolated as a brown sticky liquid (26.2 mg, 53% yield.).



(*E*)-1-Methyl-3-(1-phenylprop-1-en-1-yl)-1H-indole and (*Z*)-1-methyl-3-(1-phenylprop-1-en-1-yl)-1H-indole (1.5:1) (Scheme 4, 5). The title compound was isolated as an *E*/*Z* mixture.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.0 Hz, 0.66H), 7.64 – 7.53 (m, 6.35H), 7.51 – 7.41 (m, 6.34H), 7.37 – 7.32 (m, 0.66H), 7.27 (t, *J* = 7.3 Hz, 1H), 7.18 (s, 1H), 6.89 (s, 0.66H), 6.55 (q, *J* = 7.0 Hz, 0.66H), 6.47 (q, *J* = 6.9 Hz, 1H), 3.94 (s, 3H), 3.79 (s, 2H), 2.13 (d, *J* = 7.0 Hz, 3H), 2.08 (d, *J* = 7.0 Hz, 2H). * The *N*-methyl protons of the 1.5 ratio form were set to 3 protons.

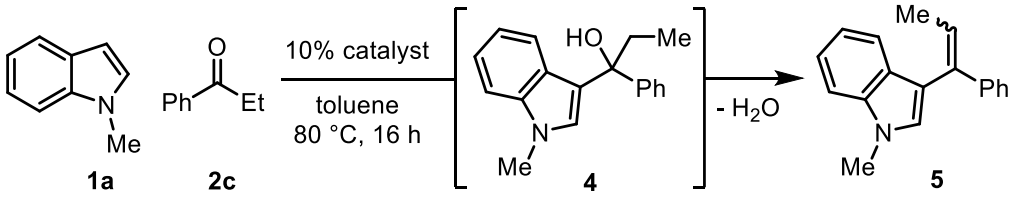
^{13}C NMR (101 MHz, $CDCl_3$) δ 144.0, 141.4, 137.8, 137.1, 136.8, 135.5, 130.1, 129.1, 128.5, 128.3 (t, *J* = 5.7 Hz), 127.5, 126.9 (d, *J* = 9.4 Hz), 126.5, 125.0, 122.0, 121.8, 121.1, 120.7, 119.8, 119.4, 118.9, 109.6, 109.4, 32.9, 16.6, 15.5.

HRMS (ESI) *m*/*z* calcd for $\mathbf{C_{18}H_{17}N}$ $[M + H]^+$: 248.1439, found: 248.1367.

Procedure for Table S3

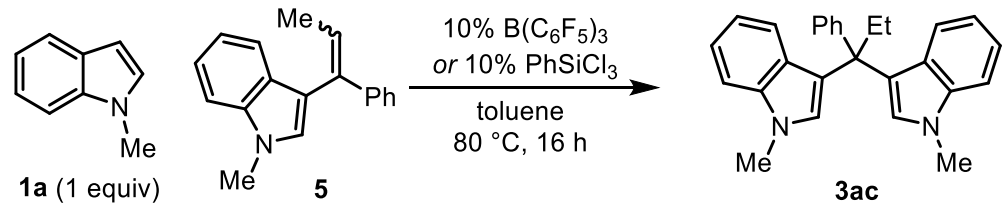
In a nitrogen-filled glovebox, propiophenone (0.050 mmol, 1.0 equiv.), 1-methylindole (1.0-3.0 equiv.), Lewis acid catalyst (10 mol% of $B(C_6F_5)_3$ or $PhSiCl_3$) and toluene (1 M) were combined in 4-mL vial equipped with a stir bar. The resulting mixture was stirred at 80 ℃ overnight. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The yield of the product was determined by ^1H NMR spectroscopy with 1,1,2,2-tetrachloroethane as an internal standard.

Table S4. *Alkene Formation: effect of catalysts and equivalents of indole*



entry	catalyst	indole equiv.	pdt : alkene a : alkene b
a	$B(C_6F_5)_3$	3	29 : 24 : 16
b	$B(C_6F_5)_3$	2	18 : **32** : **21**
c	$B(C_6F_5)_3$	1	<5 : 30 : 20
d	$PhSiCl_3$	3	80 : 12 : 8
e	$PhSiCl_3$	2	22 : 26 : 18
f	$PhSiCl_3$	1	10 : 34 : 22

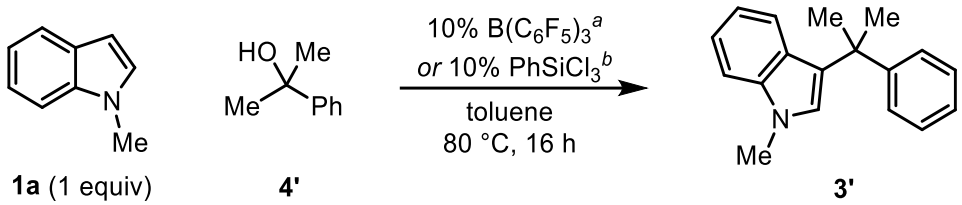
Procedure for Scheme 4(b)



In a nitrogen-filled glovebox, **5** (0.050 mmol, 1.0 equiv.), 1-methylindole (0.050 mmol, 1.0 equiv.), Lewis acid catalyst (10 mol% of $B(C_6F_5)_3$ or $PhSiCl_3$) and toluene (1 M) were combined in 4-mL vial equipped with a stir bar. The resulting mixture was stirred at 80 ℃ overnight. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The yield of the product was determined by ^1H NMR spectroscopy with 1,1,2,2-tetrachloroethane as an internal standard.

VI. Reactivity Studies: Addition of Indole to an Alcohol

Procedure for Scheme 4(c)



1a (1 equiv) **4'** **3'**

In a nitrogen-filled glovebox, 2-phenyl-2-propanol (0.050 mmol, 1.0 equiv.), 1-methylindole (0.050 mmol, 1.0 equiv.), Lewis acid catalyst (10 mol% of $B(C_6F_5)_3$ or $PhSiCl_3$) and toluene (1 M) were combined in 4-mL vial equipped with a stir bar. The resulting mixture was stirred at 80 ℃ overnight. The mixture was then exposed to air, and the solvent was evaporated under reduced pressure. The yield of the product was determined by ^1H NMR spectroscopy with 1,1,2,2-tetrachloroethane as an internal standard.

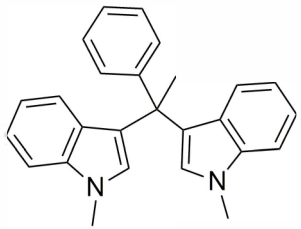


Table 2, 3aa

^{1}H NMR (300 MHz, $CDCl_3$)

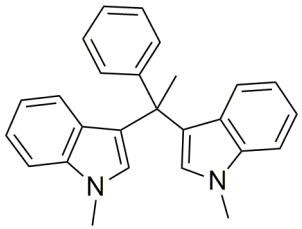


Table 2, 3aa

^{13}C NMR (75 MHz, $CDCl_3$)

148.48
137.88
128.20
127.86
126.95
125.81
123.44
122.29
121.14
118.47
109.25
43.79
32.71
29.26

S28

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

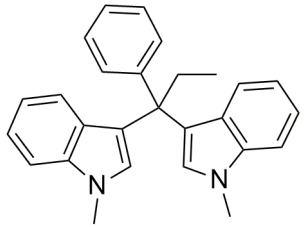


Table 2, 3ac

^{1}H NMR (400 MHz, $CDCl_3$)

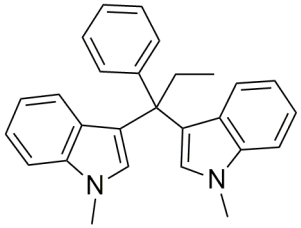


Table 2, 3ac

^{13}C NMR (101 MHz, $CDCl_3$)

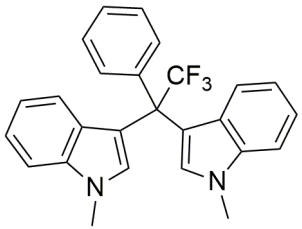


Table 2, 3ad

^{1}H NMR (400 MHz, $CDCl_3$)

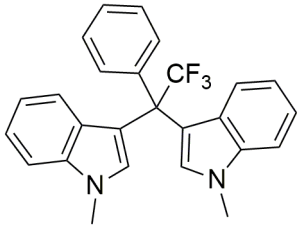


Table 2, 3ad

^{13}C NMR (101 MHz, $CDCl_3$)

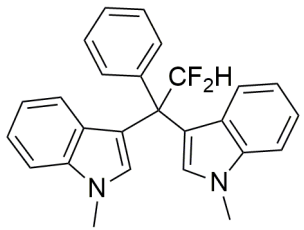


Table 2, 3ae

^{1}H NMR (400 MHz, $CDCl_3$)

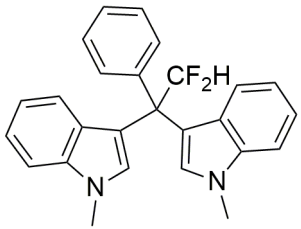


Table 2, 3ae

^{13}C NMR (101 MHz, $CDCl_3$)

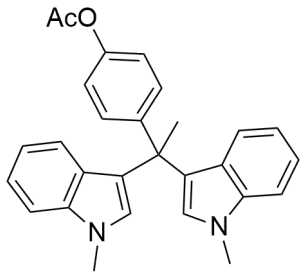
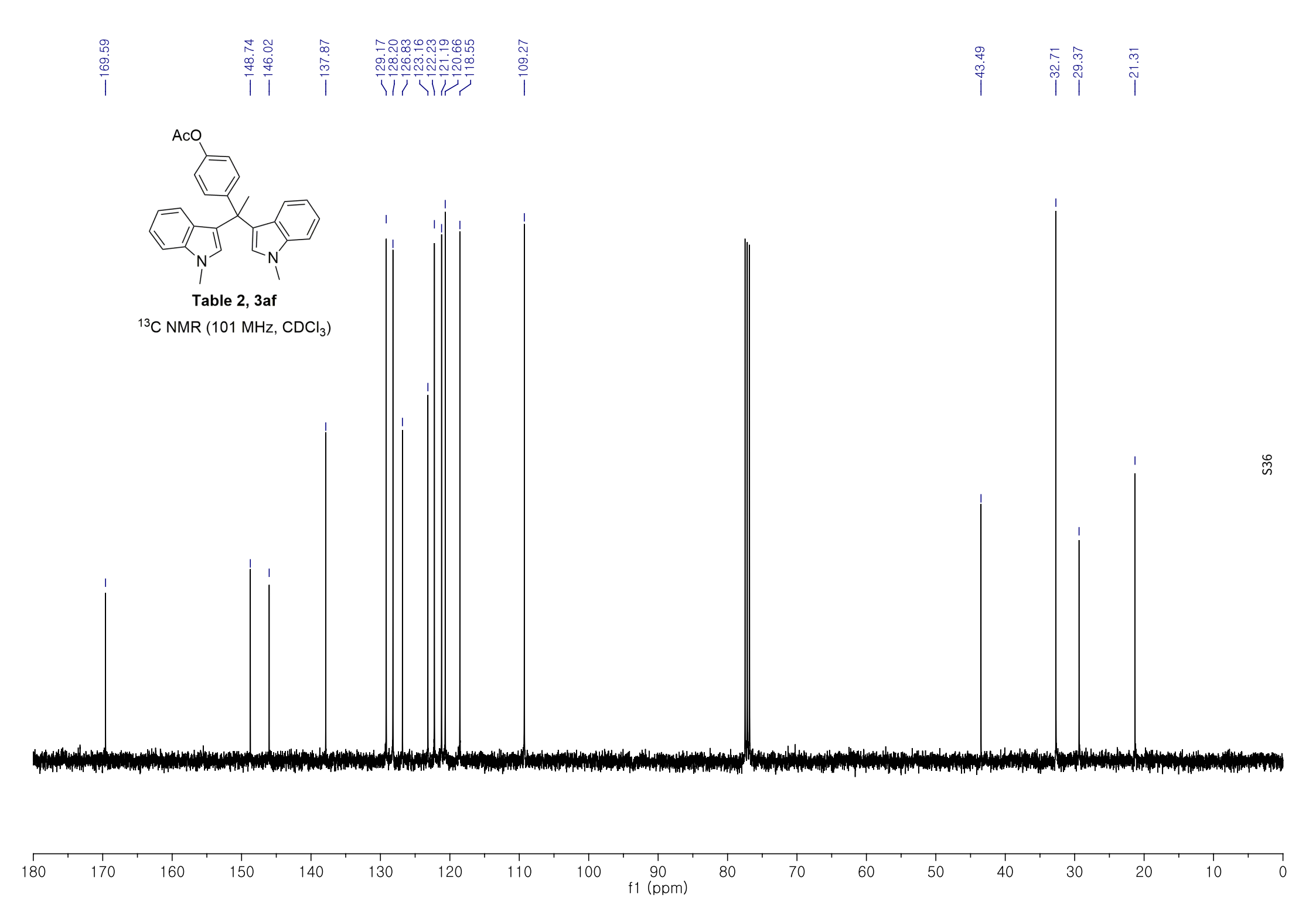
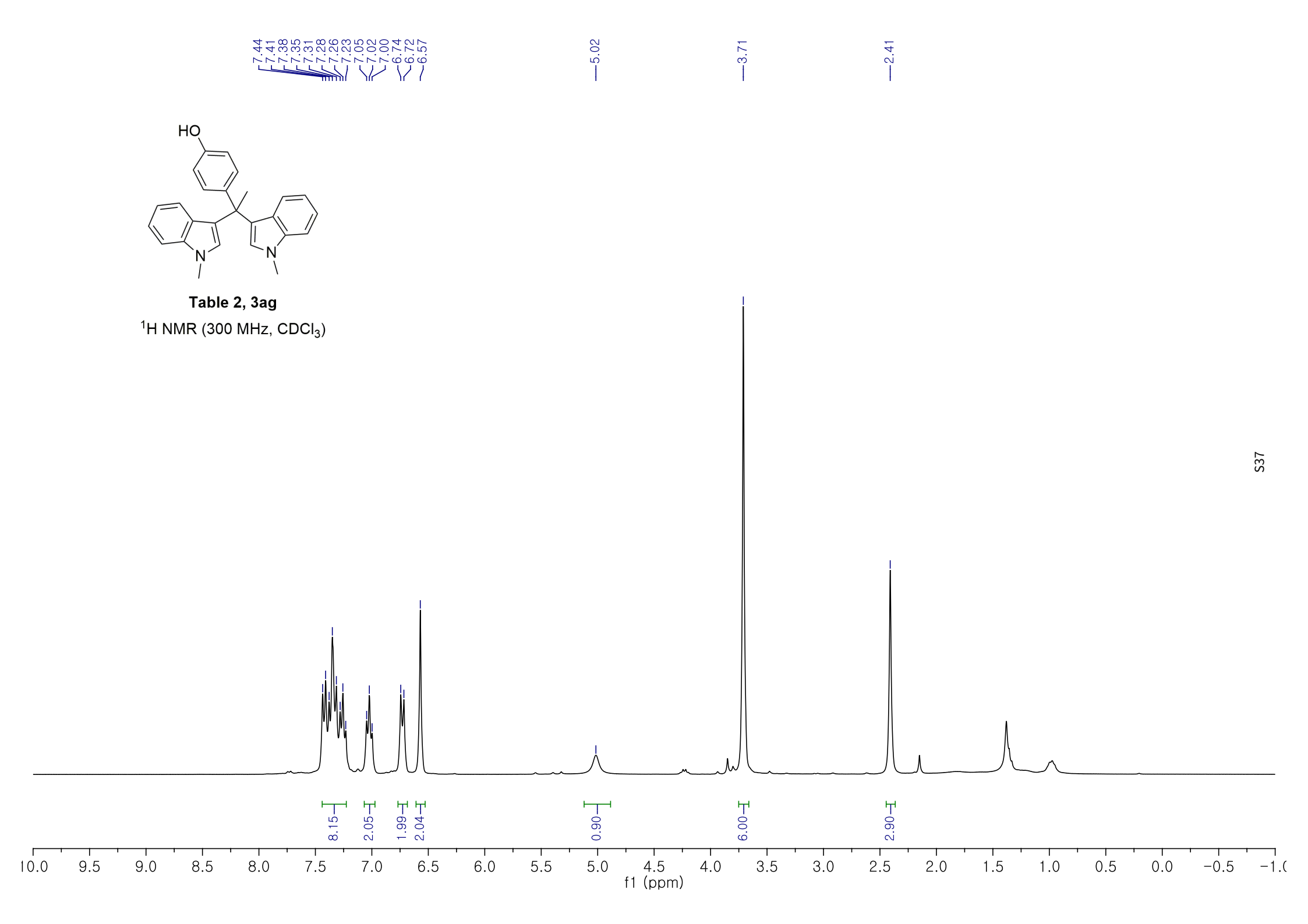


Table 2, 3af

^{1}H NMR (400 MHz, $CDCl_3$)


169.59
148.74
146.02
137.87
129.17
128.20
126.83
123.16
122.23
121.19
120.66
118.55
109.27
43.49
32.71
29.37
21.31
AcO
N
N
Table 2, 3af
13C NMR (101 MHz, CDCl3)
S36
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.44
7.41
7.38
7.35
7.31
7.28
7.26
7.23
7.05
7.02
7.00
6.74
6.72
6.57
5.02
3.71
2.41
HO
N
N
Table 2, 3ag
1H NMR (300 MHz, CDCl3)
S37
8.15
2.05
1.99
2.04
0.90
6.00
2.90
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

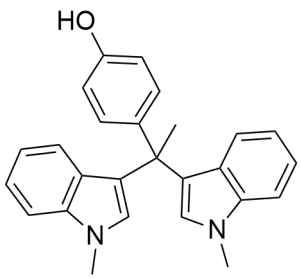


Table 2, 3ag

^{13}C NMR (101 MHz, $CDCl_3$)

153.77
140.61
137.87
129.30
128.19
126.89
123.71
122.33
121.09
118.41
114.67
109.25
43.11
32.71
29.27

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

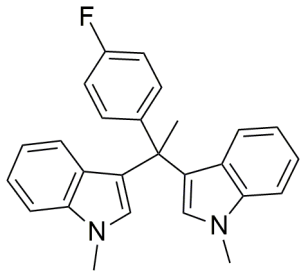
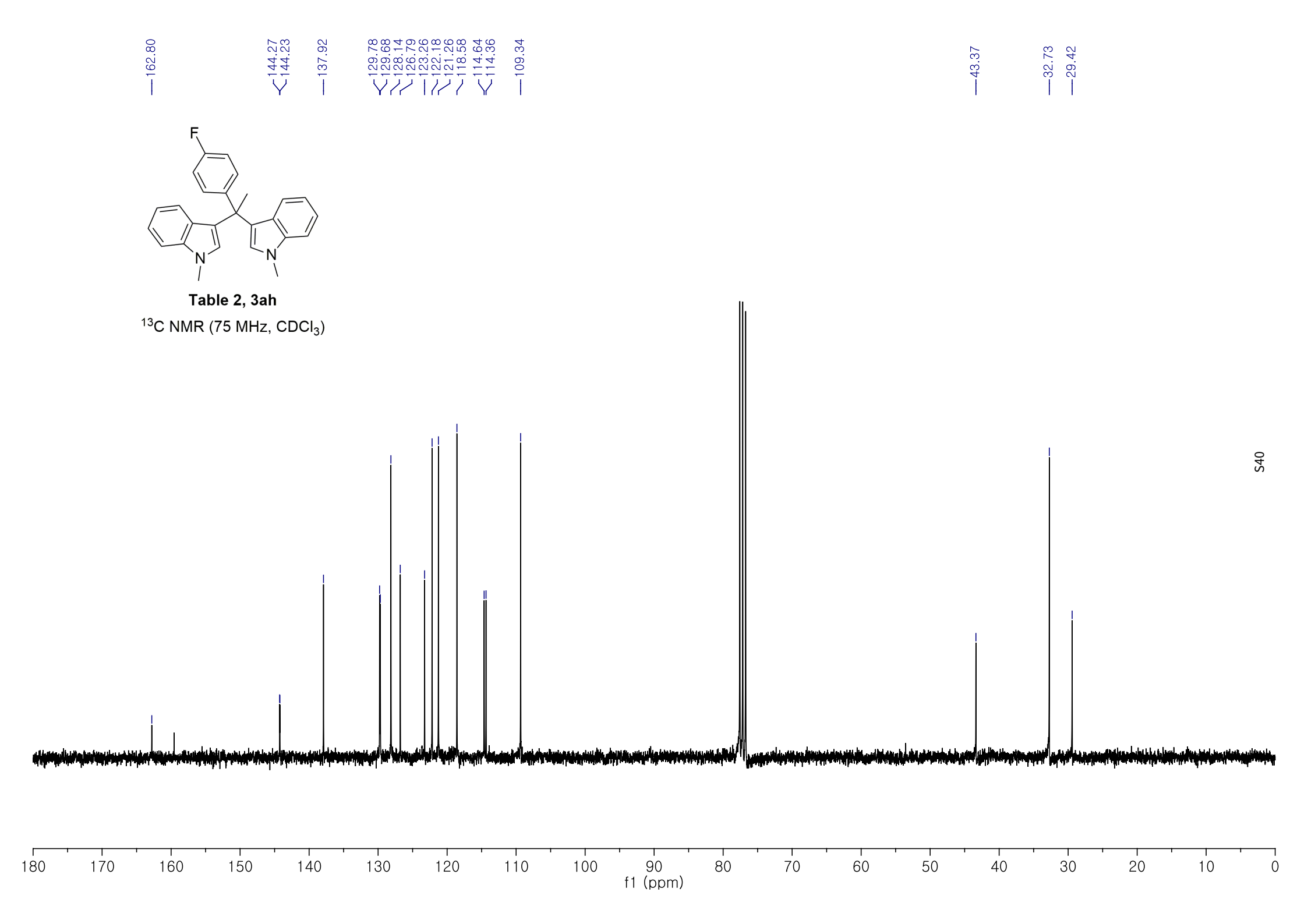


Table 2, 3ah

^{1}H NMR (300 MHz, $CDCl_3$)


162.80
144.27
144.23
137.92
129.78
129.68
128.14
126.79
123.26
122.18
121.26
118.58
114.64
114.36
109.34
43.37
32.73
29.42
F
N
N
Table 2, 3ah
13C NMR (75 MHz, CDCl3)
S40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

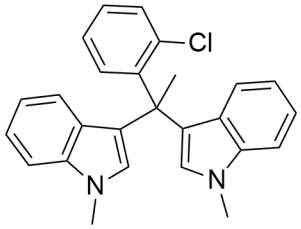
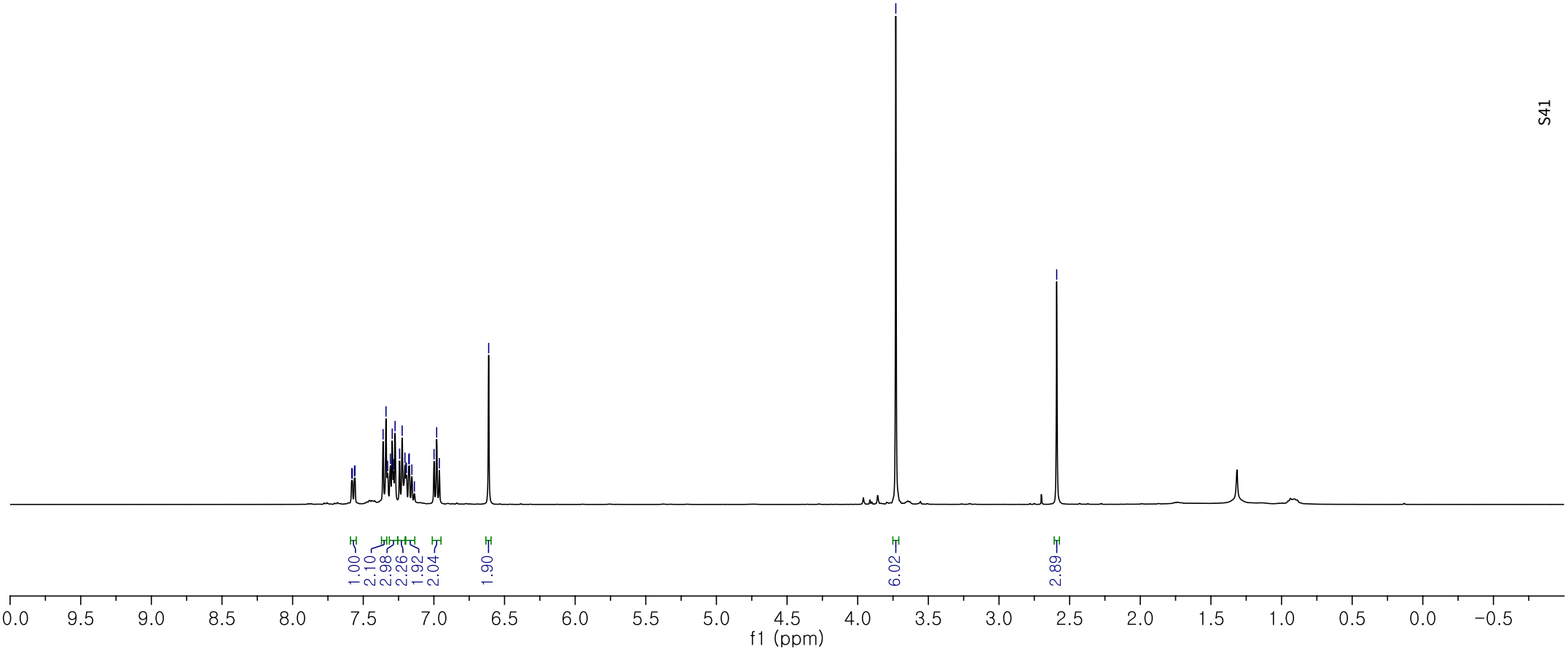


Table 2, 3ai

^{1}H NMR (400 MHz, $CDCl_3$)



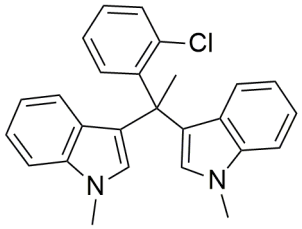


Table 2, 3ai

^{13}C NMR (101 MHz, $CDCl_3$)

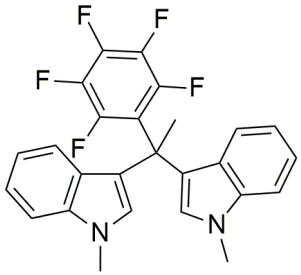


Table 2, 3aj

^{1}H NMR (400 MHz, $CDCl_3$)

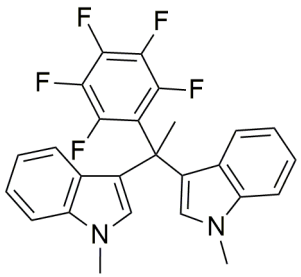


Table 2, 3aj

^{13}C NMR (101 MHz, $CDCl_3$)

147.35
144.80
139.23
137.86
136.67
127.16
126.31
121.48
120.94
120.52
118.93
109.67
42.70
32.83
32.80
29.67

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

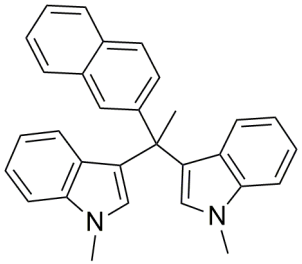


Table 2, 3ak

^{1}H NMR (300 MHz, $CDCl_3$)

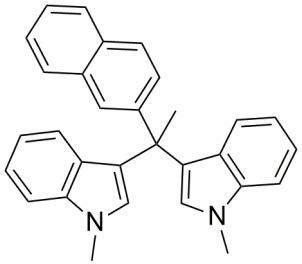


Table 2, 3ak

^{13}C NMR (75 MHz, $CDCl_3$)

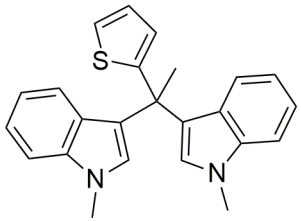


Table 2, 3al

^{1}H NMR (300 MHz, $CDCl_3$)

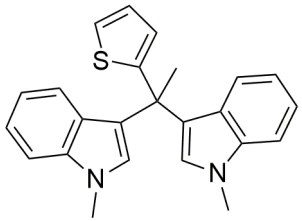


Table 2, 3al

^{13}C NMR (75 MHz, $CDCl_3$)

155.01
137.82
127.80
126.62
126.12
124.96
123.55
123.22
122.04
121.22
118.59
109.33
41.92
32.75
30.78

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)

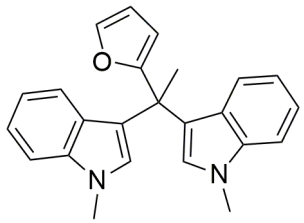


Table 2, 3am

^{1}H NMR (300 MHz, $CDCl_3$)

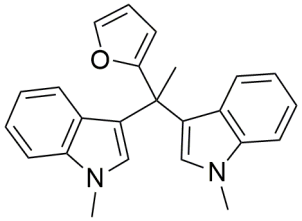


Table 2, 3am

^{13}C NMR (101 MHz, $CDCl_3$)

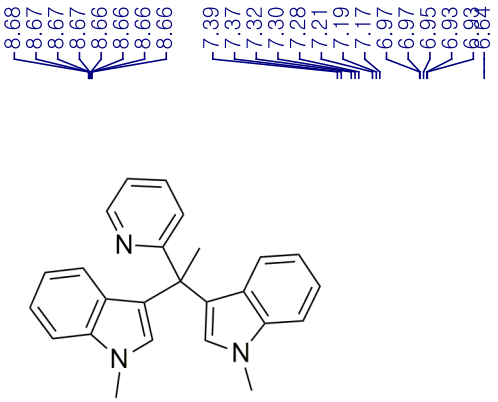
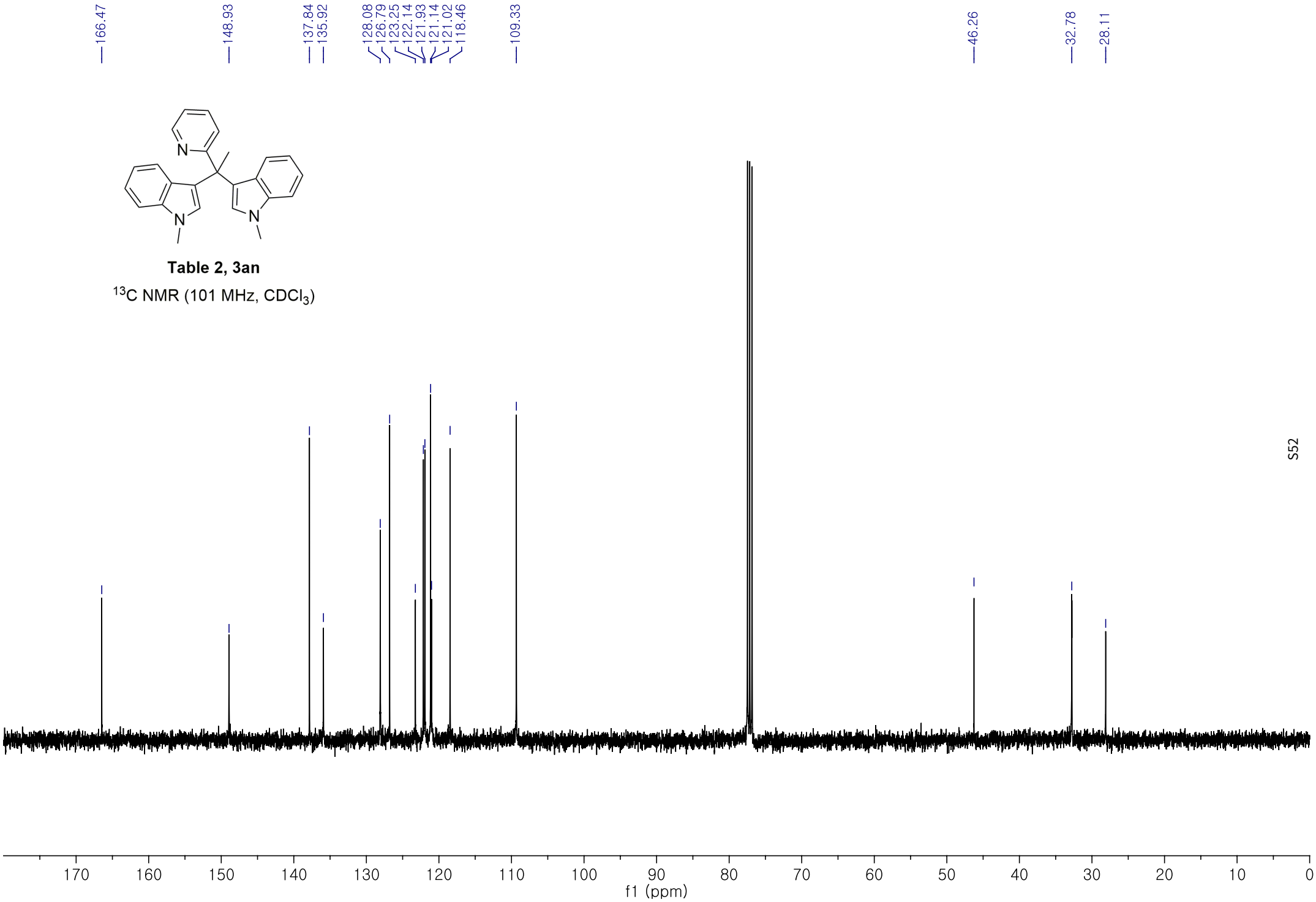


Table 2, 3an

^{1}H NMR (400 MHz, $CDCl_3$)


166.47
148.93
137.84
135.92
128.08
126.79
123.25
122.14
121.93
121.14
121.02
118.46
109.33
46.26
32.78
28.11
N
N
N
Table 2, 3an
13C NMR (101 MHz, CDCl3)
S52
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

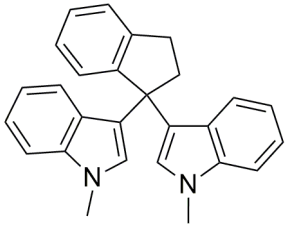


Table 2, 3ao

^{1}H NMR (300 MHz, $CDCl_3$)

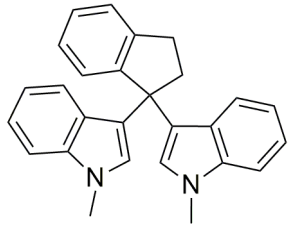


Table 2, 3ao

^{13}C NMR (75 MHz, $CDCl_3$)

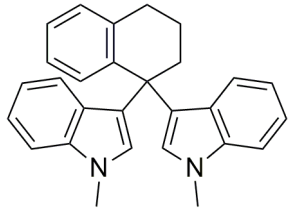


Table 2, 3ap

^{1}H NMR (300 MHz, $CDCl_3$)

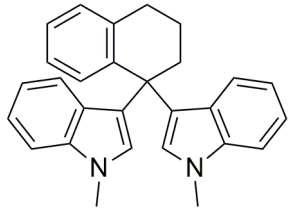
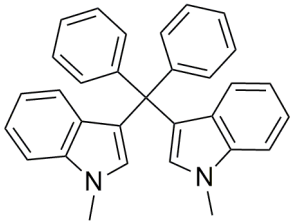


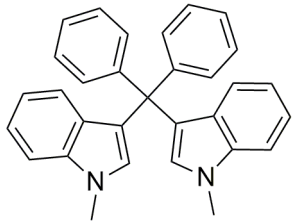
Table 2, 3ap

^{13}C NMR (75 MHz, $CDCl_3$)



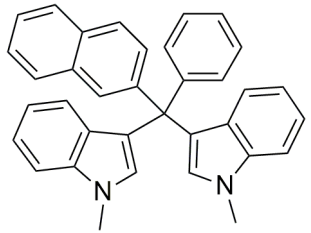
Scheme 2, 3ab

^{1}H NMR (400 MHz, $CDCl_3$)



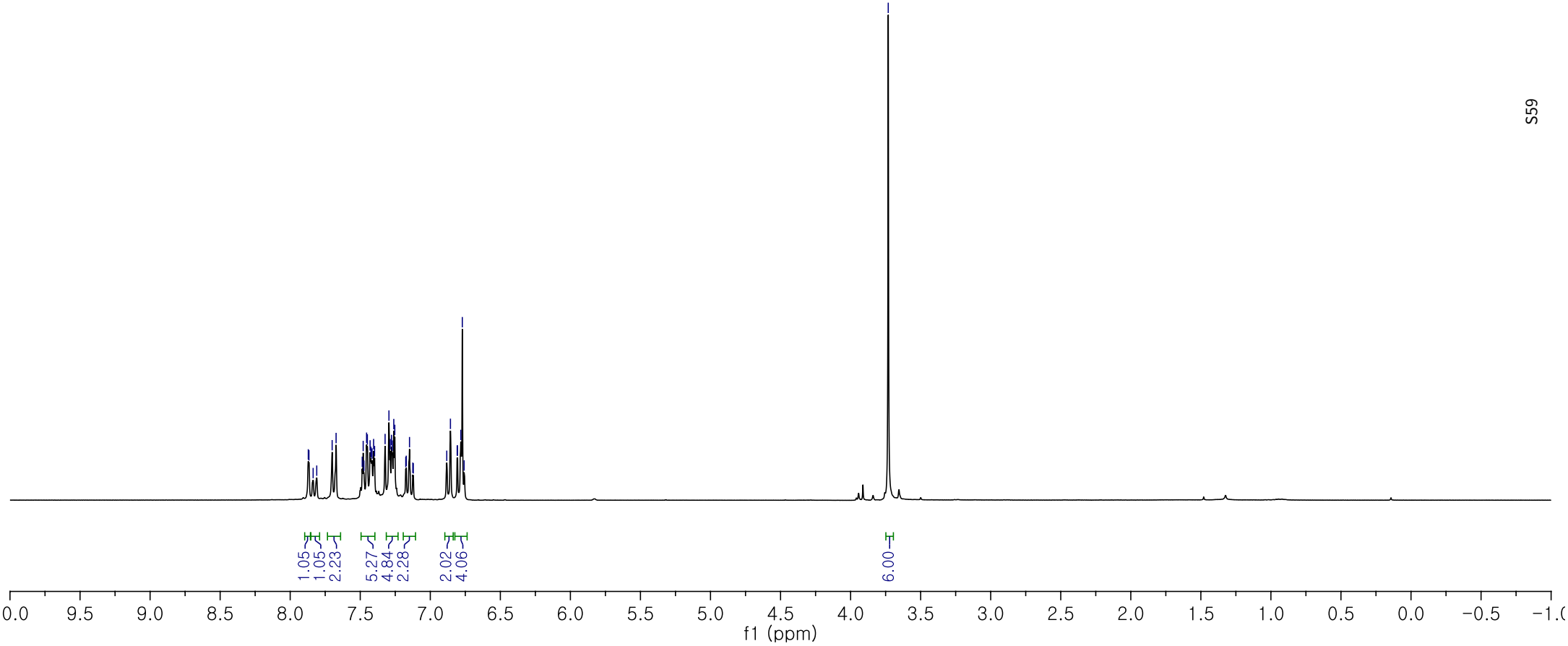
Scheme 2, 3ab

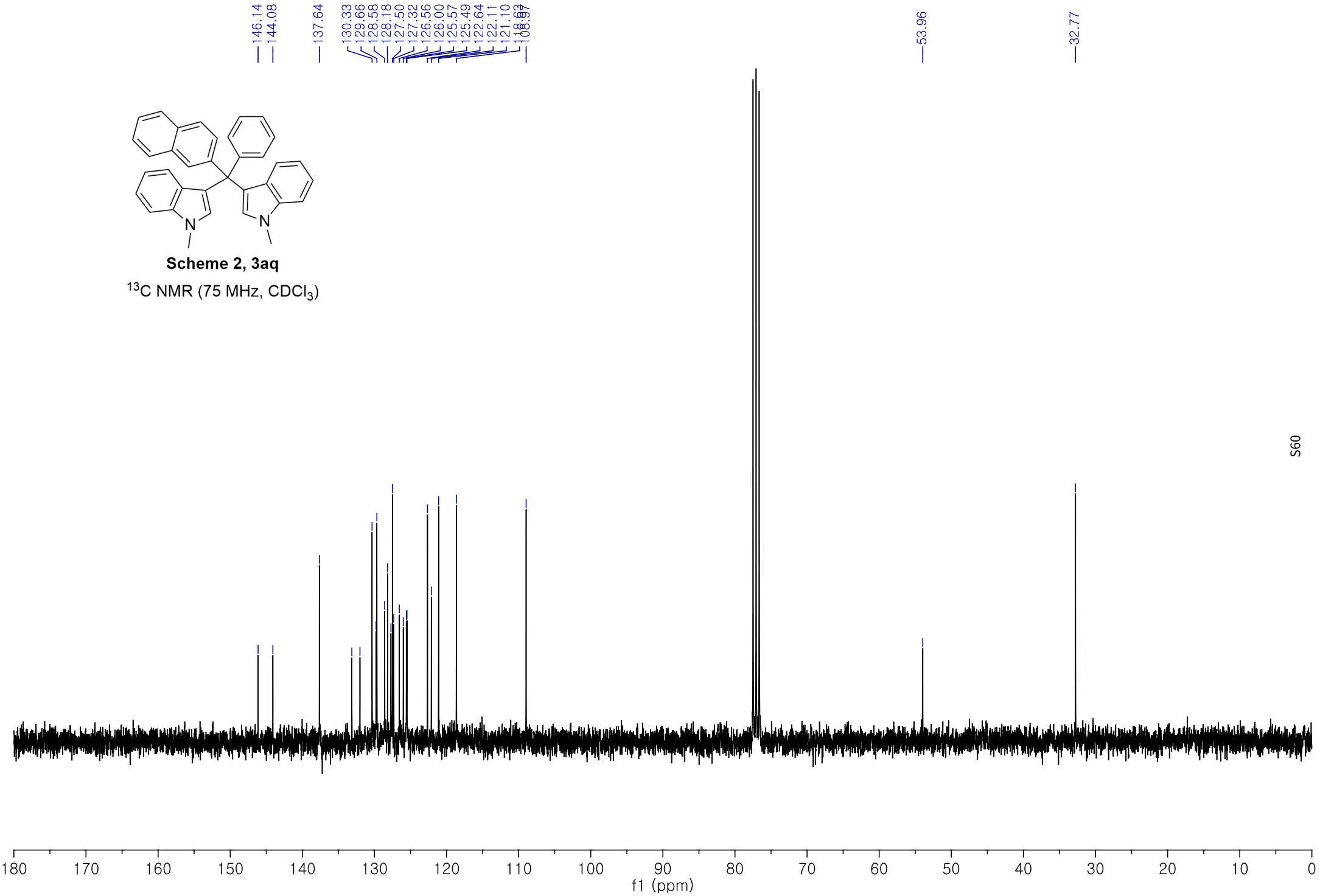
^{13}C NMR (101 MHz, $CDCl_3$)



Scheme 2, 3aq

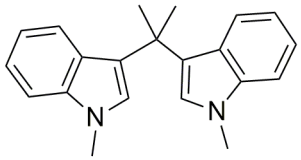
^{1}H NMR (300 MHz, $CDCl_3$)





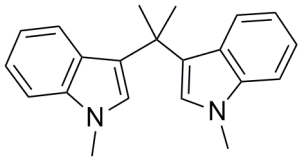
Scheme 2, 3aq

^{13}C NMR (75 MHz, $CDCl_3$)



Scheme 2, 3ar

^{1}H NMR (300 MHz, $CDCl_3$)



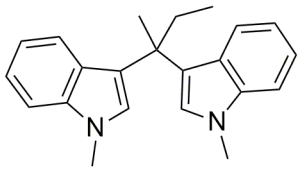
Scheme 2, 3ar

^{13}C NMR (75 MHz, $CDCl_3$)

137.87
126.82
125.62
124.19
121.59
121.01
118.18
109.19
35.07
32.78
30.46

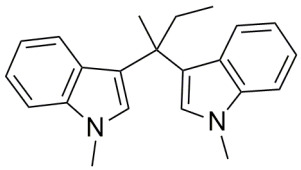
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

f1 (ppm)



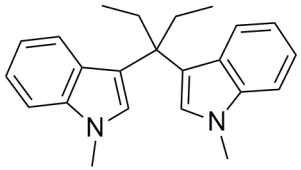
Scheme 2, 3as

^{1}H NMR (400 MHz, $CDCl_3$)



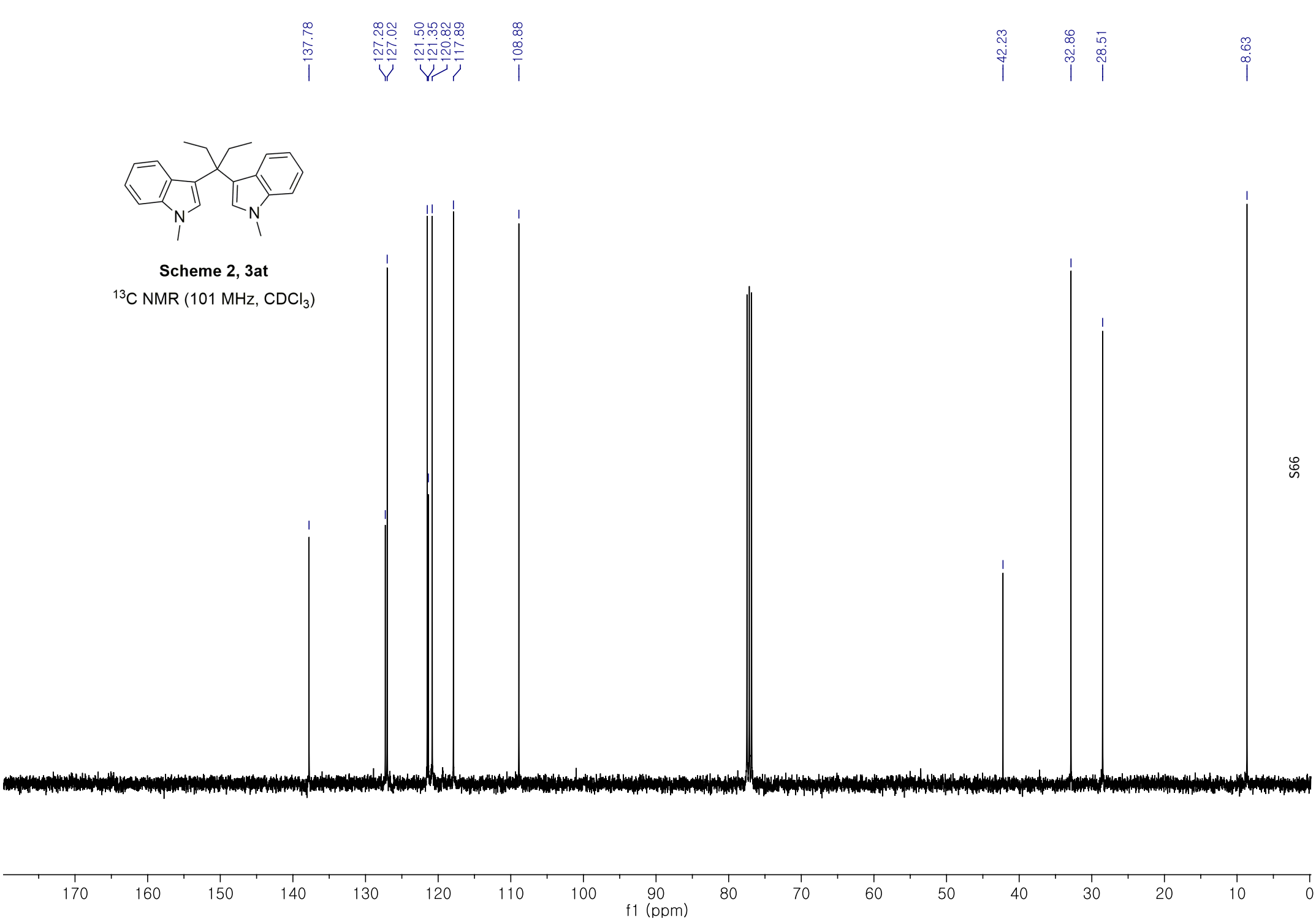
Scheme 2, 3as

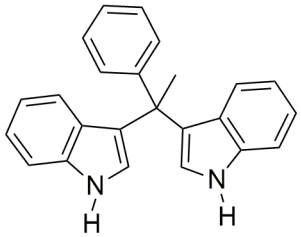
^{13}C NMR (101 MHz, $CDCl_3$)



Scheme 2, 3at

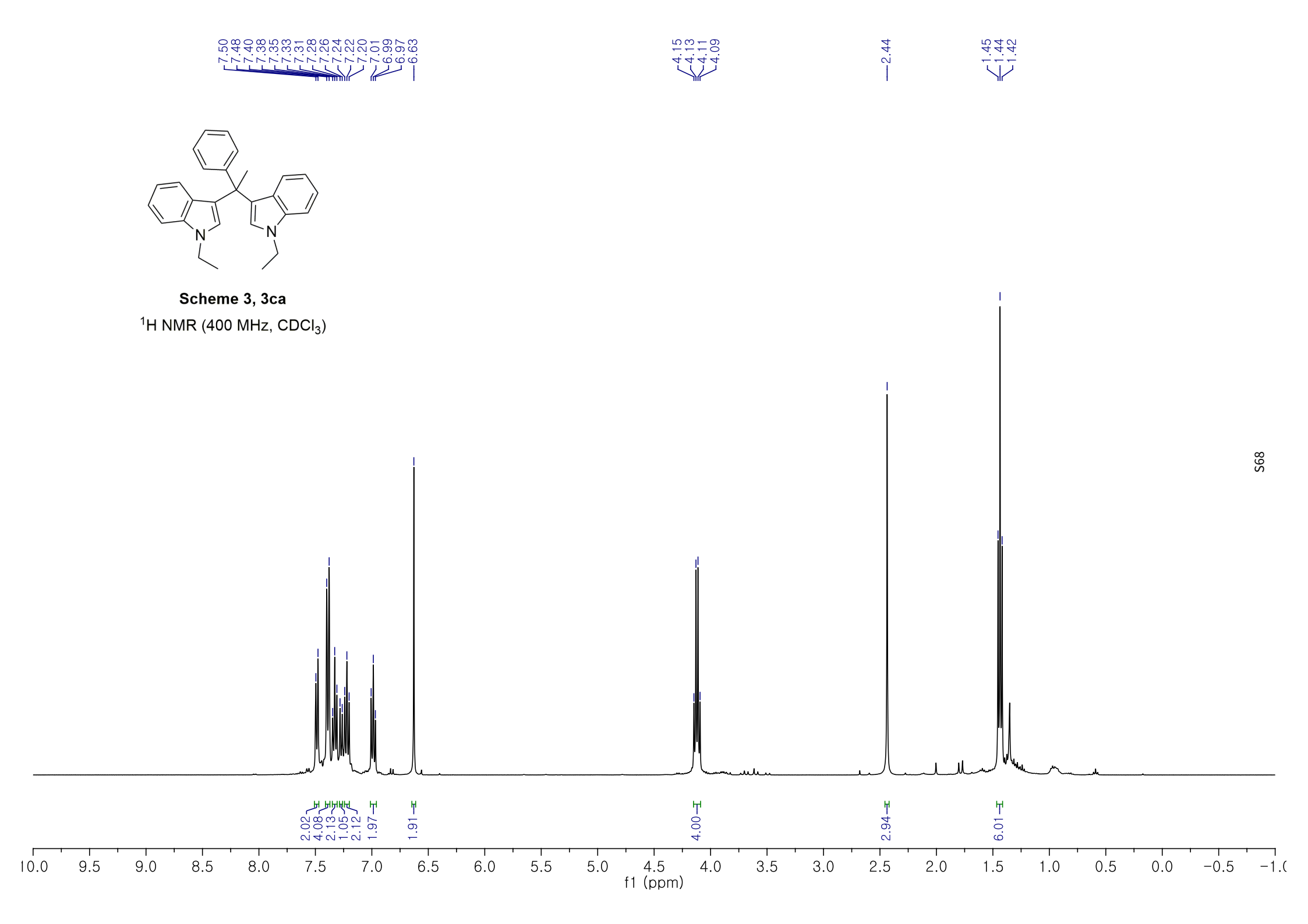
^{1}H NMR (400 MHz, $CDCl_3$)

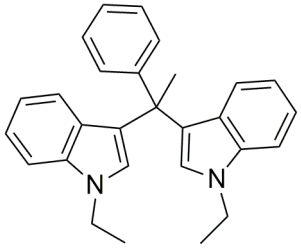

137.78
127.28
127.02
121.50
121.35
120.82
117.89
108.88
42.23
32.86
28.51
8.63
N
N
Scheme 2, 3at
13C NMR (101 MHz, CDCl3)
S66
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



Scheme 3, 3ba

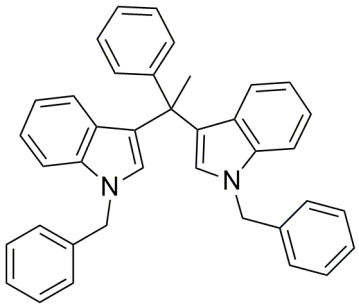
^{1}H NMR (300 MHz, $CDCl_3$)


7.50
7.48
7.40
7.38
7.35
7.33
7.31
7.28
7.26
7.24
7.22
7.20
7.01
6.99
6.97
6.63
4.15
4.13
4.11
4.09
2.44
1.45
1.44
1.42
N
N
Scheme 3, 3ca
1H NMR (400 MHz, CDCl3)
S68
2.02
4.08
2.13
1.05
2.12
1.97
1.91
4.00
2.94
6.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)



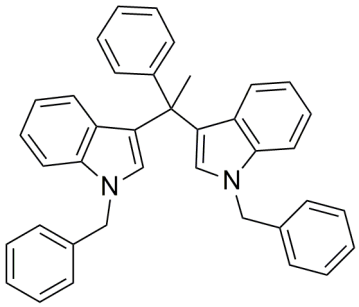
Scheme 3, 3ca

^{13}C NMR (101 MHz, $CDCl_3$)



Scheme 3, 3da

^{1}H NMR (400 MHz, $CDCl_3$)



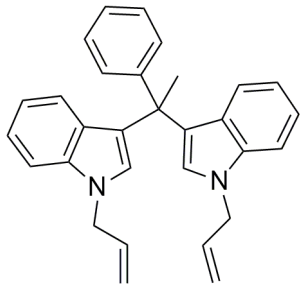
Scheme 3, 3da

^{13}C NMR (101 MHz, $CDCl_3$)

148.14
137.96
137.45
128.74
128.20
127.92
127.76
127.45
127.29
126.52
125.91
123.78
122.36
121.37
118.73
109.86
49.89
43.86
29.39

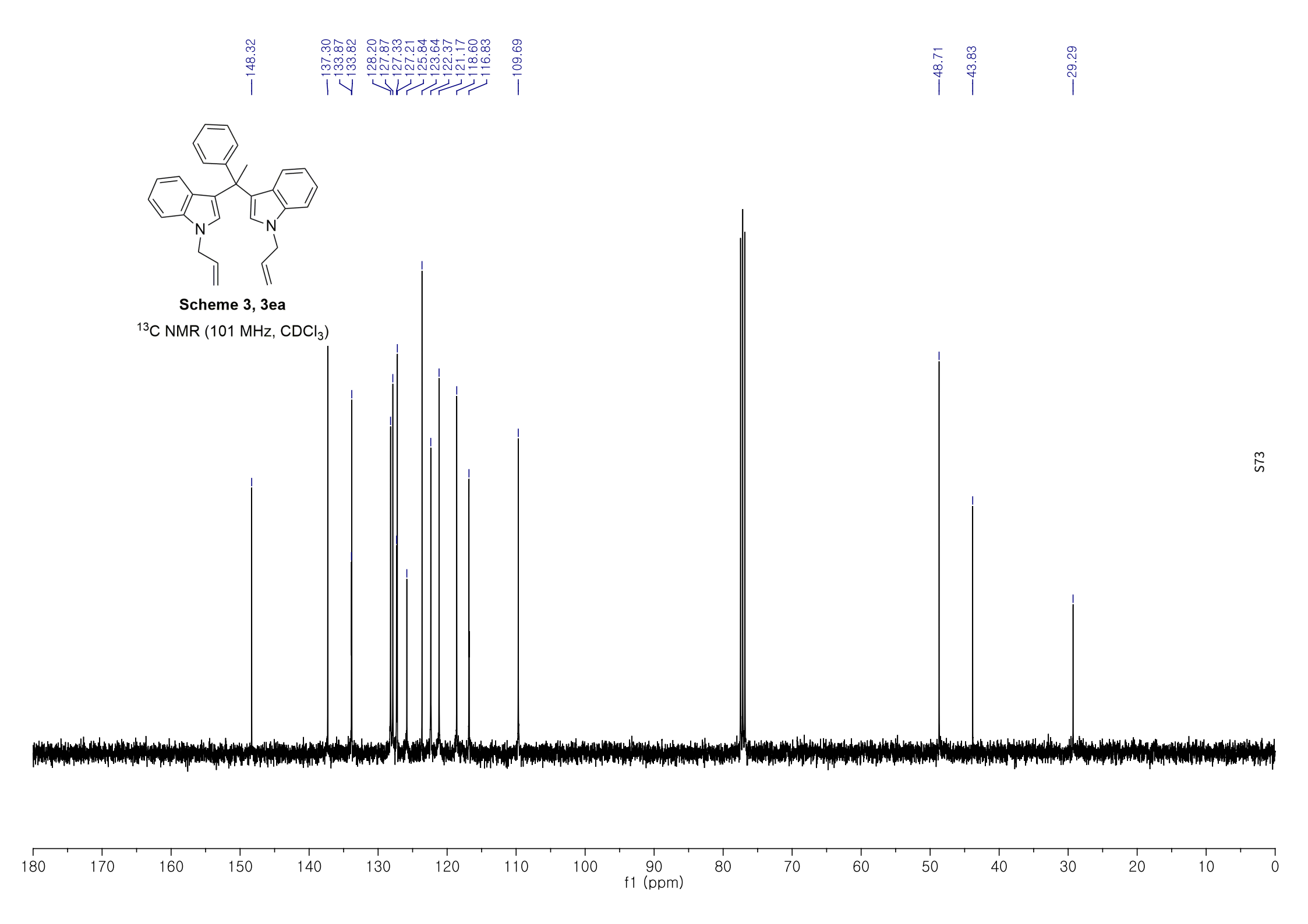
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

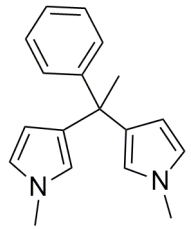
f1 (ppm)



Scheme 3, 3ea

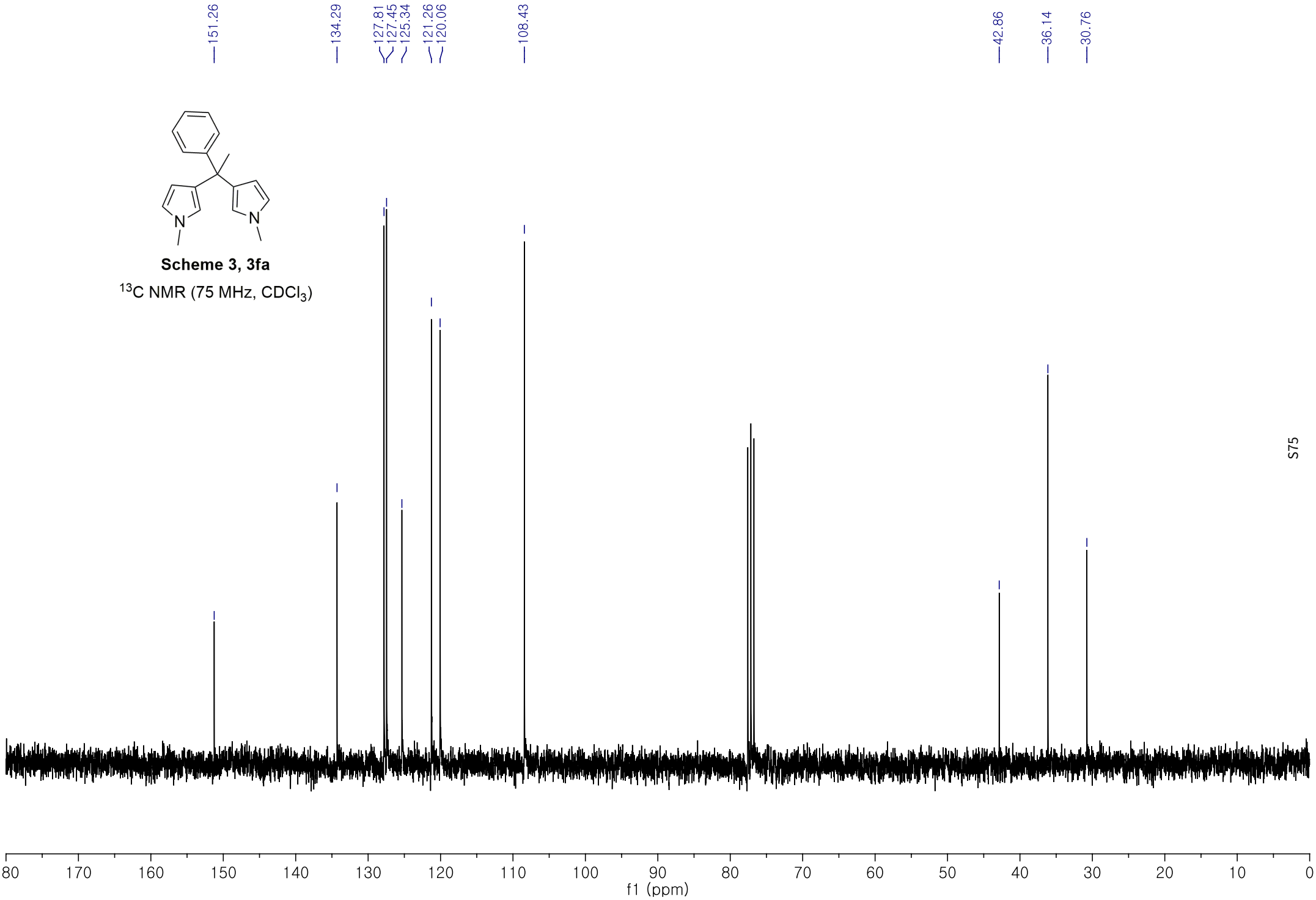
^{1}H NMR (400 MHz, $CDCl_3$)

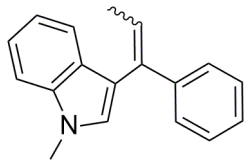

148.32
137.30
133.87
133.82
128.20
127.87
127.33
127.21
125.84
123.64
122.37
121.17
118.60
116.83
109.69
48.71
43.83
29.29
N
N
Scheme 3, 3ea
13C NMR (101 MHz, CDCl3)
S73
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



Scheme 3, 3fa

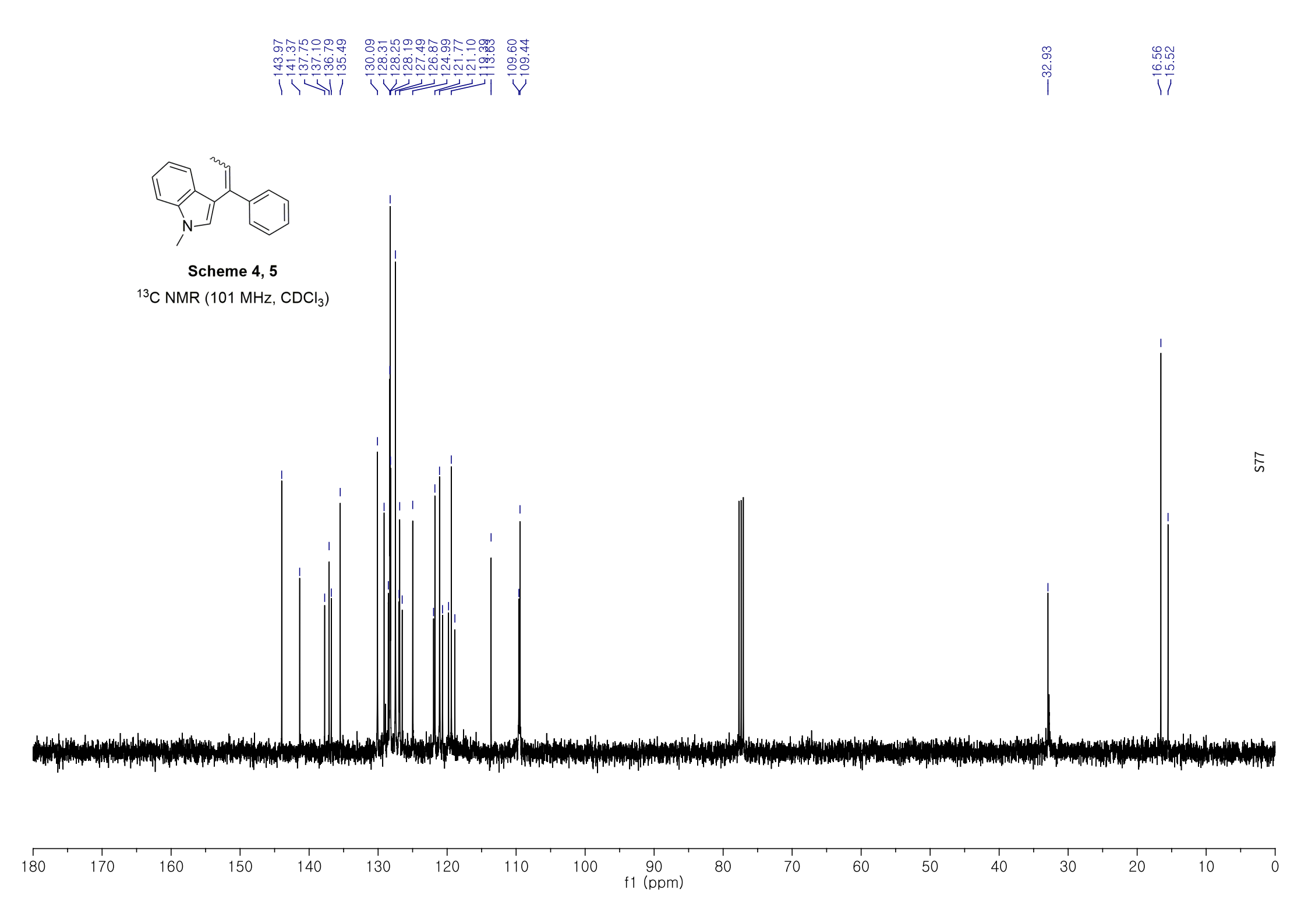
^{1}H NMR (400 MHz, $CDCl_3$)

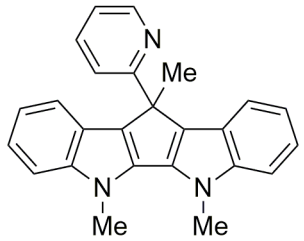

151.26
134.29
127.81
127.45
125.34
121.26
120.06
108.43
42.86
36.14
30.76
N
N
Scheme 3, 3fa
13C NMR (75 MHz, CDCl3)
S75
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



Scheme 4, 5

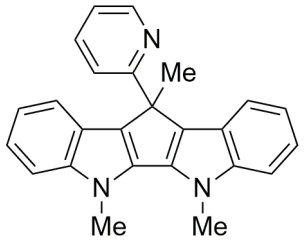
^{1}H NMR (400 MHz, $CDCl_3$)


143.97
141.37
137.75
137.10
136.79
135.49
130.09
128.31
128.25
128.19
127.49
126.87
124.99
121.77
121.10
109.60
109.44
32.93
16.56
15.52
N
Scheme 4, 5
13C NMR (101 MHz, CDCl3)
S77
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



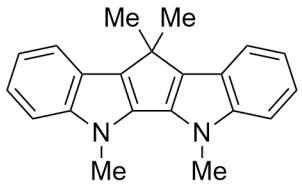
Scheme 5, 6an

^{1}H NMR (300 MHz, $CDCl_3$)



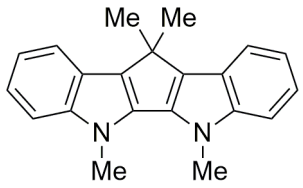
Scheme 5, 6an

^{13}C NMR (75 MHz, $CDCl_3$)



Scheme 5, 6ar

^{1}H NMR (300 MHz, $CDCl_3$)



Scheme 5, 6ar

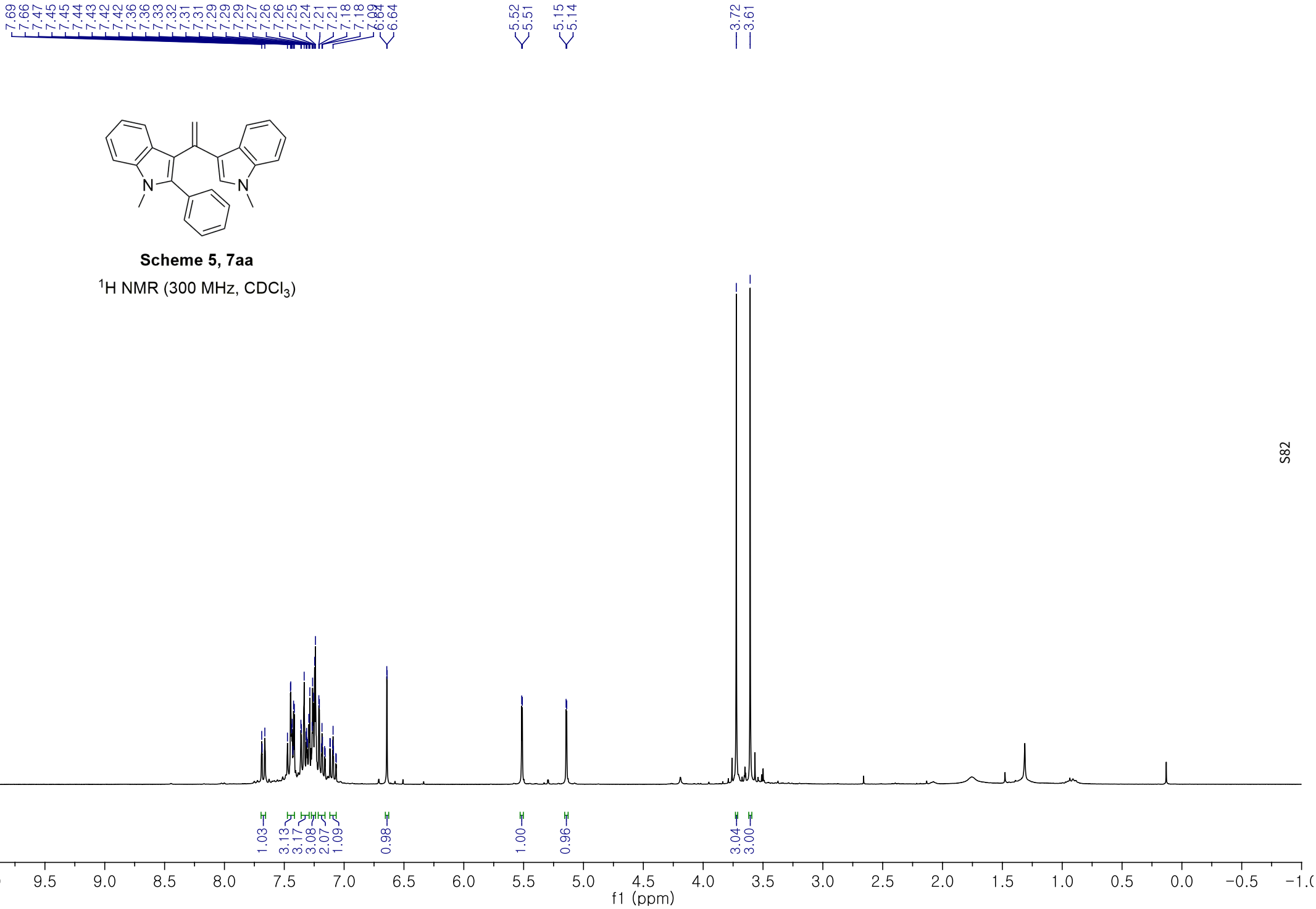
^{13}C NMR (75 MHz, $CDCl_3$)

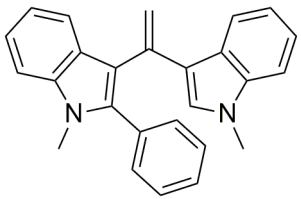
136.24
120.32
119.89
117.66
110.11
53.48
32.89
26.07

S81

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 −20

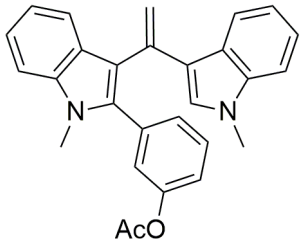
f1 (ppm)


7.69
7.66
7.47
7.45
7.45
7.44
7.43
7.42
7.42
7.36
7.33
7.32
7.31
7.31
7.29
7.29
7.29
7.27
7.26
7.26
7.25
7.24
7.21
7.18
7.18
7.09
6.64
6.64
5.52
5.51
5.15
5.14
3.72
3.61
N
N
Scheme 5, 7aa
1H NMR (300 MHz, CDCl3)
S82
1.03
3.13
3.17
3.08
2.07
1.09
0.98
1.00
0.96
3.04
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)



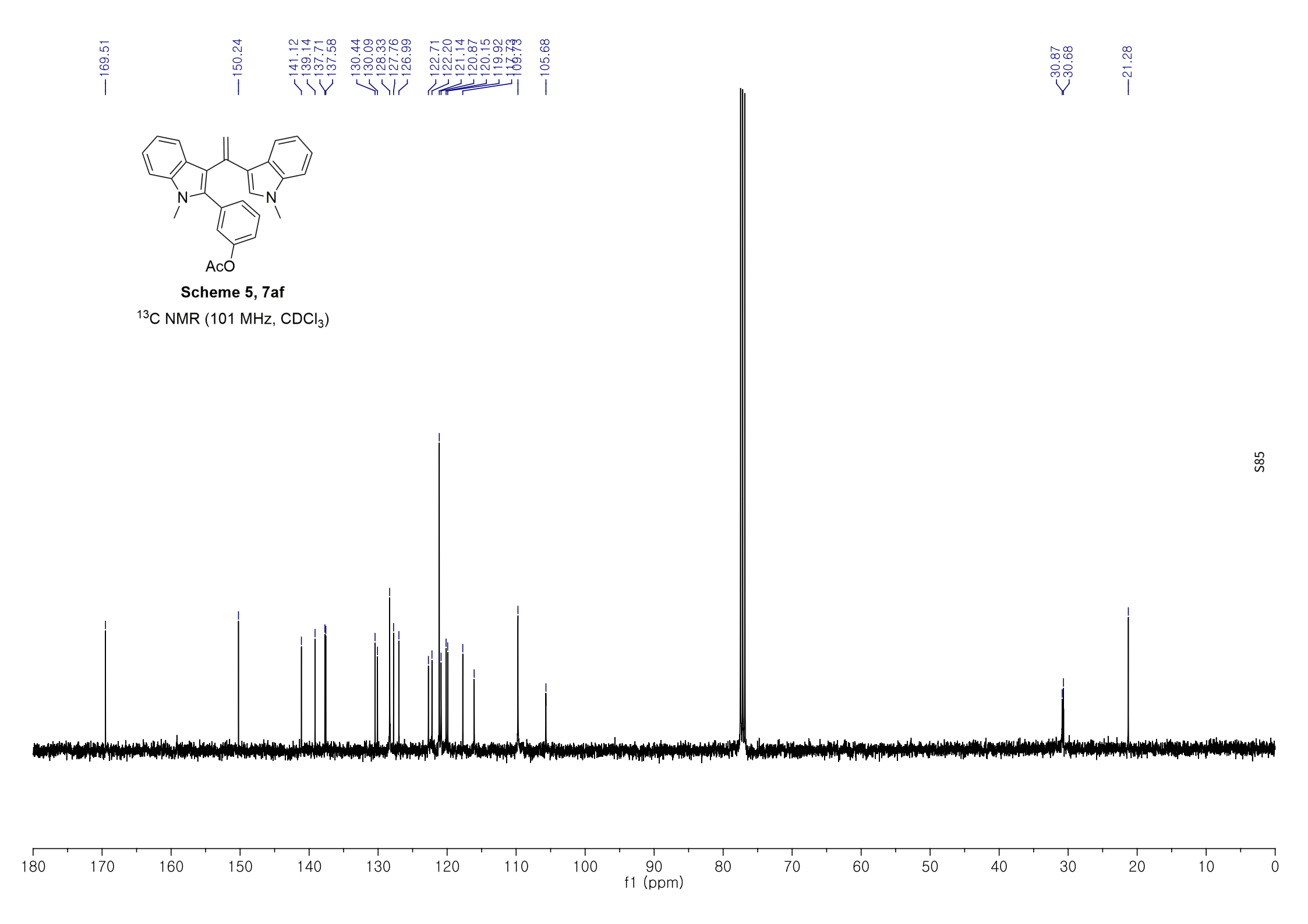
Scheme 5, 7aa

^{13}C NMR (101 MHz, $CDCl_3$)



Scheme 5, 7af

^{1}H NMR (400 MHz, $CDCl_3$)


169.51
150.24
141.12
139.14
137.71
137.58
130.44
130.09
128.33
127.76
126.99
122.71
122.20
121.14
120.87
120.15
119.92
105.68
30.87
30.68
21.28
N
N
AcO
Scheme 5, 7af
13C NMR (101 MHz, CDCl3)
S85
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)