Supporting Information

Bipyrrole Scaffold Made Easy from Diyne

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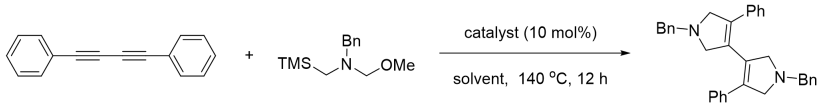
1. General information.

All reactions were carried out using oven-dried glassware and magnetic stirring under argon gas unless otherwise stated. Reaction temperatures are reported as the temperature of the bath surrounding the vessel. Analytical thin layer chromatography was performed on silica gel aluminum plates with F-254 indicator and visualized by UV light (254 nm). Column chromatography was performed using 200-300 mesh silica gel. NMR spectra were recorded on AVANCE III HD 400 MHz or Bruker AVANCE III 300 MHz spectrometer. Chemical shifts (δ) are quoted in ppm relative to TMS (^{1}H) and $CFCl_3$ (^{19}F). Coupling constants (J) are quoted in Hz. The following abbreviations were used to show the multiplicities: s: singlet, d: doublet, t: triplet, q: quadruplet, m: multiplet. The residual solvent signals were used as references ($CDCl_3$: δ_H = 7.26 ppm, δ_C = 77.00 ppm or relative to external $CFCl_3$, δ_F = 0 ppm). High-resolution mass spectrometry (HRMS) was carried out on a Waters Xevo G2-XS QTof.

2. Materials.

Toluene was distilled over sodium/benzophenone. Anhydrous *o*-xylene, TsOH, and Cu_2O was purchased from Innochem Ltd. Pyridine was purchased from Tianjin DeEn Chemical Reagent Co., Ltd. $W(CO)_6$ was purchased from Titan Technology (Shanghai) Co., Ltd. *N*-(methoxymethyl)-*N*-(trimethylsilylmethyl)-benzylamine was purchased from Shanghai Haohong Biomedical Technology Co., Ltd. All the 1,3-diyne **1** were synthesized according to the literature.[1]

3. Optimization of Cycloaddition Reaction Conditions.[a]



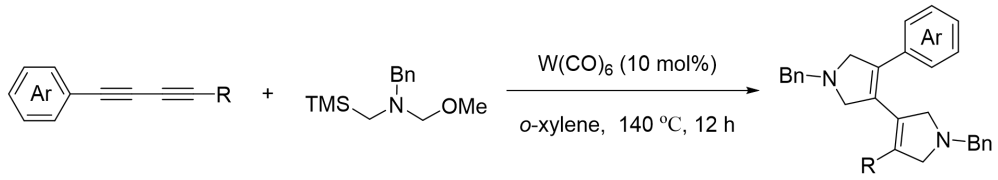
1a **2** **3a**

Entry	Cat. (X mol%)	Sol./Temp. (°C)/time (h)	Yield(%)[b]
1	$Fe_2(CO)_9$ (10)	*o*-xylene/140/12	33
2	$Re_2(CO)_{10}$ (10)	*o*-xylene/140/12	40
3	$Ru(CO)_{12}$ (10)	*o*-xylene/140/12	26
4	$Co_2(CO)_8$ (10)	*o*-xylene/140/12	29
5	$Cr(CO)_6$ (10)	*o*-xylene/140/12	26
6	$Mo(CO)_6$ (10)	*o*-xylene/140/12	56

7	$Mn_2(CO)_{10}$ (10)	*o*-xylene/140/12	47
8	$W(CO)_6$ (10)	*o*-xylene/140/12	85
9	$Cu(OAc)_2$ (10)	*o*-xylene/140/12	19
10	$Pd(OAc)_2$ (10)	*o*-xylene/140/12	28
11	WCl_6 (10)	*o*-xylene/140/12	39
12	-	*o*-xylene/140/12	7
14	$W(CO)_6$ (10)	toluene/140/12	68
15	$W(CO)_6$ (10)	PhCl/140/12	66
16	$W(CO)_6$ (10)	1,4-Dioxane/140/12	37
17	$W(CO)_6$ (10)	DMF/140/12	32
18	$W(CO)_6$ (10)	NMP/140/12	29
19	$W(CO)_6$ (10)	MeCN/140/12	36
20	$W(CO)_6$ (10)	*o*-xylene/120/12	34
21	$W(CO)_6$ (10)	*o*-xylene/130/12	51
22	$W(CO)_6$ (10)	*o*-xylene/150/12	80
23	$W(CO)_6$ (10)	*o*-xylene/140/6	37
24	$W(CO)_6$ (10)	*o*-xylene/140/24	83
25[c]	$W(CO)_6$ (10)	*o*-xylene/140/12	36
26[d]	$W(CO)_6$ (10)	*o*-xylene/140/12	78

Reaction conditions: [a]1,3-diyne **1a** (0.2 mmol, 1.0 equiv.), *N*-(methoxymethyl)-*N*-(trimethylsilylmethyl)-benzylamine **2** (1.2 mmol, 6.0 equiv.), indicated catalyst (0.02 mmol, 10 mol%), in indicated solvent (2 mL), under Ar, at indicated temperature for 12 h. [b]Isolated yield. [c]1,3-diyne **1a** (0.2 mmol, 1.0 equiv.), *N*-(methoxymethyl)-*N*-(trimethylsilylmethyl)-benzylamine **2** (1.2 mmol, 2.0 equiv.), indicated catalyst (0.02 mmol, 10 mol%), in indicated solvent (2 mL), under Ar, at indicated temperature for 12 h. [d]1,3-diyne **1a** (0.2 mmol, 1.0 equiv.), *N*-(methoxymethyl)-*N*- (trimethylsilylmethyl)-benzylamine **2** (1.2 mmol, 5.0 equiv.), indicated catalyst (0.02 mmol, 10 mol%), in indicated solvent (2 mL), under Ar, at indicated temperature for 12 h.

4. General procedure for the synthesis of derivatives 3.

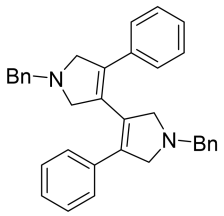


1 **2** **3**

An oven-dried 25 mL schlenk tube equipped with a stirring bar was transferred into glovebox (through standard glovebox operation), where $W(CO)_6$ (0.02 mmol, 7.0 mg, 0.1 equiv.) was added. The tube was then removed from glovebox and placed under Ar. Then 1,3-diyne **1** (0.2 mmol, 1.0 equiv.), *N*-(methoxymethyl)-*N*-(trimethylsilylmethyl)-benzylamine **2** (1.2 mmol, 6.0 equiv.),

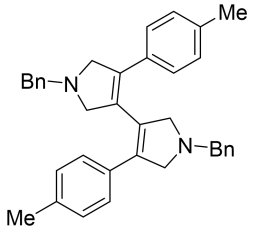
and *o*-xylene (2 mL) were added subsequently to the test tube under Ar. The resulting reaction mixture was stirred at 140 ℃ for 12 h. After reaction completed, the mixture was cooled down to room temperature, and the volatiles were removed under reduced pressure. The residue was then purified by flash column chromatography on silica gel to give the desired product **3**.

5. Purification and characterization of derivatives 3.



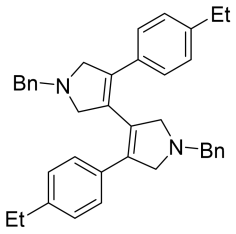
3a

1,1'-dibenzyl-4,4'-diphenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3a. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (85%, 79.6 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.44. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.18 (m, 20H), 3.95 – 3.84 (m, 4H), 3.84 – 3.73 (m, 4H), 3.65 – 3.52 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.23, 136.04, 135.18, 130.55, 128.68, 128.38, 128.27, 127.38, 127.03, 126.94, 62.90, 62.81, 60.40. **HRMS** (ESI) calcd for $C_{34}H_{33}N_2^+$ *m/z* 469.2644 $[M+H]^+$, Found 469.2647.



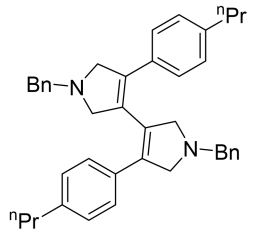
3b

1,1'-dibenzyl-4,4'-di-p-tolyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3b. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (85%, 84.4 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.41. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.18 (m, 14H), 7.11 – 7.03 (m, 4H), 3.92 – 3.83 (m, 4H), 3.82 – 3.73 (m, 4H), 3.62 – 3.49 (m, 4H), 2.31 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.33, 137.16, 135.70, 132.35, 129.94, 129.02, 128.72, 128.37, 127.02, 126.88, 62.90, 62.78, 60.46, 21.34. **HRMS** (ESI) calcd for $C_{36}H_{37}N_2^+$ *m/z* 497.2951 $[M+H]^+$, Found 497.2946.



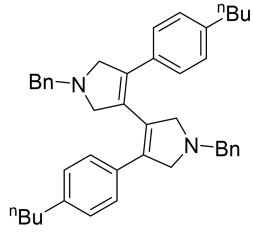
3c

1,1'-dibenzyl-4,4'-bis(4-ethylphenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3c. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a white solid (75%, 78.6 mg), mp 105.8-107.1 °C. R_f (petroleum ether/ethyl acetate = 5:1): 0.45. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 – 7.26 (m, 2H), 7.25 – 7.20 (m, 8H), 7.20 – 7.11 (m, 4H), 7.03 (d, J = 7.6 Hz, 4H), 3.84 (t, J = 3.8 Hz, 4H), 3.73 (s, 4H), 3.52 (t, J = 3.8 Hz, 4H), 2.55 (q, J = 7.6 Hz, 4H), 1.16 (t, J = 7.6 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.46, 139.36, 135.63, 132.57, 129.95, 128.70, 128.35, 127.78, 126.99, 126.92, 62.93, 62.77, 60.47, 28.68, 15.57. **HRMS** (ESI) calcd for $C_{38}H_{41}N_2^+$ *m/z* 525.3264 $[M+H]^+$, Found 525.3270.



3d

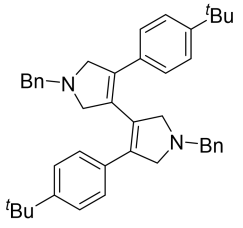
1,1'-dibenzyl-4,4'-bis(4-propylphenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3d. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (82%, 90.7 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.47. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.29 – 7.24 (m, 4H), 7.23 – 7.17 (m, 8H), 7.17 – 7.10 (m, 2H), 7.03 – 6.96 (m, 4H), 3.83 (t, J = 3.6 Hz, 4H), 3.71 (s, 4H), 3.52 (t, J = 4.0 Hz, 4H), 2.47 (t, J = 6.8 Hz, 4H), 1.62 – 1.48 (m, 4H), 0.86 (t, J = 7.2 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.92, 139.41, 135.72, 132.65, 129.98, 128.72, 128.41, 128.39, 127.02, 126.87, 63.00, 62.82, 60.51, 37.90, 24.54, 13.96. **HRMS** (ESI) calcd for $C_{40}H_{45}N_2^+$ *m/z* 553.3577 $[M+H]^+$, Found 553.3567.



3e

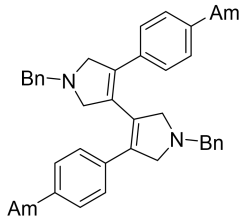
1,1'-dibenzyl-4,4'-bis(4-butylphenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3e. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a

yellow oil (69%, 80.2 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.37. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.21 (m, 14H), 7.07 (d, J = 7.6 Hz, 4H), 3.89 (t, J = 4.0 Hz, 4H), 3.78 (s, 4H), 3.57 (t, J = 3.6 Hz, 4H), 2.56 (t, J = 7.6 Hz, 4H), 1.61 – 1.52 (m, 4H), 1.39 – 1.28 (m, 4H), 0.92 (t, J = 7.2 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.10, 139.32, 135.61, 132.50, 129.86, 128.66, 128.32, 128.27, 126.95, 126.80, 62.90, 62.73, 60.45, 35.43, 33.57, 22.39, 14.02. **HRMS** (ESI) calcd for $C_{42}H_{48}N_2Na^+$ *m/z* 603.3710 $[M+Na]^+$, Found 603.3718.



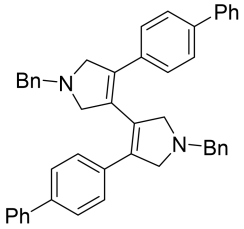
3f

1,1'-dibenzyl-4,4'-bis(4-(tert-butyl)phenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3f. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a white solid (69%, 80.2 mg), mp 186.2-187.1 ℃. R_f (petroleum ether/ethyl acetate = 5:1): 0.41. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 12H), 7.25 – 7.19 (m, 6H), 3.90 (t, J = 4.0 Hz, 4H), 3.79 (s, 4H), 3.58 (t, J = 3.6 Hz, 4H), 1.29 (s, 18H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.16, 139.33, 135.36, 132.22, 129.99, 128.65, 128.31, 126.94, 126.55, 125.10, 62.90, 62.70, 60.45, 34.56, 31.30. **HRMS** (ESI) calcd for $C_{42}H_{49}N_2^+$ *m/z* 581.3890 $[M+H]^+$, Found 581.3880.



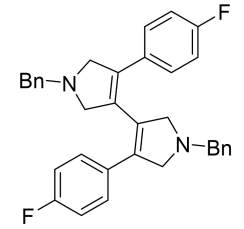
3g

1,1'-dibenzyl-4,4'-bis(4-pentylphenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3g. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (80%, 97.4 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.52. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.26 (m, 10H), 7.25 – 7.21 (m, 4H), 7.07 (d, J = 6.4 Hz, 4H), 3.89 (t, J = 4.0 Hz, 4H), 3.79 (s, 4H), 3.58 (t, J = 4.0 Hz, 4H), 2.56 (t, J = 8.0 Hz 4H), 1.61 – 1.55 (m, 4H), 1.37 – 1.27 (m, 8H), 0.89 (t, J = 6.8 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.19, 139.23, 135.62, 132.45, 129.79, 128.68, 128.33, 128.28, 126.98, 126.80, 62.86, 62.69, 60.43, 35.72, 31.55, 31.12, 22.59, 14.09. **HRMS** (ESI) calcd for $C_{44}H_{53}N_2^+$ *m/z* 609.4203 $[M+H]^+$, Found 609.4198.



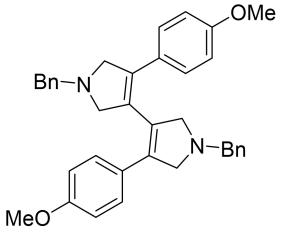
3h

4,4'-di([1,1'-biphenyl]-4-yl)-1,1'-dibenzyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3h. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (75%, 93.0 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.32. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.55 (d, J = 7.2 Hz, 4H), 7.49 (d, J = 8.4 Hz, 4H), 7.46 – 7.37 (m, 8H), 7.37 – 7.24 (m, 10H), 7.24 – 7.16 (m, 2H), 3.94 (t, J = 3.6 Hz, 4H), 3.81 (s, 4H), 3.65 (t, J = 3.6 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.71, 140.03, 139.21, 135.64, 134.14, 130.82, 128.85, 128.72, 128.43, 127.36, 127.09, 126.99, 126.94, 62.90, 62.86, 60.44, one carbon was overlapped. **HRMS** (ESI) calcd for $C_{46}H_{41}N_2^+$ *m/z* 621.3270 $[M+H]^+$, Found 621.3275.



3i

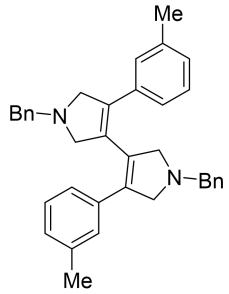
1,1'-dibenzyl-4,4'-bis(4-fluorophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3i. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a white solid (81%, 81.7 mg), mp 155.7-157.3 ℃. R_f (petroleum ether/ethyl acetate = 5:1): 0.46. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.29 (m, 7H), 7.27 – 7.19 (m, 7H), 6.98 – 6.86 (m, 4H), 3.83 (t, J = 4.0 Hz, 4H), 3.79 (s, 4H), 3.56 (t, J = 3.6 Hz, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -113.99 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.00 (d, J = 248.3 Hz), 139.00, 135.22, 131.20 (d, J = 3.3 Hz), 129.93, 128.50 (d, J = 8.0 Hz), 128.52 (d, J = 23.7 Hz), 127.11, 127.00, 115.15 (d, J = 21.5 Hz), 62.86, 62.68, 60.28. **HRMS** (ESI) calcd for $C_{34}H_{31}F_2N_2^+$ *m/z* 505.2450 $[M+H]^+$, Found 505.2446.



3j

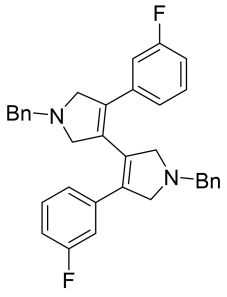
1,1'-dibenzyl-4,4'-bis(4-methoxyphenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrro

le 3j. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 10:1 to 2:1) as a yellow oil (79%, 83.5 mg). R_f (petroleum ether/ethyl acetate = 2:1): 0.53. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 – 7.24 (m, 12H), 7.22 – 7.17 (m, 2H), 6.84 – 6.74 (m, 4H), 3.86 (t, J = 4.0 Hz, 4H), 3.77 (s, 4H), 3.74 (s, 6H), 3.55 (t, J = 3.6 Hz, 4H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 158.91, 139.30, 135.19, 128.88, 128.73, 128.39, 128.19, 127.87, 127.05, 113.73, 62.90, 62.76, 60.46, 55.27. **HRMS** (ESI) calcd for $C_{36}H_{37}N_2O_2^+$ *m/z* 529.2850 $[M+H]^+$, Found 529.2840.



3k

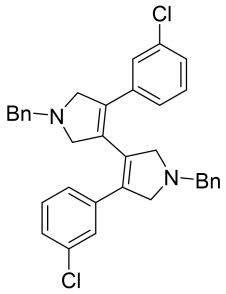
1,1'-dibenzyl-4,4'-di-m-tolyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3k. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (83%, 82.4 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.46. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 – 7.26 (m, 8H), 7.24 – 7.20 (m, 2H), 7.19 – 7.12 (m, 4H), 7.10 (s, 2H), 7.04 – 6.98 (m, 2H), 3.87 (t, J = 3.6 Hz, 4H), 3.77 (s, 4H), 3.57 (t, J = 3.6 Hz, 4H), 2.29 (s, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.39, 137.63, 136.01, 135.22, 130.50, 128.63, 128.35, 128.14, 128.07, 127.59, 126.97, 124.18, 62.95, 62.82, 60.44, 21.54. **HRMS** (ESI) calcd for $C_{36}H_{37}N_2^+$ *m/z* 497.2951 $[M+H]^+$, Found 497.2945.



3l

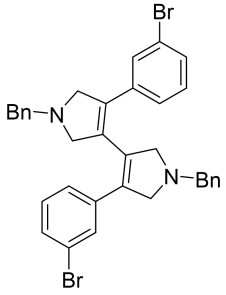
1,1'-dibenzyl-4,4'-bis(3-fluorophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3l. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a white solid (75%, 75.8 mg), mp 109.2-112.5 °C. R_f (petroleum ether/ethyl acetate = 5:1): 0.47. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.31 – 7.19 (m, 8H), 7.17 – 7.12 (m, 2H), 7.12 – 7.04 (m, 2H), 6.96 (dt, J = 8.0, 1.2 Hz, 2H), 6.89 (dt, J = 9.2, 2.4 Hz, 2H), 6.79 (td, J = 8.4, 2.4 Hz, 2H), 3.77 (t, J = 3.6 Hz, 4H), 3.72 (s, 4H), 3.53 (t, J = 4.0 Hz, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -113.27 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.57 (d, J = 246.2 Hz), 138.89, 137.01 (d, J = 7.8 Hz), 135.19 (d, J = 2.5 Hz), 131.19, 129.61 (d, J = 8.5 Hz), 128.51, 128.33, 127.04, 122.46 (d, J = 2.8 Hz), 114.15 (d, J = 21.3

Hz), 113.46 (d, J = 22.1 Hz), 62.66, 62.59, 60.10. **HRMS** (ESI) calcd for $C_{34}H_{31}F_2N_2^+$ *m/z* 505.2450 $[M+H]^+$, Found 505.2441.



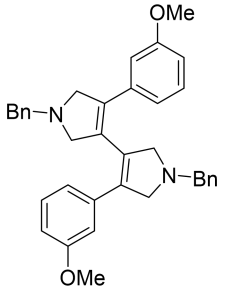
3m

1,1'-dibenzyl-4,4'-bis(3-chlorophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3m. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (82%, 88.1 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.49. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 8H), 7.26 – 7.21 (m, 2H), 7.19 – 7.10 (m, 8H), 3.88 – 3.76 (m, 8H), 3.61 (td, J = 4.0, 1.2 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.01, 136.83, 135.30, 134.07, 131.22, 129.42, 128.59, 128.42, 127.34, 127.12, 126.74, 125.00, 62.80, 62.67, 60.25. **HRMS** (ESI) calcd for $C_{34}H_{31}Cl_2N_2^+$ *m/z* 537.1859 $[M+H]^+$, Found 537.1864.



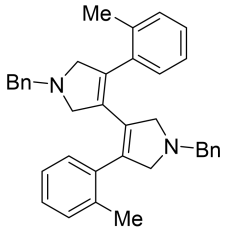
3n

1,1'-dibenzyl-4,4'-bis(3-bromophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3n. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (78%, 97.5 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.44. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 – 7.23 (m, 14H), 7.15 (d, J = 8.0 Hz, 2H), 7.07 (t, J = 8.0 Hz, 2H), 3.85 – 3.75 (m, 8H), 3.61 (t, J = 4.0 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.02, 137.13, 135.26, 131.20, 130.26, 129.70, 129.64, 128.62, 128.44, 127.15, 125.46, 122.34, 62.85, 62.67, 60.28. **HRMS** (ESI) calcd for $C_{34}H_{31}Br_2N_2^+$ *m/z* 625.0849 $[M+H]^+$, Found 625.0842.



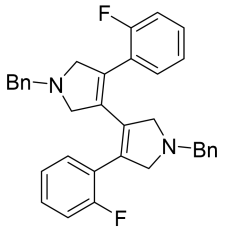
3o

1,1'-dibenzyl-4,4'-bis(3-methoxyphenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3o. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 2:1) as a white solid (65%, 68.8 mg), mp 142.3-143.8 ℃. R_f (petroleum ether/ethyl acetate = 2:1): 0.35. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 8H), 7.25 – 7.14 (m, 4H), 7.00 – 6.86 (m, 4H), 6.76 (dd, J = 9.2, 3.2 Hz, 2H), 3.88 (t, J = 3.6 Hz, 4H), 3.79 (s, 4H), 3.74 (s, 6H), 3.62 (t, J = 4 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.39, 139.22, 136.31, 135.61, 131.02, 129.22, 128.61, 128.34, 127.00, 119.32, 113.47, 111.80, 62.80, 62.70, 60.33, 55.20. **HRMS** (ESI) calcd for $C_{36}H_{37}N_2O_2^+$ *m/z* 529.2850 $[M+H]^+$, Found 529.2857.



3p

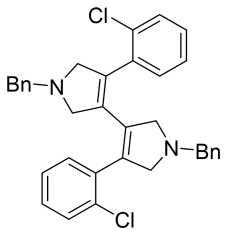
1,1'-dibenzyl-4,4'-di-o-tolyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3p. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (77%, 76.6 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.43. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.19 – 7.12 (m, 5H), 7.12 – 7.07 (m, 6H), 7.07 – 6.93 (m, 7H), 3.51 (s, 4H), 3.47 (t, J = 3.2 Hz, 4H), 3.11 (t, J = 3.6 Hz, 4H), 2.15 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.99, 137.74, 136.43, 136.00, 130.47, 129.75, 128.88, 128.85, 128.26, 127.57, 126.93, 125.41, 64.81, 62.36, 60.45, 19.84. **HRMS** (ESI) calcd for $C_{36}H_{37}N_2^+$ *m/z* 497.2951 $[M+H]^+$, Found 497.2942.



3q

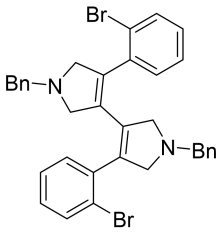
1,1'-dibenzyl-4,4'-bis(2-fluorophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3q. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a white solid (67%, 67.7 mg), mp 146.2-147.7 ℃. R_f (petroleum ether/ethyl acetate = 5:1): 0.41. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.26 – 7.18 (m, 8H), 7.17 – 7.13 (m, 2H), 7.05 – 6.96 (m, 4H), 6.85 (td, J = 7.6, 1.2 Hz, 2H), 6.78 – 6.69 (m, 2H), 3.68 (s, 4H), 3.65 (t, J = 4.0 Hz, 4H), 3.48 (t, J = 3.9 Hz, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -112.69 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.64 (d, J = 249.1 Hz), 139.13, 132.45, 132.08, 129.98 (d, J = 4.3 Hz), 128.90 (d, J = 8.2 Hz), 128.78, 128.38, 127.06, 123.71,

123.68, 123.56, 115.42 (d, J = 22.3 Hz), 63.56 (d, J = 3.5 Hz), 62.33, 60.43. **HRMS** (ESI) calcd for $C_{34}H_{31}F_2N_2^+$ *m/z* 505.2450 $[M+H]^+$, Found 505.2447.



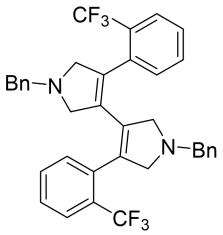
3r

1,1'-dibenzyl-4,4'-bis(2-chlorophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3r. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (65%, 69.8 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.47. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.23 – 7.12 (m, 13H), 7.09 (d, J = 2.4 Hz, 1H), 7.07 (d, J = 2.4 Hz, 1H), 7.05 (d, J = 1.6 Hz, 1H), 7.02 (d, J = 2.4 Hz, 2H), 3.63 – 3.53 (m, 8H), 3.25 (t, J = 3.2 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.82, 135.78, 135.49, 133.11, 131.61, 130.59, 129.35, 128.83, 128.79, 128.26, 126.98, 126.41, 63.93, 62.15, 60.41. **HRMS** (ESI) calcd for $C_{34}H_{31}Cl_2N_2^+$ *m/z* 537.1859 $[M+H]^+$, Found 537.1868.



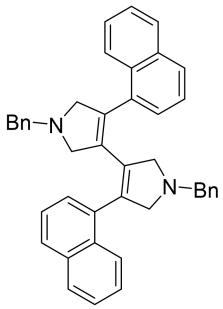
3s

1,1'-dibenzyl-4,4'-bis(2-bromophenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3s. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a white solid (62%, 77.8 mg), mp 112.2-113.5 ℃ R_f (petroleum ether/ethyl acetate = 5:1): 0.38. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 14H), 7.15 (d, J = 8.0 Hz, 2H), 7.07 (t, J = 8.0 Hz, 2H), 3.81 (m, 8H), 3.61 (t, J = 4.0 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.02, 137.13, 135.26, 131.20, 130.26, 129.70, 129.64, 128.62, 128.44, 127.15, 125.46, 122.34, 62.85, 62.67, 60.28. **HRMS** (ESI) calcd for $C_{34}H_{31}Br_2N_2$ *m/z* 627.0831 $[M+H]^+$, Found 627.0835.



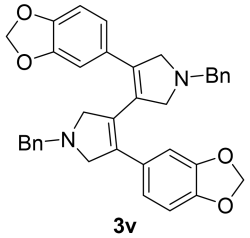
3t

1,1'-dibenzyl-4,4'-bis(2-(trifluoromethyl)phenyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3t. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (70%, 84.7 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.42. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.64 – 7.55 (m, 2H), 7.42 – 7.31 (m, 4H), 7.25 – 7.13 (m, 12H), 3.63 – 3.49 (m, 8H), 3.08 (t, J = 3.6 Hz, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -59.51 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.89, 135.66, 135.35, 131.68, 131.24, 131.00, 128.68, 128.66 (q, J = 31.3Hz), 128.13, 127.62, 126.82, 125.93 (q, J = 5.1Hz), 123.98 (q, J = 274.7Hz), 65.89, 62.45, 60.34. **HRMS** (ESI) calcd for $C_{36}H_{31}F_6N_2^+$ *m/z* 605.2386 $[M+H]^+$, Found 605.2396.



3u

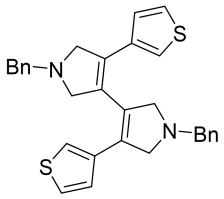
1,1'-dibenzyl-4,4'-di(naphthalen-1-yl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3u. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (57%, 64.8 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.39. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 – 7.80 (m, 4H), 7.73 (d, J = 8.0 Hz, 2H), 7.56 – 7.46 (m, 4H), 7.39 – 7.30 (m, 2H), 7.25 – 7.19 (m, 2H), 7.19 – 7.09 (m, 6H), 7.08 – 7.00 (m, 4H), 3.64 (s, 4H), 3.49 (s, 4H), 3.19 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.76, 136.71, 134.58, 133.37, 131.85, 131.64, 128.71, 128.30, 128.15, 127.70, 126.82, 126.05, 126.01, 125.81, 125.70, 125.10, 65.80, 62.57, 60.29. **HRMS** (ESI) calcd for $C_{42}H_{37}N_2^+$ *m/z* 569.2951 $[M+H]^+$, Found 569.2963.



3v

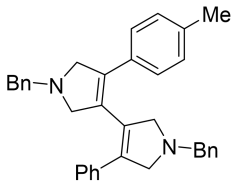
4,4'-bis(benzo[d][1,3]dioxol-5-yl)-1,1'-dibenzyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3v. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (78%, 86.9 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.37. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 – 7.26 (m, 9H), 7.24 – 7.21 (m, 1H), 6.87 – 6.82 (m, 2H), 6.81 – 6.75 (m, 2H), 6.73 – 6.67 (m, 2H), 5.92 (s, 4H), 3.82 (t, J = 3.6 Hz, 4H), 3.78 (s, 4H), 3.57 (t, J = 3.6 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.47, 146.78, 139.20, 135.37, 129.30, 129.15, 128.66, 128.35, 127.01, 120.72, 108.06, 107.25,

100.98, 62.98, 62.76, 60.35. **HRMS** (ESI) calcd for $C_{36}H_{33}N_2O_4^+$ *m/z* 557.2435 $[M+H]^+$, Found 557.2426.



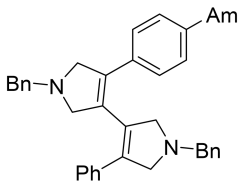
3w

1,1'-dibenzyl-4,4'-di(thiophen-3-yl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3w. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (66%, 63.5 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.36. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 – 7.32 (m, 8H), 7.29 – 7.25 (m, 2H), 7.25 – 7.17 (m, 4H), 7.17 – 7.11 (m, 2H), 3.98 – 3.91 (m, 4H), 3.89 – 3.83 (m, 4H), 3.72 – 3.65 (m, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.28, 135.74, 130.75, 129.37, 128.65, 128.40, 127.06, 126.34, 125.38, 121.95, 62.87, 62.75, 60.42. **HRMS** (ESI) calcd for $C_{30}H_{29}N_2S_2^+$ *m/z* 481.1767 $[M+H]^+$, Found 481.1765.



3x

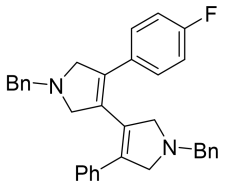
1,1'-dibenzyl-4-phenyl-4'-(p-tolyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3x. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (50%, 48.3 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.35. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.30 – 7.11 (m, 17H), 7.01 (d, J = 8.0 Hz, 2H), 3.82 (q, J = 3.6 Hz, 4H), 3.71 (d, J = 3.6 Hz, 4H), 3.51 (dt, J = 8.0, 3.6 Hz, 4H), 2.24 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.10, 137.27, 135.99, 135.85, 135.18, 132.21, 130.69, 129.54, 129.04, 128.74, 128.40, 128.30, 127.43, 127.09, 126.97, 126.88, 62.90, 62.85, 62.72, 60.41, 60.38, 21.34. **HRMS** (ESI) calcd for $C_{35}H_{35}N_2^+$ *m/z* 483.2795 $[M+H]^+$, Found 483.2788.



3y

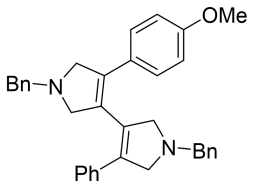
1,1'-dibenzyl-4-(4-pentylphenyl)-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3y. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (66%, 71.1mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.48. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.26 (m, 12H), 7.25 – 7.16 (m, 5H), 7.07 (d, J =

8.0 Hz, 2H), 3.96 – 3.85 (m, 4H), 3.79 (d, *J* = 6.0 Hz, 4H), 3.65 – 3.51 (m, 4H), 2.56 (t, *J* = 7.6 Hz, 2H), 1.64 – 1.53 (m, 2H), 1.36 – 1.25 (m, 4H), 0.89 (t, *J* = 6.8 Hz, 3H). **^{13}C NMR** (75 MHz, $CDCl_3$) *δ* 142.32, 139.29, 135.95, 135.83, 135.15, 132.35, 130.63, 129.40, 128.71, 128.36, 128.31, 128.25, 127.35, 127.04, 126.93, 126.83, 62.85, 62.68, 60.39, 35.71, 31.53, 31.08, 22.57, 14.07. **HRMS** (ESI) calcd for $C_{39}H_{43}N_2^+$ *m/z* 539.3421 $[M+H]^+$, Found 539.3430.



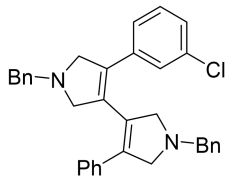
3z

1,1'-dibenzyl-4-(4-fluorophenyl)-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3z. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (75%, 73.1 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.44. **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.37 – 7.26 (m, 11H), 7.25 – 7.16 (m, 6H), 6.95 – 6.90 (m, 2H), 3.92 – 3.81 (m, 4H), 3.81 – 3.76 (m, 4H), 3.61 – 3.52 (m, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) *δ* -113.85 (s), 113.95 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 163.65, 160.37, 139.03 (d, *J* = 7.3 Hz), 136.35, 136.07, 135.26, 135.08, 134.98, 131.29, 131.20, 130.37 (d, *J* = 17.9 Hz), 130.10, 129.95, 128.68, 128.63, 128.58, 128.52, 128.48, 128.43, 128.41, 128.28, 127.46, 127.13 (d, *J* = 4.7 Hz), 126.95, 126.88, 115,17 (d, *J* = 28.6 Hz), 62.89, 62.87, 62.75, 62.71, 60.34, 60.30. **HRMS** (ESI) calcd for $C_{34}H_{32}FN_2^+$ *m/z* 487.2544 $[M+H]^+$, Found 487.2533.



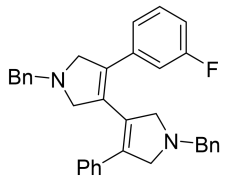
3aa

1,1'-dibenzyl-4-(4-methoxyphenyl)-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3aa. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (63%, 62.8 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.28. **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.30 – 7.11 (m, 17H), 6.73 (d, *J* = 8.4 Hz, 2H), 3.82 (dt, *J* = 9.6, 4.0 Hz, 4H), 3.71 (d, *J* = 6.8 Hz, 4H), 3.68 (s, 3H), 3.51 (dt, *J* = 10.0, 3.6 Hz, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 158.97, 139.20, 135.80, 135.54, 135.23, 130.82, 128.75, 128.57, 128.43, 128.33, 128.25, 127.78, 127.42, 127.11, 126.95, 113.77, 62.93, 62.86, 62.76, 60.45, 60.41, 55.29. **HRMS** (ESI) calcd for $C_{35}H_{35}N_2O^+$ *m/z* 499.2744 $[M+H]^+$, Found 499.2753.



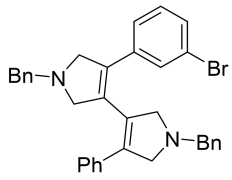
3ab

1,1'-dibenzyl-4-(3-chlorophenyl)-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ab. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (60%, 60.4 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.39. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.28 – 7.01 (m, 19H), 3.81 (t, J = 3.6 Hz, 2H), 3.75 (t, J = 4.0 Hz, 2H), 3.71 (d, J = 9.6 Hz, 4H), 3.51 (m, 4H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.22, 139.07, 137.00, 136.88, 136.83, 135.35, 135.07, 134.61, 134.12, 132.10, 131.28, 129.81, 129.47, 128.67, 128.47, 128.43, 128.30, 127.51, 127.40, 127.29, 127.17, 127.13, 127.09, 126.90, 126.85, 126.81, 125.10, 125.05, 62.95, 62.86, 62.73, 62.67, 60.38, 60.29. **HRMS** (ESI) calcd for $C_{34}H_{32}ClN_2^+$ *m/z* 503.2249 $[M+H]^+$, Found 503.2239.



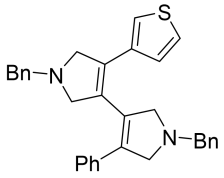
3ac

1,1'-dibenzyl-4-(3-fluorophenyl)-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ac. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (70%, 68.2 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.42. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.25 (m, 11H), 7.24 – 7.13 (m, 4H), 7.12 – 6.90 (m, 3H), 6.87 (td, J = 8.4, 2.4 Hz, 1H), 3.87 (dt, J = 17.6, 4.0 Hz, 4H), 3.79 (d, J = 6.0 Hz, 4H), 3.64 – 3.55 (m, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -113.18 (s), -113.28 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.63 (d, J = 243.8 Hz), 139.07 (d, J = 9.0 Hz), 137.23 (d, J = 7.5 Hz), 136.55, 135.23 (d, J = 2.3 Hz), 134.96, 134.66 (d, J = 2.3 Hz), 131.91, 131.27, 129.83, 129.63 (d, J = 8.3 Hz), 129.57 (d, J = 8.3 Hz), 128.57, 128.32, 128.20, 127.39, 127.01, 126.98, 126.87, 126.78, 122.56 (d, J = 3.0 Hz), 122.49 (d, J = 3.0 Hz), 114.16 (d, J = 21.0 Hz), 114.04 (d, J = 21.0 Hz), 113.56 (d, J = 22.5 Hz), 113.51 (d, J = 21.8 Hz), 62.84, 62.79, 62.73, 62.67, 60.26, 60.24, 60.17. **HRMS** (ESI) calcd for $C_{34}H_{32}FN_2^+$ *m/z* 487.2544 $[M+H]^+$, Found 487.2543.



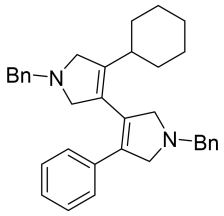
3ad

1,1'-dibenzyl-4-(3-bromophenyl)-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ad. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (59%, 64.6 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.39. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.18 (m, 17H), 7.14 – 7.01 (m, 2H), 3.93 – 3.73 (m, 8H), 3.65 – 3.53 (m, 4H). **^{13}C NMR (**75 MHz, $CDCl_3$**)** δ 139.17, 139.02, 137.26, 137.14, 136.86, 135.29, 135.03, 134.50, 132.11, 131.21, 130.29, 130.19, 129.72, 129.67, 128.67, 128.46, 128.43, 128.42, 128.29, 127.51, 127.17, 127.13, 127.09, 126.89, 125.52, 125.48, 122.36, 62.93, 62.86, 62.82, 62.69, 62.62, 60.40, 60.30. **HRMS** (ESI) calcd for $C_{34}H_{32}BrN_2^+$ *m/z* 547.1743 $[M+H]^+$, Found 547.1740.



3ae

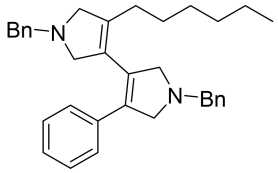
1,1'-dibenzyl-4-phenyl-4'-(thiophen-3-yl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ae. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (64%, 60.8 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.46. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.32 – 7.20 (m, 10H), 7.20 – 7.13 (m, 5H), 7.13 – 7.07 (m, 2H), 7.07 – 7.02 (m, 1H), 3.87 (t, J = 3.6 Hz, 2H), 3.81 (t, J = 4.4 Hz, 2H), 3.77 (s, 2H), 3.74 – 3.68 (m, 2H), 3.60 (t, J = 4.0 Hz, 2H), 3.57 – 3.49 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.32, 139.27, 136.07, 135.89, 135.77, 135.52, 135.21, 134.98, 131.11, 130.79, 130.59, 129.40, 128.71, 128.46, 128.45, 128.35, 127.44, 127.13, 127.10, 126.98, 126.75, 126.57, 126.39, 125.48, 122.03, 63.08, 62.93, 62.84, 62.81, 62.65, 60.48, 60.41. **HRMS** (ESI) calcd for $C_{32}H_{31}N_2S^+$ *m/z* 475.2202 $[M+H]^+$, Found 475.2199.



3af

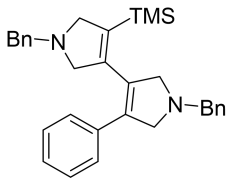
4-([1,1'-biphenyl]-4-yl)-1,1'-dibenzyl-4'-cyclohexyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3af. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a colorless oil (76%, 93.0 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.44. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.17 (m, 15H), 3.88 (t, J = 4.0 Hz, 2H), 3.84 (s, 2H), 3.73 (s, 2H), 3.67 (t, J = 4.0 Hz, 2H), 3.51 – 3.44 (m, 2H), 3.43 – 3.36 (m, 2H), 1.58 – 1.46 (m, 3H), 1.23 – 1.15 (m, 2H), 1.05 – 0.90 (m, 5H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 142.26, 139.41, 139.21, 135.81, 134.76, 130.22, 128.83, 128.79, 128.44, 128.33, 128.19, 127.22, 127.14, 127.12, 126.96, 125.96, 64.47, 63.24, 62.19, 60.84,

60.62, 59.76, 37.76, 30.99, 26.29, 26.03. **HRMS** (ESI) calcd for $C_{34}H_{39}N_2^+$ *m/z* 475.3113 $[M+H]^+$, Found 475.3115.



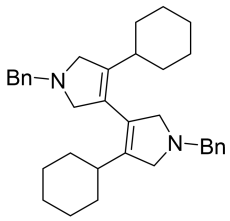
3ag

1,1'-dibenzyl-4-hexyl-4'-phenyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ag. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (68%, 64.0 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.47. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 13H), 7.25 – 7.23 (m, 2H), 3.88 (t, J = 4.0 Hz, 2H), 3.83 (s, 2H), 3.74 (s, 2H), 3.67 (t, J = 4.0 Hz, 2H), 3.51 – 3.47 (m, 2H), 3.43 – 3.38 (m, 2H), 1.84 – 1.76 (m, 2H), 1.24 – 1.14 (m, 3H), 1.09 – 1.02 (m, 5H), 0.85 – 0.80 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.41, 139.24, 137.85, 135.77, 134.76, 130.01, 128.79, 128.72, 128.41, 128.32, 128.14, 127.39, 127.17, 127.09, 127.06, 126.94, 64.21, 63.20, 62.66, 62.21, 60.70, 60.59, 31.62, 29.41, 28.26, 27.36, 22.57, 14.13. **HRMS** (ESI) calcd for $C_{34}H_{41}N_2^+$ *m/z* 477.3264 $[M+H]^+$, Found 477.3251.



3ah

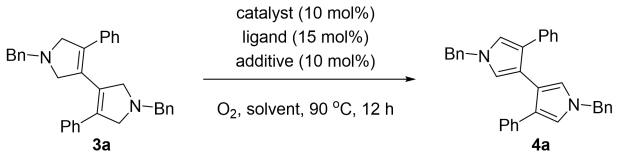
1,1'-dibenzyl-4-phenyl-4'-(trimethylsilyl)-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ah. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (72%, 66.9 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.45. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.47 – 7.28 (m, 15H), 4.02 – 3.95 (m, 2H), 3.93 (s, 2H), 3.89 (s, 2H), 3.80 – 3.59 (m, 6H), -0.03 (s, 9H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 144.66, 139.55, 139.36, 136.78, 135.04, 134.21, 132.53, 128.81, 128.75, 128.72, 128.44, 128.40, 128.25, 127.23, 127.11, 127.01, 65.38, 65.33, 64.13, 62.79, 60.55, 60.53, -1.52. **HRMS** (ESI) calcd for $C_{31}H_{37}N_2Si^+$ *m/z* 465.2726 $[M+H]^+$, Found 465.2730.



3ai

1,1'-dibenzyl-4,4'-dicyclohexyl-2,2',5,5'-tetrahydro-1H,1'H-3,3'-bipyrrole 3ai. The product was purified by flash column chromatography on silica gel (height 23 cm, width 1.7 cm, eluent: petroleum ether/ethyl acetate, gradient: 20:1 to 5:1) as a yellow oil (72%, 66.9 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.32. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.46 – 7.37 (m, 10H), 3.85 – 3.74 (m, 6H), 3.57 – 3.48 (m, 8H), 2.90 (s, 2H), 1.82 – 1.58 (m, 10H), 1.35 – 1.16 (m, 10H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 141.00, 139.49, 128.90, 128.64, 128.37, 128.33, 127.01, 126.20, 63.33, 61.00, 59.68, 59.57, 52.44, 37.51, 31.68, 26.38, 26.12. **HRMS** (ESI) calcd for $C_{34}H_{44}N_2Na^+$ *m/z* 503.3397 $[M+H]^+$, Found 503.3389.

6. Optimization of Dehydrogenation Reaction Conditions.[a]

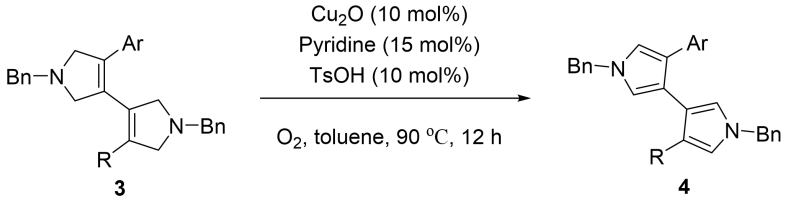


Entry	Cat. (X mol%)	Add. (Y mol%)	Sol./Temp.(°C)/time (h)	Yield(%)[b]
1	CuI (10)	Pyridine (15)/TsOH (10)	toluene/90/12	62
2	CuCl (10)	Pyridine (15)/TsOH (10)	toluene/90/12	59
3	CuBr (10)	Pyridine (15)/TsOH (10)	toluene/90/12	55
4	Cu_2O (10)	Pyridine (15)/TsOH (10)	toluene/90/12	79
5	CuCN (10)	Pyridine (15)/TsOH (10)	toluene/90/12	60
6	$CuCl_2$ (10)	Pyridine (15)/TsOH (10)	toluene/90/12	49
7	$Cu(OAc)_2$ (10)	Pyridine (15)/TsOH (10)	toluene/90/12	38
8	$Cu(MeCN)_4PF_6$ (10)	Pyridine (15)/TsOH (10)	toluene/90/12	28
9	CuF_2 (10)	Pyridine (15)/TsOH (10)	toluene/90/12	37
10	Cu_2O (10)	Pyridine (15)/TsOH (10)	*o*-xylene/90/12	69
11	Cu_2O (10)	Pyridine (15)/TsOH (10)	PhCl/90/12	66
12	Cu_2O (10)	Pyridine (15)/TsOH (10)	1,4-Dioxane/90/12	45
13	Cu_2O (10)	Pyridine (15)/TsOH (10)	DMF/90/12	47
14	Cu_2O (10)	Pyridine (15)/TsOH (10)	NMP/90/12	64
15	Cu_2O (10)	Pyridine (15)/TsOH (10)	MeCN/90/12	60
16	Cu_2O (10)	Pyridine (15)/TsOH (10)	DMSO/90/12	51
17	Cu_2O (10)	2,2'-bpy (15)/TsOH (10)	toluene/90/12	44
18	Cu_2O (10)	1,10-phen (15)/TsOH (10)	toluene/90/12	29
19	Cu_2O (10)	2,2'-bipyrimidine(15) /TsOH (10)	toluene/90/12	18
20	Cu_2O (10)	triethylamine (15)/TsOH (10)	toluene/90/12	33
21	Cu_2O (10)	Pyridine (15)/TsOH (10)	toluene/80/12	61

22	Cu_2O (10)	Pyridine (15)/TsOH (10)	toluene/70/12	40
23	Cu_2O (10)	Pyridine (15)/AcOH (10)	toluene/90/12	37
24	Cu_2O (10)	Pyridine (15)/TFA (10)	toluene/90/12	45
25	Cu_2O (10)	Pyridine (15)/CF_3SO_3H (10)	toluene/90/12	16
26[c]	Cu_2O (10)	Pyridine (15)/TsOH (10)	toluene/90/12	58

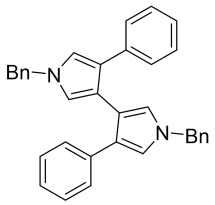
Reaction conditions: [a]tetrahydrobipyrrole **3a** (0.2 mmol, 1.0 equiv.), copper salt (0.02 mmol, 10 mol%), ligand (0.03 mmol, 15 mol%), acid additive (0.02 mmol, 10 mol%), in solvent (2 mL), under O_2 (1 atm), at indicated temperature for 12 h. [b]Isolated yield. [c]under air.

7. General procedure for the synthesis of derivatives 4.



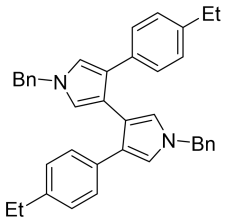
An oven-dried 25 mL reaction tube equipped with a stirring bar was charged with derivatives **3** (0.20 mmol, 1.0 equiv.), Cu_2O (2.9 mg, 0.02 mmol, 0.1 equiv.), pyridine (2.4 mg, 0.03 mmol, 0.15 equiv.), TsOH • H_2O (3.4 mg, 0.02 mmol, 0.1 equiv.) and toluene (2 mL). The tube was evacuated and filled with 1 atm O_2, and stirred rigorously at 90 °C for 12 h. After reaction completed, the mixture was cooled down to room temperature, and the volatiles were removed under reduced pressure. The residue was purified by flash chromatography on silica gel to afford the corresponding products **4**.

8. Purification and characterization of derivatives 4.



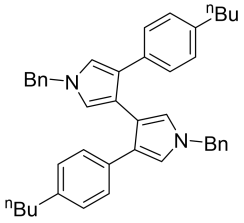
4a

1,1'-dibenzyl-4,4'-diphenyl-1H,1'H-3,3'-bipyrrole 4a. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 50:1) as a yellow oil (79%, 73.3 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.36. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.22 (m, 10H), 7.18 – 7.01 (m, 10H), 6.83 (s, 2H), 6.50 (s, 2H), 4.99 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.11, 136.23, 128.60, 127.83, 127.51, 127.37, 126.83, 124.93, 124.72, 122.00, 119.19, 116.26, 53.32. **HRMS** (ESI) calcd for $C_{34}H_{29}N_2^+$ *m/z* 465.2331 $[M+H]^+$, Found 465.2322.



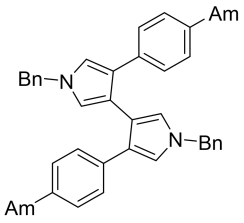
4b

1,1'-dibenzyl-4,4'-bis(4-ethylphenyl)-1H,1'H-3,3'-bipyrrole 4b. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (75%, 78.0 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.34. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 10H), 7.17 (d, J = 6.8 Hz, 4H), 7.05 (d, J = 8.0 Hz, 4H), 6.88 (d, J = 2.4 Hz, 2H), 6.56 (d, J = 2.4 Hz, 2H), 5.04 (s, 4H), 2.64 (q, J = 7.6 Hz, 4H), 1.26 (t, J = 7.2 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.84, 138.38, 133.72, 128.65, 127.55, 127.48, 127.43, 126.92, 124.86, 122.07, 119.06, 116.42, 53.39, 28.59, 15.75. **HRMS** (ESI) calcd for $C_{38}H_{37}N_2^+$ *m/z* 521.2957 $[M+H]^+$, Found 521.2950.



4c

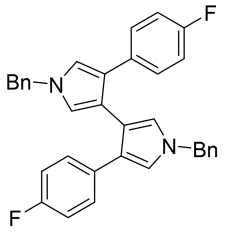
1,1'-dibenzyl-4,4'-bis(4-butylphenyl)-1H,1'H-3,3'-bipyrrole 4c. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (68%, 78.4 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.42. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.31 (m, 10H), 7.19 (d, J = 6.8 Hz, 4H), 7.05 (d, J = 8.0 Hz, 4H), 6.89 (d, J = 2.4 Hz, 2H), 6.58 (d, J = 2.4 Hz, 2H), 5.06 (s, 4H), 2.62 (t, J = 7.6 Hz, 4H), 1.69 – 1.61 (m, 4H), 1.46 – 1.39 (m, 4H), 1.01 (t, J = 7.2 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.48, 138.42, 133.70, 128.67, 128.04, 127.55, 127.36, 126.94, 124.88, 122.04, 119.08, 116.48, 53.40, 35.41, 33.81, 22.49, 14.13. **HRMS** (ESI) calcd for $C_{42}H_{45}N_2^+$ *m/z* 577.3583 $[M+H]^+$, Found 577.3580.



4d

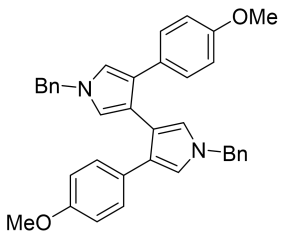
1,1'-dibenzyl-4,4'-bis(4-pentylphenyl)-1H,1'H-3,3'-bipyrrole 4d. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (64%, 77.4 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.39. **^{1}H NMR** (400 MHz, $CDCl_3$)

δ 7.37 – 7.27 (m, 10H), 7.16 (d, J = 6.8 Hz, 4H), 7.01 (d, J = 7.6 Hz, 4H), 6.86 (d, J = 2.4 Hz, 2H), 6.55 (d, J = 2.4 Hz, 2H), 5.04 (s, 4H), 2.57 (t, J = 8.0 Hz , 4H), 1.65 – 1.59 (m, 4H), 1.40 – 1.33 (m, 8H), 0.94 (t, J = 6.8 Hz, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.51, 138.37, 133.65, 128.64, 127.99, 127.52, 127.31, 126.90, 124.85, 122.02, 119.03, 116.42, 53.38, 35.67, 31.63, 31.30, 22.65, 14.13. **HRMS** (ESI) calcd for $C_{44}H_{49}N_2^+$ *m/z* 605.3896 $[M+H]^+$, Found 605.3898.



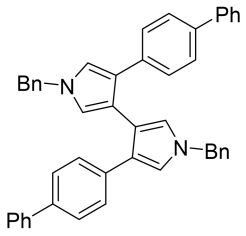
4e

1,1'-dibenzyl-4,4'-bis(4-fluorophenyl)-1H,1'H-3,3'-bipyrrole 4e. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (76%, 76.0 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.34. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 6H), 7.20 – 7.13 (m, 8H), 6.85 – 6.77 (m, 6H), 6.53 (d, J = 2.4 Hz, 2H), 5.03 (s, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -118.6 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.91 (d, J = 244.3 Hz), 137.94, 132.20 (d, J = 3.4 Hz), 128.80 (d, J = 7.5 Hz), 128.67, 127. 68, 126.93, 123.80, 121.59, 119.06, 116.18, 114.48 (d, J = 21.1 Hz), 53.40. **HRMS** (ESI) calcd for $C_{34}H_{27}N_2F_2^+$ *m/z* 501.2142 $[M+H]^+$, Found 501.2133.



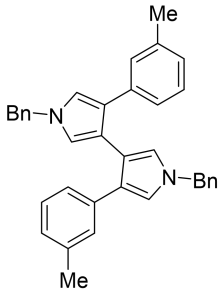
4f

1,1'-dibenzyl-4,4'-bis(4-methoxyphenyl)-1H,1'H-3,3'-bipyrrole 4f. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 10:1) as a yellow oil (68%, 71.3 mg). R_f (petroleum ether/ethyl acetate = 10:1): 0.31. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.34 – 7.19 (m, 11H), 7.11 (d, J = 7.2 Hz, 4H), 6.75 (d, J = 2.4 Hz, 2H), 6.73 – 6.66 (m, 4H), 6.47 (d, J = 2.4 Hz, 2H), 4.97 (s, 4H), 3.74 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.38, 138.32, 129.10, 128.65, 128.60, 127.56, 126.96, 124.46, 121.87, 118.67, 116.26, 113.43, 55.24, 53.37. **HRMS** (ESI) calcd for $C_{36}H_{33}N_2O_2^+$ *m/z* 525.2542 $[M+H]^+$, Found 525.2538.



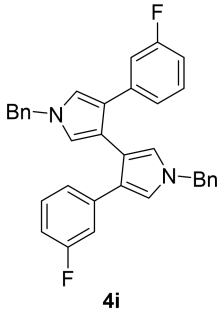
4g

4,4'-di([1,1'-biphenyl]-4-yl)-1,1'-dibenzyl-1H,1'H-3,3'-bipyrrole 4g. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 50:1) as a yellow oil (54%, 66.6 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.23. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 – 7.52 (m, 4H), 7.43 – 7.36 (m, 12H), 7.34 – 7.25 (m, 8H), 7.17 (d, J = 6.4 Hz, 4H), 6.92 (d, J = 2.4 Hz, 2H), 6.59 (d, J = 2.4 Hz, 2H), 5.05 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.29, 138.05, 137.59, 135.41, 128.65, 128.61, 127.64, 127.61, 126.95, 126.78, 126.72, 126.58, 124.29, 122.09, 119.35, 116.44, 53.44. **HRMS** (ESI) calcd for $C_{46}H_{37}N_2^+$ *m/z* 617.2957 $[M+H]^+$, Found 617.2952.



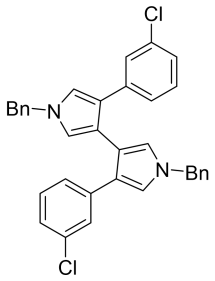
4h

1,1'-dibenzyl-4,4'-di-m-tolyl-1H,1'H-3,3'-bipyrrole 4h. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (75%, 73.8 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.34. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.35 – 7.23 (m, 6H), 7.13 (d, J = 8.0 Hz, 8H), 7.04 (t, J = 7.6 Hz, 2H), 6.92 – 6.78 (m, 4H), 6.53 (s, 2H), 5.01 (s, 4H), 2.22 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.36, 137.27, 136.34, 128.72, 128.29, 127.83, 127.59, 126.89, 125.81, 124.98, 124.72, 122.04, 119.26, 116.47, 53.40, 21.57. **HRMS** (ESI) calcd for $C_{36}H_{33}N_2^+$ *m/z* 493.2644 $[M+H]^+$, Found 493.2635.



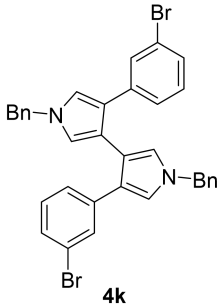
4i

1,1'-dibenzyl-4,4'-bis(3-fluorophenyl)-1H,1'H-3,3'-bipyrrole 4i. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (71%, 71.0 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.42. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.25 (m, 6H), 7.16 (d, J = 7.6 Hz, 4H), 7.10 – 7.00 (m, 4H), 6.95 (d, J = 11.2 Hz, 2H), 6.88 (s, 2H), 6.73 (t, J = 10.0 Hz, 2H), 6.58 (s, 2H), 5.06 (s, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -114.5 (s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.67 (d, J = 243.8 Hz), 138.42 (d, J = 8.6 Hz), 137.84, 129.08 (d, J = 8.8 Hz), 128.74, 127.68, 126.81, 123.61 (d, J = 2.4 Hz), 122.71 (d, J = 2.7 Hz), 122.00, 119.71, 116.15, 113.78 (d, J = 21.9 Hz), 111.57 (d, J = 21.2 Hz), 53.43. **HRMS** (ESI) calcd for $C_{34}H_{27}F_2N_2^+$ *m/z* 501.2142 $[M+H]^+$, Found 501.2134.



4j

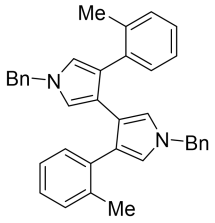
1,1'-dibenzyl-4,4'-bis(3-chlorophenyl)-1H,1'H-3,3'-bipyrrole 4j. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (72%, 76.8 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.46. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.27 (m, 6H), 7.23 – 7.12 (m, 6H), 7.09 – 6.99 (m, 6H), 6.84 (d, J = 2.4 Hz, 2H), 6.59 (d, J = 2.4 Hz, 2H), 5.06 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.05, 137.89, 133.55, 128.97, 128.82, 127.73, 127.25, 126.89, 125.42, 124.88, 123.49, 121.79, 119.77, 116.21, 53.49. **HRMS** (ESI) calcd for $C_{34}H_{27}Cl_2N_2^+$ *m/z* 533.1551 $[M+H]^+$, Found 533.1541.



4k

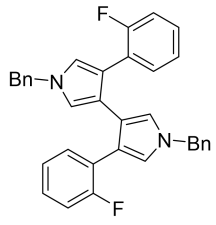
1,1'-dibenzyl-4,4'-bis(3-bromophenyl)-1H,1'H-3,3'-bipyrrole 4k. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (58%, 71.9 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.47. **^{1}H NMR** (400 MHz, $CDCl_3$)

δ 7.40 – 7.21 (m, 8H), 7.21 – 7.01 (m, 8H), 6.92 (t, J = 8.0 Hz, 2H), 6.81 (s, 2H), 6.58 (s, 2H), 5.05 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.32, 137.88, 130.18, 129.23, 128.83, 127.76, 127.73, 126.91, 125.90, 123.38, 121.93, 121.67, 119.77, 116.21, 53.49. **HRMS** (ESI) calcd for $C_{34}H_{27}Br_2N_2^+$ *m/z* 621.0541 $[M+H]^+$, Found 621.0541.



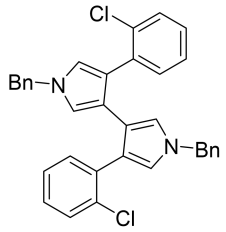
4l

1,1'-dibenzyl-4,4'-di-o-tolyl-1H,1'H-3,3'-bipyrrole 4l. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (73%, 71.9 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.18. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 – 7.28 (m, 6H), 7.22 – 7.05 (m, 12H), 6.51 (d, J = 2.4 Hz, 2H), 6.16 (d, J = 2.4 Hz, 2H), 4.91 (s, 4H), 2.16 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.53, 137.32, 136.77, 131.23, 129.67, 128.60, 127.44, 126.87, 126.32, 125.24, 123.01, 119.76, 118.83, 118.11, 53.22, 20.60. **HRMS** (ESI) calcd for $C_{36}H_{33}N_2^+$ *m/z* 493.2644 $[M+H]^+$, Found 493.2635.



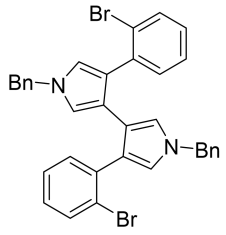
4m

1,1'-dibenzyl-4,4'-bis(2-fluorophenyl)-1H,1'H-3,3'-bipyrrole 4m. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (73%, 70.0 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.47. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 – 7.24 (m, 8H), 7.17 (d, J = 6.8 Hz, 4H), 7.08 – 7.01 (m, 2H), 7.00 – 6.88 (m, 6H), 6.57 (d, J = 2.8 Hz, 2H), 5.06 (s, 4H). **^{19}F NMR** (376 MHz, $CDCl_3$) δ -115.48(s). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 159.74 (d, J = 246.3 Hz), 138.12, 130.9 (d, J = 3.9 Hz), 128.71, 127.63, 126.98, 126.45 (d, J = 8.3 Hz), 123.80 (d, J = 14.3 Hz), 123.25 (d, J = 3.5 Hz), 121.82 (d, J = 7.1 Hz), 120.79, 117.40, 116.85, 115.17 (d, J = 23.2 Hz), 53.45. **HRMS** (ESI) calcd for $C_{34}H_{27}F_2N_2^+$ *m/z* 501.2142 $[M+H]^+$, Found 501.2139.



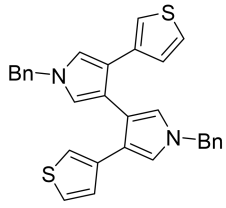
4n

1,1'-dibenzyl-4,4'-bis(2-chlorophenyl)-1H,1'H-3,3'-bipyrrole 4n. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 50:1) as a yellow oil (65%, 69.1 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.32. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 – 7.08 (m, 8H), 7.08 – 7.02 (m, 2H), 7.00 – 6.94 (m, 4H), 6.94 – 6.88 (m, 4H), 6.64 (d, J = 2.4 Hz, 2H), 6.23 (d, J = 2.4 Hz, 2H), 4.84 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.28, 135.20, 133.35, 132.42, 129.41, 128.66, 127.53, 126.88, 126.09, 121.44, 120.48, 119.62, 117.94, 53.36, one carbon was overlapped. **HRMS** (ESI) calcd for $C_{34}H_{27}Cl_2N_2^+$ *m/z* 533.1551 $[M+H]^+$, Found 533.1546.



4o

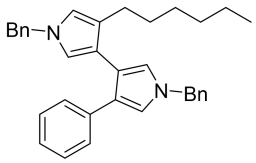
1,1'-dibenzyl-4,4'-bis(2-bromophenyl)-1H,1'H-3,3'-bipyrrole 4o. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 50:1) as a yellow oil (63%, 78.1 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.28. **^{1}H NMR (**400 MHz, $CDCl_3$) δ 7.49 – 7.42 (m, 2H), 7.34 – 7.22 (m, 6H), 7.21 – 7.14 (m, 2H), 7.14 – 7.03 (m, 6H), 7.01 – 6.91 (m, 2H), 6.74 – 6.63 (m, 2H), 6.31 – 6.20 (m, 2H), 4.95 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.30, 137.39, 132.61, 128.61, 127.46, 127.30, 126.82, 126.75, 124.41, 122.34, 121.13, 119.46, 117.73, 53.31. **HRMS** (ESI) calcd for $C_{34}H_{27}Br_2N_2^+$ *m/z* 621.0541 $[M+H]^+$, Found 621.0545.



4p

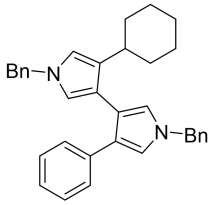
1,1'-dibenzyl-4,4'-di(thiophen-3-yl)-1H,1'H-3,3'-bipyrrole 4p. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 50:1) as a yellow oil (72%, 68.7 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.25. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.27 – 7.13 (m, 6H), 7.09 – 7.02 (m, 4H), 7.02 – 6.93 (m, 4H), 6.86 – 6.75 (m, 4H),

6.48 (d, J = 2.4 Hz, 2H), 4.91 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.14, 136.47, 128.79, 127.74, 127.06, 127.03, 124.20, 122.26, 120.79, 118.91, 117.95, 116.53, 53.48. **HRMS** (ESI) calcd for $C_{30}H_{25}S_2N_2^+$ *m/z* 477.1459 $[M+H]^+$, Found 477.1452.



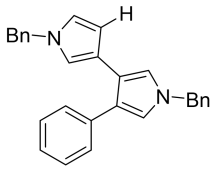
4q

1,1'-dibenzyl-4-hexyl-4'-phenyl-1H,1'H-3,3'-bipyrrole 4q The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (75%, 70.8 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.52. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 8H), 7.23 – 7.18 (m, 4H), 7.13 – 7.07 (m, 3H), 6.84 (d, J = 2.4 Hz, 1H), 6.65 (d, J = 2.4 Hz, 1H), 6.44 (s, 2H), 5.09 (s, 2H), 4.96 (s, 2H), 2.25 (t, J = 8.0 Hz, 2H), 1.44 – 1.35 (m, 2H), 1.24 – 1.16 (m, 6H), 0.84 (t, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.85, 138.03, 136.67, 128.67, 128.51, 127.86, 127.61, 127.43, 127.29, 127.13, 126.71, 124.99, 124.34, 124.24, 121.11, 120.30, 119.33, 118.43, 117.38, 116.91, 53.43, 53.11, 31.68, 30.32, 29.29, 25.83, 22.63, 14.10. **HRMS** (ESI) calcd for $C_{34}H_{37}N_2^+$ *m/z* 473.2957 $[M+H]^+$, Found 473.2951.



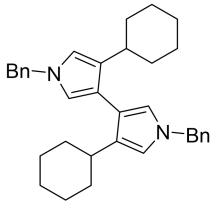
4r

1,1'-dibenzyl-4-cyclohexyl-4'-phenyl-1H,1'H-3,3'-bipyrrole 4r. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (72%, 67.7 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.31. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.13 (m, 12H), 7.13 – 7.00 (m, 3H), 6.83 (d, J = 2.4 Hz, 1H), 6.64 (d, J = 2.0 Hz, 1H), 6.48 – 6.29 (m, 2H), 5.07 (s, 2H), 4.93 (s, 2H), 2.41 – 2.29 (m, 1H), 1.84 – 1.71 (m, 2H), 1.68 – 1.54 (m, 3H), 1.23 – 1.04 (m, 5H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.83, 138.23, 136.58, 130.45, 128.66, 128.47, 127.82, 127.54, 127.43, 127.24, 126.88, 126.66, 124.93, 124.42, 121.22, 120.40, 119.28, 117.03, 116.83, 116.56, 53.35, 53.12, 35.26, 34.56, 26.94, 26.42. **HRMS** (ESI) calcd for $C_{34}H_{35}N_2^+$ *m/z* 471.2800 $[M+H]^+$, Found 471.2792.



4s

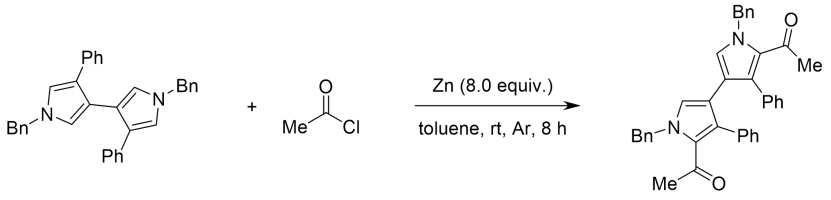
1,1'-dibenzyl-4-phenyl-1H,1'H-3,3'-bipyrrole 4s. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (78%, 71.8 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.42. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.50 – 7.31 (m, 10H), 7.27 – 7.12 (m, 5H), 6.78 (s, 2H), 6.63 (s, 1H), 6.55 (s, 1H), 6.17 (s, 1H), 5.10 (s, 2H), 5.01 (s, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 138.44, 137.83, 136.64, 128.76, 128.63, 128.57, 127.91, 127.75, 127.49, 127.40, 126.92, 125.48, 123.41, 120.82, 120.26, 119.44, 118.73, 118.53, 117.98, 108.67, 53.49, 53.30. **HRMS** (ESI) calcd for $C_{28}H_{25}N_2^+$ m/z 389.2018 $[M+H]^+$, Found 389.2015.



4t

1,1'-dibenzyl-4,4'-dicyclohexyl-1H,1'H-3,3'-bipyrrole 4t. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (78%, 71.8 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.51. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.41 – 7.30 (m, 6H), 7.19 – 7.12 (m, 4H), 6.61 (d, J = 2.4 Hz, 2H), 6.51 (d, J = 2.4 Hz, 2H), 5.09 (s, 4H), 2.58 – 2.46 (m, 2H), 1.97 – 1.87 (m, 4H), 1.79 – 1.66 (m, 7H), 1.30 – 1.21 (m, 9H). **^{13}C NMR** (75 MHz, CDCl3) δ 139.02, 130.38, 128.61, 127.33, 126.76, 119.82, 117.11, 116.84, 53.25, 35.28, 34.88, 27.06, 26.54. **HRMS** (ESI) calcd for $C_{34}H_{40}N_2Na^+$ m/z 499.3084 $[M+H]^+$, Found499.3092.

9. Procedure for the synthesis of derivative 5.

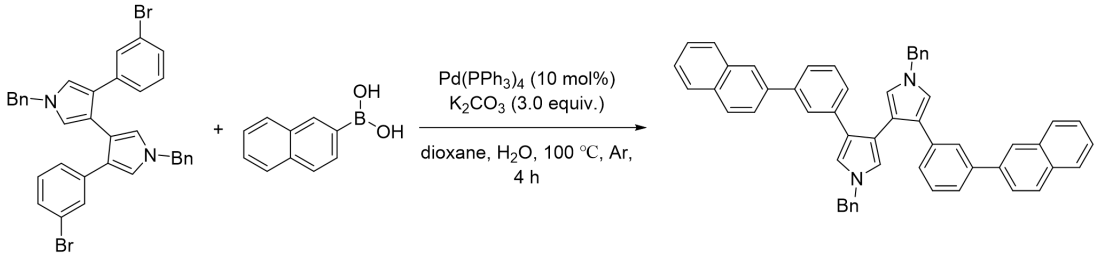


4a 5

An oven-dried 25 mL Schlenk tube equipped with a stirring bar was charged with **4a** (0.2 mmol, 1.0 equiv.), acetyl chloride (94.2 mg, 1.2 mmol, 6.0 equiv.), zinc powder (104.6 mg, 1.6 mmol, 8.0 equiv.), in toluene (2 mL) under Ar, at room temperature for 5 h. After reaction completed, the volatiles were removed under reduced pressure. The residue was purified by flash column chromatography on silica gel to afford the corresponding product **5**[2].

1,1'-(1,1'-dibenzyl-4,4'-diphenyl-1H,1'H-[3,3'-bipyrrole]-5,5'-diyl)bis(ethan-1-one) 5. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a white solid (37%, 40 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.38. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.37 – 7.32 (m, 6H), 7.26 – 7.19 (m, 10H), 6.95 – 6.86 (m, 4H), 6.15 (s, 2H), 5.30 (s, 4H), 1.77 (s, 6H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 190.19, 137.99, 136.83, 133.53, 130.61, 128.46, 128.43, 128.11, 127.89, 127.49, 127.35, 127.30, 116.66, 53.03, 30.32. **HRMS** (ESI) calcd for $C_{38}H_{33}N_2O_2^+$ m/z 549.2542 $[M+H]^+$, Found 549.2545.

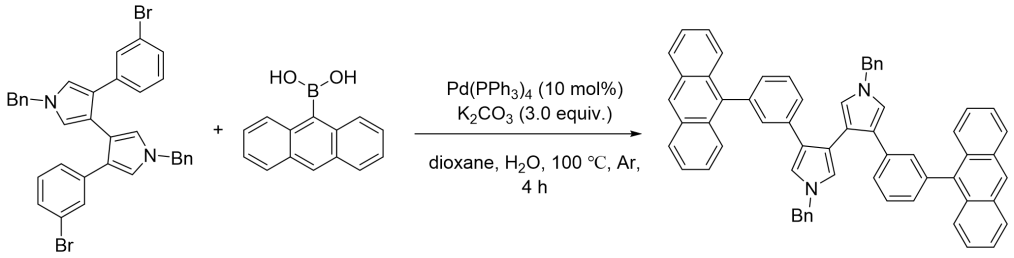
10.General procedure for the synthesis of derivatives 6.



4k **6a**

An oven-dried 25 mL Schlenk tube equipped with a stirring bar was charged with **4k** (124.0 mg, 0.2 mmol, 1.0 equiv.), 2-naphthaleneboronic acid (75.7 mg, 0.44 mmol, 2.2 equiv.), $Pd(PPh_3)_4$ (23.1 mg, 0.02 mmol, 10 mol%) and K_2CO_3 (82.9 mg, 0.6 mmol, 3.0 equiv.) in dioxane/H_2O (1:1, 2 mL) under Ar. The resulting reaction mixture was stirred at 100 ℃ for 4 h. After reaction completed, the volatiles were removed under reduced pressure. The residue was purified by flash column chromatography on silica gel (height 20 cm, width 3.5 cm, eluent: petroleum ether) to afford the corresponding product **6a**.

1,1'-dibenzyl-4,4'-bis(3-(naphthalen-2-yl)phenyl)-1H,1'H-3,3'-bipyrrole 6a. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (79%, 113.2 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.37. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 – 7.70 (m, 6H), 7.70 – 7.62 (m, 4H), 7.55 – 7.49 (m, 2H), 7.44 – 7.36 (m, 4H), 7.35 – 7.31 (m, 2H), 7.27 – 7.23 (m, 2H), 7.22 – 7.09 (m, 12H), 6.95 (d, J = 2.4 Hz, 2H), 6.69 (d, J = 2.4 Hz, 2H), 5.05 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.29, 139.03, 137.96, 136.83, 133.72, 132.51, 128.76, 128.36, 128.23, 128.14, 127.67, 127.60, 127.08, 126.42, 126.38, 126.06, 125.73, 125.66, 125.57, 124.90, 124.01, 121.93, 119.50, 116.91, 53.55. **HRMS** (ESI) calcd for $C_{54}H_{41}N_2^+$ *m/z* 717.3270 $[M+H]^+$, Found 717.3273.

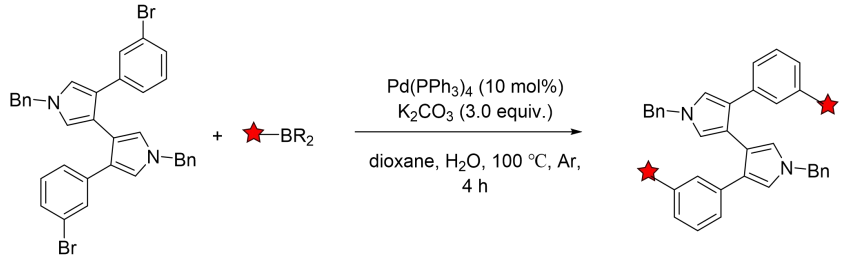


4k **6b**

An oven-dried 25 mL Schlenk tube equipped with a stirring bar was charged with **4k** (124.0 mg, 0.20 mmol, 1.0 equiv.), 9-anthraceneboronic acid (75.7 mg, 0.44 mmol, 2.2 equiv.), $Pd(PPh_3)_4$ (23.1 mg, 0.02 mmol, 10 mol%) and K_2CO_3 (82.9 mg, 0.6 mmol, 3.0 equiv.) in dioxane/H_2O (1:1, 2 mL) under Ar. The resulting reaction mixture was stirred at 100 ℃ for 4 h. After reaction completed, the volatiles were removed under reduced pressure. The residue was purified by flash column chromatography on silica gel (height 20 cm, width 3.5 cm, eluent: petroleum ether) to afford product **6b**.

4,4'-bis(3-(anthracen-9-yl)phenyl)-1,1'-dibenzyl-1H,1'H-3,3'-bipyrrole 6b. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (73%, 119.1 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.33. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.41 (s, 2H), 7.95 (d, J = 8.8 Hz, 4H), 7.62 (d, J = 8.8 Hz, 4H), 7.44 – 7.39 (m, 4H), 7.35 – 7.30 (m, 6H), 7.15 – 7.05 (m, 12H), 6.95 – 6.90 (m, 4H), 6.77 (d, J = 2.4 Hz, 2H), 6.58 (d, J = 2.4 Hz, 2H), 4.85 (s, 4H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.19, 137.86, 137.82, 136.48, 131.43, 130.28, 129.73, 128.66, 128.16, 127.93, 127.86, 127.60, 127.48, 127.06, 126.39, 126.20, 125.11, 125.08, 124.25, 121.69, 119.95, 116.85, 53.42. **HRMS** (ESI) calcd for $C_{62}H_{45}N_2^+$ *m/z* 817.3583 $[M+H]^+$, Found 817.3574.

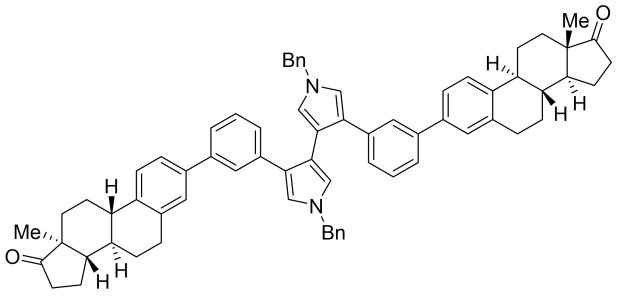
11.General procedure for the synthesis of derivatives 7, 8, 9.



4k

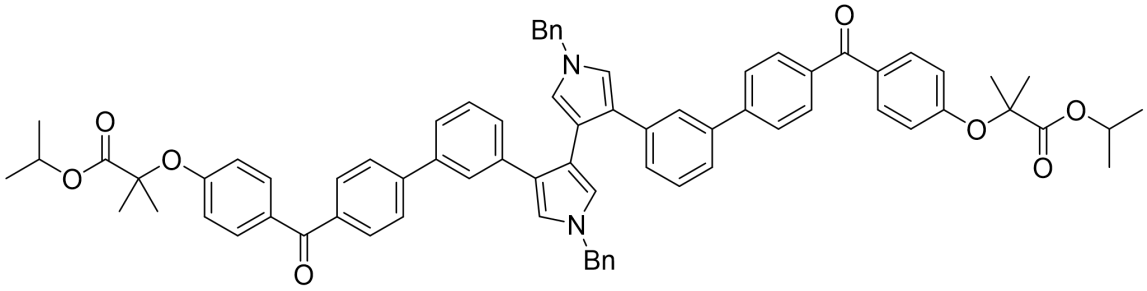
An oven-dried 25 mL Schlenk tube equipped with a stirring bar was charged with **4k** (124.0 mg, 0.2 mmol, 1.0 equiv.), borate ester[3,4] (0.44 mmol, 2.2 equiv.), $Pd(PPh_3)_4$ (23.1 mg, 0.02 mmol, 10 mol%) and K_2CO_3 (82.9 mg, 0.6 mmol, 3.0 equiv.)in dioxane/H_2O (1:1, 2 mL) under Ar. The resulting reaction mixture was stirred at 100 ℃ for 4 h. After reaction completed, the volatiles were removed under reduced pressure. The residue was purified by flash column chromatography on silica gel

(height 20 cm, width 3.5 cm, eluent: petroleum ether) to afford the corresponding product **7**, **8**, **9**.



7

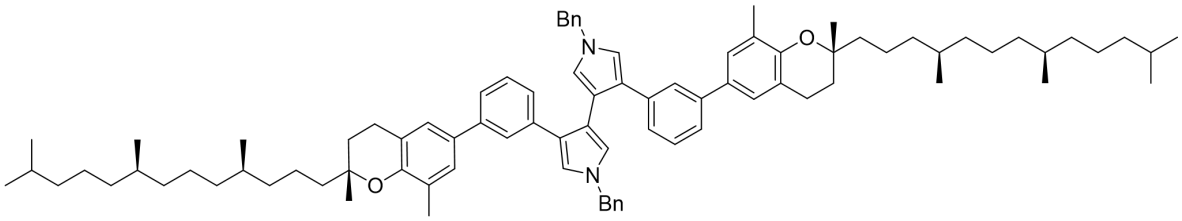
(8R,8'R,9S,9'S,13S,13'S,14S,14'S)-3,3'-((1,1'-dibenzyl-1H,1'H-[3,3'-bipyrrole]-4,4'-diyl)bis(3,1-phenylene))bis(13-methyl-6,7,8,9,11,12,13,14,15,16-decahydro-17H-cyclopenta[a]phenanthren-17-one) 7. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 20:1) as a yellow oil (74%, 143.3 mg). R_f (petroleum ether/ethyl acetate = 20:1): 0.33. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.54 (s, 2H), 7.28 – 7.22 (m, 14H), 7.18 – 7.11 (m, 8H), 6.93 (d, J = 2.4 Hz, 2H), 6.66 (d, J = 2.4 Hz, 2H), 5.08 (s, 4H), 2.93 – 2.85 (m, 4H), 2.57 – 2.49 (m, 2H), 2.38 – 2.30 (m, 2H), 2.20 – 1.98 (m, 10H), 1.66 – 1.49 (m, 12H), 0.93 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 220.93, 140.17, 139.23, 138.34, 137.99, 136.56, 136.48, 128.72, 128.16, 127.69, 127.57, 127.00, 125.99, 125.86, 125.59, 124.83, 124.46, 123.58, 121.92, 119.36, 116.75, 53.48, 50.54, 48.03, 44.41, 38.25, 35.89, 31.66, 29.52, 26.63, 25.82, 21.64, 13.89. **HRMS** (ESI) calcd for $C_{70}H_{68}N_2O_2Na^+$ *m/z* 991.5178 $[M+Na]^+$, Found 991.5179.



8

Diisopropyl2,2'-(((3',3'''-(1,1'-dibenzyl-1H,1'H-[3,3'-bipyrrole]-4,4'-diyl)bis([1'',1'''-biphenyl]-4-carbonyl))bis(4,1-phenylene))bis(oxy))bis(2-methylpropanoate) 8. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 20 to 5:1) as a yellow oil (79%, 175.8 mg). R_f (petroleum ether/ethyl acetate = 5:1): 0.18. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 – 7.68 (m, 9H), 7.53 (t, J = 1.6 Hz, 2H), 7.45 (d, J = 8.0 Hz, 4H), 7.26

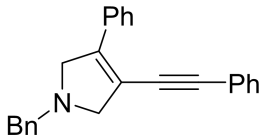
– 7.13 (m, 15H), 6.95 – 6.86 (m, 6H), 6.70 (d, *J* = 2.4 Hz, 2H), 5.13 – 5.02 (m, 6H), 1.67 (s, 12H), 1.20 (d, *J* = 6.4 Hz, 12H). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 195.12, 173.07, 159.35, 145.16, 138.99, 137.70, 136.73, 136.19, 131.87, 130.74, 130.19, 128.65, 128.29, 127.63, 126.94, 126.78, 126.53, 126.00, 124.37, 123.59, 121.68, 119.32, 117.10, 116.63, 79.25, 69.21, 53.40, 25.28, 21.42. **HRMS** (ESI) calcd for $C_{74}H_{69}N_2O_8^+$ *m/z* 1113.5054 $[M+H]^+$, Found 1113.5047.



9

1,1'-dibenzyl-4,4'-bis(3-((R)-2,8-dimethyl-2-((4R,8R)-4,8,12-trimethyltridecyl)chroman-6-yl)phenyl)-1H,1'H-3,3'-bipyrrole 9. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:1 to 50:1) as a yellow oil (83%, 204.5 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.43. **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.61 (s, 1H), 7.36 – 7.25 (m, 10H), 7.23 – 7.13 (m, 9H), 7.11 – 7.04 (m, 2H), 7.00 (d, *J* = 2.4 Hz, 2H), 6.69 (d, *J* = 2.4 Hz, 2H), 5.10 (s, 4H), 2.89 – 2.69 (m, 4H), 2.33 – 2.20 (m, 6H), 1.94 – 1.78 (m, 4H), 1.71 – 1.17 (m, 48H), 0.99 – 0.90 (m, 25H). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 151.47, 140.56, 137.99, 136.38, 132.29, 128.61, 127.97, 127.48, 127.10, 126.91, 126.11, 125.58, 125.46, 125.28, 124.97, 123.27, 121.85, 120.27, 119.22, 116.76, 76.04, 53.38, 40.32, 39.35, 37.47, 37.44, 37.27, 32.78, 32.70, 31.25, 27.96, 24.79, 24.44, 24.27, 22.72, 22.63, 22.41, 21.01, 19.76, 19.65, 16.20. **HRMS** (ESI) calcd for $C_{88}H_{116}N_2O_2Na^+$ *m/z* 1255.8935 $[M+Na]^+$, Found 1255.8921.

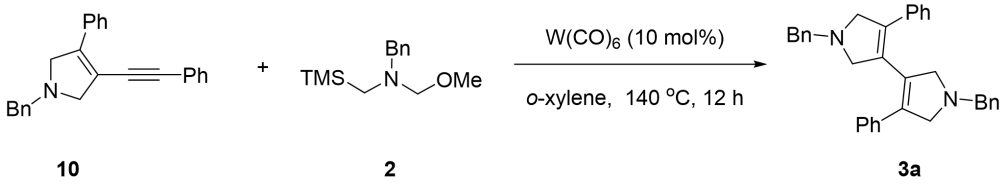
12.General procedures for control experiments.



10

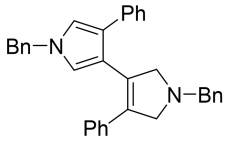
benzyl-3-phenyl-4-(phenylethynyl)-2,5-dihydro-1H-pyrrole 10. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 100:0 to 50:1) as a white solid (53%, 35.5 mg). R_f (petroleum ether/ethyl acetate = 50:1): 0.5. **^{1}H NMR** (400 MHz, $CDCl_3$) *δ* 7.81 (d, *J* = 7.6 Hz, 2H), 7.48 – 7.42 (m, 2H), 7.42 – 7.37 (m, 2H), 7.37 – 7.25 (m, 9H), 3.98 (t, *J* = 4.0 Hz, 2H), 3.85 (s, 2H), 3.81 (t, *J* = 4.0 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) *δ* 142.32, 139.11, 134.16, 131.57, 128.78, 128.54, 128.48, 128.35,

128.28, 127.26, 126.78, 123.38, 115.65, 96.53, 85.51, 64.41, 61.84, 60.35, one carbon was overlapped. **HRMS** (ESI) calcd for $C_{25}H_{22}N^+$ *m/z* 336.1752 $[M+H]^+$, Found 336.1754.



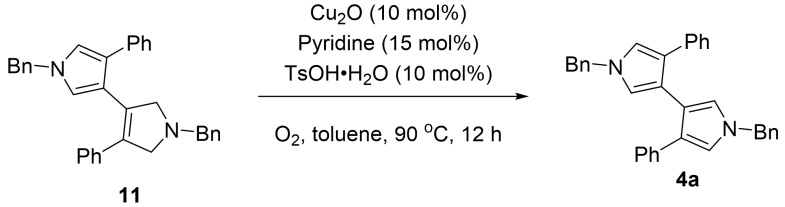
10 **2** **3a**

An oven-dried 25 mL schlenk tube equipped with a stirring bar was transferred into glovebox (through standard glovebox operation), where $W(CO)_6$ (0.02 mmol, 7.0 mg, 0.1 equiv.) was added. The tube was then removed from glovebox and placed under Ar. Then derivatives **10** (0.2 mmol, 1.0 equiv.), *N*-(methoxymethyl)-*N*-(trimethylsilylmethyl)-benzylamine **2** (1.2 mmol, 6.0 equiv.), and *o*-xylene (2 mL) were added subsequently to the test tube under Ar. The resulting reaction mixture was stirred at 140 ℃ for 12 h. After reaction completed, the mixture was cooled down to room temperature, and the volatiles were removed under reduced pressure. The residue was then purified by flash column chromatography on silica gel to give the desired product **3a**.



11

1,1'-dibenzyl-4,4'-diphenyl-2,5-dihydro-1H,1'H-3,3'-bipyrrole 11. The product was purified by flash column chromatography on silica gel (height 18 cm, width 1.5 cm, eluent: petroleum ether/ethyl acetate, gradient: 50:1 to 10:1) as a yellow oil (43%, 40.1 mg). R_f (petroleum ether/ethyl acetate = 10:1): 0.2. **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.40 (d, J = 7.2 Hz, 2H), 7.35 – 7.21 (m, 12H), 7.14 – 7.05 (m, 6H), 6.80 (d, J = 2.4 Hz, 1H), 6.49 (d, J = 2.4 Hz, 1H), 4.96 (s, 2H), 3.95 (t, J = 3.6 Hz, 2H), 3.82 (s, 2H), 3.67 (t, J = 4.0 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 139.37, 137.80, 135.99, 135.81, 133.35, 130.65, 128.76, 128.75, 128.33, 127.82, 127.76, 127.29, 126.95, 126.91, 126.87, 126.54, 125.52, 124.30, 121.02, 119.83, 116.89, 65.58, 63.03, 60.51, 53.46, one carbon was overlapped. **HRMS** (ESI) calcd for $C_{34}H_{31}N_2^+$ *m/z* 467.2487 $[M+H]^+$, Found 467.2491.



11 **4a**

An oven-dried 25 mL reaction tube equipped with a stirring bar was charged with

derivatives **11** (0.20 mmol, 1.0 equiv.), Cu_2O (2.9 mg, 0.02 mmol, 0.1 equiv.), pyridine (2.4 mg, 0.03 mmol, 0.15 equiv.), TsOH•H_2O (3.4 mg, 0.02 mmol, 0.1 equiv.) and toluene (2 mL). The tube was evacuated and filled with 1 atm O_2, and stirred rigorously at 90 °C for 12 h. After reaction completed, the mixture was cooled down to room temperature, and the volatiles were removed under reduced pressure. The residue was purified by flash chromatography on silica gel to afford the corresponding products **4a**.

13.References.

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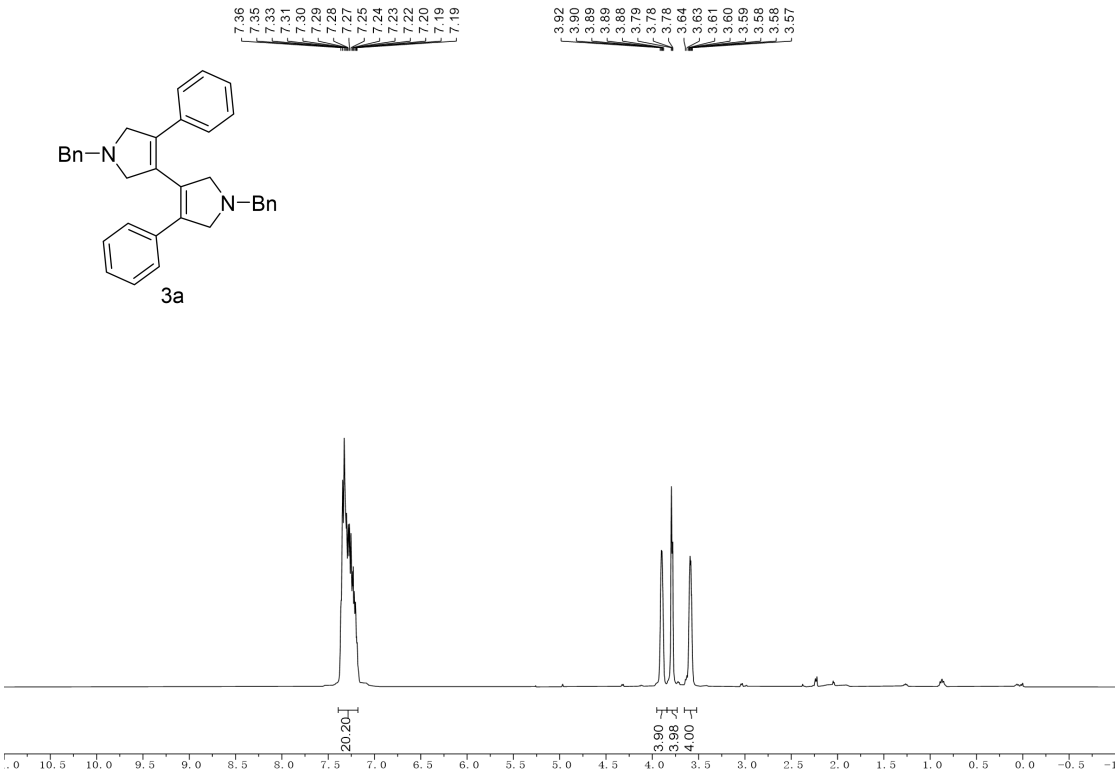
[3] S. S. Zhang, X.-Y. Liu, Z. B. Chang, X. X. Qiao, H.-Y. Xiong and G. W. Zhang, The [3+2] Annulation of CF3-Ketimines by Re Catalysis: Access to CF_3-Containing Amino Heterocycles and Polyamides, *iScience*., 2020, **23**, 101705.

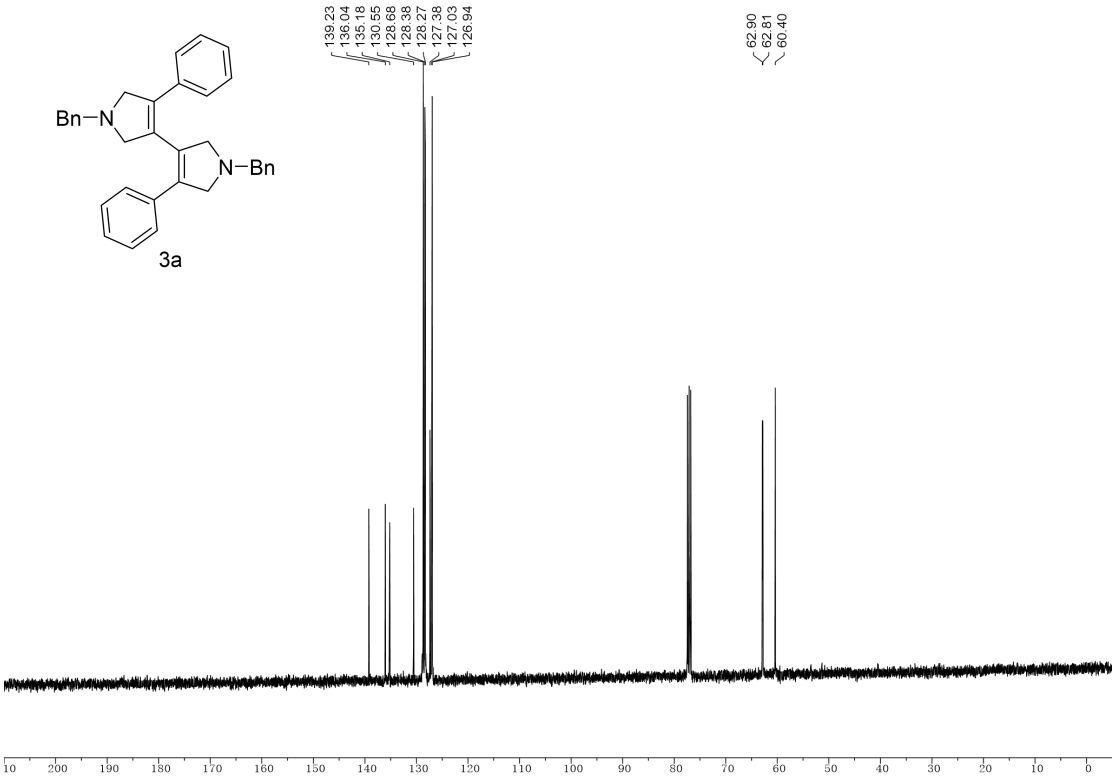
[4] Y. L. Zhou, L. P. Qiu, J. Li and W. L. Xie, A General Copper Catalytic System for Suzuki–Miyaura Cross-Coupling of Unactivated Secondary and Primary Alkyl Halides with Arylborons, *J. Am. Chem, Soc*., 2023, **145**, 28146-28155.

14.NMR spectra copies of the compounds 3.

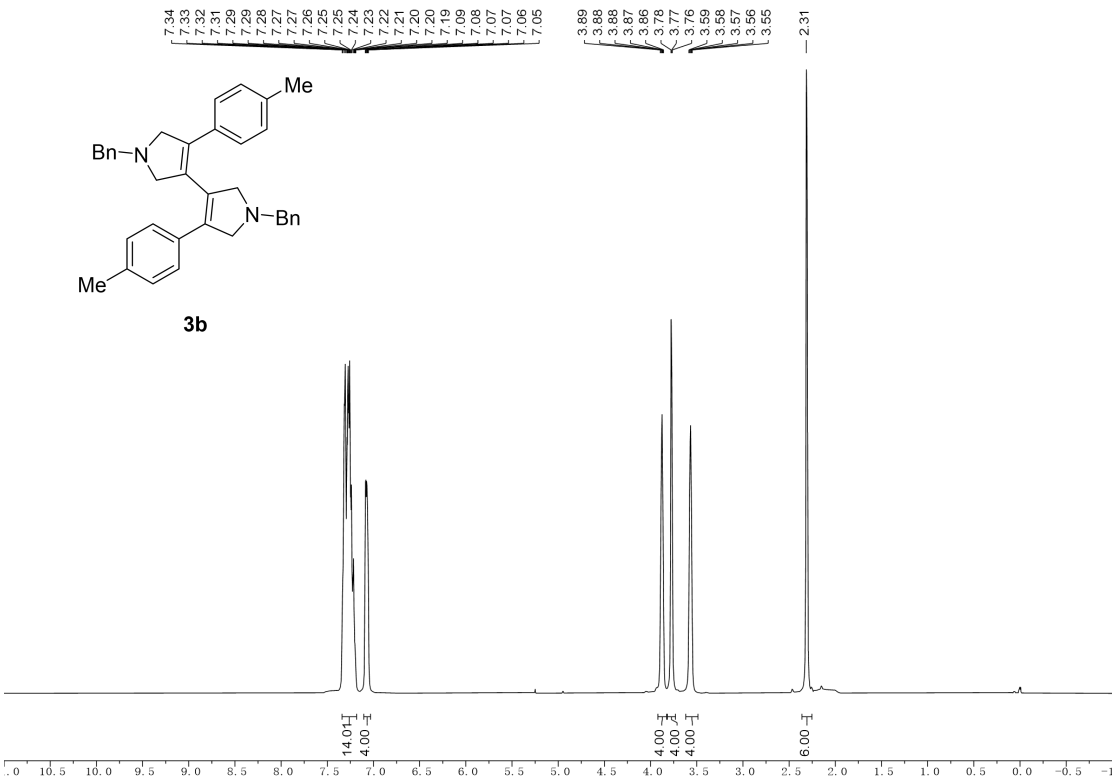
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3a



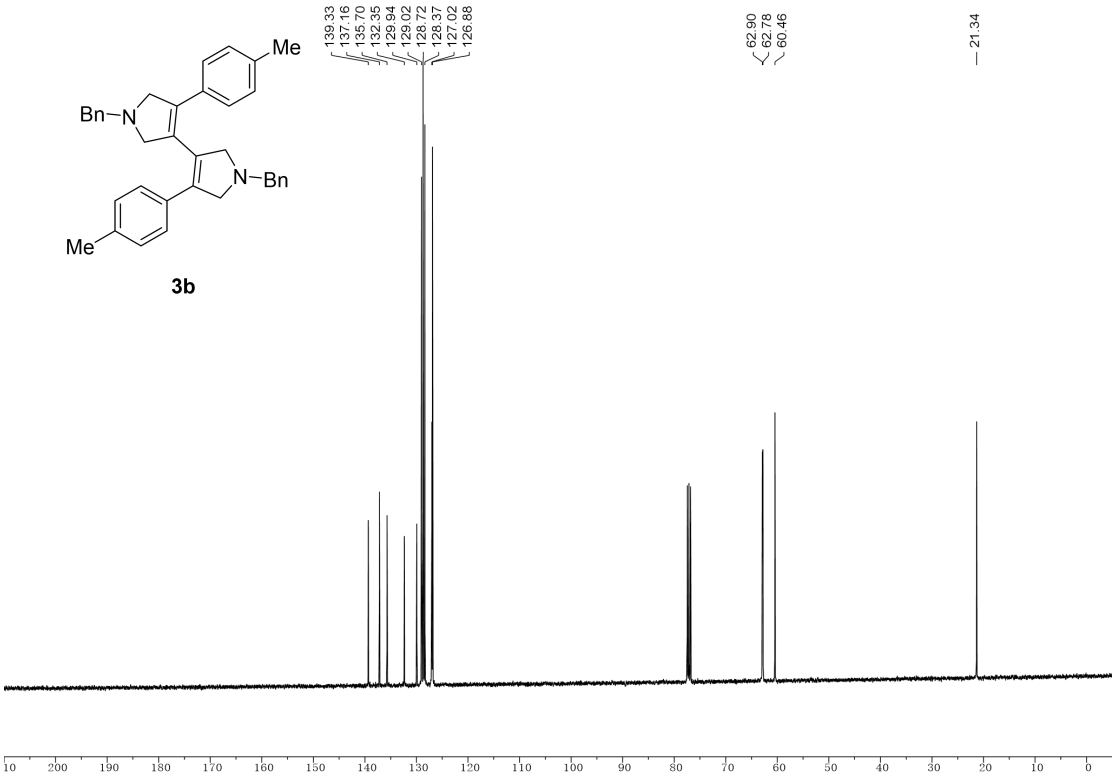
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3a



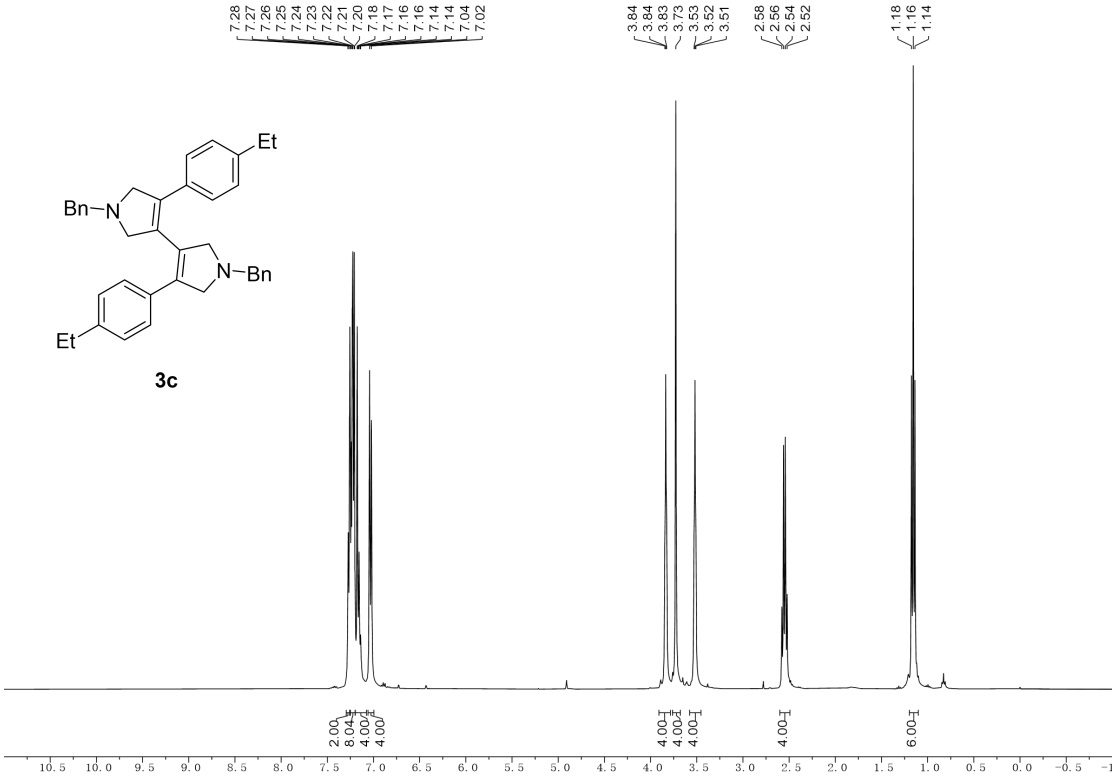
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3b:



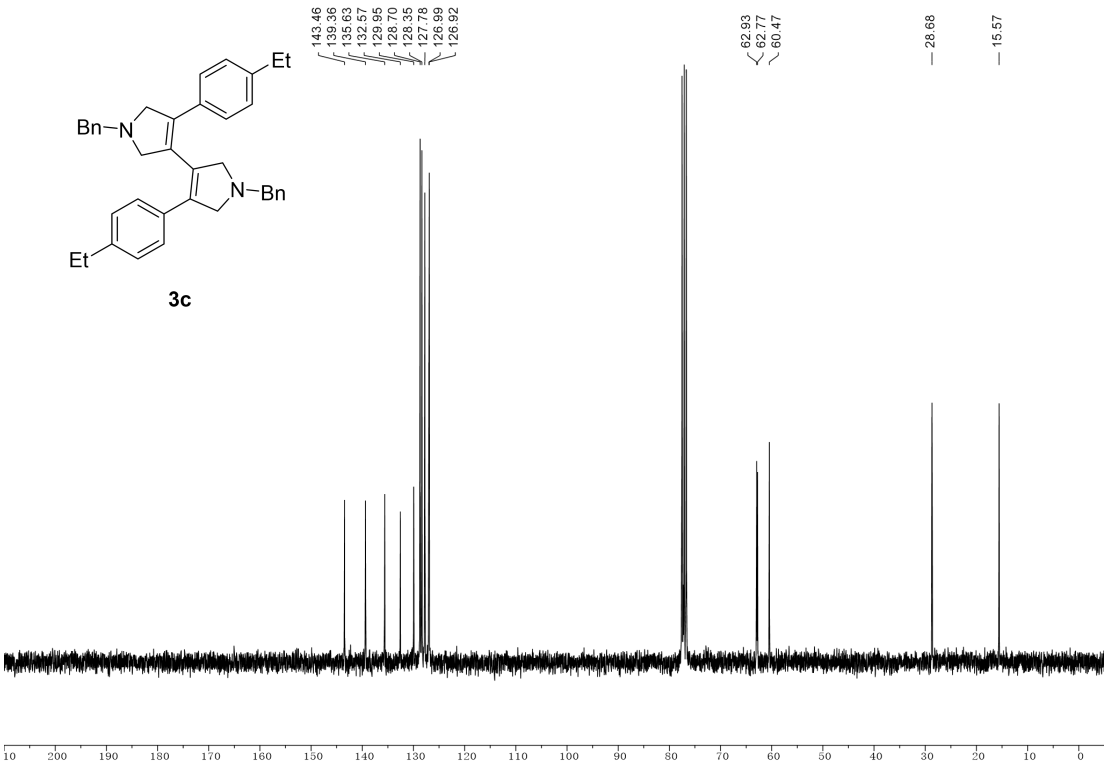
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3b:



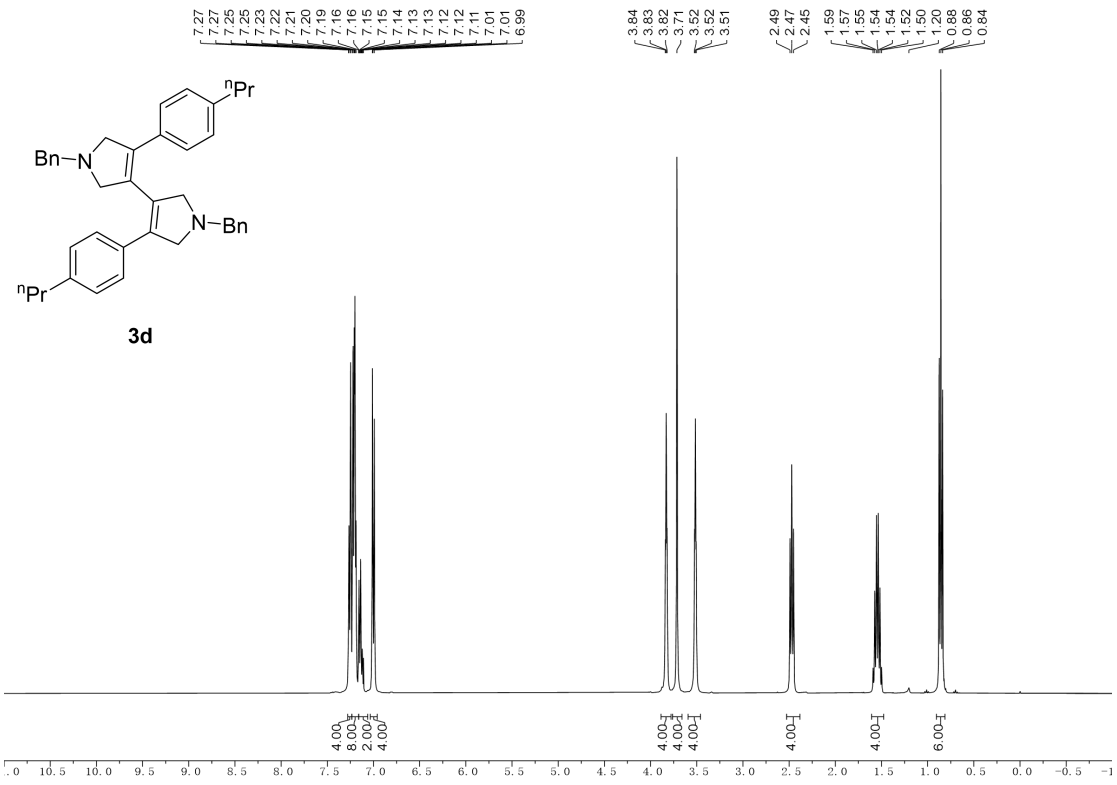
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3c:



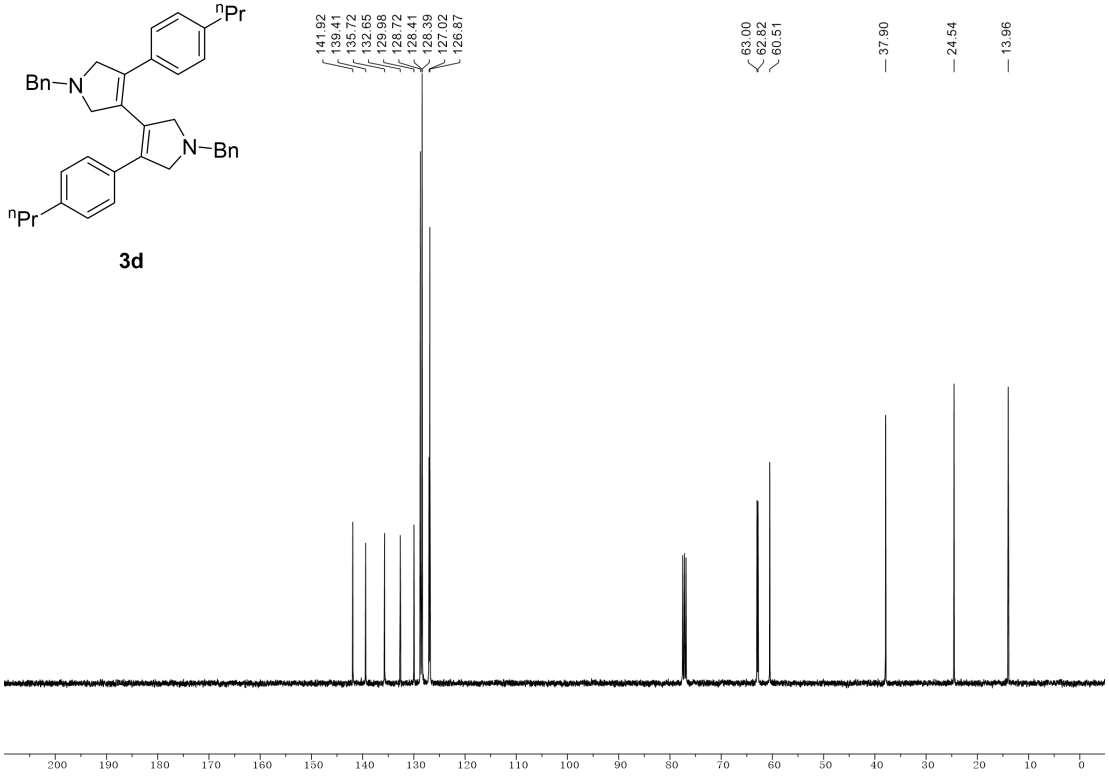
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3c:



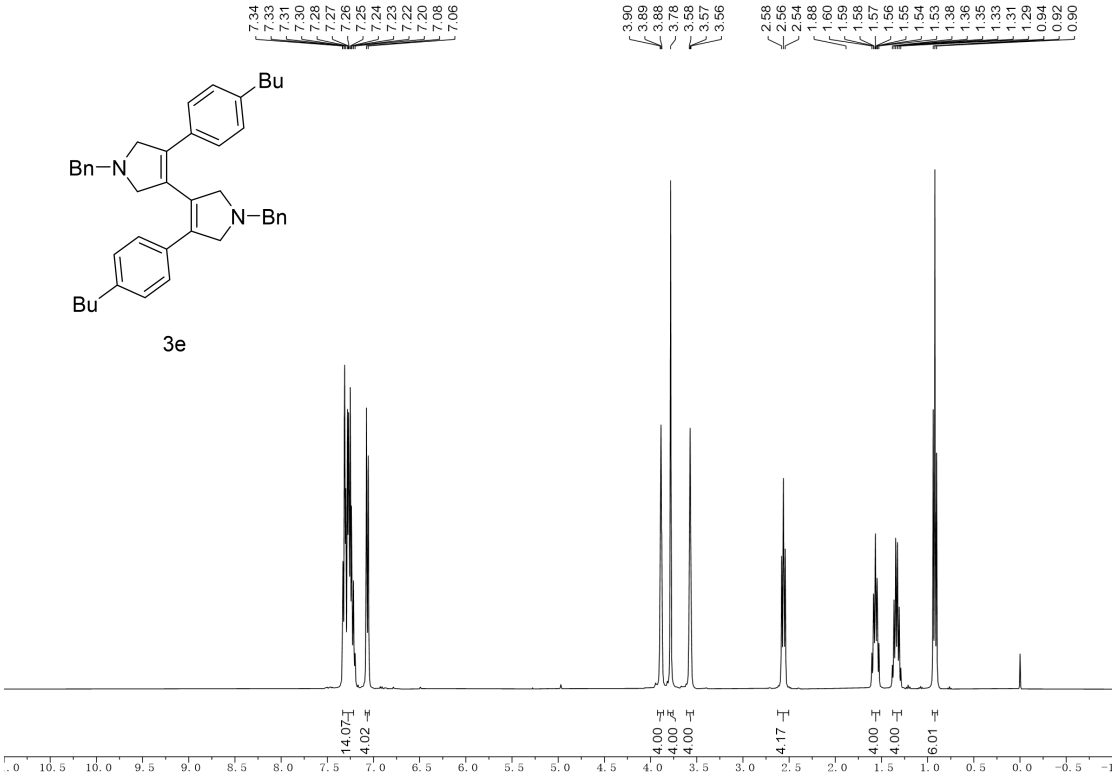
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3d:



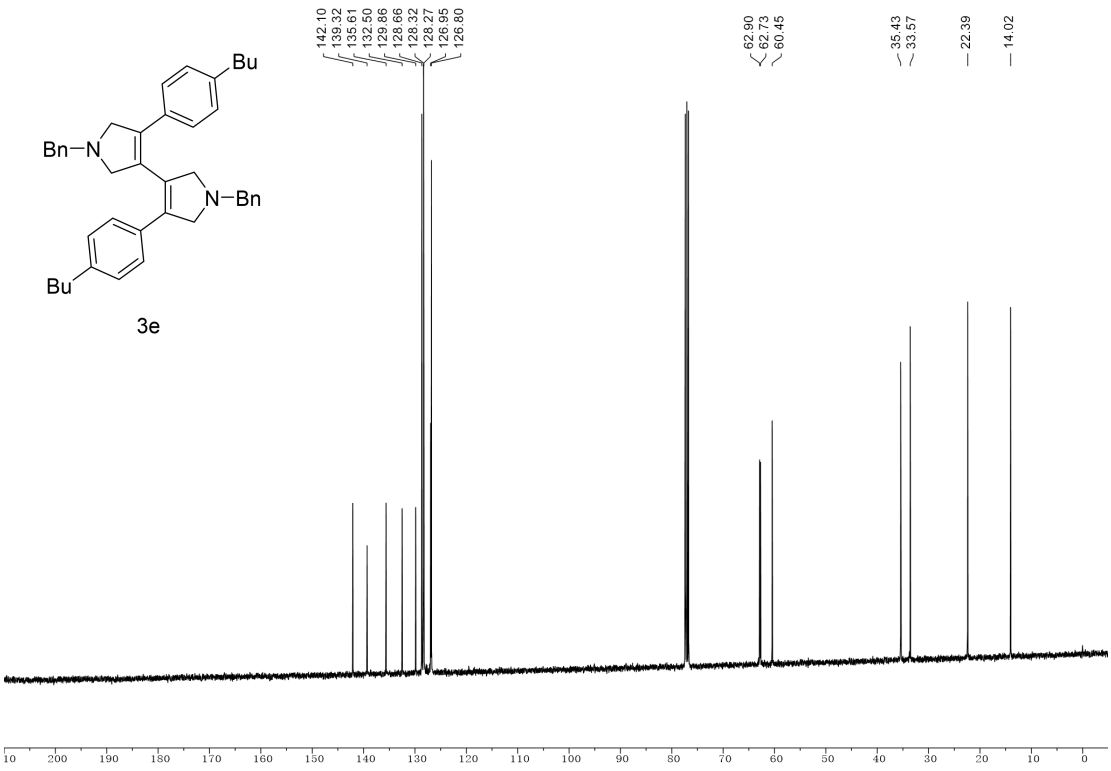
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3d:



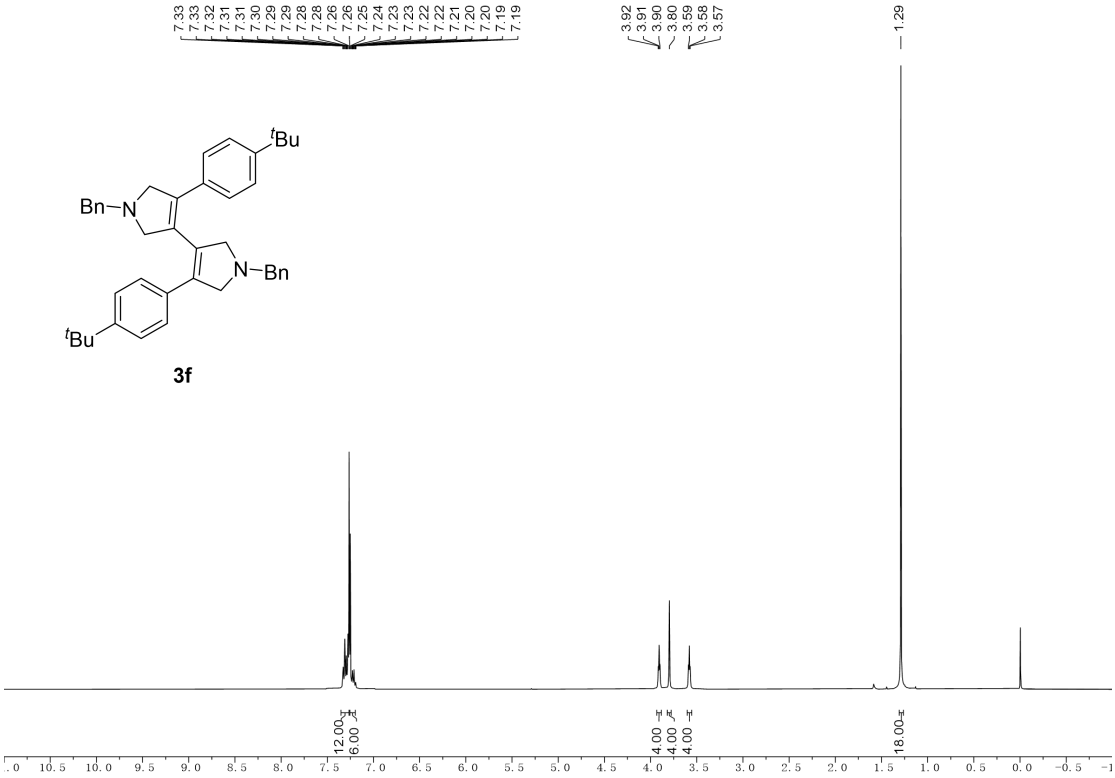
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3e:



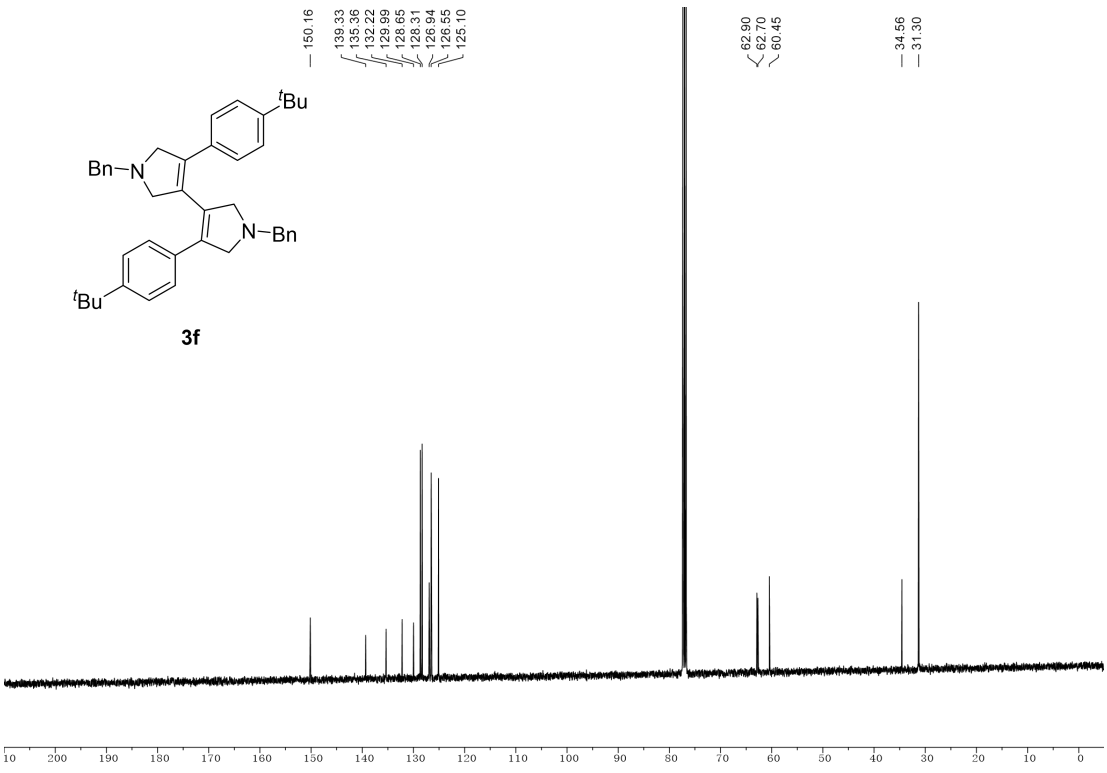
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3e:



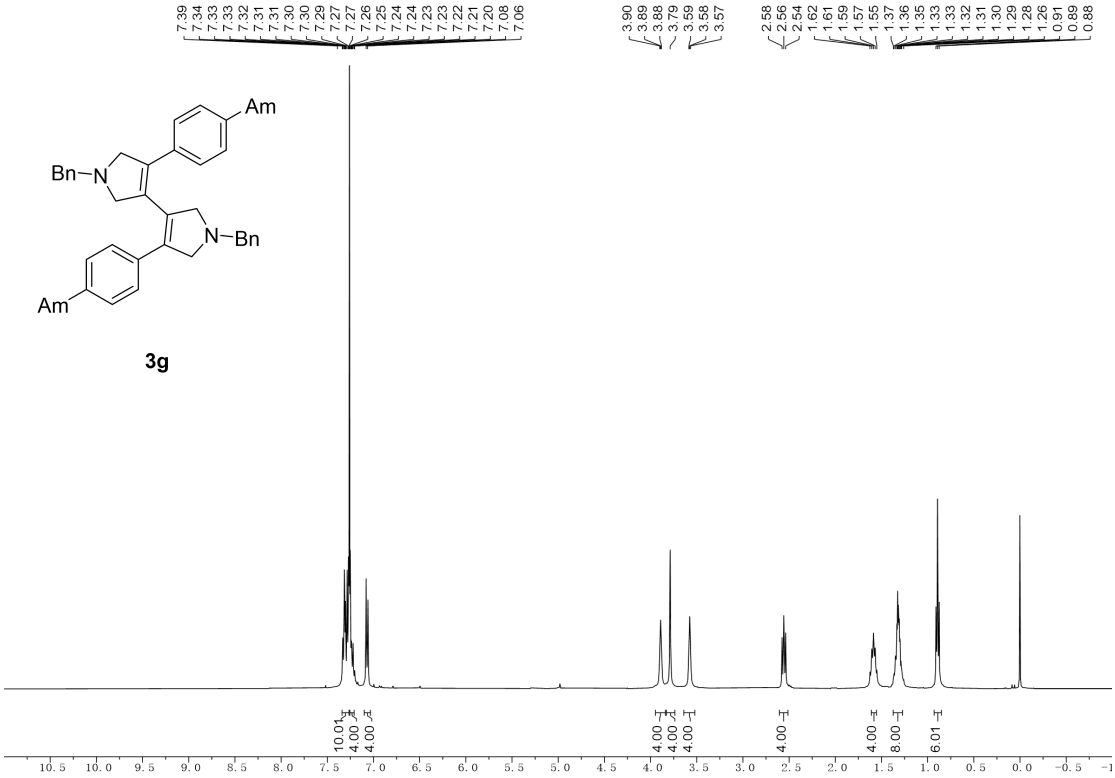
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3f:



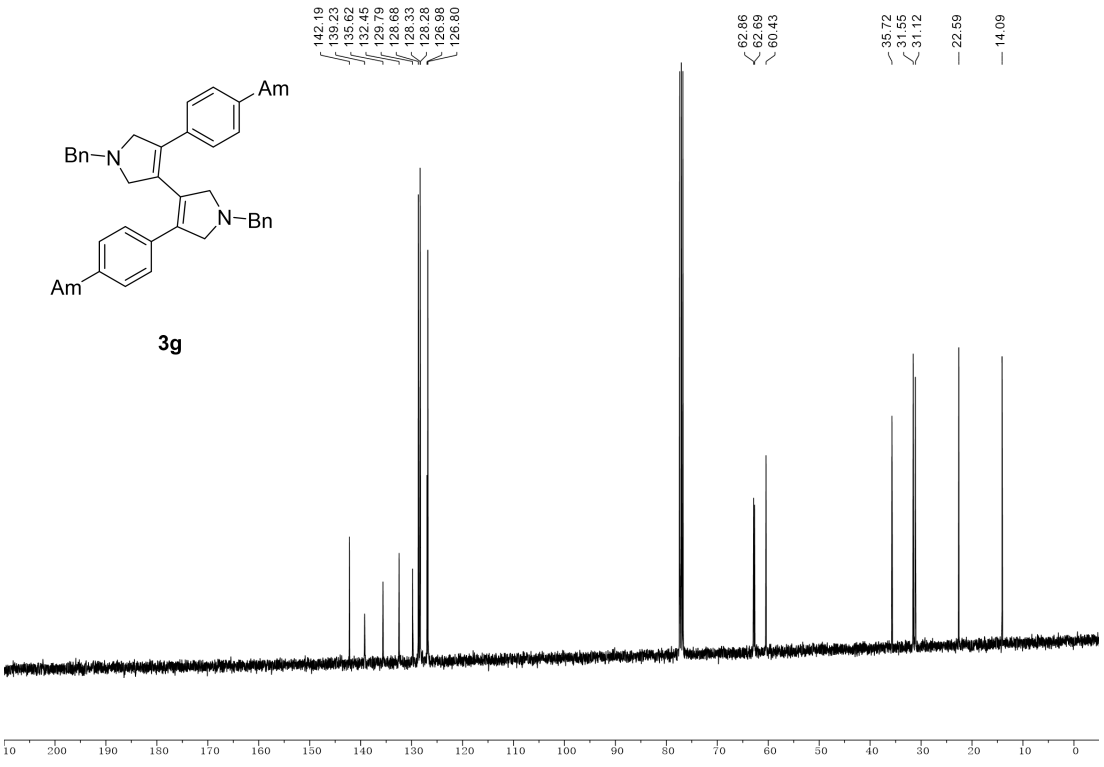
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3f:



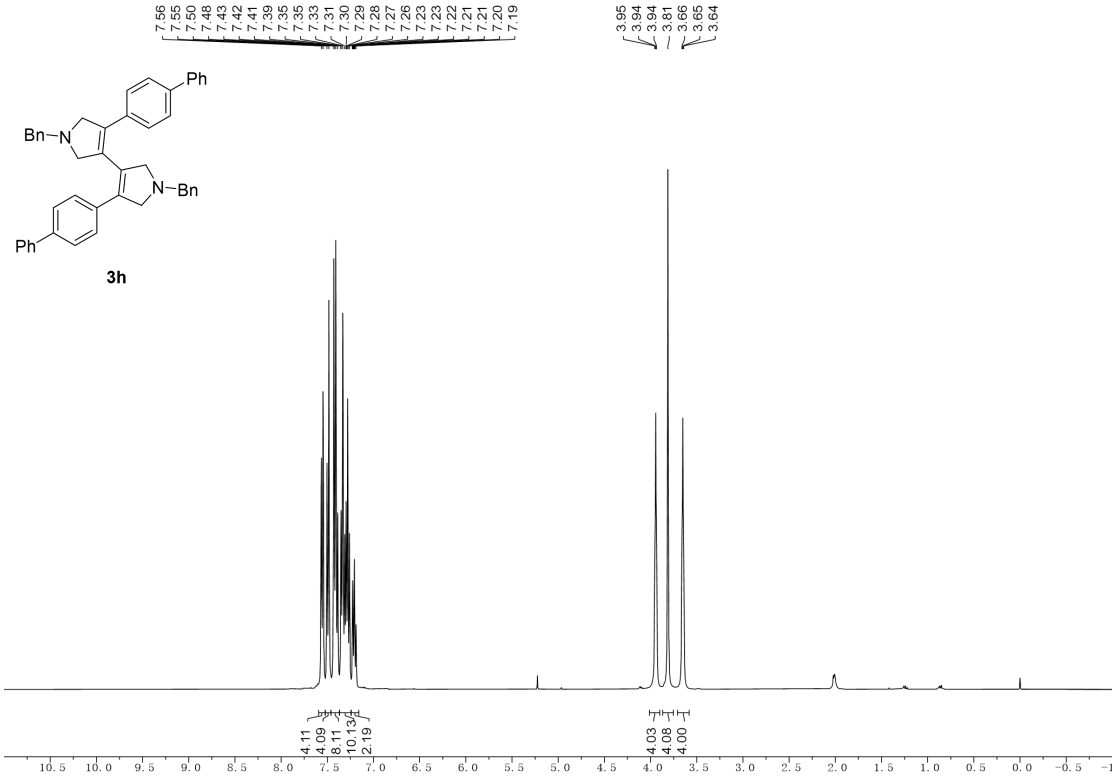
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3g:



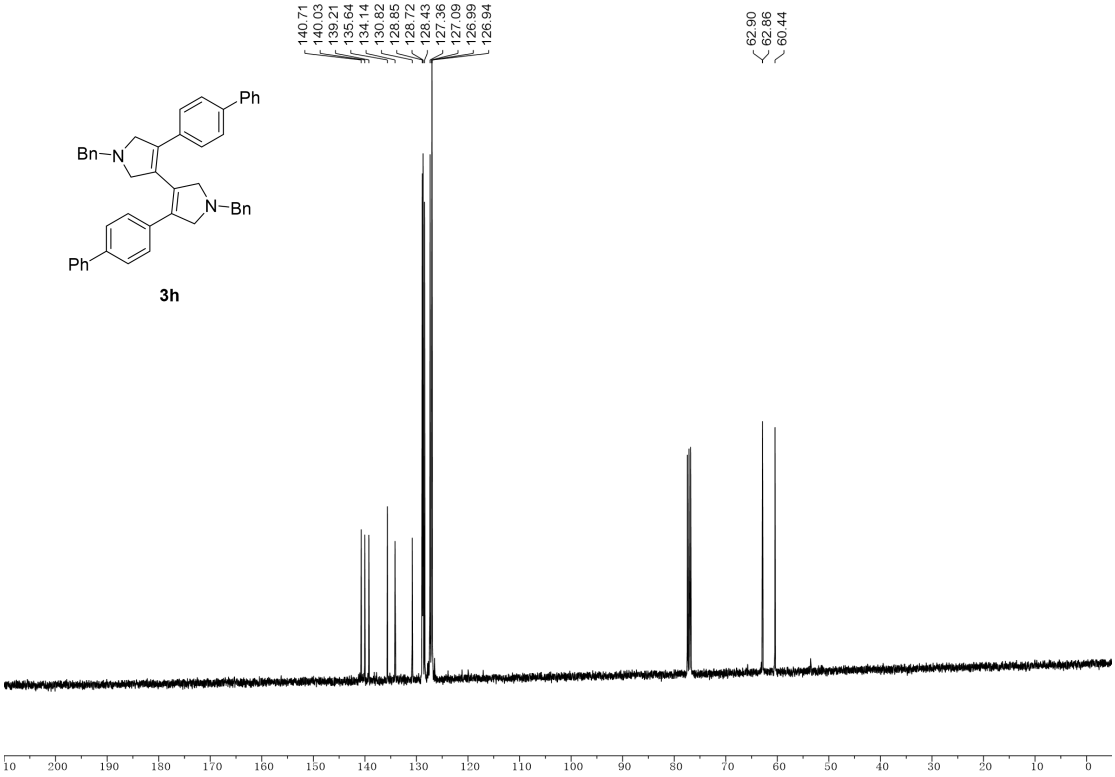
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3g:



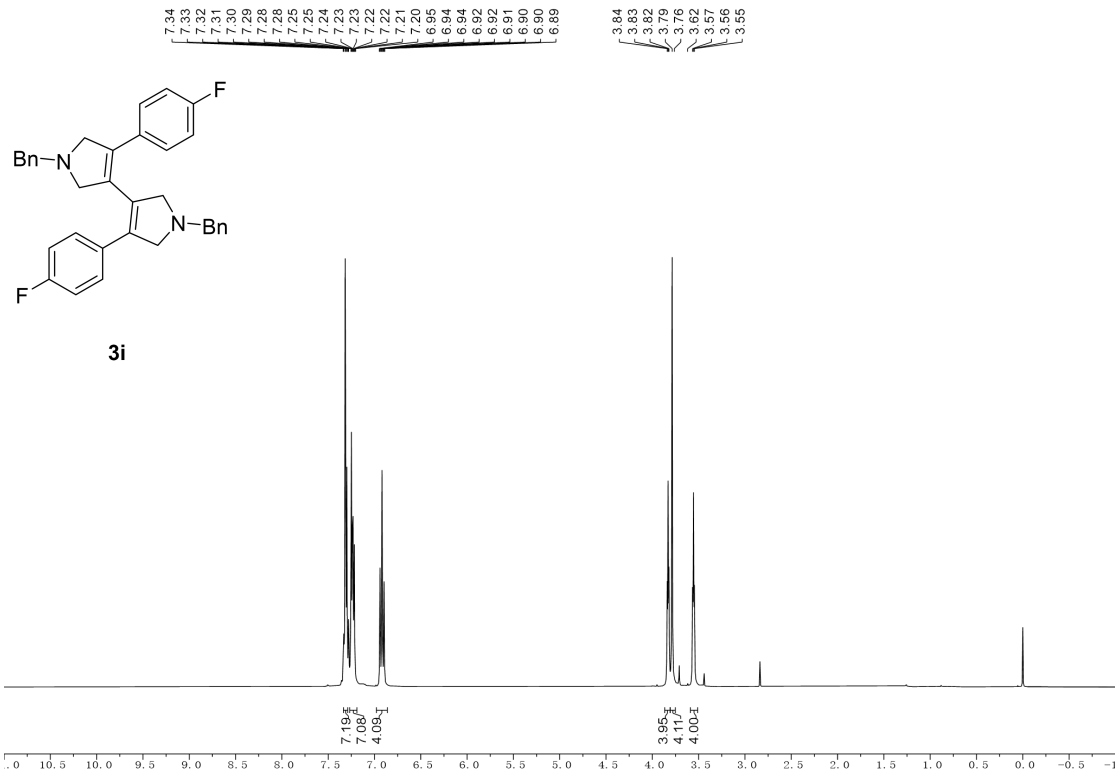
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3h:



^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 3h:

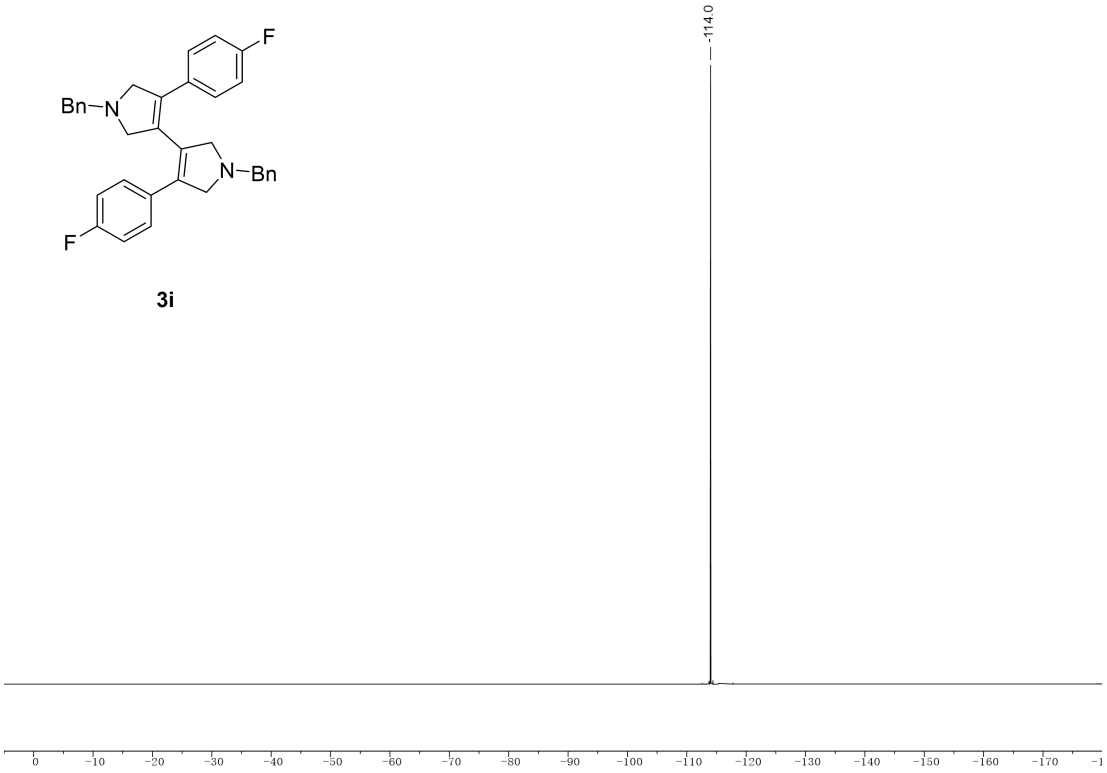


^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3i:



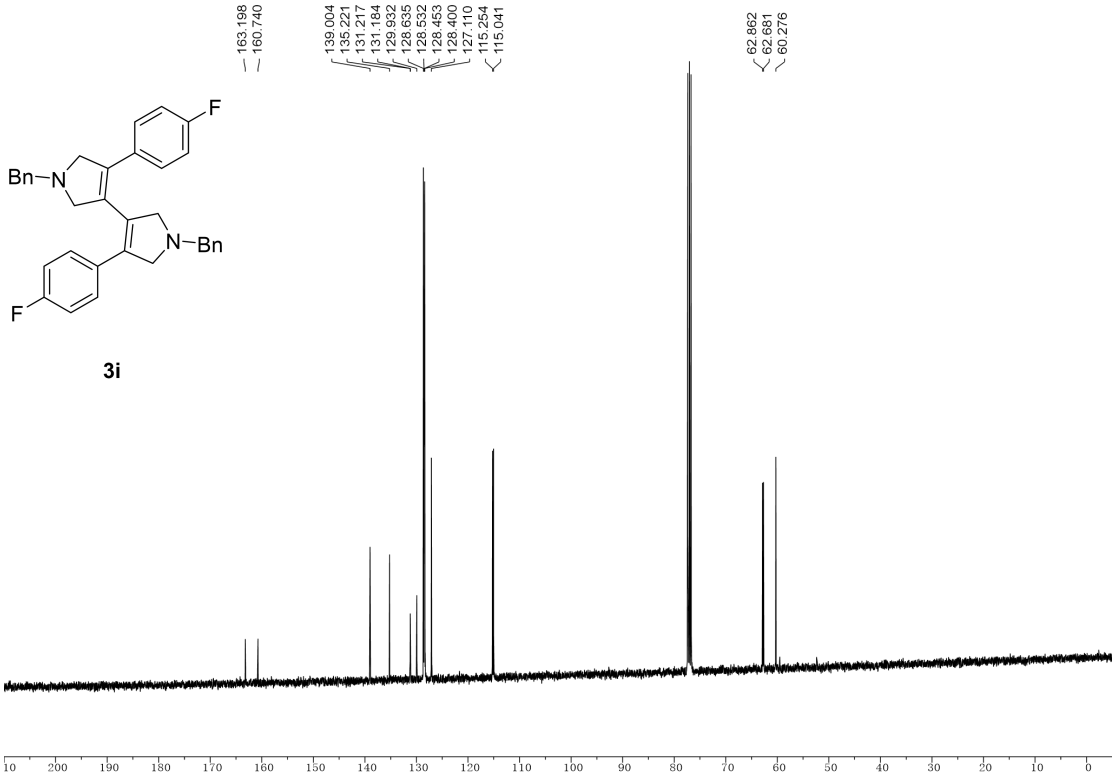
3i

^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) spectra of compound 3i:

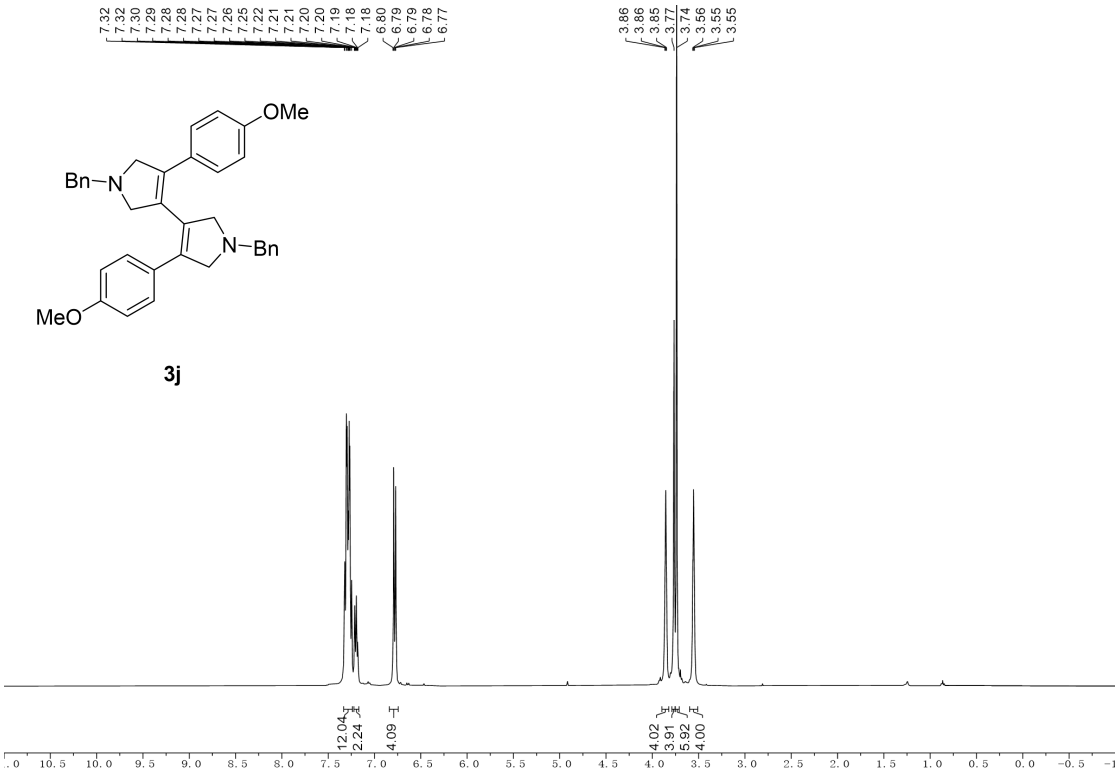


3i

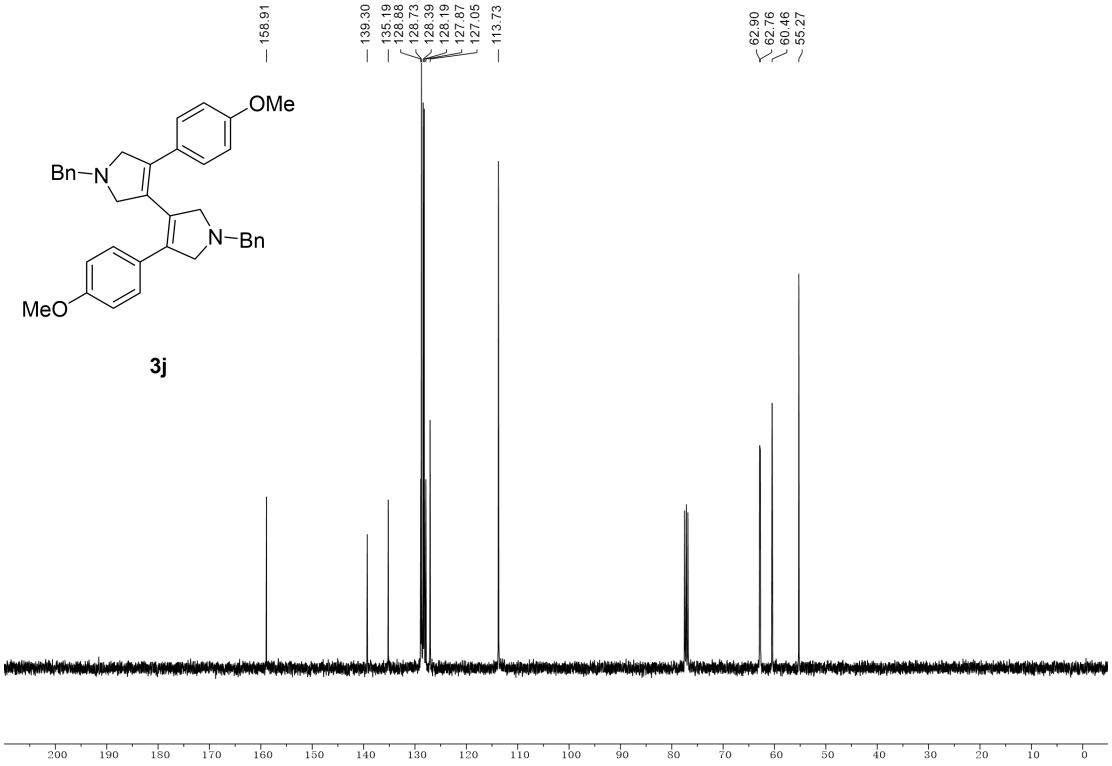
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 3i:



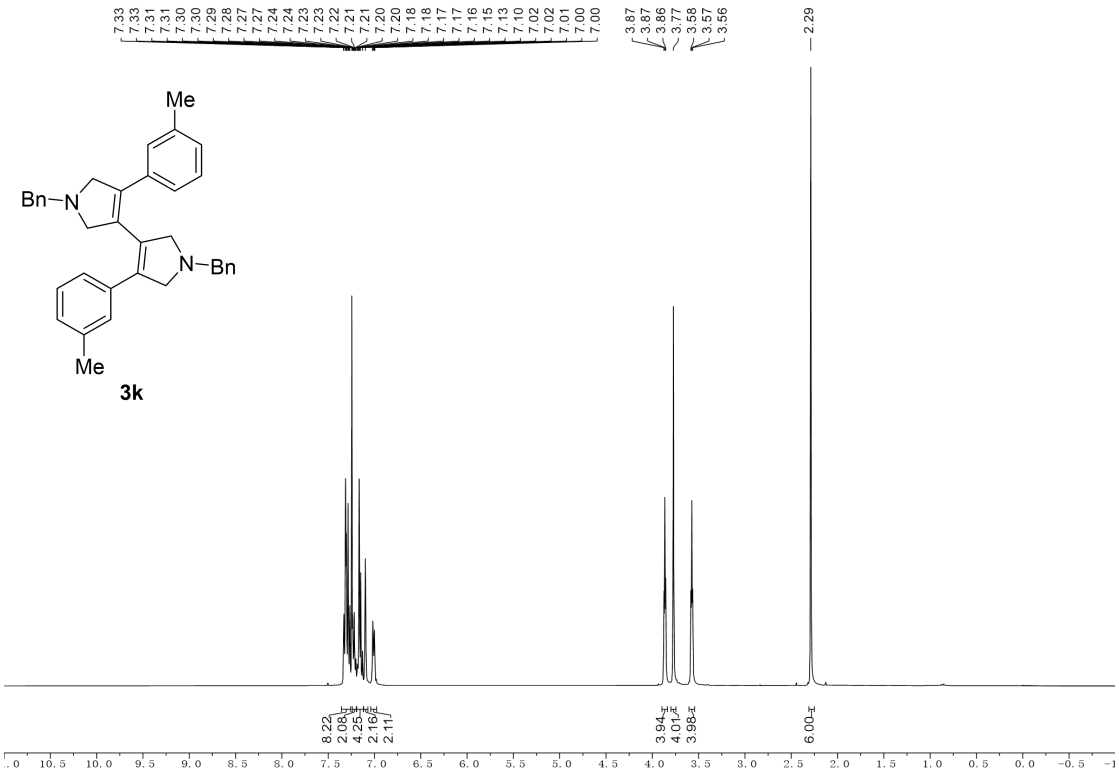
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3j:



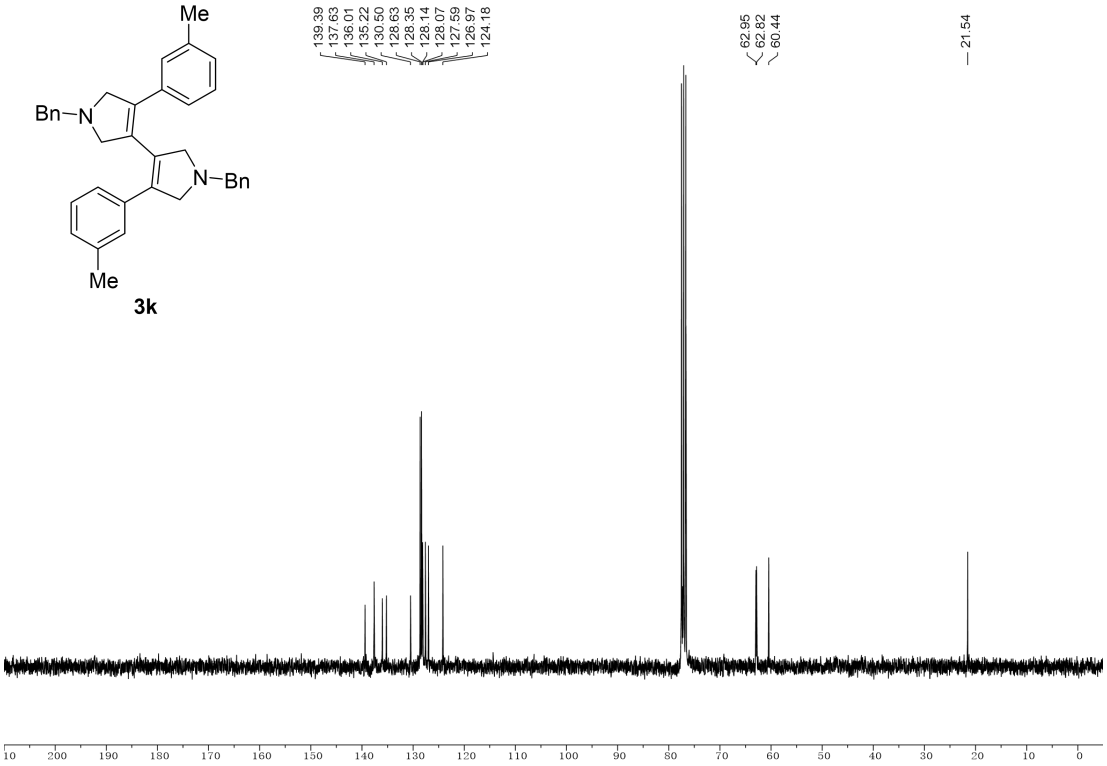
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound 3j:



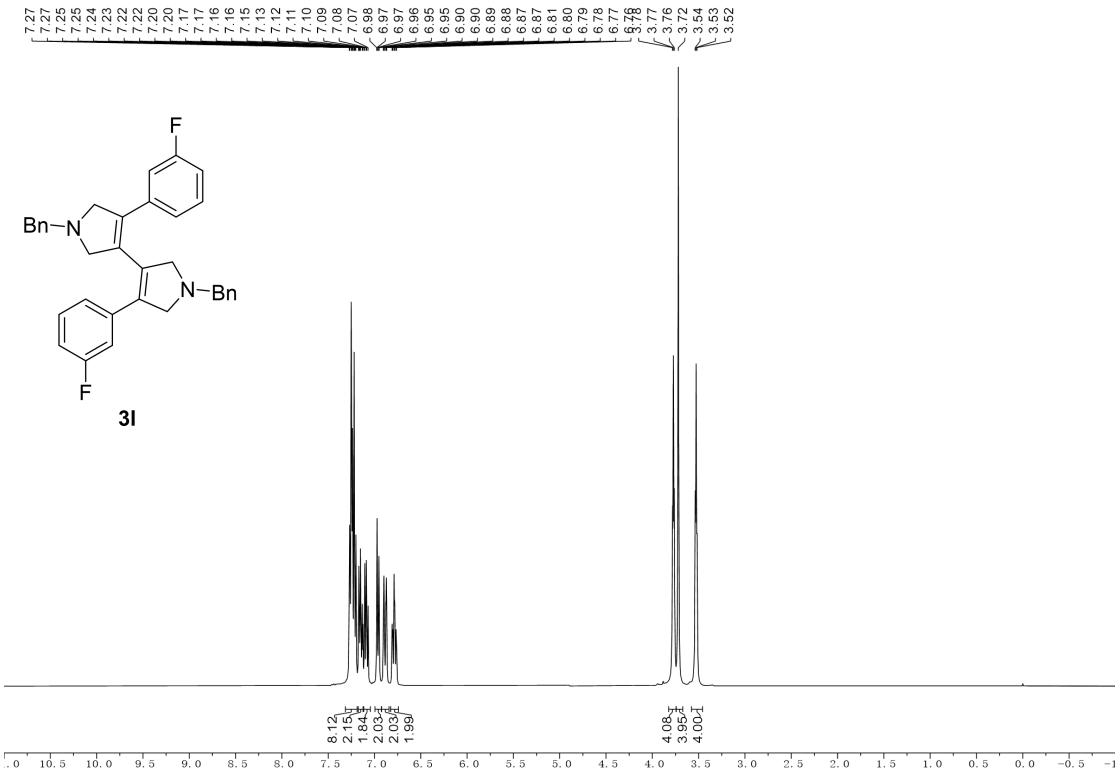
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3k:



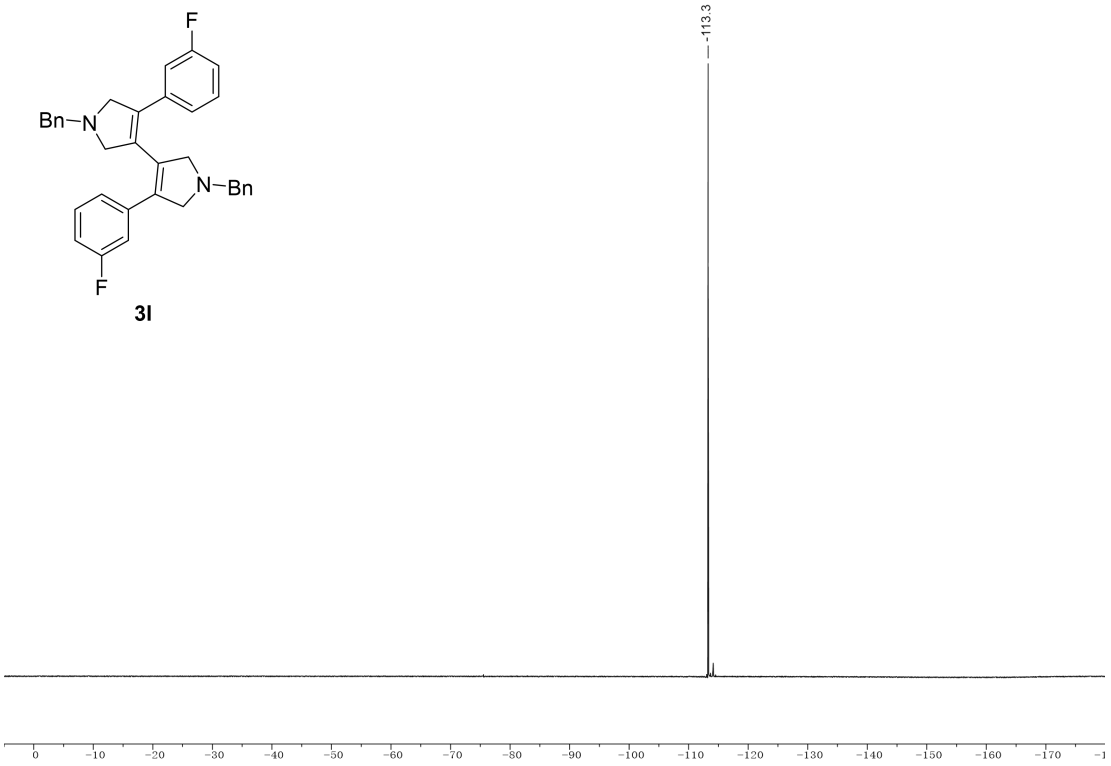
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound 3k:



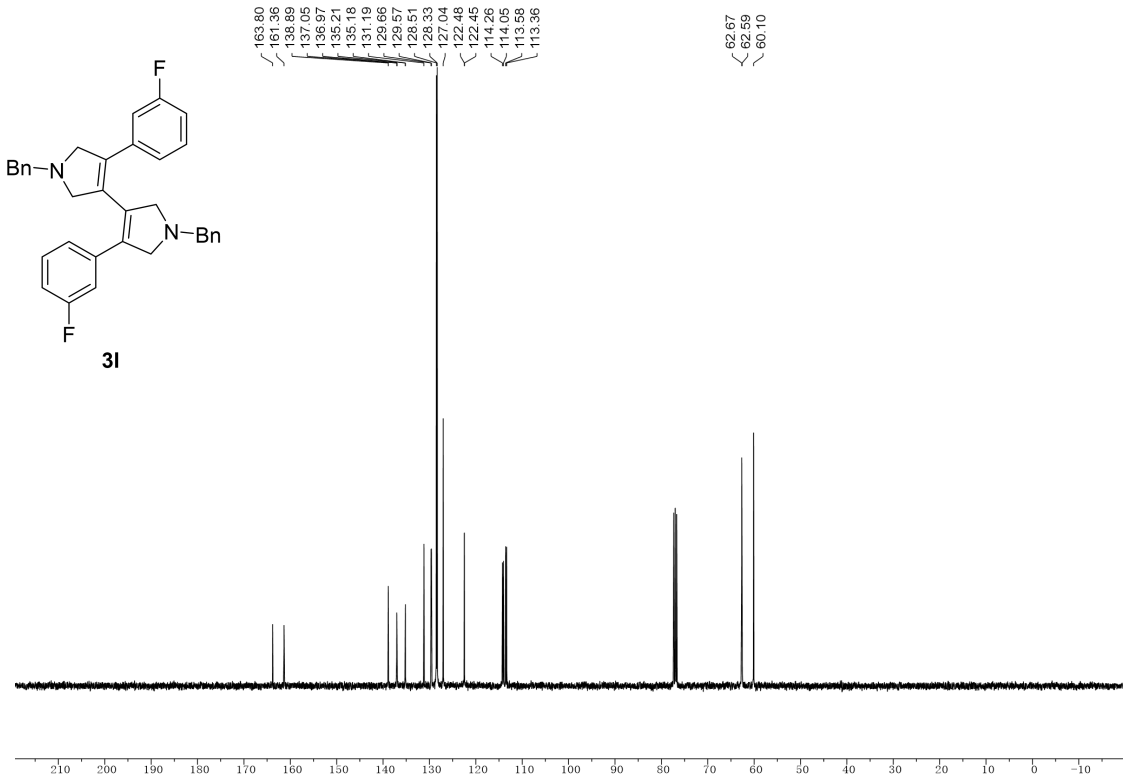
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3l:



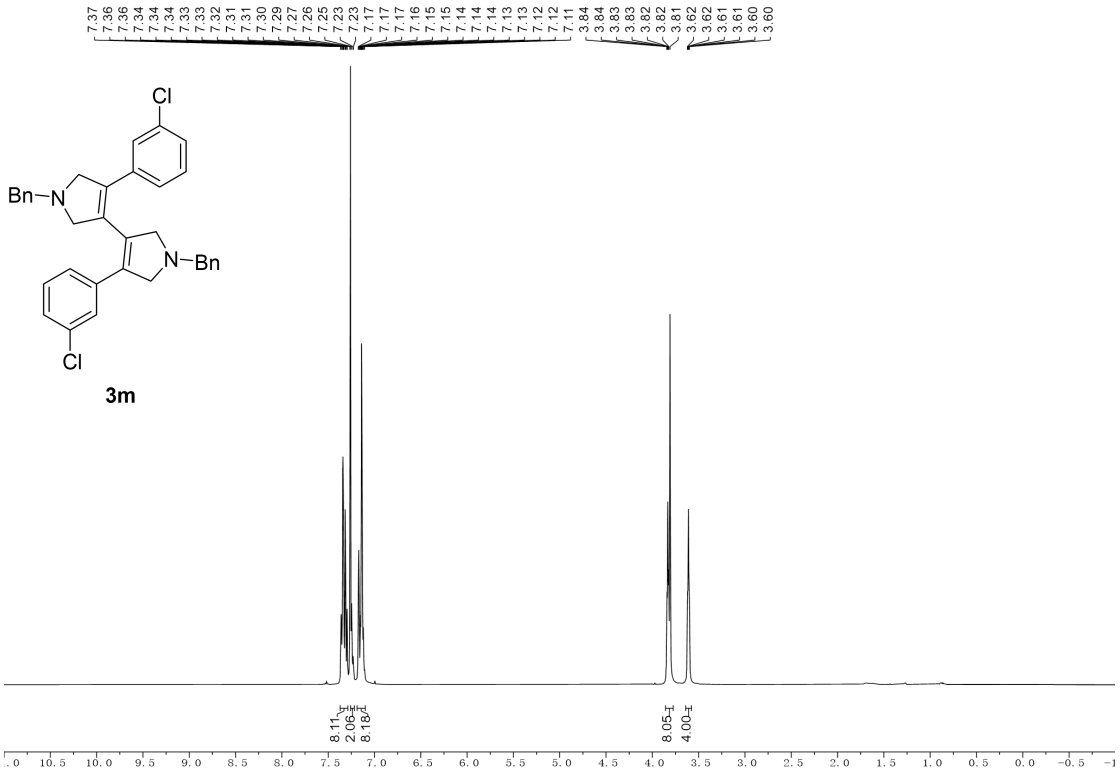
$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$) spectra of compound 3l:



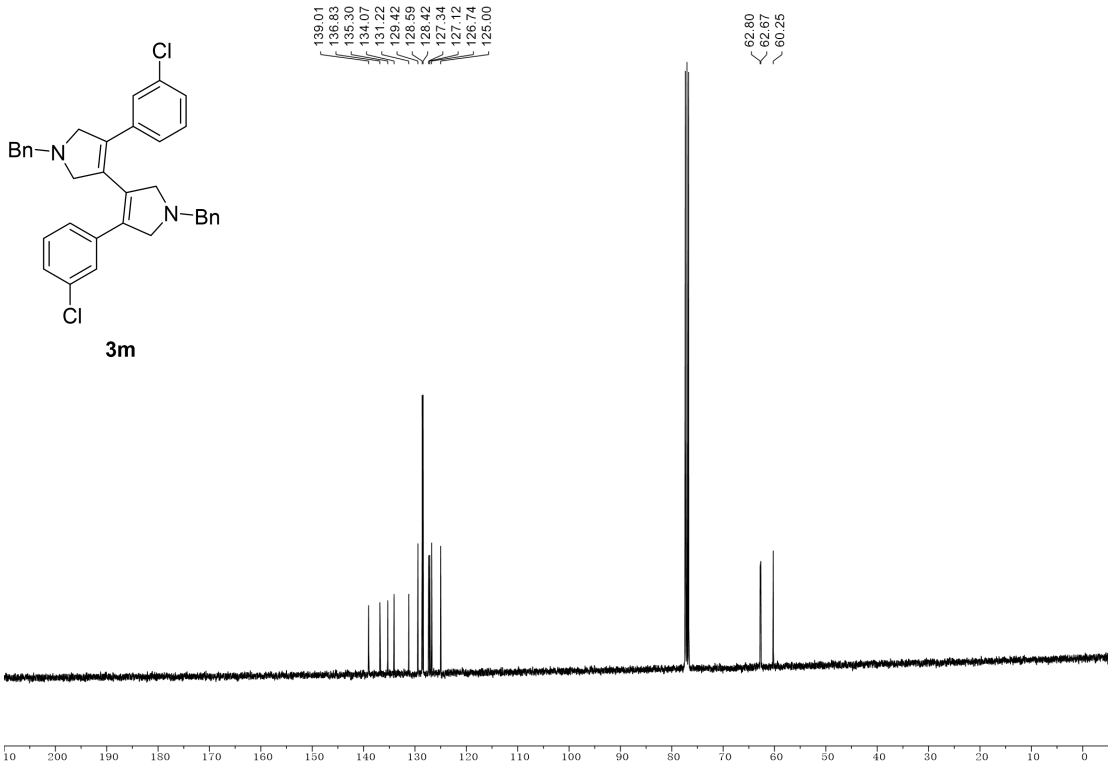
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 3l:



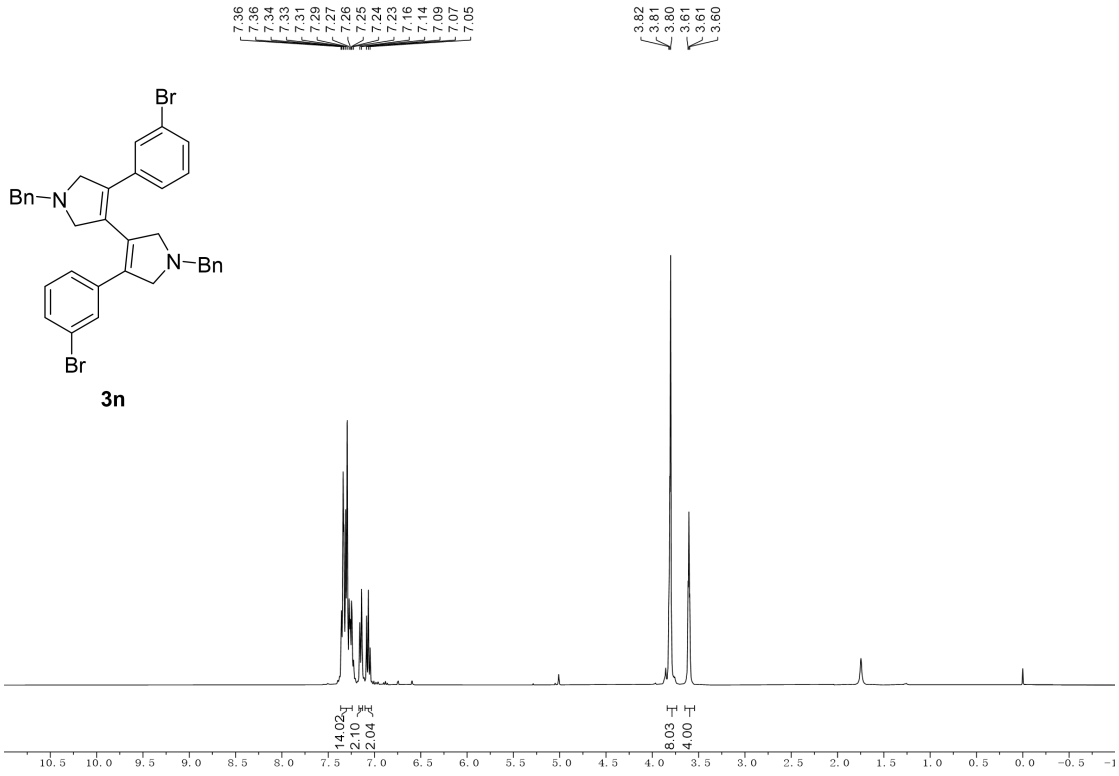
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3m:



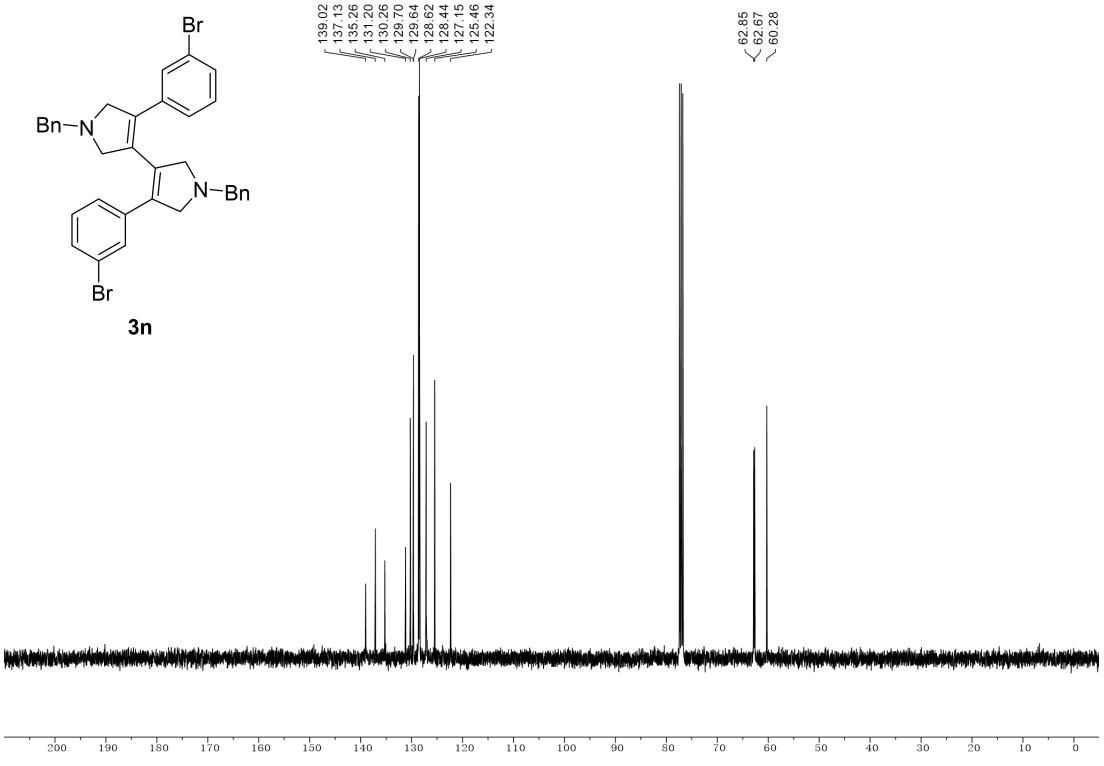
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3m:



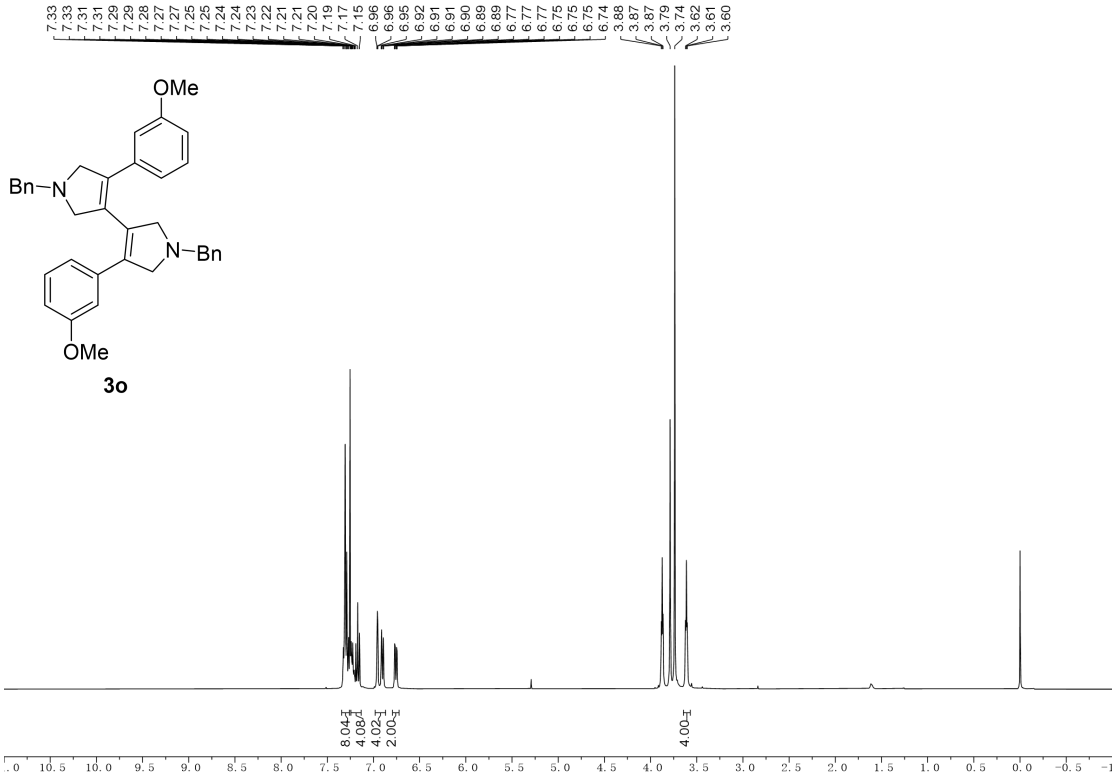
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3n:



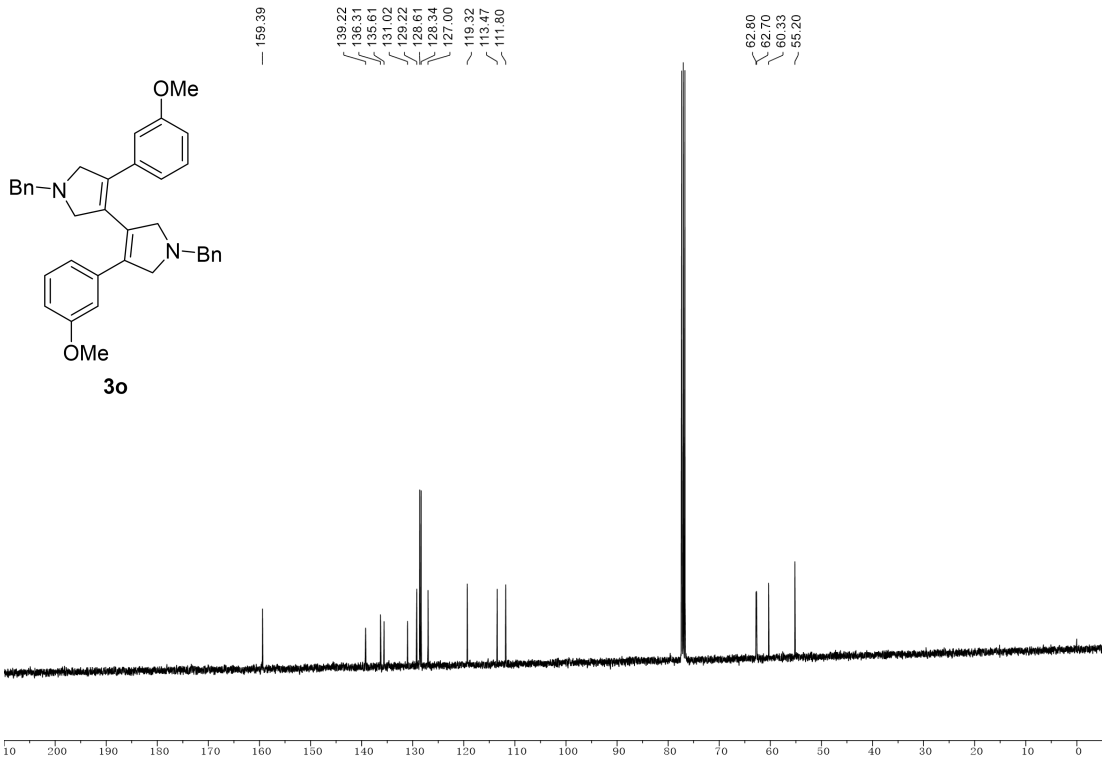
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3n:



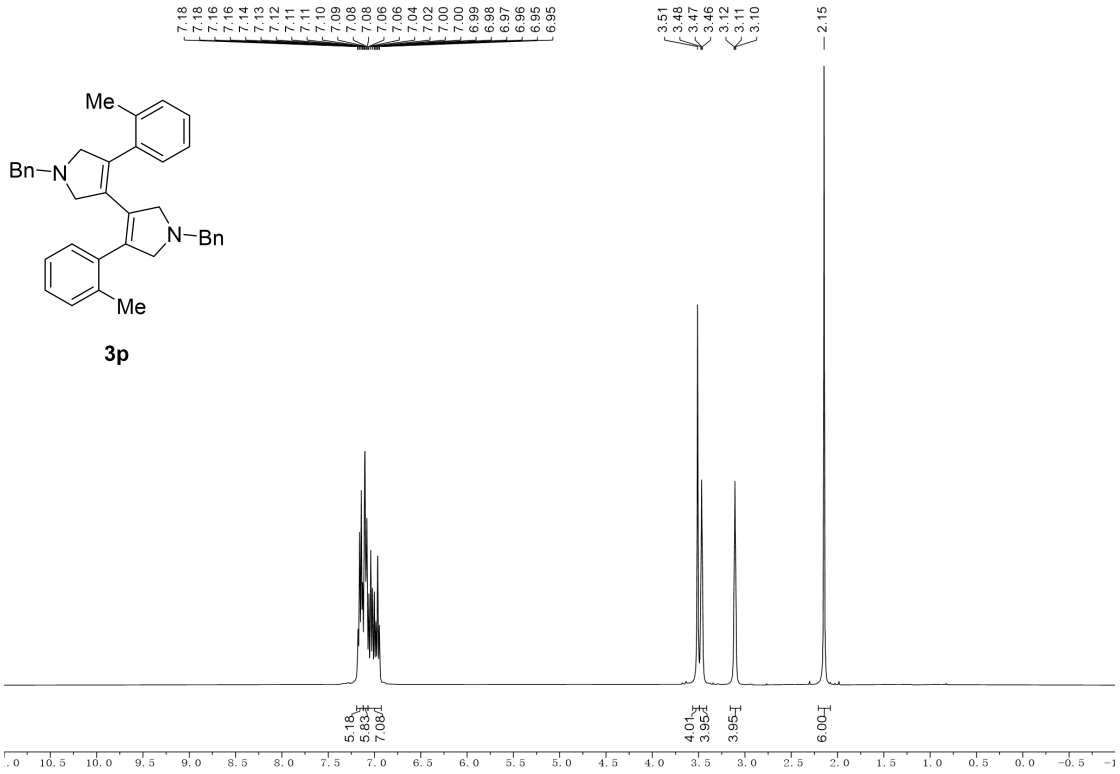
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3o:



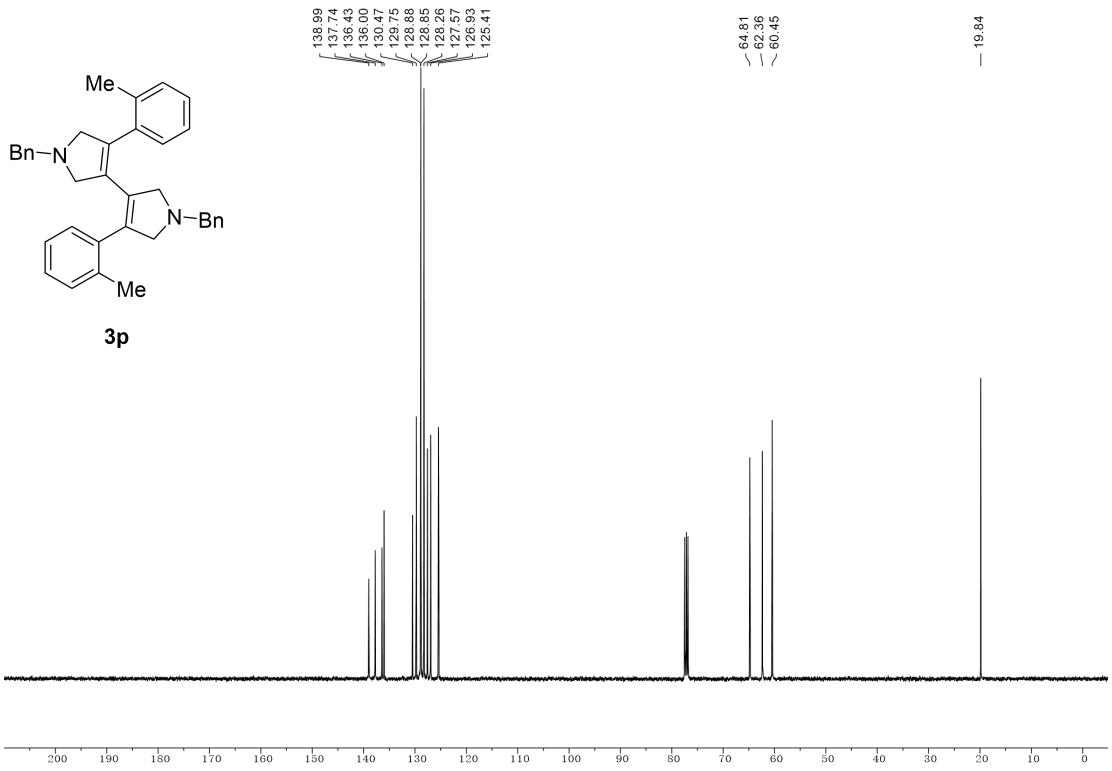
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3o:



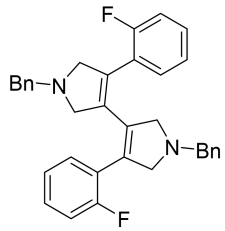
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3p:



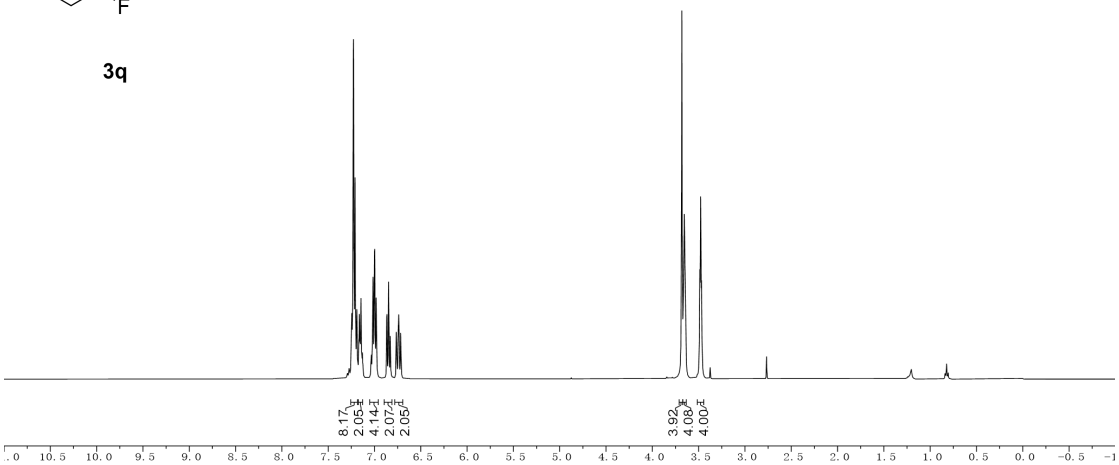
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3p:



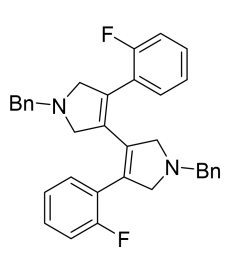
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3q:



3q

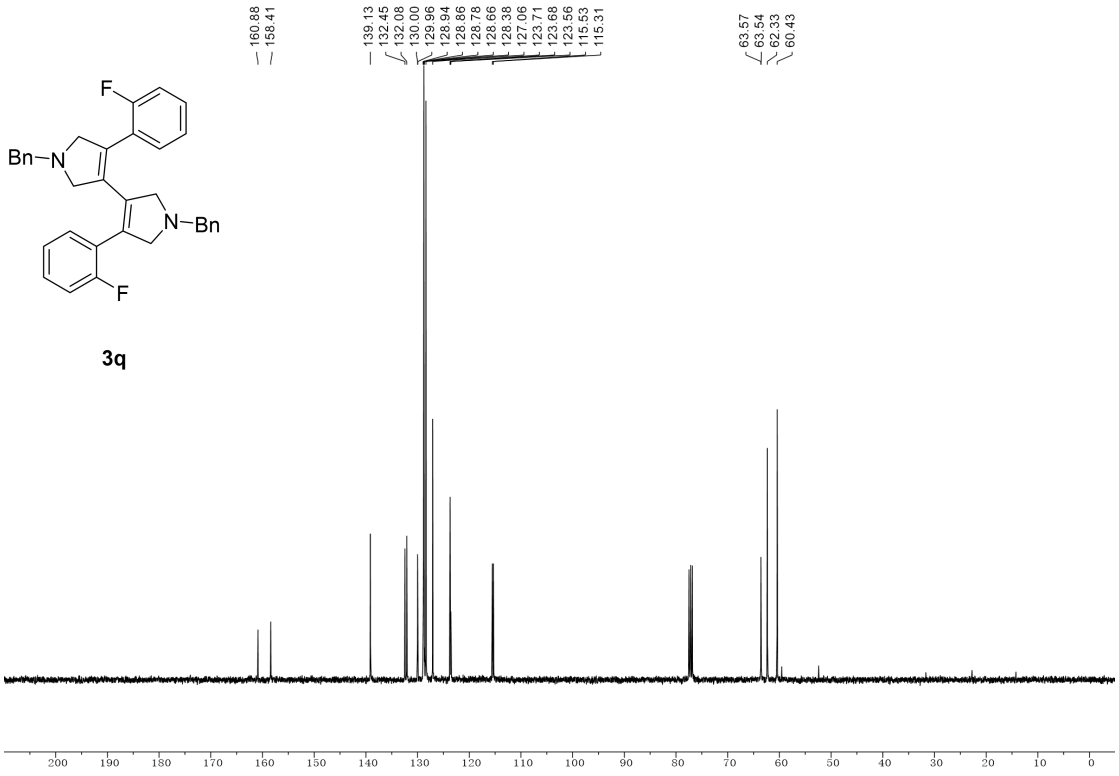


^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) spectra of compound 3q:

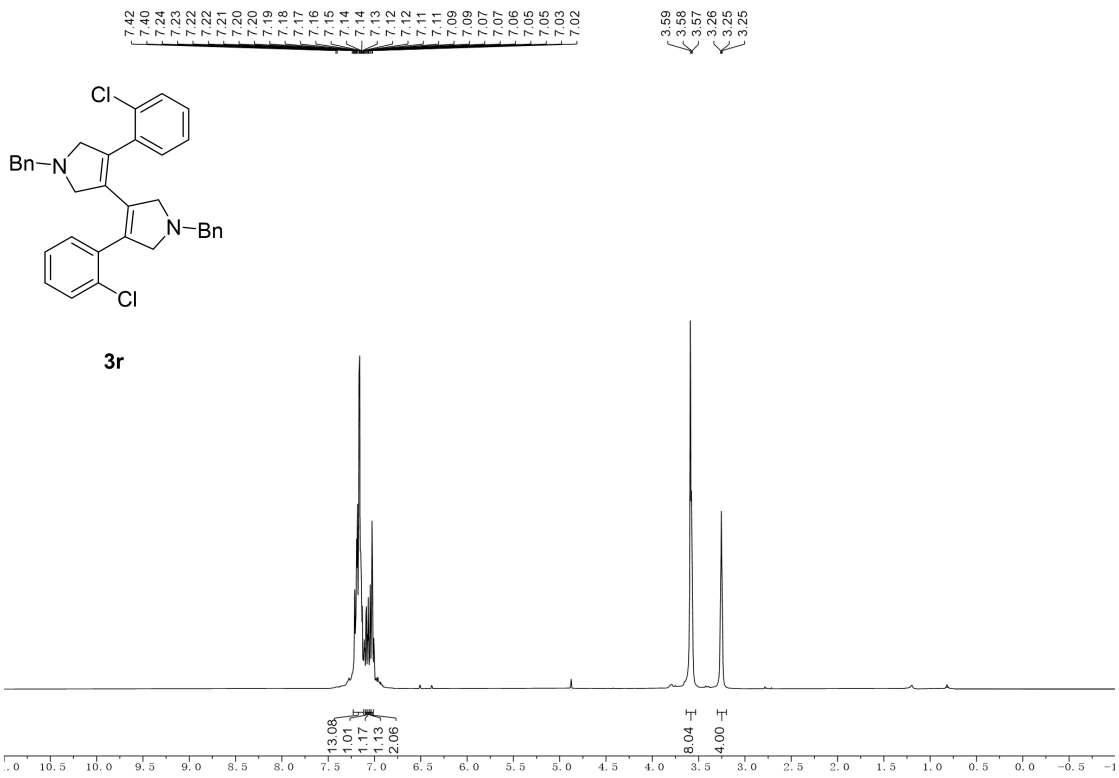


3q

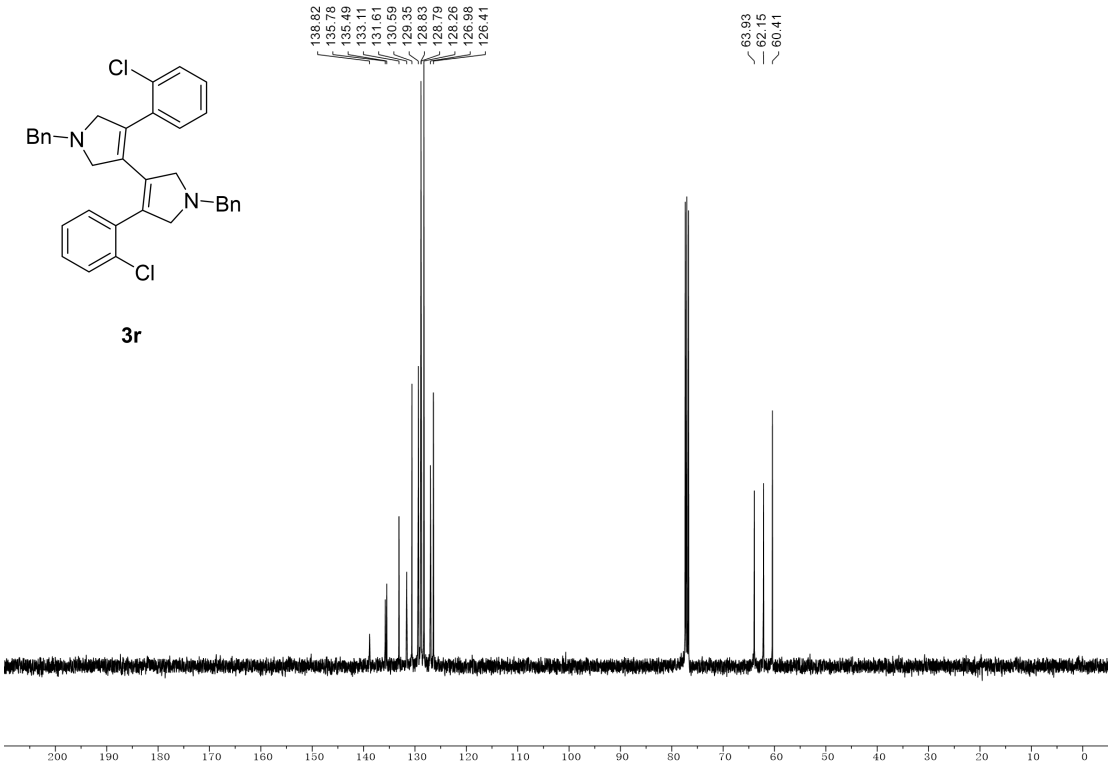
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 3q:



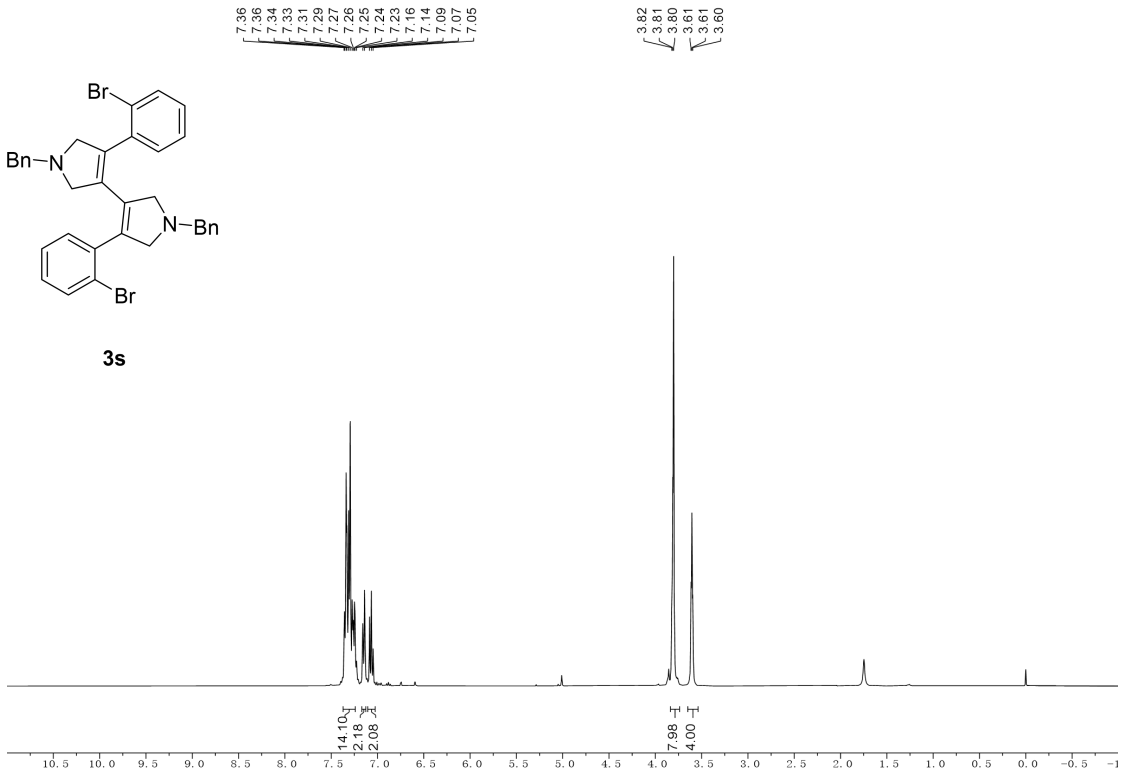
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3r:



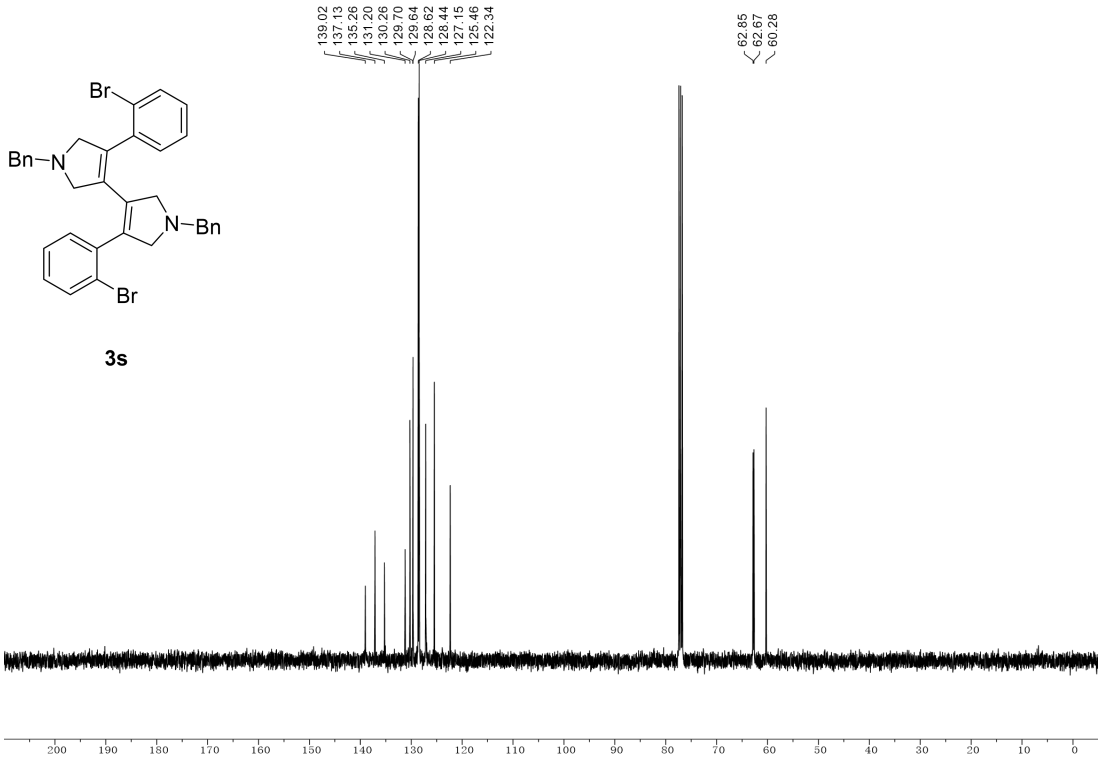
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of compound 3r:



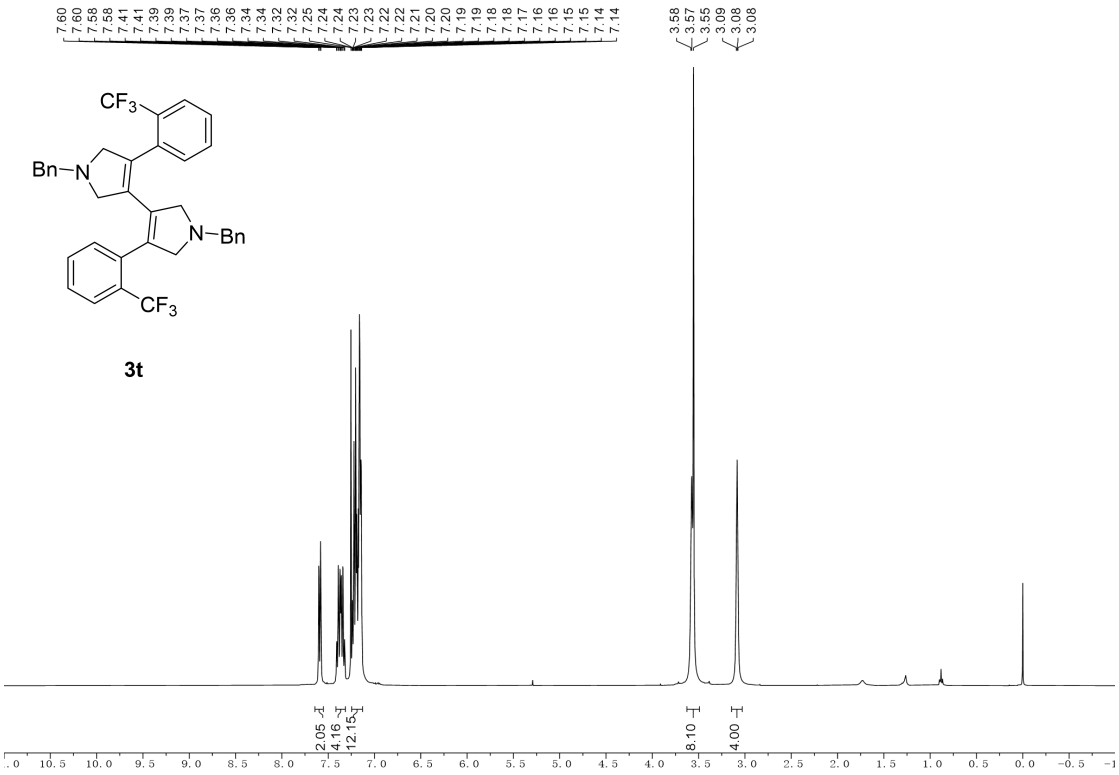
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3s:



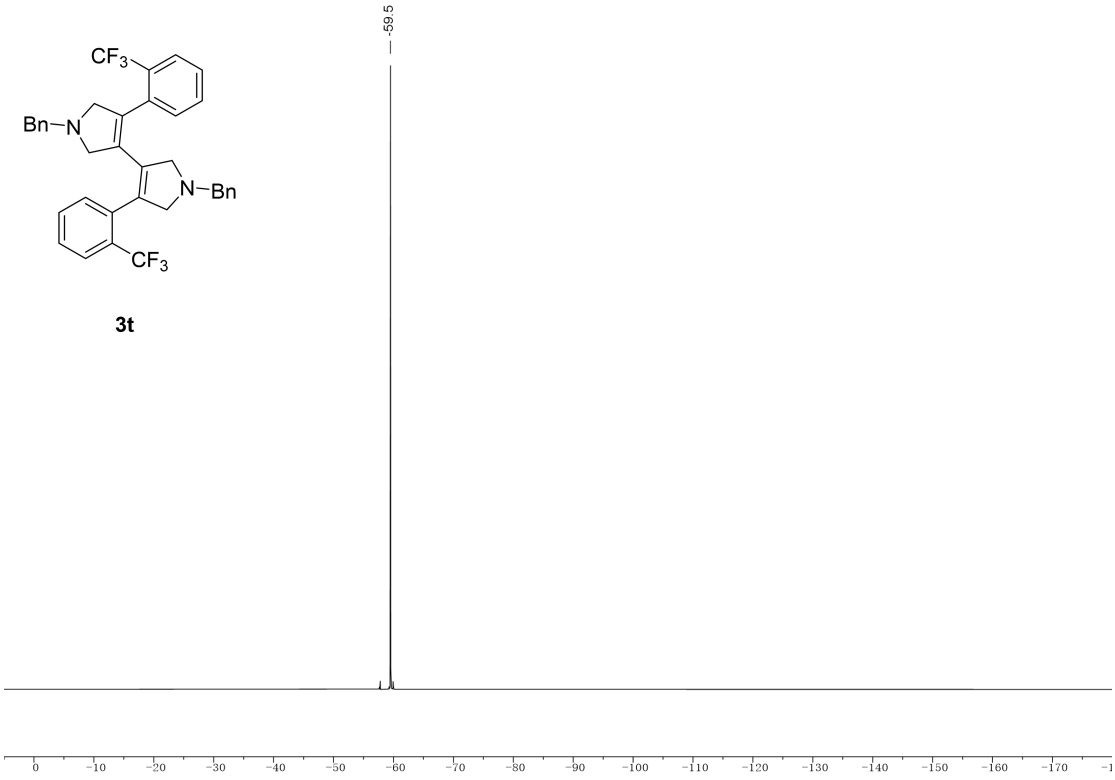
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of compound 3s:



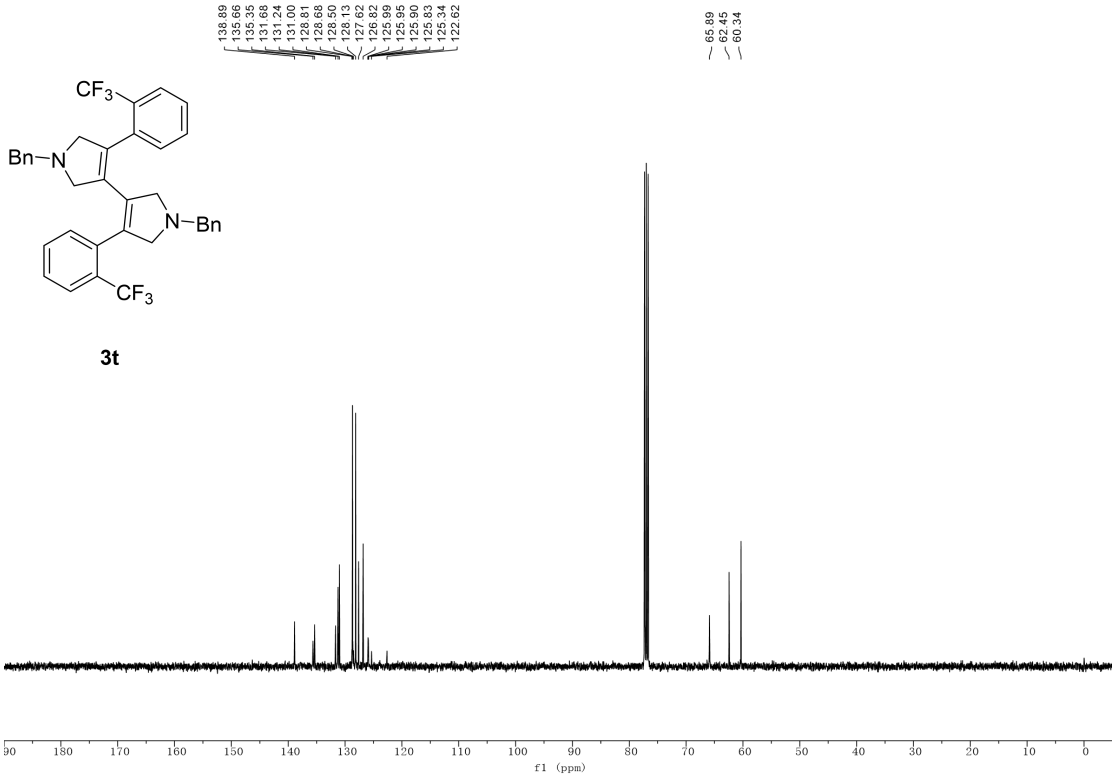
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3t:



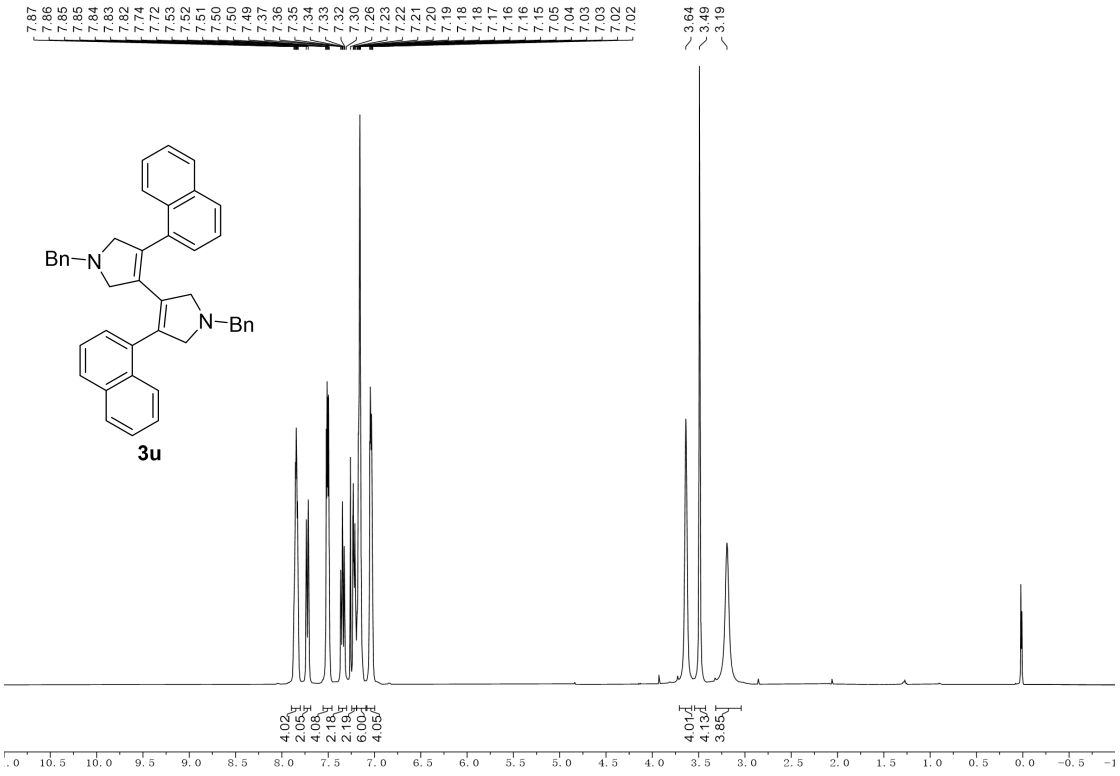
$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$) spectra of compound 3t:



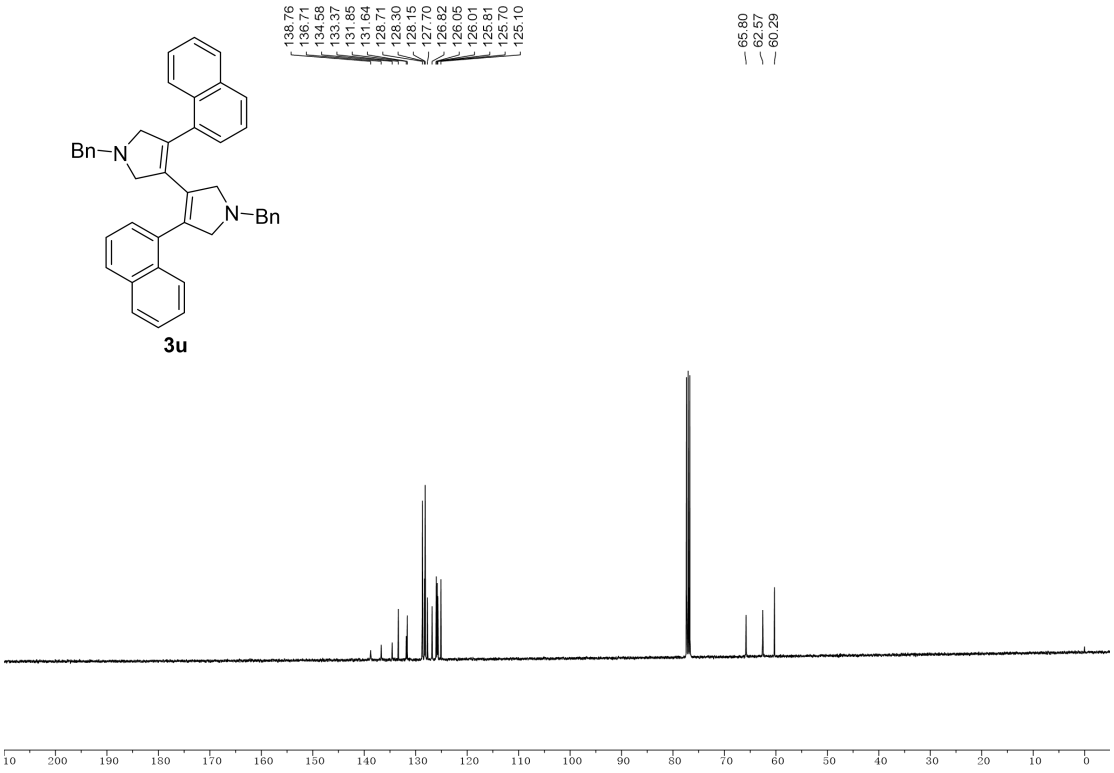
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 3t:



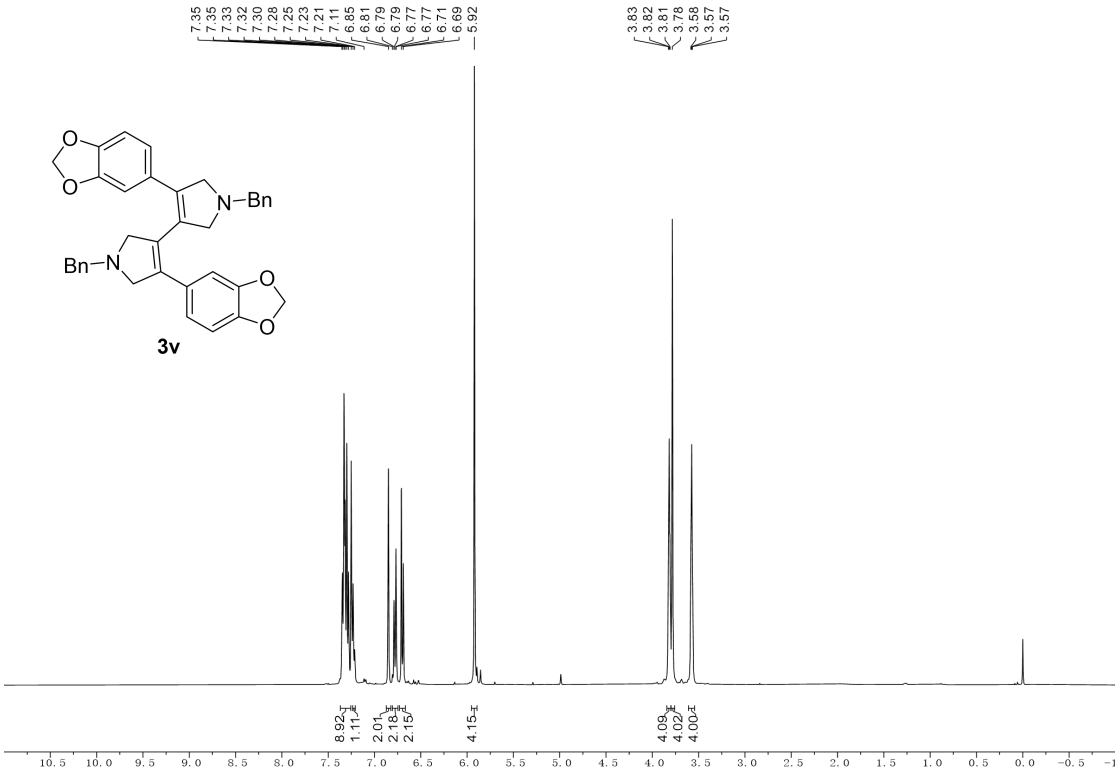
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3u:



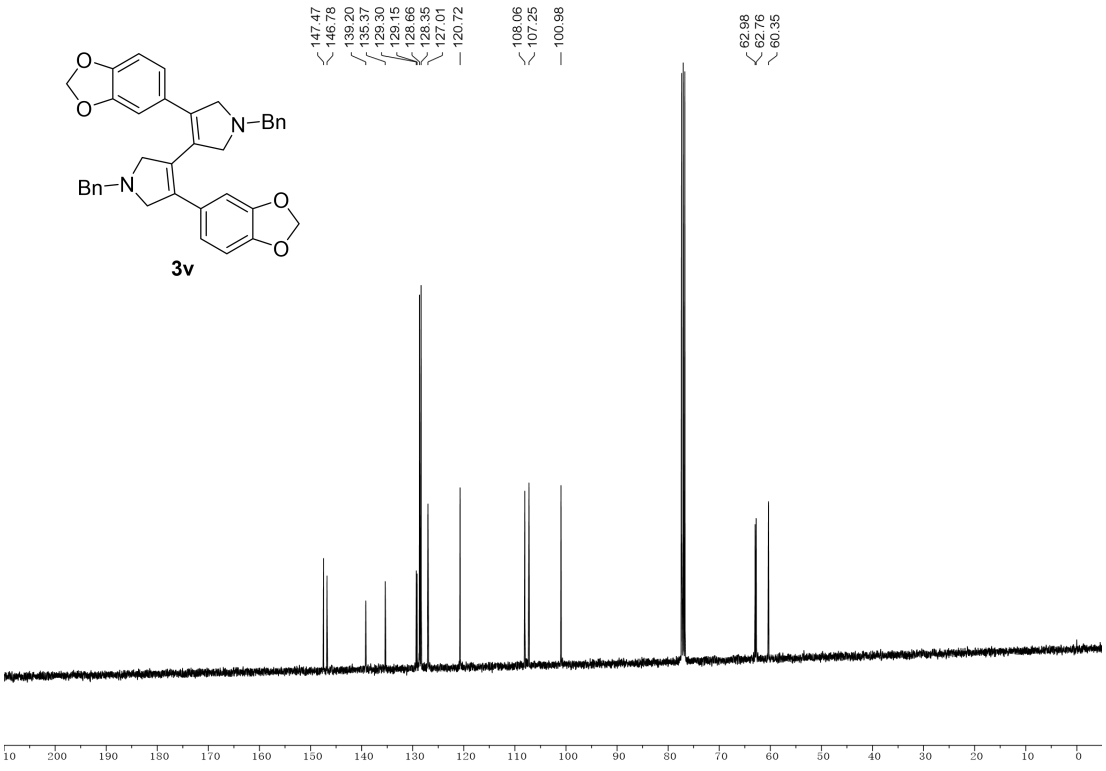
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3u:



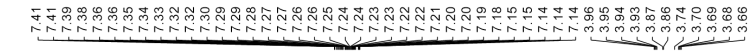
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3v:

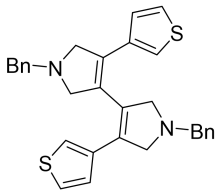


^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3v:

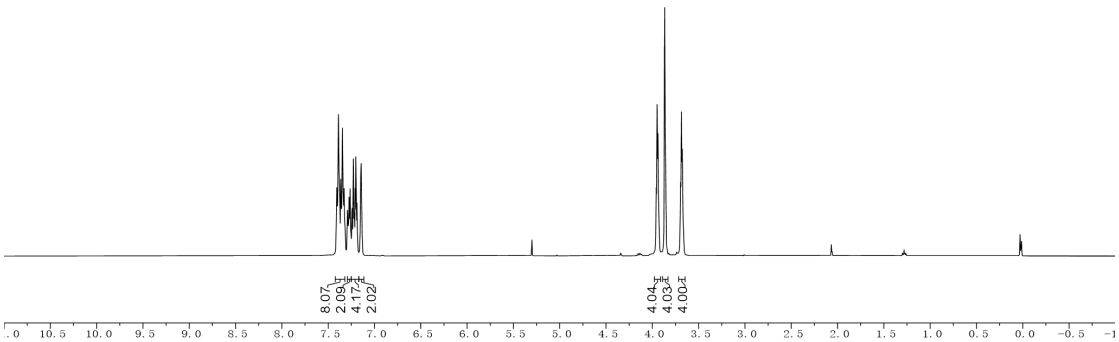


^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3w:

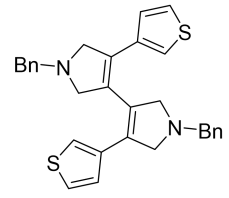




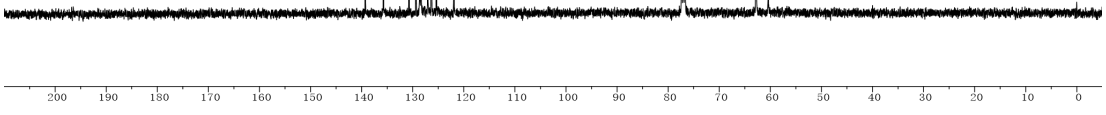
3w



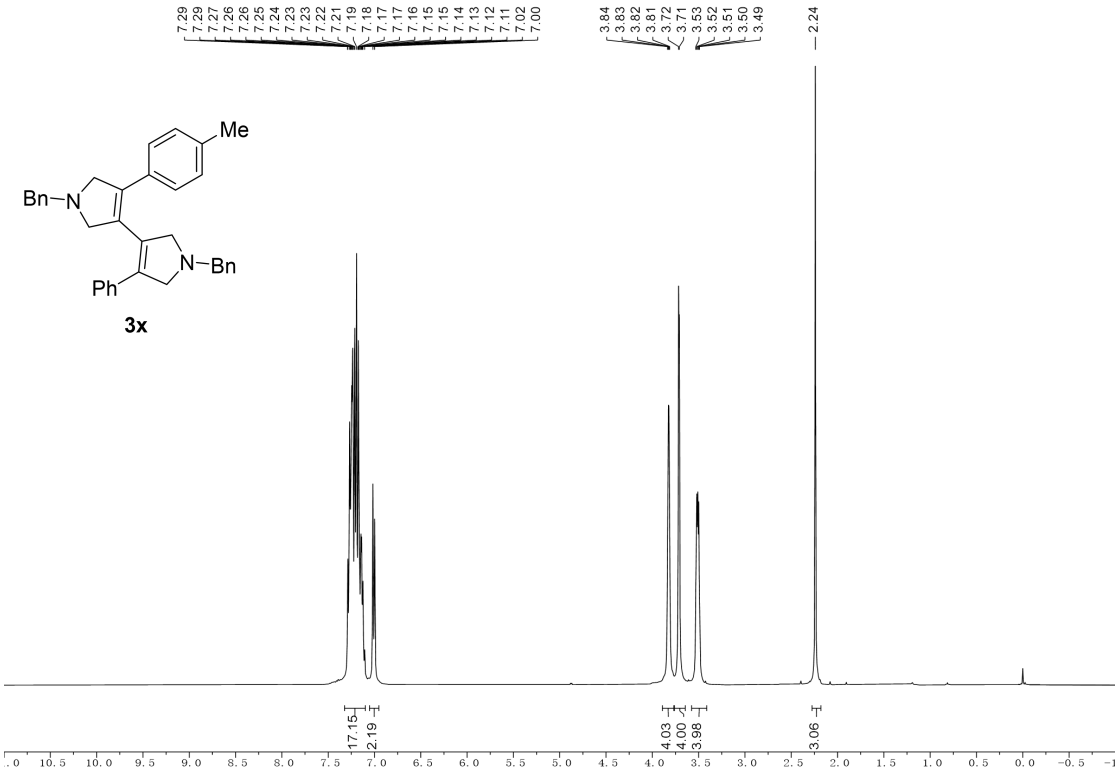
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3w:



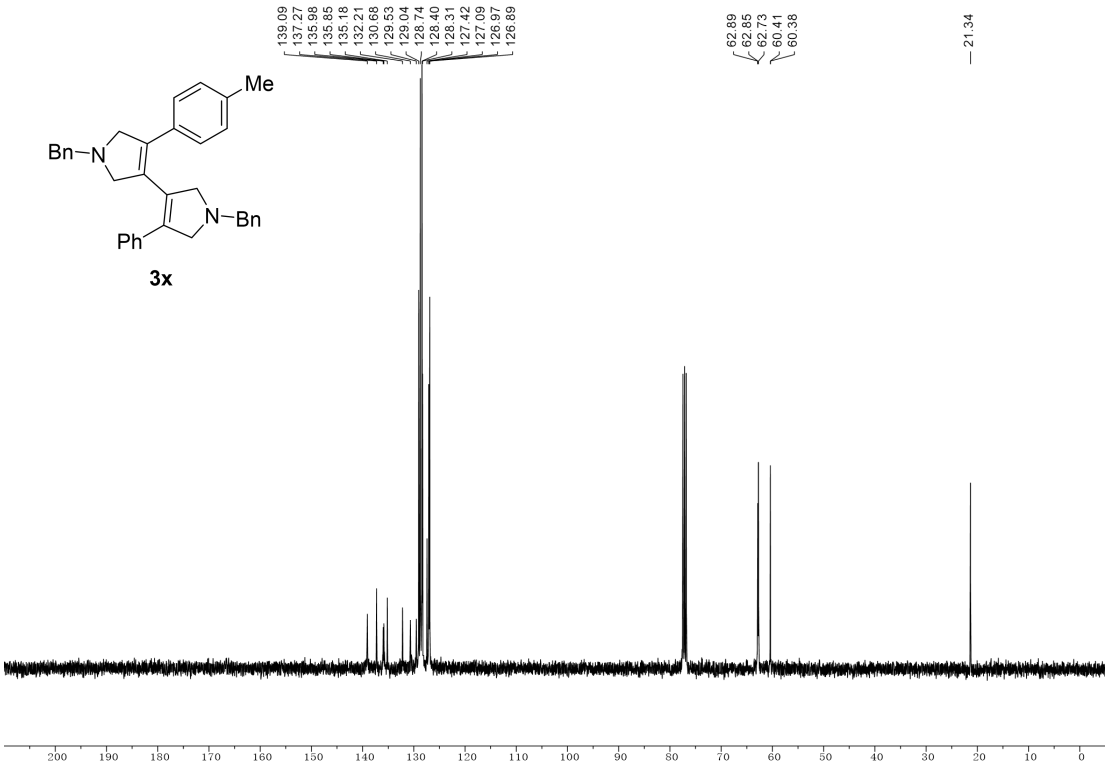
3w



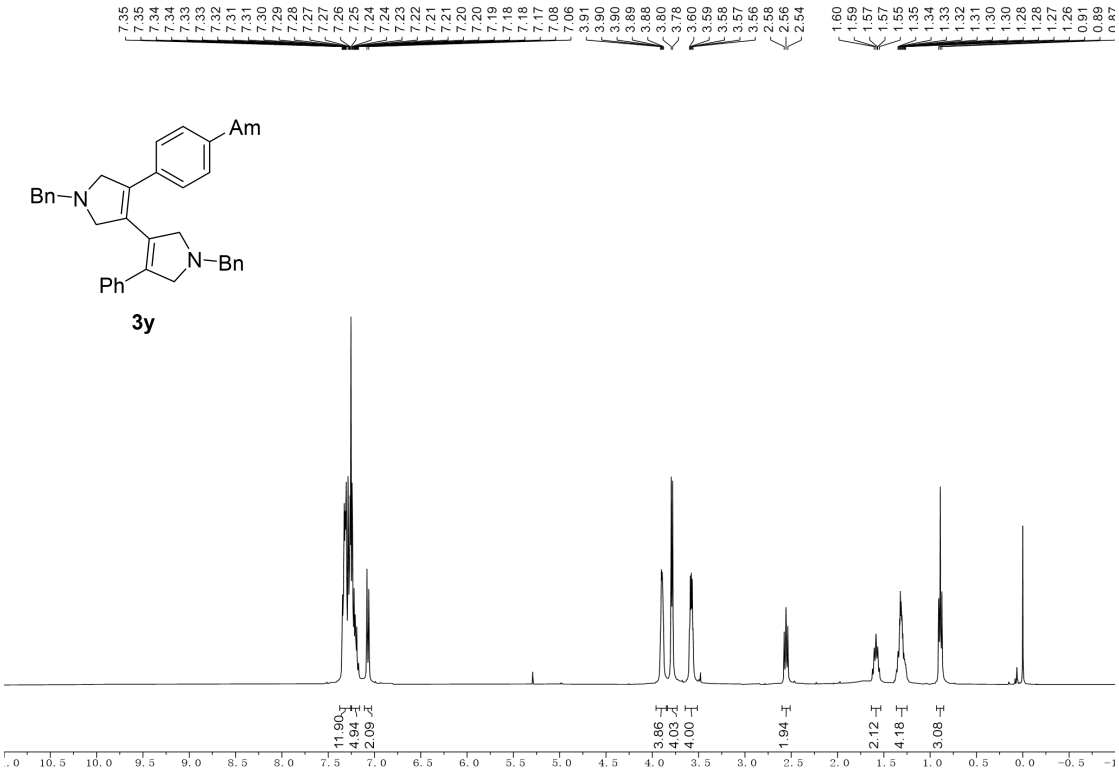
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3x:



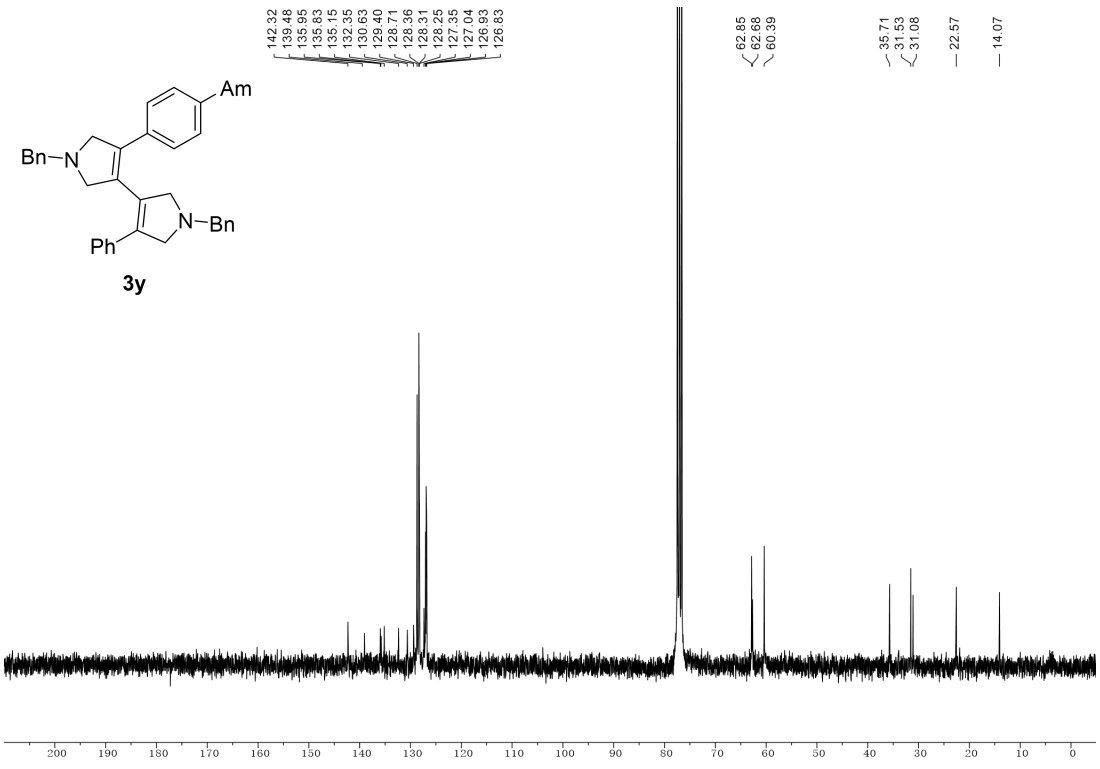
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3x:



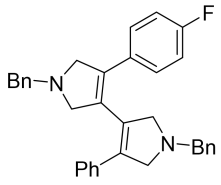
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3y:



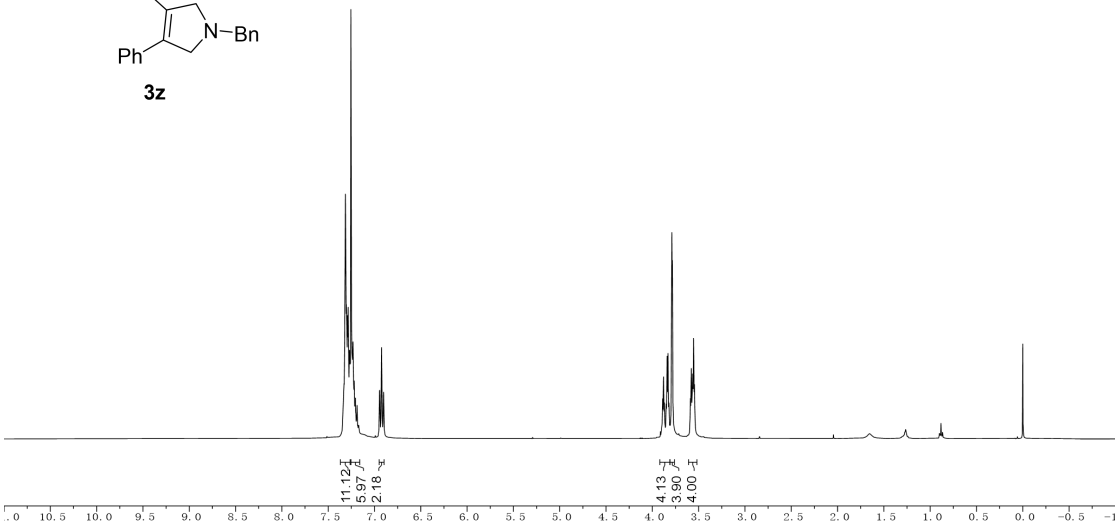
^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) spectrum of compound 3y:



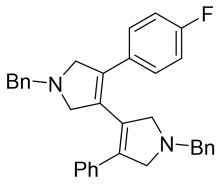
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3z:



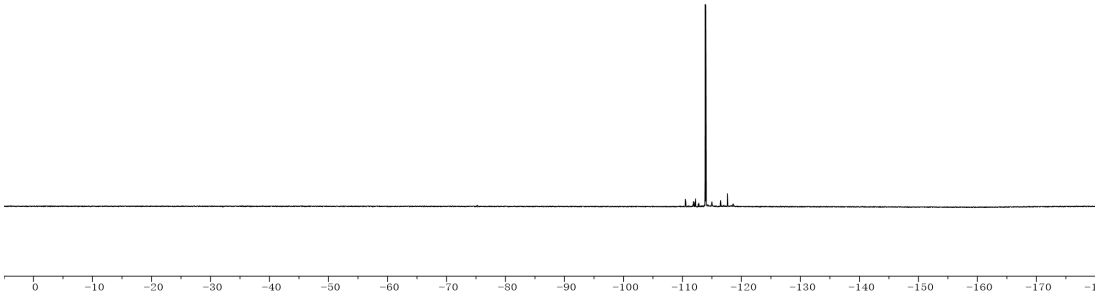
3z



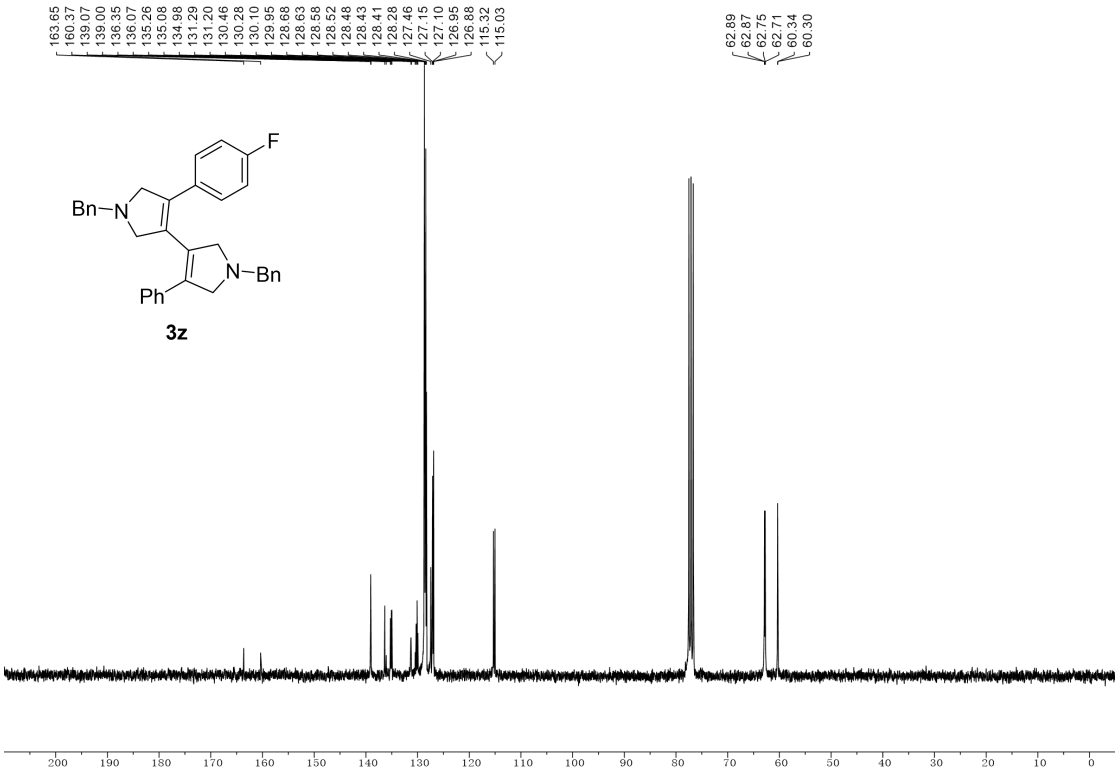
^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) spectra of compound 3z:



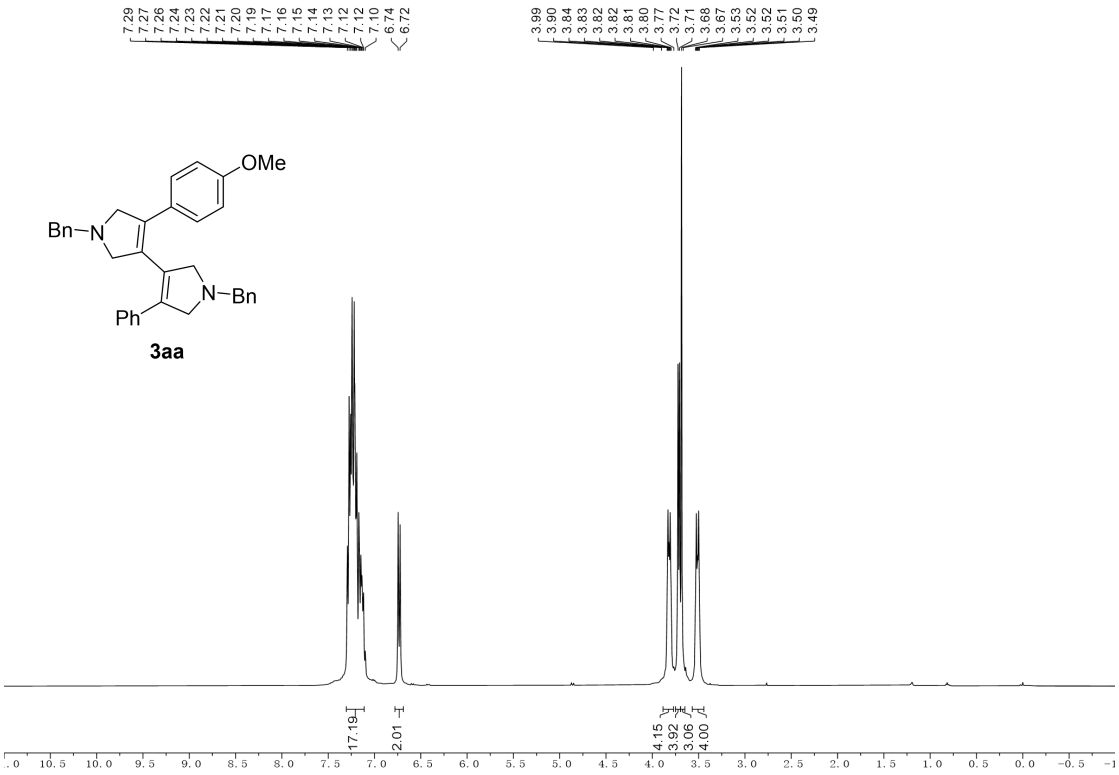
3z



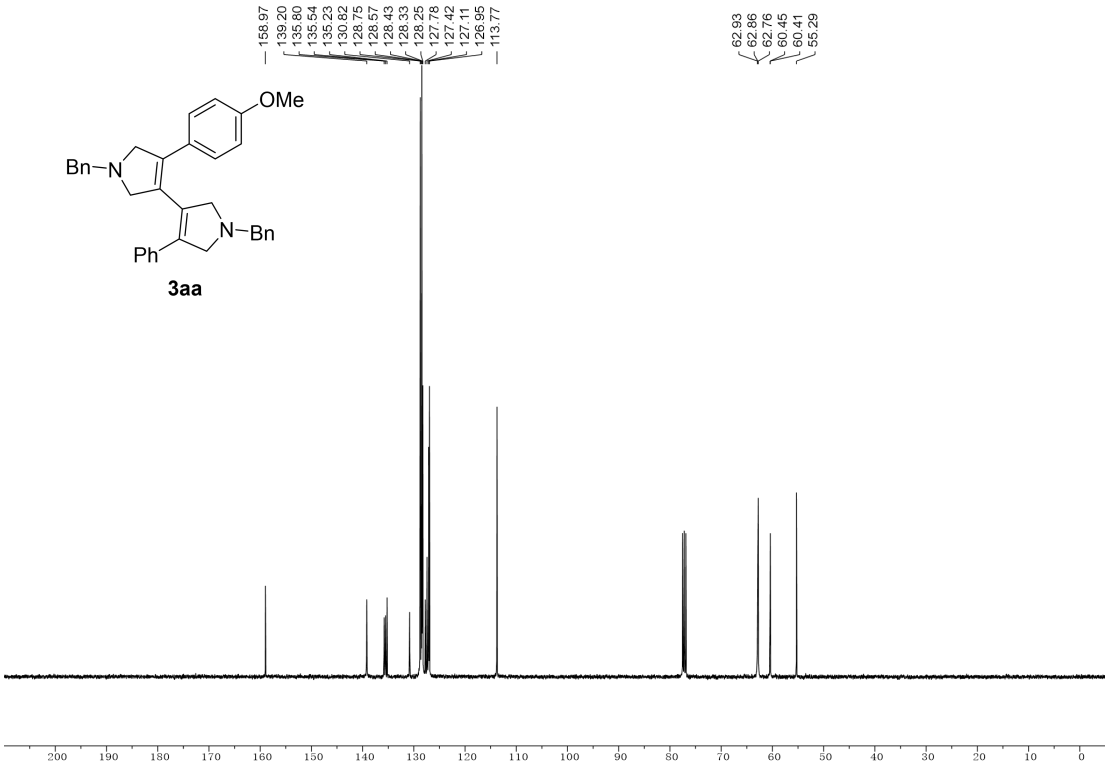
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 3z:



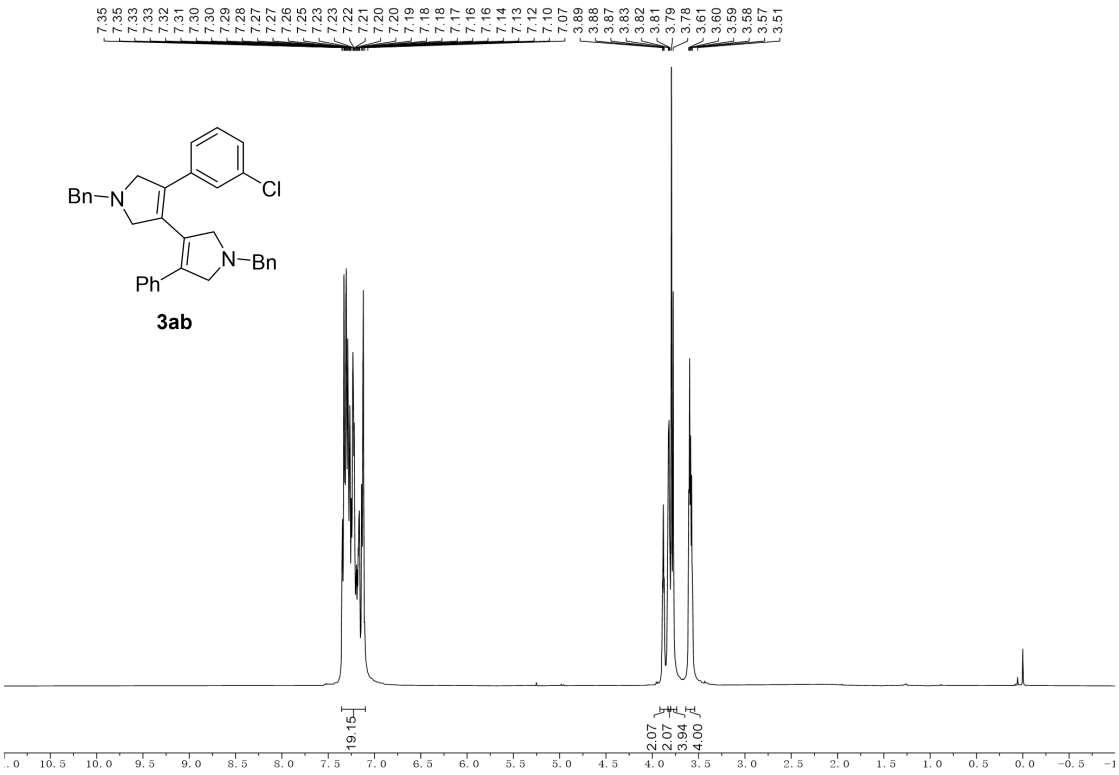
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3aa:



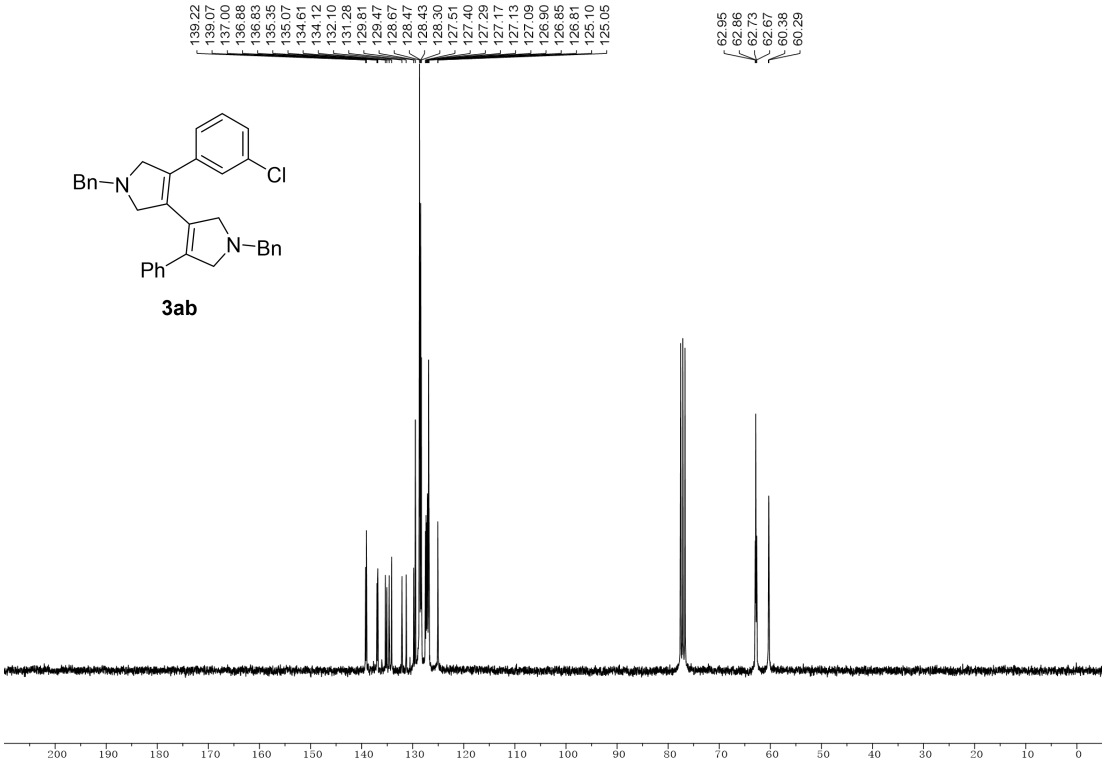
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectrum of compound 3aa:



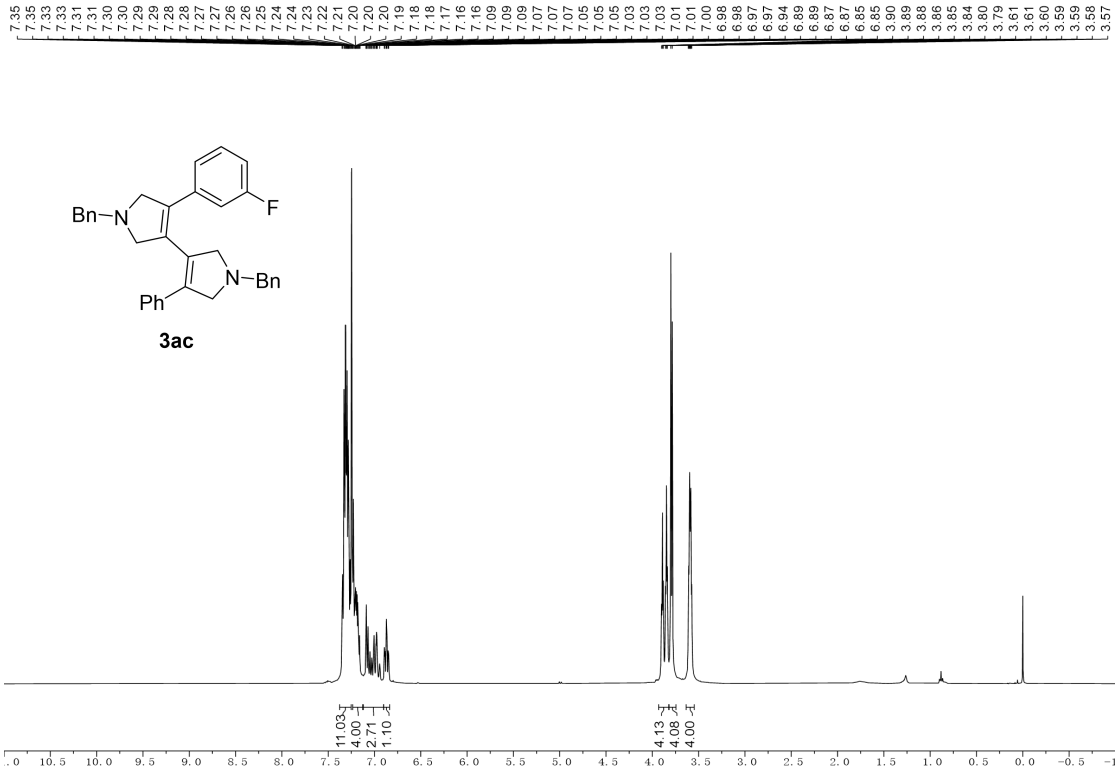
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3ab:



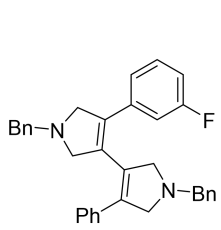
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectrum of compound 3ab:



^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3ac:

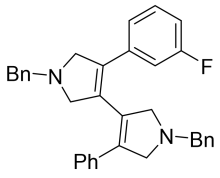


$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$) spectra of compound 3ac:



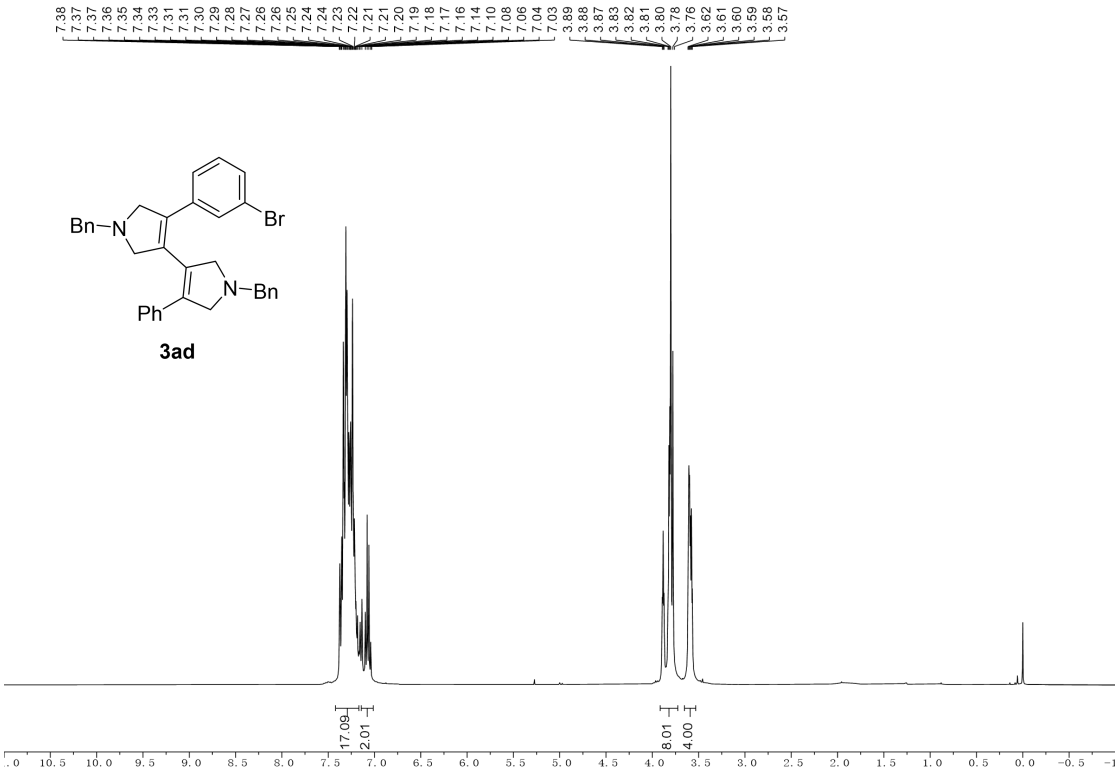
3ac

$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 3ac:

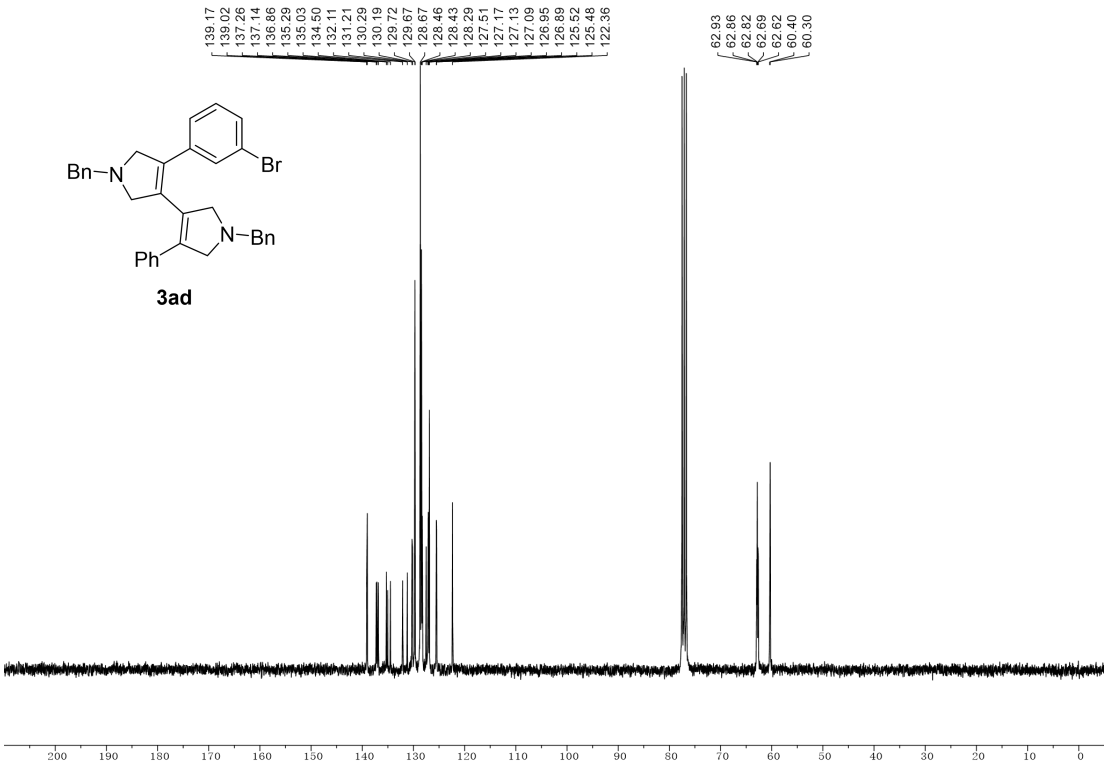


3ac

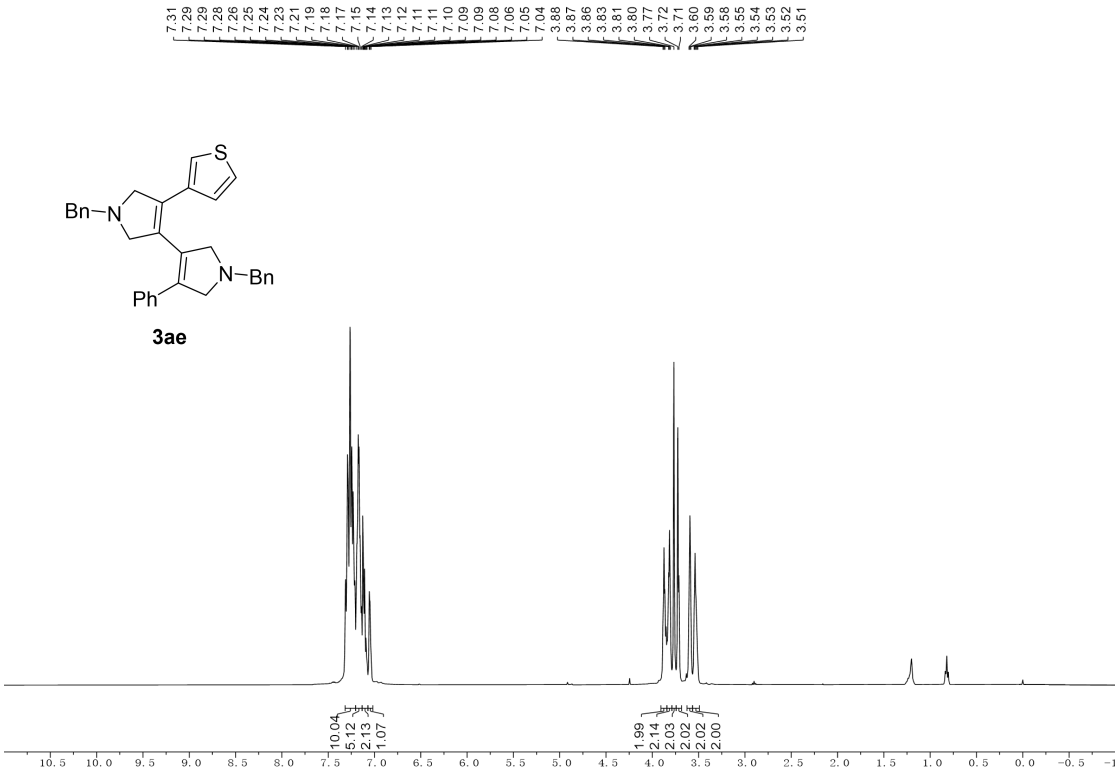
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3ad:



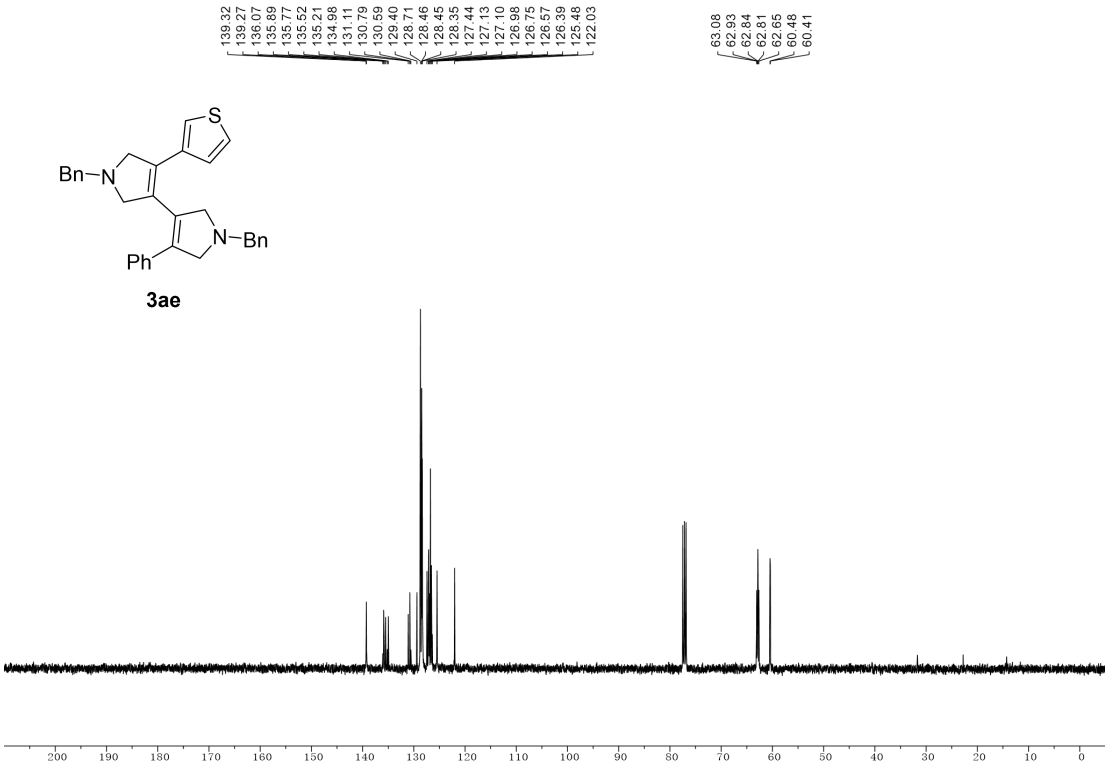
^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) spectrum of compound 3ad:



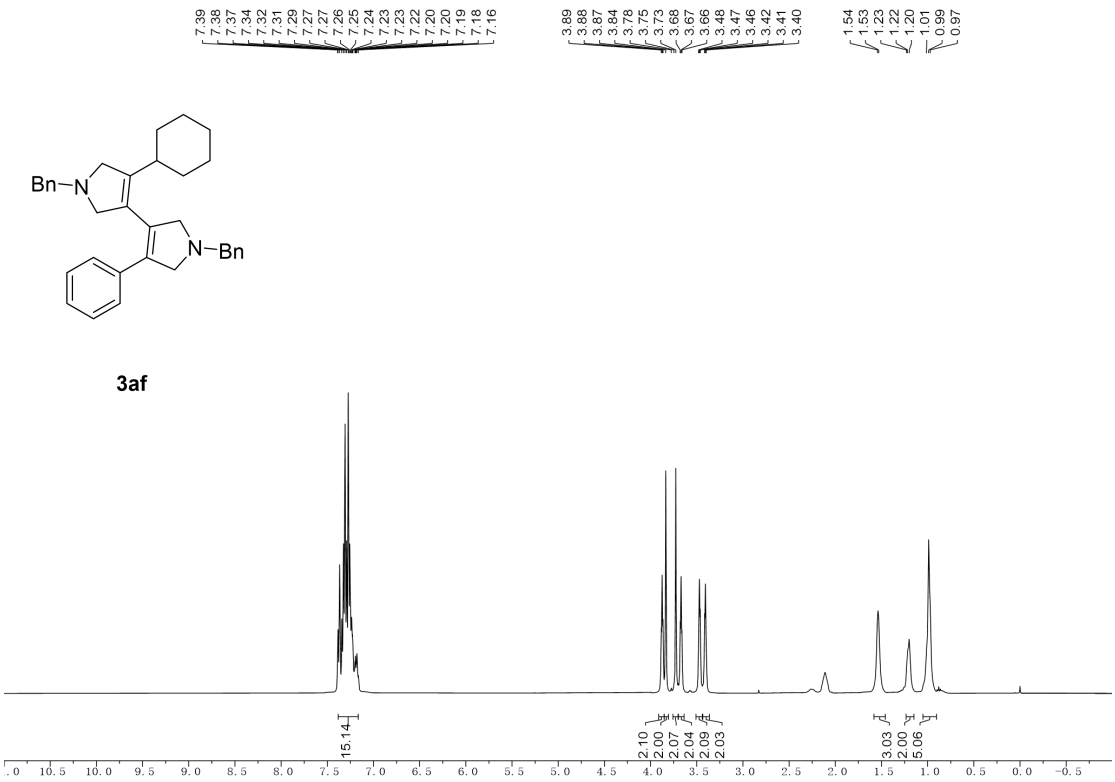
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of compound 3ae:



^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectrum of compound 3ae:

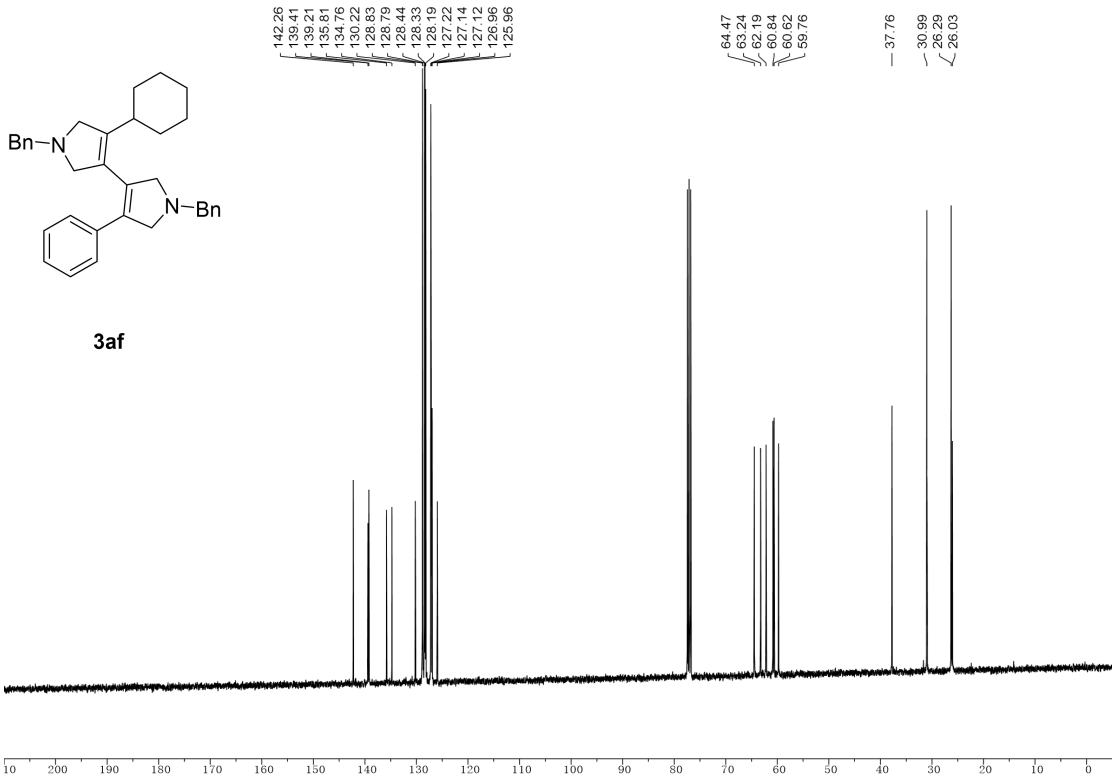


^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3af:



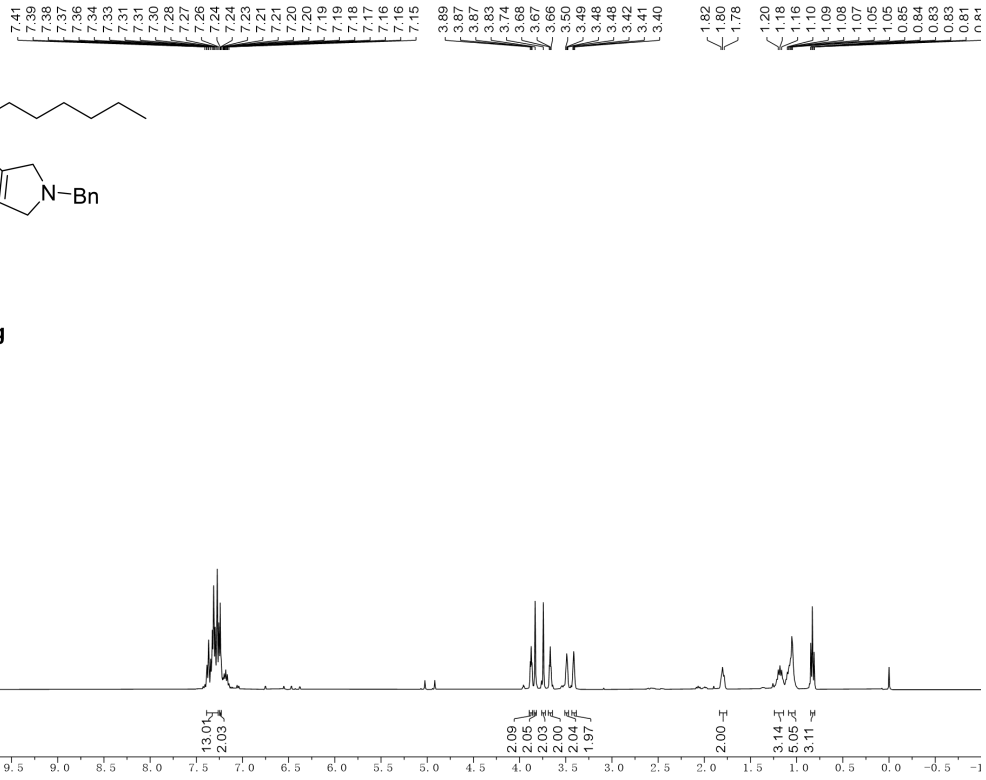
3af

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 3af:



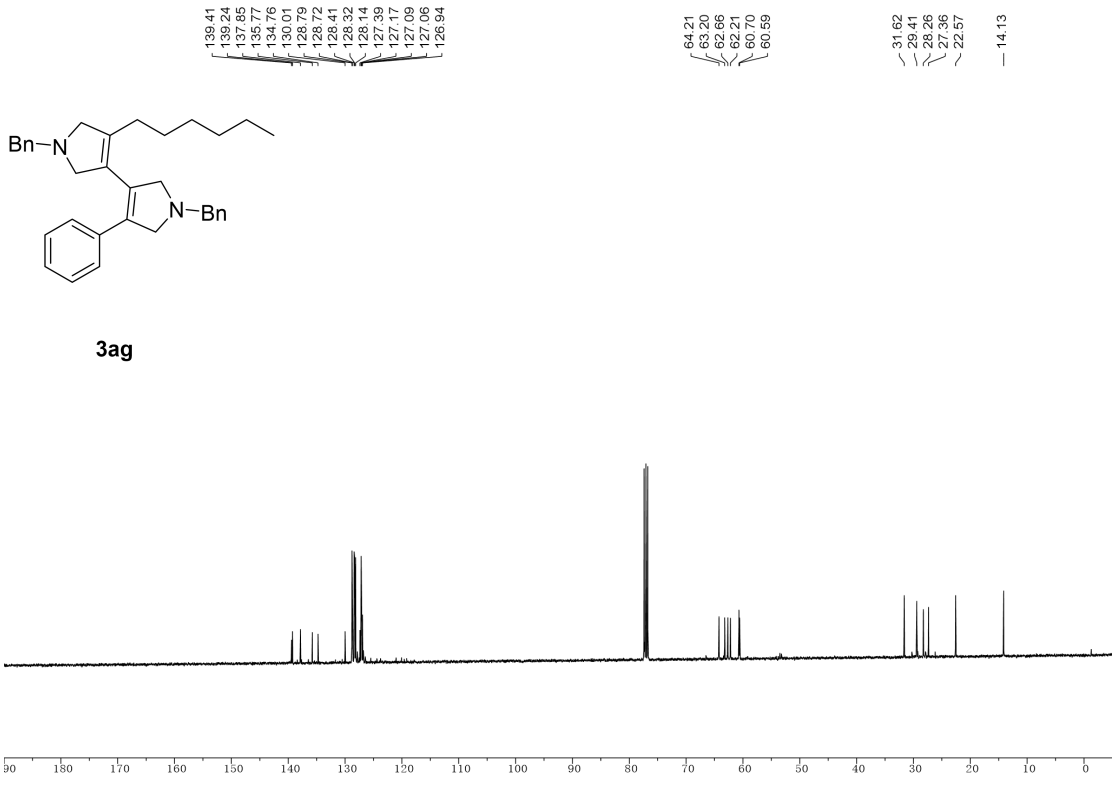
3af

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 3ag:



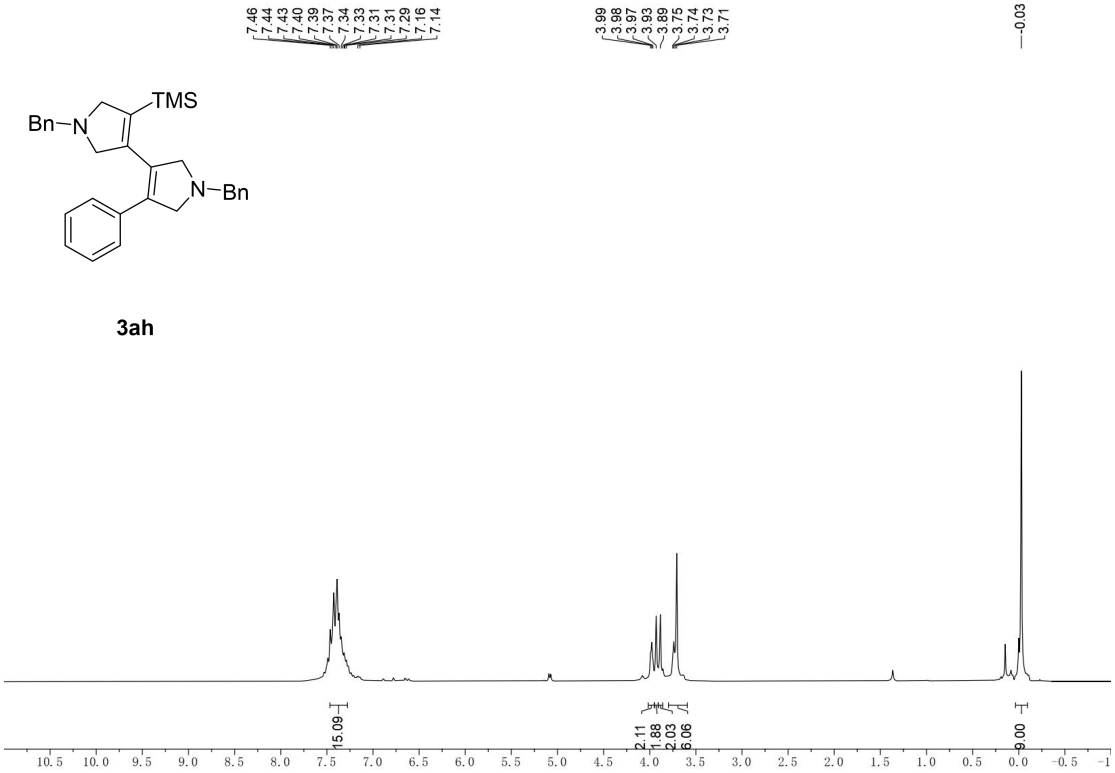
3ag

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 3ag:



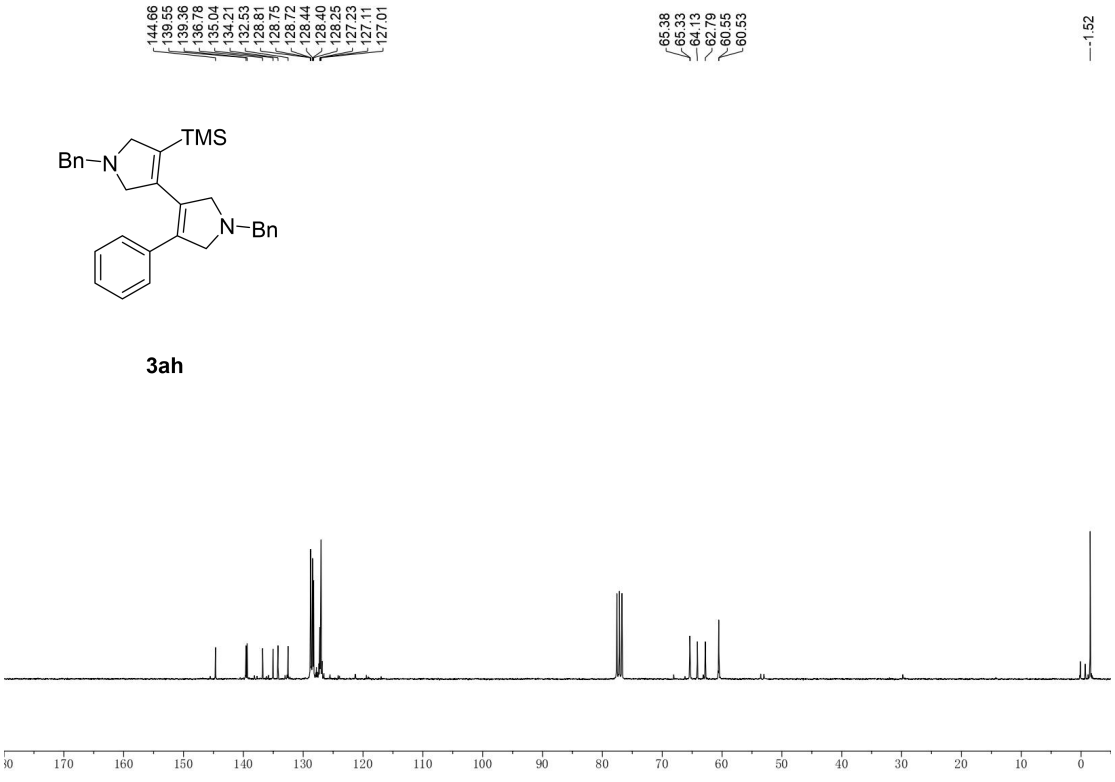
3ag

^{1}H NMR (300 MHz, $CDCl_3$) spectra of compound 3ah:



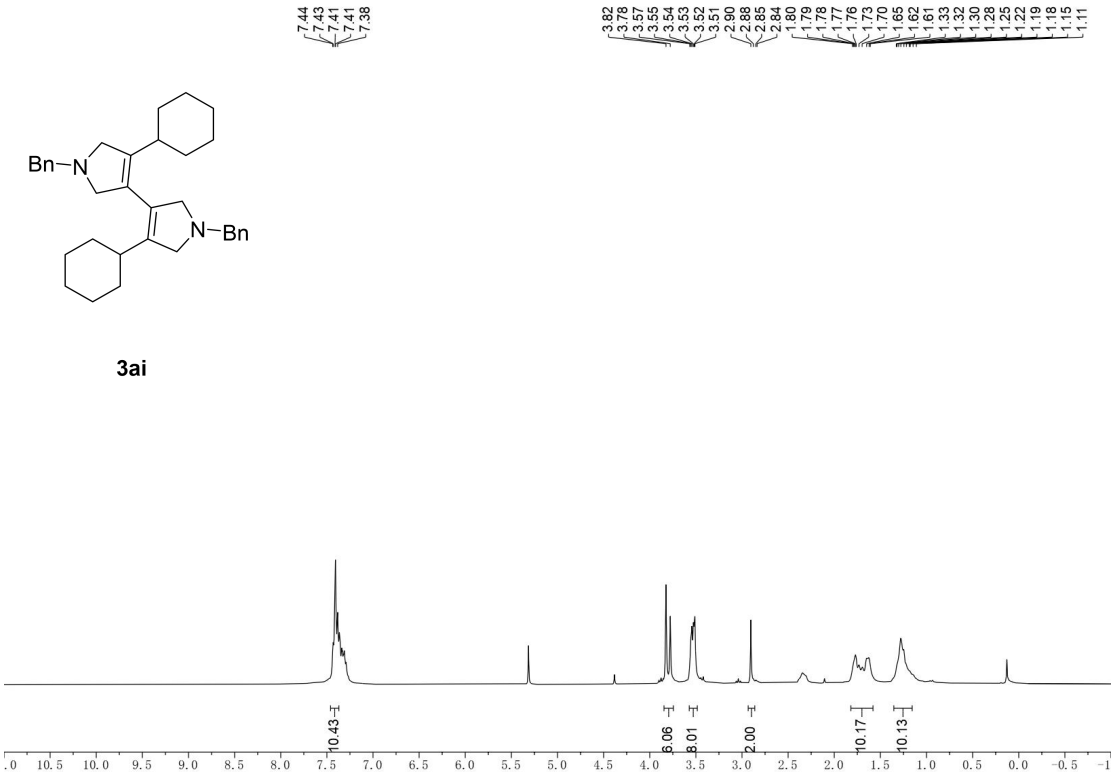
3ah

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) spectra of compound 3ah:



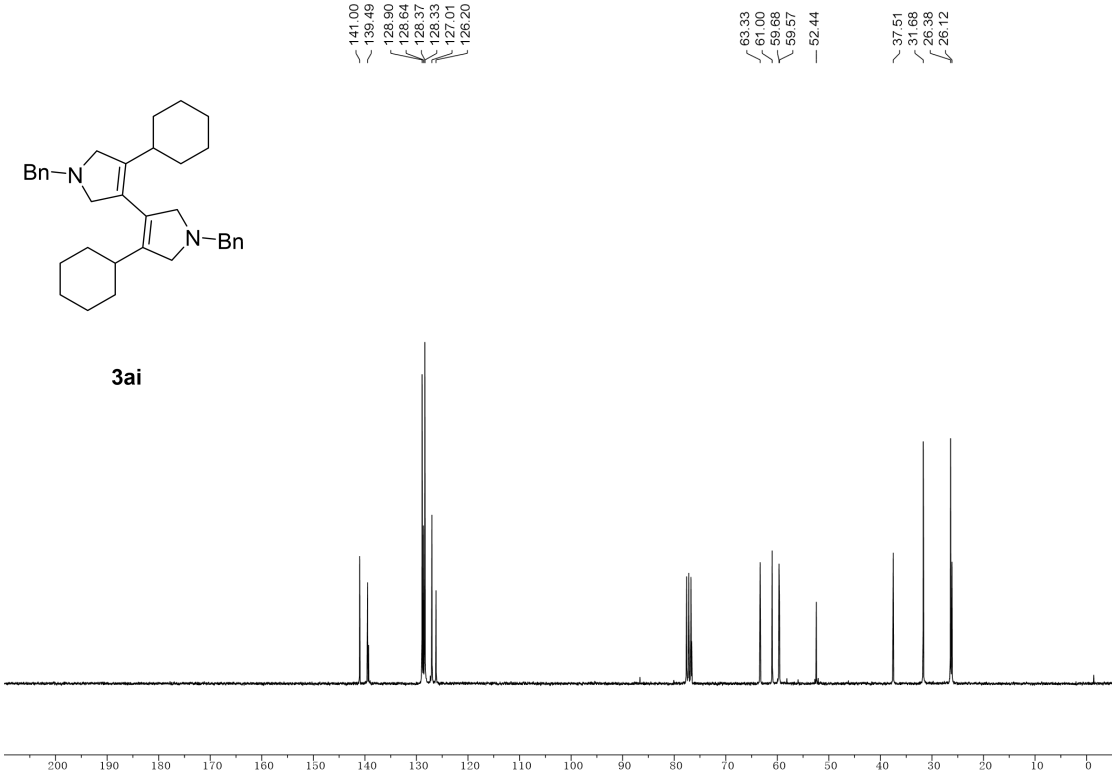
3ah

^{1}H NMR (300 MHz, $CDCl_3$) spectra of compound 3ai:



3ai

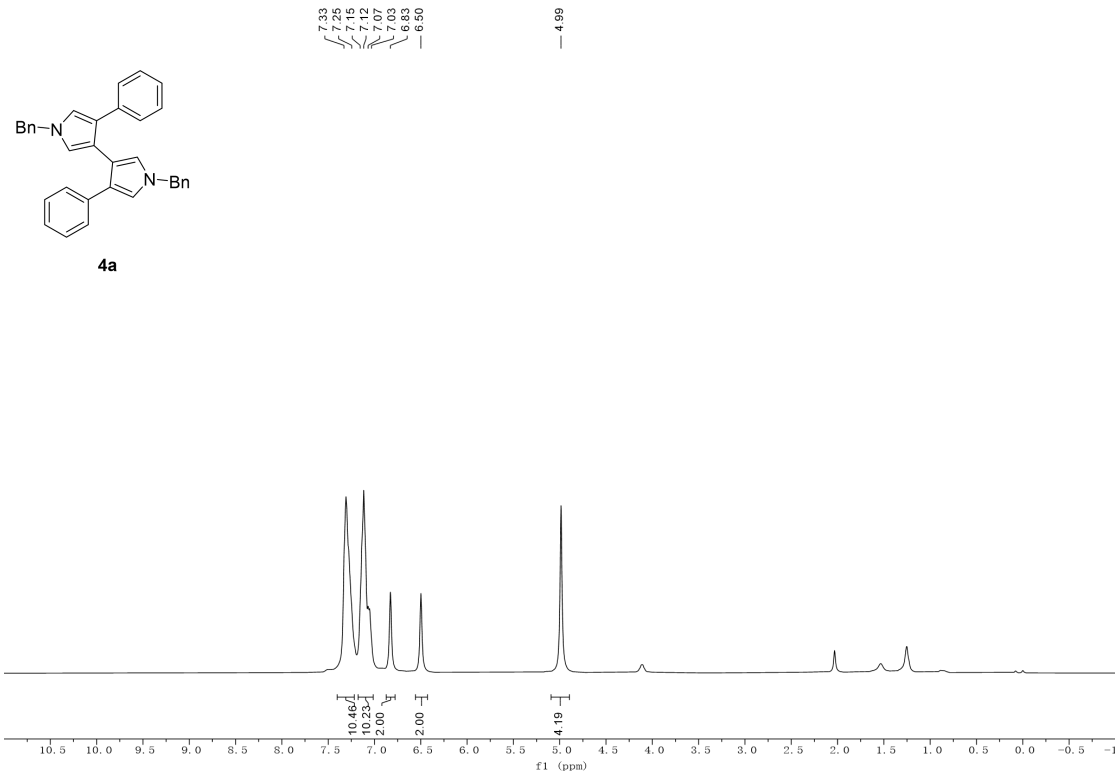
^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$) spectra of compound 3ai:



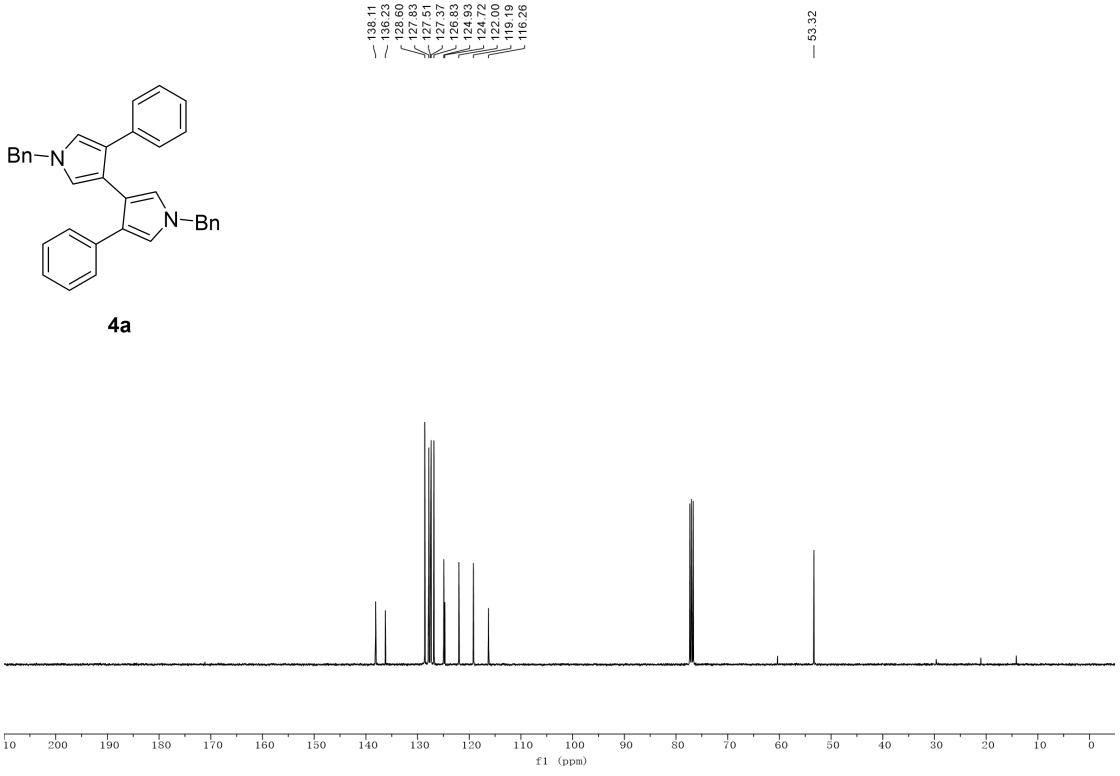
3ai

15.NMR spectra copies of the compounds 4.

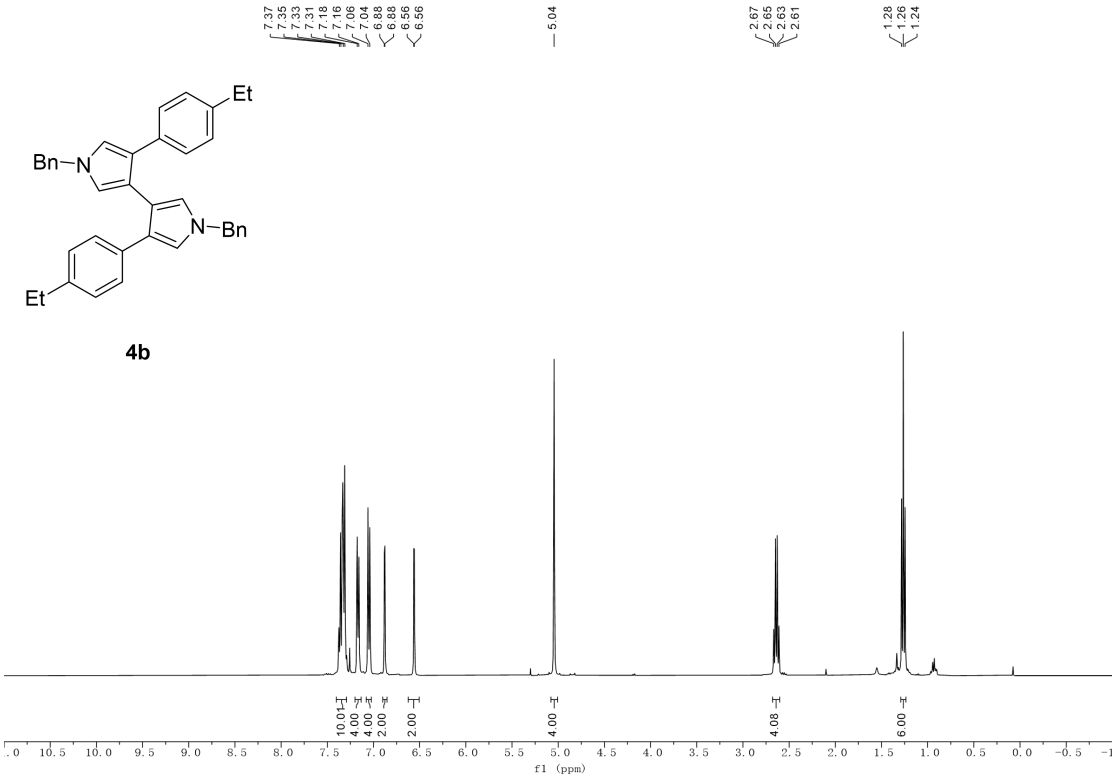
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4a:



^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4a:

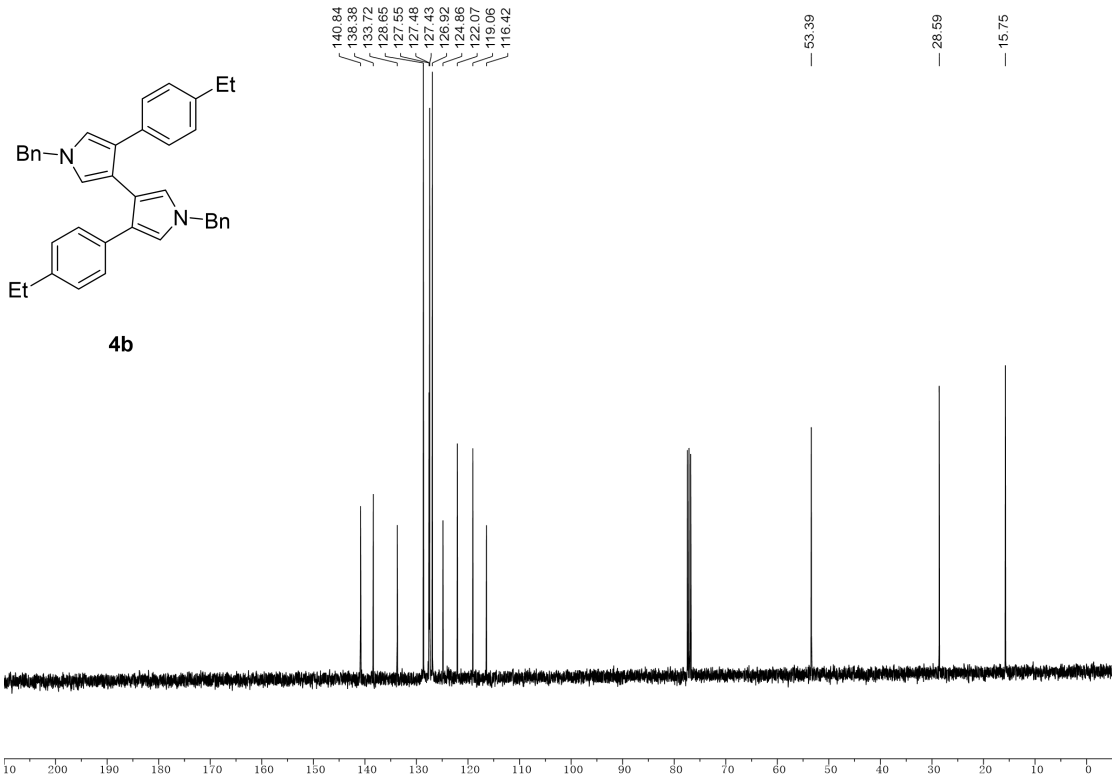


^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4b:



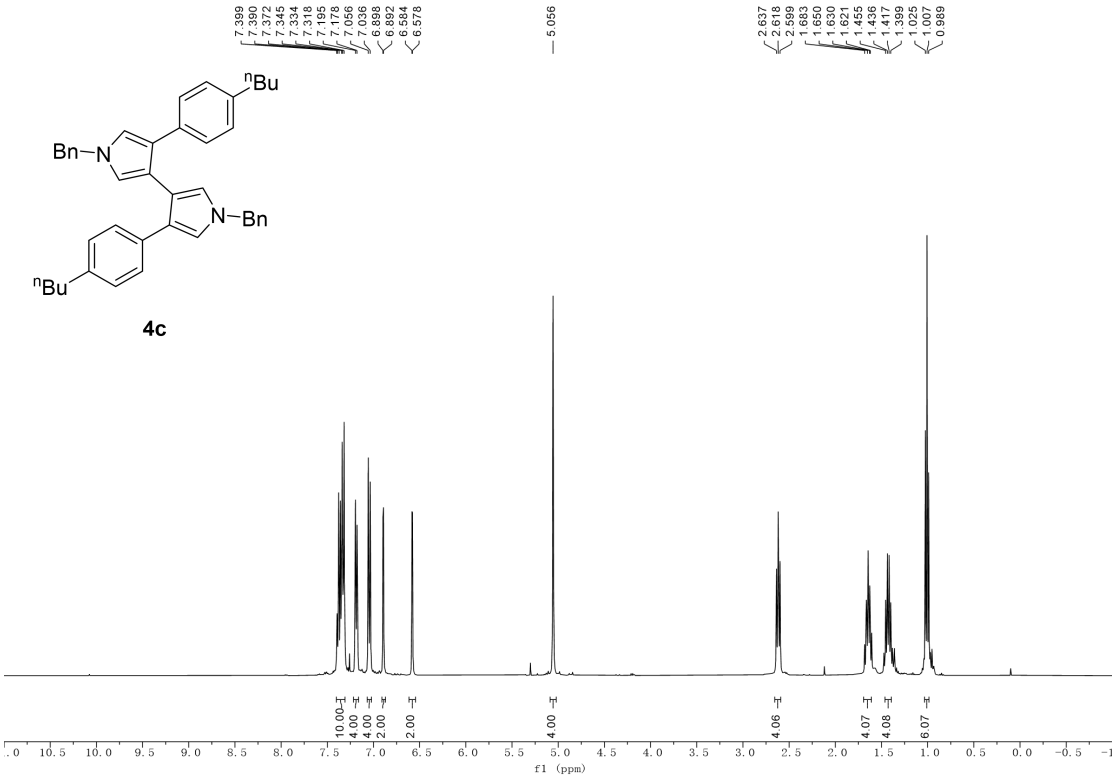
4b

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4b:



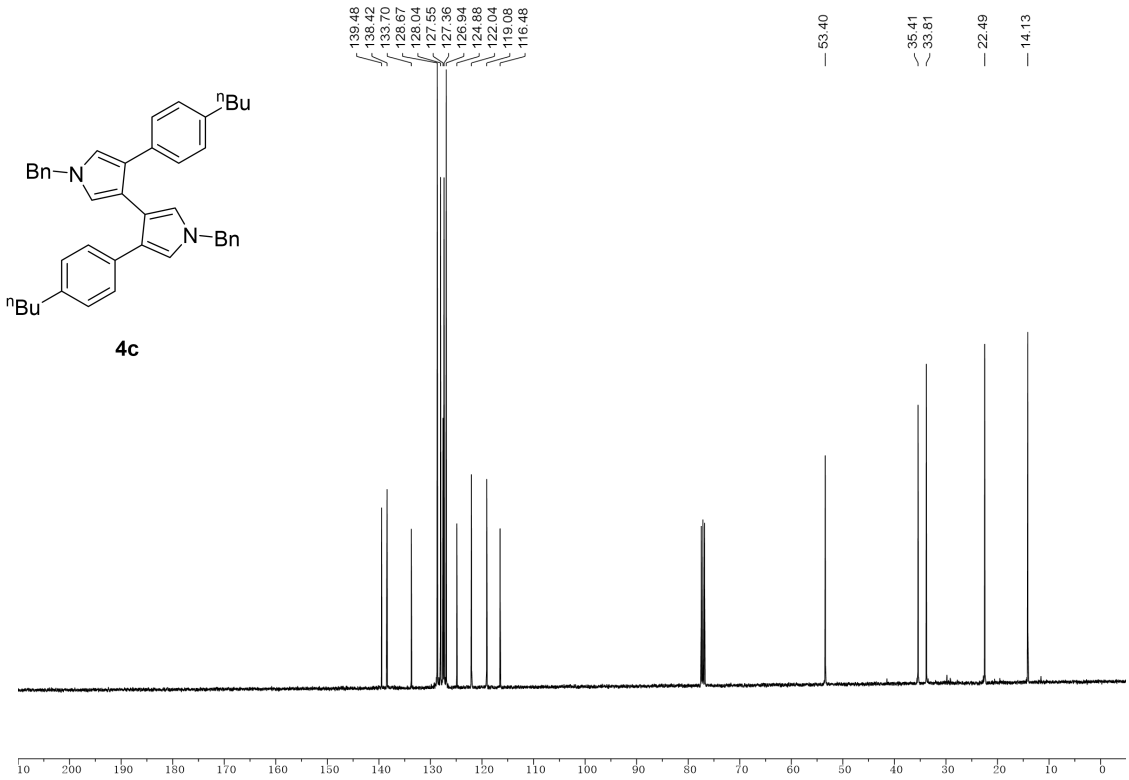
4b

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4c:



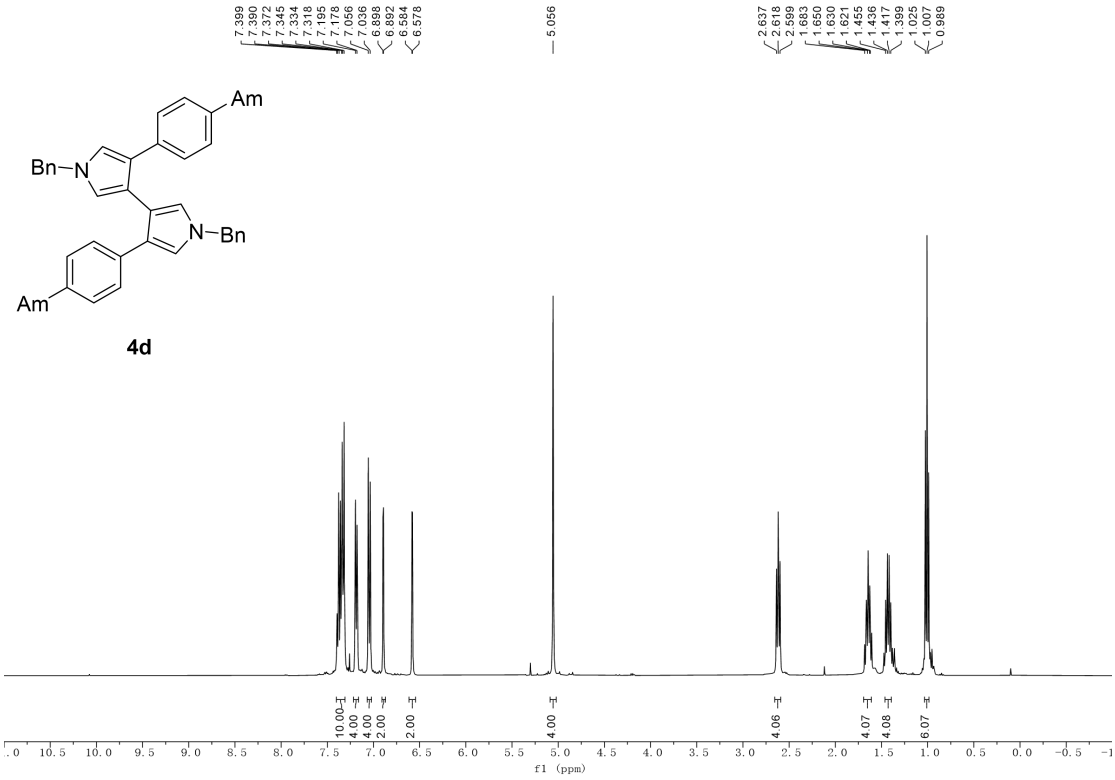
4c

^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4c:

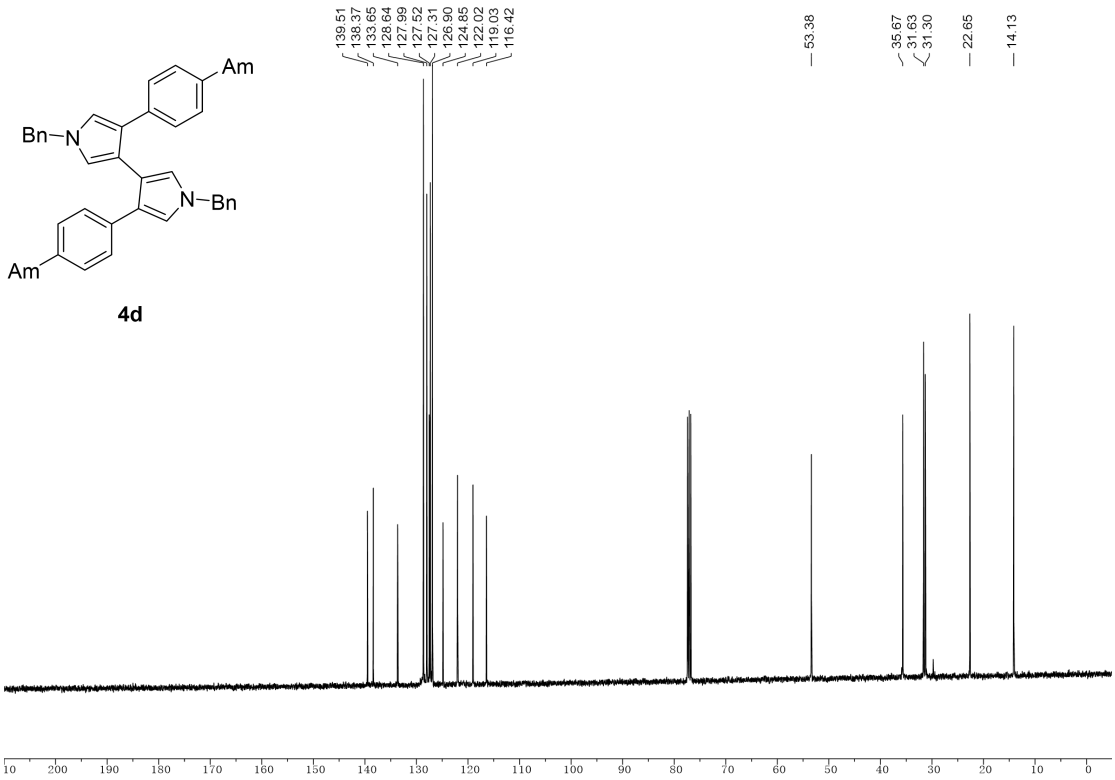


4c

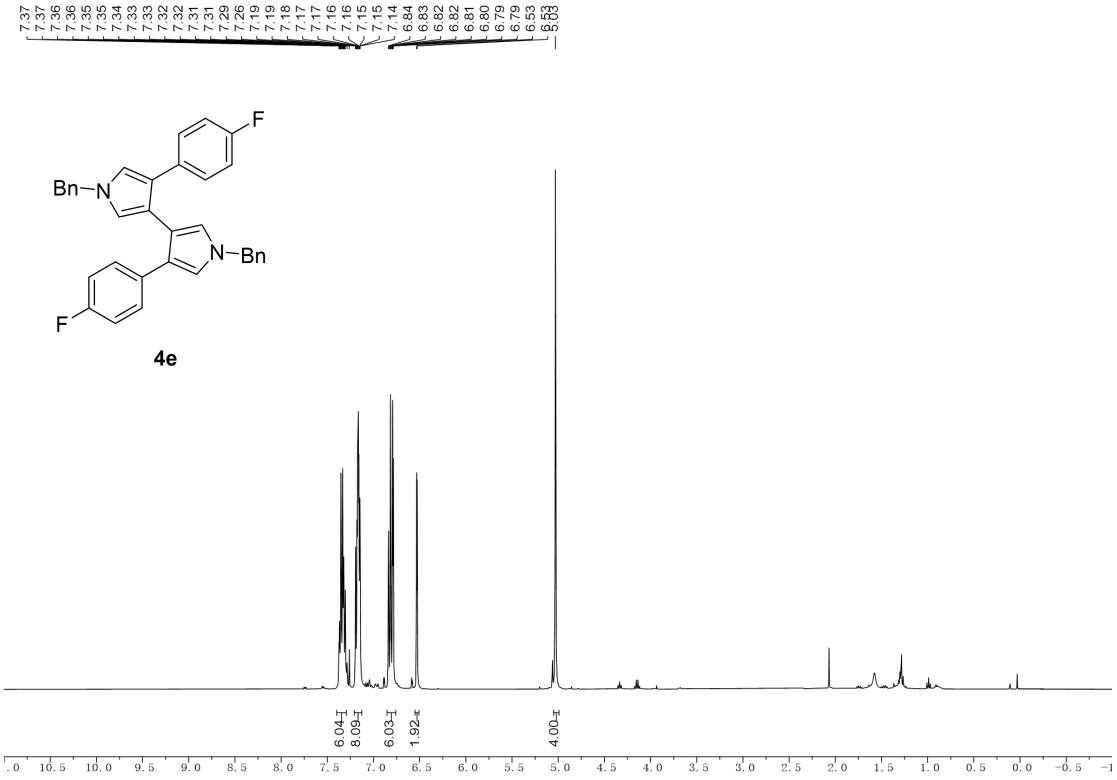
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4d:



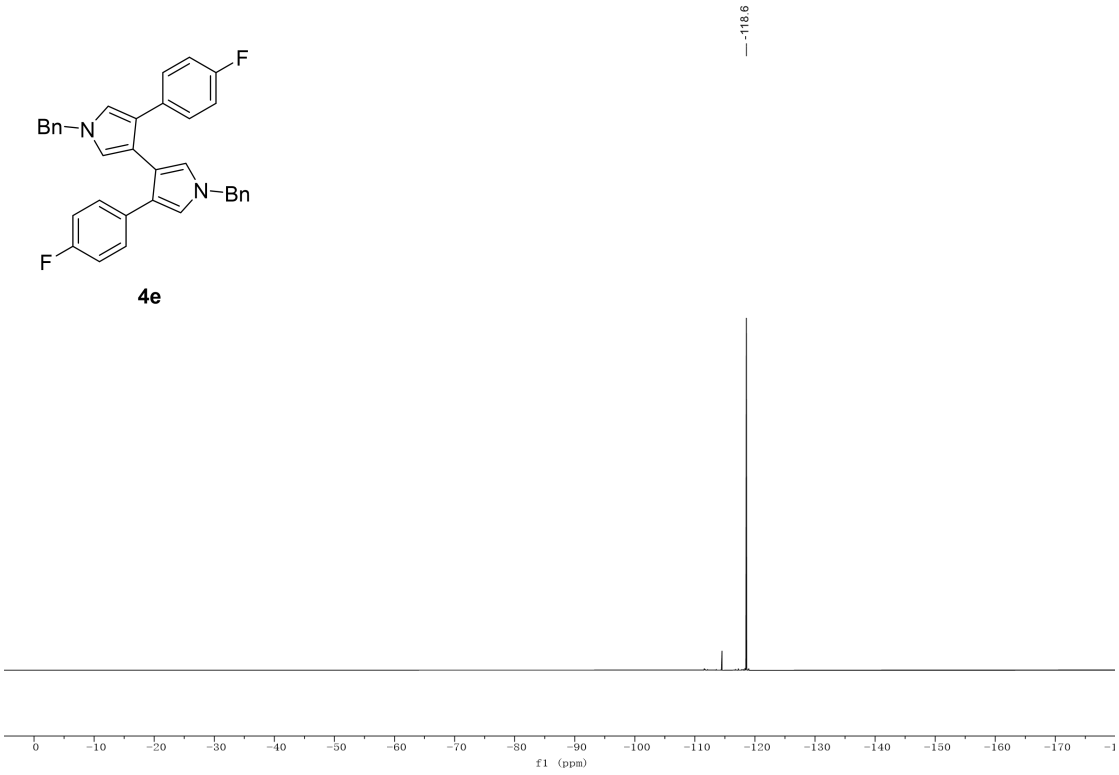
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4d:



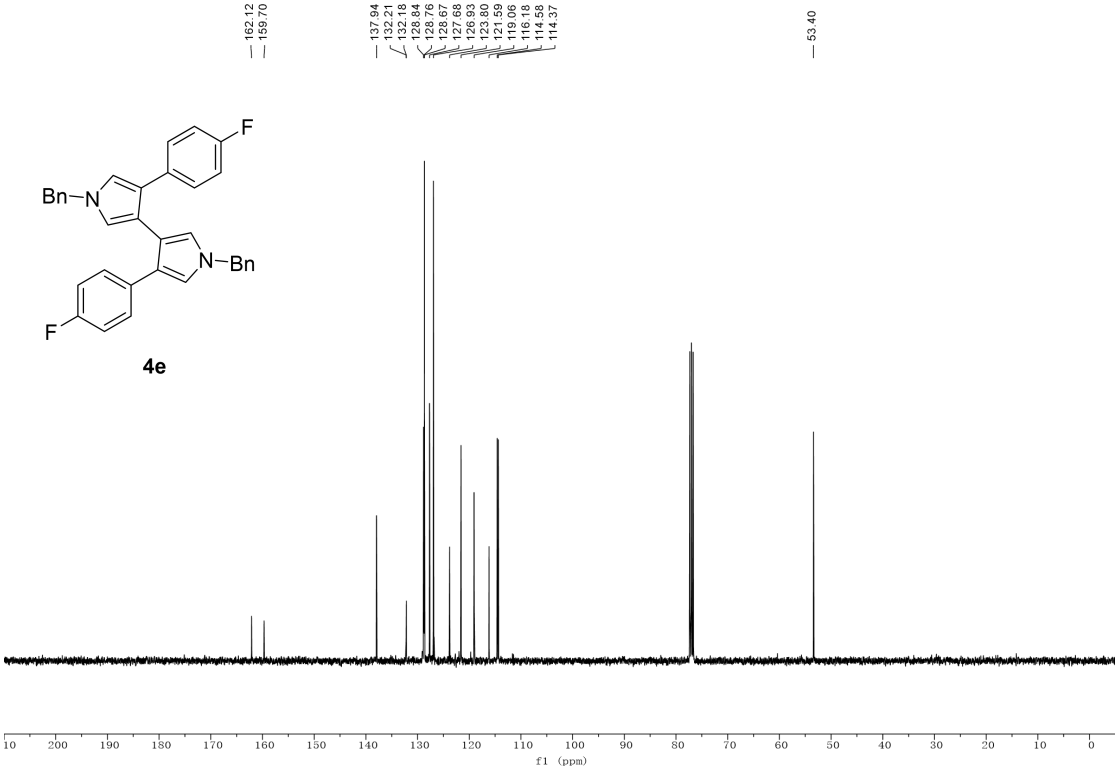
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4e:



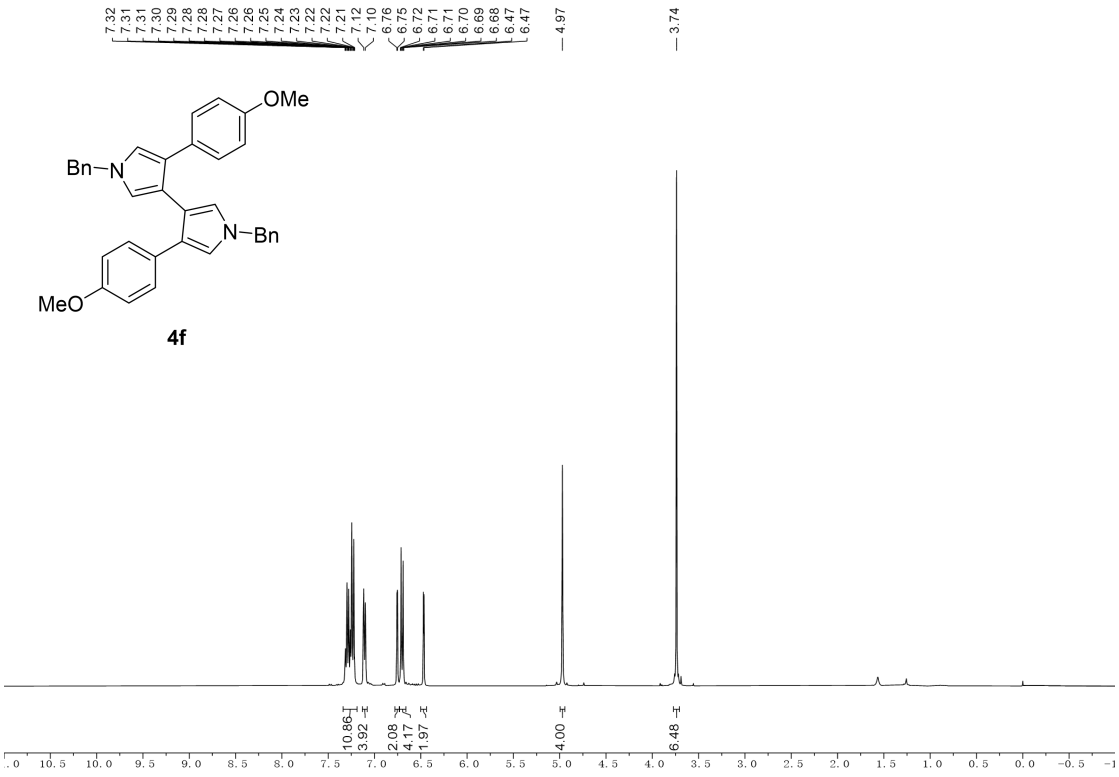
^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) spectra of compound 4e:



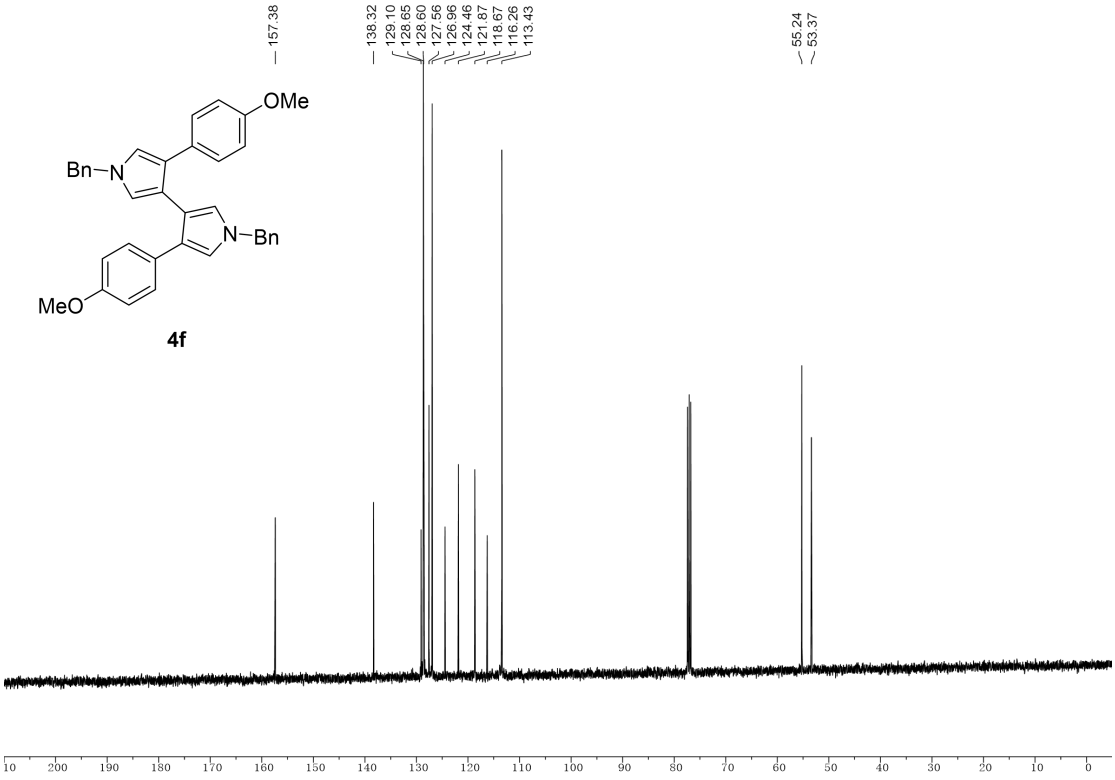
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4e:



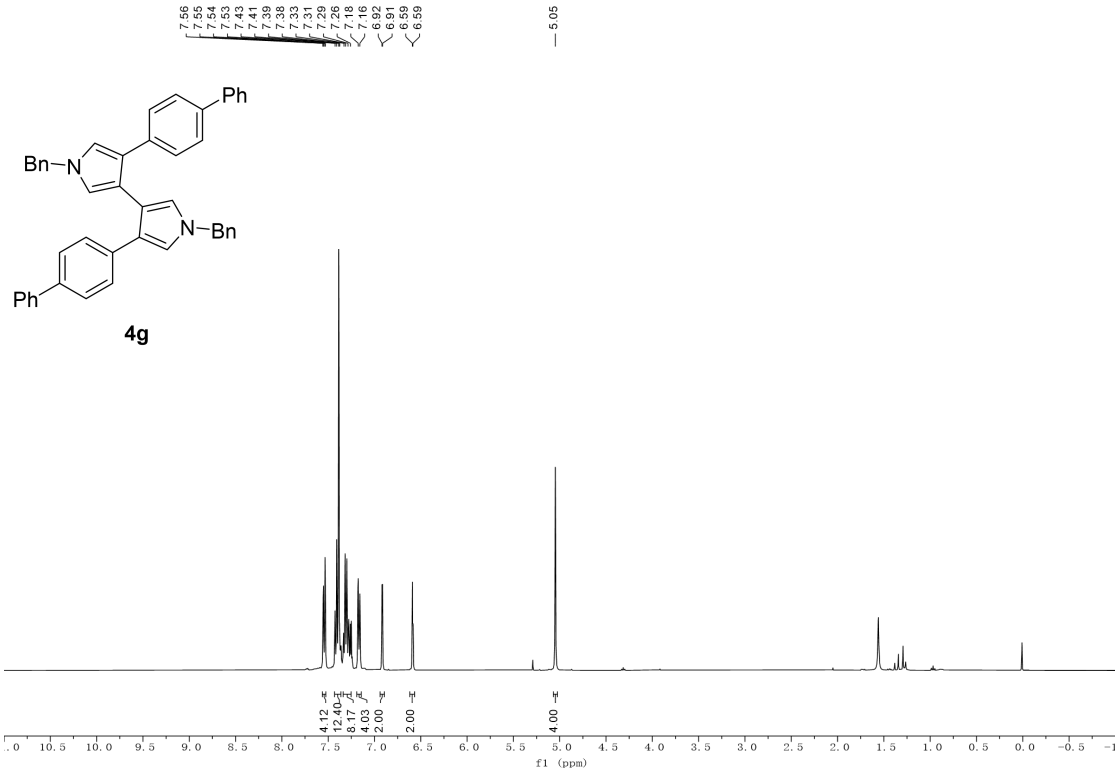
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4f:



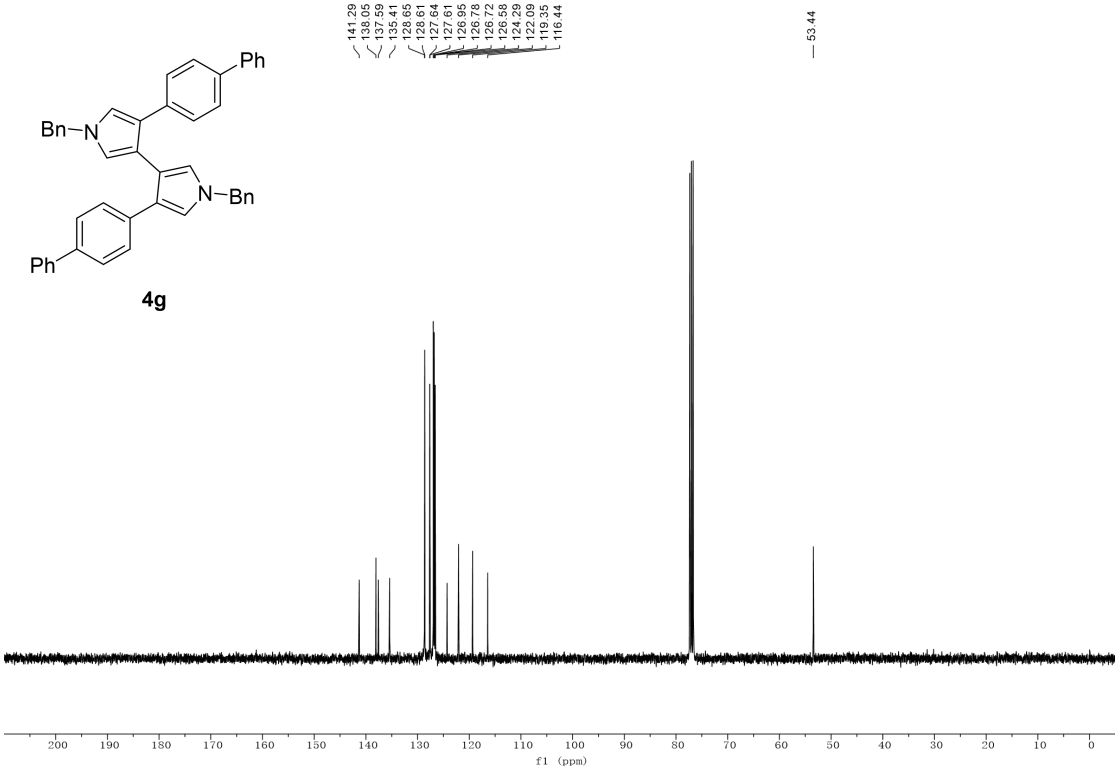
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4f:



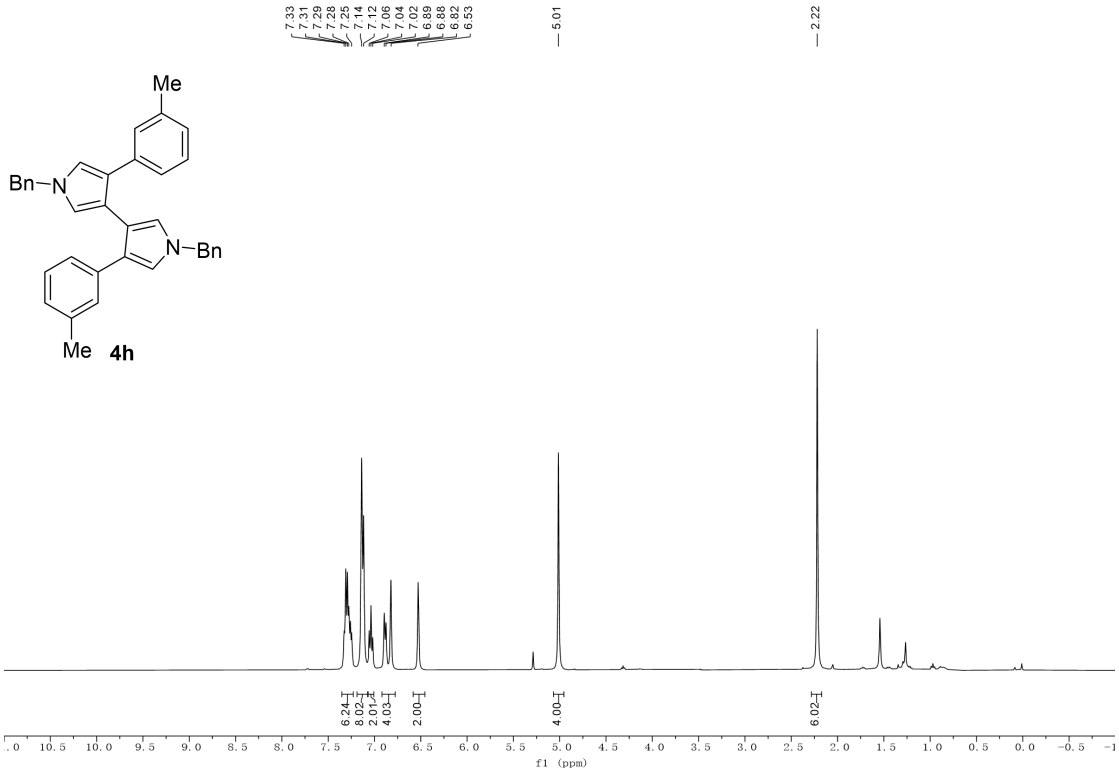
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4g:



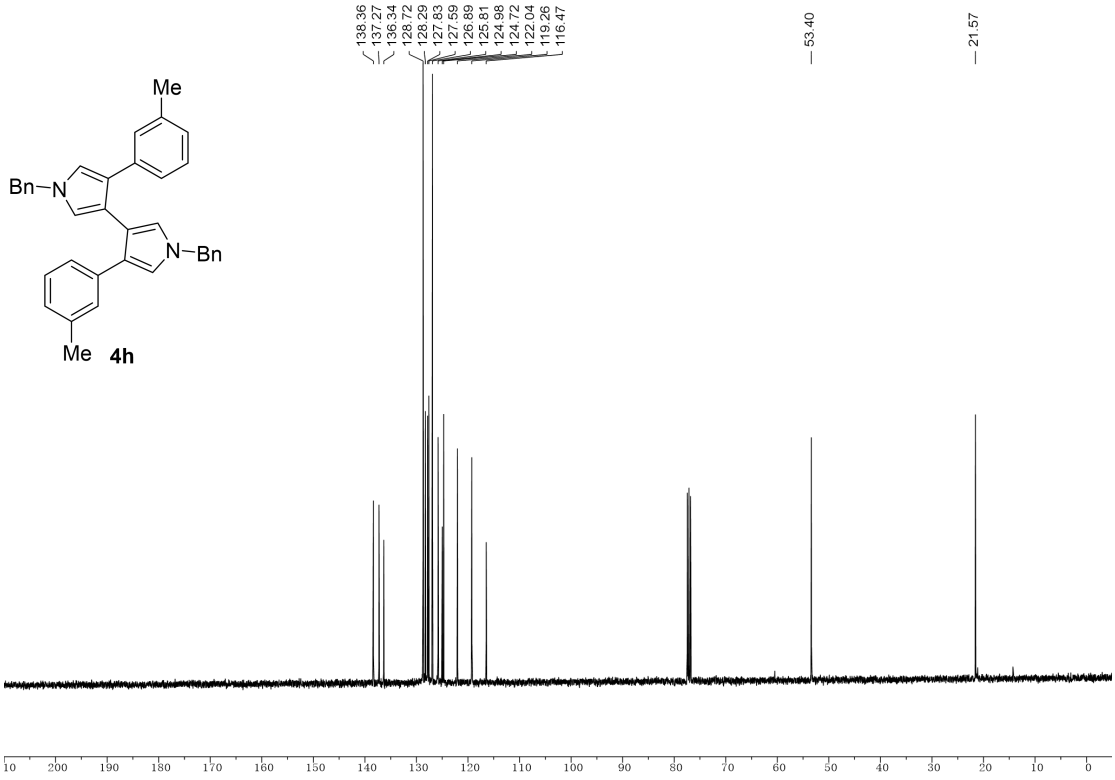
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4g:



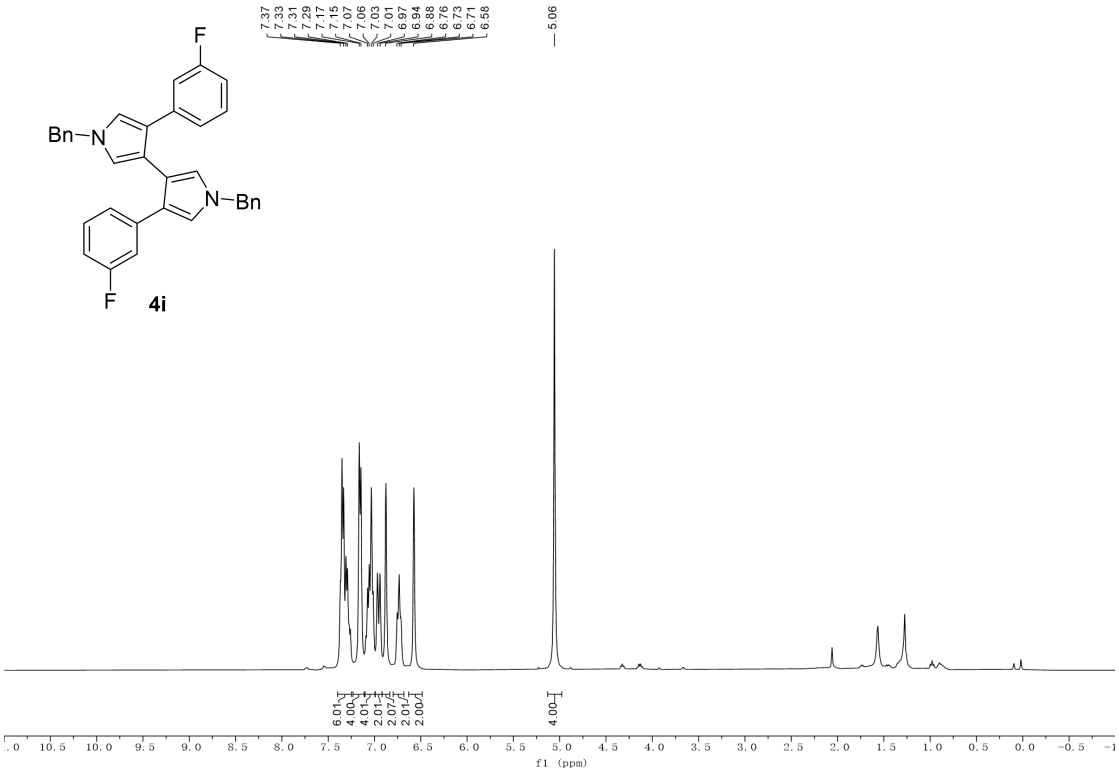
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4h:



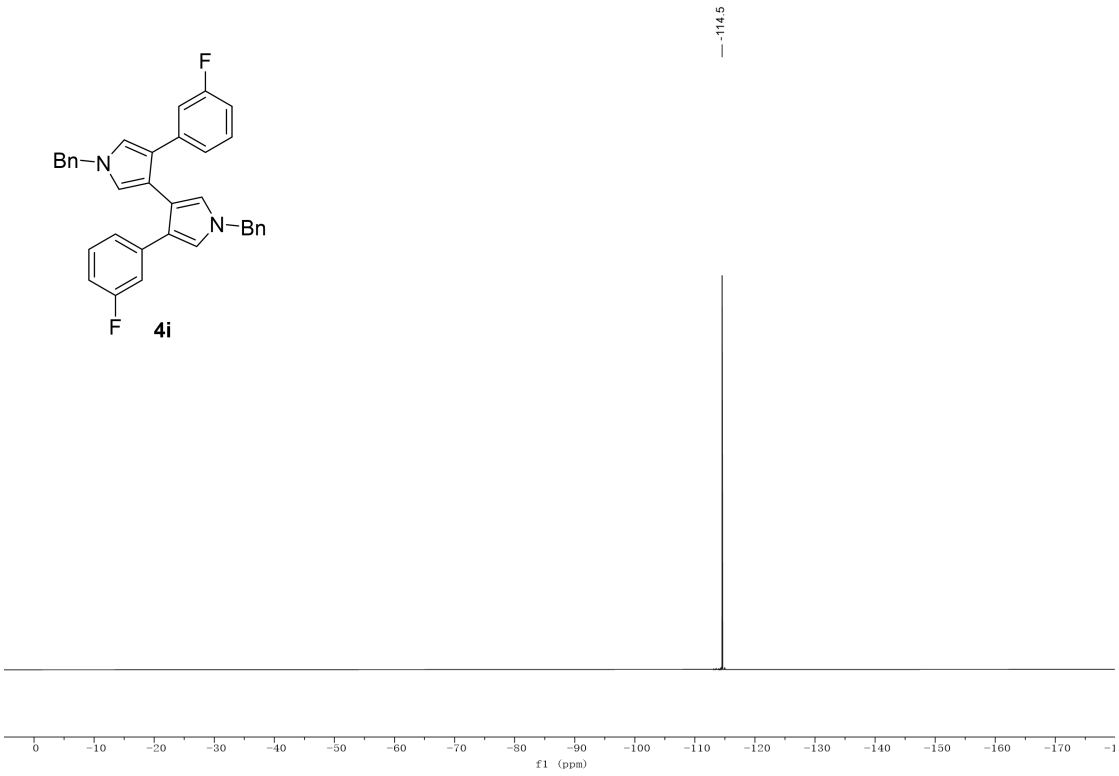
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4h:



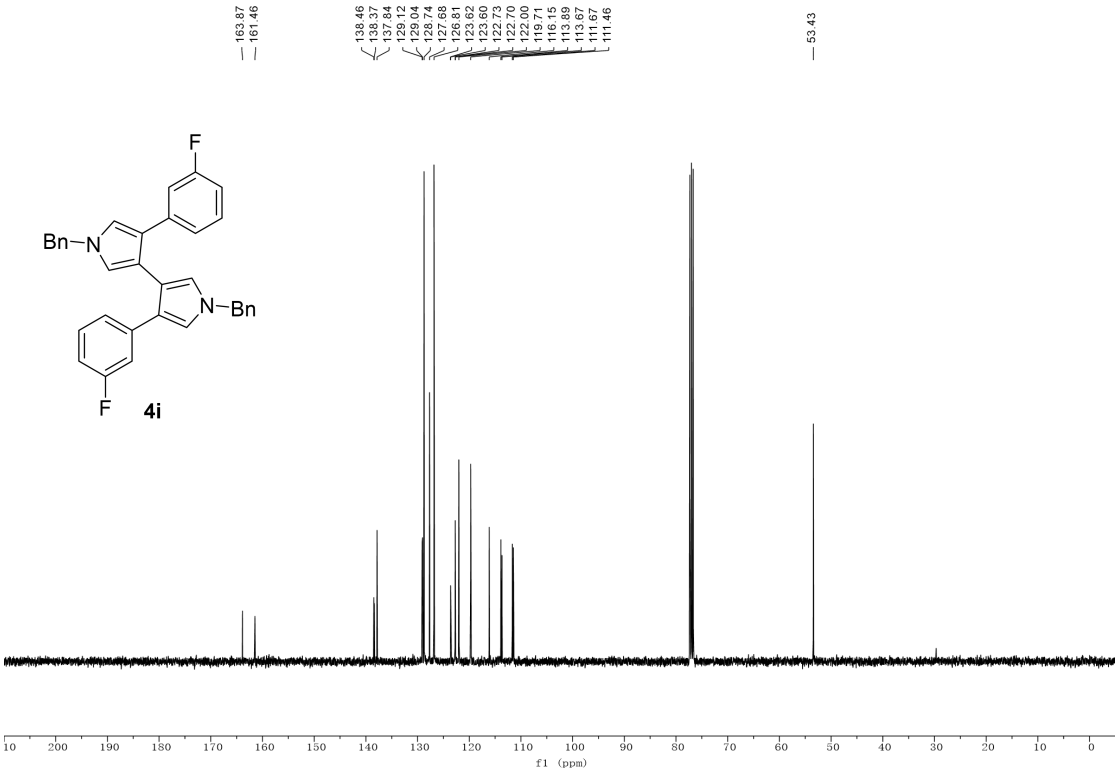
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4i:



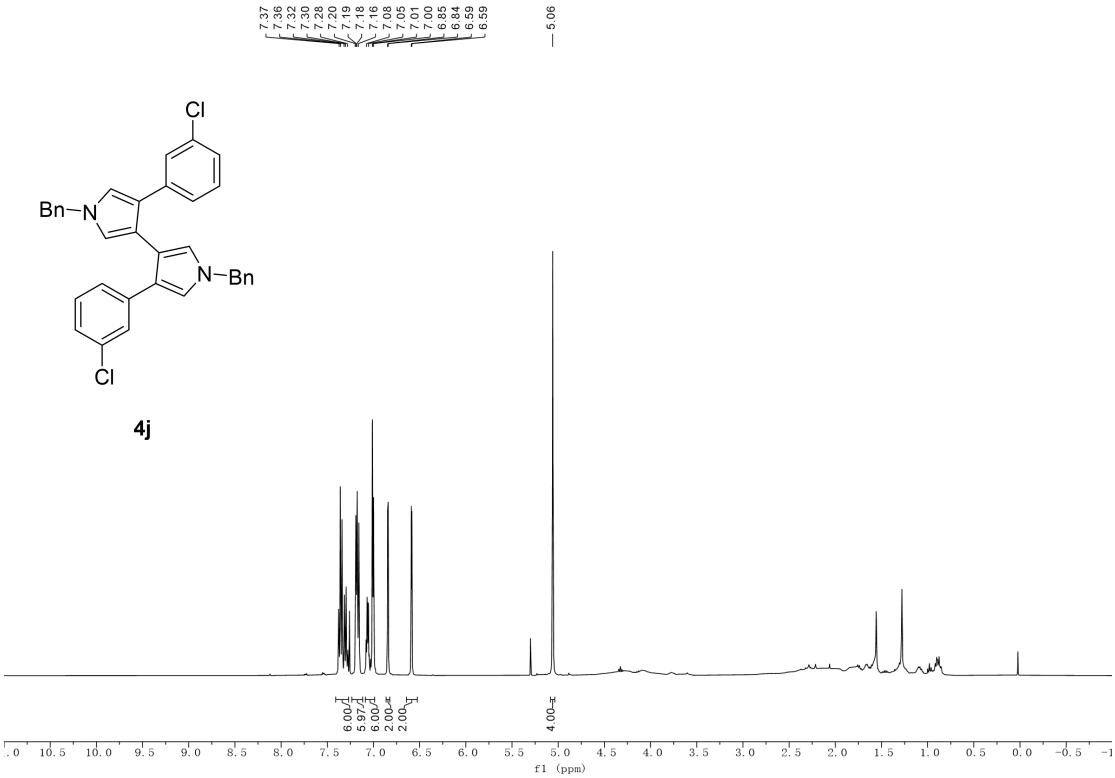
$^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$) spectrum of compound 4i



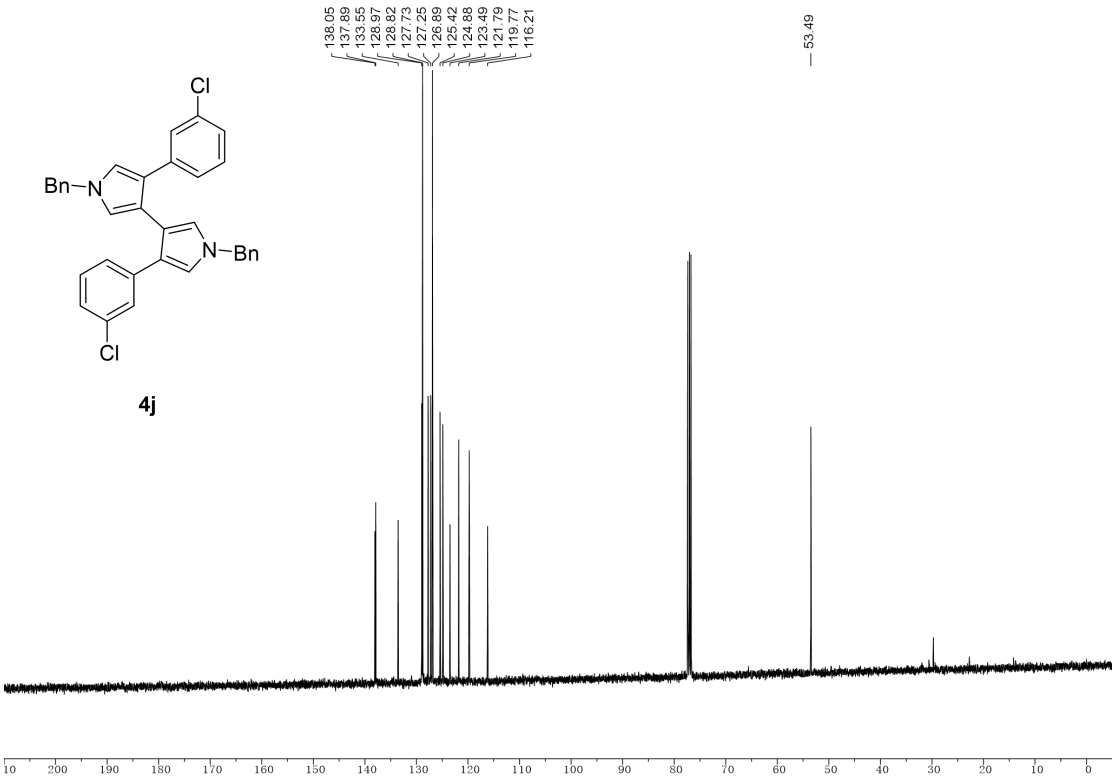
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4i:



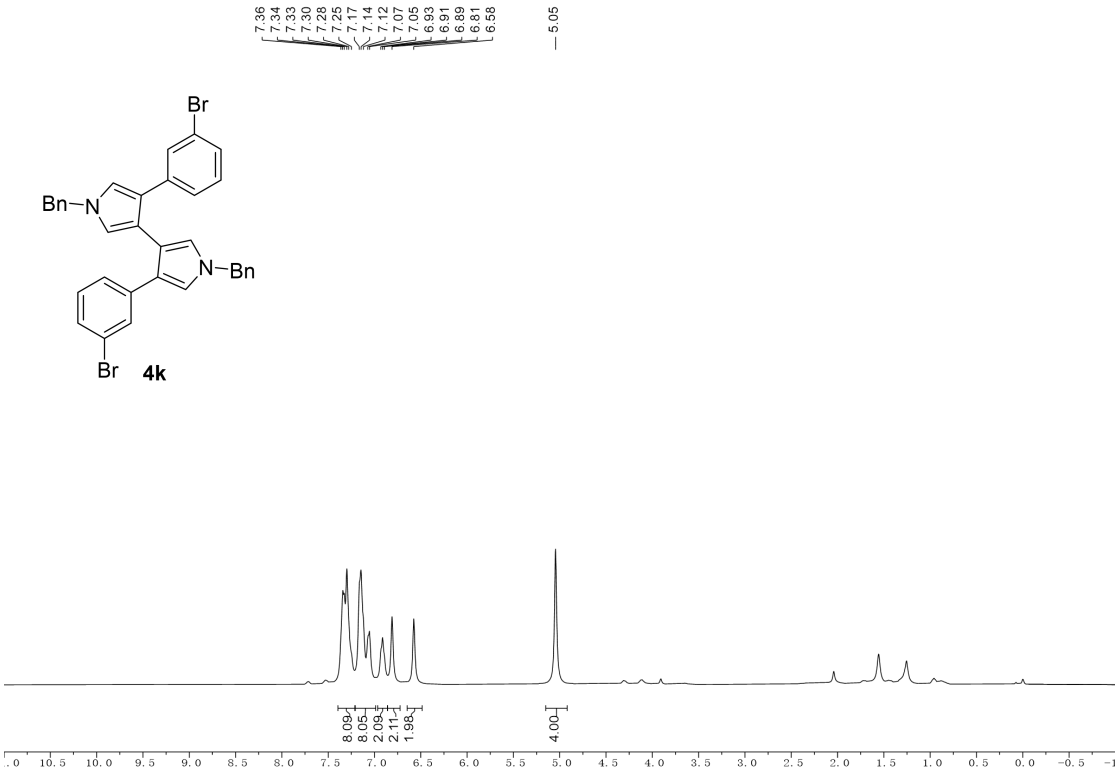
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4j:



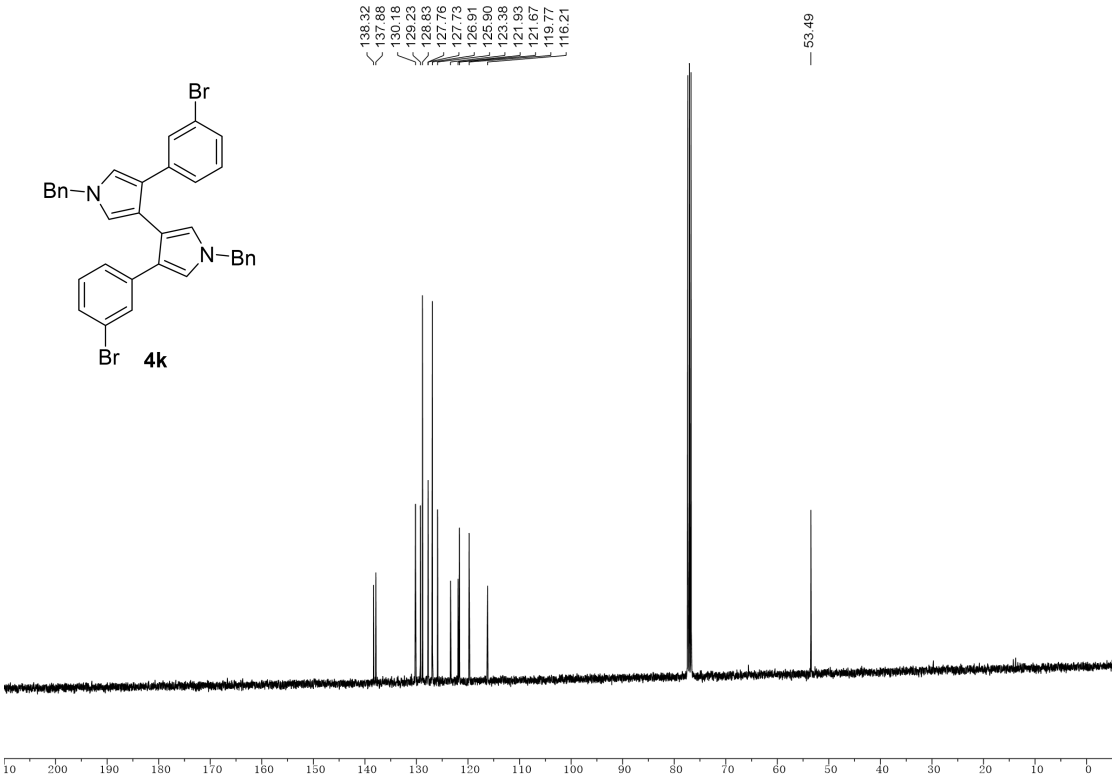
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4j:



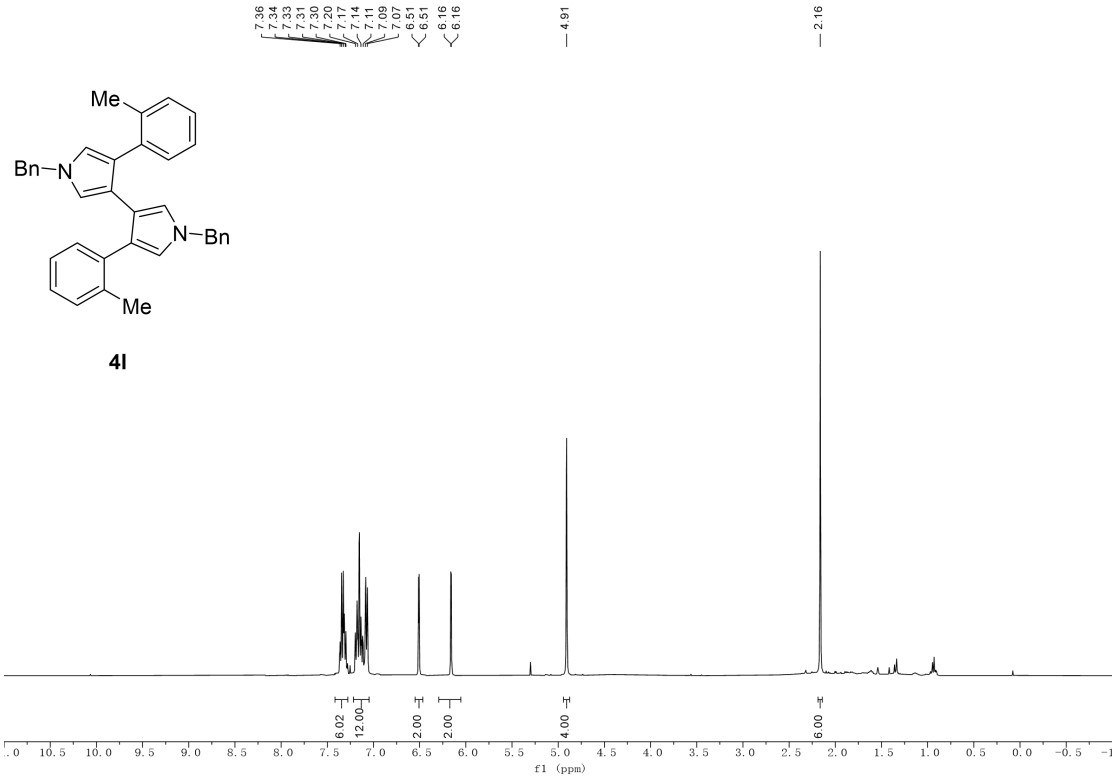
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4k:



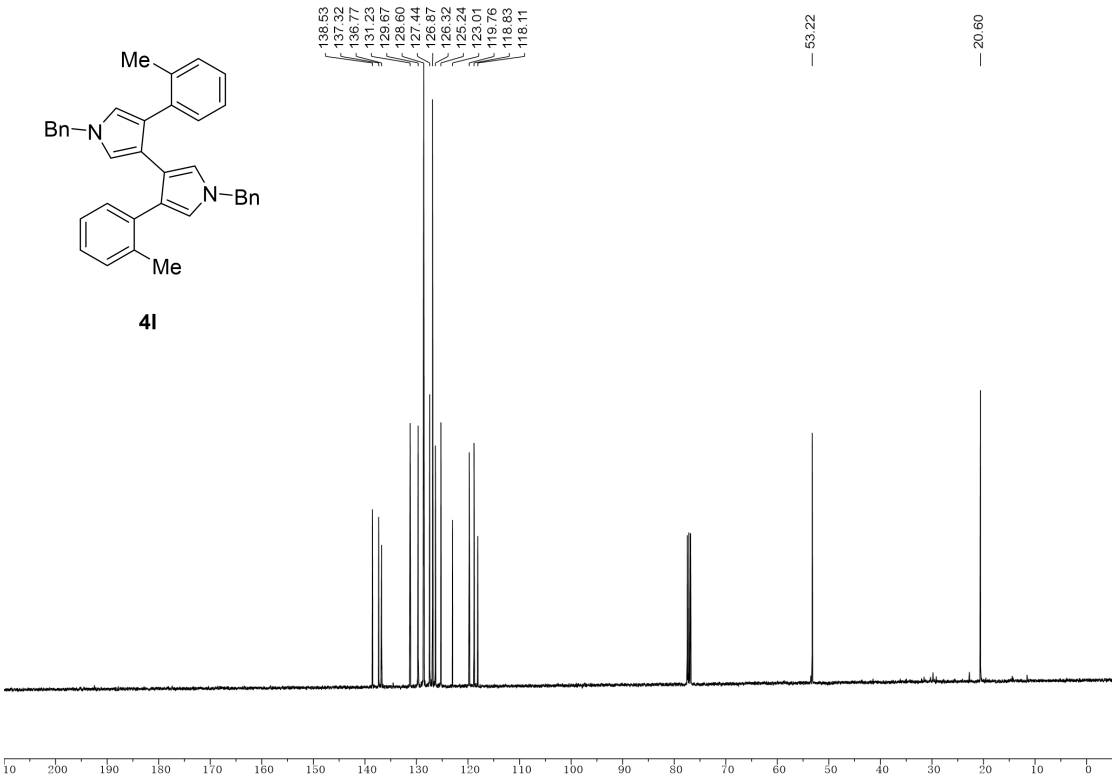
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4k:



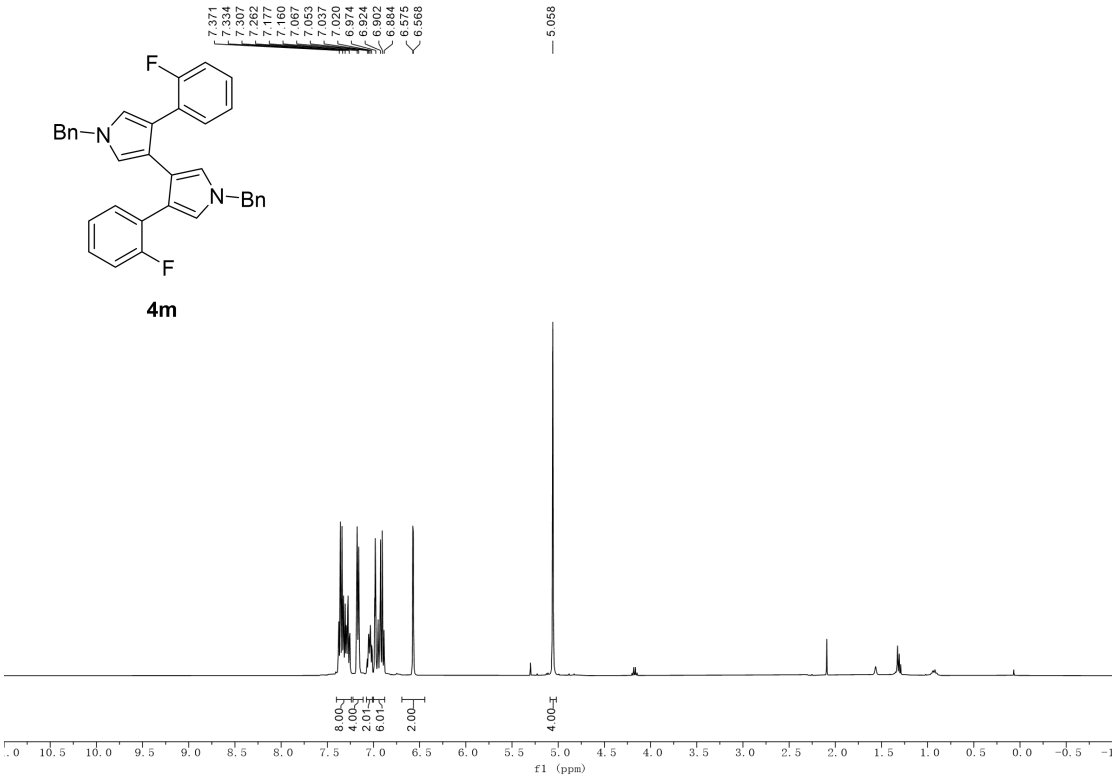
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4l:



^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) spectra of compound 4l:

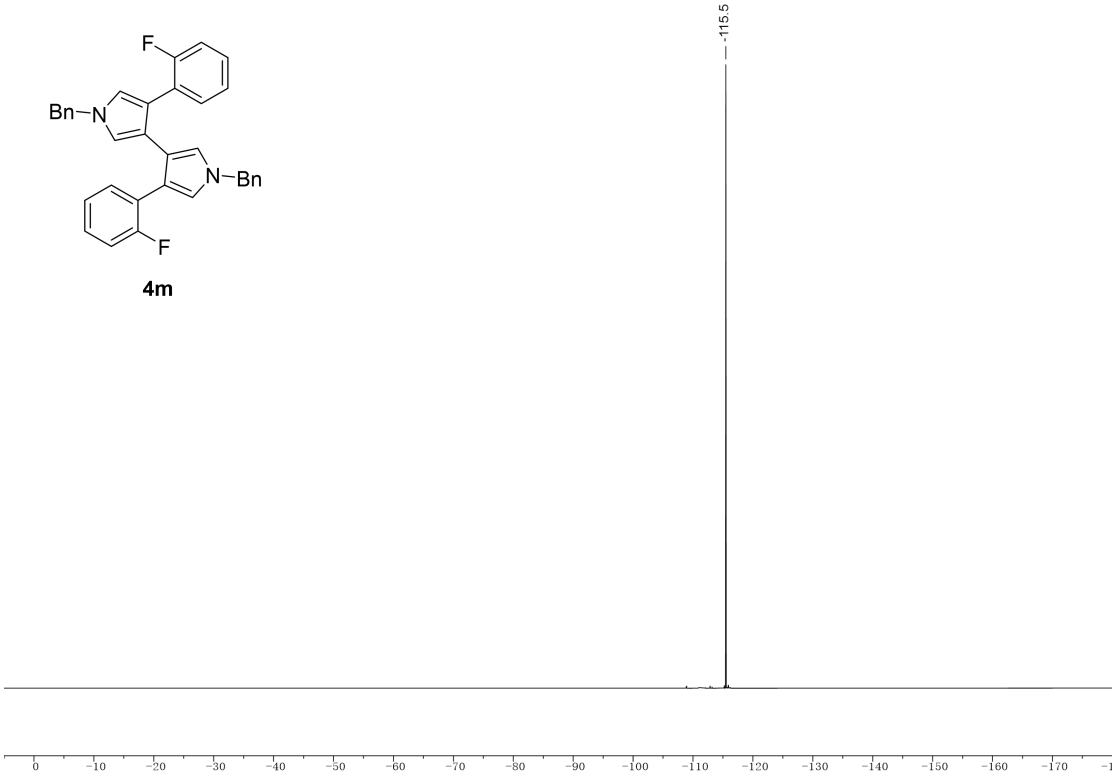


^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4m:



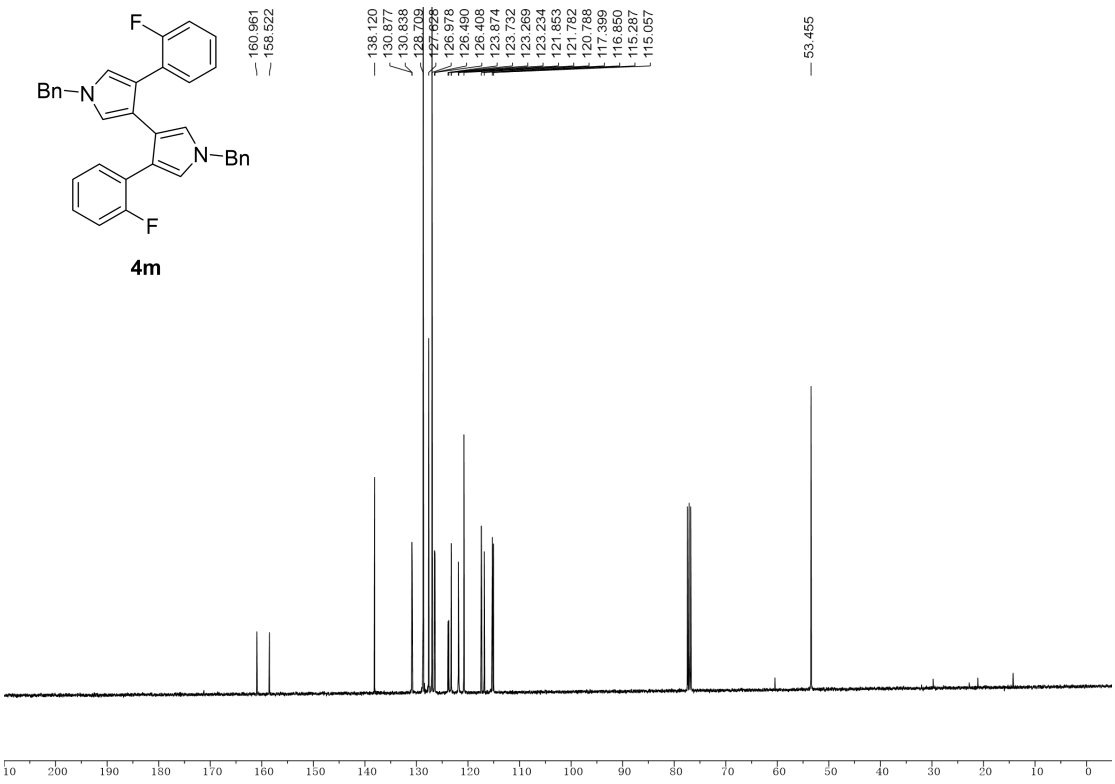
4m

^{19}F{^{1}H} NMR (376 MHz, $CDCl_3$) spectrum of compound 4m



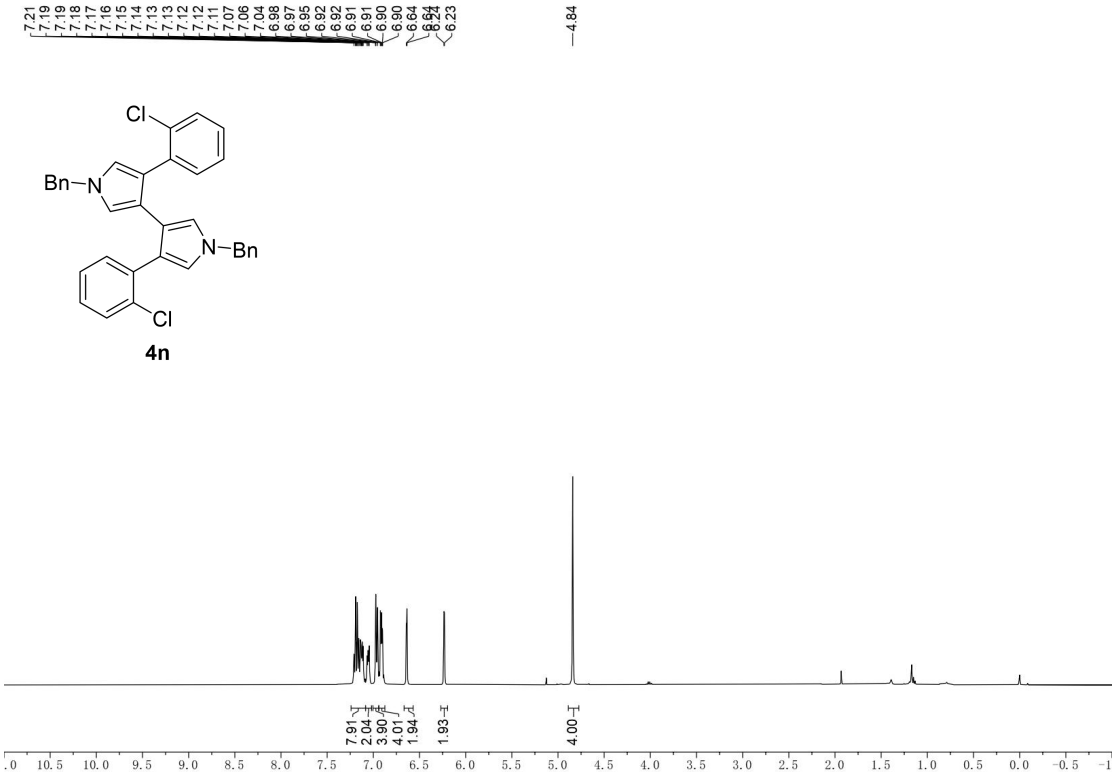
4m

$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4m:



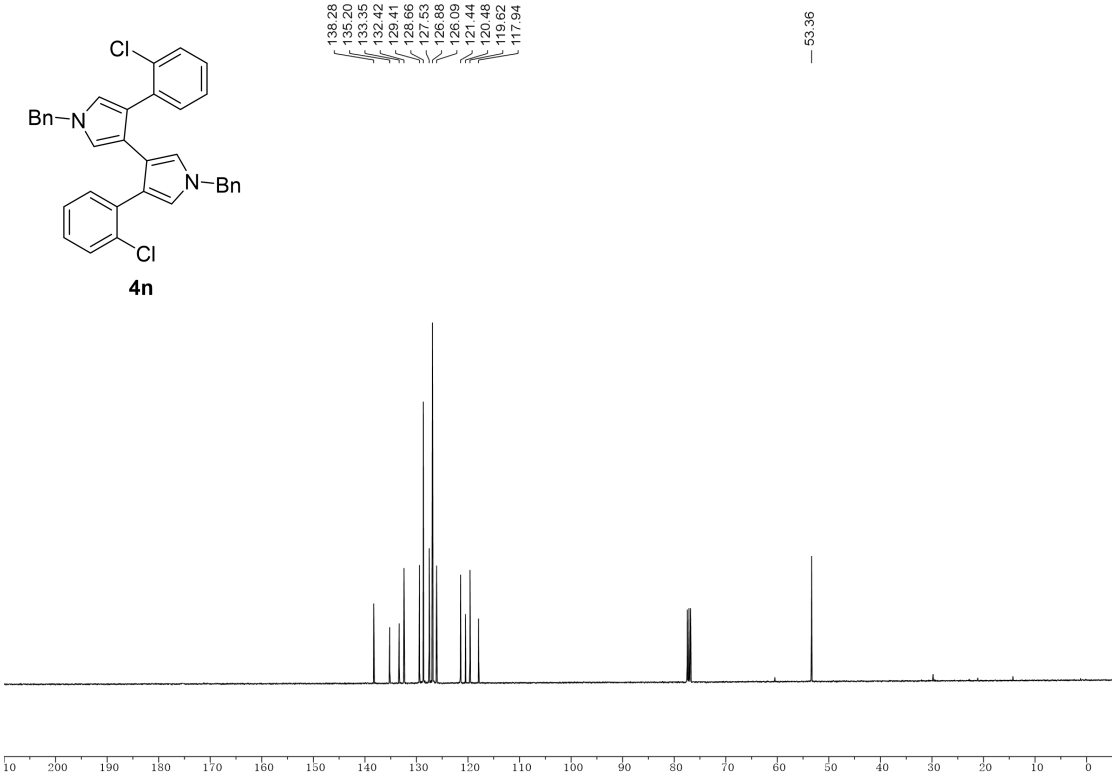
4m

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4n:

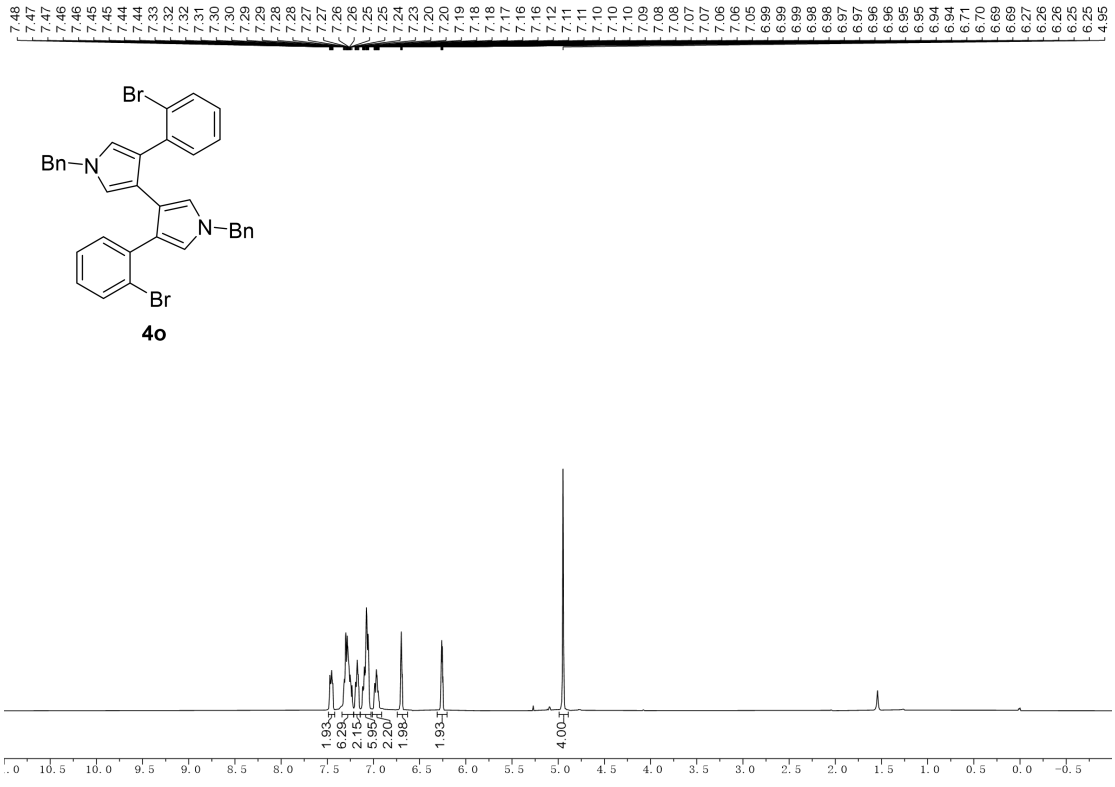


4n

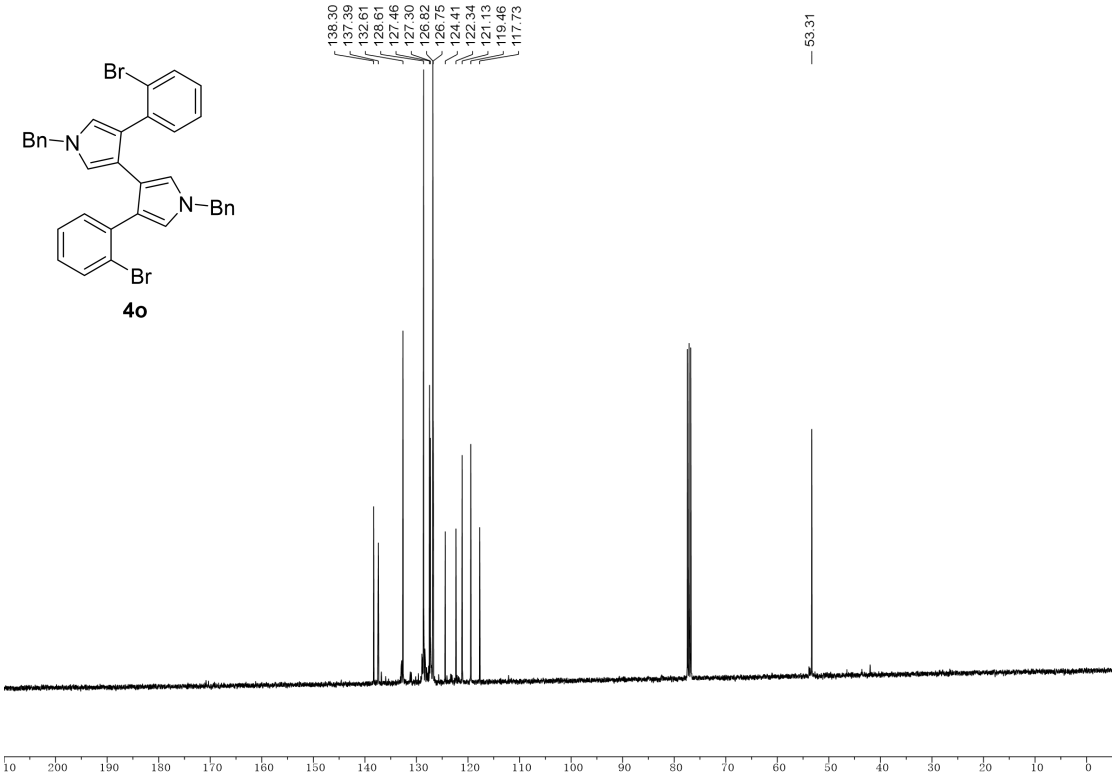
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4n:



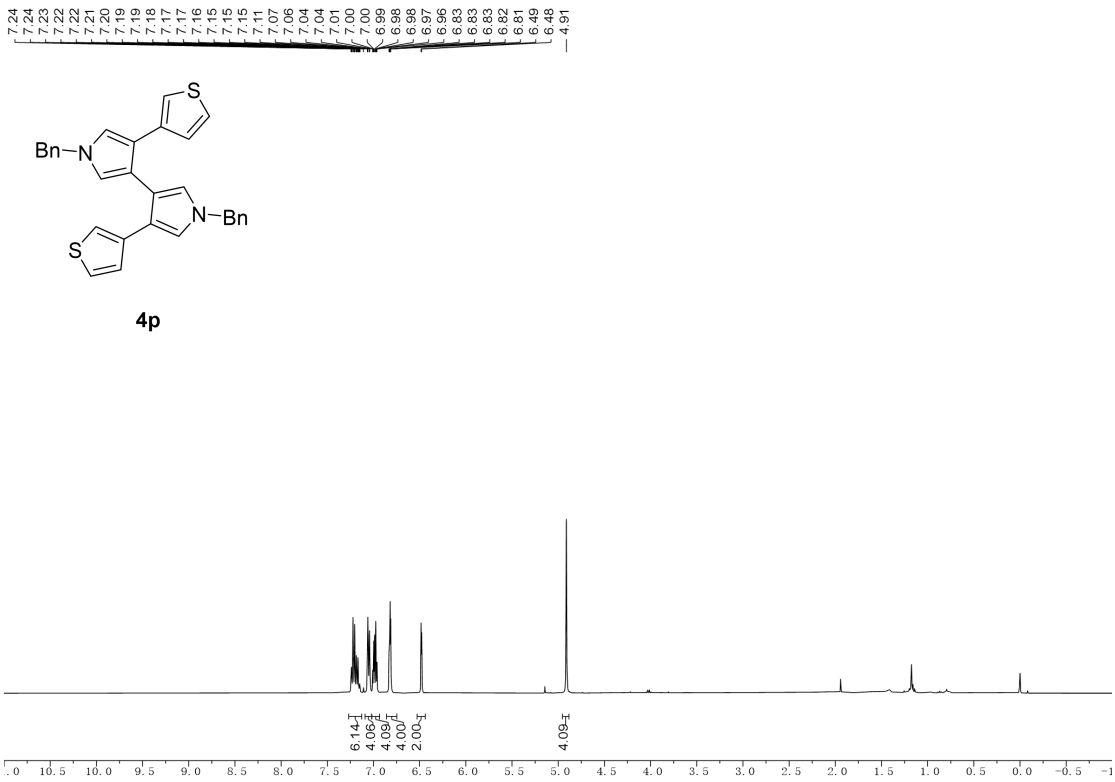
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4o:



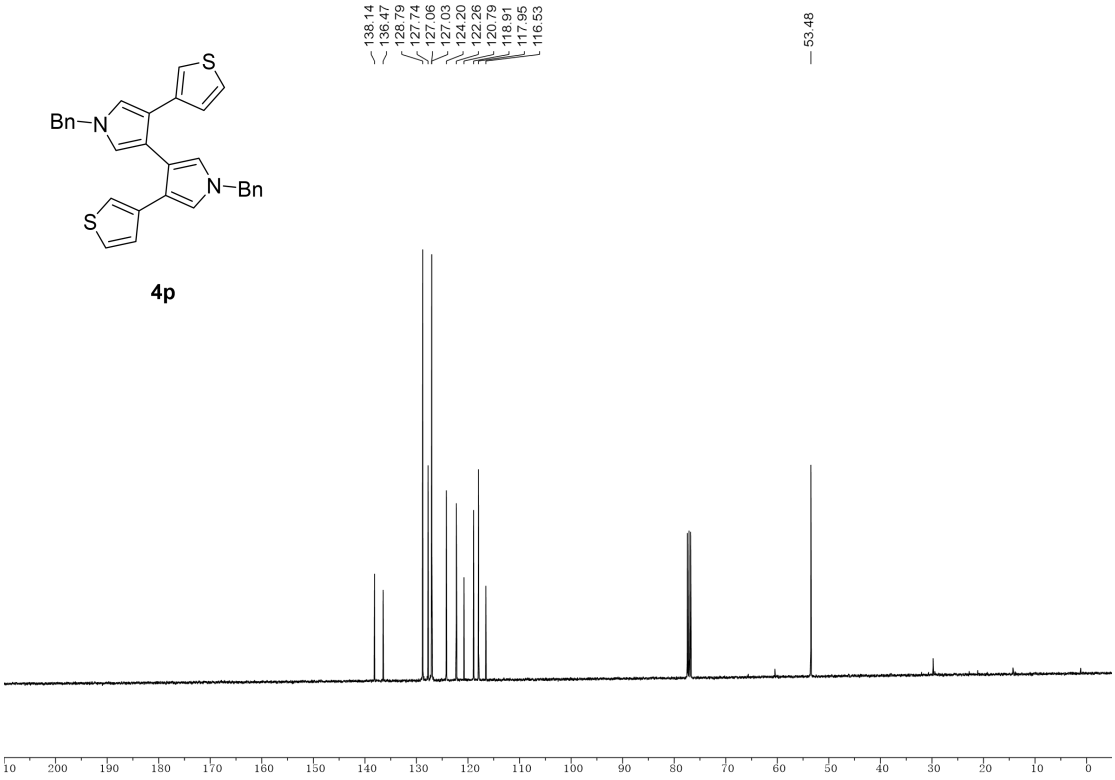
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4o:



^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4p:

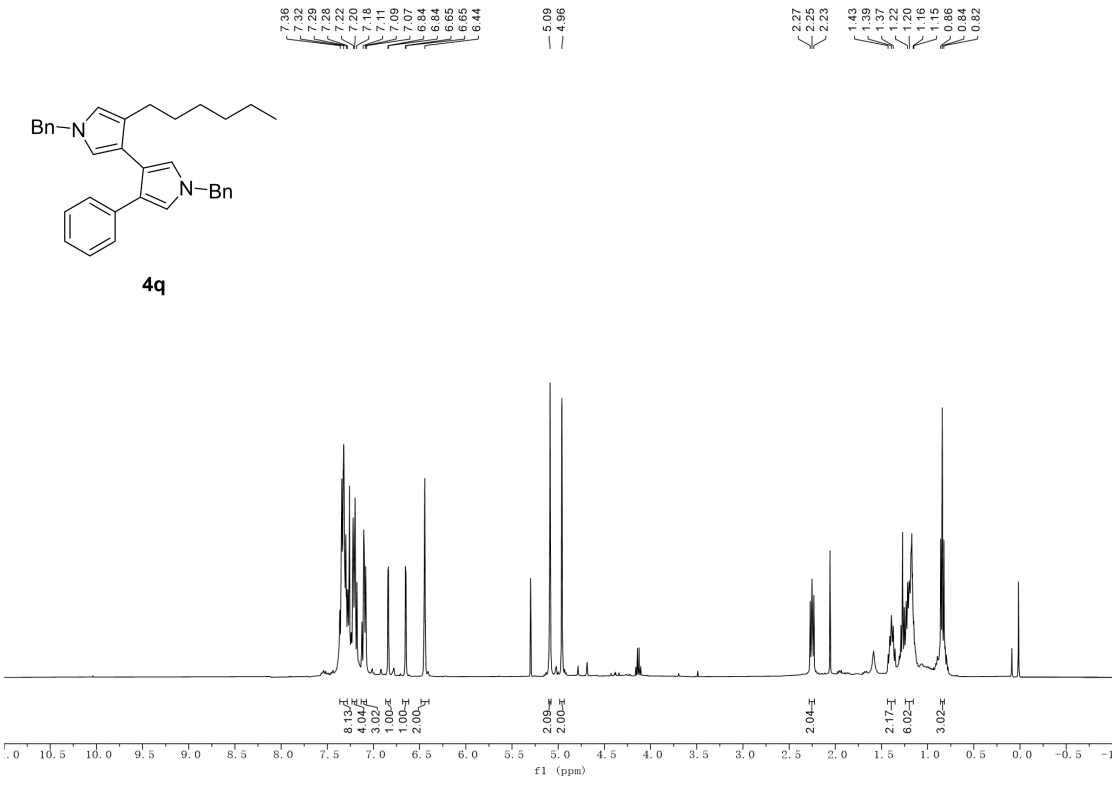


$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4p:



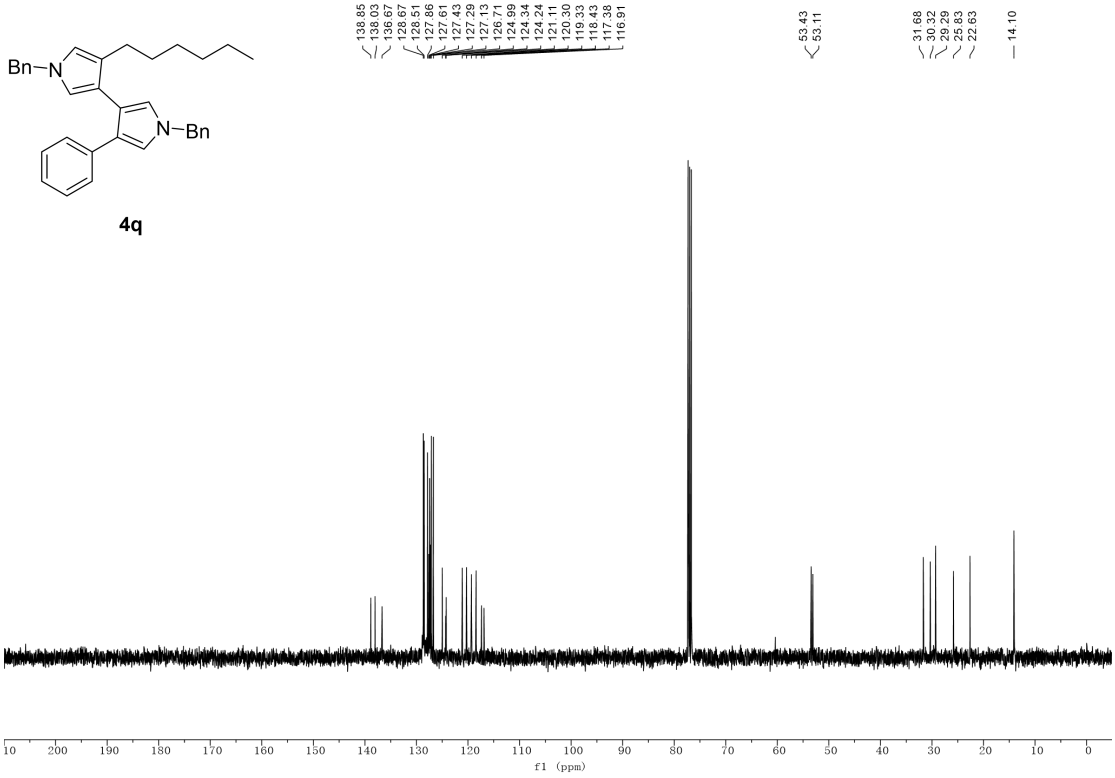
4p

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4q:

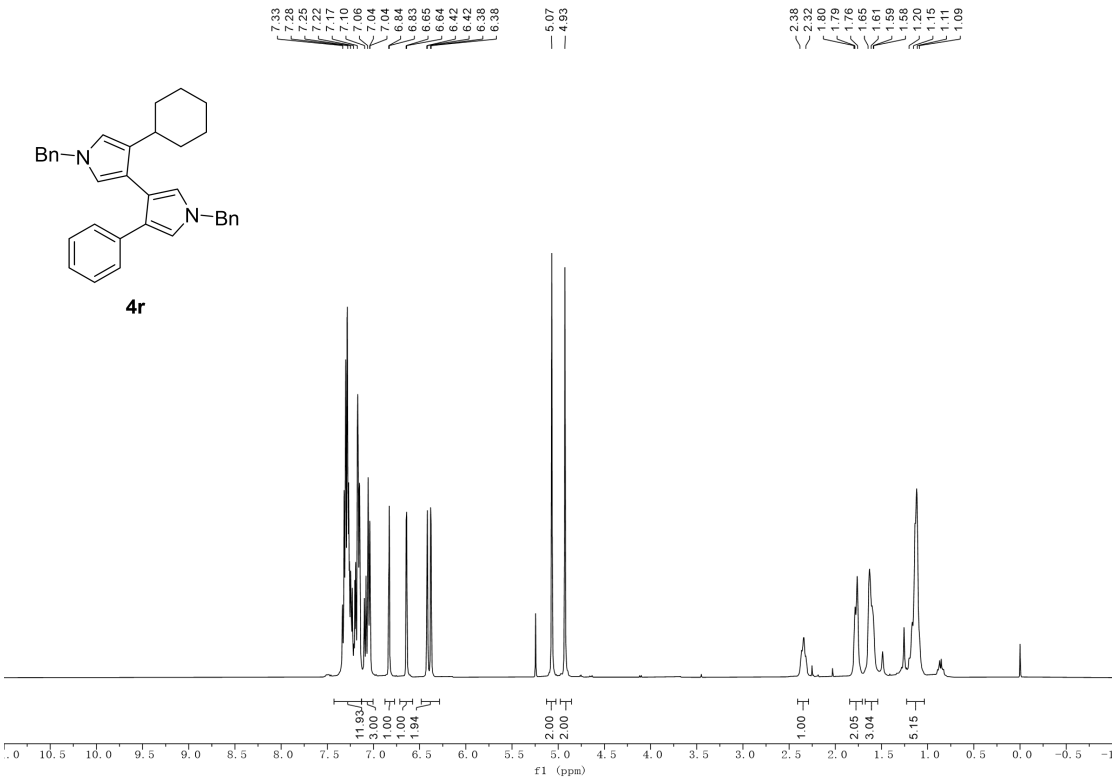


4q

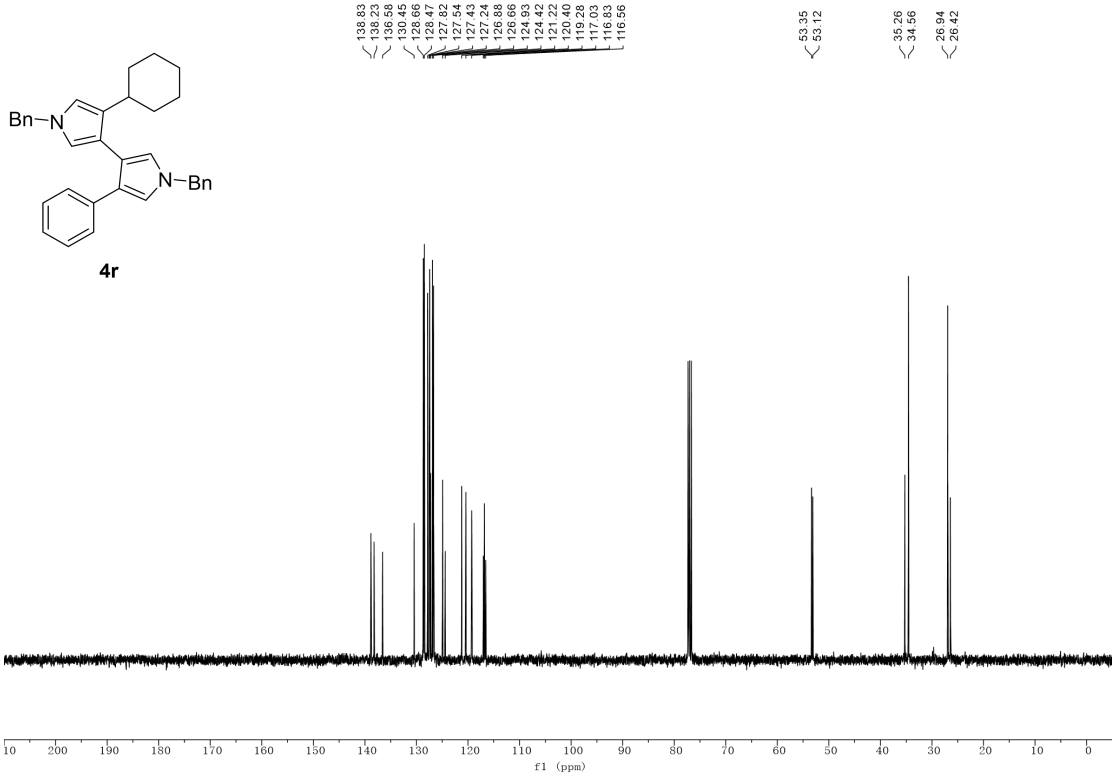
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4q:



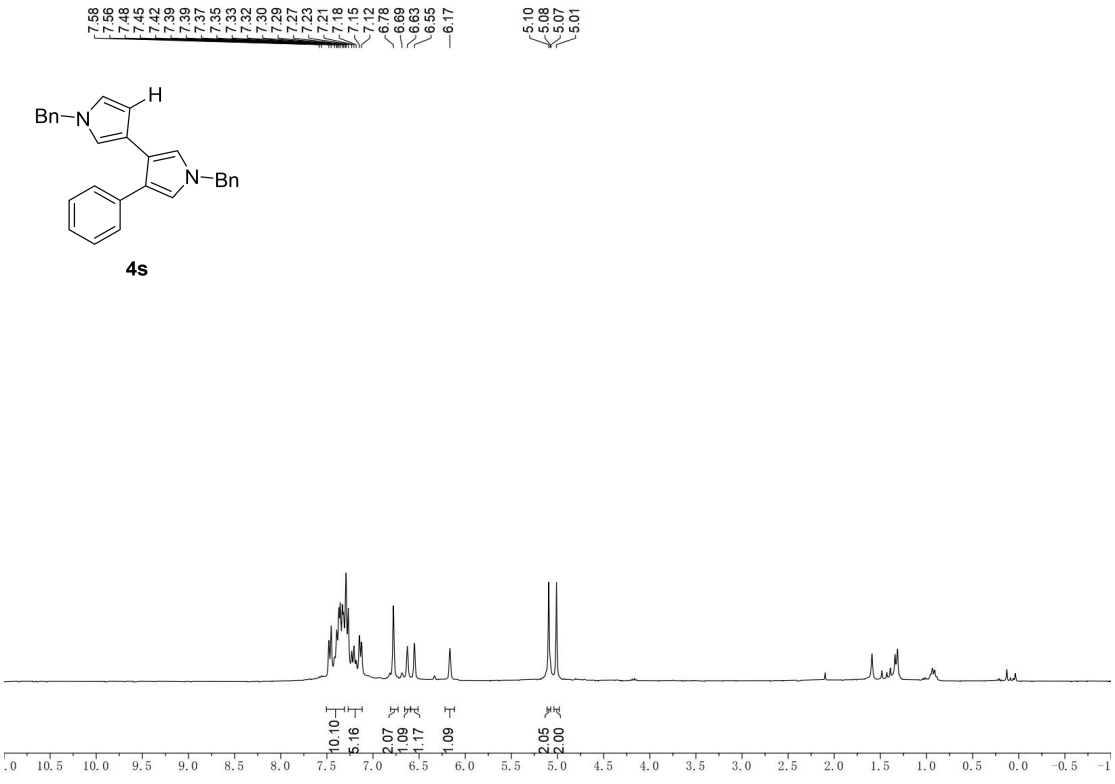
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 4r:



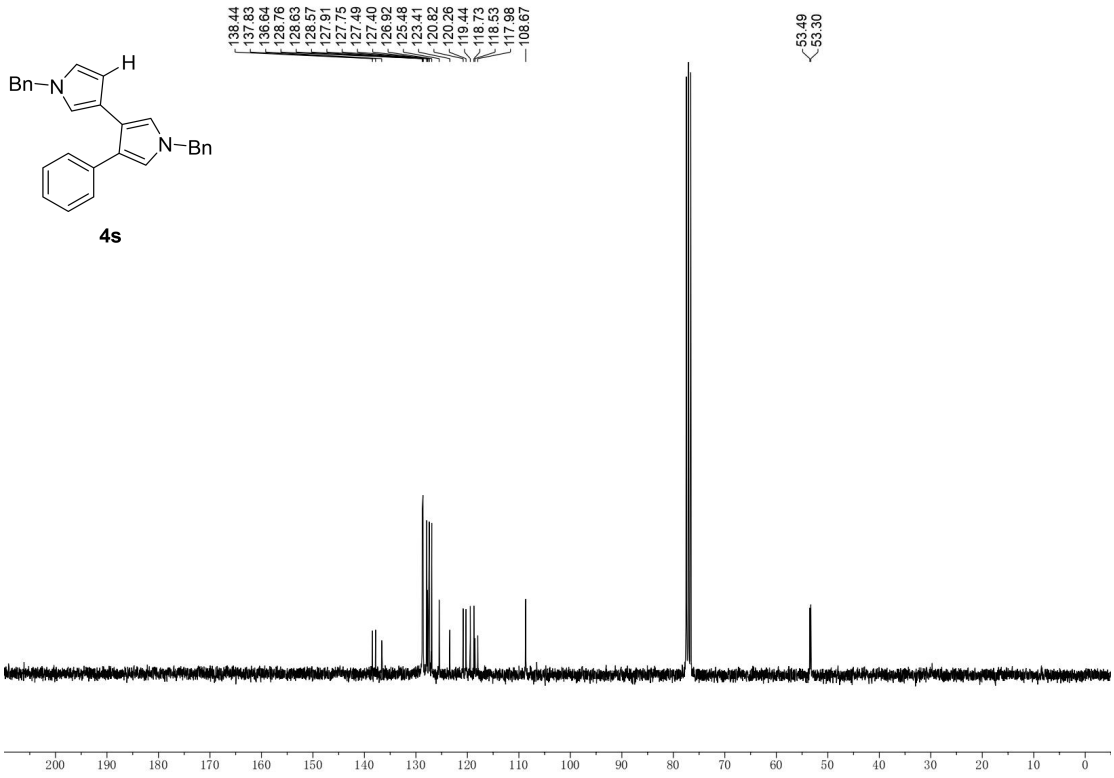
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 4r:



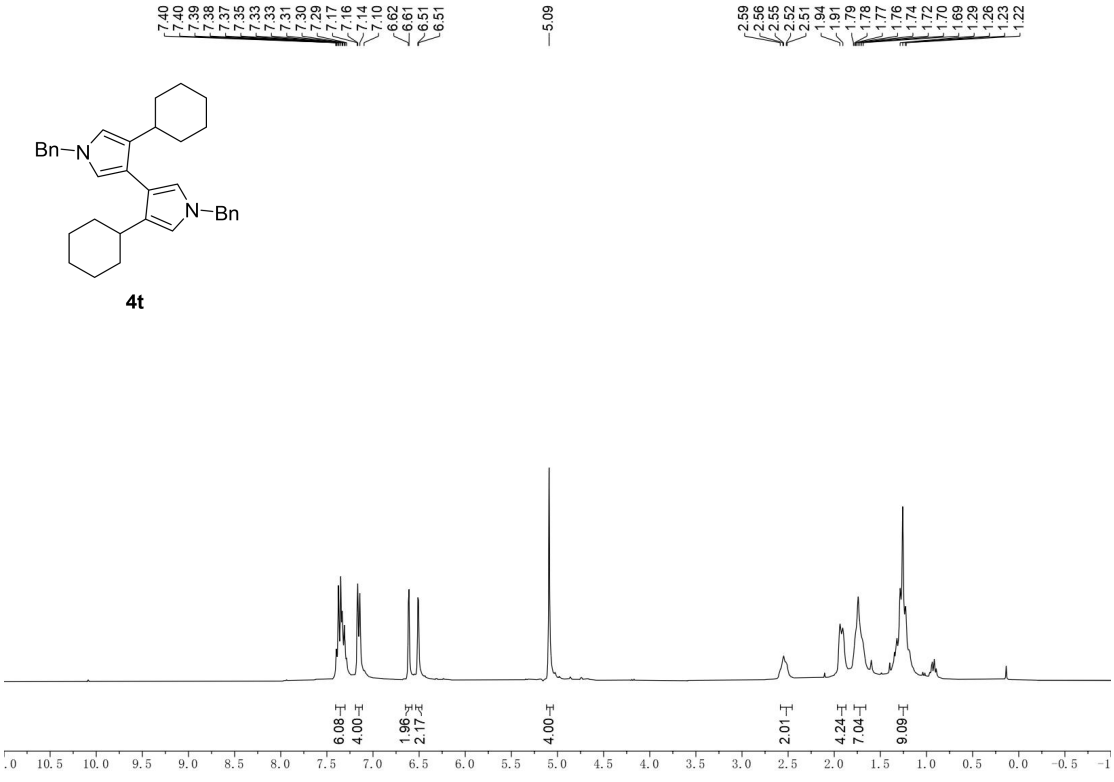
^{1}H NMR (300 MHz, $CDCl_3$) spectra of compound 4s:



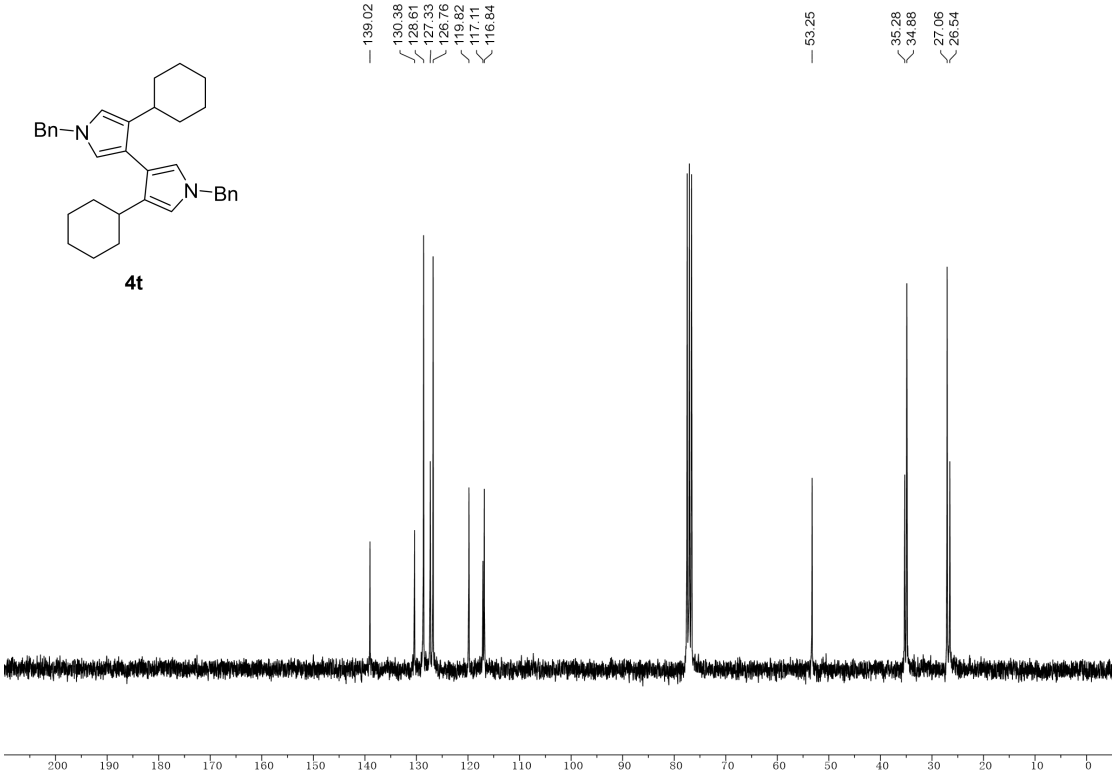
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectra of compound 4s:



^{1}H NMR (300 MHz, $CDCl_3$) spectra of compound 4t:

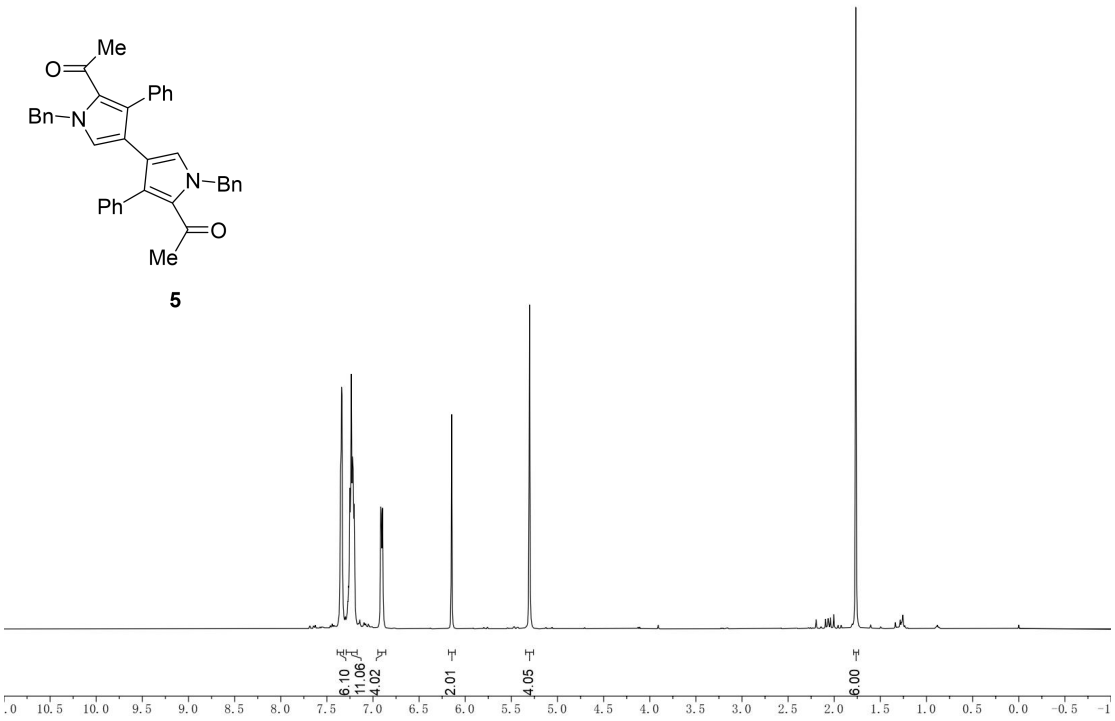


$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectra of compound 4t:

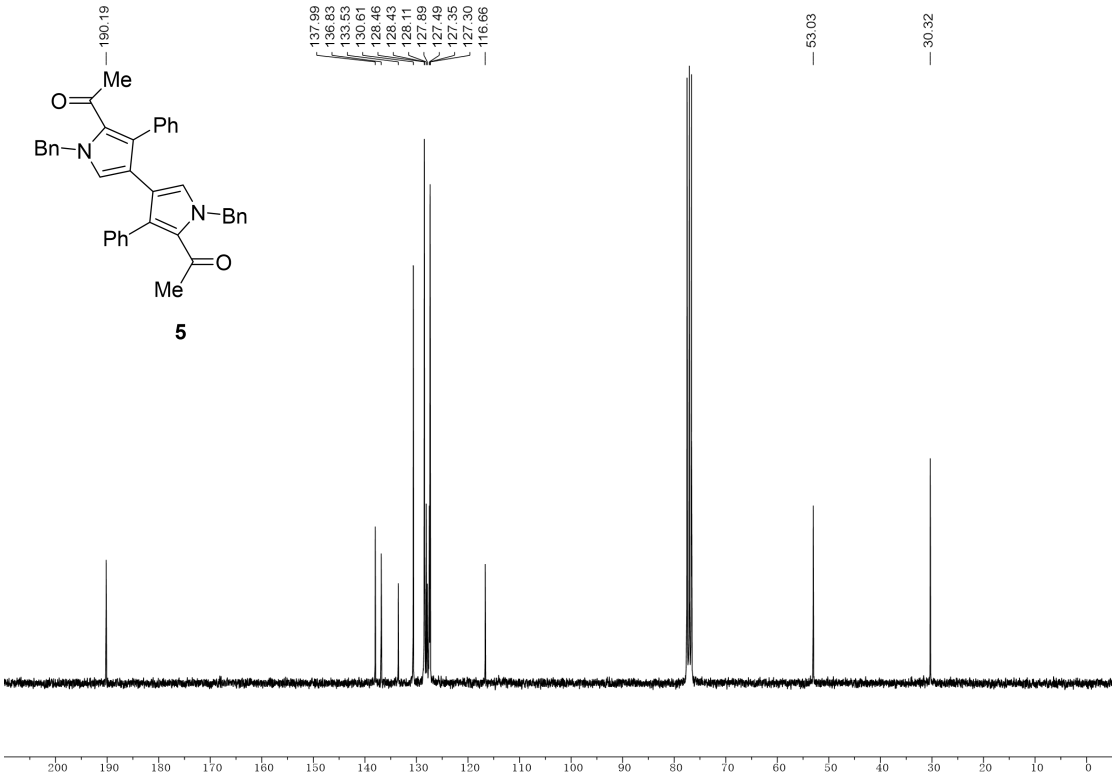


16.NMR spectra copies of the compounds 5, 6a, 6b, 7, 8, 9, 10, 11.

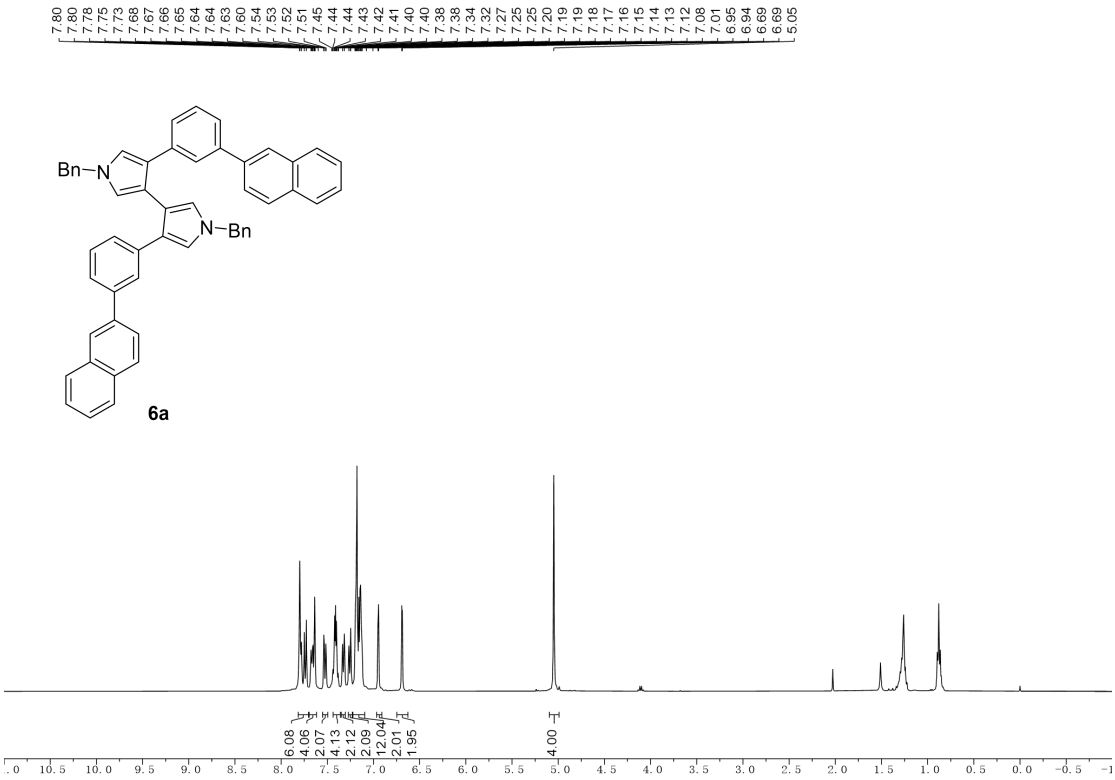
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 5:



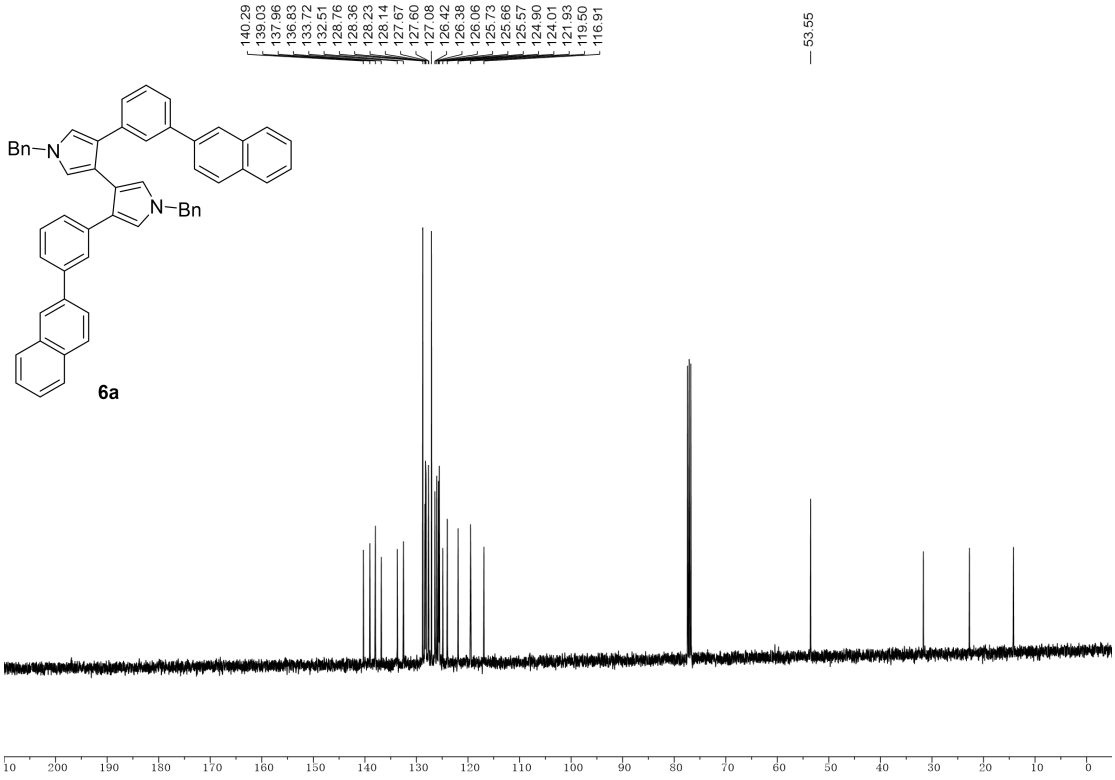
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$) spectra of compound 5:



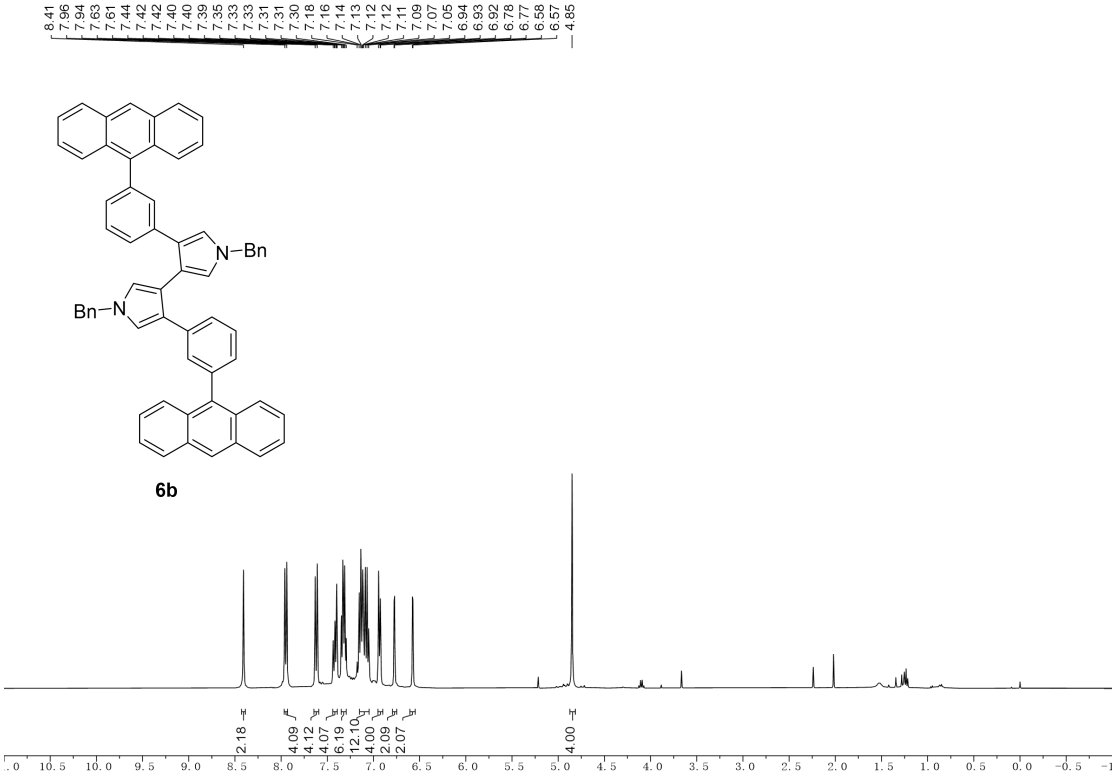
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 6a:



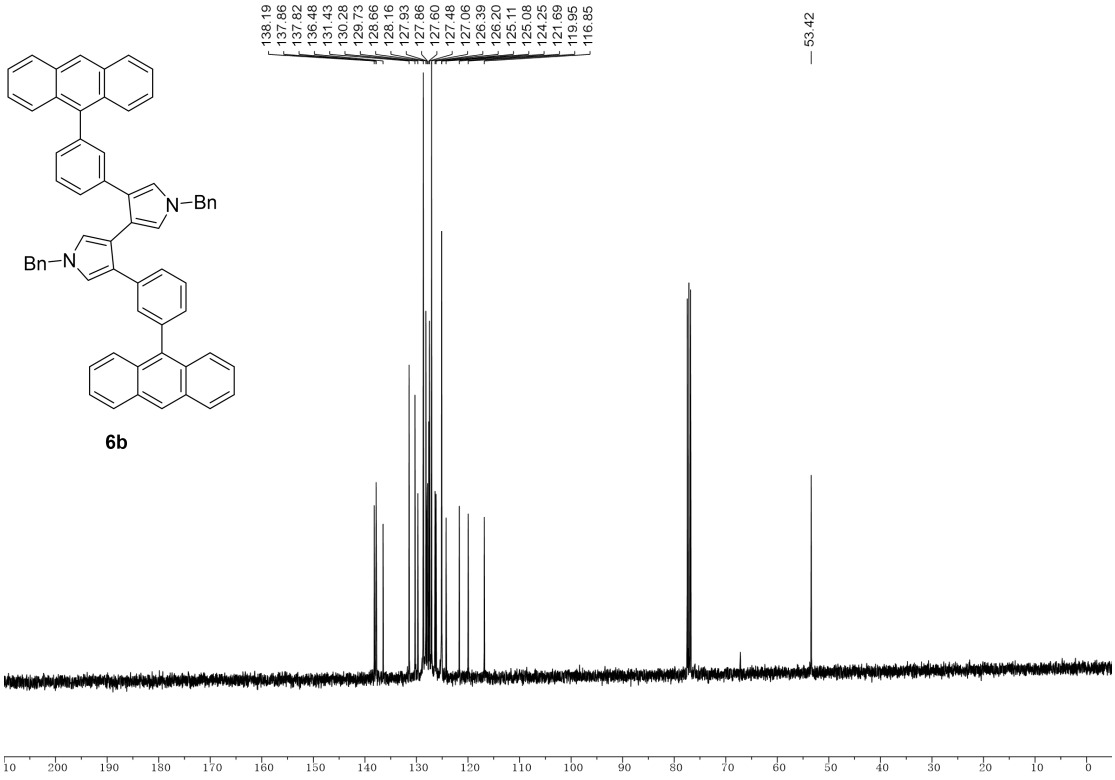
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 6a:



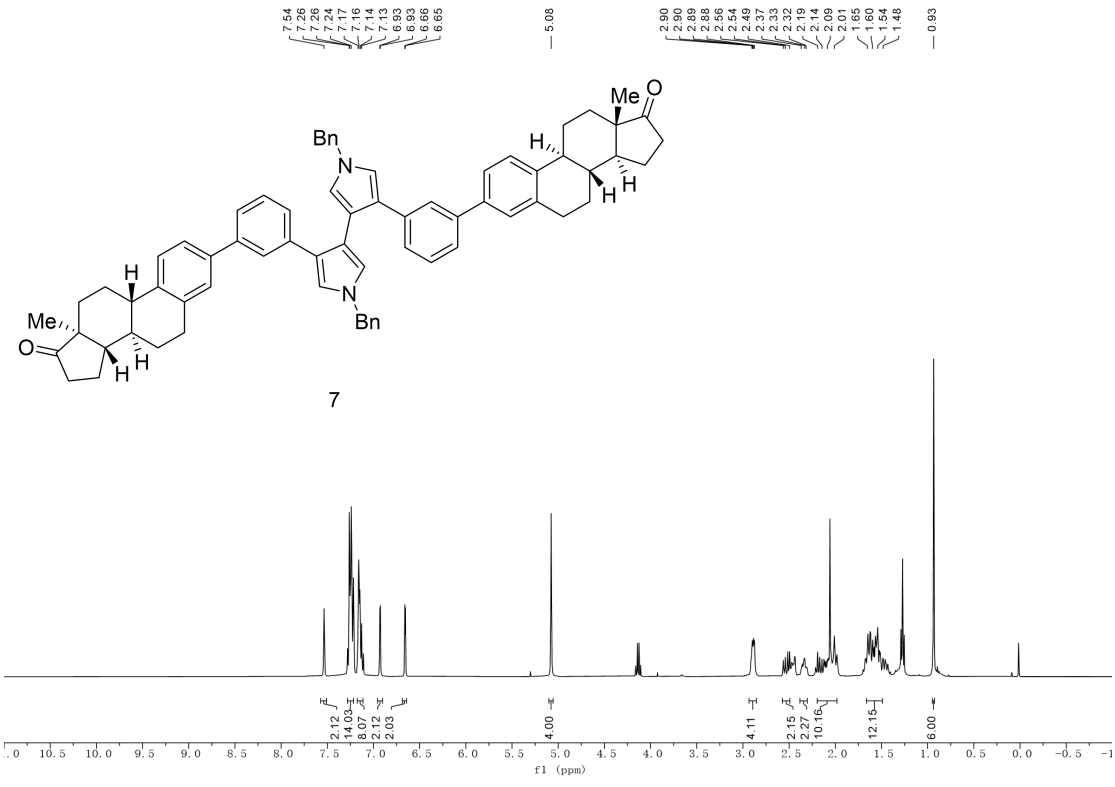
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 6b:



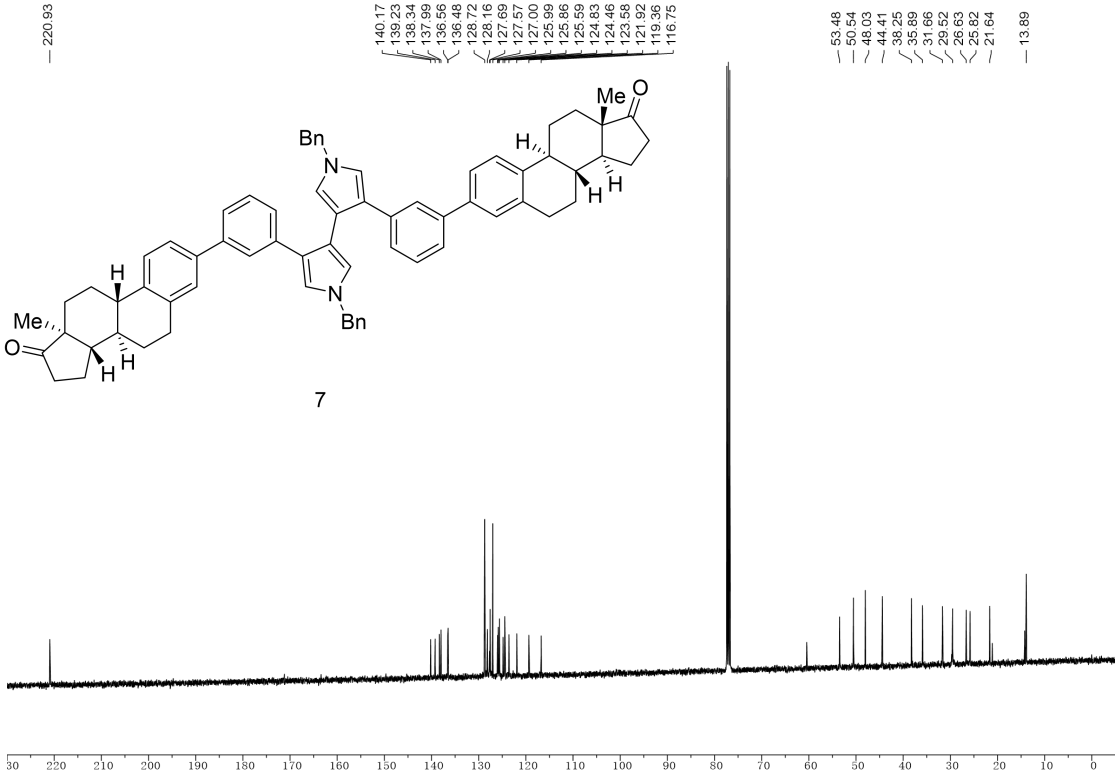
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 6b:



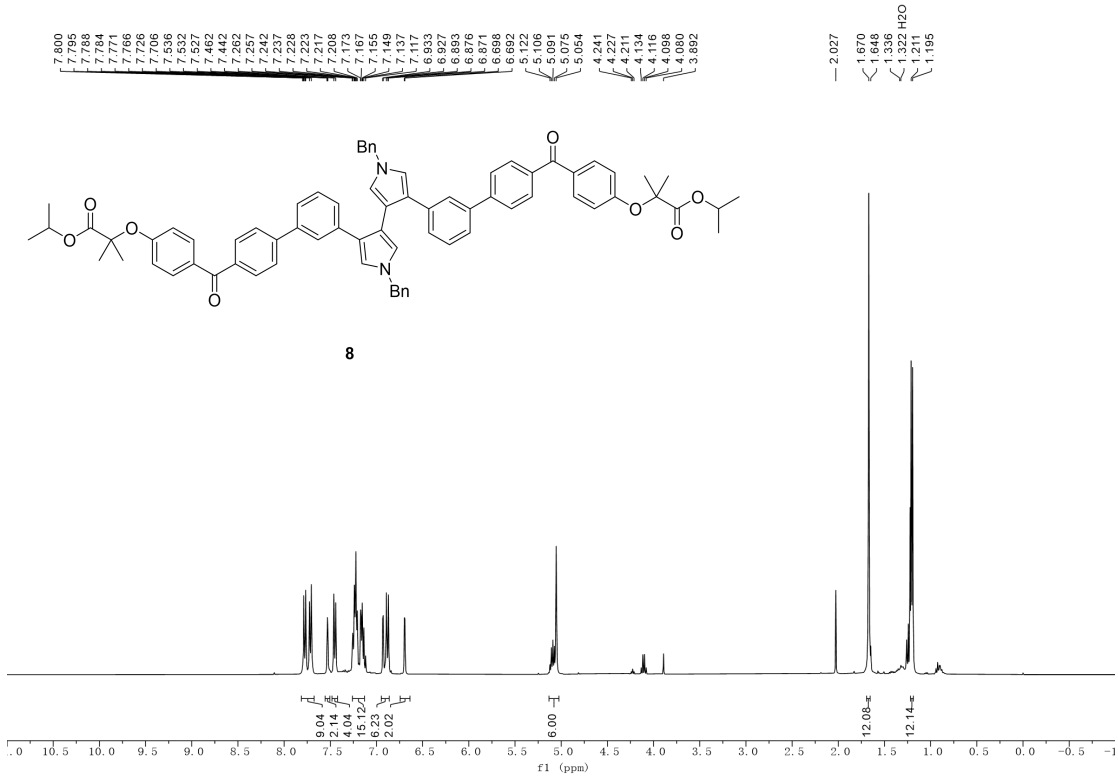
^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 7:



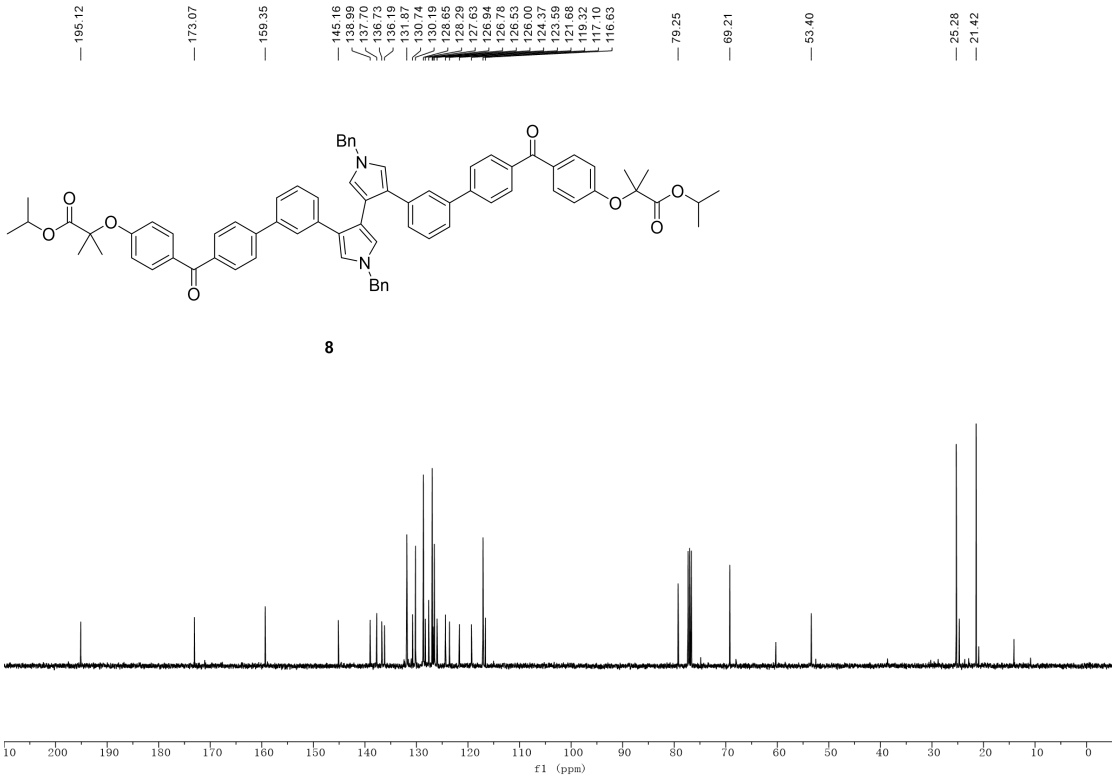
$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 7:



^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 8

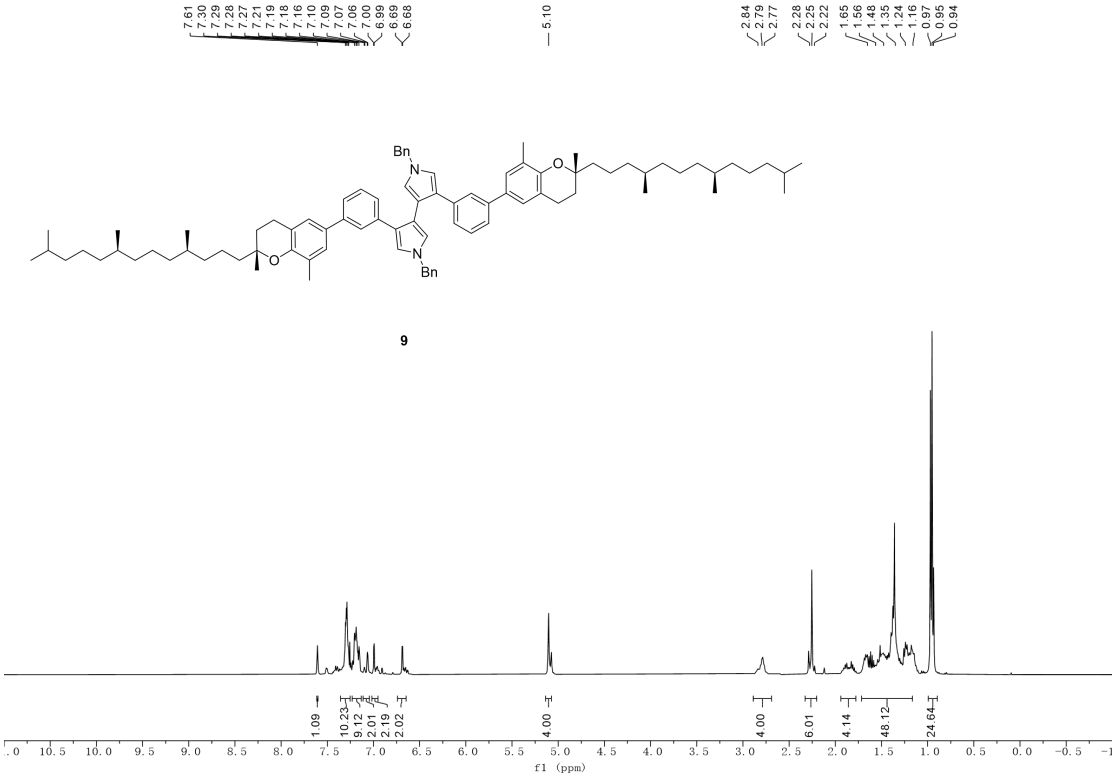


$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 8



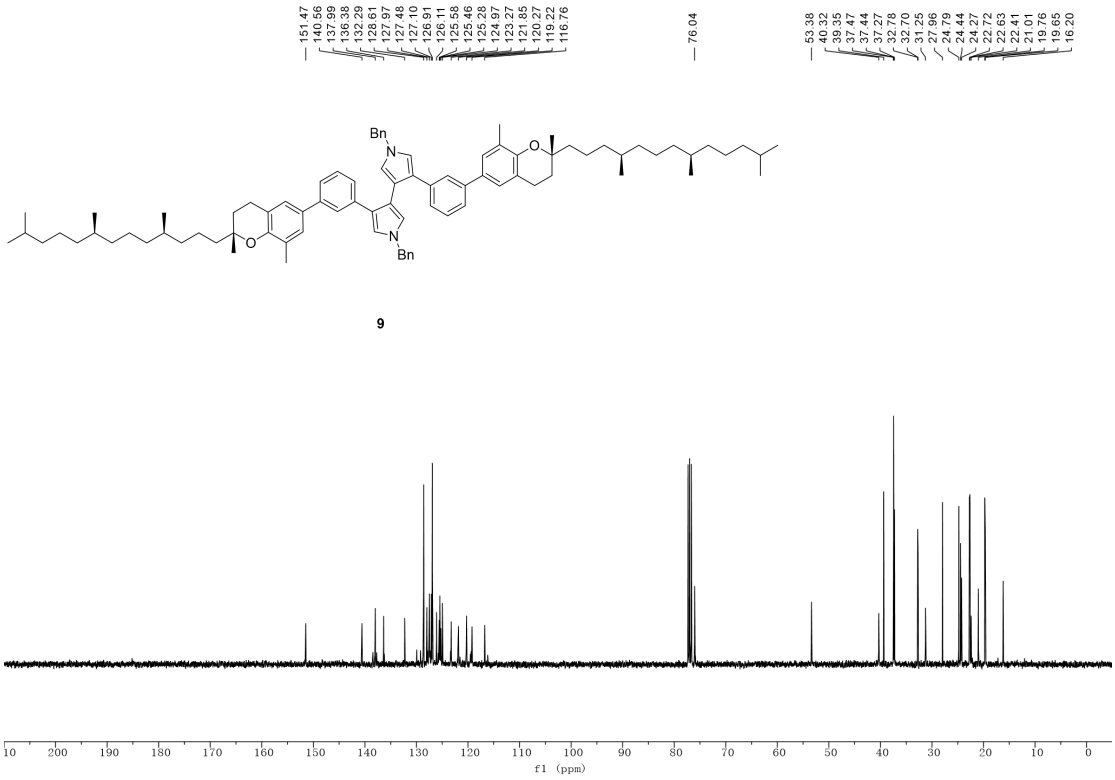
8

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 9



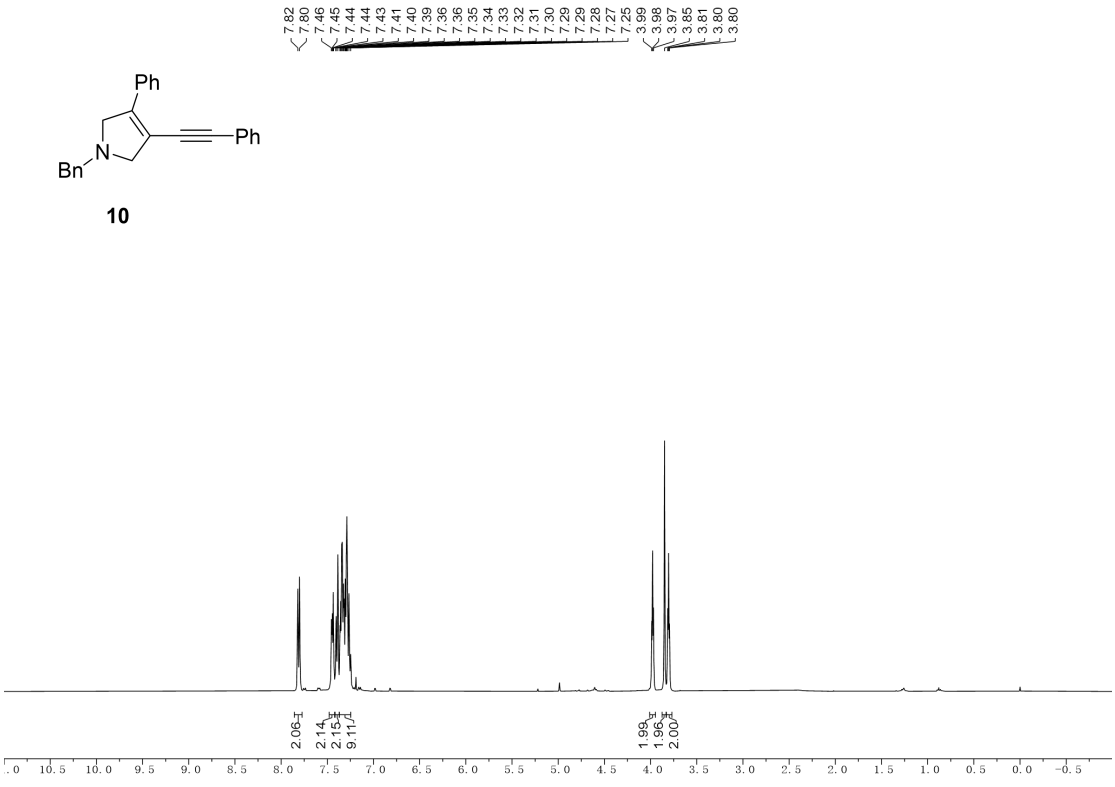
9

$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 9



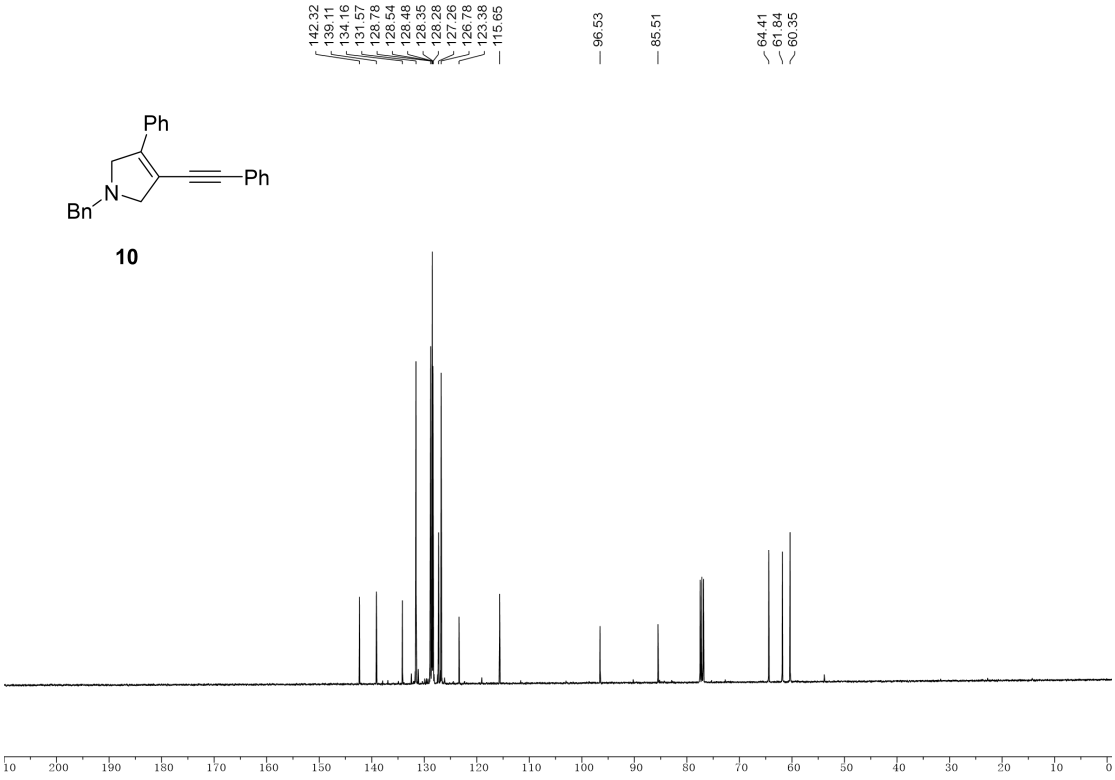
9

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 10



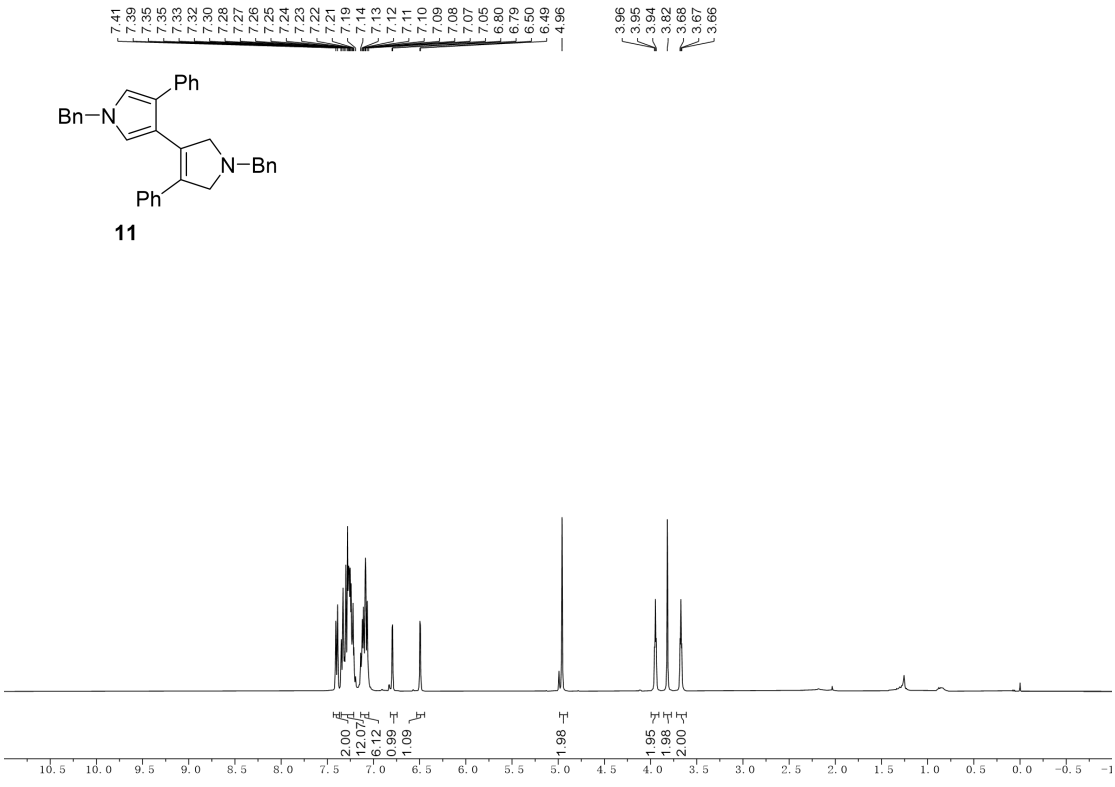
10

$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 10



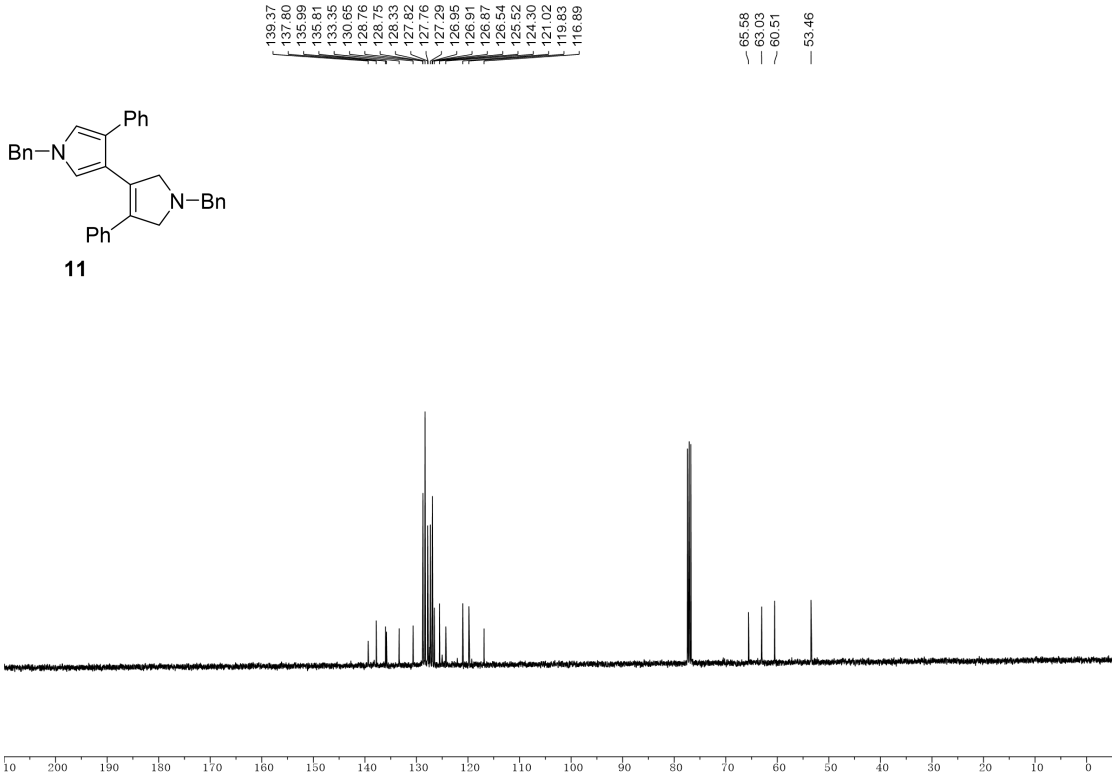
10

^{1}H NMR (400 MHz, $CDCl_3$) spectra of compound 11



11

$^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) spectra of compound 11



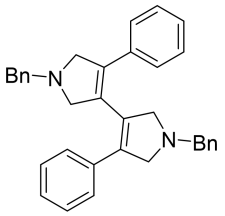
17.HRMS copies of the compounds

3a. HRMS (ESI) calcd for $C_{34}H_{33}N_2^+$ *m/z* 469.2644 $[M+H]^+$, Found 469.2647.

Elemental Composition Report **Page 1**

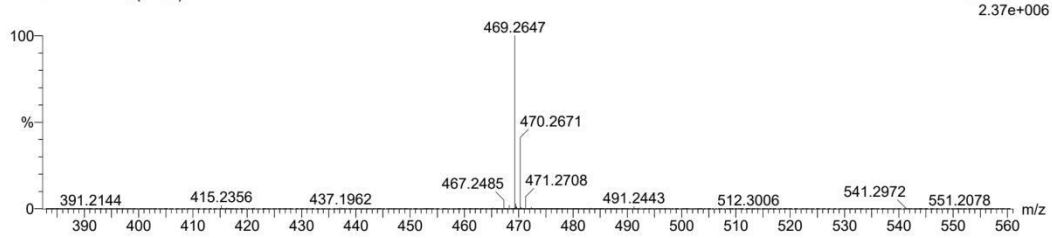
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3132 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 33-33 N: 0-30 O: 0-100 Na: 0-8
32
241102-2-T-471 13 (0.105)



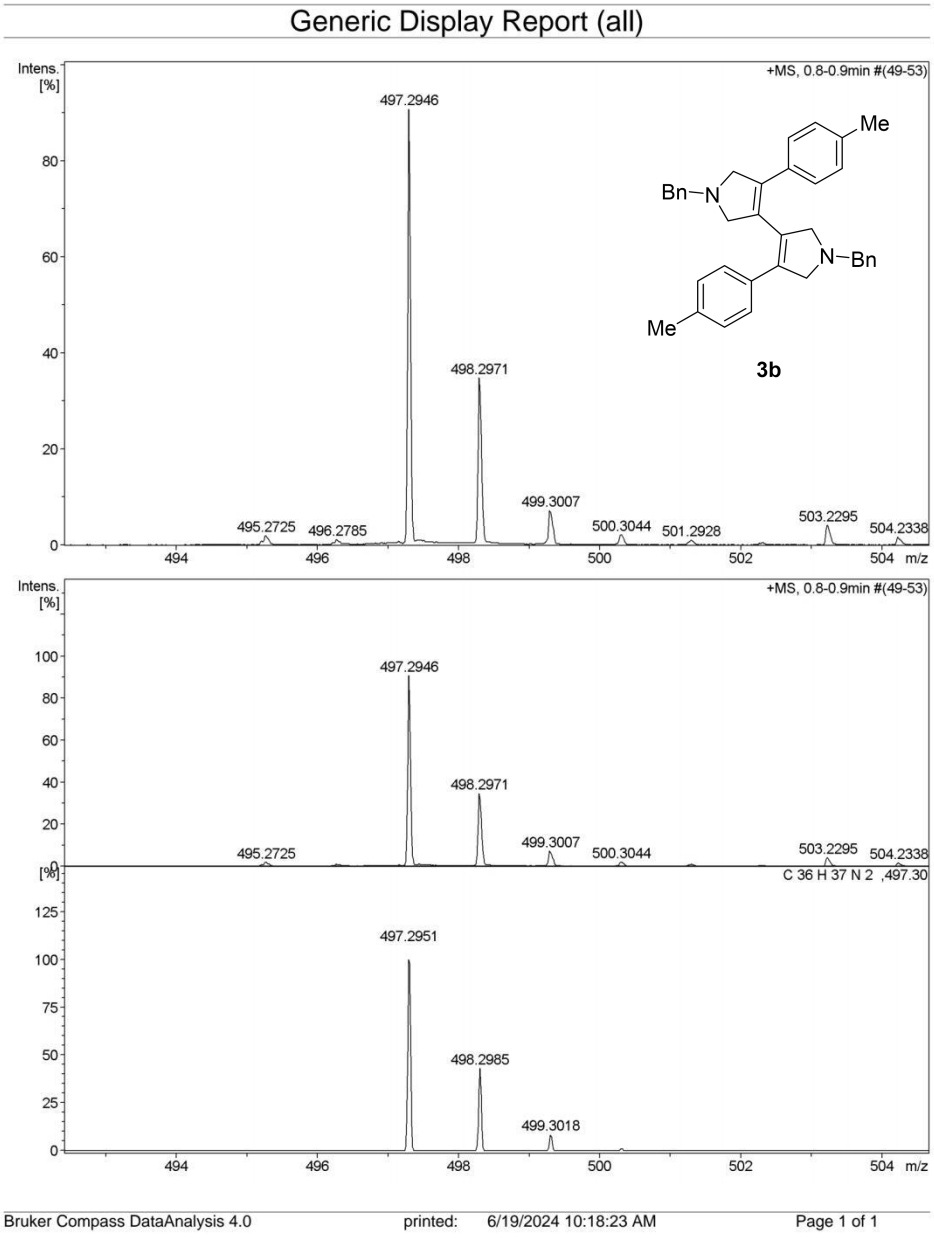
3a

1: TOF MS ES+
2.37e+006

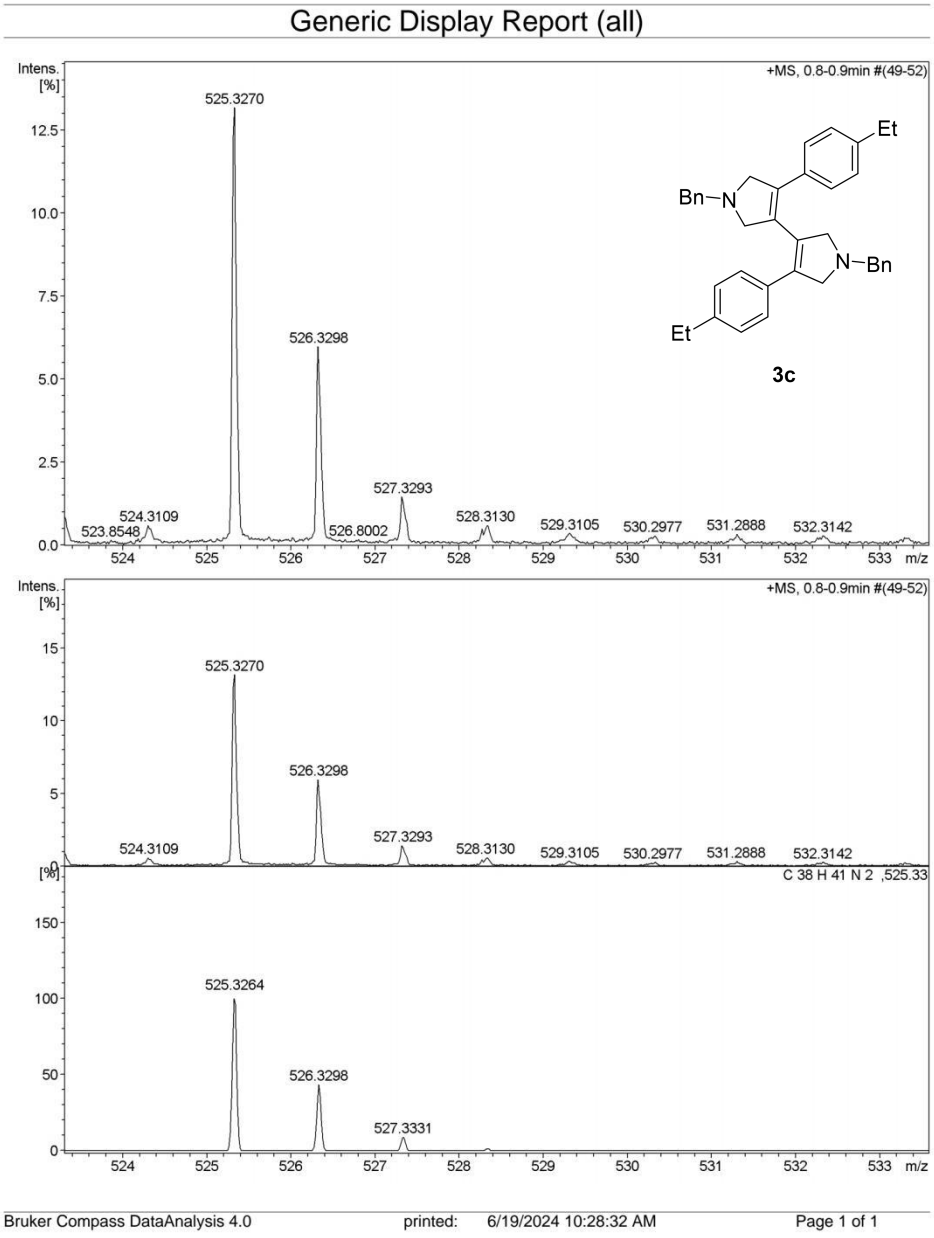


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
469.2647	469.2644	0.3	0.6	19.5	835.5	n/a	n/a	C34 H33 N2

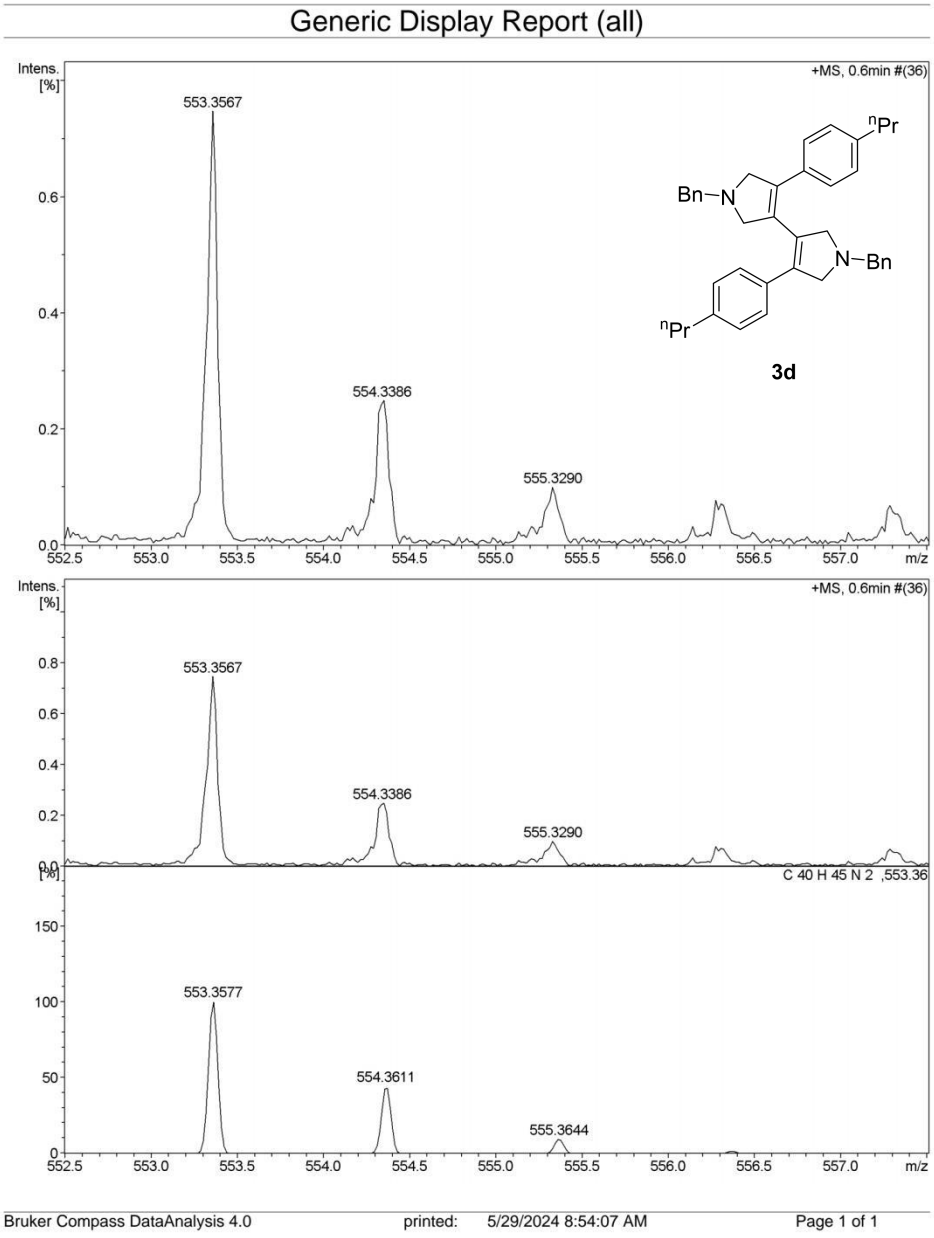
3b. HRMS (ESI) calcd for $C_{36}H_{37}N_2^+$ *m/z* 497.2951 $[M+H]^+$, Found 497.2946.



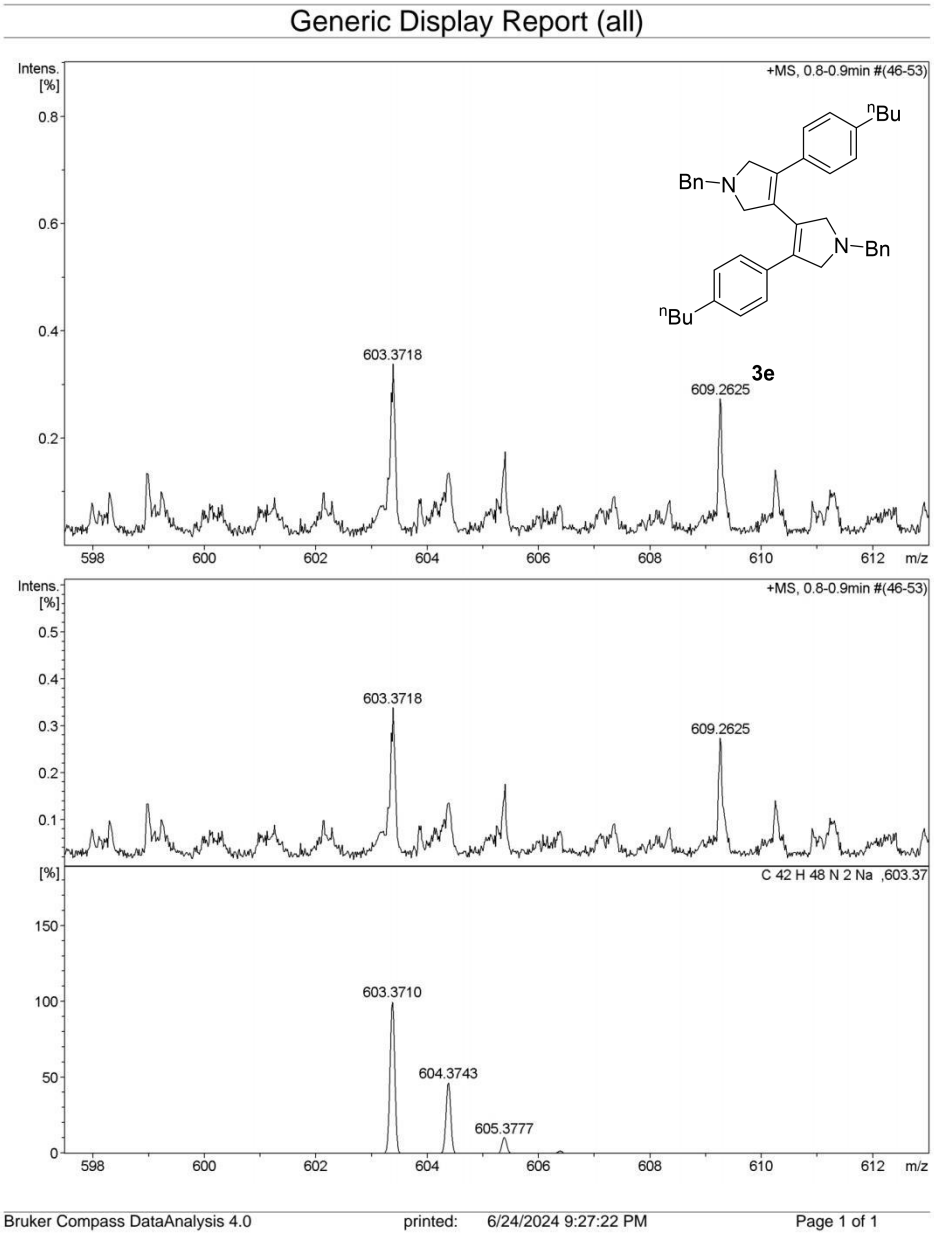
3c. HRMS (ESI) calcd for $C_{38}H_{41}N_2^+$ *m/z* 525.3264 $[M+H]^+$, Found 525.3270.



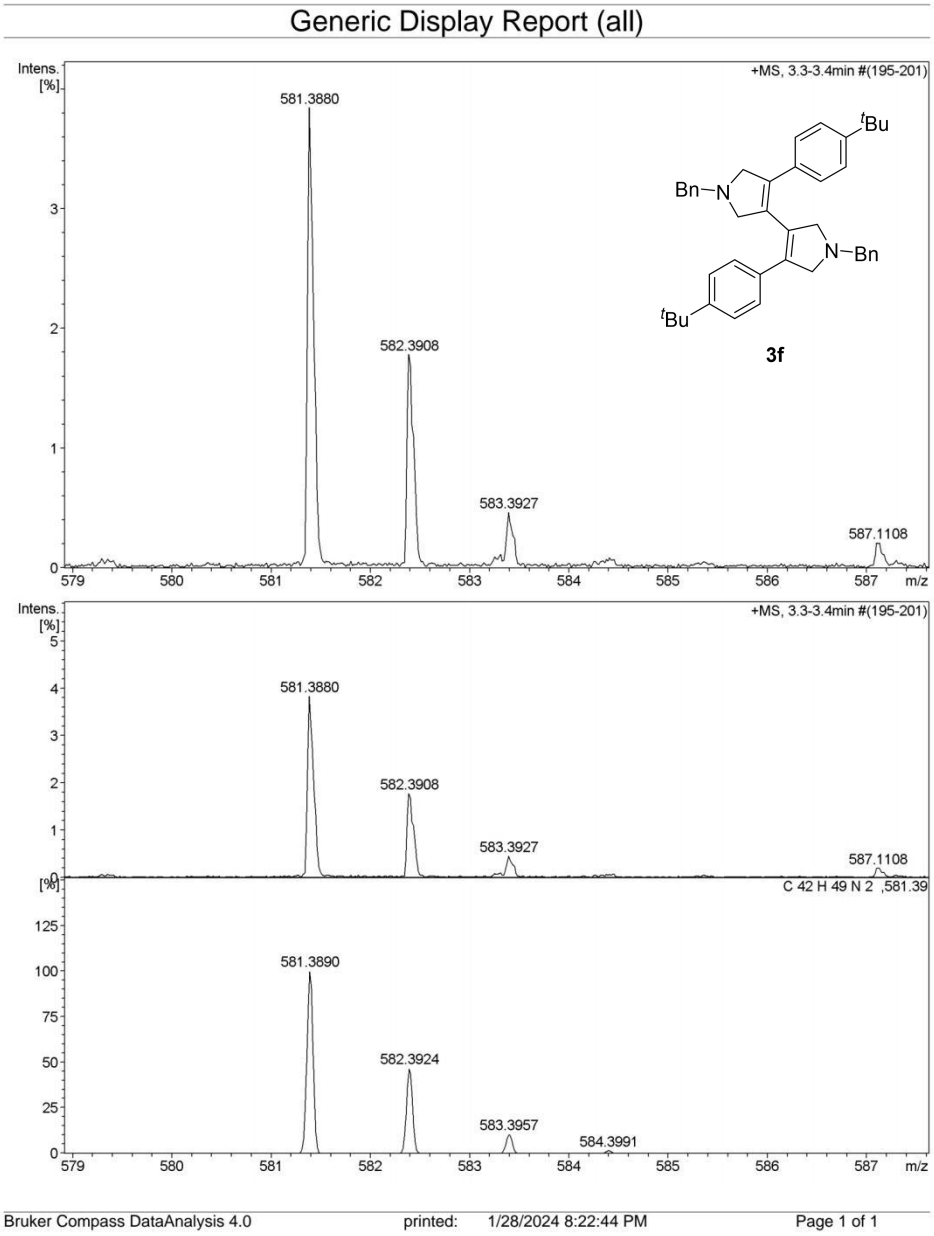
3d. HRMS (ESI) calcd for $C_{40}H_{45}N_2^+$ *m/z* 553.3577 $[M+H]^+$, Found 553.3567.



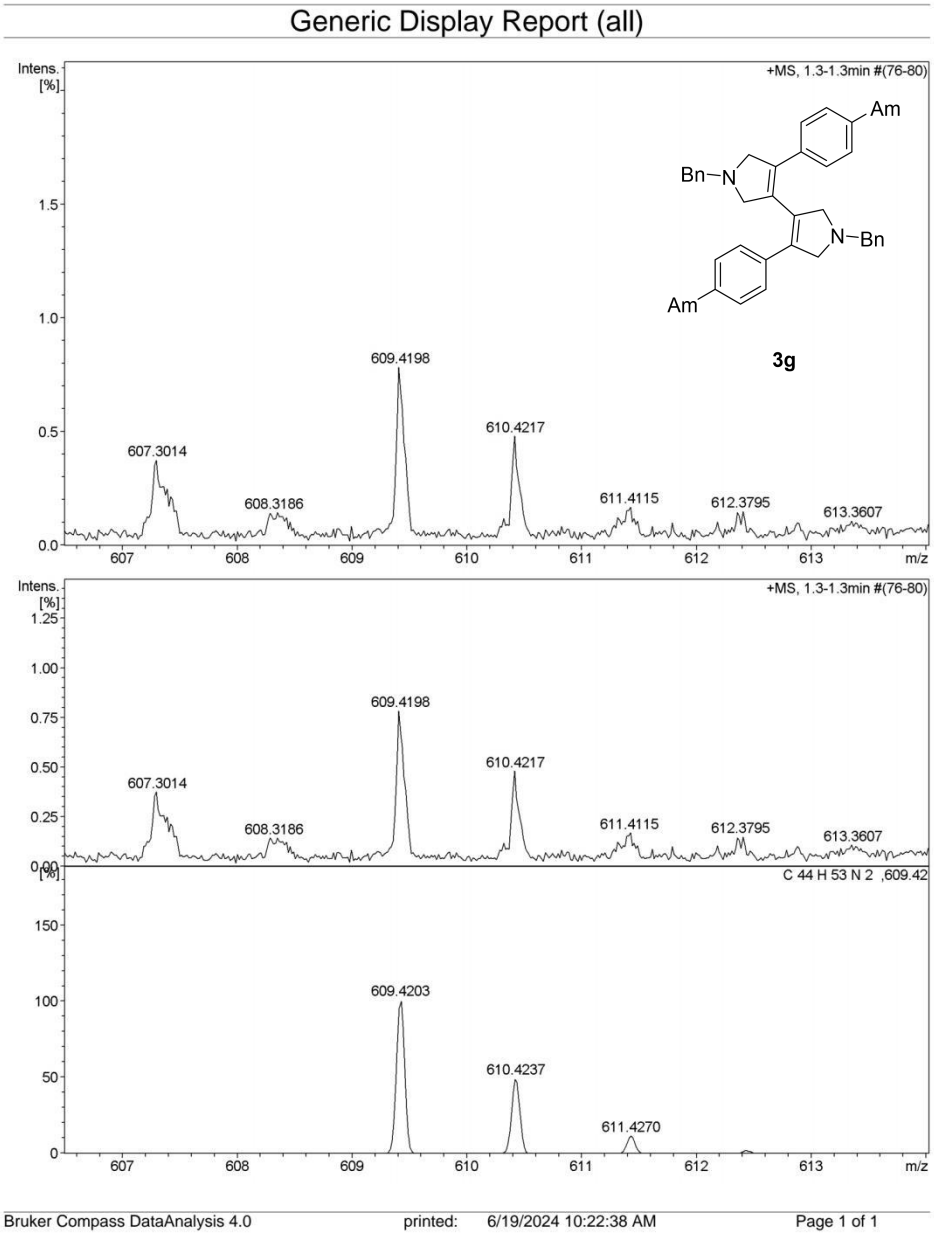
3e. HRMS (ESI) calcd for $C_{42}H_{48}N_2Na^+$ *m/z* 603.3710 $[M+Na]^+$, Found 603.3718.



3f. HRMS (ESI) calcd for $C_{42}H_{49}N_2^+$ *m/z* 581.3890 $[M+H]^+$, Found 581.3880.



3g. HRMS (ESI) calcd for $C_{44}H_{53}N_2^+$ *m/z* 609.4203 $[M+H]^+$, Found 609.4198.



3h. HRMS (ESI) calcd for $C_{46}H_{41}N_2^+$ *m/z* 621.3270 $[M+H]^+$, Found 621.3275.

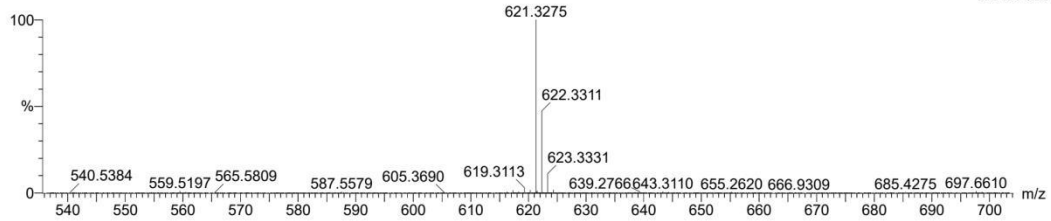
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
5678 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 46-46 H: 41-41 N: 0-30 O: 0-100 Na: 0-8

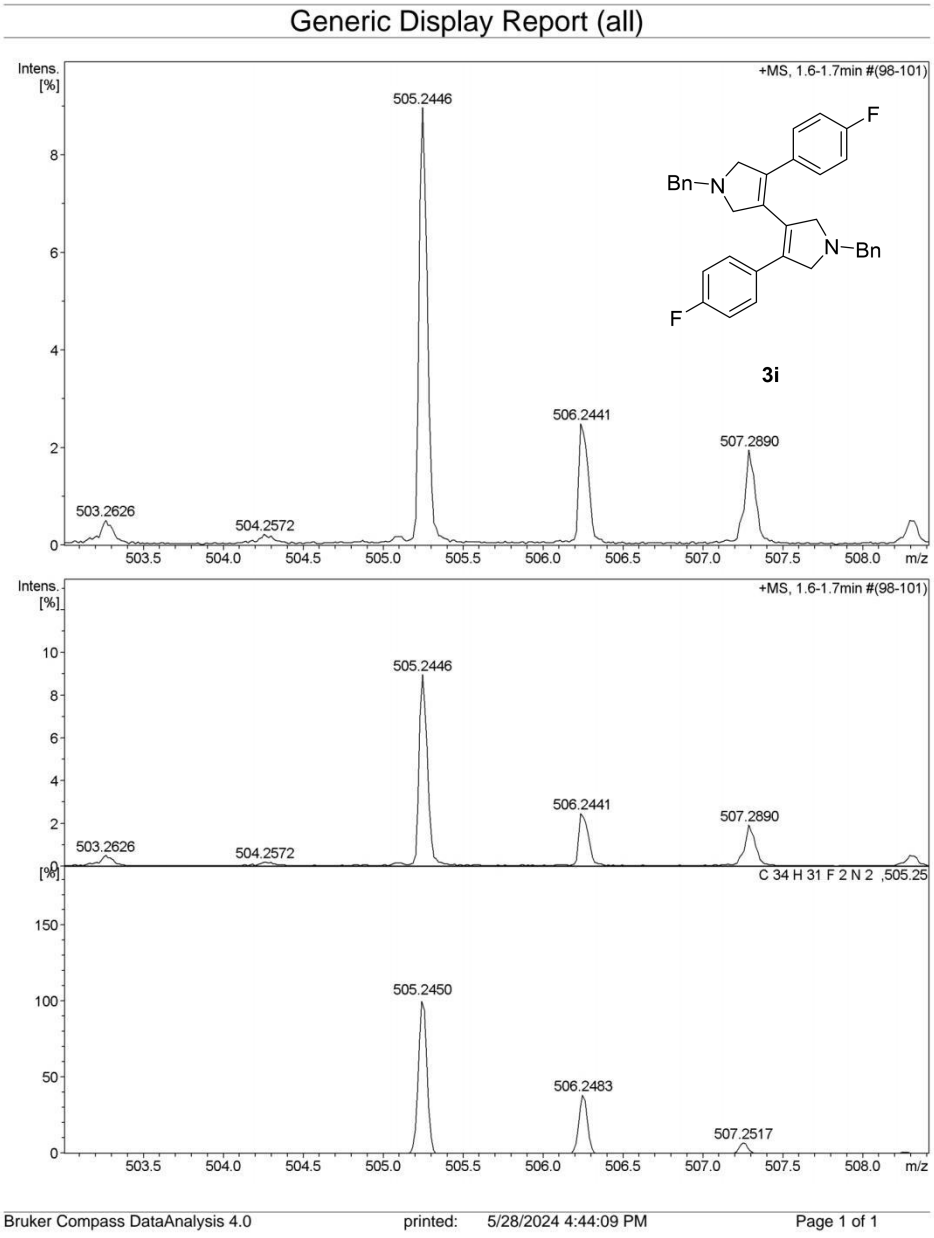
32
241102-2-T-443-1 26 (0.173)

1: TOF MS ES+
8.04e+005

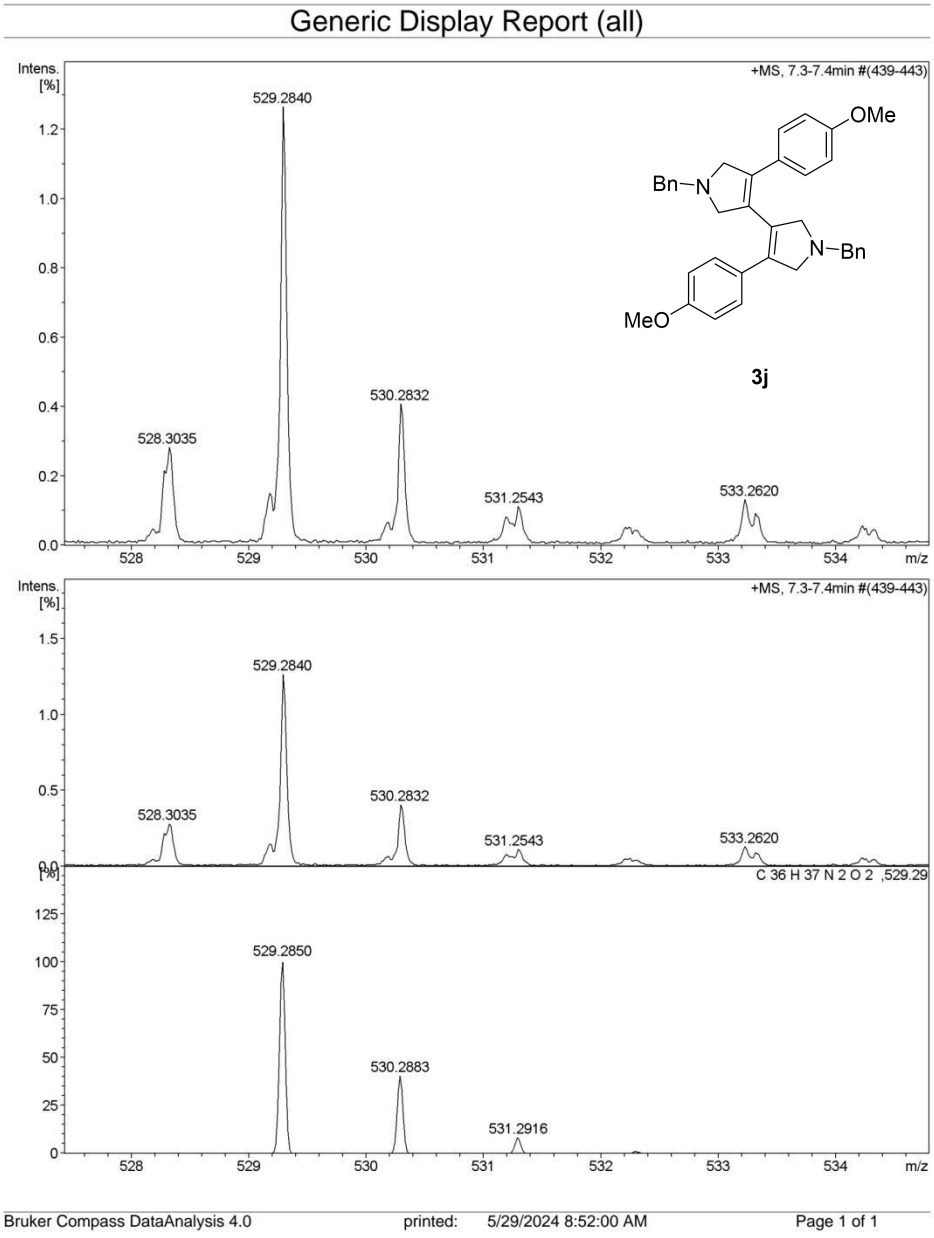


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
621.3275	621.3270	0.5	0.8	27.5	666.0	n/a	n/a	C46 H41 N2

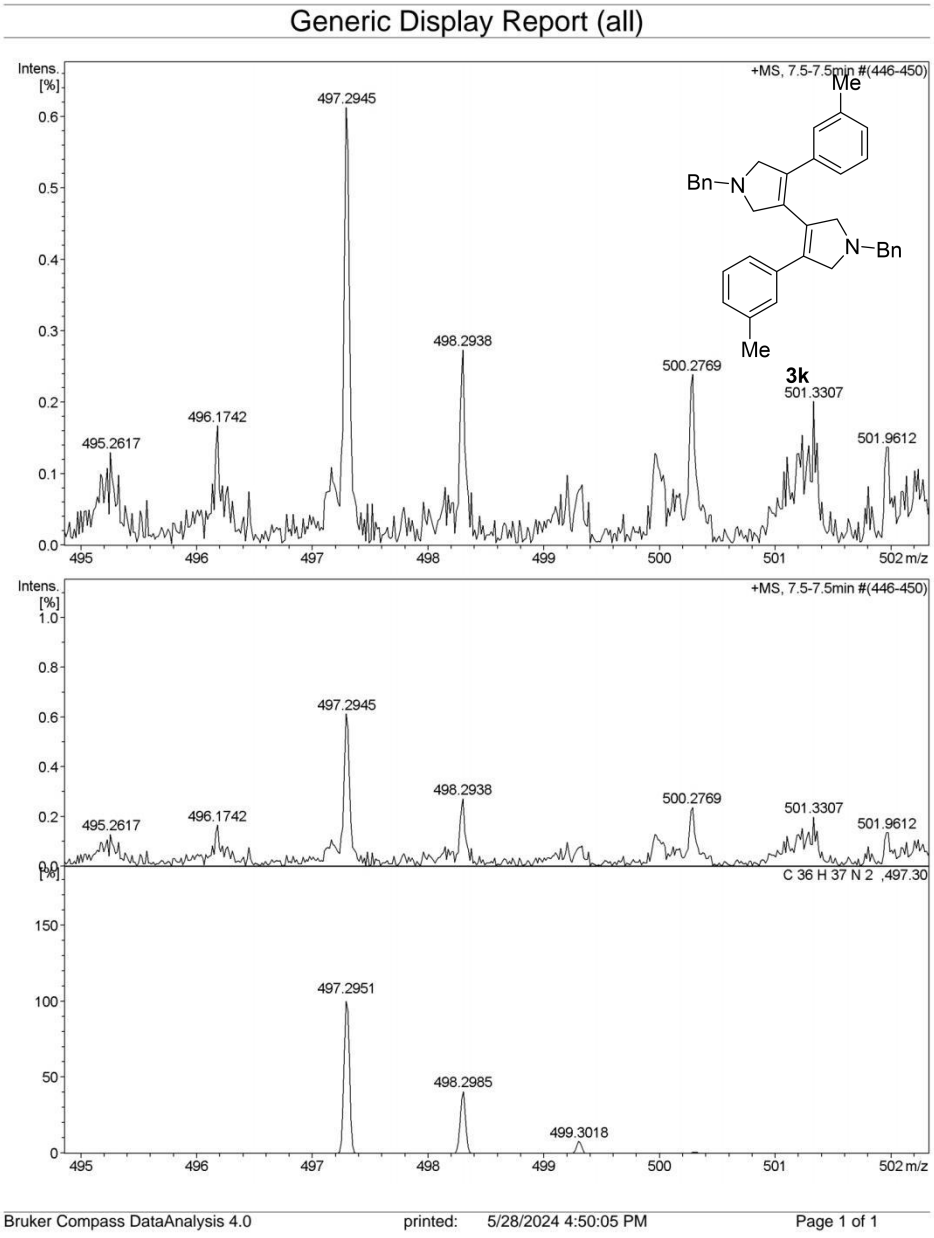
3i. HRMS (ESI) calcd for $C_{34}H_{31}F_2N_2^+$ *m/z* 505.2450 $[M+H]^+$, Found 505.2446.



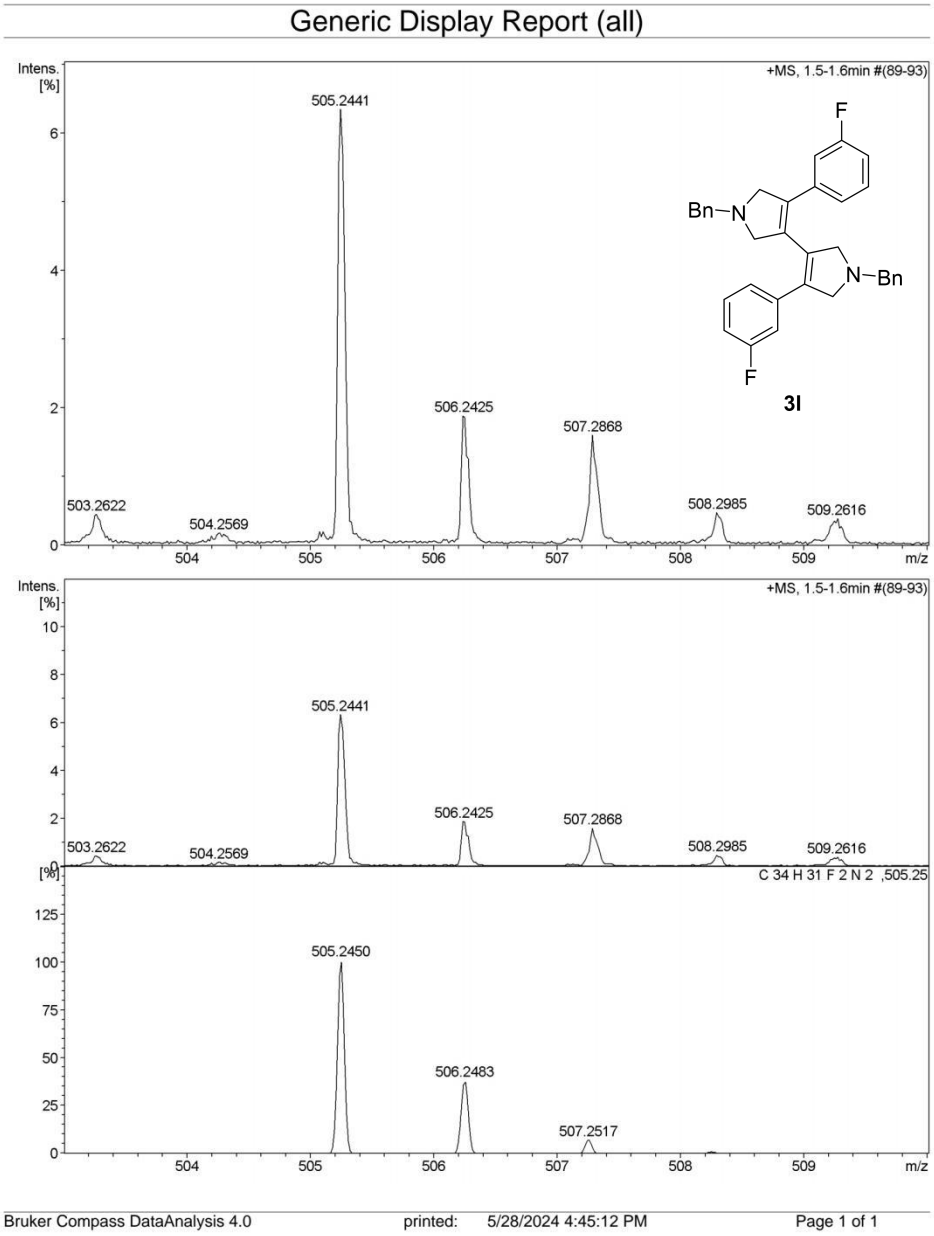
3j. HRMS (ESI) calcd for $C_{36}H_{37}N_2O_2^+$ *m/z* 529.2850 $[M+H]^+$, Found 529.2840.



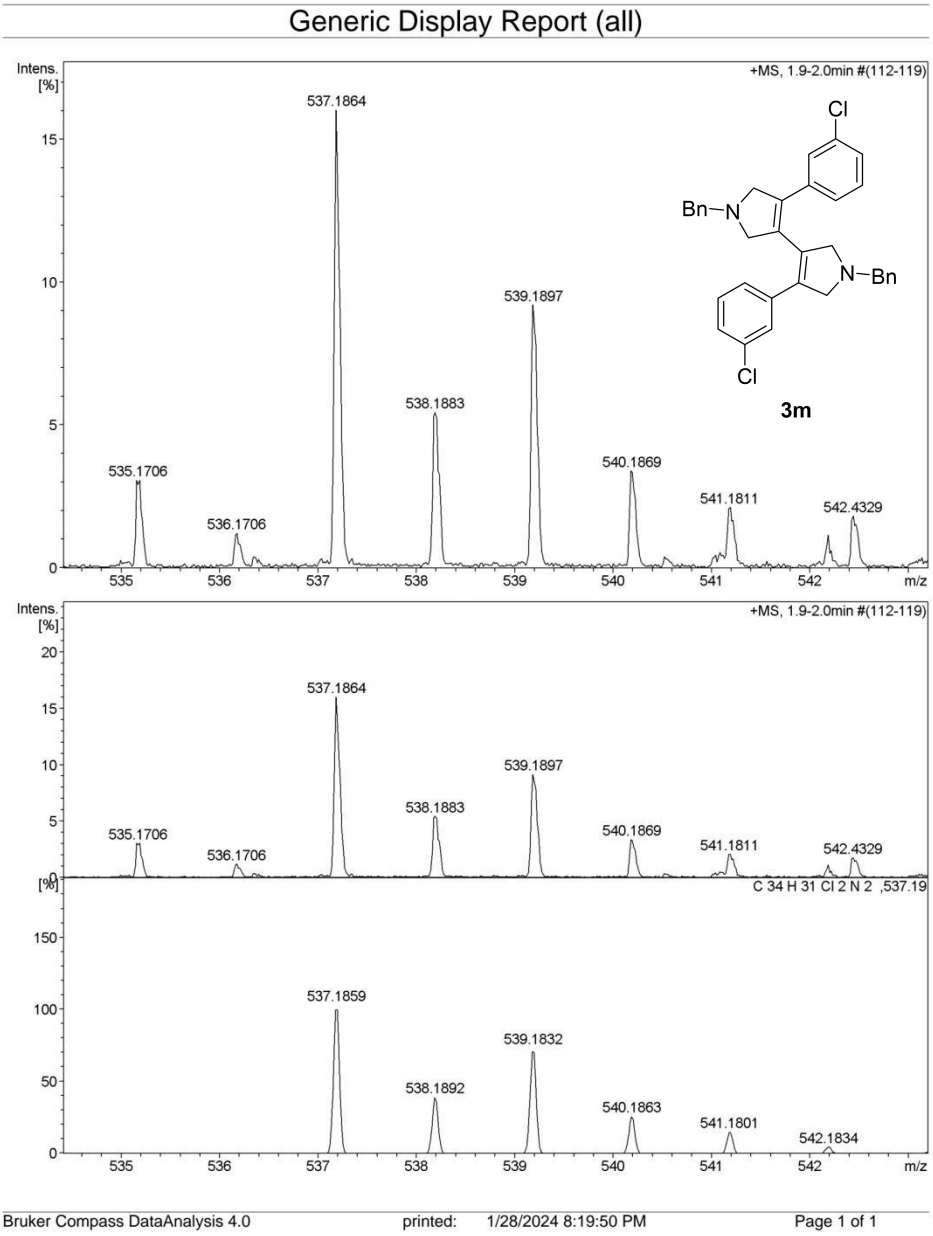
3k. HRMS (ESI) calcd for $C_{36}H_{37}N_2^+$ *m/z* 497.2951 $[M+H]^+$, Found 497.2945.



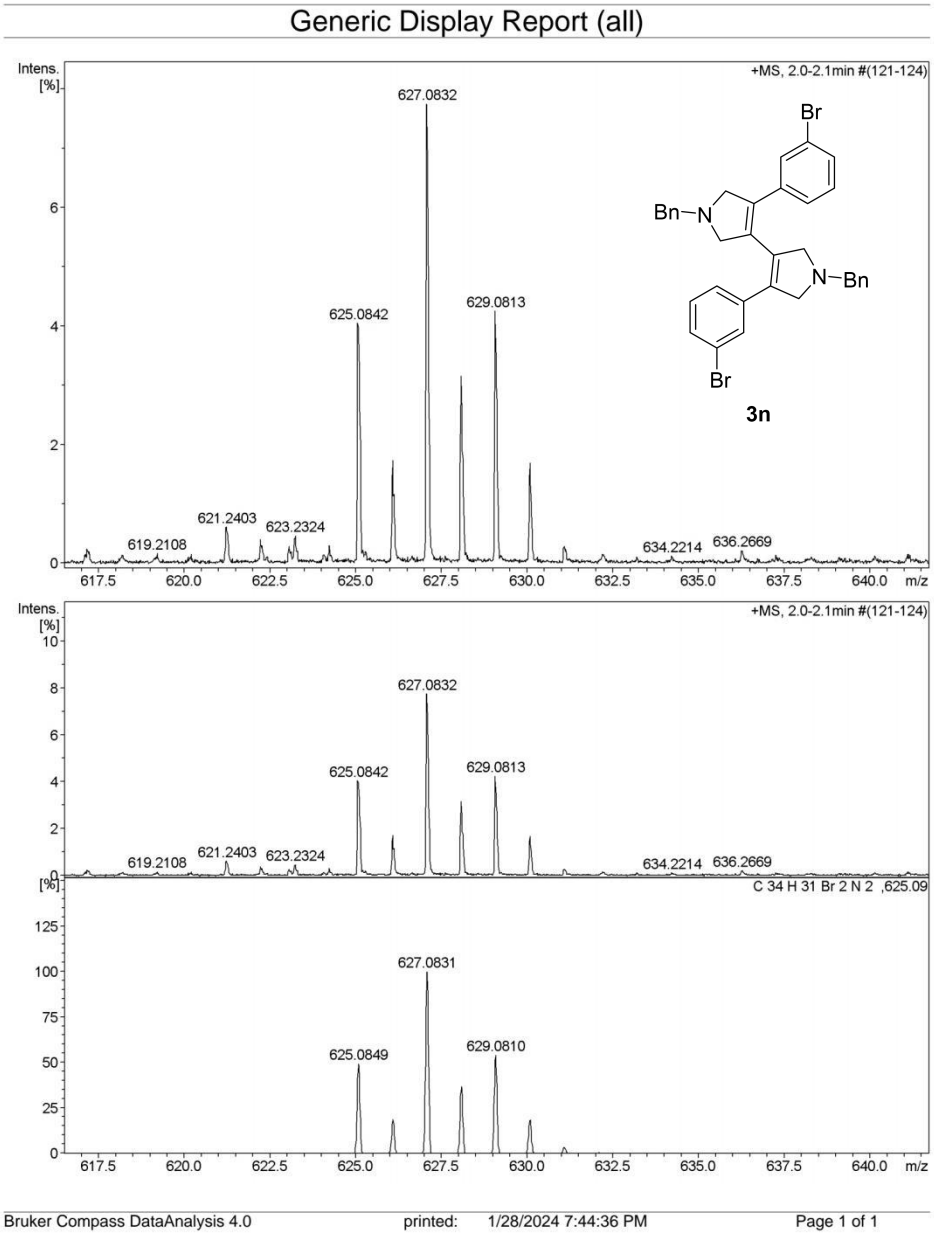
3l.HRMS (ESI) calcd for $C_{34}H_{31}F_2N_2^+$ *m/z* 505.2450 $[M+H]^+$, Found 505.2441.



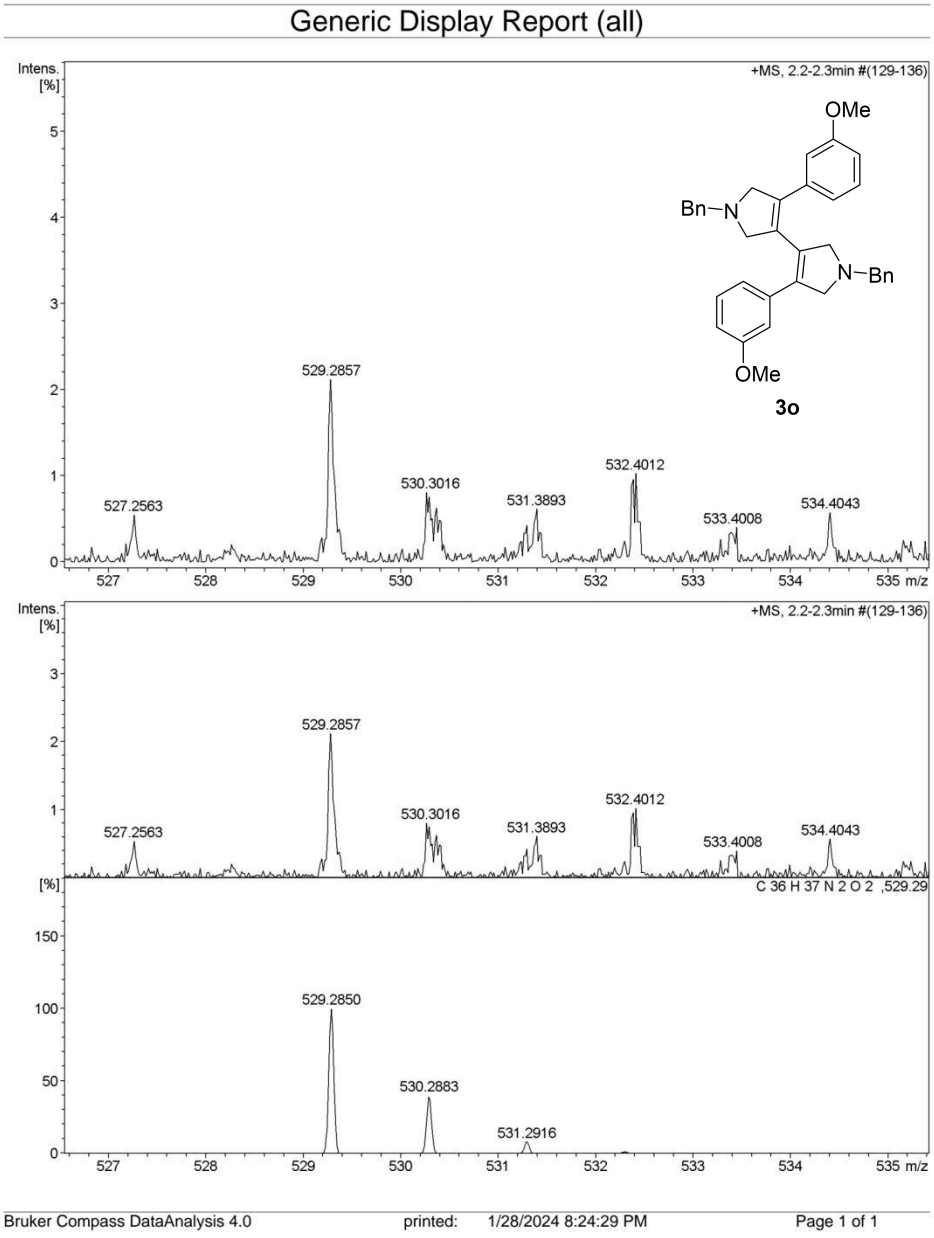
3m. HRMS (ESI) calcd for $C_{34}H_{31}Cl_2N_2^+$ *m/z* 537.1859 $[M+H]^+$, Found 537.1864.



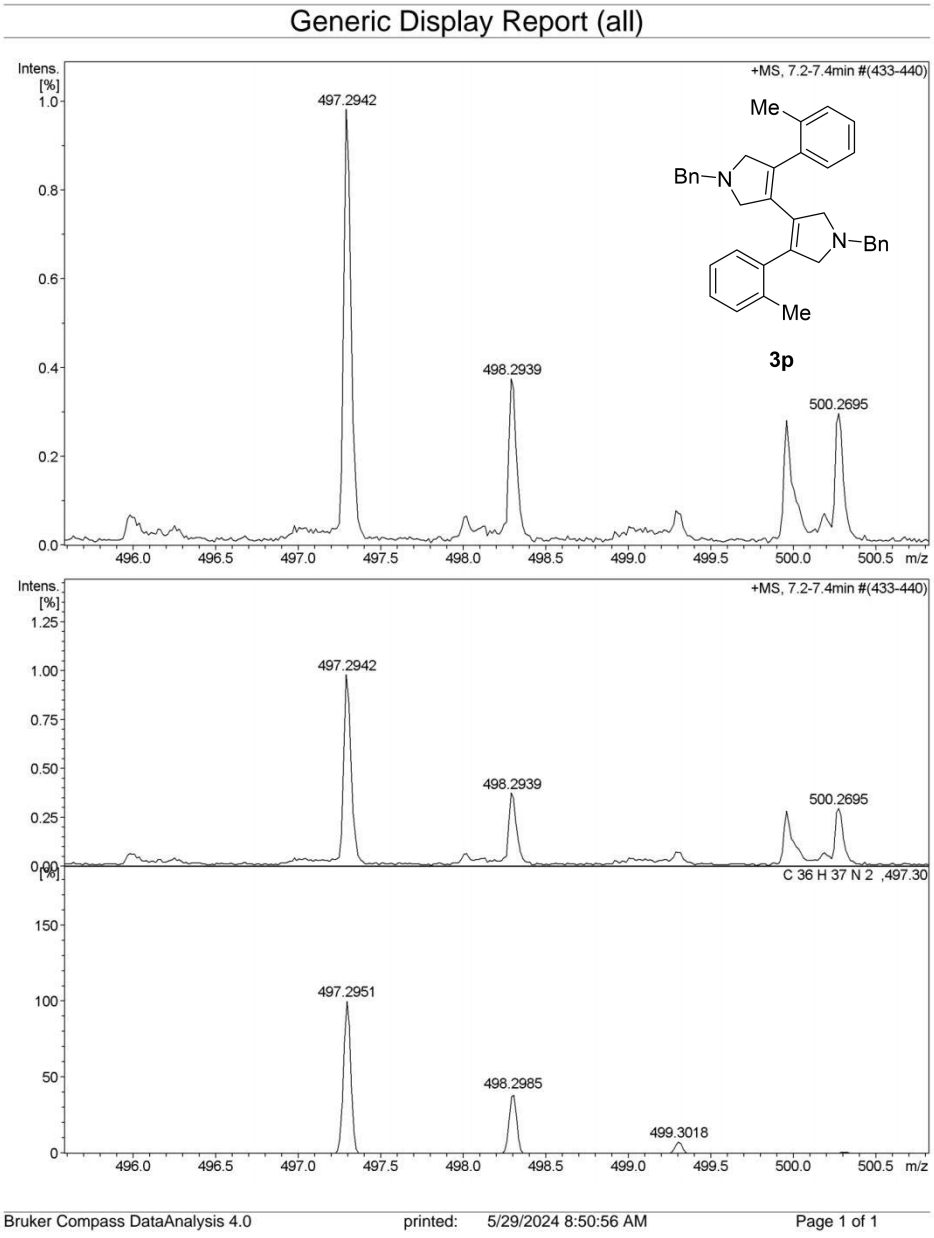
3n. HRMS (ESI) calcd for $C_{34}H_{31}Br_2N_2^+$ *m/z* 625.0849 $[M+H]^+$, Found 625.0842.



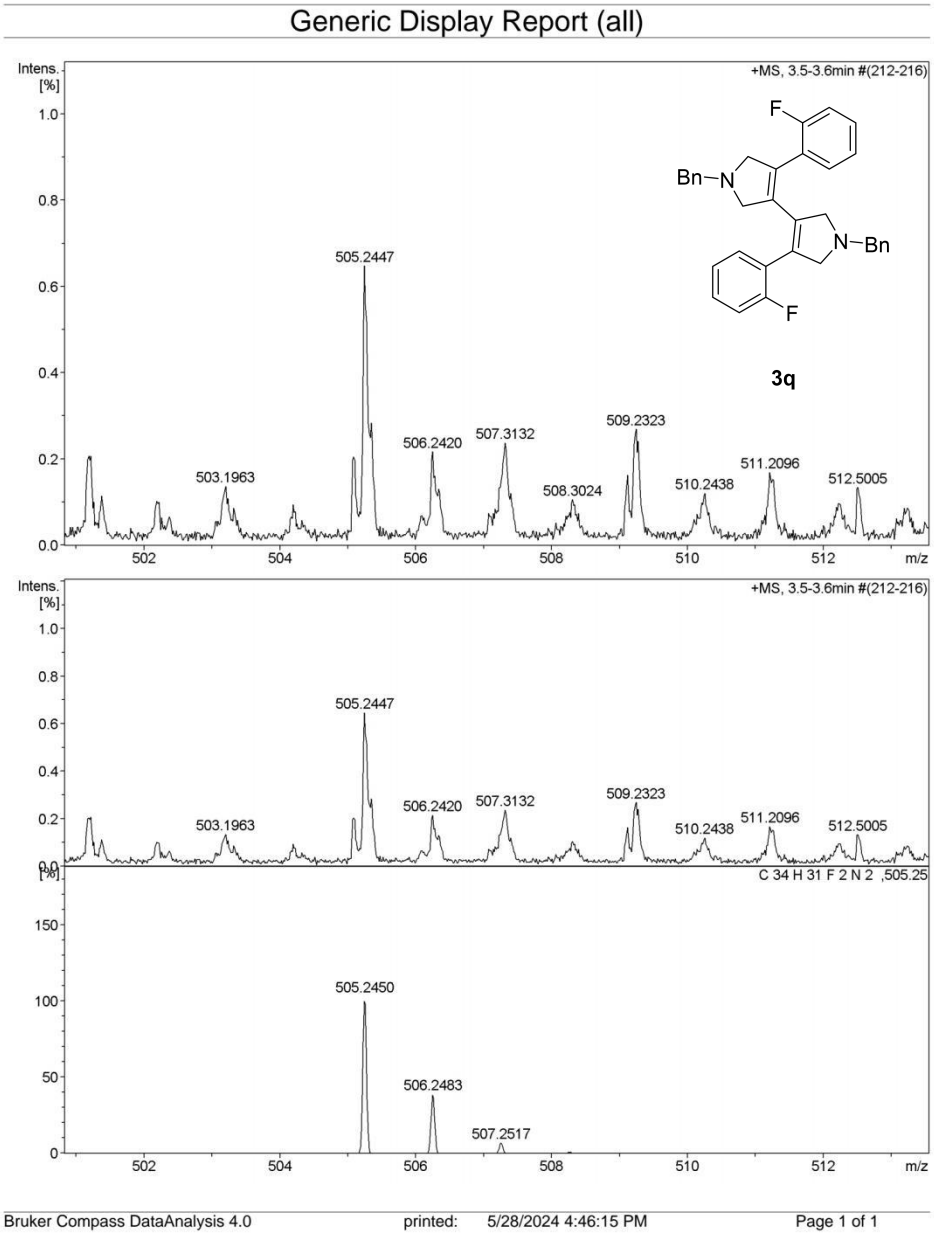
3o. HRMS (ESI) calcd for $C_{36}H_{37}N_2O_2^+$ *m/z* 529.2850 $[M+H]^+$, Found 529.2857.



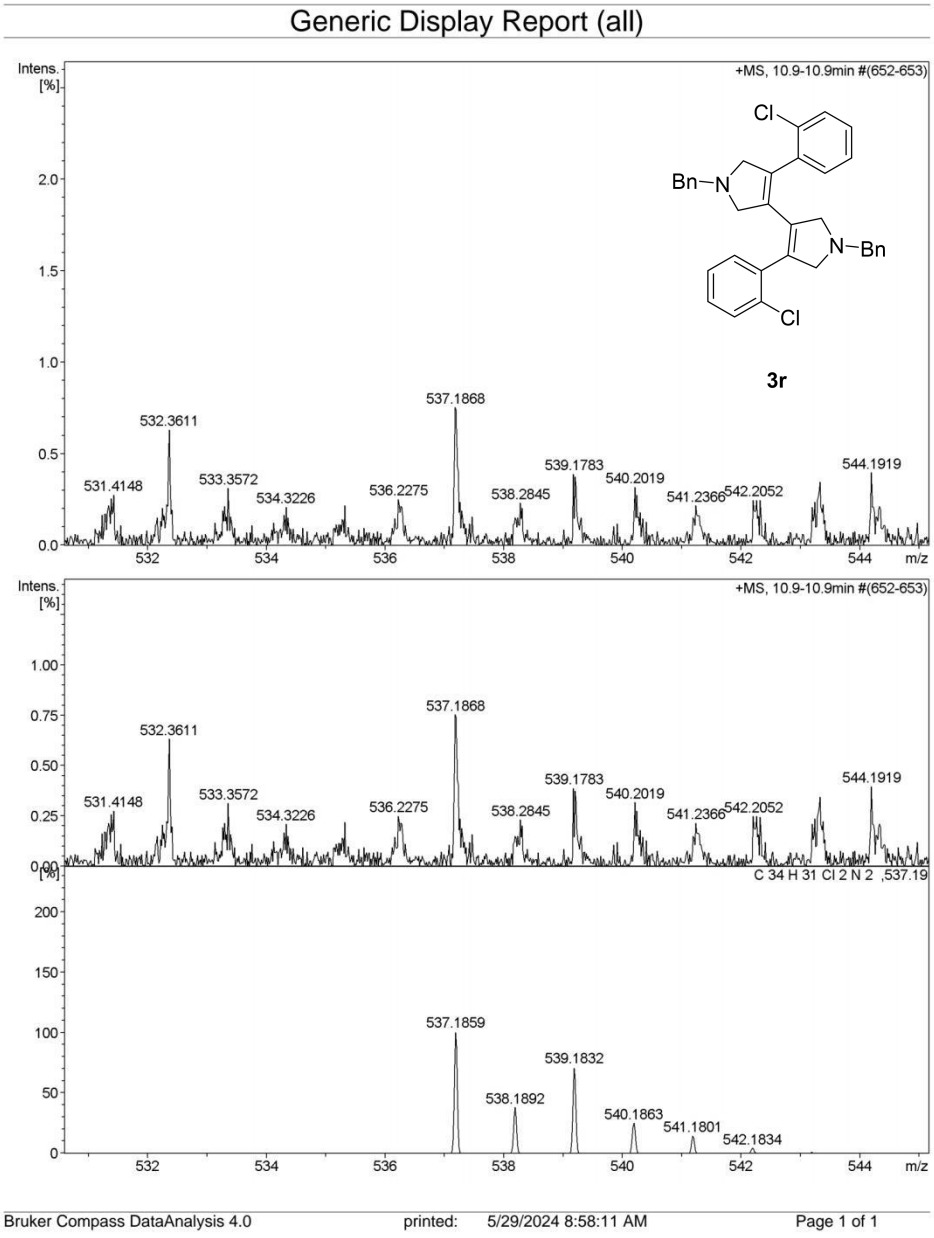
3p. HRMS (ESI) calcd for $C_{36}H_{37}N_2^+$ *m/z* 497.2951 $[M+H]^+$, Found 497.2942.



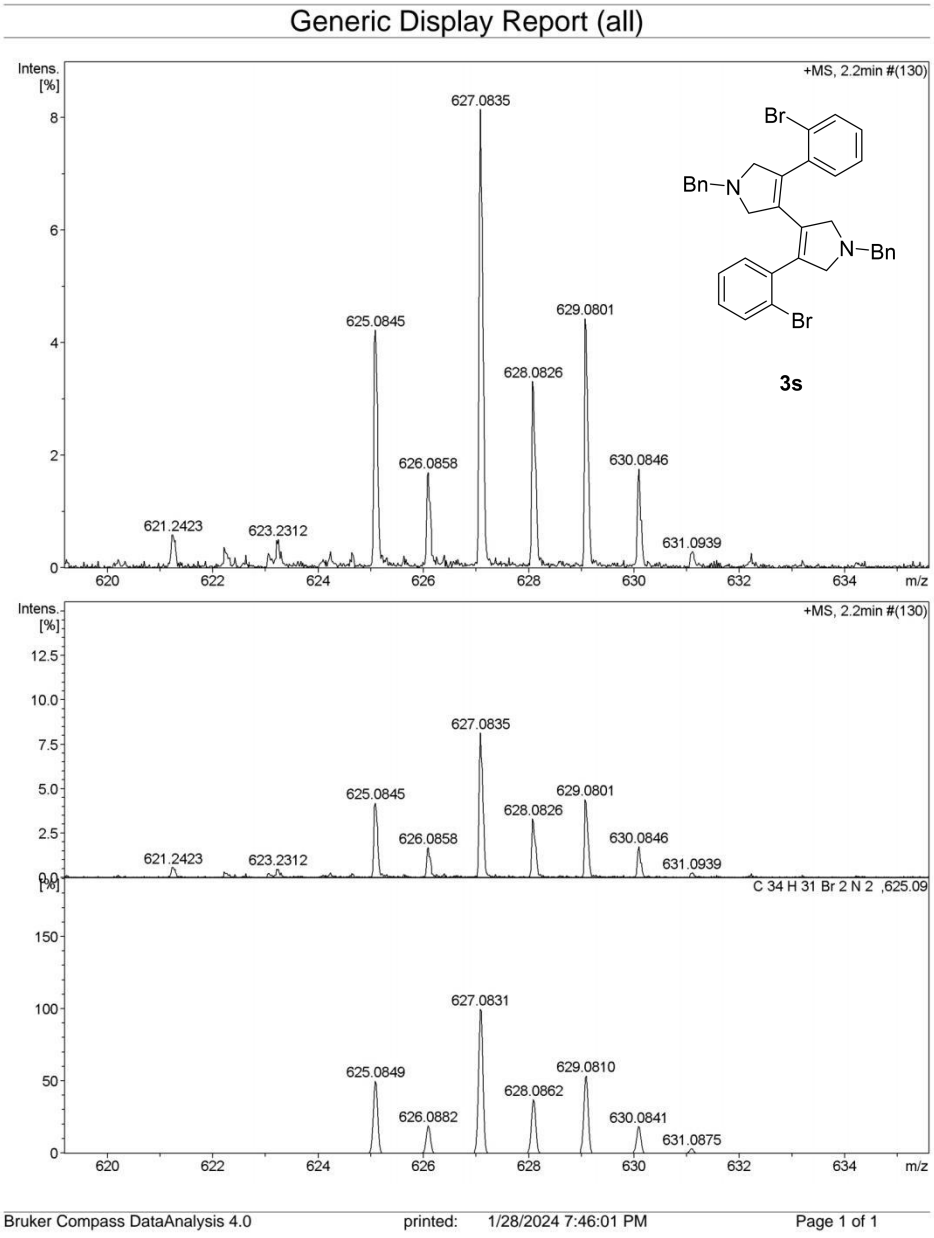
3q. HRMS (ESI) calcd for $C_{34}H_{31}F_2N_2^+$ *m/z* 505.2450 $[M+H]^+$, Found 505.2447.



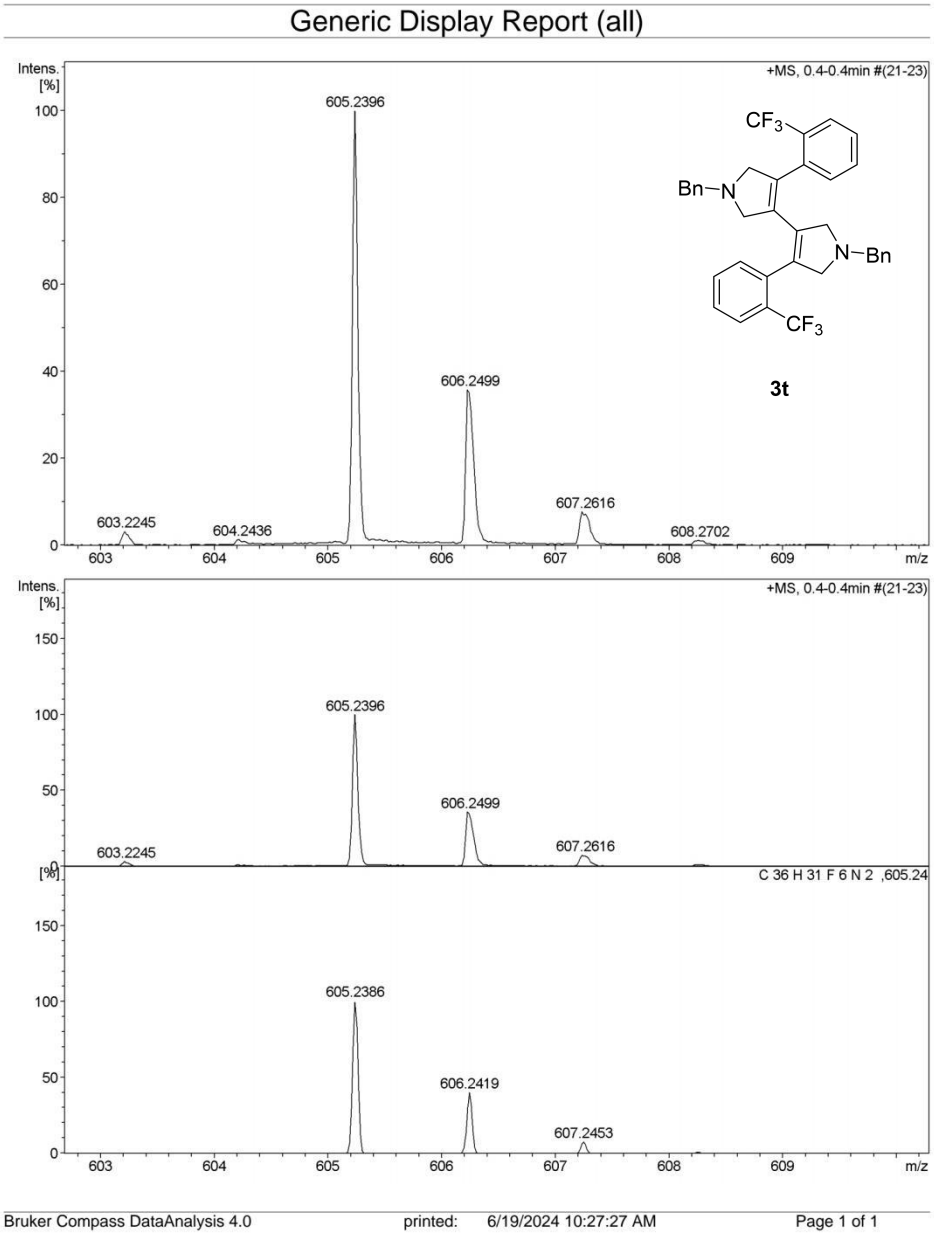
3r. HRMS (ESI) calcd for $C_{34}H_{31}Cl_2N_2^+$ *m/z* 537.1859 $[M+H]^+$, Found 537.1868.



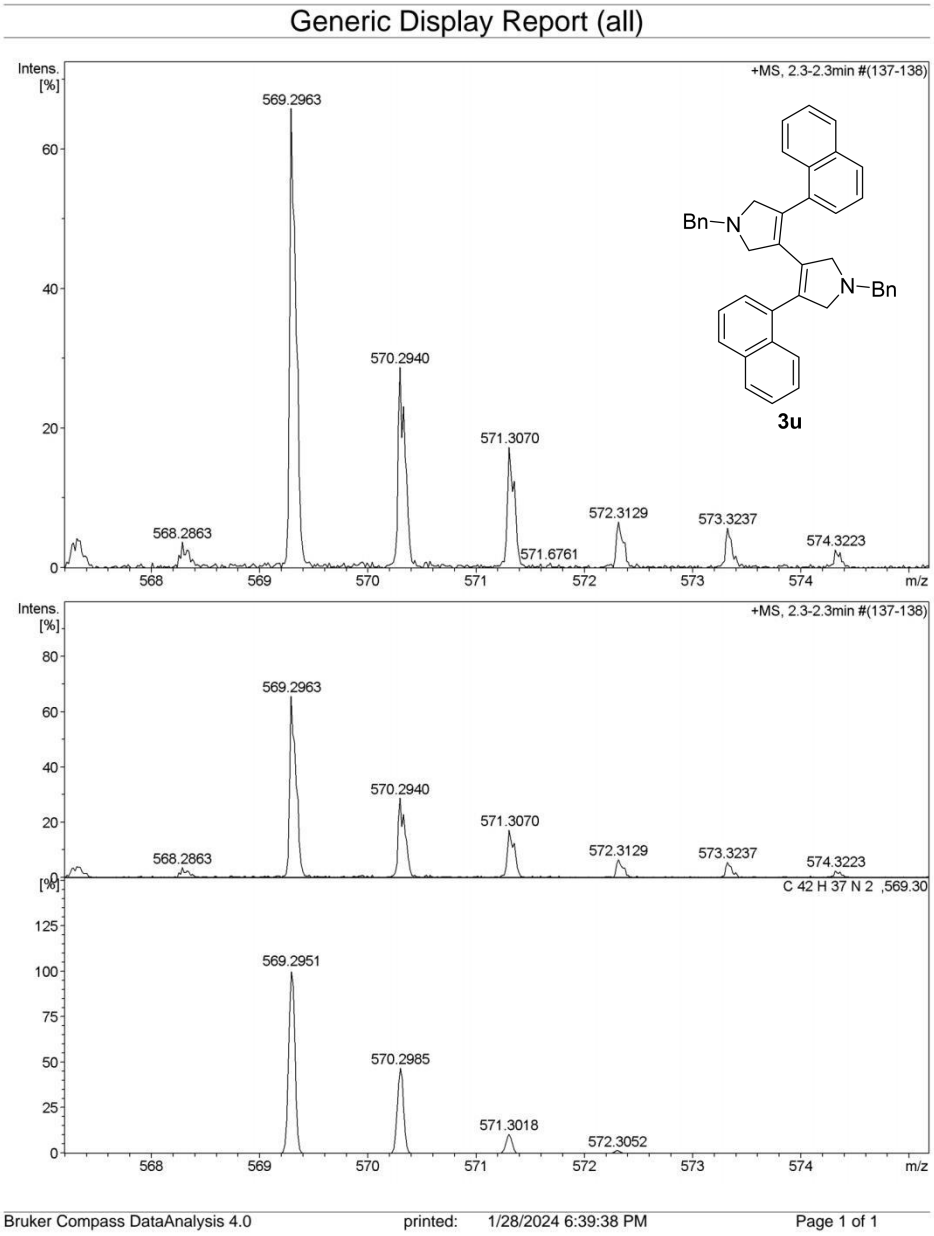
3s. HRMS (ESI) calcd for $C_{34}H_{31}Br_2N_2$ *m/z* 627.0831 $[M+H]^+$, Found 627.0835.



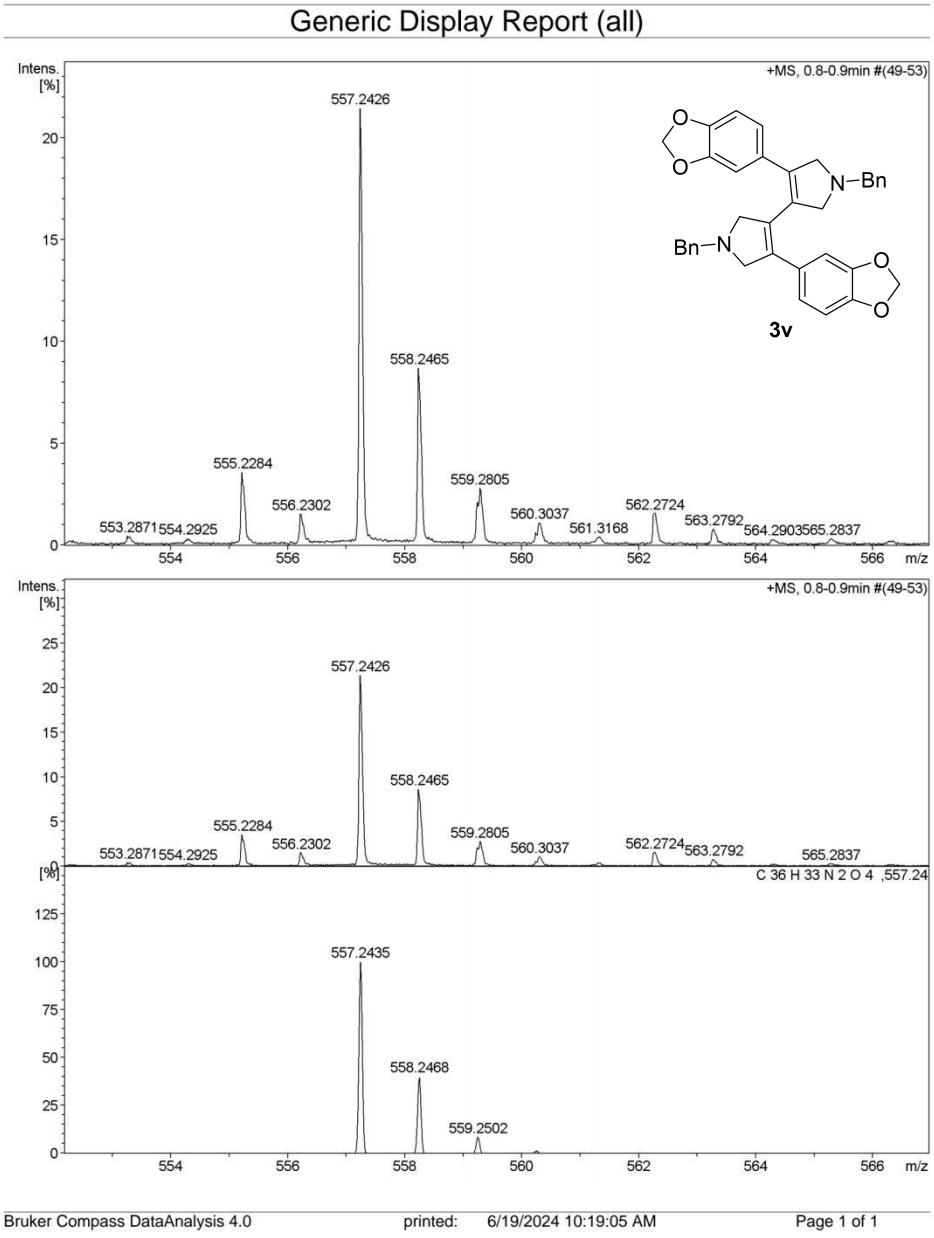
3t. HRMS (ESI) calcd for $C_{36}H_{31}F_6N_2^+$ *m/z* 605.2386 $[M+H]^+$, Found 605.2396.



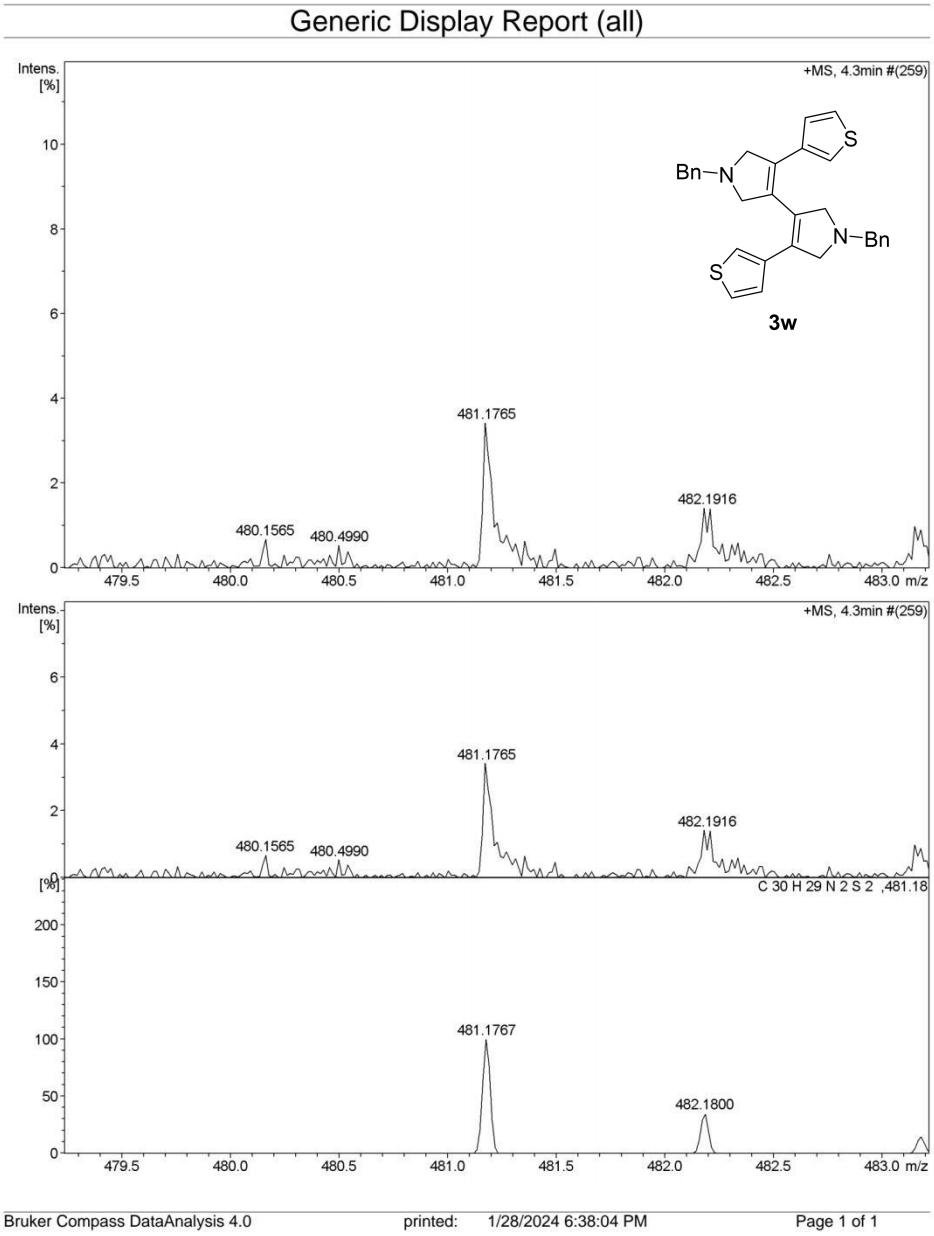
3u. HRMS (ESI) calcd for $C_{42}H_{37}N_2^+$ *m/z* 569.2951 $[M+H]^+$, Found 569.2963.



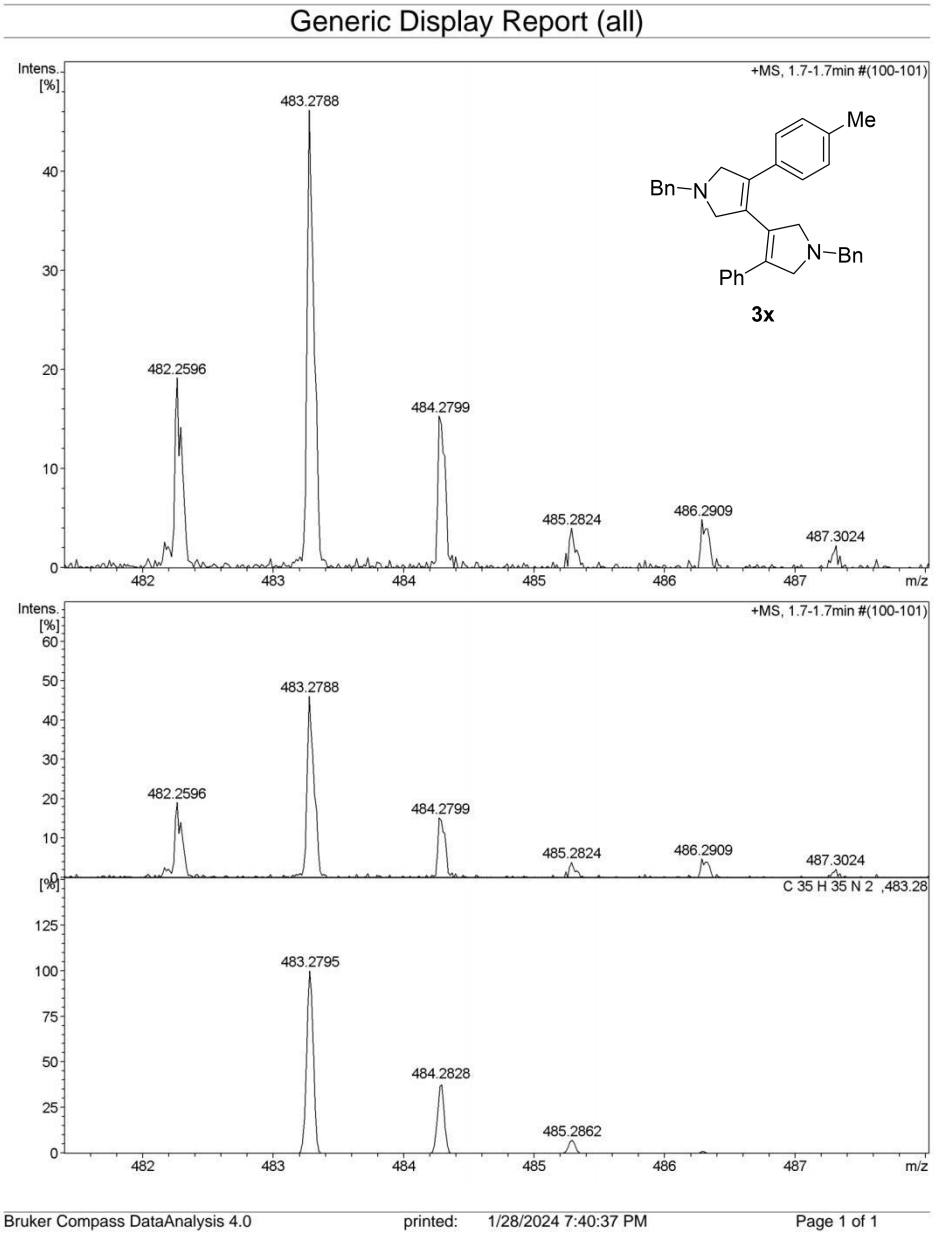
3v. HRMS (ESI) calcd for $C_{36}H_{33}N_2O_4^+$ *m/z* 557.2435 $[M+H]^+$, Found 557.2426.



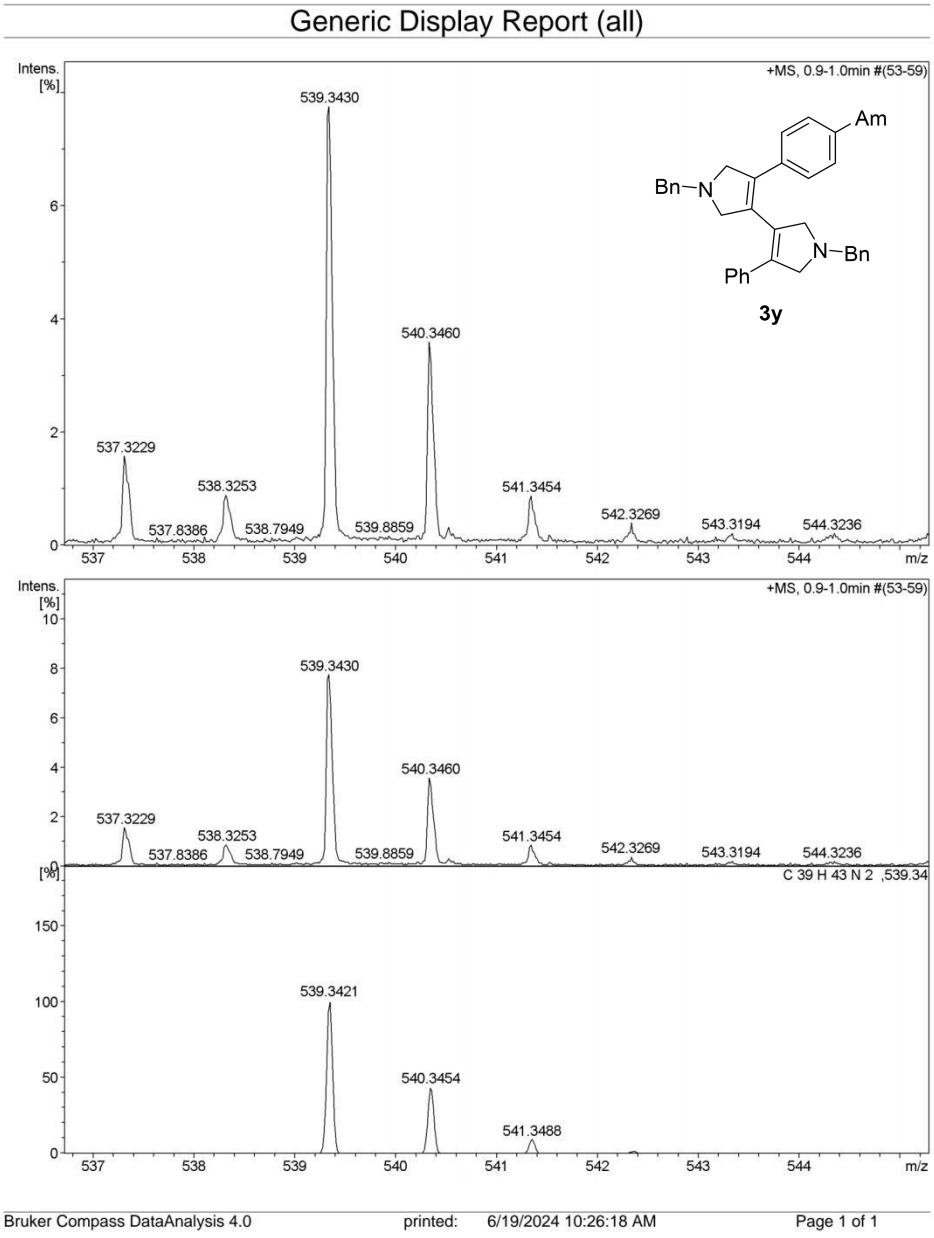
3w. HRMS (ESI) calcd for $C_{30}H_{29}N_2S_2^+$ *m/z* 481.1767 $[M+H]^+$, Found 481.1765.



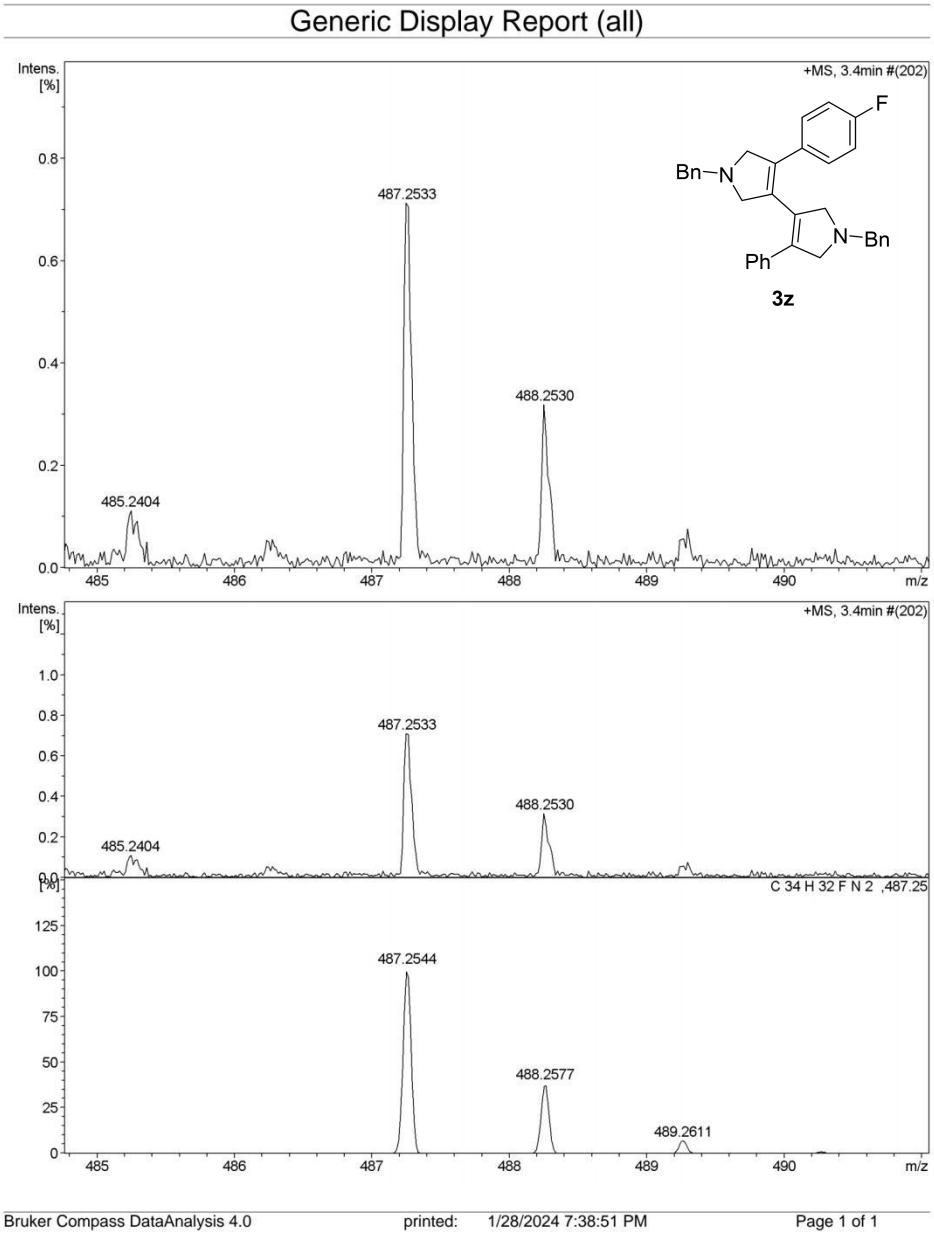
3x. HRMS (ESI) calcd for $C_{35}H_{35}N_2^+$ *m/z* 483.2795 $[M+H]^+$, Found 483.2788.



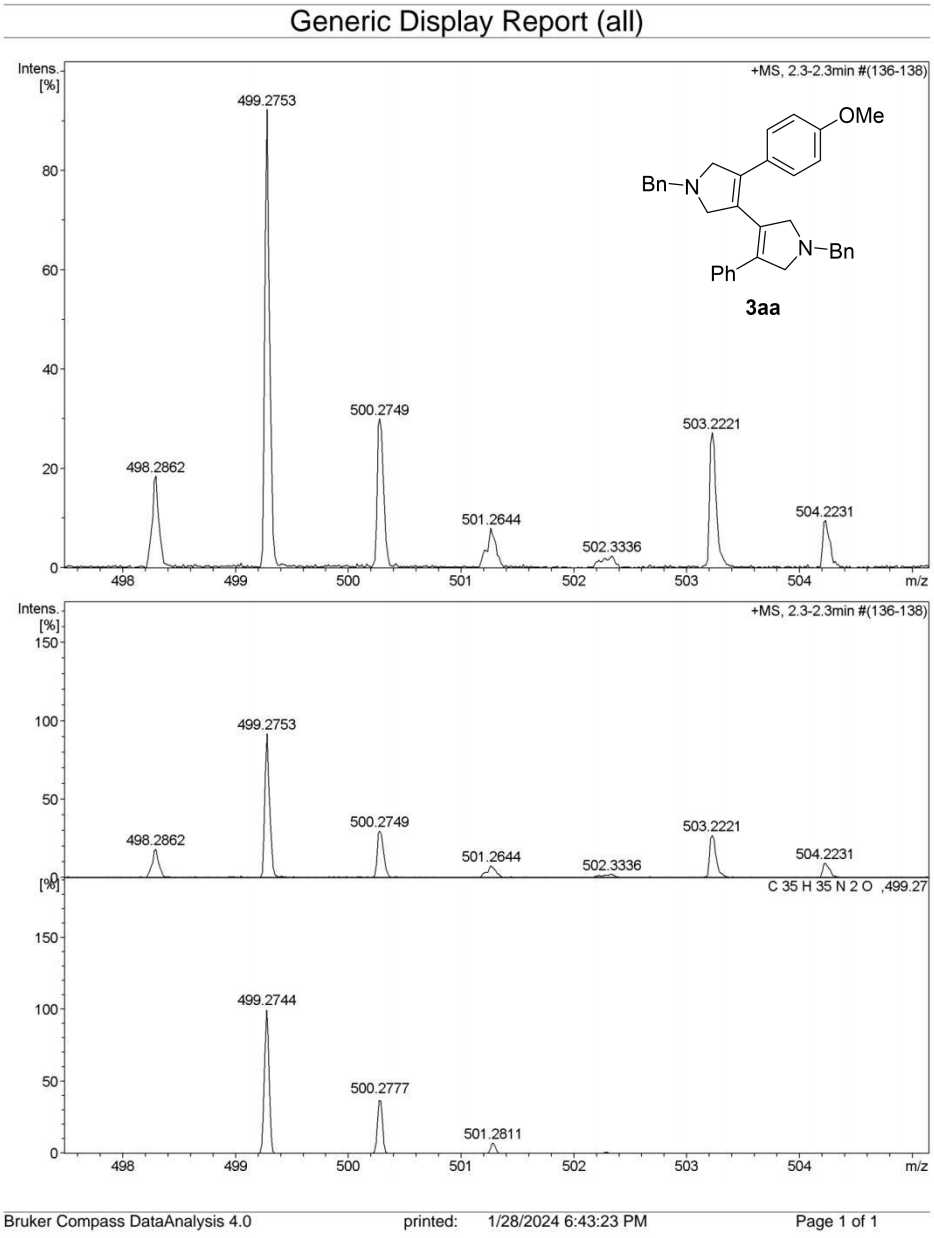
3y. HRMS (ESI) calcd for $C_{39}H_{43}N_2^+$ *m/z* 539.3421 $[M+H]^+$, Found 539.3430.



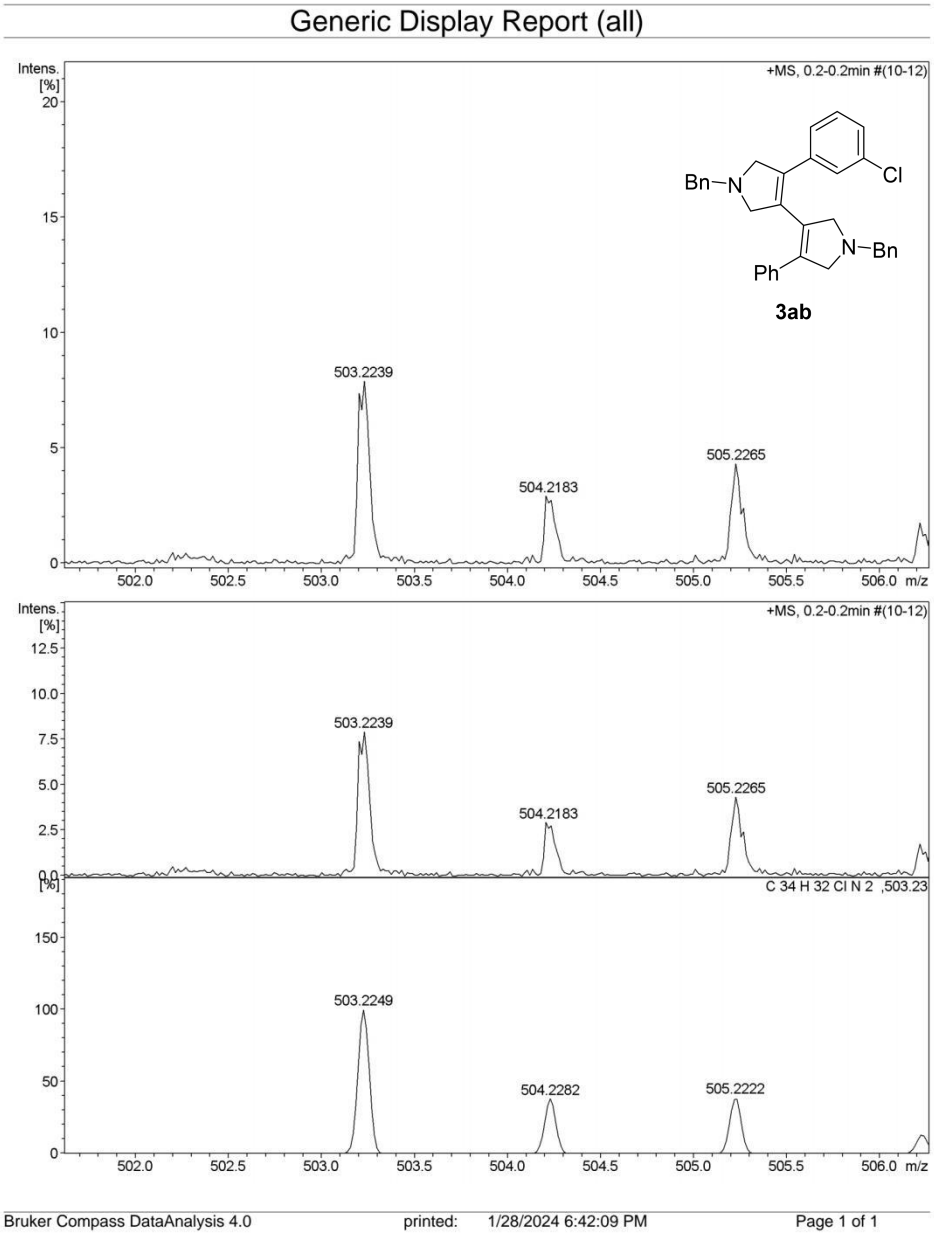
3z. HRMS (ESI) calcd for $C_{34}H_{32}FN_2^+$ *m/z* 487.2544 $[M+H]^+$, Found 487.2533.



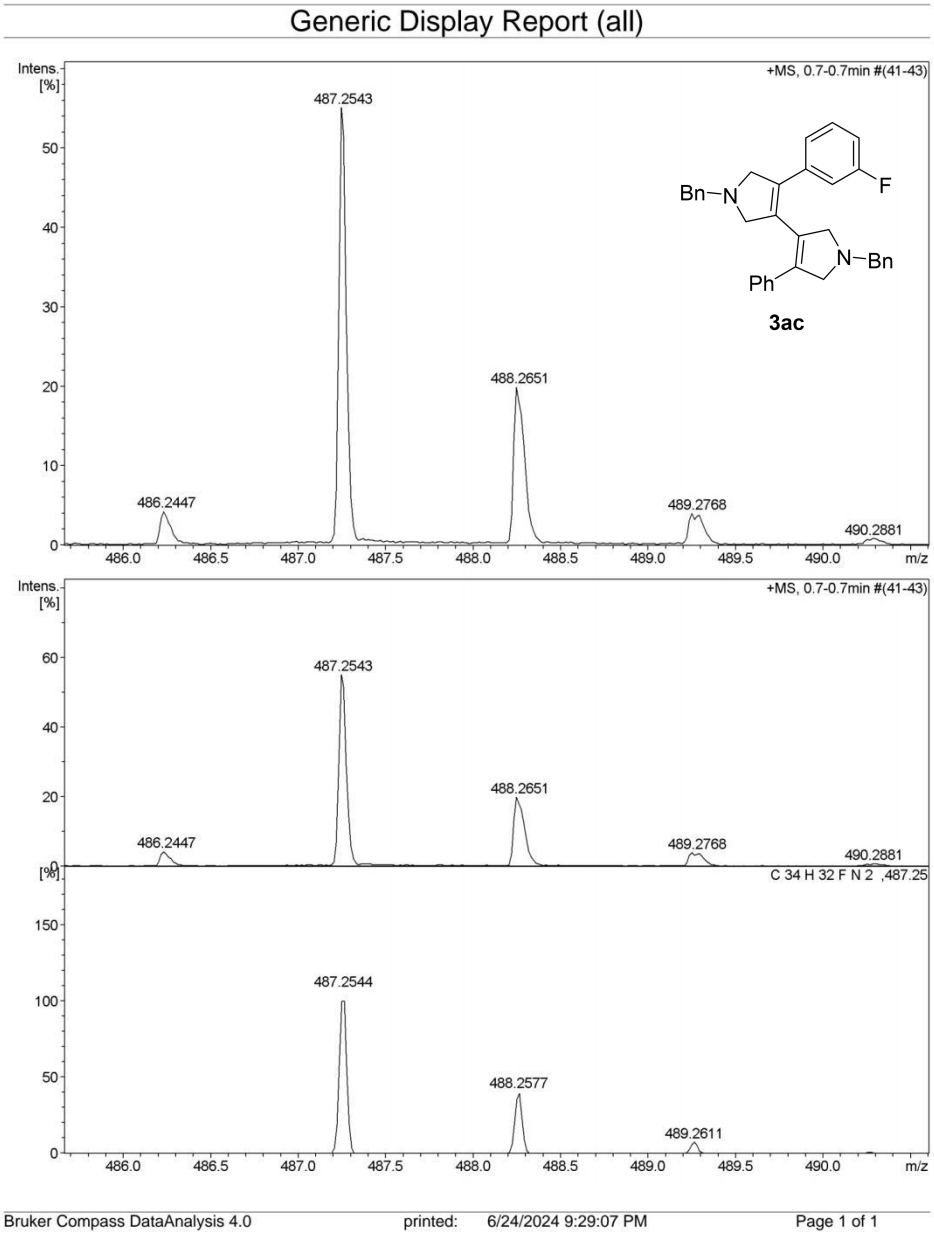
3aa. HRMS (ESI) calcd for $C_{35}H_{35}N_2O^+$ *m/z* 499.2744 $[M+H]^+$, Found 499.2753.



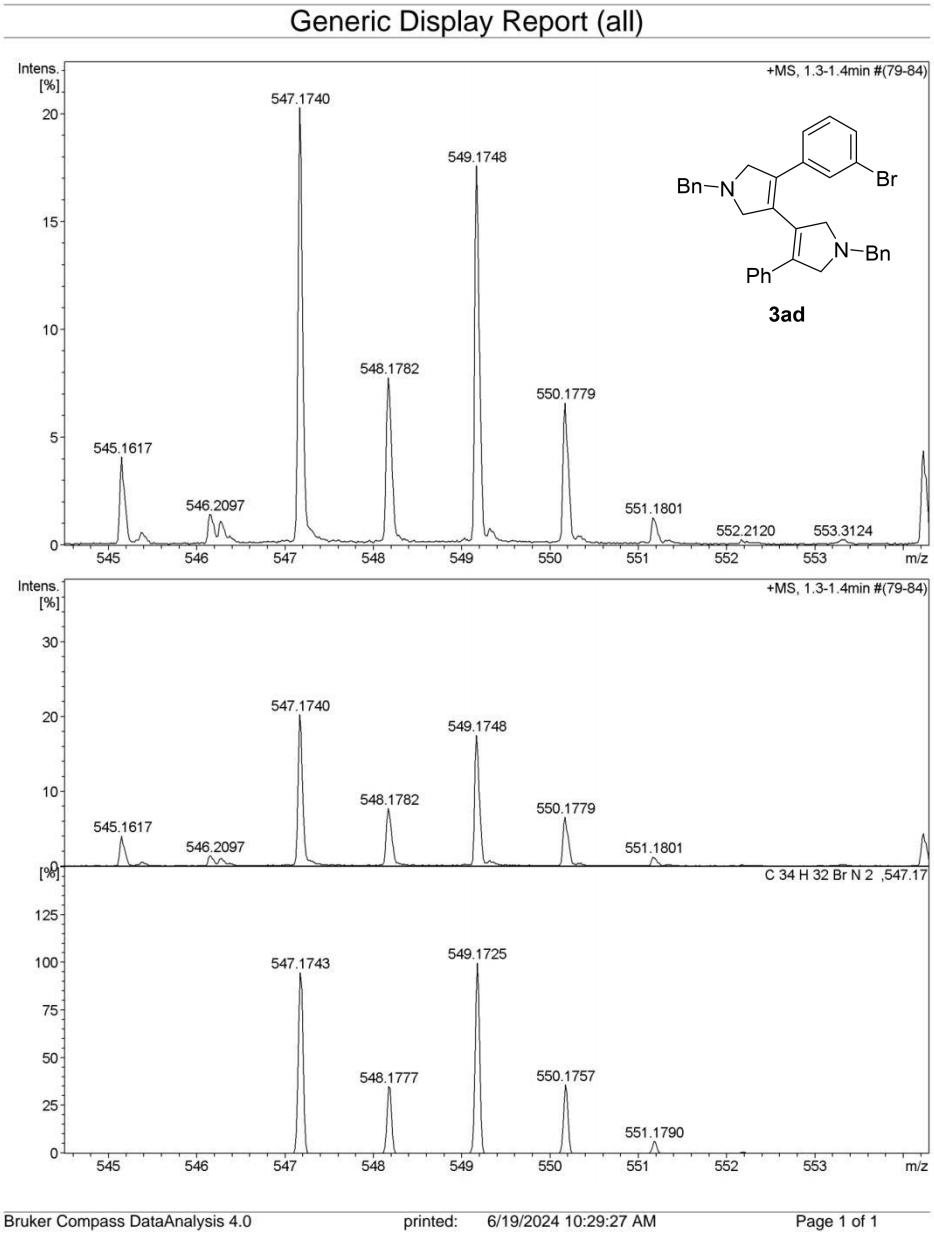
3ab. HRMS (ESI) calcd for $C_{34}H_{32}ClN_2^+$ *m/z* 503.2249 $[M+H]^+$, Found 503.2239.



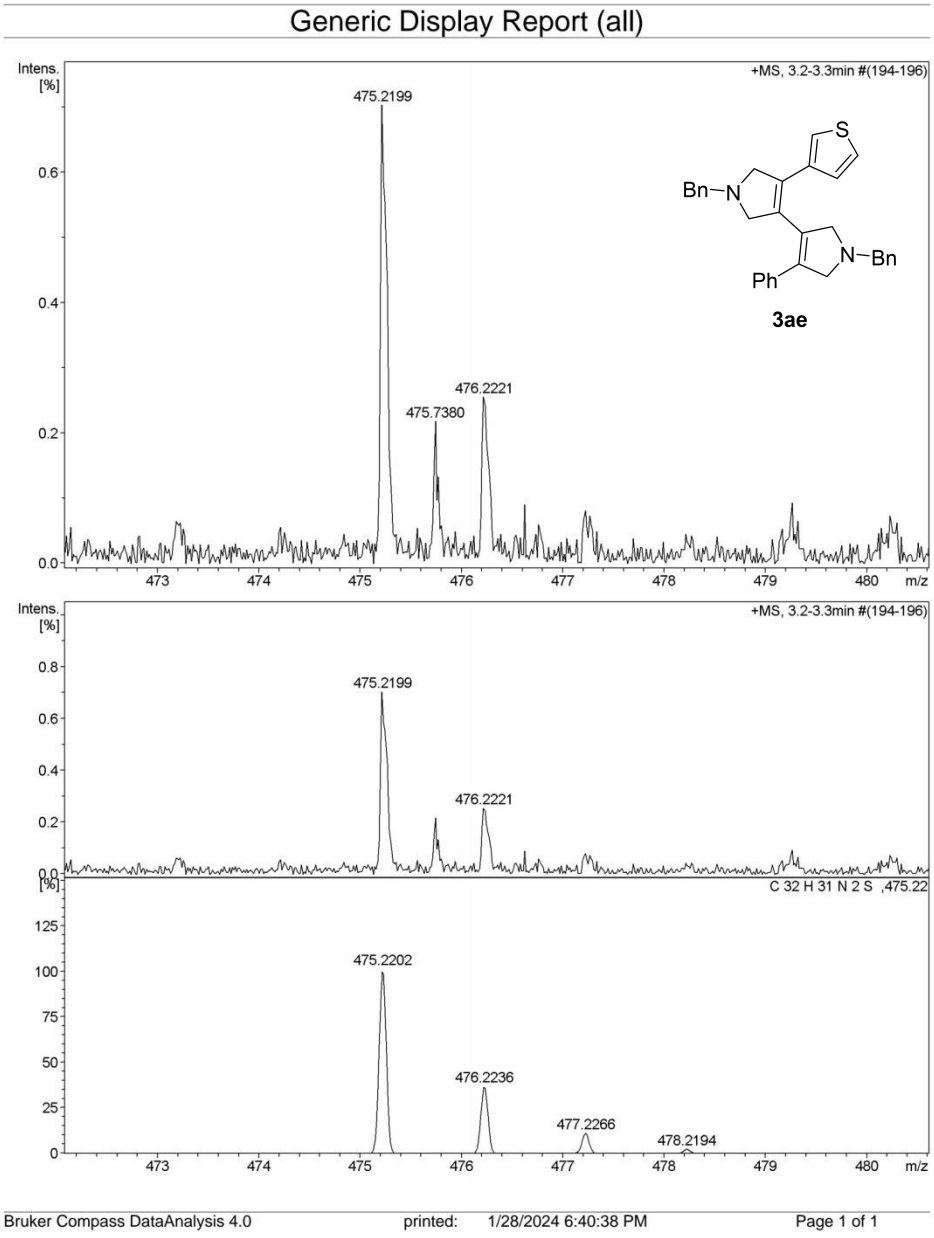
3ac. HRMS (ESI) calcd for $C_{34}H_{32}FN_2^+$ *m/z* 487.2544 $[M+H]^+$, Found 487.2543.



3ad. HRMS (ESI) calcd for $C_{34}H_{32}BrN_2^+$ *m/z* 547.1743 $[M+H]^+$, Found 547.1740.



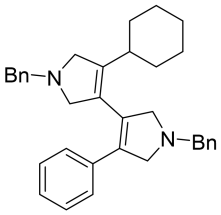
3ae. HRMS (ESI) calcd for $C_{32}H_{31}N_2S^+$ *m/z* 475.2202 $[M+H]^+$, Found 475.2199.



3af. HRMS (ESI) calcd for $C_{34}H_{39}N_2^+$ *m/z* 475.3113 $[M+H]^+$, Found 475.3115.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

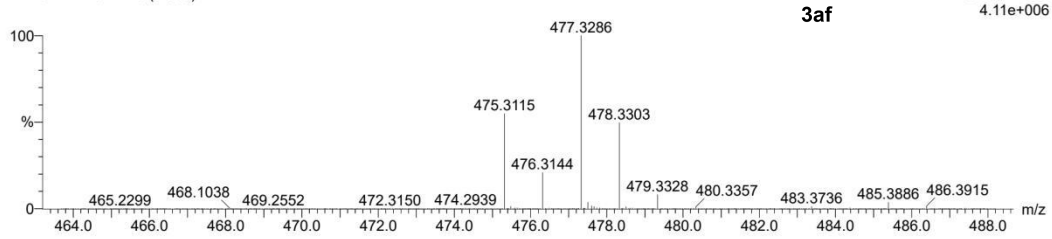


Monoisotopic Mass, Even Electron Ions
3220 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 39-39 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-461-1 10 (0.089)

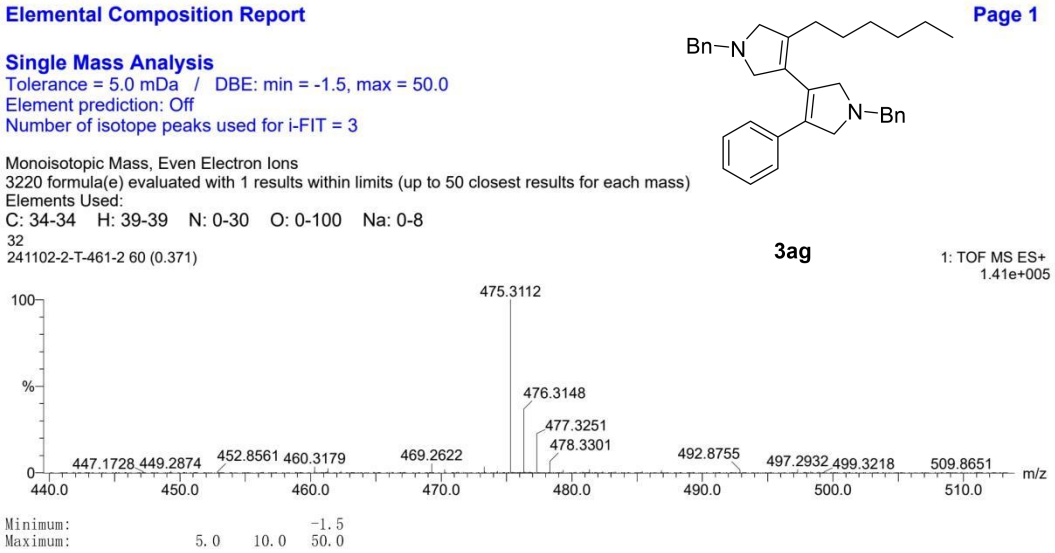
1: TOF MS ES+
4.11e+006

3af



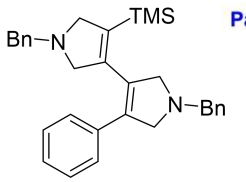
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
475.3115	475.3113	0.2	0.4	16.5	974.3	n/a	n/a	C34 H39 N2

3ag. HRMS (ESI) calcd for $C_{34}H_{41}N_2^+$ *m/z* 477.3264 $[M+H]^+$, Found 477.3251.



3ah. HRMS (ESI) calcd for $C_{31}H_{37}N_2Si^+$ *m/z* 465.2726 $[M+H]^+$, Found 465.2730.

Elemental Composition Report



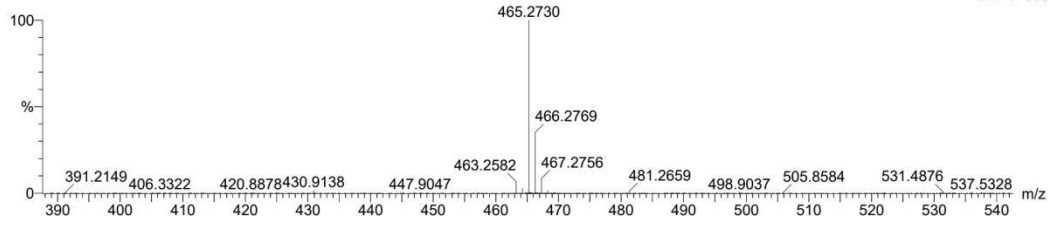
3ah

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
441 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 31-31 H: 37-37 N: 0-100 O: 0-100 Si: 1-1

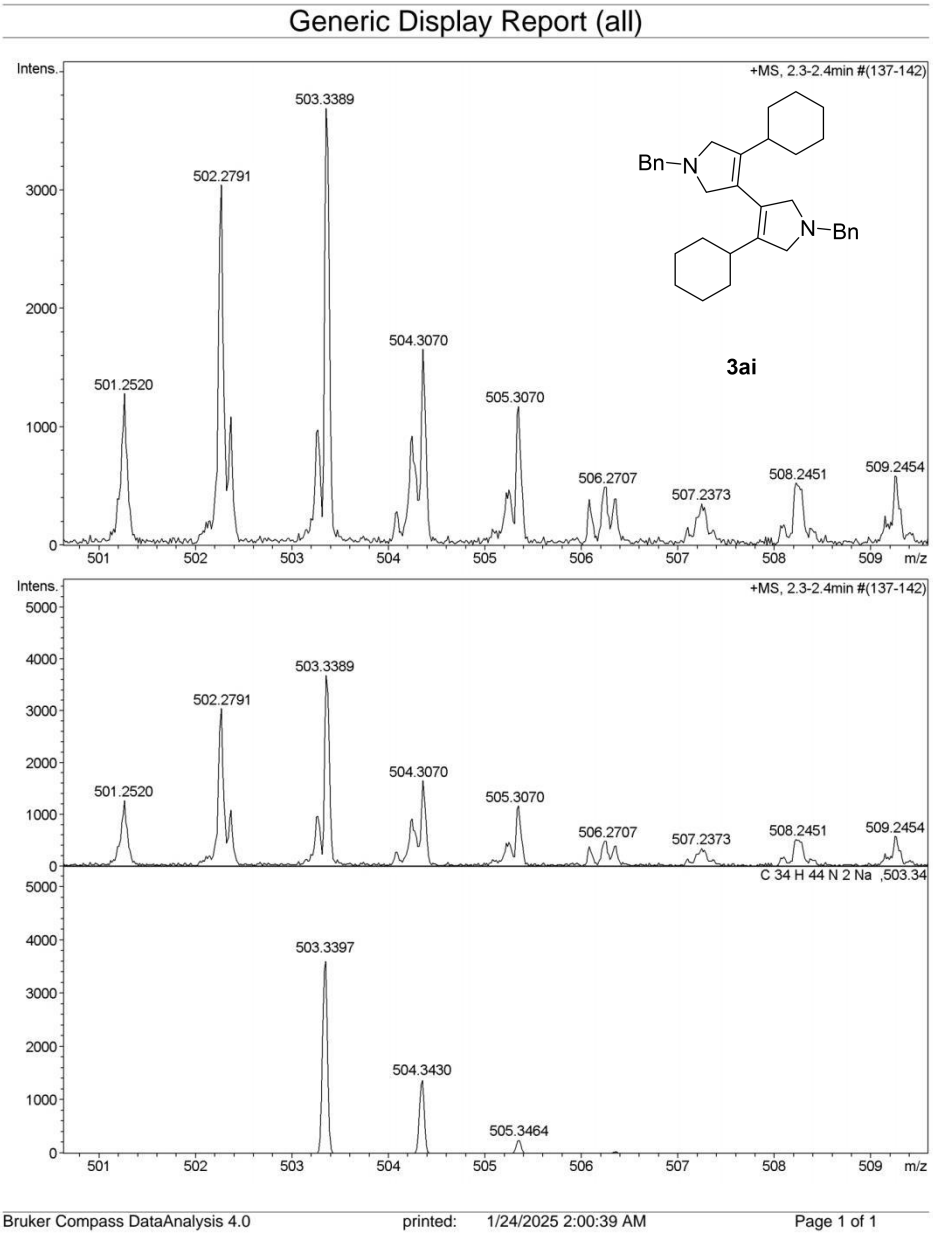
31
250103-3-T-494-1 257 (0.566)

1: TOF MS ES+
2.47e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
465.2730	465.2726	0.4	0.9	15.5	461.7	n/a	n/a	C31 H37 N2 Si

3ai. HRMS (ESI) calcd for $C_{34}H_{44}N_2Na^+$ *m/z* 503.3397 $[M+H]^+$, Found 503.3389.

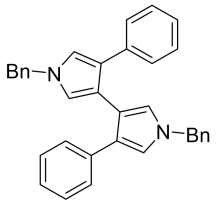


4a. HRMS (ESI) calcd for $C_{34}H_{29}N_2^+$ *m/z* 465.2331 $[M+H]^+$, Found 465.2322.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

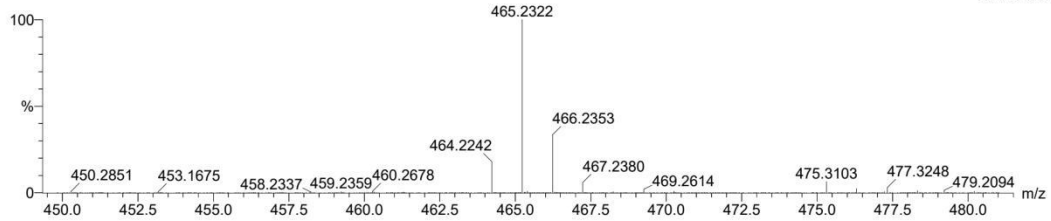
Monoisotopic Mass, Even Electron Ions
3074 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 29-29 N: 0-30 O: 0-100 Na: 0-8



4a

32
241102-2-T-472 20 (0.142)

1: TOF MS ES+
5.47e+005



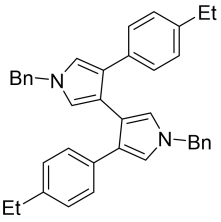
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
465.2322	465.2331	-0.9	-1.9	21.5	726.5	n/a	n/a	C34 H29 N2

4b. HRMS (ESI) calcd for $C_{38}H_{37}N_2^+$ *m/z* 521.2957 $[M+H]^+$, Found 521.2950.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

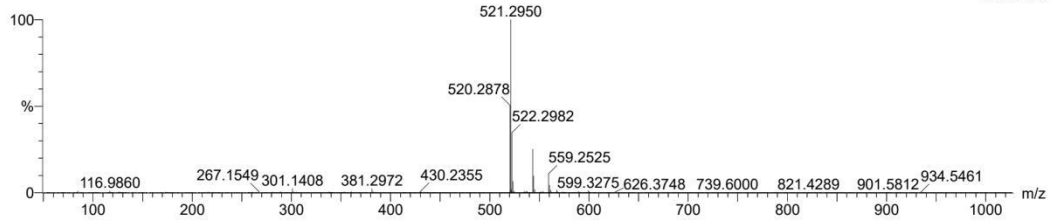
Monoisotopic Mass, Even Electron Ions
3962 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 38-38 H: 37-37 N: 0-30 O: 0-100 Na: 0-8



4b

32
241102-2-T-233-6 24 (0.162)

1: TOF MS ES+
1.32e+006



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
521.2950	521.2957	-0.7	-1.3	21.5	796.6	n/a	n/a	C38 H37 N2

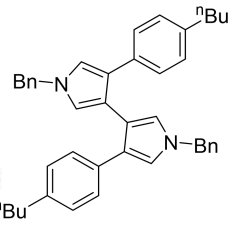
4c. HRMS (ESI) calcd for $C_{42}H_{45}N_2^+$ *m/z* 577.3583 $[M+H]^+$, Found 577.3580.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

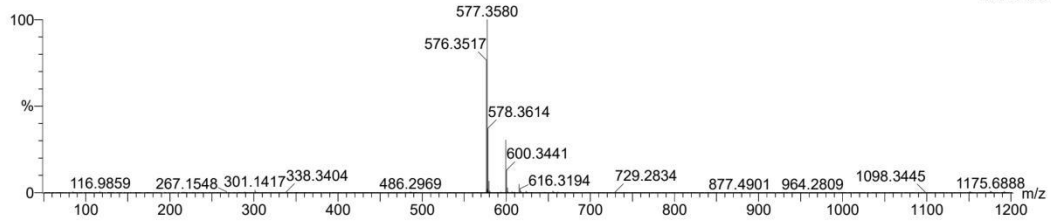
Monoisotopic Mass, Even Electron Ions
4907 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 42-42 H: 45-45 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-415-3 35 (0.240)



4c

1: TOF MS ES+
1.87e+006



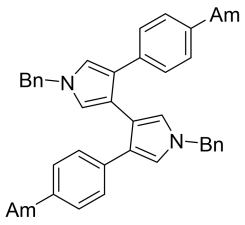
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
577.3580	577.3583	-0.3	-0.5	21.5	739.1	n/a	n/a	C42 H45 N2

4d. HRMS (ESI) calcd for $C_{44}H_{49}N_2^+$ *m/z* 605.3896 $[M+H]^+$, Found 605.3898.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

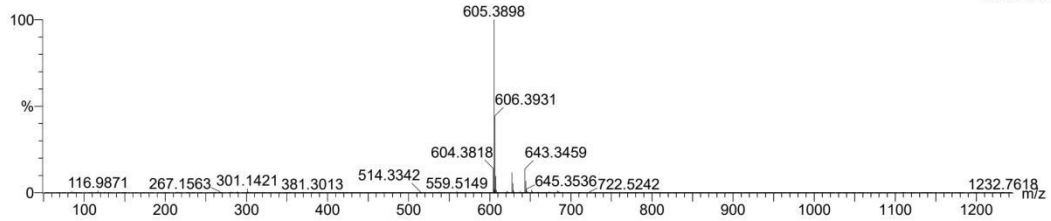
Monoisotopic Mass, Even Electron Ions
5390 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 44-44 H: 49-49 N: 0-30 O: 0-100 Na: 0-8



4d

32
241102-2-T-415-4 57 (0.355)

1: TOF MS ES+
1.56e+006



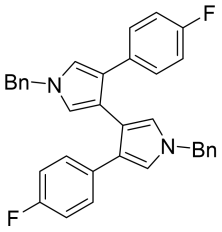
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
605.3898	605.3896	0.2	0.3	21.5	761.6	n/a	n/a	C44 H49 N2

4e. HRMS (ESI) calcd for $C_{34}H_{27}N_2F_2^+$ *m/z* 501.2142 $[M+H]^+$, Found 501.2133.

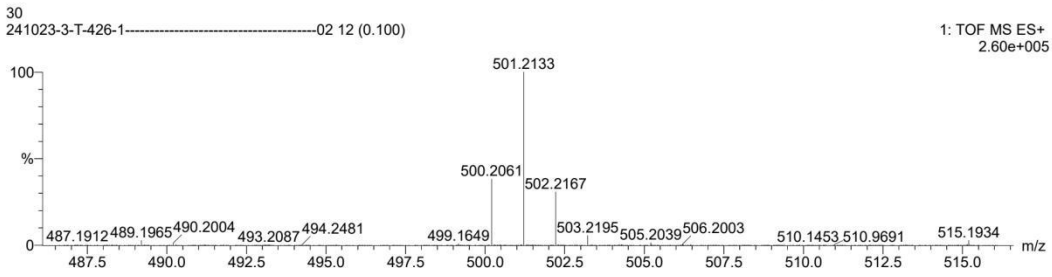
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
9139 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 27-27 N: 0-30 O: 0-100 Na: 0-8 F: 1-3



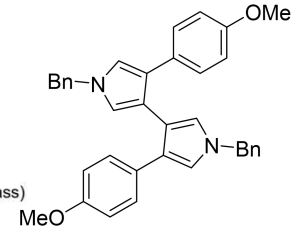
4e



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
501.2133	501.2142	-0.9	-1.8	21.5	573.8	n/a	n/a	C34 H27 N2 F2

4f. HRMS (ESI) calcd for $C_{36}H_{33}N_2O_2^+$ *m/z* 525.2542 $[M+H]^+$, Found 525.2538.

Elemental Composition Report **Page 1**



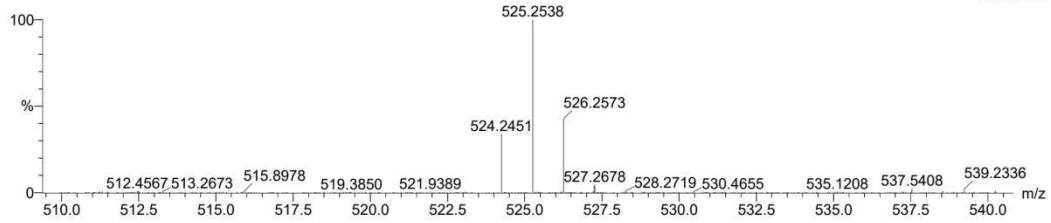
4f

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3997 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 36-36 H: 33-33 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-467-1 31 (0.219)

1: TOF MS ES+
7.58e+004



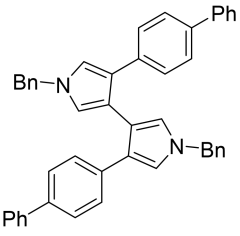
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
525.2538	525.2542	-0.4	-0.8	21.5	466.5	n/a	n/a	C36 H33 N2 O2

4g. HRMS (ESI) calcd for $C_{46}H_{37}N_2^+$ *m/z* 617.2957 $[M+H]^+$, Found 617.2952.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

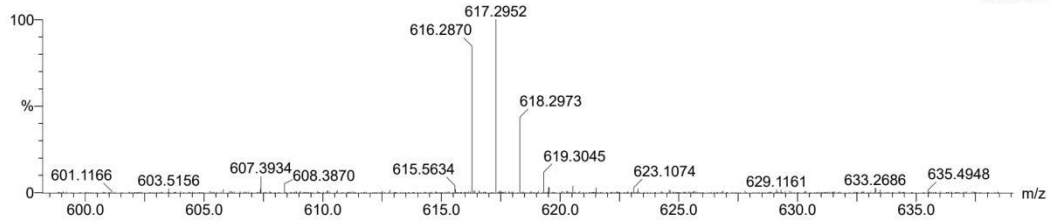
Monoisotopic Mass, Even Electron Ions
5611 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 46-46 H: 37-37 N: 0-30 O: 0-100 Na: 0-8



4g

30
241023-3-T-446-1----------02 85 (0.522)

1: TOF MS ES+
1.80e+004



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
617.2952	617.2957	-0.5	-0.8	29.5	290.6	n/a	n/a	C46 H37 N2

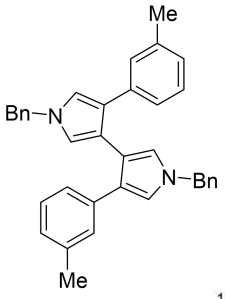
4h. HRMS (ESI) calcd for $C_{36}H_{33}N_2^+$ *m/z* 493.2644 $[M+H]^+$, Found 493.2635.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

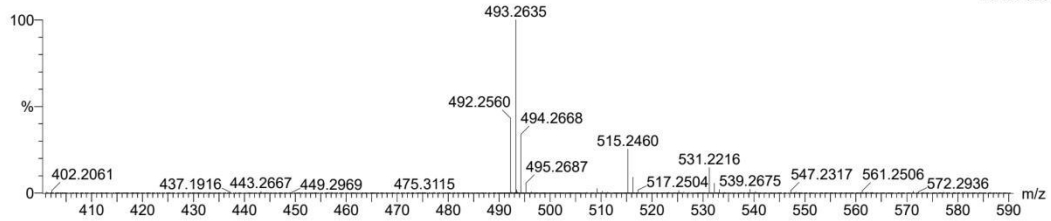
Monoisotopic Mass, Even Electron Ions
3510 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 36-36 H: 33-33 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-467-2 20 (0.142)



4h

1: TOF MS ES+
1.24e+006



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
493.2635	493.2644	-0.9	-1.8	21.5	722.2	n/a	n/a	C36 H33 N2

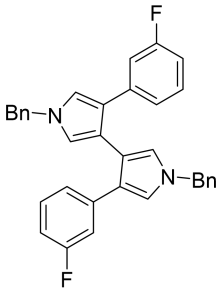
4i. HRMS (ESI) calcd for $C_{34}H_{27}F_2N_2^+$ *m/z* 501.2142 $[M+H]^+$, Found 501.2134.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

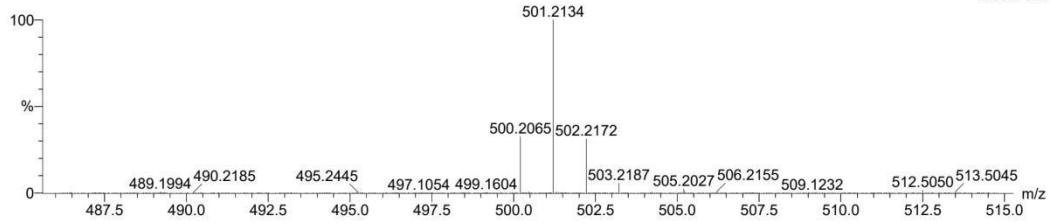
Monoisotopic Mass, Even Electron Ions
9139 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 27-27 N: 0-30 O: 0-100 F: 1-3 Na: 0-8

30
241023-3-T-426-2----------02 12 (0.100)



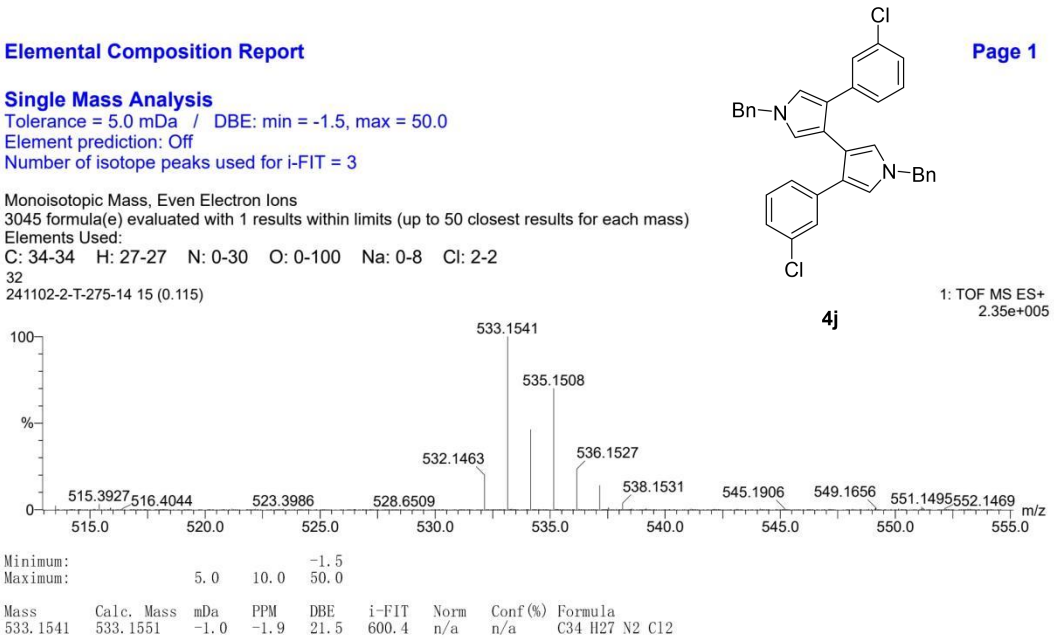
4i

1: TOF MS ES+
3.53e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
501.2134	501.2142	-0.8	-1.6	21.5	590.9	n/a	n/a	C34 H27 N2 F2

4j. HRMS (ESI) calcd for $C_{34}H_{27}Cl_2N_2^+$ *m/z* 533.1551 $[M+H]^+$, Found 533.1541.

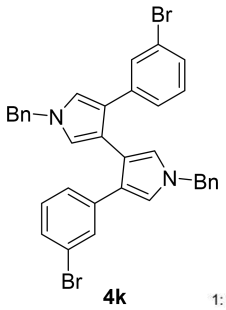


4k. HRMS (ESI) calcd for $C_{34}H_{27}Br_2N_2^+$ *m/z* 621.0541 $[M+H]^+$, Found 621.0541.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

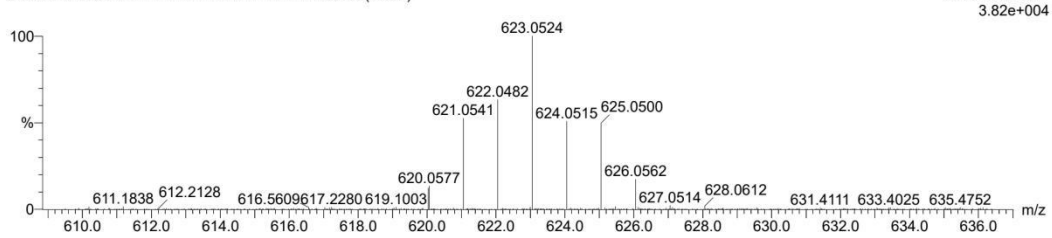
Monoisotopic Mass, Even Electron Ions
10336 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 27-27 N: 0-30 O: 0-100 Na: 0-8 Br: 1-4



4k

30
241023-3-T-424----------02 20 (0.142)

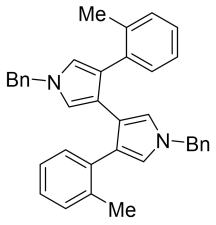
1: TOF MS ES+
3.82e+004



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
621.0541	621.0541	0.0	0.0	21.5	418.4	n/a	n/a	C34 H27 N2 Br2

4l. HRMS (ESI) calcd for $C_{36}H_{33}N_2^+$ *m/z* 493.2644 $[M+H]^+$, Found 493.2635.

Elemental Composition Report **Page 1**



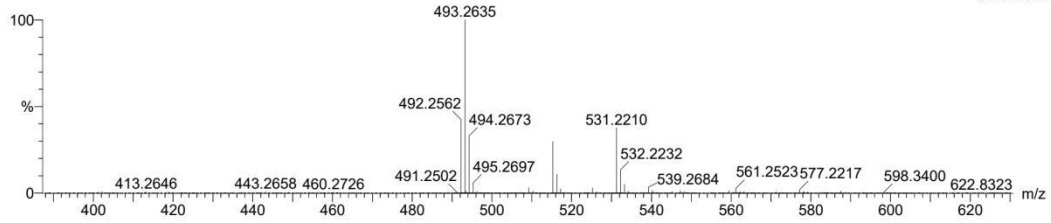
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3510 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 36-36 H: 33-33 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-275-3 24 (0.162)

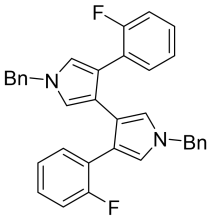
4l

1: TOF MS ES+
6.23e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
493.2635	493.2644	-0.9	-1.8	21.5	708.3	n/a	n/a	C36 H33 N2

4m. HRMS (ESI) calcd for $C_{34}H_{27}F_2N_2^+$ *m/z* 501.2142 $[M+H]^+$, Found 501.2139.



4m

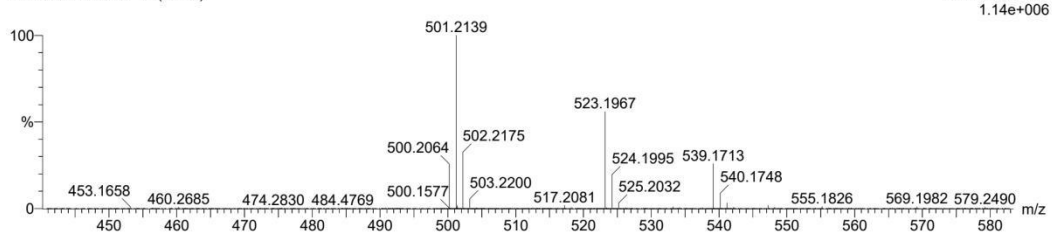
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
9139 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 27-27 N: 0-30 O: 0-100 Na: 0-8 F: 1-3

32
241102-2-T-275-12 12 (0.100)

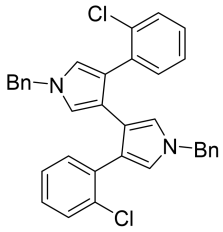
1: TOF MS ES+
1.14e+006



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
501.2139	501.2142	-0.3	-0.6	21.5	779.2	n/a	n/a	C34 H27 N2 F2

4n. HRMS (ESI) calcd for $C_{34}H_{27}Cl_2N_2^+$ *m/z* 533.1551 $[M+H]^+$, Found 533.1546.

Elemental Composition Report **Page 1**

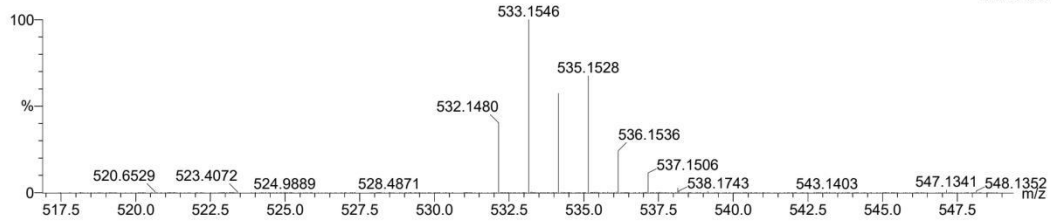


Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
11232 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 27-27 N: 0-30 O: 0-100 Na: 0-8 Cl: 1-4

30
241023-3-T-433-1----------02 17 (0.126)

4n 1: TOF MS ES+
1.77e+005



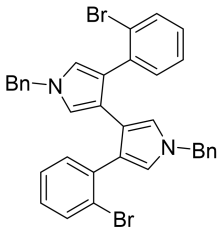
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
533.1546	533.1551	-0.5	-0.9	21.5	564.3	n/a	n/a	C34 H27 N2 Cl2

4o. HRMS (ESI) calcd for $C_{34}H_{27}Br_2N_2^+$ *m/z* 621.0541 $[M+H]^+$, Found 621.0545.

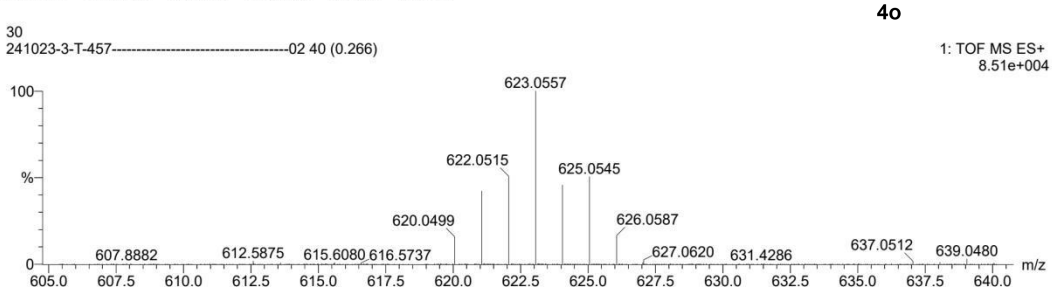
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
10336 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 27-27 N: 0-30 O: 0-100 Na: 0-8 Br: 1-4



4o



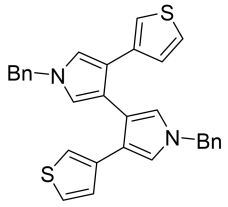
Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
621.0545	621.0541	0.4	0.6	21.5	397.6	n/a	n/a	C34 H27 N2 Br2

4p. HRMS (ESI) calcd for $C_{30}H_{25}S_2N_2^+$ *m/z* 477.1459 $[M+H]^+$, Found 477.1452.

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

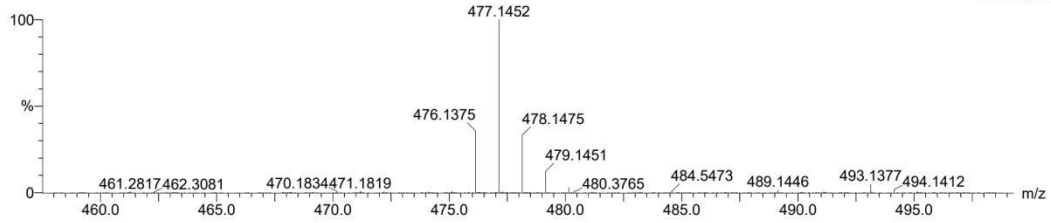
Monoisotopic Mass, Even Electron Ions
5073 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 30-30 H: 25-25 N: 0-30 O: 0-100 Na: 0-8 S: 1-2



4p

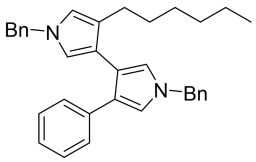
30
241023-3-T-446-3----------02 32 (0.224)

1: TOF MS ES+
1.74e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
477.1452	477.1459	-0.7	-1.5	19.5	431.2	n/a	n/a	C30 H25 N2 S2

4q. HRMS (ESI) calcd for $C_{34}H_{37}N_2^+$ *m/z* 473.2957 $[M+H]^+$, Found 473.2951.



4q

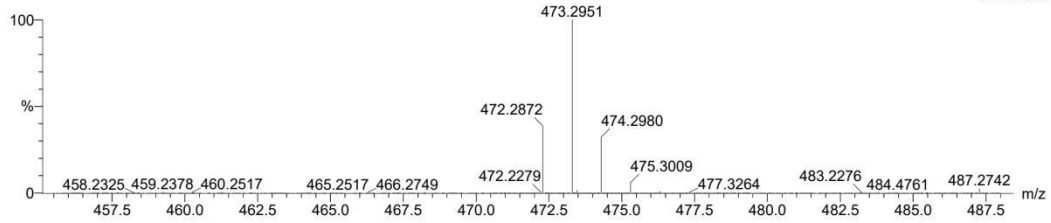
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3189 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 37-37 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-474 21 (0.147)

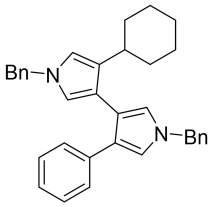
1: TOF MS ES+
8.12e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
473.2951	473.2957	-0.6	-1.3	17.5	821.3	n/a	n/a	C34 H37 N2

4r. HRMS (ESI) calcd for $C_{34}H_{35}N_2^+$ *m/z* 471.2800 $[M+H]^+$, Found 471.2792.

Elemental Composition Report **Page 1**

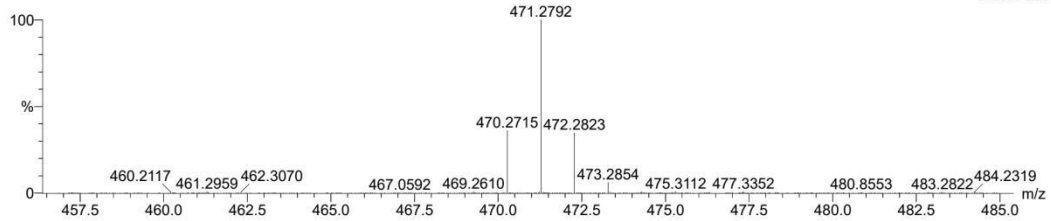


4r

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3161 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 35-35 N: 0-30 O: 0-100 Na: 0-8
32
241102-2-T-473 31 (0.219)

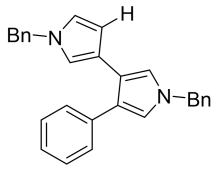
1: TOF MS ES+
2.08e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
471.2792	471.2800	-0.8	-1.7	18.5	529.4	n/a	n/a	C34 H35 N2

4s. HRMS (ESI) calcd for $C_{28}H_{25}N_2^+$ m/z 389.2018 $[M+H]^+$, Found 389.2015.

Elemental Composition Report **Page 1**

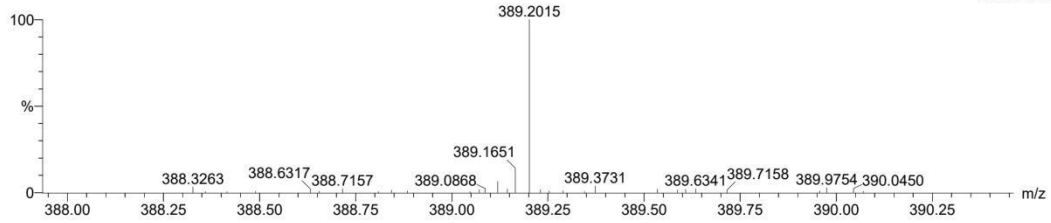


4s

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

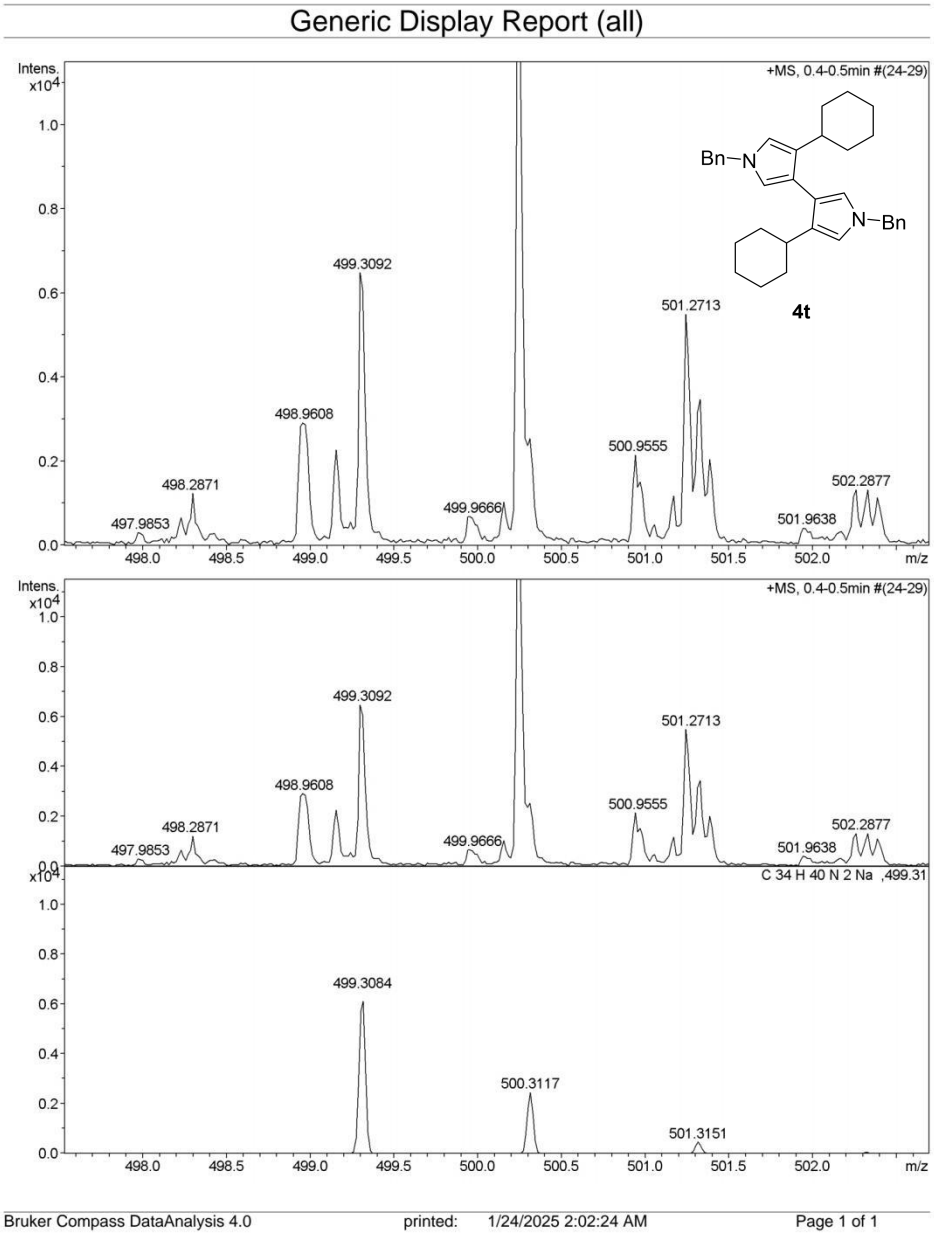
Monoisotopic Mass, Even Electron Ions
353 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 28-28 H: 25-25 N: 0-100 O: 0-100
31
250103-3-T-494-2 21 (0.077)

1: TOF MS ES+
3.65e+003



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
389.2015	389.2018	-0.3	-0.8	17.5	138.4	n/a	n/a	C28 H25 N2

4t. HRMS (ESI) calcd for $C_{34}H_{40}N_2Na^+$ m/z 499.3084 $[M+H]^+$, Found499.3092.



5. HRMS (ESI) calcd for $C_{38}H_{33}N_2O_2^+$ m/z 549.2542 $[M+H]^+$, Found 549.2545.

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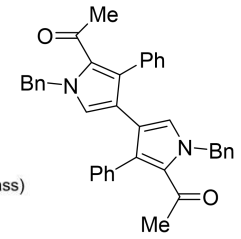
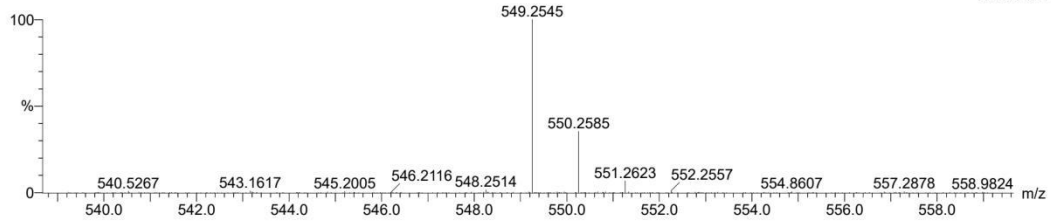


5

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

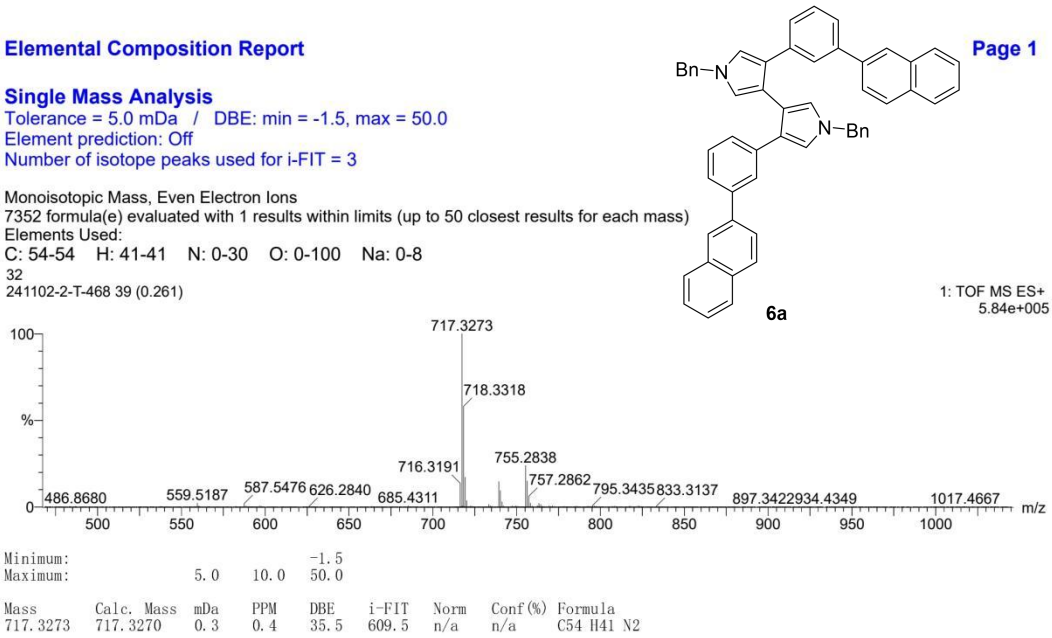
Monoisotopic Mass, Even Electron Ions
682 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 38-38 H: 33-33 N: 0-100 O: 0-100
31
250103-3-T-496 41 (0.115)

1: TOF MS ES+
8.53e+004

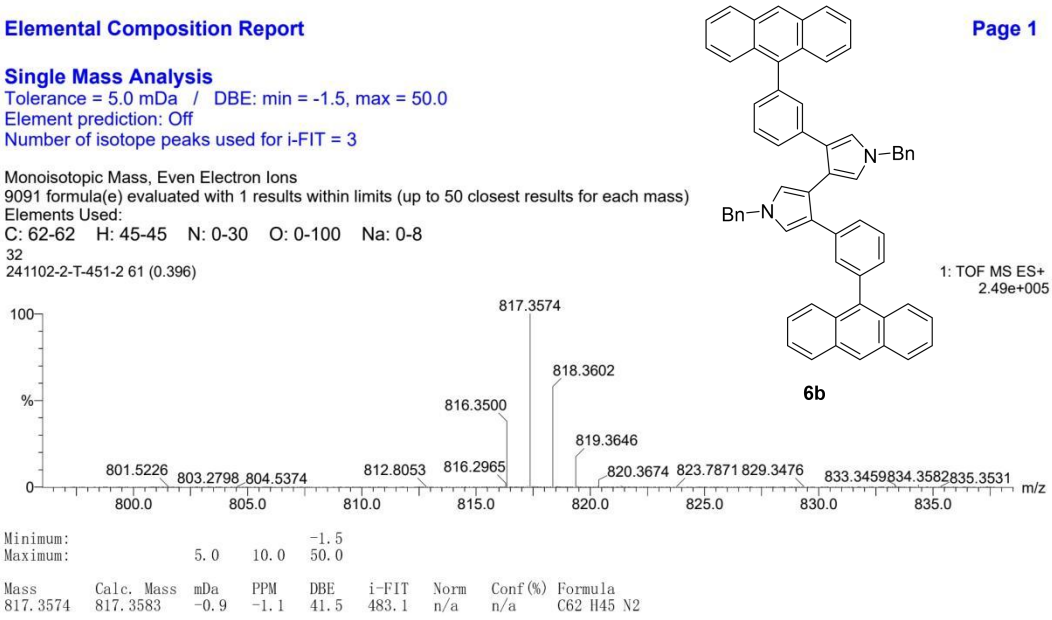


Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
549.2545	549.2542	0.3	0.5	23.5	391.9	n/a	n/a	C38 H33 N2 O2

6a. HRMS (ESI) calcd for $C_{54}H_{41}N_2^+$ *m/z* 717.3270 $[M+H]^+$, Found 717.3273.

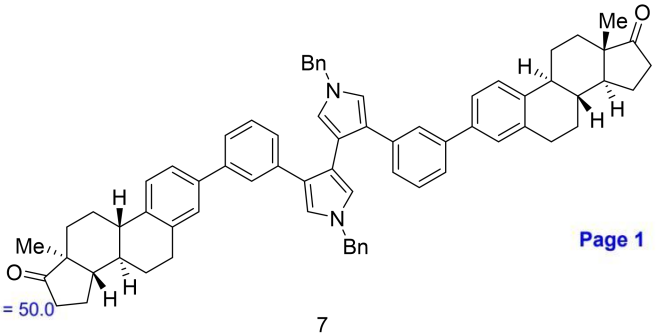


6b. HRMS (ESI) calcd for $C_{62}H_{45}N_2^+$ *m/z* 817.3583 $[M+H]^+$, Found 817.3574.



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
817.3574	817.3583	-0.9	-1.1	41.5	483.1	n/a	n/a	C62 H45 N2

7. HRMS (ESI) calcd for $C_{70}H_{68}N_2O_2Na^+$ *m/z* 991.5178 $[M+Na]^+$, Found 991.5179.



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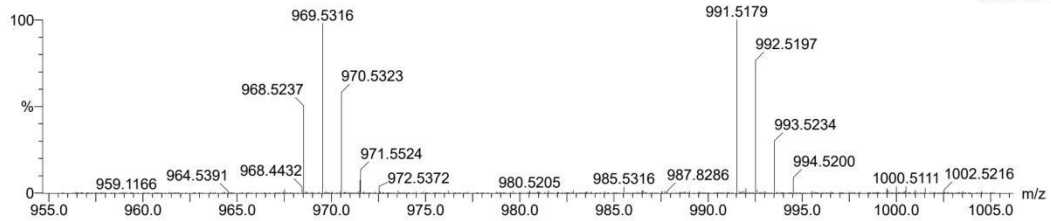
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
11964 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 70-70 H: 68-68 N: 0-30 O: 0-100 Na: 0-8

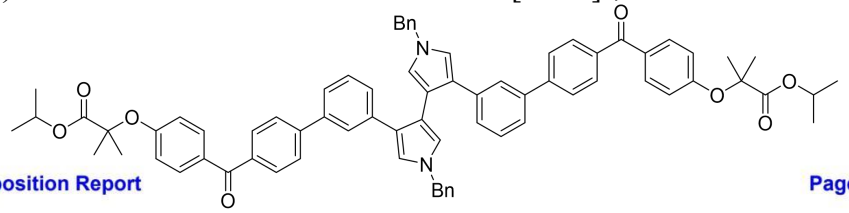
32
241102-2-T-465 60 (0.371)

1: TOF MS ES+
3.54e+004



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
991.5179	991.5178	0.1	0.1	37.5	311.4	n/a	n/a	C70 H68 N2 O2 Na

8. HRMS (ESI) calcd for $C_{74}H_{69}N_2O_8^+$ *m/z* 1113.5054 $[M+H]^+$, Found 1113.5047.



Elemental Composition Report **Page 1**

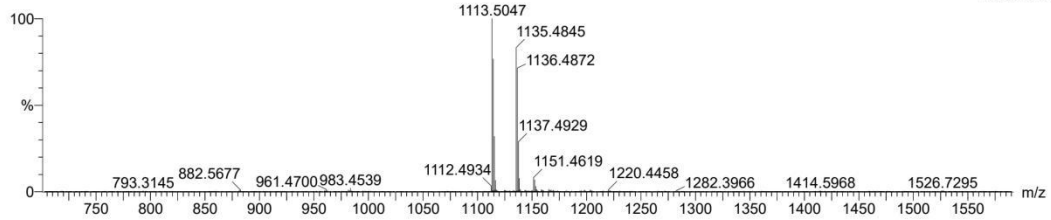
Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

8

Monoisotopic Mass, Even Electron Ions
13744 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 74-74 H: 69-69 N: 0-30 O: 0-100 Na: 0-8

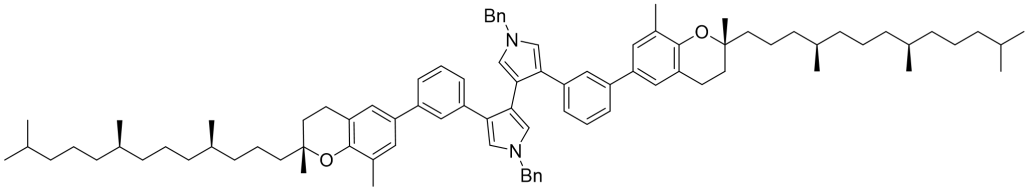
32
241102-2-T-466 39 (0.261)

1: TOF MS ES+
1.98e+005



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
1113.5047	1113.5054	-0.7	-0.6	41.5	462.7	n/a	n/a	C74 H69 N2 O8

9. HRMS (ESI) calcd for $C_{88}H_{116}N_2O_2Na^+$ *m/z* 1255.8935 $[M+Na]^+$, Found 1255.8921.



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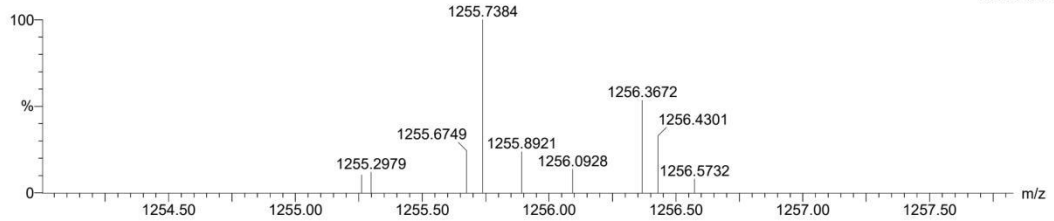
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

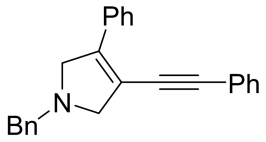
Monoisotopic Mass, Even Electron Ions
16385 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 88-88 H: 116-116 N: 0-30 O: 0-100 Na: 0-8
32
241102-2-T-464 32 (0.224)

1: TOF MS ES+
2.29e+002



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
1255.8921	1255.8935	-1.4	-1.1	31.5	50.5	n/a	n/a	C88 H116 N2 O2 Na

10. HRMS (ESI) calcd for $C_{25}H_{22}N^+$ *m/z* 336.1752 $[M+H]^+$, Found 336.1754.



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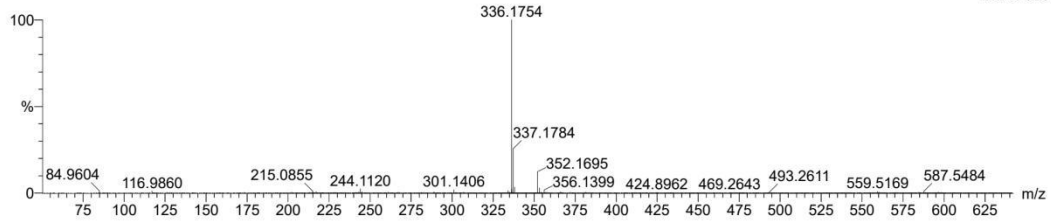
Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
1414 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 25-25 H: 22-22 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-452 42 (0.277) 1: TOF MS ES+
1.17e+006



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
336.1754	336.1752	0.2	0.6	15.5	822.8	n/a	n/a	C25 H22 N

11. HRMS (ESI) calcd for $C_{34}H_{31}N_2^+$ *m/z* 467.2487 $[M+H]^+$, Found 467.2491.

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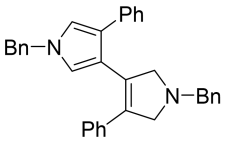


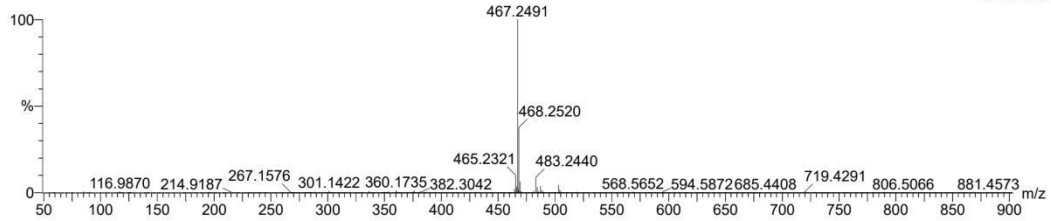
11

Single Mass Analysis
Tolerance = 5.0 mDa / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
3102 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)
Elements Used:
C: 34-34 H: 31-31 N: 0-30 O: 0-100 Na: 0-8

32
241102-2-T-453 33 (0.229)

1: TOF MS ES+
1.77e+006



Minimum:				-1.5				
Maximum:		5.0	10.0	50.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
467.2491	467.2487	0.4	0.9	20.5	764.6	n/a	n/a	C34 H31 N2