# Supporting Information

## 4,5-Disubstituted Pyrenes from Phenangermoles

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# List of Content

## 1 General information

### 1.1 Reagent information

All reagents were purchased from commercial source and used without further purification unless otherwise noted. Dry DCE、TMEDA、n-BuLi and $CD_3Cl$ were purchased from J&K Scientific Ltd., $PdBr_2$ were purchased form Sinocompound company. o-chloranil was purchased from TCI. $Me_2GeCl_2$ was purchased from Alfa, $AgPF_6(collidine)_2$[1] was synthesized according to reported literatures. Silica gel (300-400 mesh, pH = 6-7, HG/T2354-2010) was purchased from Branch Qingdao Haiyang Chemical Co., Ltd.. Other reagents and solvents were used as received unless otherwise noted.

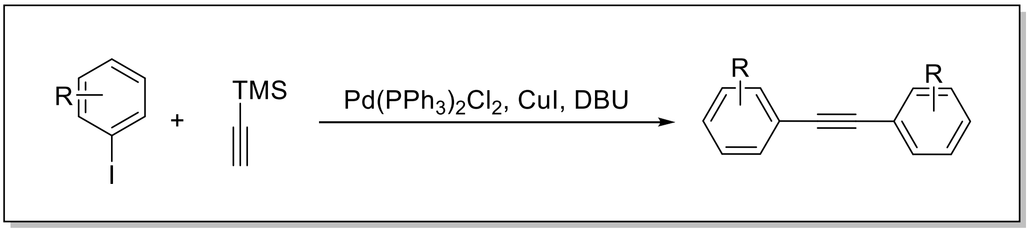
### 1.2 Analytical information

$^1$H-NMR spectra were recorded on 400 MHz spectrometers. Chemical shifts of $^1$H-NMR spectra were reported in parts per million relative to tetramethylsilane (δ = 0). Data for $^1$H-NMR were reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz), and integration. $^{13}$C-NMR spectra were recorded on 101 MHz spectrometers. Chemical shifts were reported in parts per million relative to the solvent resonance as the internal standard ($CDCl_3$, δ 77.2 ppm). Data for $^{13}$C-NMR are reported in terms of chemical shift (δ ppm), multiplicity, and coupling constant (Hz). High-resolution mass spectra (HRMS) were recorded on an Acquity UPLC-Xevo G2 QTof instrument with ESI mode or Thermo Scientific Q Exactive GC Mass Spectrometer with EI mode unless otherwise stated. Organic solutions were concentrated under reduced pressure on a Buchi rotary evaporator. Chromatographic purification of products was accomplished using column chromatography on silica gel.

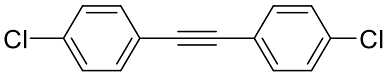
## 2. Compounds preparation and characterization

### 2.1 Preparation and characterization of diarylacetylene

**General Procedure A:**



Starting material iodide (2 mmol)、$PdCl_2(PPh_3)_2$ (85.9 mg, 0.12 mmol, 6 mol%) and CuI (38.1 mg, 0.2 mmol, 10 mol%) was placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. While stirring, dry THF (10 mL) was added, DBU (1.8 mL, 12 mmol, 6 eq.) and distilled water (14.5 μL, 0.8 mmol, 40 mol%) were then added. Finally trimethylsilylacetylene (144 μL, 1 mmol, 0.5 eq.) was added.[2] The reaction mixture was stirred at room temperature for 18 h, at the end of which the reaction mixture is partitioned in EtOAc and distilled water (20 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography.



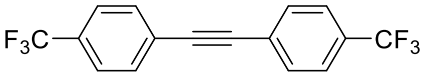
**1,2-bis(4-chlorophenyl)ethyne(2c)**

According to General Procedure A, starting material: 1-chloro-4-iodobenzene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 81% yield, 199.9 mg, yellow solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 7.48 – 7.42 (m, 4H), 7.36 – 7.30 (m, 4H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 134.51, 132.80, 128.77, 121.40, 89.17.

**HRMS (EI) m/z (M+•)** Calcd for $C_{14}H_8Cl_2$: 246.0003; Found:245.99988.



**1,2-bis(4-(trifluoromethyl)phenyl)ethyne(2f)**

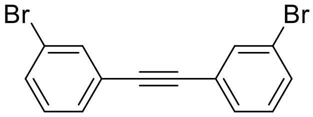
According to General Procedure A, starting material: 4-iodobenzotrifluoride, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 75% yield, 236.1 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 7.76 – 7.58 (m, 8H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 131.98, 130.48 (q, J = 33 Hz), 126.36, 125.39 (q, J = 4 Hz), 123.83 (q, J = 271 Hz), 90.12.

**$^{19}$F NMR (471 MHz, CDCl$_3$)** δ -62.86.

**HRMS (ESI) *m/z* ([M+Na]$^+$)** Calcd for $C_{16}H_8F_6Na$: 337.0428; Found:337.0439.



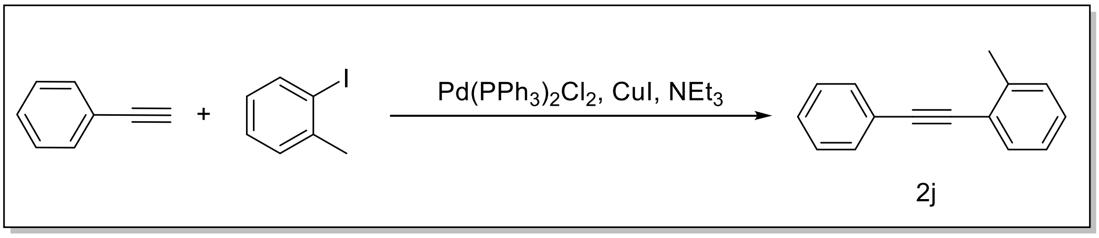
**1,2-bis(3-bromophenyl)ethyne(2g)**

According to General Procedure A, starting material: 1-bromo-3-iodobenzene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 73% yield, 244.9 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 7.68 (t, J = 1.8 Hz, 2H), 7.49 (ddd, J = 8.1, 2.0, 1.0 Hz, 2H), 7.44 (dt, J = 7.7, 1.3 Hz, 2H), 7.23 (t, J = 7.9 Hz, 2H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 134.37, 131.76, 130.20, 129.85, 124.74, 122.22, 89.00.

**HRMS (EI) m/z (M+•)** Calcd for $C_{14}H_8Br_2$: 333.8993; Found: 333.8987.



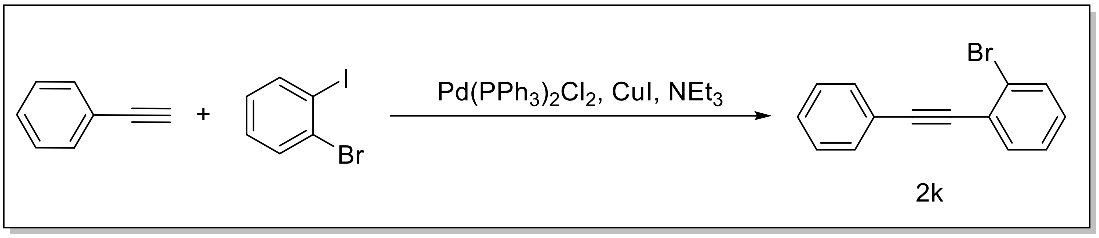
**1-methyl-2-(phenylethynyl)benzene(2j)**

1-iodo-2-methylbenzene (0.26 mL, 2 mmol), $Pd(PPh_3)_2Cl_2$ (28.6 mg, 0.04 mmol, 2 mol%) and CuI (15.2 mg, 0.08 mmol, 4 mol%) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The $NEt_3$(4.8 mL) was added under argon, then phenylacetylene ( 0.22 mL, 2 mmol, 1eq.) was added dropwise at room temperature. The reaction mixture was stirred at room temperature for 2 h, at the end of which the reaction mixture is partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography (petroleum ether) to give 345.6 mg title product as a white oil. 90% yield.

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.59 – 7.47 (m, 3H), 7.39 – 7.29 (m, 3H), 7.23 (d, J = 4.5 Hz, 2H), 7.16 (dt, J = 8.6, 4.5 Hz, 1H), 2.52 (d, J = 1.5 Hz, 3H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 140.23, 131.87, 131.56, 129.52, 128.41, 128.36, 128.23, 125.64, 123.56, 123.03, 93.39, 88.38, 20.84.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{15}H_{13}$: 193.1017; Found:193.1014.



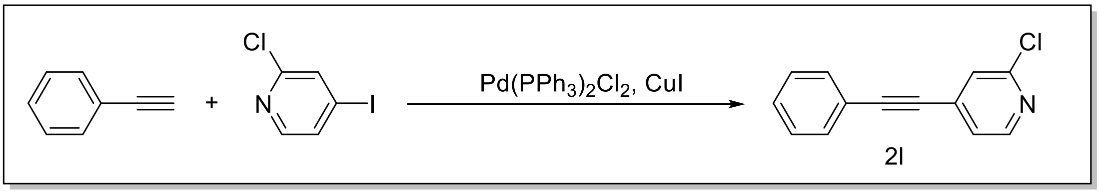
**1-bromo-2-(phenylethynyl)benzene(2k)**

1-bromo-2-iodobenzene (0.25 mL, 2 mmol), $Pd(PPh_3)_2Cl_2$ (28.6 mg, 0.04 mmol, 2 mol%) and CuI (15.2 mg, 0.08 mmol, 4 mol%) was placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The $NEt_3$ (4.8 mL) was added under argon, then phenylacetylene ( 0.22 mL, 2 mmol, 1eq.) was added dropwise at room temperature. The reaction mixture was stirred at room temperature for 2 h, at the end of which the reaction mixture was partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography (petroleum ether) to give 488.5 mg title product as a white oil. 95% yield.

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.63 – 7.53 (m, 4H), 7.35 (dd, J = 4.7, 2.0 Hz, 3H), 7.27 (td, J = 7.6, 1.2 Hz, 1H), 7.16 (td, J = 7.8, 1.7 Hz, 1H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 133.27, 132.49, 131.75, 129.45, 128.71, 128.44, 127.09, 125.68, 125.40, 122.91, 93.96, 88.07.

**HRMS (EI) m/z (M+•)** Calcd for $C_{14}H_9Br$: 255.9888; Found: 255.98851.



**2-chloro-4-(phenylethynyl)pyridine(2l)**

2-chloro-4-iodopyridine (0.4789 g, 2 mmol), $Pd(PPh_3)_2Cl_2$ (28.6 mg, 0.04 mmol, 2 mol%) and CuI (15.2 mg, 0.08 mmol, 4 mol%) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The $NEt_3$ (4.8 mL) was added under argon, then phenylacetylene ( 0.22 mL, 2 mmol, 1eq.) was added dropwise at room temperature. The reaction mixture was stirred at room temperature for 2 h, at the end of which the reaction mixture was partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography (petroleum ether) to give 312.8 mg title product as a white oil. 73% yield.

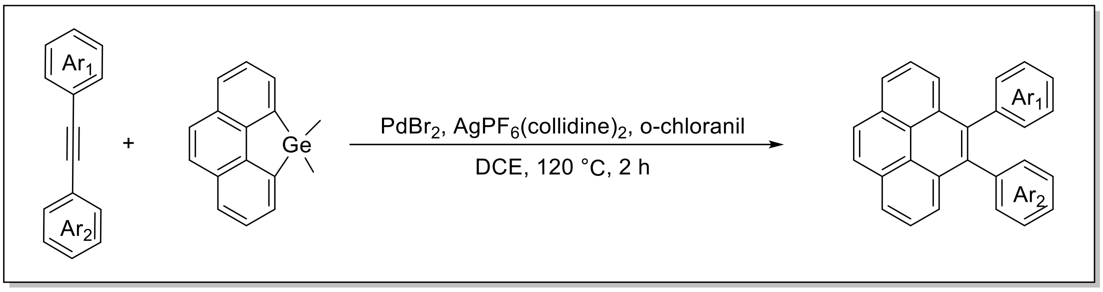
**$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.54 (d, J = 2.3 Hz, 1H), 7.76 (dd, J = 8.3, 2.3 Hz, 1H), 7.57 – 7.51 (m, 2H), 7.41 – 7.35 (m, 3H), 7.33 (dd, J = 8.3, 0.7 Hz, 1H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 152.04, 150.39, 140.93, 131.70, 129.07, 128.52, 123.94, 122.16, 119.39, 93.76, 84.68.

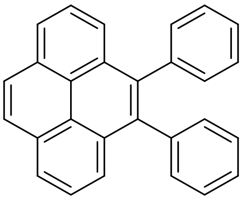
**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{13}H_9ClN$: 214.0424; Found:214.0424.

**2.2 Preparation and characterization of 4,5-diaryl pyrene**

**General Procedure B:**



4,4-dimethyl-4H-phenanthro[4,5-bcd]germole (0.0418g, 0.15 mmol, 1.5 eq.)、Diarylacetylene (0.1 mmol)、 $PdBr_2$ (0.0014g, 0.5 umol, 5 mol%)、$AgPF_6(collidine)_2$ (0.0050g, 1 umol, 10 mol%) and o-chloranil (0.0507g, 0.2 mmol, 2 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, DCE(0.5 mL) was added. The reaction mixture was stirred at 120℃ for 2 h before being quenched with water. The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography on silica gel.



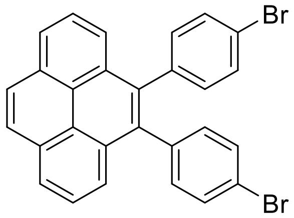
**4,5-diphenylpyrene(3a)**

According to General Procedure B, using 1,2-diphenylethyne, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 78% yield, 27.5 mg, white solid.

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.20 (dd, J = 7.5, 1.2 Hz, 2H), 8.14 (s, 2H), 7.91 (t, J = 7.7 Hz, 2H), 7.84 (dd, J = 7.9, 1.2 Hz, 2H), 7.32 – 7.21 (m, 10H).

**$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 139.50, 137.77, 131.37, 131.13, 127.68, 127.41, 126.60, 125.86, 125.05, 124.86, 124.22.

**HRMS (ESI) m/z ([M+H]+)** Calcd for $C_{28}H_{19}$: 355.1487; Found:355.1473.



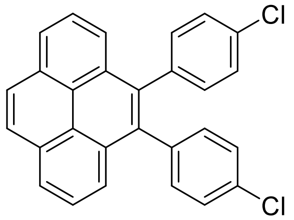
**4,5-bis(4-bromophenyl)pyrene(3b)**

According to General Procedure B, using 1,2-bis(4-bromophenyl)ethyne, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 87% yield, 44.5 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.21 (dd, J = 7.6, 1.1 Hz, 2H), 8.13 (s, 2H), 7.91 (t, J = 7.7 Hz, 2H), 7.76 (dd, J = 7.9, 1.1 Hz, 2H), 7.52 – 7.43 (m, 4H), 7.16 – 7.09 (m, 4H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 138.18, 136.63, 132.70, 131.23, 131.07, 130.88, 127.53, 126.06, 125.51, 124.65, 124.24, 121.12.

**HRMS (EI) m/z (M+•)** Calcd for $C_{28}H_{16}Br_2$: 509.9619; Found:509.96168.



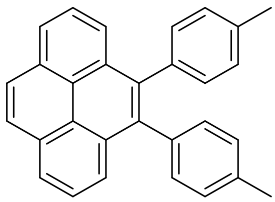
**4,5-bis(4-chlorophenyl)pyrene(3c)**

According to General Procedure B, using 1,2-bis(4-chlorophenyl)ethyne at 100℃ for 12 h, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 99% yield, 42.1 mg, yellow solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.22 (dd, J = 7.7, 1.1 Hz, 2H), 8.14 (s, 2H), 7.91 (t, J = 7.8 Hz, 2H), 7.77 (dd, J = 7.9, 1.1 Hz, 2H), 7.30 (d, J = 8.4 Hz, 4H), 7.21 – 7.14 (m, 4H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 137.73, 136.74, 132.86, 132.36, 131.08, 130.97, 128.27, 127.53, 126.05, 125.48, 124.65, 124.25.

**HRMS (EI) m/z (M+•)** Calcd for $C_{28}H_{16}Cl_2$: 422.0629; Found:422.06243.



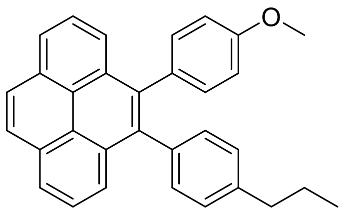
**4,5-di-p-tolylpyrene(3d)**

According to General Procedure B, using 1,2-di-p-tolylethyne, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 78% yield, 29.9 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.16 (dd, J = 7.3, 1.3 Hz, 2H), 8.10 (s, 2H), 7.92 – 7.78 (m, 4H), 7.14 (dd, J = 8.0, 1.8 Hz, 4H), 7.09 (dd, J = 7.9, 1.7 Hz, 4H), 2.34 (s, 6H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 137.79, 136.61, 135.97, 131.71, 131.03, 131.00, 128.49, 127.42, 125.83, 124.93 (d, J = 2.2 Hz), 124.22, 21.38.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{30}H_{23}$: 383.1800; Found:383.1816.



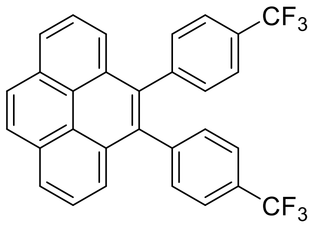
**4-(4-methoxyphenyl)-5-(4-propylphenyl)pyrene(3e)**

According to General Procedure B, using 1-methoxy-4-((4-propylphenyl)ethynyl)benzene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 88% yield, 37.8 mg, yellow solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.22 – 8.17 (m, 2H), 8.14 (s, 2H), 7.94 – 7.86 (m, 4H), 7.20 – 7.13 (m, 4H), 7.10 (d, J = 7.9 Hz, 2H), 6.87 – 6.80 (m, 2H), 3.81 (s, 3H), 2.60 (t, J = 7.6 Hz, 2H), 1.67 (q, J = 7.4 Hz, 2H), 0.93 (t, J = 7.3 Hz, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 158.06, 140.68, 138.17, 137.45, 136.86, 132.23, 131.92, 131.77, 131.62, 131.01, 130.94, 127.83, 127.41, 127.39, 125.81, 124.95, 124.91, 124.83, 124.23, 113.11, 55.20, 37.72, 24.39, 13.72.

**HRMS (EI) m/z (M+•)** Calcd for $C_{32}H_{26}O$: 426.1984; Found:426.19792.



**4,5-bis(4-(trifluoromethyl)phenyl)pyrene(3f)**

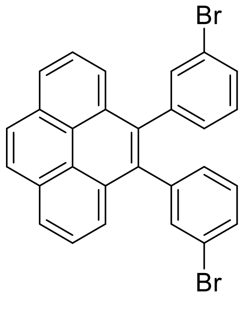
According to General Procedure B, using 1,2-bis(4-(trifluoromethyl)phenyl)ethyne, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 79% yield, 38.6 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.26 (dd, J = 7.6, 1.1 Hz, 2H), 8.17 (s, 2H), 7.95 (t, J = 7.8 Hz, 2H), 7.73 (dd, J = 8.0, 1.1 Hz, 2H), 7.59 (d, J = 7.9 Hz, 4H), 7.38 (d, J = 7.9 Hz, 4H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 142.93, 136.56, 131.39, 131.13, 130.57, 129.26 (q, J = 32 Hz), 127.61, 126.20, 125.79, 125.00 (q, J = 4 Hz), 124.60, 124.15 (q, J = 271 Hz), 124.31.

**$^{19}$F NMR (471 MHz, CDCl$_3$)** δ -62.46.

**HRMS (EI) m/z (M+•)** Calcd for $C_{30}H_{16}F_6$: 490.1156; Found: 490.11502.



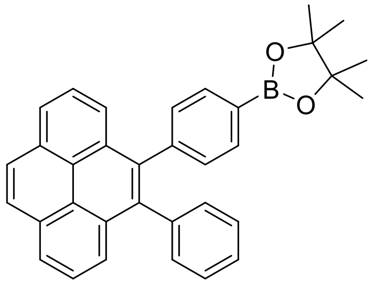
**4,5-bis(3-bromophenyl)pyrene(3g)**

According to General Procedure B, using 1,2-bis(3-bromophenyl)ethyne, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 42% yield, 21.3 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.23 (dd, J = 7.6, 1.1 Hz, 2H), 8.14 (s, 2H), 7.94 (t, J = 7.8 Hz, 2H), 7.80 (d, J = 7.9 Hz, 2H), 7.53 – 7.39 (m, 4H), 7.28 – 7.15 (m, 4H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 141.24, 141.21, 136.53, 133.97, 133.80, 131.06, 130.70, 130.12, 129.79, 129.67, 129.62, 129.43, 127.54, 126.13, 125.60, 124.73, 124.25, 122.09, 121.94.

**HRMS (EI) m/z (M+•)** Calcd for $C_{28}H_{16}Br_2$: 509.9619; Found:509.96155.



**4,4,5,5-tetramethyl-2-(4-(5-phenylpyren-4-yl)phenyl)-1,3,2-dioxaborolane(3h)**

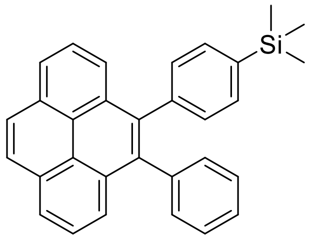
According to General Procedure B, using 4,4,5,5-tetramethyl-2-(4-(phenylethynyl)phenyl)-1,3,2-dioxaborolane, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 49% yield, 23.3 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.19 (ddd, J = 7.5, 3.4, 1.1 Hz, 2H), 8.12 (s, 2H), 7.96 – 7.72 (m, 6H), 7.37 – 7.25 (m, 7H), 1.36 (s, 12H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 142.66, 139.35, 137.63, 137.56, 134.18, 131.41, 131.25, 131.11, 131.01, 130.60, 127.84, 127.44, 127.43, 126.72, 125.89, 125.11, 125.09, 124.91, 124.89, 124.24, 124.21, 83.82, 24.99.

**$^{11}$B NMR (160 MHz, CDCl$_3$)** δ 30.36.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{34}H_{30}BO_2$: 481.2339; Found:481.2347.



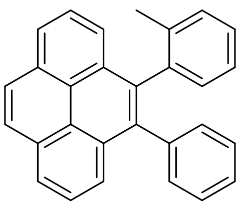
**trimethyl(4-(5-phenylpyren-4-yl)phenyl)silane(3i)**

According to General Procedure B, using trimethyl(4-(phenylethynyl)phenyl)silane, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 76% yield, 32.3 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.17 (dd, J = 7.4, 1.4 Hz, 2H), 8.11 (s, 2H), 7.93 – 7.79 (m, 4H), 7.46 – 7.37 (m, 2H), 7.33 – 7.17 (m, 7H), 0.32 – 0.22 (m, 9H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 140.85, 140.48, 139.31, 138.77, 138.67, 133.62, 132.42, 132.39, 132.16, 132.02, 131.43, 128.70, 128.43, 127.56, 126.87, 126.06, 125.99, 125.88, 125.24, 0.00.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{31}H_{27}Si$: 427.1882; Found:427.1892.



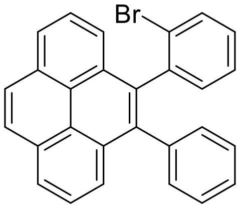
**4-phenyl-5-(o-tolyl)pyrene(3j)**

According to General Procedure B, using 1-methyl-2-(phenylethynyl)benzene at 100℃ for 12 h, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 65% yield, 24.0 mg, white solid.

**$^{1}$H NMR (400 MHz, CDCl$_3$)** δ 8.19 (ddd, J = 7.7, 4.0, 1.1 Hz, 2H), 8.13 (s, 2H), 7.92 – 7.87 (m, 2H), 7.84 (ddd, J = 8.0, 6.2, 1.1 Hz, 1H), 7.64 (dt, J = 7.9, 1.4 Hz, 1H), 7.33 – 7.23 (m, 5H), 7.19 – 7.09 (m, 4H), 2.00 (d, J = 1.2 Hz, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 139.47, 138.94, 137.54, 137.20, 136.81, 131.57, 131.49, 131.37, 131.15, 131.05, 130.86, 129.62, 129.43, 127.78, 127.65, 127.48, 127.45, 127.24, 126.82, 126.14, 125.87, 125.24, 125.13, 125.10, 124.83, 124.39, 124.34, 20.16.

**HRMS (ESI) *m/z* ([M+Na]$^+$)** Calcd for $C_{29}H_2Na$: 391.1463; Found:391.1457.



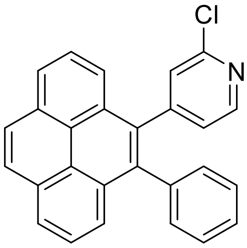
**4-(2-bromophenyl)-5-phenylpyrene(3k)**

According to General Procedure B, using 1-bromo-2-(phenylethynyl)benzene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 68% yield, 29.3 mg, white solid.

**$^{1}$H NMR (400 MHz, CDCl$_3$)** δ 8.21 (dt, J = 7.7, 1.7 Hz, 2H), 8.13 (s,2H), 7.91 (td, J = 7.7, 5.6 Hz, 2H), 7.84 (d, J = 8.4 Hz, 1H), 7.63 (d, J = 7.9 Hz, 1H), 7.59 (d, J = 8.0 Hz, 2H), 7.50 (dd, J = 7.7, 1.9 Hz, 2H), 7.34 – 7.25 (m, 4H), 7.22 (d, J = 4.6 Hz, 2H), 7.12 (dt, J = 8.7, 4.7 Hz, 1H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 140.54, 139.13, 137.88, 136.71, 133.00, 132.38, 131.39, 131.20, 131.10, 130.85, 130.16, 129.68, 128.76, 128.08, 127.54, 127.50, 127.46, 127.04, 126.99, 126.17, 125.92, 125.42, 125.26, 125.19, 125.05, 124.52, 124.17.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{28}H_{18}Br$: 433.0592; Found:433.0605.



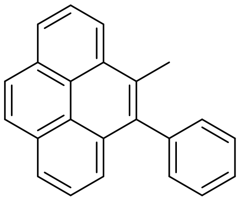
**2-chloro-4-(5-phenylpyren-4-yl)pyridine(3l)**

According to General Procedure B, using 2-chloro-4-(phenylethynyl)pyridine, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 74% yield, 32.4 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.30 (d, J = 2.4 Hz, 1H), 8.22 (dd, J = 7.6, 1.1 Hz, 2H), 8.13 (s, 2H), 7.91 (td, J = 7.7, 5.6 Hz, 2H), 7.81 (dd, J = 8.0, 1.1 Hz, 1H), 7.70 (dd, J = 7.9, 1.0 Hz, 1H), 7.52 (dd, J = 8.1, 2.4 Hz, 1H), 7.32 (tddd, J = 8.2, 6.5, 4.0, 2.0 Hz, 3H), 7.25 – 7.17 (m, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 151.49, 149.75, 141.31, 139.35, 138.57, 134.40, 132.49, 131.21, 131.18, 131.06, 131.04, 130.96, 130.74, 128.50, 128.08, 127.69, 127.62, 127.47, 127.33, 126.19, 126.14, 125.84, 125.58, 125.21, 124.44, 124.21, 123.93, 123.54.

**HRMS (ESI) m/z ([M+H]$^+$)** Calcd for $C_{27}H_{17}ClN$ : 390.1050 ; Found:390.1055.



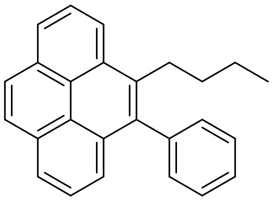
**4-methyl-5-phenylpyrene(3m)**

According to General Procedure B, using prop-1-yn-1-ylbenzene at 100℃ for 12 h, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 37% yield, 10.9 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.41 (dd, J = 7.9, 1.1 Hz, 1H), 8.23 (dd, J = 7.7, 1.0 Hz, 1H), 8.22 – 8.02 (m, 4H), 7.85 (td, J = 7.8, 1.2 Hz, 1H), 7.65 (dq, J = 8.0, 1.1 Hz, 1H), 7.62 – 7.53 (m, 2H), 7.55 – 7.46 (m, 1H), 7.47 – 7.37 (m, 2H), 2.63 (s, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 140.63, 137.62, 131.76, 131.34, 131.31, 130.84, 130.61, 130.50, 128.51, 127.40, 127.36, 127.19, 125.96, 125.68, 125.02, 124.62, 124.50, 124.21, 123.52, 122.02, 17.40.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{23}H_{17}$: 293.1330; Found:293.1324.



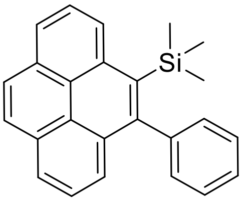
**4-butyl-5-phenylpyrene(3n)**

According to General Procedure B, using hex-1-yn-1-ylbenzene at 100℃ for 12 h, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 48% yield, 16.1 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.43 (dt, J = 8.0, 1.3 Hz, 1H), 8.21 (d, J = 7.6 Hz, 1H), 8.17 – 8.00 (m, 4H), 7.83 (td, J = 7.8, 1.7 Hz, 1H), 7.62 – 7.50 (m, 3H), 7.55 – 7.46 (m, 1H), 7.45 – 7.36 (m, 2H), 3.08 – 2.98 (m, 2H), 1.70 (qdd, J = 7.9, 6.2, 5.4, 1.9 Hz, 2H), 1.36 (hd, J = 7.3, 2.0 Hz, 2H), 0.84 (td, J = 7.3, 2.3 Hz, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 140.42, 137.39, 135.63, 131.94, 131.53, 130.84, 130.35, 128.43, 127.43, 127.34, 127.18, 125.90, 125.65, 124.91, 124.69, 124.66, 124.43, 123.54, 122.21, 33.11, 30.41, 23.29, 13.84.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{26}H_{13}$: 335.1800; Found:335.1783.



**trimethyl(5-phenylpyren-4-yl)silane(3o)**

According to General Procedure B, using trimethyl(phenylethynyl)silane at 100℃ for 12 h, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 48% yield, 16.9 mg, white solid.

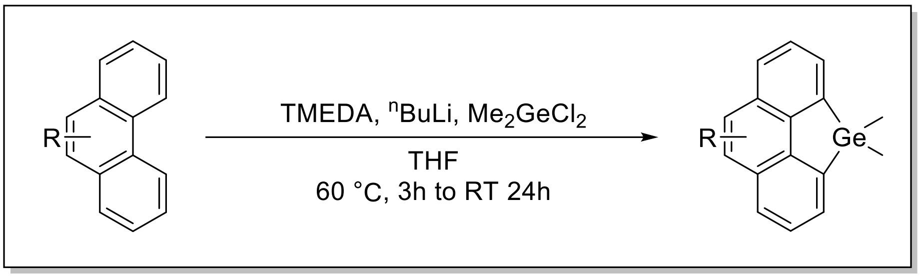
**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.63 (dd, J = 8.0, 1.0 Hz, 1H), 8.20 (dd, J = 7.6, 1.0 Hz, 1H), 8.16 (dd, J = 7.6, 1.1 Hz, 1H), 8.13 – 8.06 (m, 2H), 8.04 (t, J = 7.8 Hz, 1H), 7.84 (t, J = 7.8 Hz, 1H), 7.67 (dd, J = 8.0, 1.1 Hz, 1H), 7.53 (dd, J = 5.2, 1.9 Hz, 3H), 7.48 (dt, J = 6.4, 2.2 Hz, 2H), 0.20 (s, 9H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 147.59, 142.49, 135.20, 134.00, 131.51, 131.44, 130.79, 128.05, 127.62, 127.01, 126.65, 125.55, 125.46, 125.04, 124.80, 124.68, 124.38, 2.74.

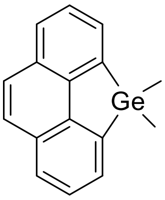
**HRMS (EI) m/z (M+•)** Calcd for $C_{25}H_{22}Si$: 350.1491; Found:350.14798.

## 2.3 Preparation and characterization of phenangermoles

### General Procedure C:



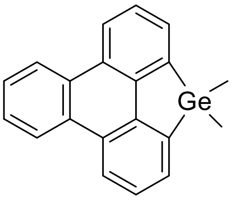
Derivative of phenanthrene (2 mmol) and TMEDA (0.72 mL, 4.8 mmol 2.4 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, a solution of n-BuLi (2.4 M in Hexane, 1.84 mL, 4.4 mmol, 2.2 eq.) was added dropwise at 0 ℃. After stirring at 60 ℃ for 3 h,[3] dry THF (4.8 mL) was added, dichlorodimethylgermane (0.4 mL, 3.4 mmol, 1.7 eq.) was added at –78 ℃. The reaction mixture was stirred at room temperature for 24 h before being quenched with water. The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography on silica gel.



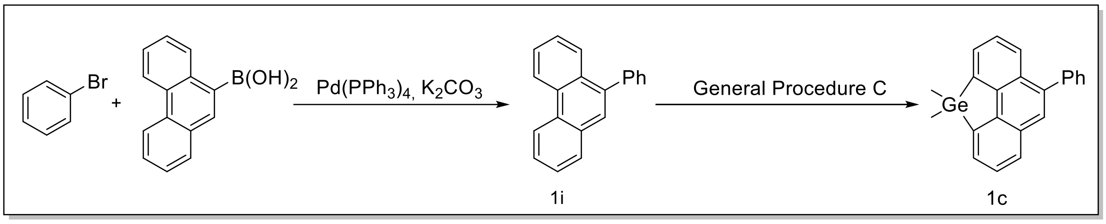
**4,4-dimethyl-4H-phenanthro[4,5-bcd]germole (1a)**

According to General Procedure C, using phenanthrene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 30% yield, 167.4 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 7.90 – 7.86 (m, 4H), 7.77 (s, 2H), 7.63 (dd, J = 7.9, 6.8 Hz, 2H), 0.73 (s, 6H).
**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 142.82, 142.47, 132.45, 132.22, 129.44, 129.09, 128.78, 128.44, 0.00.
**HRMS (EI) *m/z* (M$^{+•}$)** Calcd for $C_{16}H_{14}Ge$: 280.0307; Found:280.02992.



**4,4-dimethyl-4H-triphenyleno[1,12-bcd]germole(1b)**
According to General Procedure C, using triphenylene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 51% yield, 337.9 mg, white solid.
**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.70 – 8.64 (m, 2H), 8.56 (dd, *J* = 8.2, 1.0 Hz, 2H), 7.88 (dd, *J* = 6.8, 1.0 Hz, 2H), 7.71 – 7.64 (m, 4H), 0.73 (s, 6H).
**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 142.84, 142.28, 133.49, 132.51, 130.57, 129.59, 129.34, 125.76, 125.20, 2.34.
**HRMS (EI) m/z (M+•)** Calcd for $C_{20}H_{16}Ge$: 330.0464; Found:330.04562.



9-Phenanthracenylboronic acid (0.5329 g, 2.4 mmol, 1.2 eq.), Pd(PPh$_3$)$_4$ (0.0924 g, 0.08 mmol, 4 mol%) and $K_2CO_3$ (0.6634 g, 4.8 mmol, 2.4 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The dry THF (20 mL) was added under argon, then bromobenzene (0.21 mL, 2 mmol, 1 eq.) was added dropwise. The reaction mixture was refluxed for 24 h, at the end of which the reaction mixture is partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography (petroleum ether) to give 278.7 mg title product as a white oil. 55% yield.
According general procedure C to get compound 1c.

**9-phenylphenanthrene(1i)**
**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.83 (d, J = 8.3 Hz, 1H), 8.78 (d, J = 8.1 Hz, 1H), 8.00 (dd, J = 8.2, 1.3 Hz, 1H), 7.95 (dd, J = 7.7, 1.5 Hz, 1H), 7.78 – 7.65 (m, 4H), 7.64 – 7.49 (m, 6H).
**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 140.87, 138.84, 131.63, 131.20, 130.70, 130.15, 130.03, 128.75, 128.40, 127.61, 127.45, 127.01, 126.93, 126.67, 126.59, 126.54, 122.99, 122.62, 77.34.
**HRMS (EI) m/z (M+•)** Calcd for $C_{20}H_{14}$: 254.1096; Found: 254.10904 .

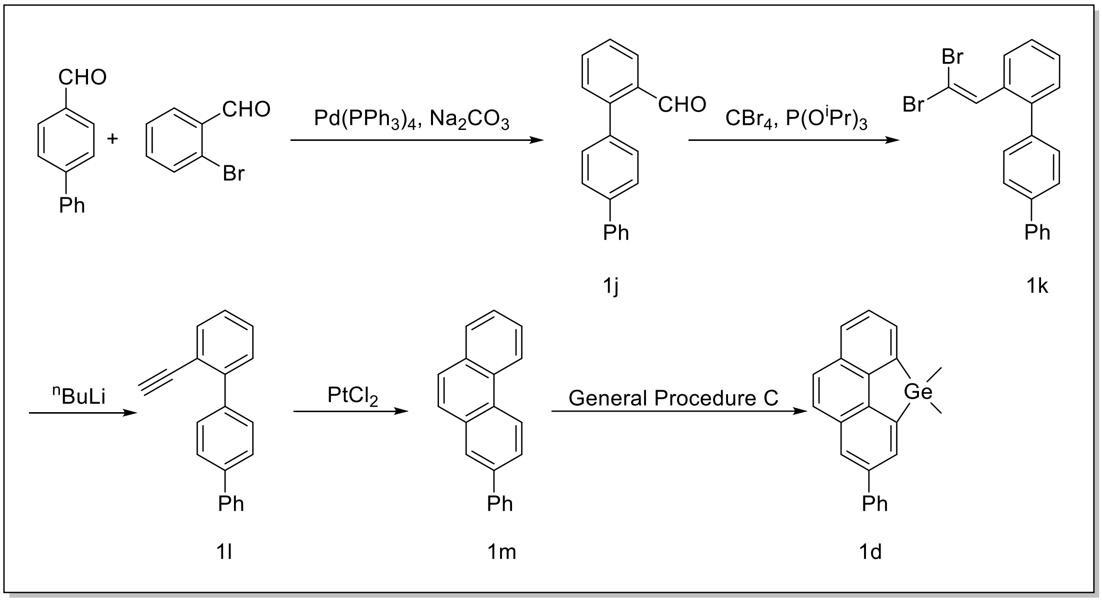
**4,4-dimethyl-8-phenyl-4H-phenanthro[4,5-bcd]germole(1c)**

According to General Procedure C, using 9-phenylphenanthrene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 22% yield, 155.9 mg, white oil.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 7.90 (dd, J = 8.2, 1.0 Hz, 1H), 7.88 – 7.84 (m, 3H), 7.70 (s, 1H), 7.64 – 7.61 (m, 1H), 7.57 (ddd, J = 9.1, 7.4, 1.5 Hz, 3H), 7.52 – 7.47 (m, 2H), 7.46 – 7.41 (m, 1H), 0.73 (s, 6H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 143.06, 142.80, 142.78, 142.52, 142.10, 141.07, 132.54, 132.43, 132.20, 131.85, 131.37, 130.53, 129.54, 129.53, 129.34, 129.08, 128.88, 128.15, 0.00.

**HRMS (EI) m/z (M+•)** Calcd for $C_{22}H_{18}Ge$: 356.0620; Found:356.06132.



4-Biphenylboronic acid (0.9902 g, 5 mmol, 1 eq.), Pd(PPh$_3$)$_4$ (0.3178 g, 0.275 mmol, 5.5 mol%) and Na$_2$CO$_3$ (5.2995 g, 50 mmol, 10 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The toluene/EtOH/H$_2$O (25/25/25 mL) was added under argon, then 2-bromobenzaldehyde ( 0.58 mL, 1 eq.) was added dropwise. The reaction mixture was stirred at 80℃ for 3 h, at the end of which the reaction mixture is partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over Na$_2$SO$_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 1.172 g title product as a white oil. 91% yield.

Cool a solution of the above compound (1.0667 g, 4.13 mmol, 1 eq.) and CBr$_4$ (2.7952g, 8.26 mmol, 2 eq.) in DCM (30 mL) to 0 °C. Add P(Oi-Pr)$_3$ (4 mL , 16.52 mmol, 4 eq.) dropwise. The mixture was stirred for 2.5 h and warmed to RT,[4] at the end of which the reaction mixture was partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over Na$_2$SO$_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 1.4419 g title product as a white solid. 85% yield.

The above compound (1.4129 g, 3.4 mmol, 1 eq.) was dissolved in dry THF (23 mL) and cooled to -78 °C. n-BuLi (5.7 mL, 2.4 M in Hexane, 13.6 mmol, 4 eq.) was added dropwise. The mixture was stirred under nitrogen for 6 h. The cold solution was quenched with H$_2$O dropwise and warmed to RT. The solution was extracted with MTBE. The combined organic layers were washed with brine, and then dried over Na$_2$SO$_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 605.31 mg title product as a white oil. 70% yield.

The above compound (0.6060 g, 2.38 mmol, 1 eq.) and $PtCl_2$ (0.0505 g, 0.19 mmol, 8 mol%) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. the DCE (14 mL) was added under argon. The reaction mixture was stirred at 100℃ for 24 h, at the end of which the reaction mixture is partitioned in $CH_2Cl_2$ and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 473.7 mg title product as a white solid. 78% yield.

According general procedure C to get compound 1d.

**[1,1':4',1''-terphenyl]-2-carbaldehyde(1j)**

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 10.07 (d, J = 0.7 Hz, 1H), 8.06 (dt, J = 7.6, 1.2 Hz, 1H), 7.73 – 7.68 (m, 2H), 7.68 – 7.64 (m, 3H), 7.55 – 7.45 (m, 6H), 7.42 – 7.37 (m, 1H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 192.48, 145.55, 141.06, 140.29, 136.65, 133.77, 133.64, 130.77, 130.58, 128.93, 127.84, 127.71, 127.69, 127.16.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{19}H_{15}O$: 259.1123; Found: 259.1122.

**2-(2,2-dibromovinyl)-1,1':4',1''-terphenyl(1k)**

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.80 – 7.63 (m, 5H), 7.53 – 7.34 (m, 8H), 7.28 (s, 1H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 139.56, 139.47, 139.27, 137.96, 136.49, 132.81, 128.92, 128.79, 128.32, 127.82, 127.69, 126.44, 126.15, 126.06, 125.93, 89.86.

**HRMS (EI) m/z (M+•)** Calcd for $C_{20}H_{14}Br_2$: 411.9462; Found: 411.94601.

**2-ethynyl-1,1':4',1''-terphenyl(1l)**

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.68 – 7.62 (m, 7H), 7.48 – 7.41 (m, 4H), 7.38 – 7.29 (m, 2H), 3.08 (d, J = 1.0 Hz, 1H).

**$^{13}$C NMR (126 MHz, $CDCl_3$)** δ 143.93, 140.78, 140.35, 139.17, 134.01, 129.66, 129.57, 129.06, 128.80, 127.37, 127.14, 127.07, 126.75, 120.40, 83.16, 80.36.

**HRMS (EI) m/z (M+•)** Calcd for $C_{20}H_{14}$: 254.1096; Found: 254.10927.

**2-phenylphenanthrene(1m)**

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.76 (d, J = 8.7 Hz, 1H), 8.72 (d, J = 8.2 Hz, 1H), 8.11 (d, J = 2.0 Hz, 1H), 7.92 (ddd, J = 7.9, 4.6, 1.7 Hz, 2H), 7.84 – 7.76 (m, 4H), 7.68 (ddd, J = 8.3, 6.9, 1.6 Hz, 1H), 7.62 (ddd, J = 8.1, 6.9, 1.3 Hz, 1H), 7.52 (dd, J = 8.4, 7.0 Hz, 2H), 7.44 – 7.38 (m, 1H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 140.81, 139.24, 132.38, 132.07, 130.16, 129.43, 128.93, 128.63, 127.47, 127.39, 127.38, 127.12, 126.72, 126.61, 125.85, 123.28, 122.72.

**HRMS (EI) m/z (M+•)** Calcd for $C_{20}H_{14}$: 254.1096; Found:254.10870.

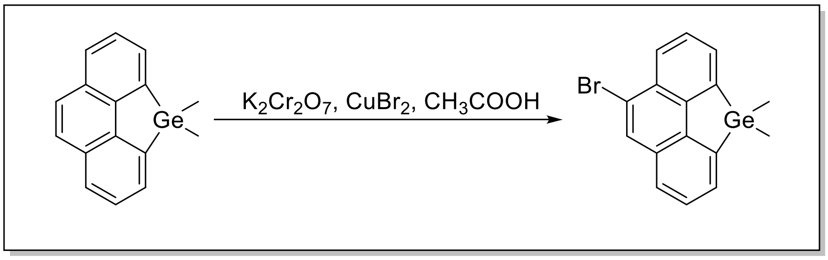
**4,4-dimethyl-2-phenyl-4H-phenanthro[4,5-bcd]germole（1d）**

According to General Procedure C, using 2-phenylphenanthrene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 20% yield, 144.2 mg, white oil.

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.10 (d, J = 1.7 Hz, 1H), 8.06 (d, J = 1.6 Hz, 1H), 7.87 (ddd, J = 6.9, 4.6, 1.0 Hz, 2H), 7.83 – 7.75 (m, 4H), 7.62 (dd, J = 7.9, 6.8 Hz, 1H), 7.50 (dd, J = 8.4, 7.0 Hz, 2H), 7.42 – 7.35 (m, 1H), 0.75 (s, 6H).

**$^{13}$C NMR (126 MHz, CDCl$_3$)** δ 144.07, 143.15, 142.56, 142.49, 142.11, 141.95, 132.50, 132.22, 132.18, 131.90, 130.95, 129.82, 129.41, 129.31, 129.04, 128.79, 128.50, 127.93, 0.00.

**HRMS (EI) m/z (M+•)** Calcd for $C_{22}H_{18}Ge$: 356.0620; Found: 356.06104.



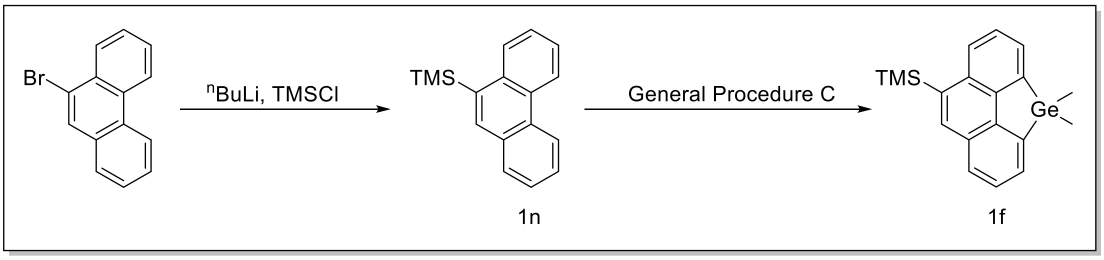
**8-bromo-4,4-dimethyl-4H-phenanthro[4,5-bcd]germole(1e)**

4,4-dimethyl-4H-phenanthro[4,5-bcd]germole (0.2789g, 1 mmol, 1 eq.)、$K_2Cr_2O_7$ (0.3236g, 1.1 mmol, 1.1 eq) and $CuBr_2$ (0.2234g, 1 mmol, 1 eq.) was placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, $CH_3COOH$ (1 mL) was added. The reaction mixture was stirred at room temperature for 1 h,[5] at the end of which the reaction mixture is partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 125.1 mg title product as a white oil. 35% yield.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.23 (dd, J = 8.2, 1.0 Hz, 1H), 8.10 (s, 1H), 7.89 (ddd, J = 12.4, 6.8, 1.0 Hz, 2H), 7.80 (dd, J = 8.0, 1.0 Hz, 1H), 7.74 – 7.70 (m, 1H), 7.62 (dd, J = 8.0, 6.8 Hz, 1H), 0.74 (s, 6H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 143.53, 142.77, 142.39, 142.36, 133.52, 132.93, 132.60, 131.64, 131.38, 130.03, 129.69, 129.27, 128.71, 123.75, 0.00.

**HRMS (EI) m/z (M+•)** Calcd for $C_{16}H_{13}BrGe$: 357.9412; Found: 357.93997.



9-Bromophenanthrene (1.0285 g, 4 mmol, 1 eq.) was dissolved in dry $Et_2O$ (8 mL) and cooled to -78 °C. n-BuLi (1.7 mL, 2.4 M in Hexane, 4.2 mmol, 1 eq.) was added dropwise. The mixture was stirred under nitrogen for 30 min. Then TMSCl (0.52 mL, 1.05 eq.) was added. The reaction mixture was stirred at room temperature for 3 h, at the end of which the reaction mixture is partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 908.6 mg title product as a white oil. 91% yield.

According general procedure C to get compound 1f.

**trimethyl(phenanthren-9-yl)silane(1n)**

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.74 – 8.71 (m, 1H), 8.64 (d, J = 8.4 Hz, 1H), 8.16 – 8.13 (m, 1H), 7.95 (s, 1H), 7.87 (dd, J = 7.8, 1.5 Hz, 1H), 7.66 – 7.55 (m, 4H), 0.51 (s, 9H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 136.37, 134.98, 134.63, 130.84, 130.68, 129.79, 128.60, 128.54, 126.84, 126.31, 125.92, 125.70, 123.04, 122.13, 0.00.

**HRMS (EI) m/z (M+•)** Calcd for $C_{17}H_{18}Si$: 250.1178; Found:250.11693.

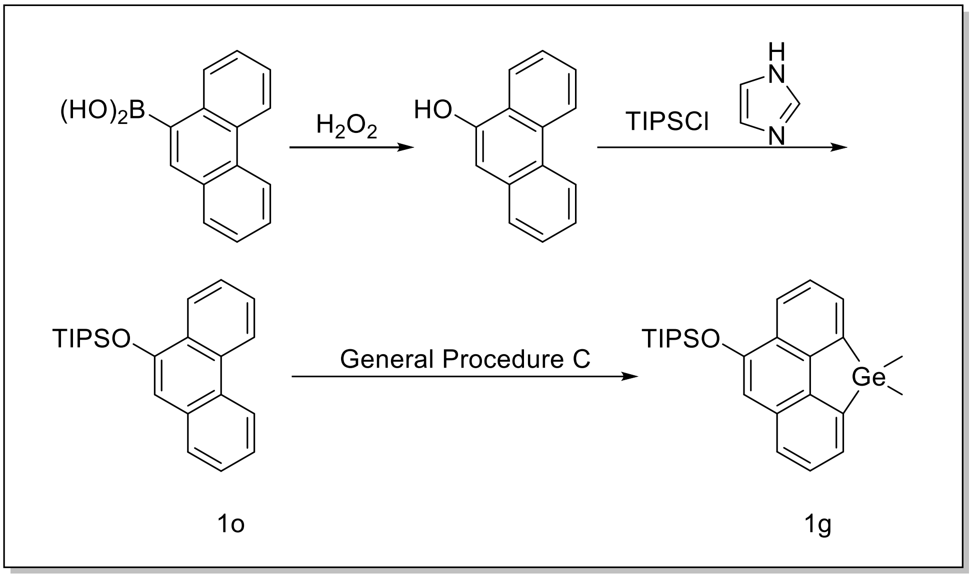
**(4,4-dimethyl-4H-phenanthro[4,5-bcd]germol-8-yl)trimethylsilane(1f)**

According to General Procedure C, using 2-phenylphenanthrene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 25% yield, 174.9 mg, white oil.

**$^1$H NMR (500 MHz, CDCl$_3$)** δ 8.11 (dd, *J* = 8.1, 1.0 Hz, 1H), 7.96 (s, 1H), 7.86 – 7.83 (m, 3H), 7.64 – 7.57 (m, 2H), 0.70 (s, 6H), 0.52 (s, 9H).

**$^{13}$C NMR (126 MHz, CDCl$_3$)** δ 141.15, 140.98, 139.99, 139.64, 136.51, 133.99, 132.80, 130.53, 129.63, 129.14, 127.65, 127.17, 126.67, 126.24, 0.00, -2.52.

**HRMS (EI) m/z (M+•)** Calcd for $C_{19}H_{22}GeSi$: 352.0703; Found:352.06921.



9-Phenanthracenylboronic acid (1.1103 g, 5 mmol, 1 eq.) was placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The dry THF (10 mL) was added under argon and cooled to 0℃, then $H_2O_2$ ( 0.6 mL) was added dropwise. The reaction mixture was stirred at room temperature for 2 h, at the end of which the reaction mixture is partitioned in DCM and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product without purification .

The above compound (0.8239 g, 2 mmol, 1 eq.) and imidazole (0.3404 g, 5 mmol, 2.5 eq.) was placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. The DCM (25 mL) was added under argon, then TIPSCl (0.51 mL, 1.2 eq.) was added dropwise. The reaction mixture was stirred at room temperature for 1 h, at the end of which the reaction mixture is partitioned in $CH_2Cl_2$ and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 322.5 mg title product as a white oil. 46% yield. According general procedure C to get compound 1g.

**triisopropyl(phenanthren-9-yloxy)silane(1o)**

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.69 (dd, J = 7.6, 1.7 Hz, 1H), 8.63 (dd, J = 7.6, 1.7 Hz, 1H), 8.47 – 8.41 (m, 1H), 7.78 – 7.73 (m, 1H), 7.73 – 7.64 (m, 2H), 7.54 (pd, J = 7.0, 1.6 Hz, 2H), 7.11 (s, 1H), 1.52 (dt, J = 14.9, 7.4 Hz, 3H), 1.24 (d, J = 7.5 Hz, 18H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 150.10, 133.05, 131.69, 128.59, 127.11, 126.91, 126.75, 126.70, 126.34, 124.27, 123.30, 122.61, 122.53, 109.78, 18.24, 13.17.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{23}H_{31}OSi$: 351.2144; Found: 351.2143.

**((4,4-dimethyl-4H-phenanthro[4,5-bcd]germol-8-yl)oxy)triisopropylsilane(1g)**

According to General Procedure C, using triisopropyl(phenanthren-9-yloxy)silane, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 31% yield, 278.9 mg, white oil.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.28 (dd, J = 8.1, 1.1 Hz, 1H), 7.89 (dd, J = 6.8, 1.1 Hz, 1H), 7.73 (d, J = 7.3 Hz, 2H), 7.66 (dd, J = 8.1, 6.7 Hz, 1H), 7.60 – 7.55 (m, 1H), 7.09 (s, 1H), 1.52 (dt, J = 14.9, 7.5 Hz, 3H), 1.24 (d, J = 7.5 Hz, 18H), 0.74 (s, 6H).

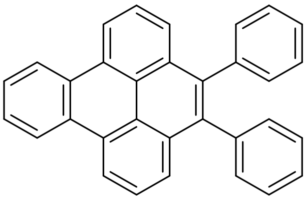
**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 153.14, 144.18, 142.23, 141.83, 139.25, 132.92, 132.80, 129.95, 129.17, 128.93, 128.77, 127.97, 124.98, 110.97, 20.43, 15.34.

**HRMS (EI) m/z (M+•)** Calcd for $C_{25}H_{34}GeOSi$: 452.1591; Found:452.15848.

## 2.4 Preparation and characterization of multisubstitued pyrene

### General Procedure D:

Phenangermole (0.15 mmol)、diphenylethyne (0.0178g, 0.1 mmol)、$PdBr_2$ (0.0014g, 5 umol, 5 mol%)、$AgPF_6(collidine)_2$ (0.0050g, 10 umol, 10 mol%) and o-chloranil(0.0507g, 0.2 mmol, 2 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, DCE (0.5 mL) was added. The reaction mixture was stirred at 120℃ for 2 h before being quenched with water. The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography on silica gel.



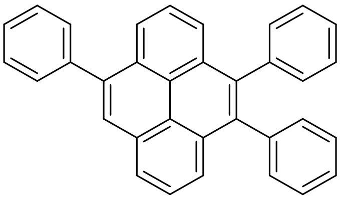
**4,5-diphenylbenzo[e]pyrene(4a)**

According to General Procedure D, using 4,4-dimethyl-4H-triphenyleno[1,12-bcd]germole at 100℃ for 12 h, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 41% yield, 16.6 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.95 (dd, J = 7.9, 1.1 Hz, 2H), 8.90 (dt, J = 6.3, 3.5 Hz, 2H), 7.94 (t, J = 7.9 Hz, 2H), 7.85 (dd, J = 8.0, 1.1 Hz, 2H), 7.78 (dt, J = 6.2, 3.4 Hz, 2H), 7.32 – 7.24 (m, 11H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 139.69, 137.70, 131.79, 131.14, 130.19, 129.03, 127.72, 127.58, 126.62, 126.07, 125.96, 123.98, 123.83, 120.33.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{32}H_{21}$: 405.1643; Found: 405.1646.



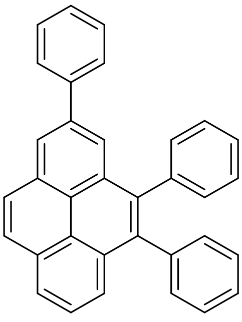
**4,5,9-triphenylpyrene(4b)**

According to General Procedure D, using 4,4-dimethyl-8-phenyl-4H-phenanthro[4,5-bcd]germole, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 67% yield, 28.7 mg, white solid.

**$^1$H NMR (500 MHz, CDCl$_3$)** δ 8.21 (td, J = 6.2, 5.4, 1.5 Hz, 2H), 8.09 (s, 1H), 7.92 (t, J = 7.7 Hz, 1H), 7.88 – 7.81 (m, 3H), 7.72 – 7.68 (m, 2H), 7.58 (t, J = 7.4 Hz, 2H), 7.53 – 7.48 (m, 1H), 7.33 – 7.22 (m, 10H).

**$^{13}$C NMR (126 MHz, CDCl$_3$)** δ 140.97, 139.64, 139.53, 139.51, 137.96, 137.76, 131.61, 131.31, 131.18, 131.17, 130.65, 130.36, 130.18, 128.47, 127.90, 127.75, 127.74, 127.57, 126.66, 126.17, 125.74, 125.27, 125.09, 124.84, 124.58, 123.98, 123.80.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{34}H_{23}$: 431.1800; Found: 431.1780.



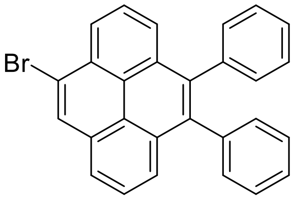
**2,4,5-triphenylpyrene(4c)**

According to General Procedure D, using 4,4-dimethyl-2-phenyl-4H-phenanthro[4,5-bcd]germole, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 43% yield, 18.5 mg, yellow solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.35 (d, J = 1.7 Hz, 1H), 8.15 – 8.06 (m, 3H), 8.00 (d, J = 1.7 Hz, 1H), 7.83 (t, J = 7.6 Hz, 1H), 7.79 – 7.75 (m, 1H), 7.62 (dd, J = 7.5, 1.6 Hz, 2H), 7.39 (t, J = 7.5 Hz, 2H), 7.30 (d, J = 7.2 Hz, 1H), 7.28 – 7.18 (m, 10H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 141.65, 139.51, 139.35, 138.79, 138.24, 137.98, 131.86, 131.49, 131.39, 131.16, 131.02, 128.90, 128.05, 127.86, 127.79, 127.73, 127.58, 127.31, 126.72, 126.65, 125.94, 125.24, 125.09, 124.14, 123.84, 123.67, 123.49.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{34}H_{22}$: 430.1722 ; Found: 430.17130 .



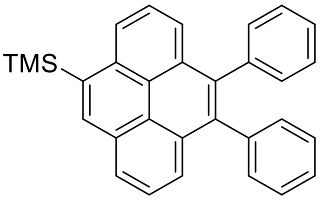
**9-bromo-4,5-diphenylpyrene(4d)**

According to General Procedure D, using 8-bromo-4,4-dimethyl-4H-phenanthro[4,5-bcd]germole, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 45% yield, 19.3 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.63 (d, J = 7.7 Hz, 1H), 8.50 (s, 1H), 8.13 (d, J = 7.1 Hz, 1H), 7.99 (t, J = 7.9 Hz, 1H), 7.89 (dt, J = 11.0, 6.7 Hz, 3H), 7.35 – 7.20 (m, 12H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 139.29, 139.18, 137.91, 137.87, 131.52, 131.41, 131.14, 131.09, 131.08, 130.92, 129.55, 127.80, 127.76, 126.79, 126.77, 126.45, 126.42, 126.12, 125.56, 125.13, 125.07, 124.67, 123.37, 122.47.

**HRMS (EI) m/z (M+•)** Calcd for $C_{28}H_{17}Br$: 432.0514; Found:432.05044.



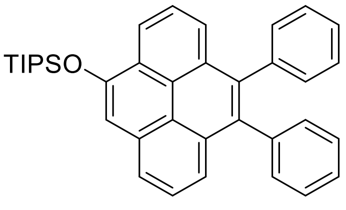
**(9,10-diphenylpyren-4-yl)trimethylsilane(4e)**

According to General Procedure D, using (4,4-dimethyl-4H-phenanthro[4,5-bcd]germol-8-yl)trimethylsilane, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 79% yield, 33.5 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.44 (dd, J = 7.7, 1.1 Hz, 1H), 8.36 (s, 1H), 8.23 – 8.18 (m, 1H), 7.92 (q, J = 7.5 Hz, 2H), 7.84 (dt, J = 7.9, 1.3 Hz, 2H), 7.33 – 7.23 (m, 10H), 0.62 (s, 9H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 139.40, 139.24, 137.65, 137.21, 135.34, 133.61, 131.63, 130.87, 130.84, 129.89, 127.41, 127.39, 126.29, 125.59, 125.38, 125.13, 125.01, 124.98, 124.30, 123.93, 0.00.

**HRMS (EI) m/z (M+•)** Calcd for $C_{31}H_{26}Si$: 426.1804; Found: 426.17980.



**((9,10-diphenylpyren-4-yl)oxy)triisopropylsilane(4f)**

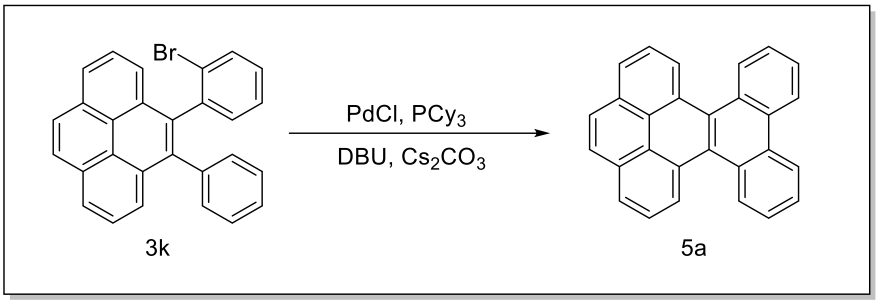
According to General Procedure D, using ((4,4-dimethyl-4H-phenanthro[4,5-bcd]germol-8-yl)oxy)triisopropylsilane, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 50% yield, 26.4 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.66 (dd, J = 7.7, 1.2 Hz, 1H), 8.04 (dd, J = 7.6, 1.0 Hz, 1H), 7.93 (t, J = 7.8 Hz, 1H), 7.88 – 7.81 (m, 2H), 7.68 (dd, J = 7.9, 1.1 Hz, 1H), 7.33 – 7.20 (m, 12H), 1.57 (q, J = 7.5 Hz, 3H), 1.26 (d, J = 7.5 Hz, 18H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 150.90, 139.63, 137.74, 137.63, 132.04, 131.33, 131.18, 127.68, 127.62, 126.57, 126.54, 126.07, 125.52, 125.35, 123.62, 122.79, 120.80, 120.34, 110.08, 18.27, 14.16, 13.23.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{37}H_{38}OSi$: 527.2770; Found: 527.2756.

## 3. Aromatization reaction



Compound 3k (0.0433g, 0.1 mmol)、PdCl$_2$ (0.0071g, 0.04 mmol, 0.4 eq.)、PCy$_3$ (0.0229g, 0.08 mmol, 0.8 eq.) and Cs$_2$CO$_3$ (0.0665g, 0.2 mmol, 2 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, DBU (30 uL, 0.2 mmol, 2 eq.) and toluene (2.5 mL) was added. The reaction mixture was stirred at 100℃ for 48 h before being quenched with water.[6] The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over Na$_2$SO$_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography on silica to give 24.9 mg title product as a yellow solid. 71% yield.

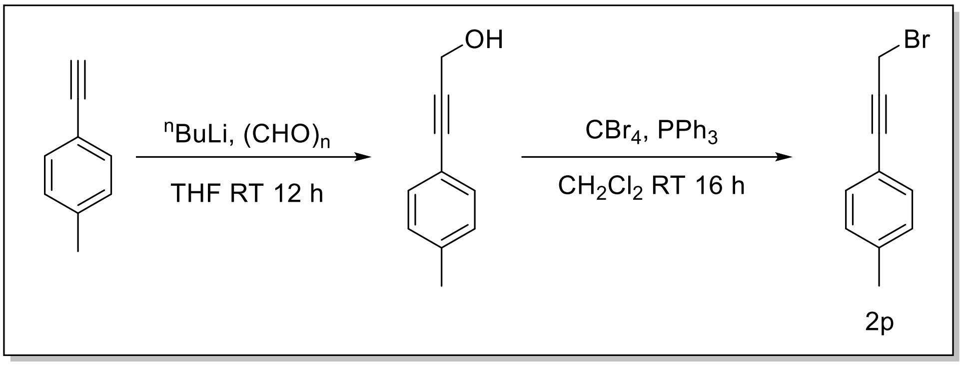
**tribenzo[f,ij,no]tetraphene (5a)**

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.95 (d, J = 8.0 Hz, 1H), 8.85 (d, J = 8.2 Hz, 1H), 8.70 (d, J = 8.1 Hz, 1H), 8.15 (d, J = 7.6 Hz, 1H), 8.06 (s, 1H), 7.98 (t, J = 7.8 Hz, 1H), 7.69 – 7.63 (m, 1H), 7.62 – 7.56 (m, 1H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 131.22, 130.93, 129.73, 128.75, 128.40, 128.03, 127.36, 126.86, 126.68, 126.30, 125.57, 125.15, 124.72, 123.68.

**HRMS (EI) m/z (M+•)** Calcd for $C_{28}H_{16}$: 352.1252; Found: 352.12462.

## 4. Conversion of propargyl bromide



A 25 mL Schlenk tube was charged with 4-ethynyltoluene (580.8 mg, 0.65mL, 5 mmol) and dry THF (5 mL). The solution was cooled to −78 ℃ and n-BuLi (2.4 M in Hexane, 2.3 mL, 5.5 mmol) was added dropwise. Paraformaldehyde (0.18 g, 6 mmol) was added under argon. The reaction was stirred at ambient temperature for 12 h and acidified with HCl at 0°C.[7] The reaction mixture was extracted with EtOAc/$H_2O$. The combined organic layer was washed with brine, dried over $Na_2SO_4$, filtered, and concentrated under vacuum. The crude 3-(4-methylphenyl)prop-2-yn-1-ol (white solid) was kept for a few hours under vacuum and used for next step without further purification.

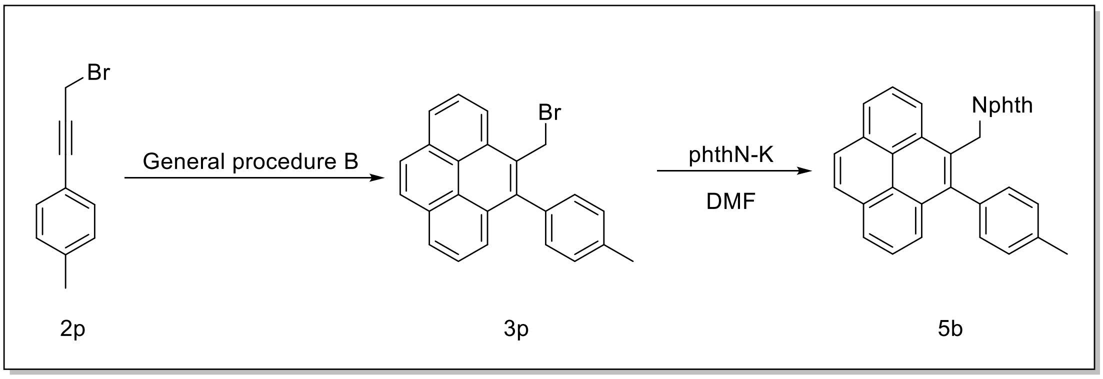
The propargyl alcohol (estimated as 5 mmol) in dry $CH_2Cl_2$ (10 mL) was loaded into a 25 mL Schlenk tube. Carbon tetrabromide (2.03 g, 6 mol) was added under argon. The homogeneous solution was cooled to 0°C and $PPh_3$ (1.57 g, 6 mmol) was added in small portions under argon (highly exothermic). The reaction mixture was stirred at room temperature for 16 h. EtOH (5 mL) was added to quench the reaction, followed by ice-water . The combined organic layers were washed with brine, and then dried over Na2SO4. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography (petroleum ether) to give 894.1 mg title product as a yellow oil. 94% yield.

**1-(3-bromoprop-1-yn-1-yl)-4-methylbenzene(2p)**

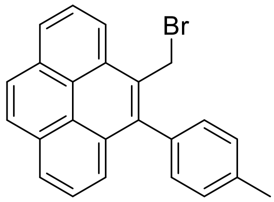
**$^1$H NMR (400 MHz, CDCl$_3$)** δ 7.34 (d, J = 8.2 Hz, 2H), 7.13 (d, J = 7.9 Hz, 2H), 4.17 (s, 2H), 2.36 (s, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 139.13, 131.80, 129.12, 119.03, 86.99, 83.59, 21.58, 15.70.

**HRMS (EI) m/z (M+•)** Calcd for $C_{10}H_9Br$: 207.9888; Found: 207.98819.



Compound 3p (0.0385g, 0.1 mmol. 1 eq.) and Potassium phthalimide (0.0189g, 0.1 mmol, 1 eq.) were placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, DMF (0.5 mL) was added.[8] The reaction mixture was stirred at room temperature for 4 h before being quenched with water. The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography on silica to give 30.3 mg title product as a white solid. 67% yield.



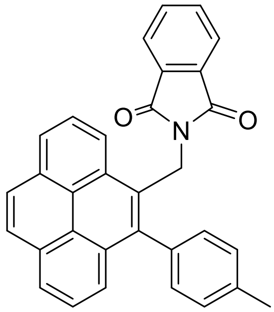
**4-(bromomethyl)-5-(p-tolyl)pyrene (3p)**

According to General Procedure B, using 1-(3-bromoprop-1-yn-1-yl)-4-methylbenzene, the title compound was isolated by silica gel column chromatography using petroleum ether as eluent in 37% yield, 14.3 mg, white solid.

**$^1$H NMR (400 MHz, CDCl$_3$)** δ 8.54 (dd, J = 8.0, 1.1 Hz, 1H), 8.25 (dd, J = 7.7, 1.1 Hz, 1H), 8.18 (dd, J = 7.6, 1.2 Hz, 1H), 8.19 – 8.05 (m, 3H), 7.92 – 7.79 (m, 1H), 7.77 – 7.66 (m, 1H), 7.44 – 7.32 (m, 4H), 5.04 (s, 2H), 2.53 (s, 3H).

**$^{13}$C NMR (101 MHz, CDCl$_3$)** δ 140.44, 137.67, 135.36, 131.49, 131.28, 130.90, 130.06, 129.78, 129.34, 129.29, 128.92, 127.61, 127.57, 127.35, 126.33, 125.97, 125.90, 125.88, 125.77, 125.49, 124.67, 124.55, 122.09, 42.60, 21.47.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{24}H_{18}Br$: 385.0592; Found:385.0580.



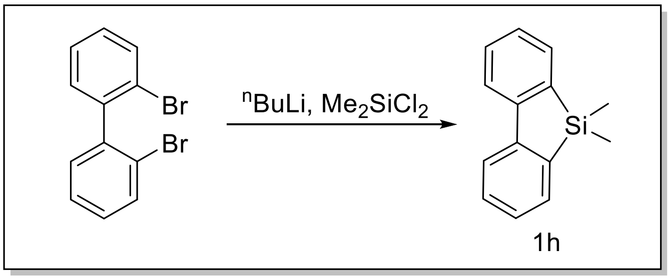
**2-((5-(p-tolyl)pyren-4-yl)methyl)isoindoline-1,3-dione (5b)**

**$^1$H NMR (500 MHz, CDCl$_3$)** δ 8.34 (dd, J = 8.0, 1.0 Hz, 1H), 8.16 (ddd, J = 7.7, 5.3, 1.0 Hz, 2H), 8.10 – 8.05 (m, 2H), 7.97 (t, J = 7.8 Hz, 1H), 7.87 (t, J = 7.7 Hz, 1H), 7.74 (dd, J = 7.9, 1.1 Hz, 1H), 7.70 (dd, J = 5.5, 3.1 Hz, 2H), 7.61 – 7.57 (m, 2H), 7.56 – 7.54 (m, 2H), 7.38 (d, J = 7.7 Hz, 2H), 5.40 (s, 2H), 2.49 (s, 3H).

**$^{13}$C NMR (126 MHz, CDCl$_3$)** δ 168.22, 141.08, 137.21, 136.22, 133.80, 131.86, 131.74, 131.56, 131.07, 130.87, 129.35, 129.00, 127.42, 127.38, 127.20, 126.24, 125.78, 125.64, 125.49, 125.09, 124.54, 124.35, 123.07, 121.32, 39.50, 21.44.

**HRMS (ESI) *m/z* ([M+H]$^+$)** Calcd for $C_{32}H_{22}NO_2$: 452.1651; Found:452.1642.

## 5. Competition experiments



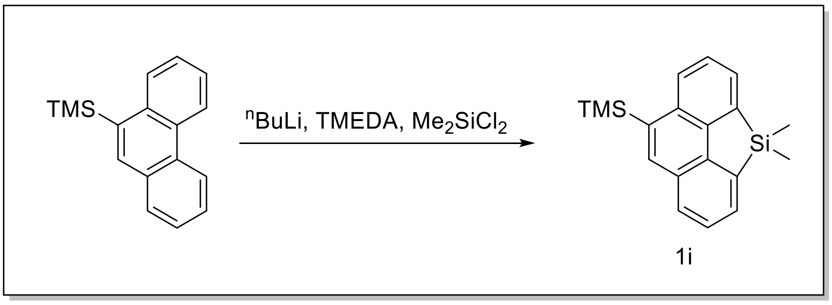
2,2'-Dibromo-1,1'-biphenyl (0.6367 g, 2 mmol, 1 eq.) was dissolved in dry THF (10 mL) and cooled to -78 °C. n-BuLi (2.4 M in Hexane, 1.7 mL, 4 mmol, 2 eq.) was added dropwise. The mixture was stirred under nitrogen for 1 h. Then $Me_2SiCl_2$ (0.24 mL, 2.4 mmol, 1.2 eq.) was added. The reaction mixture was stirred at room temperature for 2 h,[9] at the end of which the reaction mixture is partitioned in EtOAc and distilled water (10 mL each). The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The crude product is purified by silica gel column chromatography to give 397.7 mg title product as a white solid. 95% yield.

**5,5-dimethyl-5H-dibenzo[b,d]silole(2h)**

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 7.84 (d, J = 7.8 Hz, 2H), 7.64 (ddd, J = 7.0, 1.4, 0.7 Hz, 2H), 7.44 (td, J = 7.6, 1.4 Hz, 2H), 7.29 (td, J = 7.3, 0.9 Hz, 2H), 0.43 (s, 6H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 147.78, 138.92, 132.73, 130.17, 127.35, 120.82, -3.22.

**HRMS (EI) m/z (M+•)** Calcd for $C_{14}H_{14}Si$: 210.0865; Found: 210.08557.



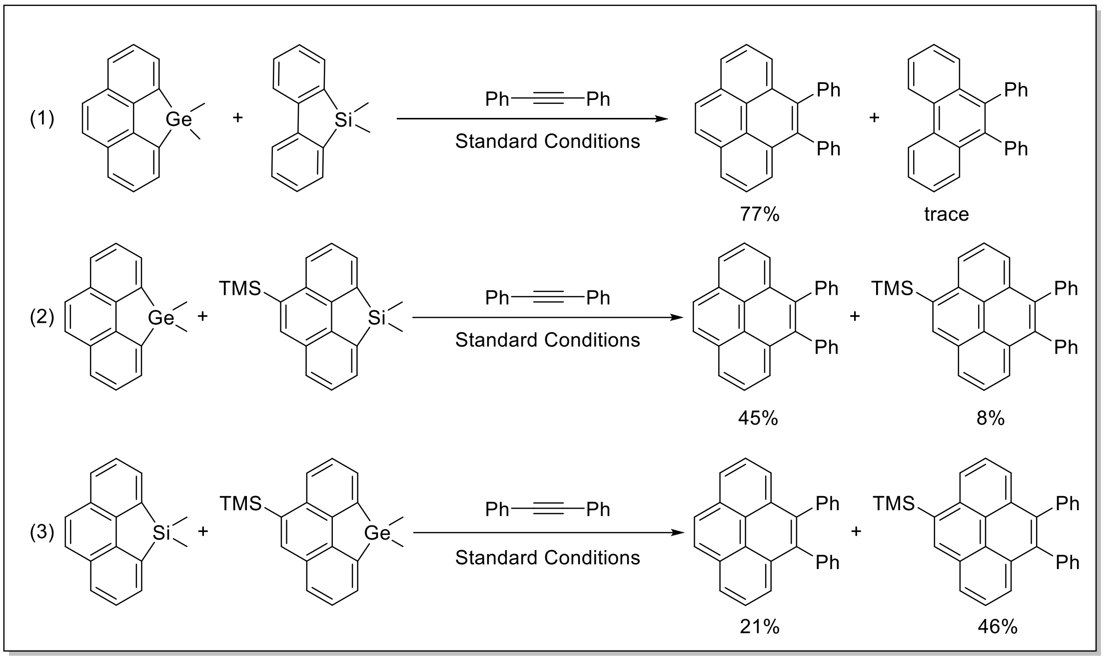
Trimethyl(phenanthren-9-yl)silane (1.0017g, 4 mmol, 1 eq.) and TMEDA (1.44mL, 9.6 mmol, 2.4 eq) were placed into a Schlenk tube equipped with a stir bar. the tube was vacuumed and backfilled with argon for three cycles. Then, a solution of n-BuLi (2.4 M in Hexane, 3.68mL, 8.8 mmol, 2.2 eq.) was added dropwise at 0 °C. After stirring at 60 °C for 3 h, anhydrous THF (9.6 mL) was added, $Me_2SiCl_2$ (0.67mL, 6.8 mmol, 1.7 eq.) was added at –78 °C. The reaction mixture was stirred at room temperature for 24 h before being quenched with water. The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, and then dried over $Na_2SO_4$. After filtration, the solvents were removed under reduced pressure to give the crude product. The purification by flash column chromatography on silica gel to give 245.5 mg title product as a white oil. 20% yield.

**4,4-dimethyl-8-(trimethylsilyl)-4H-phenanthro[4,5-bcd]silole(2i)**

**$^1$H NMR (400 MHz, $CDCl_3$)** δ 8.47 (dd, J = 8.2, 1.0 Hz, 1H), 8.32 (s, 1H), 8.24 – 8.17 (m, 3H), 7.95 (ddd, J = 11.8, 8.1, 6.8 Hz, 2H), 0.89 (s, 6H), 0.87 (s, 9H).

**$^{13}$C NMR (101 MHz, $CDCl_3$)** δ 142.41, 141.22, 138.57, 137.25, 136.44, 133.66, 132.19, 130.55, 129.66, 128.56, 128.16, 127.63, 126.76, 126.36, 0.00, -3.10.

**HRMS (EI) m/z (M+•)** Calcd for $C_{19}H_{22}Si_2$: 306.1260; Found: 306.12518.



Competition experiment 1 : 4,4-dimethyl-4H-phenanthro[4,5-bcd]germole (0.1 mmol)、5,5-dimethyl-5H-dibenzo[b,d]silole (0.1 mmol)、diphenylethyne(0.1 mmol)、$PdBr_2$ (5 mol%)、$AgPF_6(collidine)_2$ (10 mol%) and o-chloranil (2 eq.) was placed into a Schlenk tube equipped with a stir bar. The tube was vacuumed and backfilled with argon for three cycles. Then, DCE (0.5 mL) was added. The reaction mixture was stirred at 120℃ for 2 h before being quenched with water. Then triphenylmethane (0.1 mmol) was added, the reaction mixture was extracted with $Et_2O$. The solvents were removed under reduced pressure to give the crude product. The yield was determined by $^1$H NMR. The competition experiment 2 and 3 were same as above.

## 6. Crystal structure determination of 5b

### 6.1 Experimental procedure and crystal data of 5b

Single crystals of $C_{32}H_{21}NO_2$ (5b) were grown by slow cooling of saturated EtOAc/$Et_2O$ solution from 50 ℃ to room temperature. A suitable crystal was selected and intensity data was collected on a 'Bruker APEX-II CCD' diffractometer. The crystal was kept at 296.15 K during data collection. Using Olex2[10], the structure was solved with the SHELXT[11] structure solution program using Intrinsic Phasing and refined with the SHELXL[11] refinement package using Least Squares minimisation. **Crystal Data** for $C_{32}H_{21}NO_2$ (M =451.50 g/mol): monoclinic, space group C2/c (no. 15), a = 30.6180(6) Å, b = 14.6059(3) Å, c = 20.4158(5) Å, β = 92.2370(10)°, V = 9123.1(3) Å3, Z = 16, T = 213.00 K, μ(GaKα) = 0.410 $mm^{-1}$, Dcalc = 1.315 $g/cm^3$, 42094 reflections measured (5.026° ≤ 2Θ ≤ 109.974°), 8685 unique ($R_{int}$ = 0.1352, $R_{sigma}$ = 0.1167) which were used in all calculations. The final $R_1$ was 0.0763 (I > 2 σ (I)) and $wR_2$ was 0.2144 (all data).

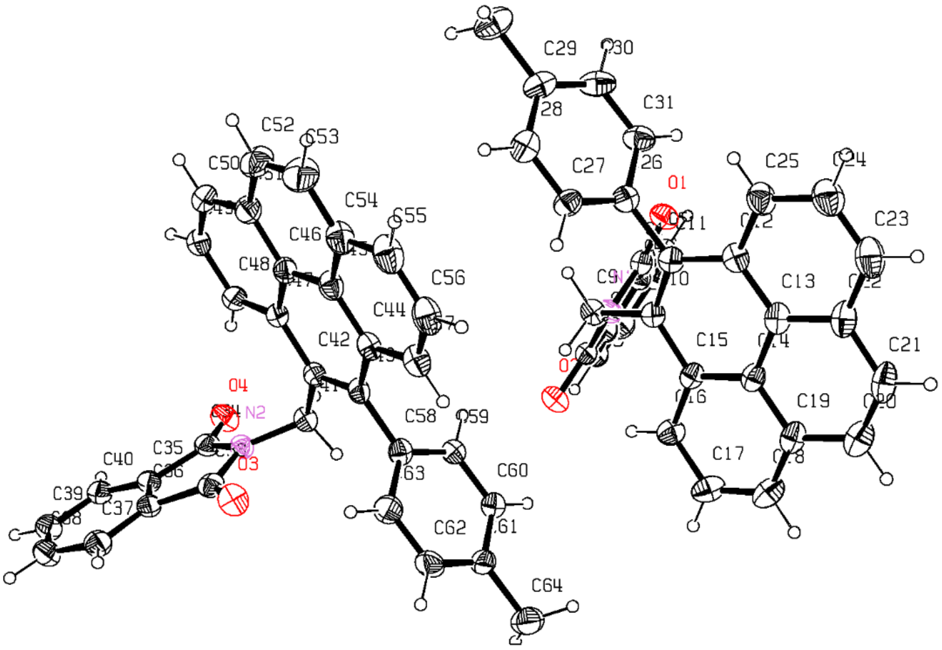


**Figure S1. X-ray Structure of compound 5b.**

**Table 1 Crystal data and structure refinement for $C_{32}H_{21}NO_2$ (5b).**

| | |
|---|---|
| Identification code | $C_{32}H_{21}NO_2$ |
| Empirical formula | $C_{32}H_{21}NO_2$ |
| Formula weight | 451.50 |
| Temperature/K | 213.00 |
| Crystal system | monoclinic |
| Space group | C2/c |
| a/Å | 30.6180(6) |
| b/Å | 14.6059(3) |
| c/Å | 20.4158(5) |
| α/° | 90 |
| β/° | 92.2370(10) |
| γ/° | 90 |
| Volume/Å$^3$ | 9123.1(3) |
| Z | 16 |
| $\rho_{calc}$g/cm$^3$ | 1.315 |
| μ/mm$^{-1}$ | 0.410 |
| F(000) | 3776.0 |
| Crystal size/mm$^3$ | 0.07 × 0.07 × 0.05 |
| Radiation | GaKα (λ = 1.34139) |
| 2Θ range for data collection/° | 5.026 to 109.974 |
| Index ranges | $-36 \le h \le 37$, $-17 \le k \le 15$, $-24 \le l \le 24$ |

| | |
|---|---|
| Reflections collected | 42094 |
| Independent reflections | 8685 [$R_{int}$ = 0.1352, $R_{sigma}$ = 0.1167] |
| Data/restraints/parameters | 8685/0/633 |
| Goodness-of-fit on $F^2$ | 1.001 |
| Final R indexes [I>=2σ (I)] | $R_1$ = 0.0763, $wR_2$ = 0.1542 |
| Final R indexes [all data] | $R_1$ = 0.2010, $wR_2$ = 0.2144 |
| Largest diff. peak/hole / e $Å^{-3}$ | 0.20/-0.27 |

**Table 2 Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$) for $C_{32}H_{21}NO_2$ (5b). $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| O1 | 3300.4(8) | 7788.3(18) | 6040.9(13) | 55.9(8) |
| O2 | 2893.0(8) | 8978.1(18) | 4040.0(13) | 54.1(7) |
| N1 | 3213.6(9) | 8379(2) | 4990.3(14) | 41.8(8) |
| C1 | 3075.3(12) | 8162(3) | 5620(2) | 44.3(10) |
| C2 | 2868.2(12) | 8749(2) | 4609(2) | 41.0(9) |
| C3 | 2487.1(11) | 8801(2) | 5032.3(18) | 39.6(9) |
| C4 | 2612.6(11) | 8465(2) | 5643.1(18) | 38.2(9) |
| C5 | 2329.9(13) | 8446(3) | 6144(2) | 50.4(10) |
| C6 | 1907.2(13) | 8773(3) | 6017(2) | 55.7(11) |
| C7 | 1781.7(13) | 9105(3) | 5401(2) | 52.3(11) |
| C8 | 2070.4(12) | 9119(2) | 4891(2) | 46.3(10) |
| C9 | 3640.5(11) | 8122(3) | 4736.1(18) | 44.1(10) |
| C10 | 4023.7(11) | 8576(2) | 5098.8(17) | 39.9(9) |
| C11 | 4299.0(11) | 8065(3) | 5492.7(18) | 43.2(10) |
| C12 | 4639.3(11) | 8498(3) | 5896.5(18) | 46.4(10) |
| C13 | 4698.7(11) | 9467(3) | 5842.7(19) | 47.0(10) |
| C14 | 4433.6(12) | 9981(3) | 5389.0(19) | 43.3(10) |
| C15 | 4091.2(11) | 9557(2) | 5015.8(18) | 41.6(10) |
| C16 | 3846.0(12) | 10075(3) | 4565(2) | 49.9(11) |
| C17 | 3929.7(14) | 10999(3) | 4492(2) | 63.7(13) |
| C18 | 4250.5(14) | 11427(3) | 4866(2) | 63.3(12) |
| C19 | 4507.8(12) | 10933(3) | 5318(2) | 51.3(11) |
| C20 | 4841.6(14) | 11357(3) | 5716(2) | 66.3(13) |

**Table 2 Fractional Atomic Coordinates (×$10^4$) and Equivalent Isotropic Displacement Parameters (Å$^2$×$10^3$) for $C_{32}H_{21}NO_2$ (5b). $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| C21 | 5089.1(14) | 10873(3) | 6151(2) | 69.3(14) |
| C22 | 5029.0(13) | 9908(3) | 6230(2) | 56.4(12) |
| C23 | 5282.8(14) | 9399(4) | 6684(2) | 74.3(14) |
| C24 | 5220.4(14) | 8469(4) | 6734(2) | 71.9(14) |
| C25 | 4909.0(13) | 8021(3) | 6342(2) | 60.0(12) |
| C26 | 4280.6(11) | 7033(3) | 5504(2) | 45.1(10) |
| C27 | 4437.7(12) | 6557(3) | 4979(2) | 49.3(10) |
| C28 | 4451.2(12) | 5609(3) | 4978(2) | 55.8(11) |
| C29 | 4307.7(12) | 5104(3) | 5502(2) | 52.7(11) |
| C30 | 4147.3(13) | 5584(3) | 6022(2) | 63.2(13) |
| C31 | 4131.7(13) | 6531(3) | 6031(2) | 56.6(11) |
| C32 | 4331.2(13) | 4071(3) | 5499(3) | 75.3(15) |
| O3 | 3445.6(9) | 7047.3(19) | 747.5(14) | 60.4(8) |
| O4 | 2047.5(9) | 6280.5(19) | 1230.2(13) | 58.9(8) |
| N2 | 2775.8(10) | 6658(2) | 1158.0(15) | 43.7(8) |
| C33 | 3072.9(13) | 6764(2) | 667(2) | 46.8(10) |
| C34 | 2365.9(14) | 6386(3) | 900(2) | 48.0(10) |
| C35 | 2417.4(13) | 6266(2) | 186.0(19) | 44.7(10) |
| C36 | 2843.1(13) | 6478(2) | 43.8(19) | 45.0(10) |
| C37 | 2992.6(14) | 6421(3) | -580(2) | 55.2(11) |
| C38 | 2697.6(17) | 6117(3) | -1063(2) | 65.7(13) |
| C39 | 2268.2(16) | 5900(3) | -926(2) | 62.2(12) |
| C40 | 2119.5(14) | 5979(2) | -297(2) | 52.2(11) |
| C41 | 2860.8(12) | 6911(3) | 1846.1(17) | 45.5(10) |
| C44 | 3984.7(12) | 6471(3) | 2685.3(18) | 44.7(10) |
| C42 | 3252.3(12) | 6424(3) | 2168.9(17) | 41.6(9) |
| C47 | 3238.1(12) | 5444(3) | 2245.7(18) | 42.8(10) |
| C43 | 3606.9(11) | 6929(3) | 2381.5(17) | 41.0(9) |
| C45 | 3978.5(12) | 5509(3) | 2757.7(18) | 48.3(10) |
| C58 | 3620.4(11) | 7951(3) | 2331.0(19) | 43.2(10) |
| C46 | 3607.9(13) | 4991(3) | 2543.2(18) | 46.1(10) |
| C48 | 2881.1(13) | 4910(3) | 2043.8(19) | 49.0(11) |
| C49 | 2884.1(14) | 3967(3) | 2108.5(19) | 56.7(12) |
| C50 | 3240.3(15) | 3531(3) | 2395(2) | 61.8(12) |

**Table 2 Fractional Atomic Coordinates (×$10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2$×$10^3$) for $C_{32}H_{21}NO_2$ (5b). $U_{eq}$ is defined as 1/3 of the trace of the orthogonalised $U_{IJ}$ tensor.**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| C51 | 3601.6(14) | 4025(3) | 2626(2) | 55.9(11) |
| C52 | 3975.7(16) | 3592(3) | 2930(2) | 75.7(15) |
| C53 | 4330.1(16) | 4084(3) | 3131(3) | 81.8(16) |
| C54 | 4349.4(13) | 5054(3) | 3053(2) | 60.2(12) |
| C55 | 4715.9(14) | 5572(4) | 3252(2) | 69.2(13) |
| C56 | 4717.0(13) | 6510(4) | 3184(2) | 64.1(13) |
| C57 | 4358.3(12) | 6954(3) | 2906.3(19) | 55.9(11) |
| C59 | 3406.3(12) | 8482(3) | 2778.1(18) | 44.8(10) |
| C60 | 3423.5(12) | 9424(3) | 2757.8(19) | 48.2(10) |
| C61 | 3658.0(13) | 9869(3) | 2296(2) | 48.9(10) |
| C62 | 3870.4(14) | 9337(3) | 1849(2) | 61.1(12) |
| C63 | 3855.3(13) | 8397(3) | 1861(2) | 57.5(11) |
| C64 | 3678.5(15) | 10903(3) | 2278(2) | 74.6(14) |

**Table 3 Anisotropic Displacement Parameters ($Å^2$×$10^3$) for $C_{32}H_{21}NO_2$ (5b). The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| O1 | 52.6(17) | 64.4(19) | 50.1(18) | 9.5(15) | -5.1(14) | 1.4(14) |
| O2 | 52.5(17) | 69(2) | 41.1(17) | 5.3(14) | -0.1(14) | 1.2(14) |
| N1 | 36.6(18) | 51(2) | 38.5(19) | 5.4(16) | 3.5(15) | 1.0(15) |
| C1 | 47(2) | 42(2) | 44(3) | -5(2) | -2(2) | -4.4(19) |
| C2 | 45(2) | 38(2) | 40(2) | -0.6(19) | 1(2) | -5.2(18) |
| C3 | 37(2) | 34(2) | 47(3) | -2.8(19) | 1.1(19) | -6.4(17) |
| C4 | 39(2) | 36(2) | 41(2) | -2.9(18) | 4.7(19) | -5.6(17) |
| C5 | 55(3) | 49(3) | 47(3) | -2(2) | 4(2) | -6(2) |
| C6 | 53(3) | 56(3) | 59(3) | -7(2) | 18(2) | -5(2) |
| C7 | 44(2) | 45(3) | 68(3) | -2(2) | 2(2) | -4.8(19) |
| C8 | 49(3) | 42(3) | 48(3) | -0.5(19) | 1(2) | -8.5(19) |
| C9 | 39(2) | 51(3) | 42(2) | -0.5(19) | 2.0(19) | 0.7(19) |
| C10 | 35(2) | 44(2) | 41(2) | -2.0(19) | 4.3(18) | -0.5(18) |
| C11 | 39(2) | 47(3) | 44(2) | -1(2) | 6.7(19) | -1.6(19) |
| C12 | 38(2) | 56(3) | 45(3) | -5(2) | 3.0(19) | 5(2) |
| C13 | 33(2) | 56(3) | 52(3) | -12(2) | 6(2) | -0.8(19) |

**Table 3 Anisotropic Displacement Parameters (Å$^2$×10$^3$) for $C_{32}H_{21}NO_2$ (5b). The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| C14 | 40(2) | 43(3) | 47(3) | -10(2) | 11(2) | -1.9(19) |
| C15 | 38(2) | 40(3) | 47(2) | -4.8(19) | 6.5(19) | -2.8(18) |
| C16 | 44(2) | 48(3) | 58(3) | 4(2) | 0(2) | 2(2) |
| C17 | 61(3) | 47(3) | 83(4) | 12(3) | 5(3) | 3(2) |
| C18 | 56(3) | 46(3) | 88(4) | -2(3) | 12(3) | -7(2) |
| C19 | 40(2) | 53(3) | 61(3) | -15(2) | 12(2) | -8(2) |
| C20 | 56(3) | 58(3) | 85(4) | -17(3) | 11(3) | -8(2) |
| C21 | 52(3) | 75(4) | 80(4) | -33(3) | 0(3) | -16(3) |
| C22 | 42(2) | 65(3) | 62(3) | -22(2) | 2(2) | -2(2) |
| C23 | 53(3) | 95(4) | 74(4) | -20(3) | -13(3) | -3(3) |
| C24 | 56(3) | 87(4) | 71(3) | -6(3) | -14(3) | 6(3) |
| C25 | 52(3) | 66(3) | 61(3) | -6(2) | -12(2) | 5(2) |
| C26 | 36(2) | 48(3) | 51(3) | 1(2) | -1.6(19) | -0.5(19) |
| C27 | 48(2) | 43(3) | 57(3) | 1(2) | 10(2) | -3(2) |
| C28 | 42(2) | 56(3) | 69(3) | -7(2) | 5(2) | -1(2) |
| C29 | 31(2) | 45(3) | 82(3) | 9(3) | -1(2) | 0.0(19) |
| C30 | 53(3) | 61(3) | 77(3) | 26(3) | 16(3) | 8(2) |
| C31 | 58(3) | 58(3) | 54(3) | 12(2) | 12(2) | 7(2) |
| C32 | 54(3) | 52(3) | 119(4) | 9(3) | 3(3) | -5(2) |
| O3 | 44.0(17) | 75(2) | 62(2) | -2.0(15) | 2.3(15) | 3.7(15) |
| O4 | 49.7(17) | 76(2) | 50.7(18) | 6.1(15) | 0.8(15) | -9.9(15) |
| N2 | 44.6(19) | 46(2) | 40(2) | 1.0(16) | -1.3(16) | 1.5(16) |
| C33 | 48(3) | 40(3) | 53(3) | 3(2) | 4(2) | 11(2) |
| C34 | 51(3) | 45(3) | 47(3) | 7(2) | -8(2) | 1(2) |
| C35 | 53(3) | 40(2) | 40(2) | 3.6(19) | -6(2) | 5(2) |
| C36 | 56(3) | 38(2) | 41(3) | 3.4(19) | -2(2) | 10.5(19) |
| C37 | 62(3) | 51(3) | 53(3) | 4(2) | 2(2) | 12(2) |
| C38 | 92(4) | 61(3) | 44(3) | 4(2) | 3(3) | 13(3) |
| C39 | 82(4) | 56(3) | 48(3) | 3(2) | -11(3) | 10(3) |
| C40 | 63(3) | 45(3) | 48(3) | 4(2) | -5(2) | 1(2) |
| C41 | 47(2) | 50(3) | 40(2) | -1.5(19) | 1.1(19) | 1.5(19) |
| C44 | 43(2) | 56(3) | 36(2) | -4(2) | 2.7(19) | 1(2) |
| C42 | 44(2) | 45(3) | 35(2) | -2.1(19) | -0.3(18) | 4.0(19) |
| C47 | 51(2) | 41(3) | 36(2) | -2.2(18) | -2.2(19) | 1(2) |

**Table 3 Anisotropic Displacement Parameters (Å$^2$×10$^3$) for $C_{32}H_{21}NO_2$ (5b). The Anisotropic displacement factor exponent takes the form: -2π$^2$[h$^2$a*$^2$U$_{11}$+2hka*b*U$_{12}$+…].**

| Atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| C43 | 44(2) | 45(3) | 33(2) | -6.1(18) | 3.4(18) | 2.9(19) |
| C45 | 44(2) | 58(3) | 42(2) | -8(2) | -1(2) | 9(2) |
| C58 | 37(2) | 49(3) | 44(2) | -4(2) | 1.0(19) | -4.3(19) |
| C46 | 51(3) | 47(3) | 40(2) | -5(2) | 0(2) | 5(2) |
| C48 | 55(3) | 47(3) | 45(3) | 2(2) | -5(2) | 1(2) |
| C49 | 72(3) | 53(3) | 44(3) | -3(2) | -9(2) | -5(2) |
| C50 | 85(3) | 43(3) | 57(3) | -5(2) | -5(3) | 1(2) |
| C51 | 64(3) | 52(3) | 51(3) | -4(2) | 0(2) | 16(2) |
| C52 | 82(4) | 53(3) | 91(4) | -1(3) | -18(3) | 22(3) |
| C53 | 75(4) | 72(4) | 97(4) | -2(3) | -22(3) | 36(3) |
| C54 | 55(3) | 69(3) | 55(3) | -9(2) | -11(2) | 21(2) |
| C55 | 49(3) | 95(4) | 62(3) | -15(3) | -11(2) | 15(3) |
| C56 | 45(3) | 89(4) | 57(3) | -14(3) | -7(2) | 1(3) |
| C57 | 52(3) | 68(3) | 48(3) | -6(2) | -1(2) | 5(2) |
| C59 | 50(2) | 45(3) | 40(2) | 0(2) | 3.9(19) | -1(2) |
| C60 | 55(3) | 45(3) | 45(3) | -2(2) | 1(2) | 1(2) |
| C61 | 54(3) | 44(3) | 48(3) | 3(2) | -8(2) | -9(2) |
| C62 | 69(3) | 69(3) | 45(3) | 7(2) | 12(2) | -23(3) |
| C63 | 61(3) | 59(3) | 54(3) | -1(2) | 17(2) | -7(2) |
| C64 | 86(3) | 60(3) | 77(3) | 16(3) | -11(3) | -9(3) |

**Table 4 Bond Lengths for $C_{32}H_{21}NO_2$ (5b).**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| O1 | C1 | 1.210(4) | O3 | C33 | 1.219(4) |
| O2 | C2 | 1.214(4) | O4 | C34 | 1.217(4) |
| N1 | C1 | 1.405(5) | N2 | C33 | 1.389(5) |
| N1 | C2 | 1.398(4) | N2 | C34 | 1.399(5) |
| N1 | C9 | 1.473(4) | N2 | C41 | 1.466(4) |
| C1 | C4 | 1.486(5) | C33 | C36 | 1.489(5) |
| C2 | C3 | 1.481(5) | C34 | C35 | 1.483(5) |
| C3 | C4 | 1.381(5) | C35 | C36 | 1.382(5) |
| C3 | C8 | 1.378(5) | C35 | C40 | 1.381(5) |
| C4 | C5 | 1.366(5) | C36 | C37 | 1.373(5) |

**Table 4 Bond Lengths for $C_{32}H_{21}NO_2$ (5b).**

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|---|---|---|---|---|---|
| C5 | C6 | 1.394(5) | C37 | C38 | 1.384(6) |
| C6 | C7 | 1.388(5) | C38 | C39 | 1.391(6) |
| C7 | C8 | 1.391(5) | C39 | C40 | 1.385(6) |
| C9 | C10 | 1.515(5) | C41 | C42 | 1.522(5) |
| C10 | C11 | 1.365(5) | C44 | C43 | 1.454(5) |
| C10 | C15 | 1.458(5) | C44 | C45 | 1.414(5) |
| C11 | C12 | 1.449(5) | C44 | C57 | 1.403(5) |
| C11 | C26 | 1.508(5) | C42 | C47 | 1.441(5) |
| C12 | C13 | 1.431(5) | C42 | C43 | 1.369(5) |
| C12 | C25 | 1.391(5) | C47 | C46 | 1.427(5) |
| C13 | C14 | 1.422(5) | C47 | C48 | 1.392(5) |
| C13 | C22 | 1.415(5) | C43 | C58 | 1.496(5) |
| C14 | C15 | 1.415(5) | C45 | C46 | 1.419(5) |
| C14 | C19 | 1.417(5) | C45 | C54 | 1.429(5) |
| C15 | C16 | 1.389(5) | C58 | C59 | 1.383(5) |
| C16 | C17 | 1.382(5) | C58 | C63 | 1.384(5) |
| C17 | C18 | 1.371(6) | C46 | C51 | 1.420(5) |
| C18 | C19 | 1.393(6) | C48 | C49 | 1.384(5) |
| C19 | C20 | 1.423(5) | C49 | C50 | 1.374(5) |
| C20 | C21 | 1.346(6) | C50 | C51 | 1.388(5) |
| C21 | C22 | 1.431(6) | C51 | C52 | 1.429(5) |
| C22 | C23 | 1.400(6) | C52 | C53 | 1.353(6) |
| C23 | C24 | 1.376(6) | C53 | C54 | 1.427(6) |
| C24 | C25 | 1.386(5) | C54 | C55 | 1.400(6) |
| C26 | C27 | 1.380(5) | C55 | C56 | 1.378(6) |
| C26 | C31 | 1.393(5) | C56 | C57 | 1.378(5) |
| C27 | C28 | 1.386(5) | C59 | C60 | 1.377(5) |
| C28 | C29 | 1.385(5) | C60 | C61 | 1.371(5) |
| C29 | C30 | 1.379(6) | C61 | C62 | 1.380(5) |
| C29 | C32 | 1.511(5) | C61 | C64 | 1.512(5) |
| C30 | C31 | 1.384(6) | C62 | C63 | 1.374(5) |

**Table 5 Bond Angles for $C_{32}H_{21}NO_2$ (5b).**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| C1 | N1 | C9 | 124.6(3) | C33 | N2 | C34 | 111.3(3) |
| C2 | N1 | C1 | 110.4(3) | C33 | N2 | C41 | 124.6(3) |
| C2 | N1 | C9 | 124.4(3) | C34 | N2 | C41 | 123.7(3) |
| O1 | C1 | N1 | 124.6(4) | O3 | C33 | N2 | 125.2(4) |
| O1 | C1 | C4 | 129.0(4) | O3 | C33 | C36 | 128.3(4) |
| N1 | C1 | C4 | 106.5(3) | N2 | C33 | C36 | 106.5(3) |
| O2 | C2 | N1 | 124.4(3) | O4 | C34 | N2 | 123.6(4) |
| O2 | C2 | C3 | 128.7(4) | O4 | C34 | C35 | 130.4(4) |
| N1 | C2 | C3 | 106.9(3) | N2 | C34 | C35 | 106.0(3) |
| C4 | C3 | C2 | 108.1(3) | C36 | C35 | C34 | 108.5(3) |
| C8 | C3 | C2 | 129.8(4) | C40 | C35 | C34 | 130.2(4) |
| C8 | C3 | C4 | 122.1(4) | C40 | C35 | C36 | 121.3(4) |
| C3 | C4 | C1 | 108.0(3) | C35 | C36 | C33 | 107.6(3) |
| C5 | C4 | C1 | 130.6(4) | C37 | C36 | C33 | 130.2(4) |
| C5 | C4 | C3 | 121.4(3) | C37 | C36 | C35 | 122.2(4) |
| C4 | C5 | C6 | 117.6(4) | C36 | C37 | C38 | 116.7(4) |
| C7 | C6 | C5 | 120.7(4) | C37 | C38 | C39 | 121.7(4) |
| C6 | C7 | C8 | 121.4(4) | C40 | C39 | C38 | 121.0(4) |
| C3 | C8 | C7 | 116.7(4) | C35 | C40 | C39 | 117.1(4) |
| N1 | C9 | C10 | 113.4(3) | N2 | C41 | C42 | 113.8(3) |
| C11 | C10 | C9 | 119.8(3) | C45 | C44 | C43 | 119.2(3) |
| C11 | C10 | C15 | 121.3(3) | C57 | C44 | C43 | 122.1(4) |
| C15 | C10 | C9 | 118.9(3) | C57 | C44 | C45 | 118.7(4) |
| C10 | C11 | C12 | 120.7(3) | C47 | C42 | C41 | 119.0(3) |
| C10 | C11 | C26 | 122.2(3) | C43 | C42 | C41 | 119.2(3) |
| C12 | C11 | C26 | 117.0(3) | C43 | C42 | C47 | 121.8(3) |
| C13 | C12 | C11 | 118.6(3) | C46 | C47 | C42 | 118.7(3) |
| C25 | C12 | C11 | 123.3(4) | C48 | C47 | C42 | 123.4(3) |
| C25 | C12 | C13 | 118.1(4) | C48 | C47 | C46 | 117.8(4) |
| C14 | C13 | C12 | 120.1(3) | C44 | C43 | C58 | 117.7(3) |
| C22 | C13 | C12 | 119.9(4) | C42 | C43 | C44 | 119.7(3) |
| C22 | C13 | C14 | 120.0(4) | C42 | C43 | C58 | 122.6(3) |
| C15 | C14 | C13 | 120.8(4) | C44 | C45 | C46 | 120.8(3) |
| C15 | C14 | C19 | 119.6(4) | C44 | C45 | C54 | 119.5(4) |
| C19 | C14 | C13 | 119.7(4) | C46 | C45 | C54 | 119.6(4) |

**Table 5 Bond Angles for $C_{32}H_{21}NO_2$ (5b).**

| Atom | Atom | Atom | Angle/° | Atom | Atom | Atom | Angle/° |
|---|---|---|---|---|---|---|---|
| C14 | C15 | C10 | 118.2(3) | C59 | C58 | C43 | 120.0(3) |
| C16 | C15 | C10 | 122.5(3) | C59 | C58 | C63 | 117.7(4) |
| C16 | C15 | C14 | 119.2(3) | C63 | C58 | C43 | 122.3(4) |
| C17 | C16 | C15 | 120.4(4) | C45 | C46 | C47 | 119.7(4) |
| C18 | C17 | C16 | 121.1(4) | C45 | C46 | C51 | 120.5(4) |
| C17 | C18 | C19 | 120.5(4) | C51 | C46 | C47 | 119.8(4) |
| C14 | C19 | C20 | 118.9(4) | C49 | C48 | C47 | 121.8(4) |
| C18 | C19 | C14 | 119.2(4) | C50 | C49 | C48 | 120.3(4) |
| C18 | C19 | C20 | 121.9(4) | C49 | C50 | C51 | 120.9(4) |
| C21 | C20 | C19 | 121.4(4) | C46 | C51 | C52 | 118.5(4) |
| C20 | C21 | C22 | 121.3(4) | C50 | C51 | C46 | 119.3(4) |
| C13 | C22 | C21 | 118.6(4) | C50 | C51 | C52 | 122.1(4) |
| C23 | C22 | C13 | 119.6(4) | C53 | C52 | C51 | 121.1(4) |
| C23 | C22 | C21 | 121.8(4) | C52 | C53 | C54 | 122.0(4) |
| C24 | C23 | C22 | 119.9(4) | C53 | C54 | C45 | 118.3(4) |
| C23 | C24 | C25 | 121.2(4) | C55 | C54 | C45 | 119.1(4) |
| C24 | C25 | C12 | 121.3(4) | C55 | C54 | C53 | 122.7(4) |
| C27 | C26 | C11 | 118.5(4) | C56 | C55 | C54 | 120.9(4) |
| C27 | C26 | C31 | 118.0(4) | C55 | C56 | C57 | 120.3(4) |
| C31 | C26 | C11 | 123.5(4) | C56 | C57 | C44 | 121.5(4) |
| C26 | C27 | C28 | 121.0(4) | C60 | C59 | C58 | 121.4(4) |
| C29 | C28 | C27 | 121.4(4) | C61 | C60 | C59 | 121.0(4) |
| C28 | C29 | C32 | 120.8(4) | C60 | C61 | C62 | 117.5(4) |
| C30 | C29 | C28 | 117.2(4) | C60 | C61 | C64 | 120.8(4) |
| C30 | C29 | C32 | 122.0(4) | C62 | C61 | C64 | 121.7(4) |
| C29 | C30 | C31 | 122.1(4) | C63 | C62 | C61 | 122.3(4) |
| C30 | C31 | C26 | 120.3(4) | C62 | C63 | C58 | 120.1(4) |

**Table 6 Torsion Angles for $C_{32}H_{21}NO_2$ (5b).**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| O1 | C1 | C4 | C3 | -176.0(4) | O3 | C33 | C36 | C35 | -176.0(4) |
| O1 | C1 | C4 | C5 | 3.7(7) | O3 | C33 | C36 | C37 | 3.2(7) |
| O2 | C2 | C3 | C4 | -179.9(4) | O4 | C34 | C35 | C36 | 179.5(4) |
| O2 | C2 | C3 | C8 | -0.3(6) | O4 | C34 | C35 | C40 | -1.7(7) |

**Table 6 Torsion Angles for $C_{32}H_{21}NO_2$ (5b).**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| N1 | C1 | C4 | C3 | 2.9(4) | N2 | C33 | C36 | C35 | 3.3(4) |
| N1 | C1 | C4 | C5 | -177.4(4) | N2 | C33 | C36 | C37 | -177.5(4) |
| N1 | C2 | C3 | C4 | -0.1(4) | N2 | C34 | C35 | C36 | -0.9(4) |
| N1 | C2 | C3 | C8 | 179.5(3) | N2 | C34 | C35 | C40 | 177.9(4) |
| N1 | C9 | C10 | C11 | 109.8(4) | N2 | C41 | C42 | C47 | -64.2(4) |
| N1 | C9 | C10 | C15 | -70.4(4) | N2 | C41 | C42 | C43 | 116.1(4) |
| C1 | N1 | C2 | O2 | -178.2(3) | C33 | N2 | C34 | O4 | -177.3(4) |
| C1 | N1 | C2 | C3 | 2.0(4) | C33 | N2 | C34 | C35 | 3.1(4) |
| C1 | N1 | C9 | C10 | -65.0(4) | C33 | N2 | C41 | C42 | -58.7(5) |
| C1 | C4 | C5 | C6 | -179.3(4) | C33 | C36 | C37 | C38 | 179.3(4) |
| C2 | N1 | C1 | O1 | 176.0(3) | C34 | N2 | C33 | O3 | 175.4(4) |
| C2 | N1 | C1 | C4 | -3.0(4) | C34 | N2 | C33 | C36 | -4.0(4) |
| C2 | N1 | C9 | C10 | 124.9(4) | C34 | N2 | C41 | C42 | 129.7(4) |
| C2 | C3 | C4 | C1 | -1.7(4) | C34 | C35 | C36 | C33 | -1.4(4) |
| C2 | C3 | C4 | C5 | 178.5(3) | C34 | C35 | C36 | C37 | 179.3(3) |
| C2 | C3 | C8 | C7 | -178.1(3) | C34 | C35 | C40 | C39 | -177.7(4) |
| C3 | C4 | C5 | C6 | 0.4(5) | C35 | C36 | C37 | C38 | -1.6(6) |
| C4 | C3 | C8 | C7 | 1.4(5) | C36 | C35 | C40 | C39 | 1.0(5) |
| C4 | C5 | C6 | C7 | -0.1(6) | C36 | C37 | C38 | C39 | 1.4(6) |
| C5 | C6 | C7 | C8 | 0.4(6) | C37 | C38 | C39 | C40 | -0.1(6) |
| C6 | C7 | C8 | C3 | -1.1(5) | C38 | C39 | C40 | C35 | -1.1(6) |
| C8 | C3 | C4 | C1 | 178.6(3) | C40 | C35 | C36 | C33 | 179.7(3) |
| C8 | C3 | C4 | C5 | -1.1(5) | C40 | C35 | C36 | C37 | 0.4(6) |
| C9 | N1 | C1 | O1 | 4.7(6) | C41 | N2 | C33 | O3 | 2.9(6) |
| C9 | N1 | C1 | C4 | -174.3(3) | C41 | N2 | C33 | C36 | -176.4(3) |
| C9 | N1 | C2 | O2 | -6.9(6) | C41 | N2 | C34 | O4 | -4.7(6) |
| C9 | N1 | C2 | C3 | 173.3(3) | C41 | N2 | C34 | C35 | 175.7(3) |
| C9 | C10 | C11 | C12 | -173.5(3) | C41 | C42 | C47 | C46 | 179.4(3) |
| C9 | C10 | C11 | C26 | 9.7(5) | C41 | C42 | C47 | C48 | -0.5(5) |
| C9 | C10 | C15 | C14 | 176.1(3) | C41 | C42 | C43 | C44 | -179.4(3) |
| C9 | C10 | C15 | C16 | -6.9(5) | C41 | C42 | C43 | C58 | 2.2(5) |
| C10 | C11 | C12 | C13 | -3.9(5) | C44 | C43 | C58 | C59 | -100.1(4) |
| C10 | C11 | C12 | C25 | 175.2(4) | C44 | C43 | C58 | C63 | 77.5(5) |
| C10 | C11 | C26 | C27 | 73.5(5) | C44 | C45 | C46 | C47 | 0.5(6) |
| C10 | C11 | C26 | C31 | -109.5(4) | C44 | C45 | C46 | C51 | -179.1(4) |

**Table 6 Torsion Angles for $C_{32}H_{21}NO_2$ (5b).**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C10 | C15 | C16 | C17 | -178.3(4) | C44 | C45 | C54 | C53 | 178.7(4) |
| C11 | C10 | C15 | C14 | -4.2(5) | C44 | C45 | C54 | C55 | -1.4(6) |
| C11 | C10 | C15 | C16 | 172.8(4) | C42 | C47 | C46 | C45 | 0.2(5) |
| C11 | C12 | C13 | C14 | -1.5(5) | C42 | C47 | C46 | C51 | 179.8(3) |
| C11 | C12 | C13 | C22 | -179.9(3) | C42 | C47 | C48 | C49 | 178.1(4) |
| C11 | C12 | C25 | C24 | -177.7(4) | C42 | C43 | C58 | C59 | 78.3(5) |
| C11 | C26 | C27 | C28 | 176.4(3) | C42 | C43 | C58 | C63 | -104.1(4) |
| C11 | C26 | C31 | C30 | -176.3(4) | C47 | C42 | C43 | C44 | 0.9(5) |
| C12 | C11 | C26 | C27 | -103.3(4) | C47 | C42 | C43 | C58 | -177.4(3) |
| C12 | C11 | C26 | C31 | 73.6(5) | C47 | C46 | C51 | C50 | 2.3(6) |
| C12 | C13 | C14 | C15 | 4.0(5) | C47 | C46 | C51 | C52 | -179.6(4) |
| C12 | C13 | C14 | C19 | -177.2(3) | C47 | C48 | C49 | C50 | 1.9(6) |
| C12 | C13 | C22 | C21 | 178.2(4) | C43 | C44 | C45 | C46 | -0.6(5) |
| C12 | C13 | C22 | C23 | -2.8(6) | C43 | C44 | C45 | C54 | 179.7(3) |
| C13 | C12 | C25 | C24 | 1.4(6) | C43 | C44 | C57 | C56 | -179.0(4) |
| C13 | C14 | C15 | C10 | -1.3(5) | C43 | C42 | C47 | C46 | -0.9(5) |
| C13 | C14 | C15 | C16 | -178.4(3) | C43 | C42 | C47 | C48 | 179.1(4) |
| C13 | C14 | C19 | C18 | 179.1(4) | C43 | C58 | C59 | C60 | 178.0(3) |
| C13 | C14 | C19 | C20 | -1.4(5) | C43 | C58 | C63 | C62 | -177.7(4) |
| C13 | C22 | C23 | C24 | 2.3(6) | C45 | C44 | C43 | C42 | -0.2(5) |
| C14 | C13 | C22 | C21 | -0.3(6) | C45 | C44 | C43 | C58 | 178.3(3) |
| C14 | C13 | C22 | C23 | 178.8(4) | C45 | C44 | C57 | C56 | 0.6(6) |
| C14 | C15 | C16 | C17 | -1.3(6) | C45 | C46 | C51 | C50 | -178.1(4) |
| C14 | C19 | C20 | C21 | 0.7(6) | C45 | C46 | C51 | C52 | 0.0(6) |
| C15 | C10 | C11 | C12 | 6.8(5) | C45 | C54 | C55 | C56 | 1.9(6) |
| C15 | C10 | C11 | C26 | -170.0(3) | C58 | C59 | C60 | C61 | -0.6(6) |
| C15 | C14 | C19 | C18 | -2.1(5) | C46 | C47 | C48 | C49 | -1.8(6) |
| C15 | C14 | C19 | C20 | 177.4(3) | C46 | C45 | C54 | C53 | -1.0(6) |
| C15 | C16 | C17 | C18 | -0.9(6) | C46 | C45 | C54 | C55 | 178.9(4) |
| C16 | C17 | C18 | C19 | 1.6(7) | C46 | C51 | C52 | C53 | -0.4(7) |
| C17 | C18 | C19 | C14 | -0.1(6) | C48 | C47 | C46 | C45 | -179.9(3) |
| C17 | C18 | C19 | C20 | -179.6(4) | C48 | C47 | C46 | C51 | -0.2(5) |
| C18 | C19 | C20 | C21 | -179.9(4) | C48 | C49 | C50 | C51 | 0.2(6) |
| C19 | C14 | C15 | C10 | 179.9(3) | C49 | C50 | C51 | C46 | -2.3(6) |
| C19 | C14 | C15 | C16 | 2.8(5) | C49 | C50 | C51 | C52 | 179.7(4) |

**Table 6 Torsion Angles for $C_{32}H_{21}NO_2$ (5b).**

| A | B | C | D | Angle/° | A | B | C | D | Angle/° |
|---|---|---|---|---|---|---|---|---|---|
| C19 | C20 | C21 | C22 | 0.3(7) | C50 | C51 | C52 | C53 | 177.7(5) |
| C20 | C21 | C22 | C13 | -0.5(6) | C51 | C52 | C53 | C54 | 0.0(8) |
| C20 | C21 | C22 | C23 | -179.6(4) | C52 | C53 | C54 | C45 | 0.7(7) |
| C21 | C22 | C23 | C24 | -178.6(4) | C52 | C53 | C54 | C55 | -179.2(5) |
| C22 | C13 | C14 | C15 | -177.6(3) | C53 | C54 | C55 | C56 | -178.2(4) |
| C22 | C13 | C14 | C19 | 1.3(5) | C54 | C45 | C46 | C47 | -179.7(3) |
| C22 | C23 | C24 | C25 | 0.0(7) | C54 | C45 | C46 | C51 | 0.7(6) |
| C23 | C24 | C25 | C12 | -1.9(7) | C54 | C55 | C56 | C57 | -1.3(7) |
| C25 | C12 | C13 | C14 | 179.3(4) | C55 | C56 | C57 | C44 | 0.0(6) |
| C25 | C12 | C13 | C22 | 0.9(5) | C57 | C44 | C43 | C42 | 179.4(4) |
| C26 | C11 | C12 | C13 | 173.0(3) | C57 | C44 | C43 | C58 | -2.2(5) |
| C26 | C11 | C12 | C25 | -7.8(5) | C57 | C44 | C45 | C46 | 179.9(4) |
| C26 | C27 | C28 | C29 | 0.1(6) | C57 | C44 | C45 | C54 | 0.1(5) |
| C27 | C26 | C31 | C30 | 0.6(6) | C59 | C58 | C63 | C62 | 0.0(6) |
| C27 | C28 | C29 | C30 | 0.5(6) | C59 | C60 | C61 | C62 | 0.8(6) |
| C27 | C28 | C29 | C32 | -179.0(4) | C59 | C60 | C61 | C64 | -179.6(4) |
| C28 | C29 | C30 | C31 | -0.6(6) | C60 | C61 | C62 | C63 | -0.5(6) |
| C29 | C30 | C31 | C26 | 0.0(6) | C61 | C62 | C63 | C58 | 0.1(6) |
| C31 | C26 | C27 | C28 | -0.7(6) | C63 | C58 | C59 | C60 | 0.2(5) |
| C32 | C29 | C30 | C31 | 179.0(4) | C64 | C61 | C62 | C63 | 179.9(4) |

**Table 7 Hydrogen Atom Coordinates (Å×$10^4$) and Isotropic Displacement Parameters (Å$^2$×$10^3$) for $C_{32}H_{21}NO_2$ (5b).**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H5 | 2417.11 | 8219.93 | 6560.12 | 60 |
| H6 | 1705.05 | 8767.83 | 6351.51 | 67 |
| H7 | 1495.74 | 9326.25 | 5326.74 | 63 |
| H8 | 1985.35 | 9334.17 | 4471.65 | 56 |
| H9A | 3645 | 8289.91 | 4271.57 | 53 |
| H9B | 3674.69 | 7456.5 | 4766.92 | 53 |
| H16 | 3621.75 | 9796.59 | 4308.49 | 60 |
| H17 | 3763.81 | 11339.24 | 4180.86 | 76 |
| H18 | 4297.07 | 12059.38 | 4816.22 | 76 |
| H20 | 4890.26 | 11989.47 | 5674.87 | 80 |

**Table 7 Hydrogen Atom Coordinates (Å×$10^4$) and Isotropic Displacement Parameters (Å$^2$×$10^3$) for $C_{32}H_{21}NO_2$ (5b).**

| Atom | *x* | *y* | *z* | U(eq) |
|---|---|---|---|---|
| H21 | 5306.01 | 11174.06 | 6408.8 | 83 |
| H23 | 5495.46 | 9691.78 | 6954.71 | 89 |
| H24 | 5392.11 | 8131.54 | 7040.28 | 86 |
| H25 | 4879.36 | 7382.68 | 6377.08 | 72 |
| H27 | 4536.99 | 6881.11 | 4615.56 | 59 |
| H28 | 4560.21 | 5301.5 | 4614.62 | 67 |
| H30 | 4045.23 | 5257.96 | 6382.75 | 76 |
| H31 | 4020.26 | 6836.49 | 6393.5 | 68 |
| H32A | 4122.39 | 3830.77 | 5173.32 | 113 |
| H32B | 4262.63 | 3838.51 | 5928.38 | 113 |
| H32C | 4623.59 | 3879.06 | 5394.06 | 113 |
| H37 | 3280.81 | 6580.56 | -674.41 | 66 |
| H38 | 2789.75 | 6055.72 | -1494.98 | 79 |
| H39 | 2076.12 | 5696.79 | -1265.99 | 75 |
| H40 | 1828.56 | 5844.03 | -202.04 | 63 |
| H41A | 2600.72 | 6771.17 | 2092.7 | 55 |
| H41B | 2909.53 | 7573.61 | 1872.06 | 55 |
| H48 | 2631.13 | 5198.13 | 1858.36 | 59 |
| H49 | 2641.44 | 3622.71 | 1955.75 | 68 |
| H50 | 3239.46 | 2889.91 | 2435.07 | 74 |
| H52 | 3975.46 | 2953.58 | 2990.42 | 91 |
| H53 | 4571.08 | 3778.15 | 3329.17 | 98 |
| H55 | 4964.5 | 5275.05 | 3434.02 | 83 |
| H56 | 4963.42 | 6849.27 | 3328.23 | 77 |
| H57 | 4364.05 | 7594.42 | 2863.79 | 67 |
| H59 | 3245.53 | 8194.93 | 3102.75 | 54 |
| H60 | 3272.19 | 9767.78 | 3065.44 | 58 |
| H62 | 4031.06 | 9627.81 | 1525.74 | 73 |
| H63 | 4004.56 | 8056.36 | 1549.5 | 69 |
| H64A | 3408.3 | 11141.38 | 2081.09 | 112 |
| H64B | 3921.95 | 11093.6 | 2020.08 | 112 |
| H64C | 3718.21 | 11136.96 | 2720.75 | 112 |

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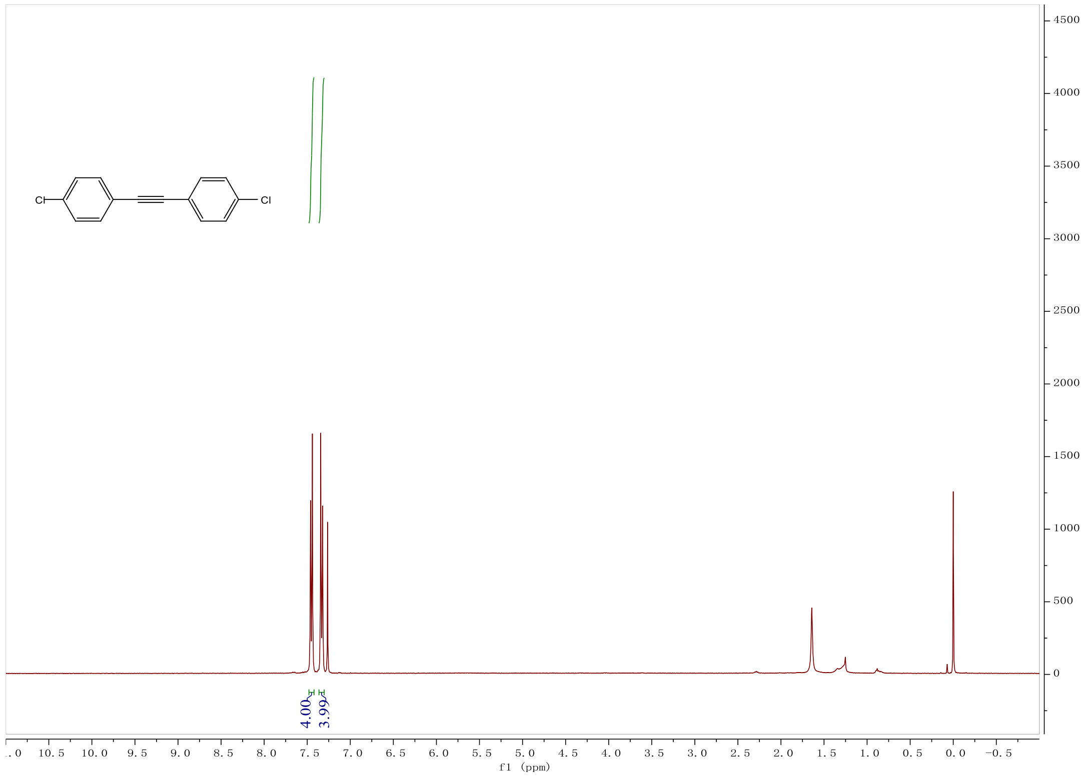
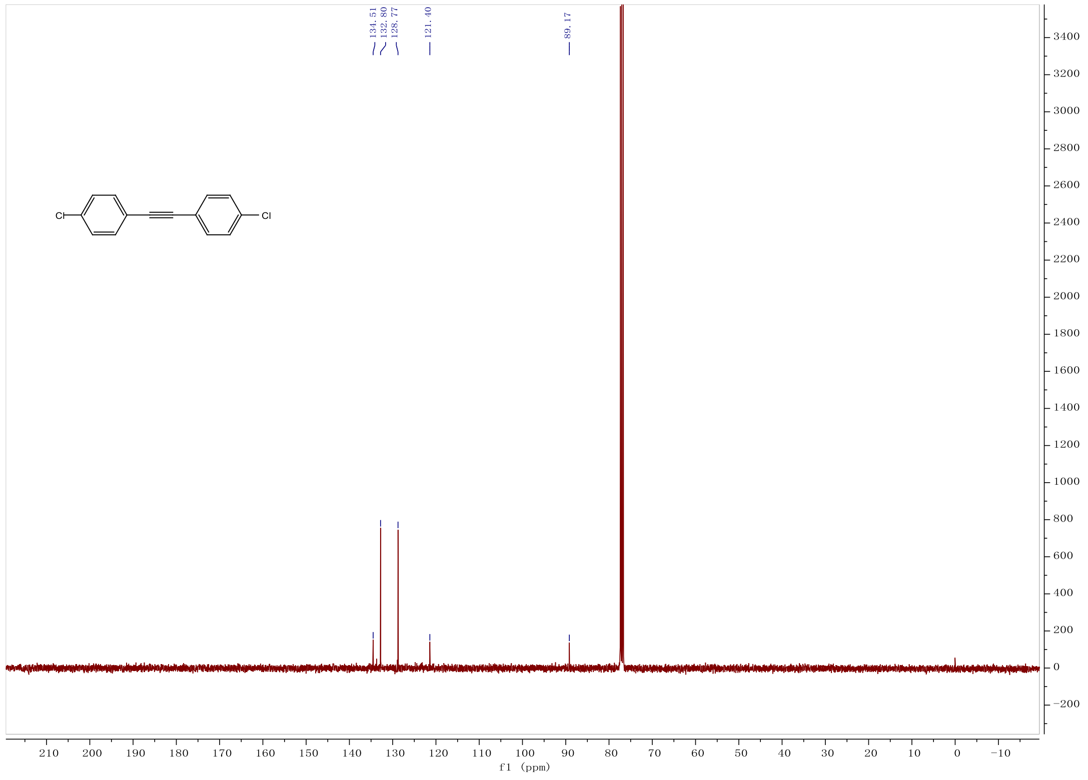
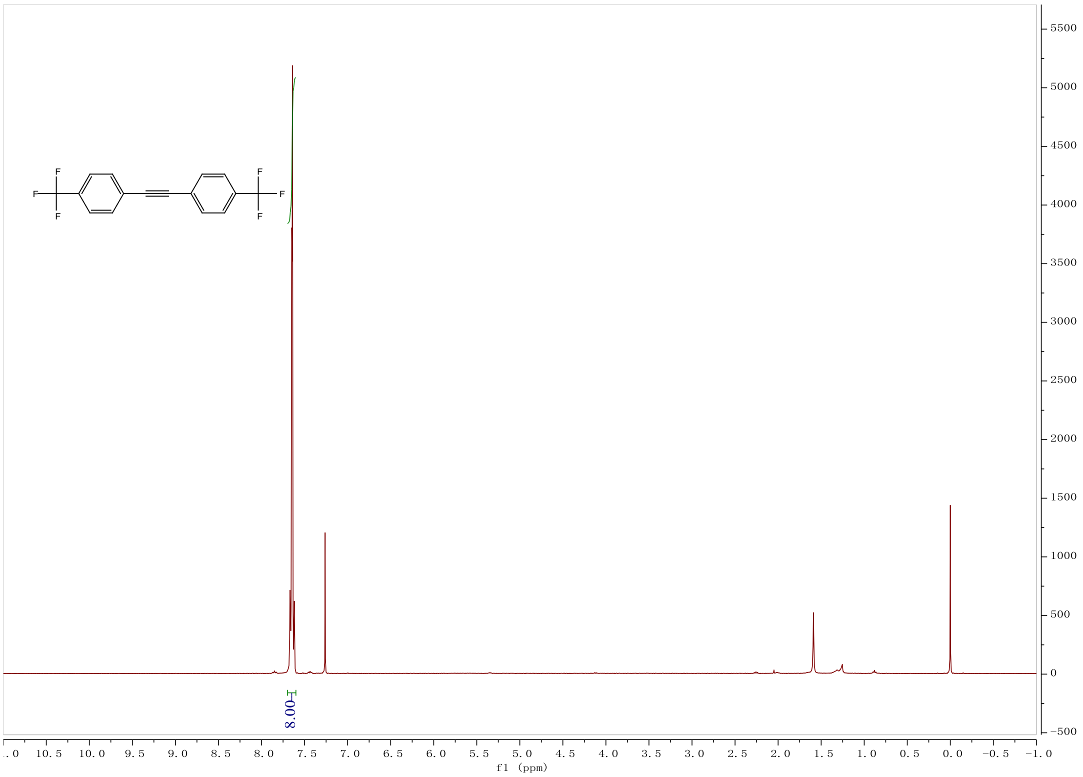
## 8. NMR spectra



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F
F
F
F
F
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11.0
10.5
10.0
9.5
9.0
8.5
8.0
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7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)
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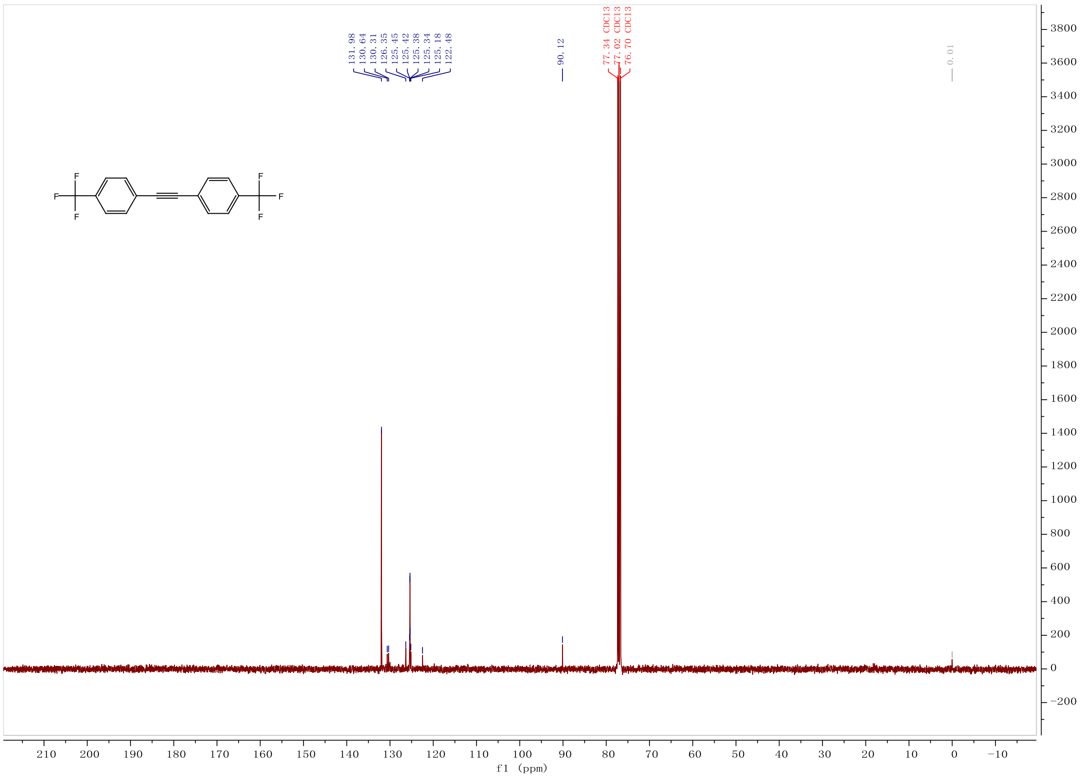
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500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130.64
130.31
126.35
125.45
125.42
125.38
125.34
125.18
122.48
90.12
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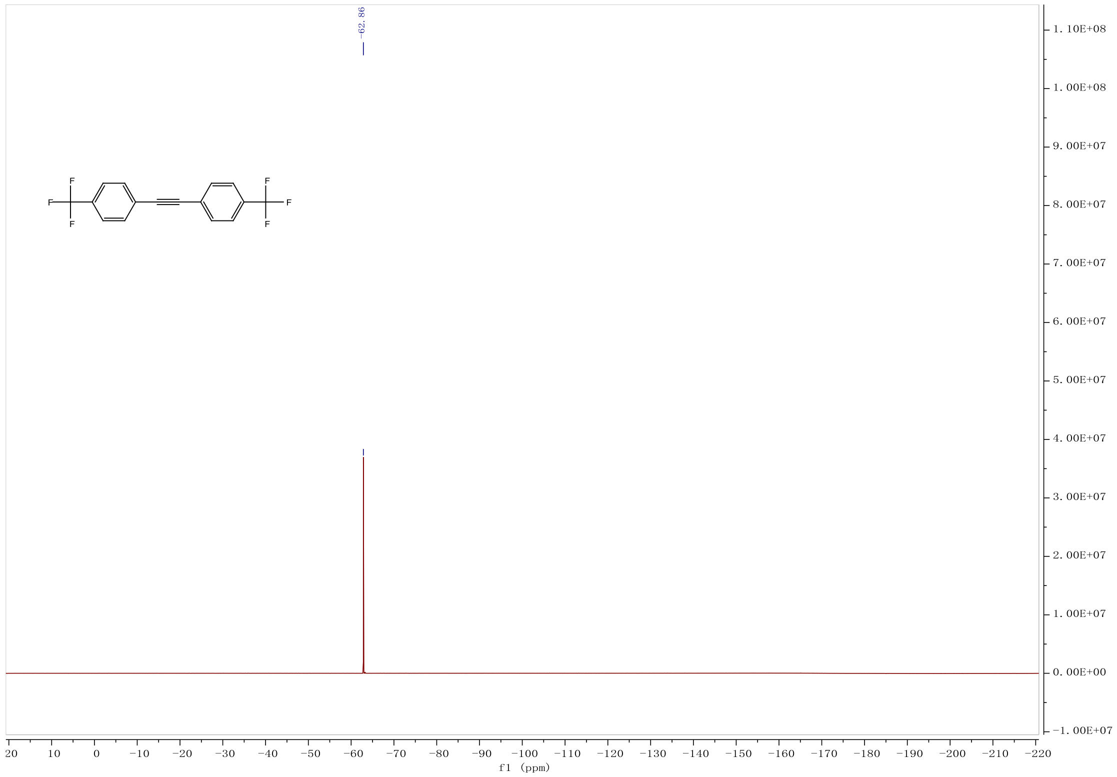
77.02 CDCl3
76.70 CDCl3
0.01
F
F
F
F
F
F
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200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F
F
F
F
F
F
1.10E+08
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6.00E+07
5.00E+07
4.00E+07
3.00E+07
2.00E+07
1.00E+07
0.00E+00
-1.00E+07
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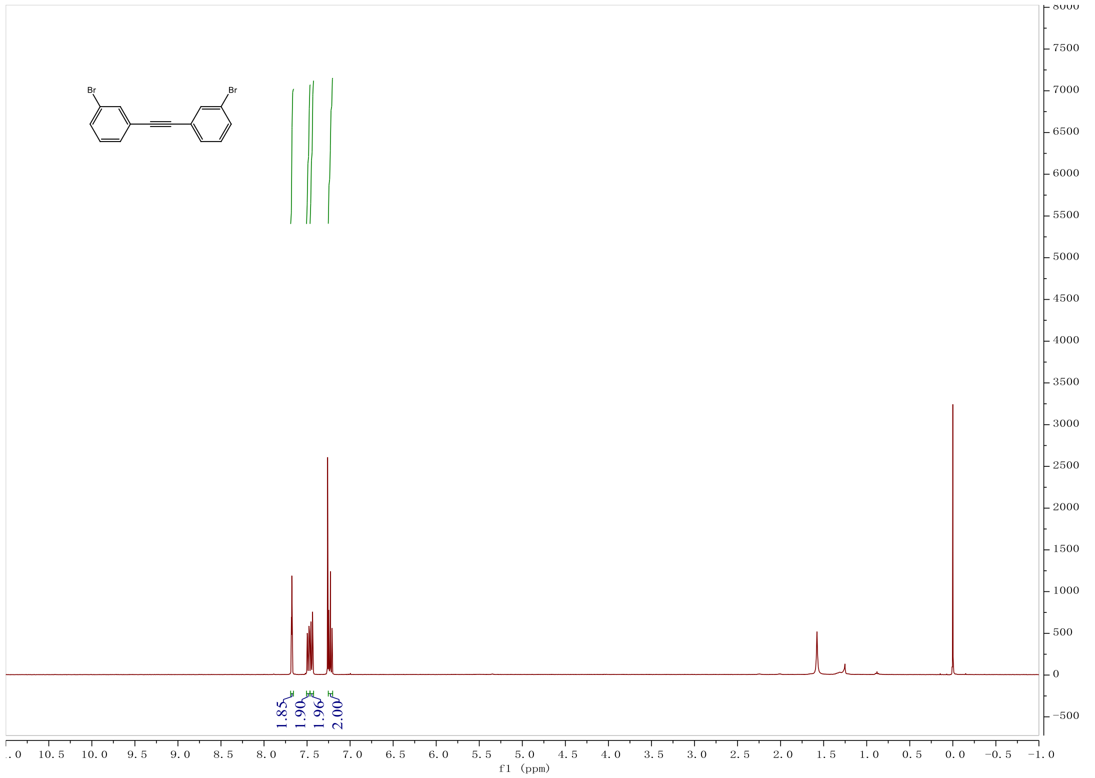
f1 (ppm)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7000
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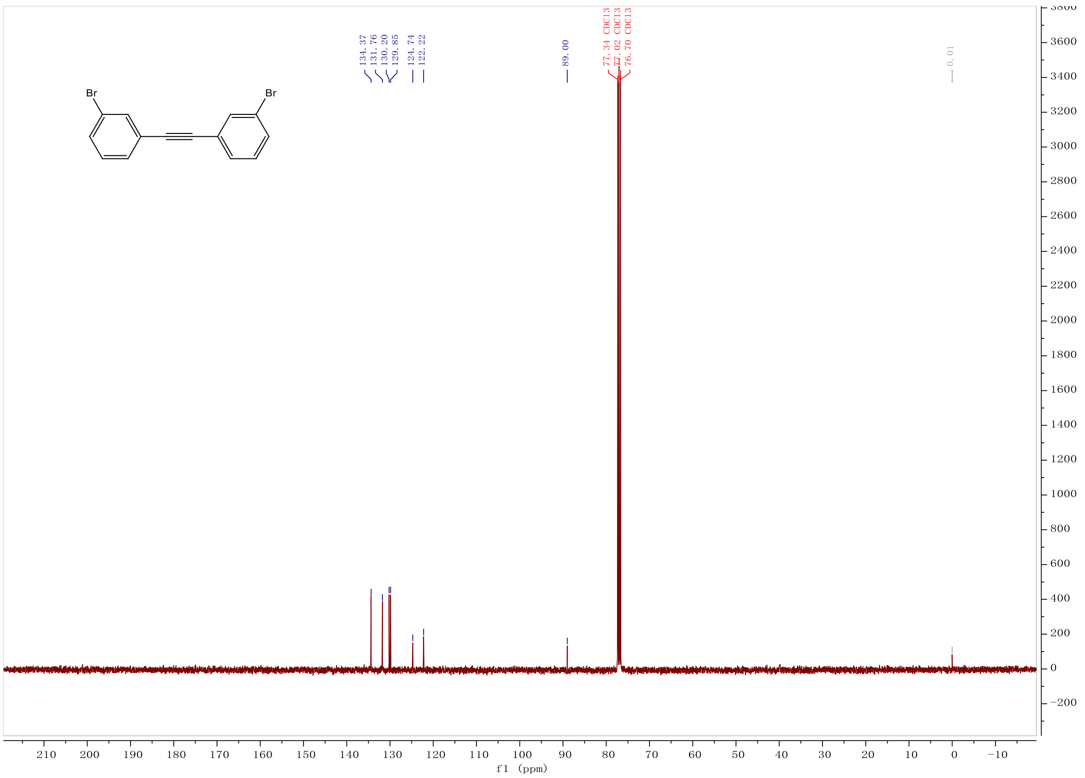
4000
3500
3000
2500
2000
1500
1000
500
0
-500
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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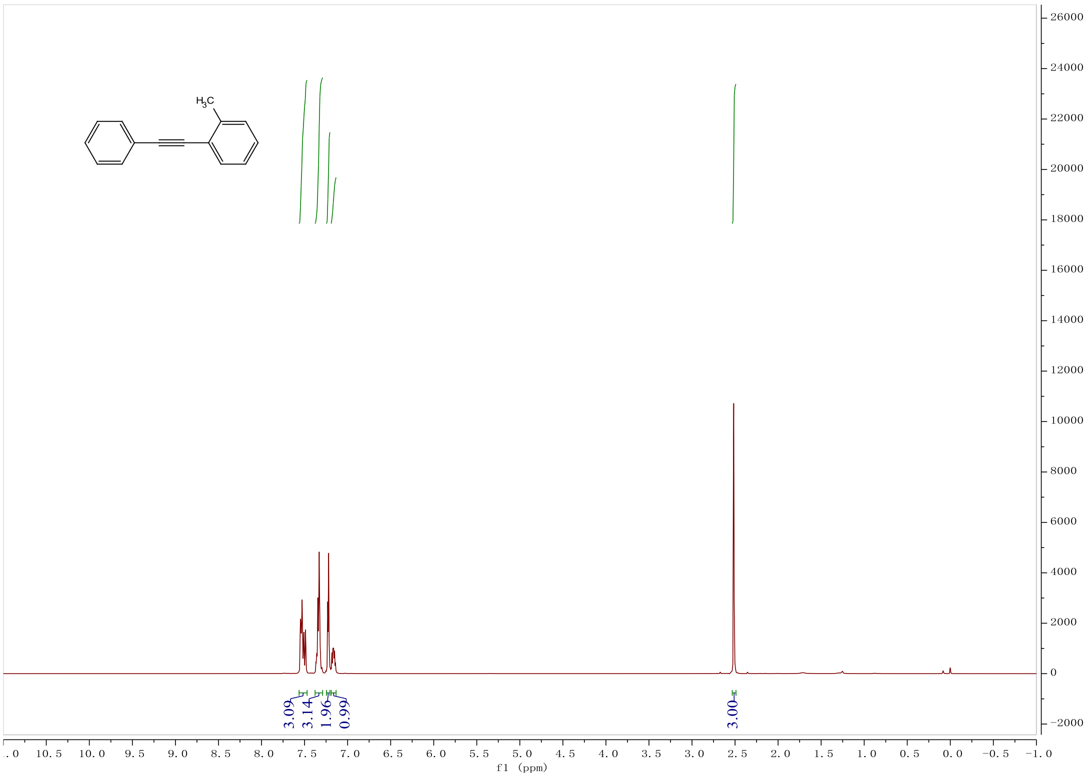
Br
Br
f1 (ppm)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3.14
1.96
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3.00
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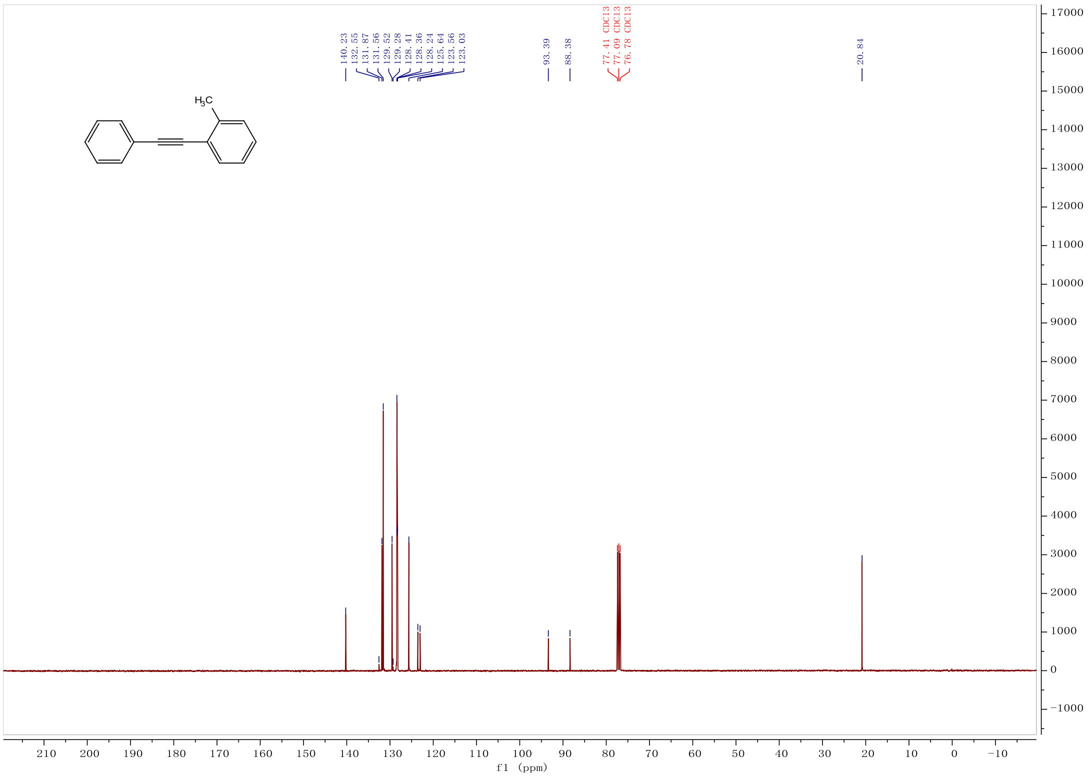
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128.24
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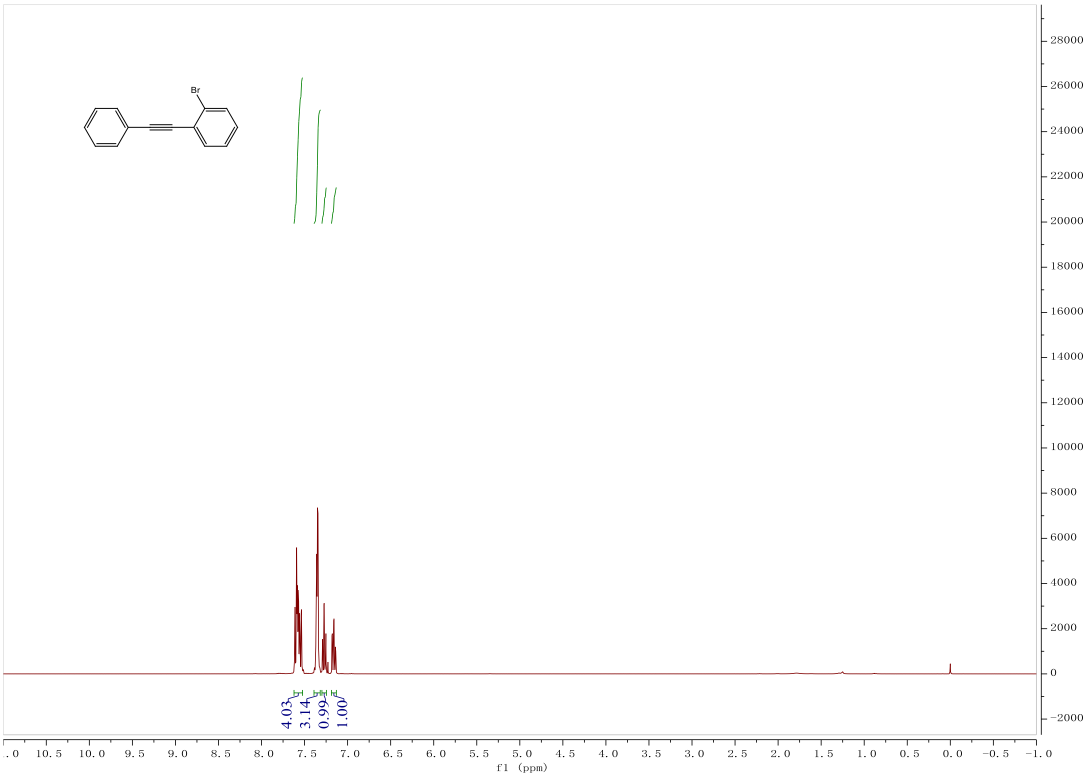
123.03
93.39
88.38
77.41 CDCl3
77.09 CDCl3
76.78 CDCl3
20.84
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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16000
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10000
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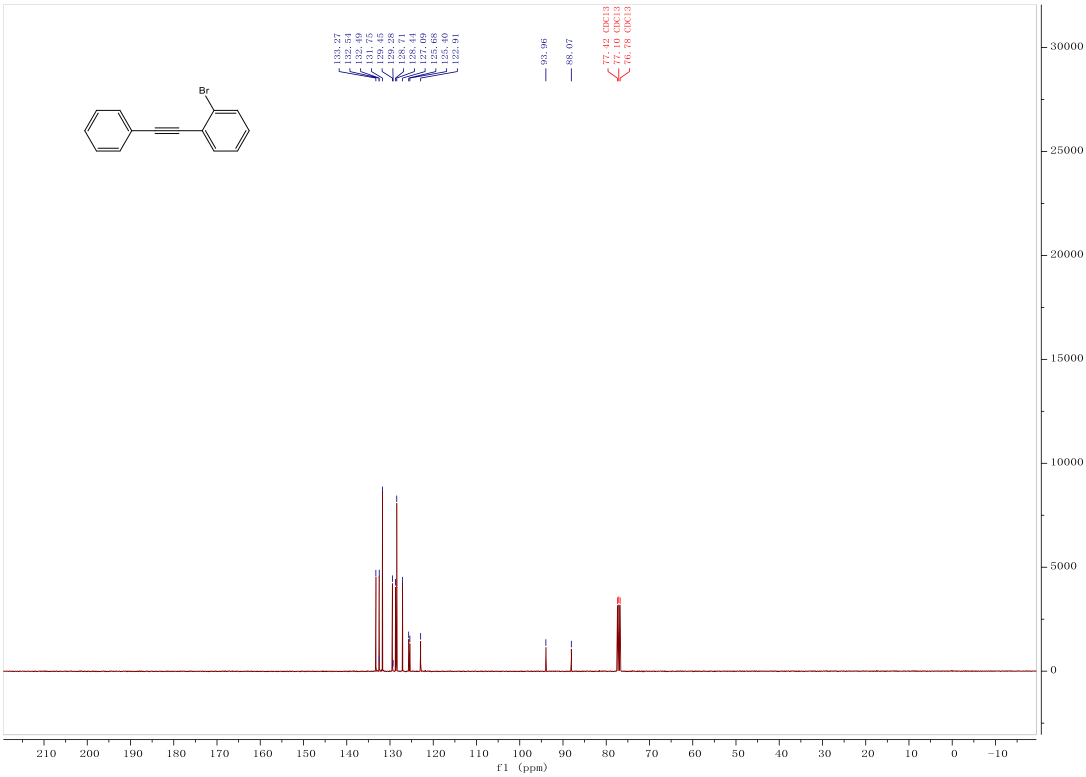
6000
4000
2000
0
−2000
4.03
3.14
0.99
1.00
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)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125.40
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93.96
88.07
77.42 CDCl3
77.10 CDCl3
76.78 CDCl3
Br
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20000
15000
10000
5000
0
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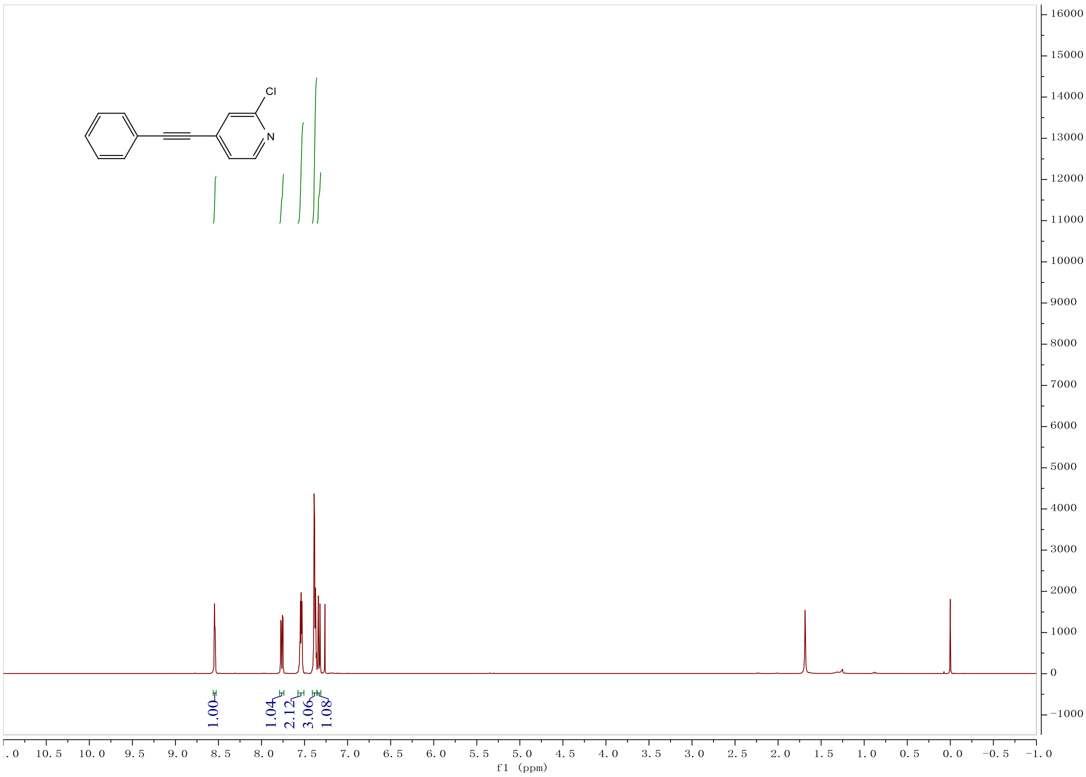
f1 (ppm)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9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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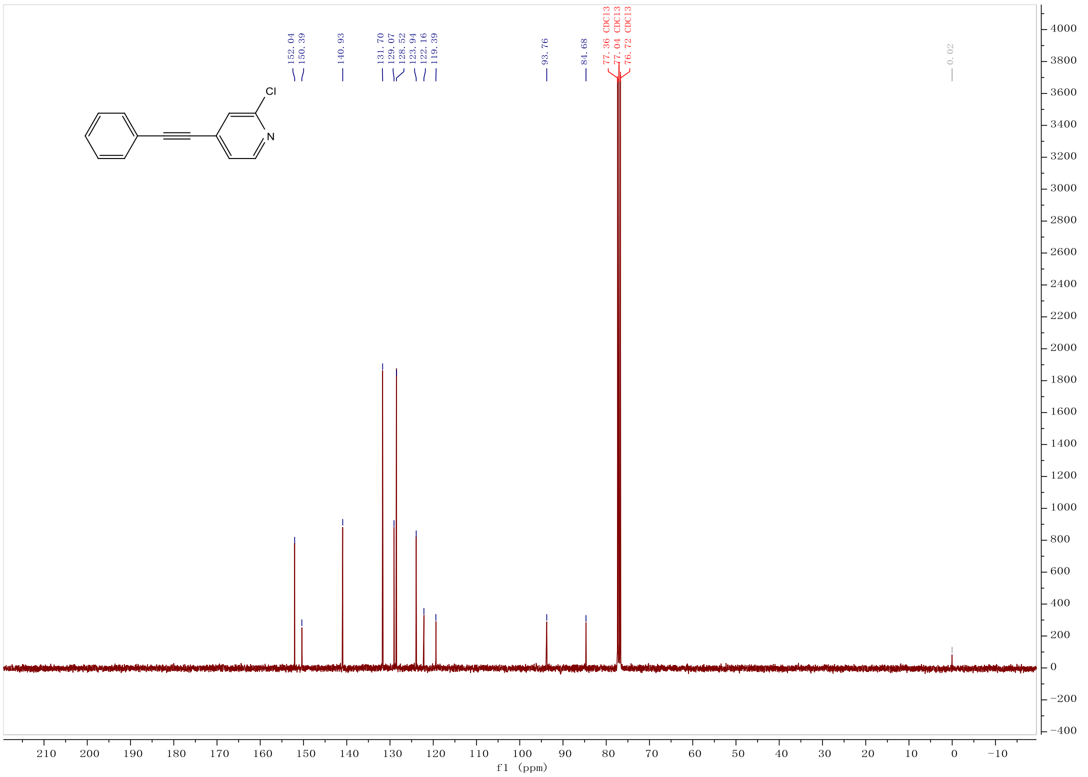
f1 (ppm)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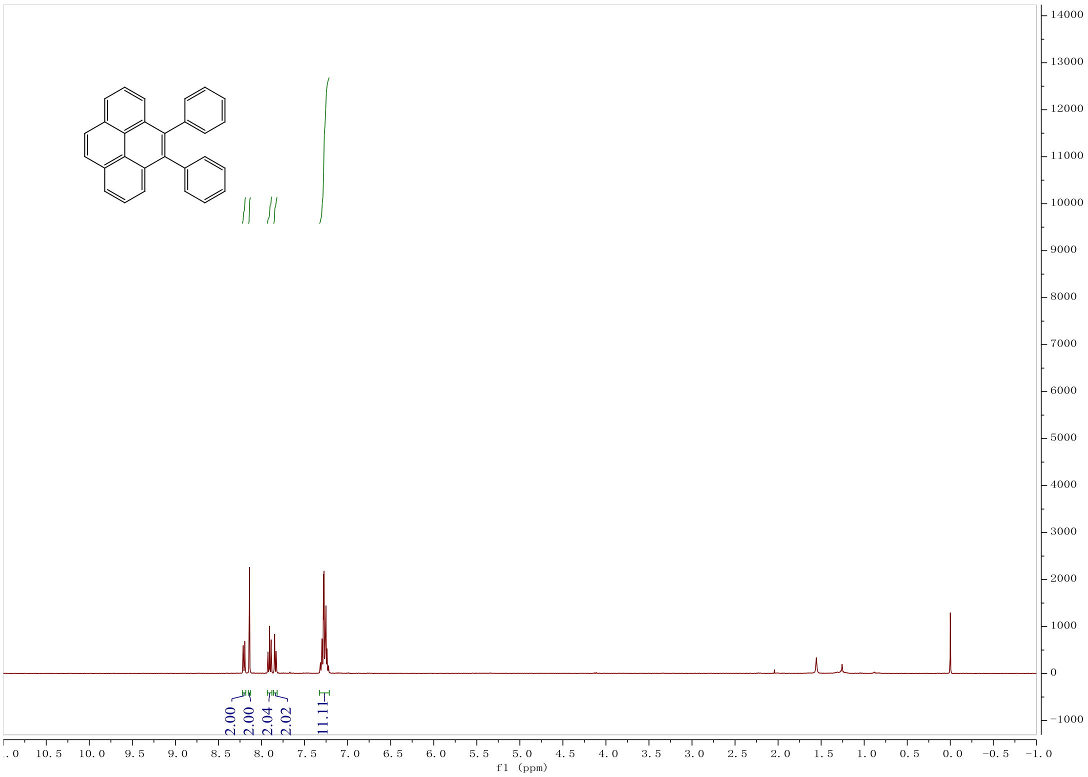
76.72 CDCl3
0.02
Cl
N
f1 (ppm)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2.00
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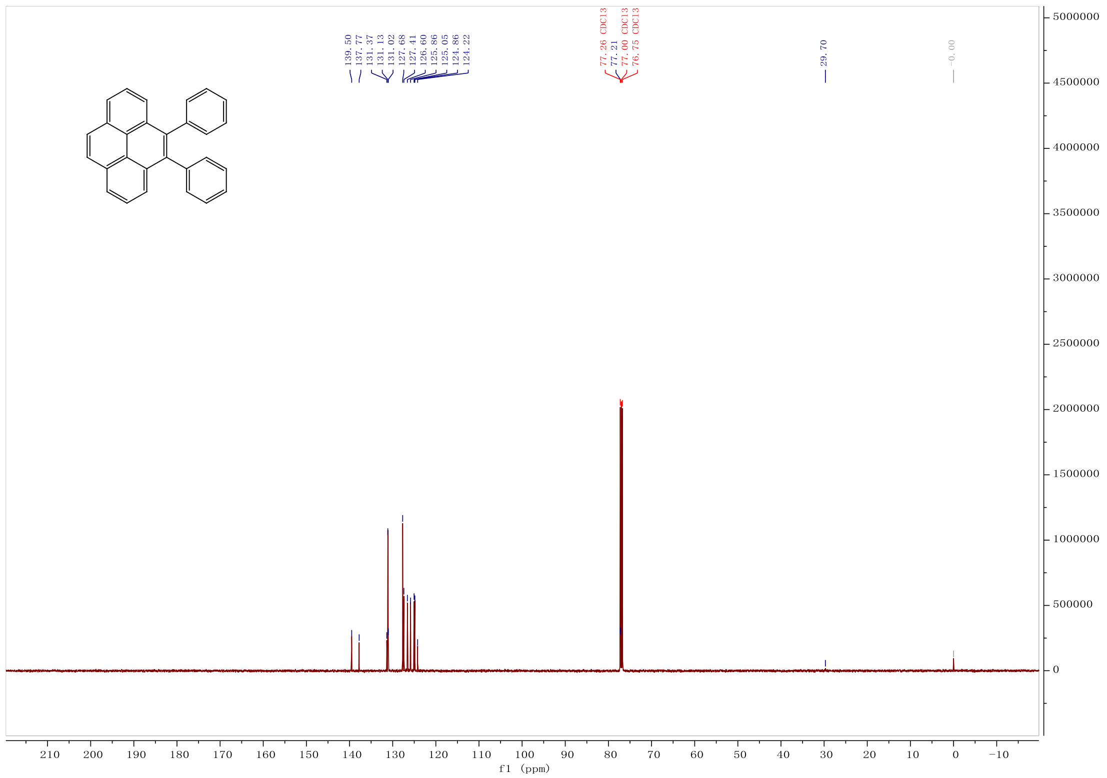
2.02
11.11
f1 (ppm)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127.41
126.60
125.86
125.05
124.86
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77.26 CDCl3
77.21
77.00 CDCl3
76.75 CDCl3
29.70
-0.00
5000000
4500000
4000000
3500000
3000000
2500000
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1500000
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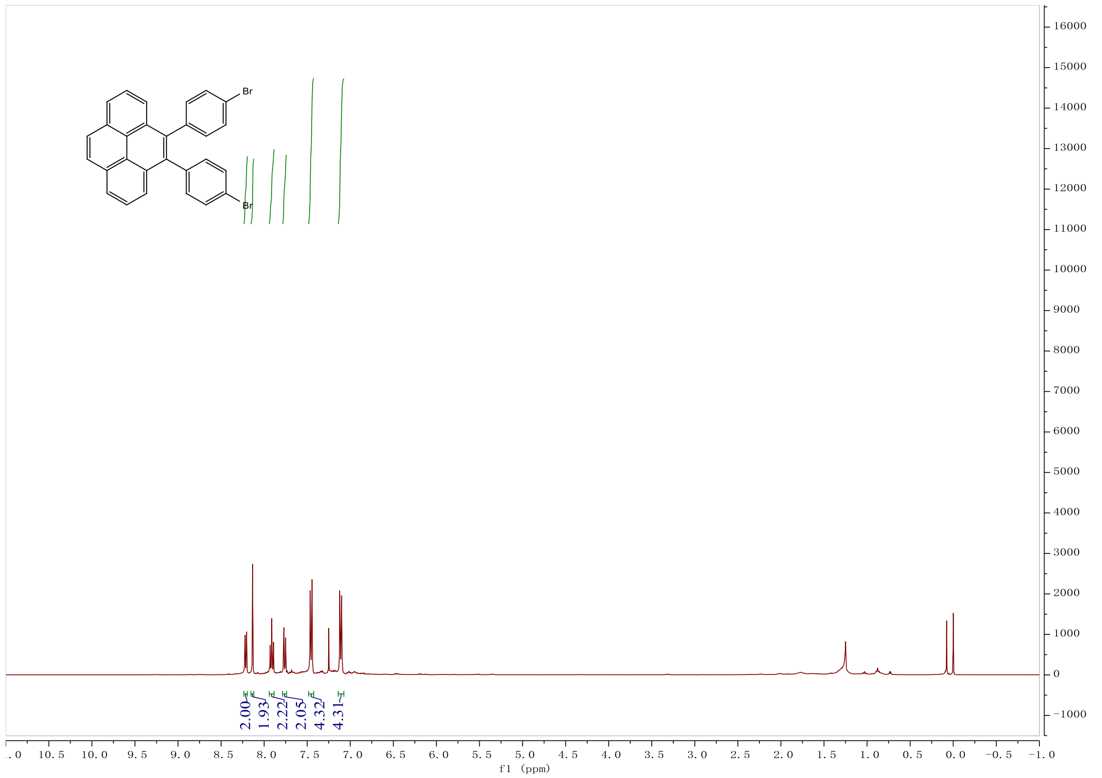
f1 (ppm)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10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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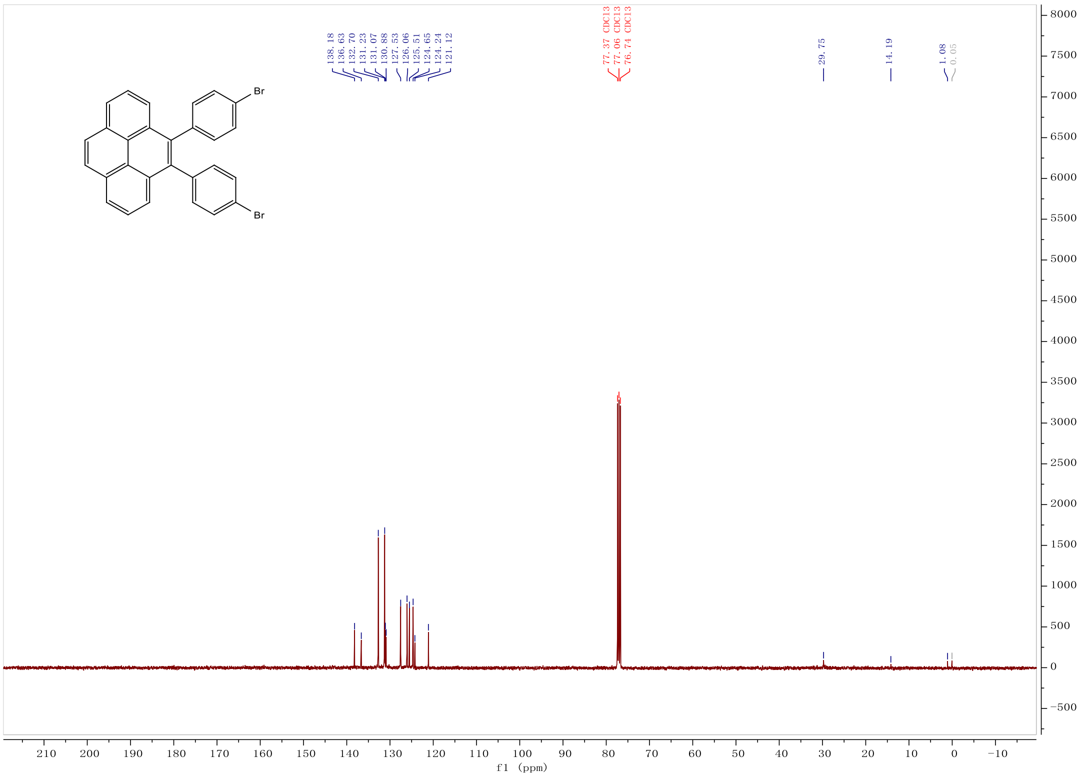
f1 (ppm)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131.07
130.88
127.53
126.06
125.51
124.65
124.24
121.12
77.37 CDCl3
77.06 CDCl3
76.74 CDCl3
29.75
14.19
1.08
0.05
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
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210
200
190
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170
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110
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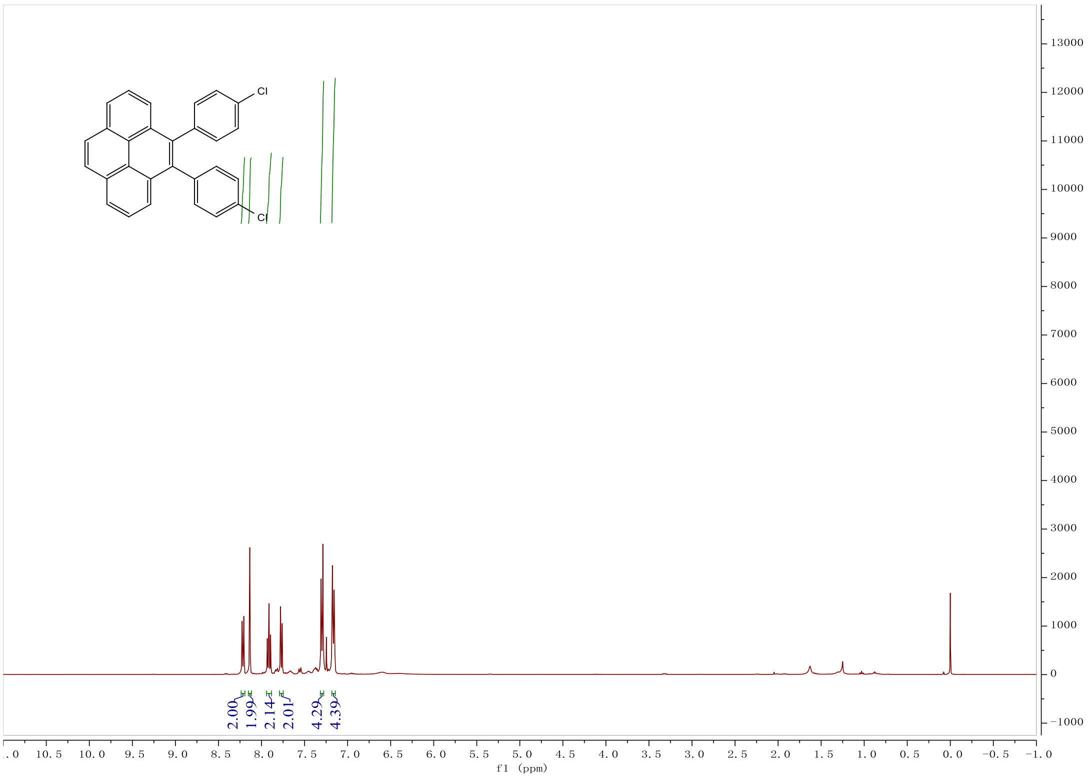
40
30
20
10
0
−10
f1 (ppm)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11000
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7000
6000
5000
4000
3000
2000
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0
−1000
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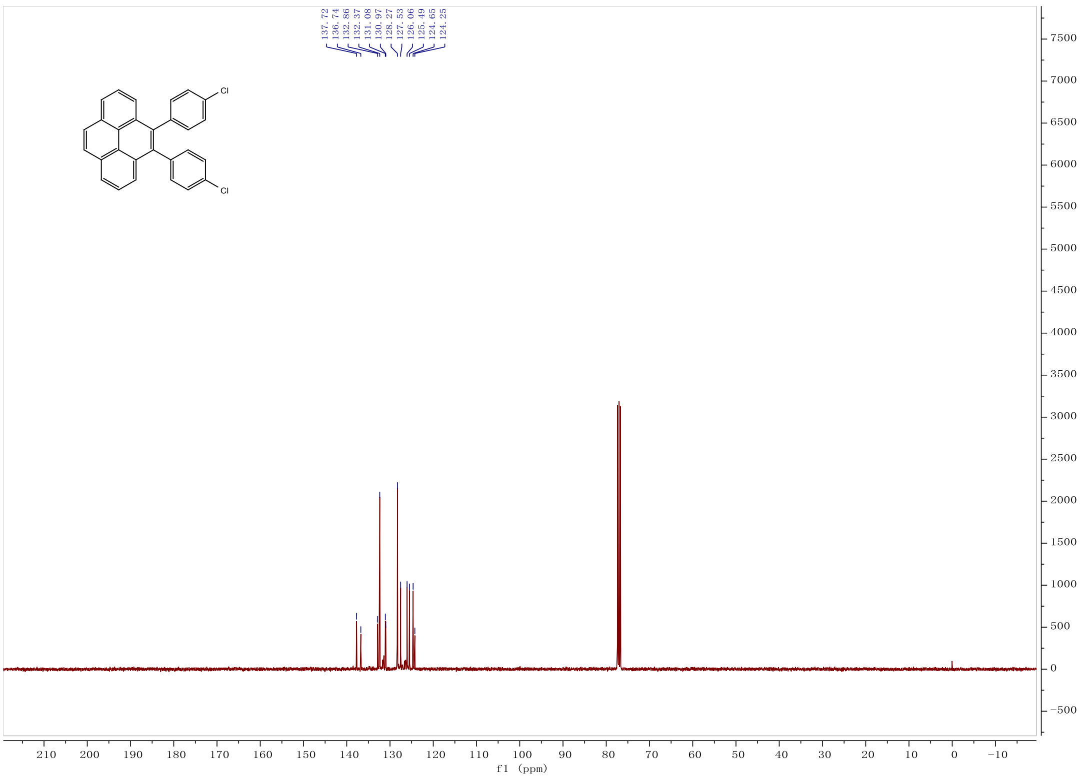
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)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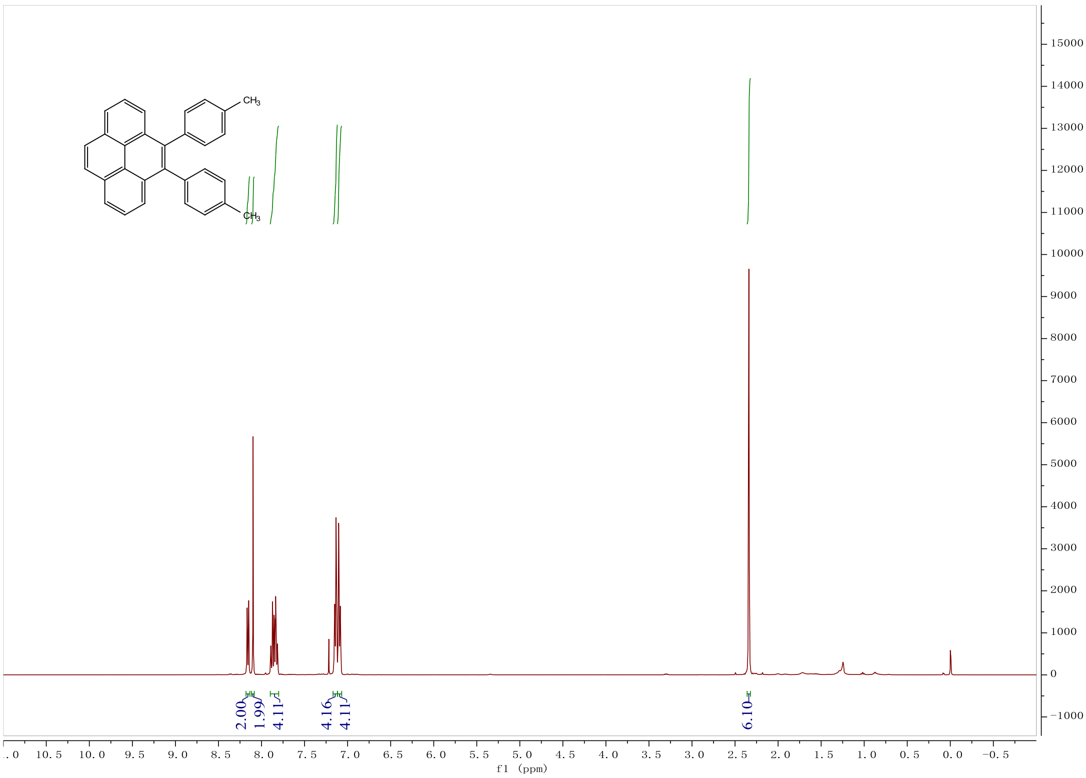
126.06
125.49
124.65
124.25
Cl
Cl
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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4.11
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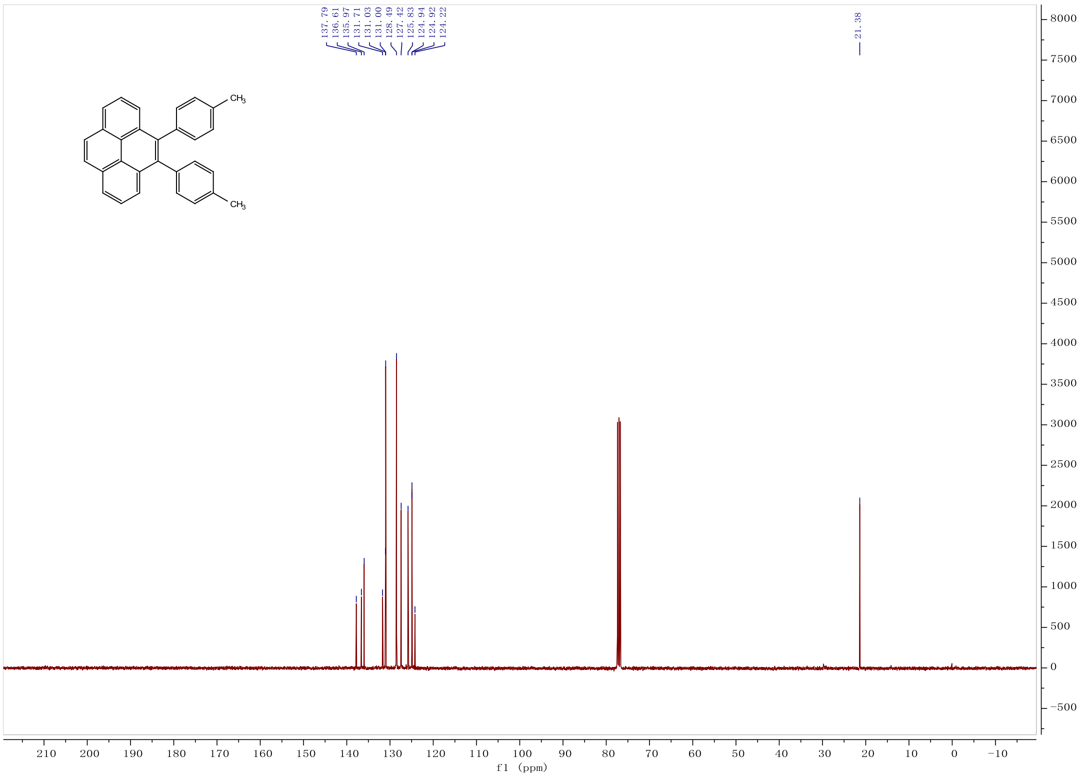
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5
f1 (ppm)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124.92
124.22
21.38
CH3
CH3
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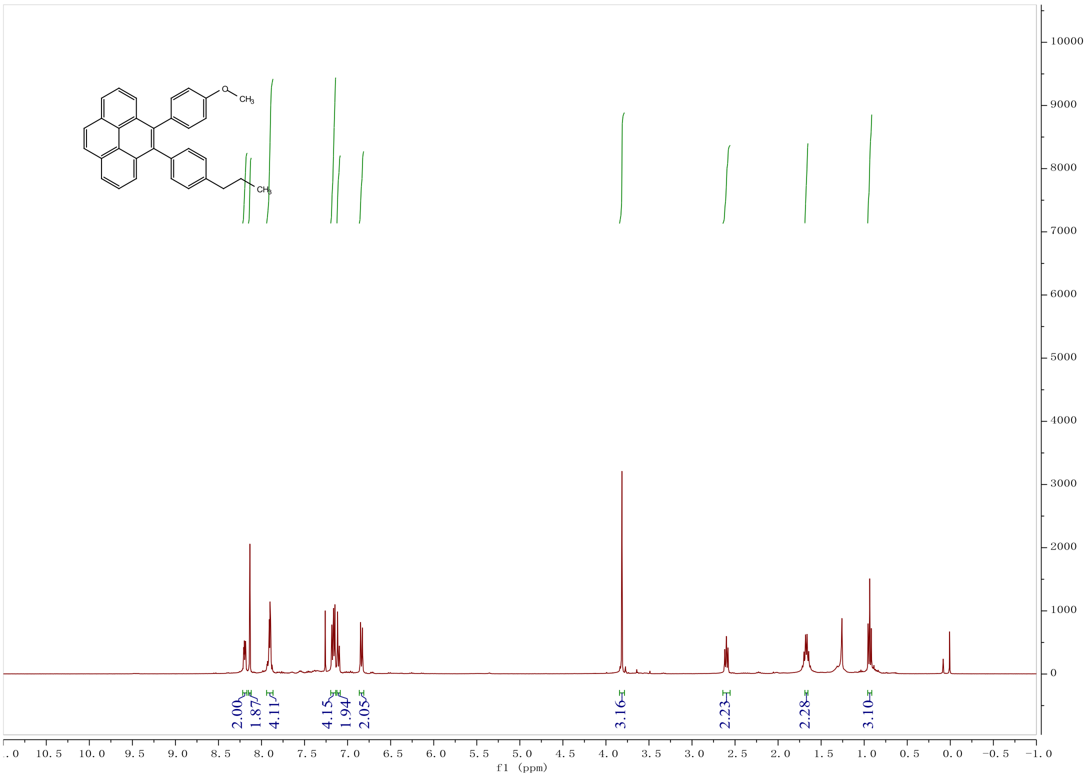
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1.94
2.05
3.16
2.23
2.28
3.10
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)
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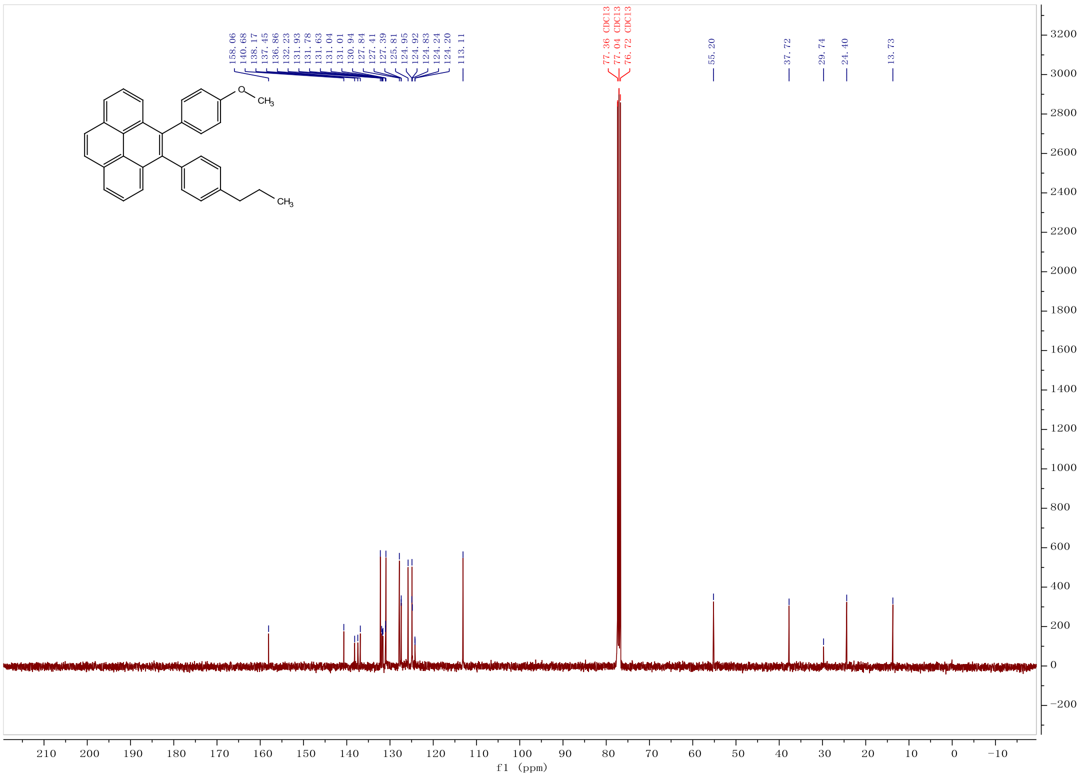
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131.63
131.04
131.01
130.94
127.84
127.41
127.39
125.81
124.95
124.92
124.83
124.24
124.20
113.11
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
55.20
37.72
29.74
24.40
13.73
O
CH3
CH3
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
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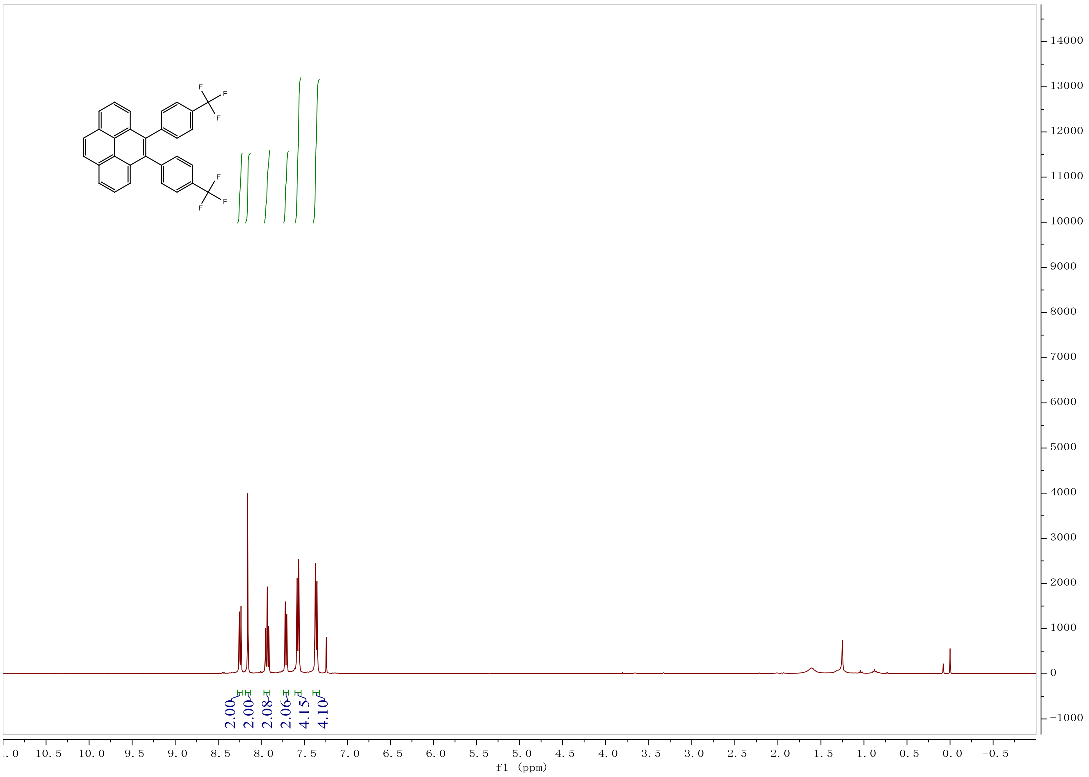
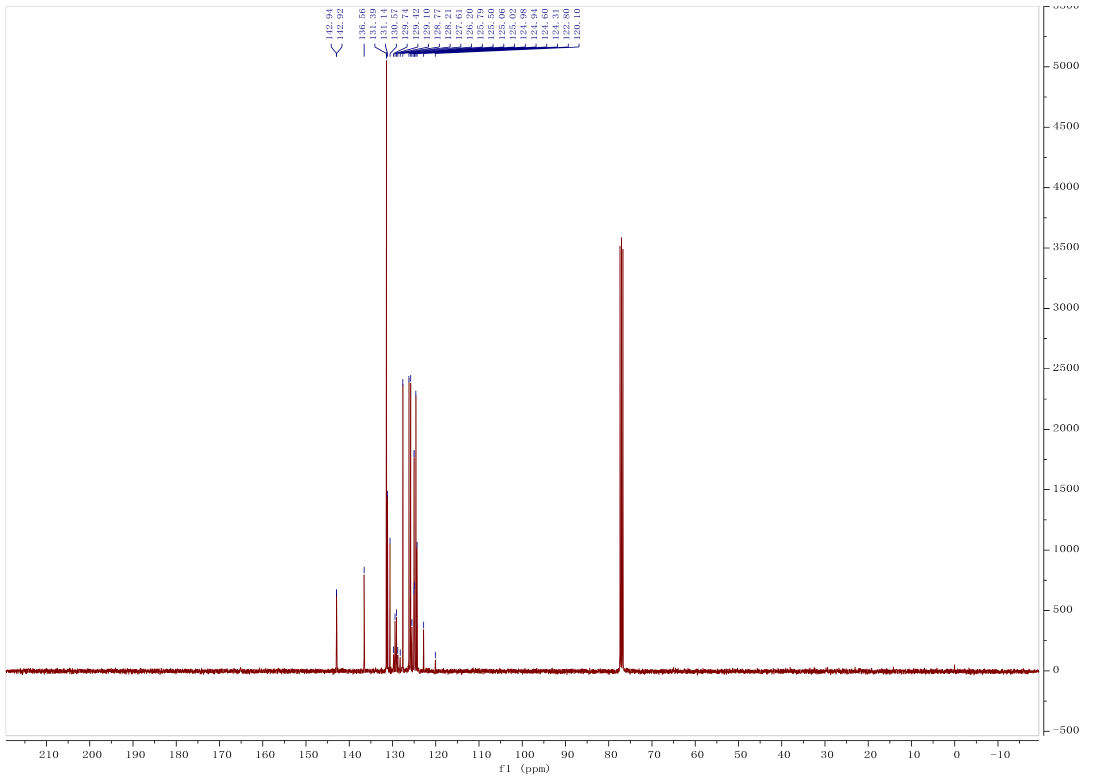
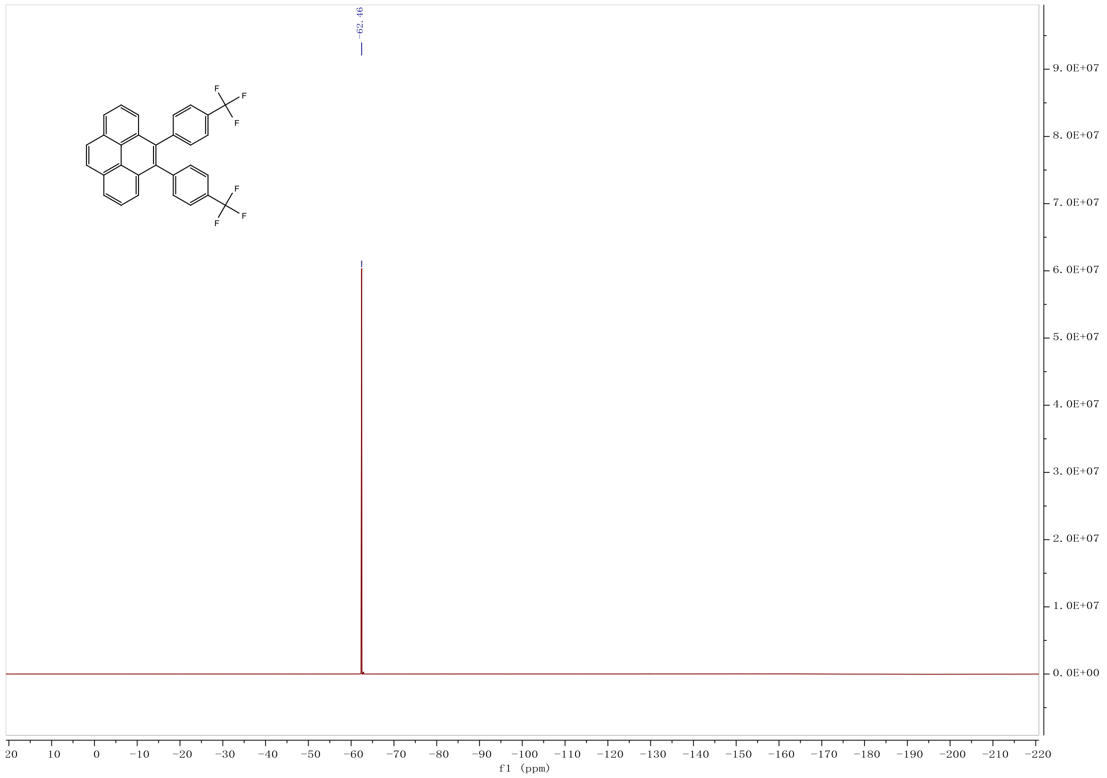
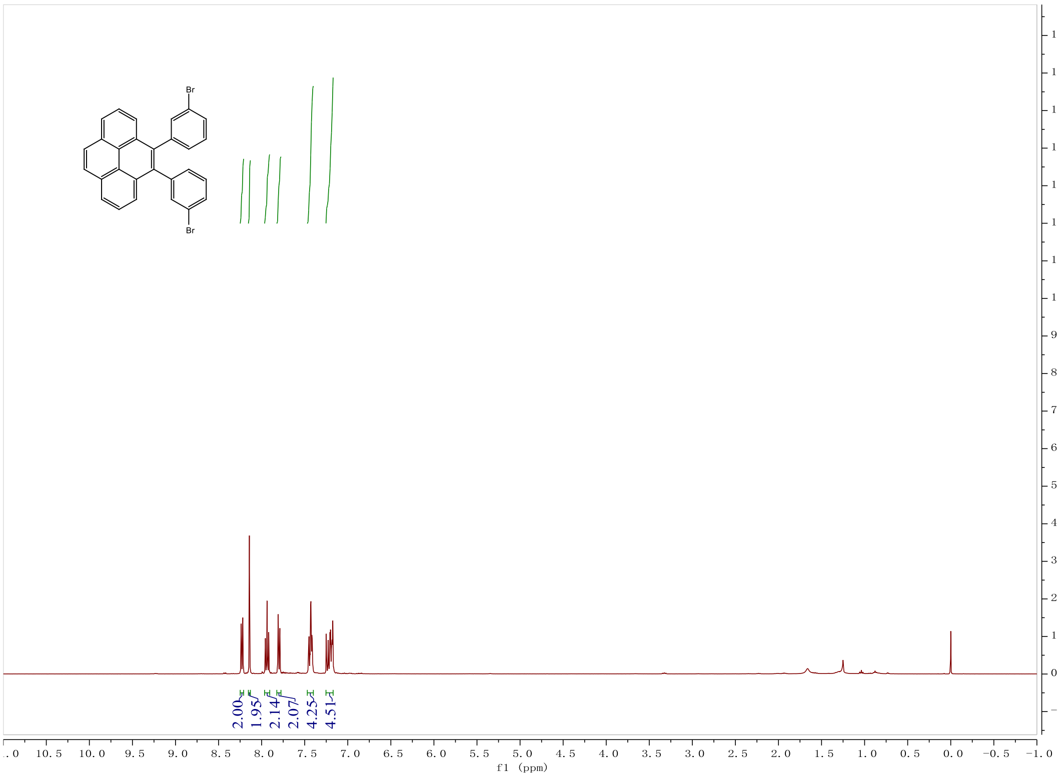
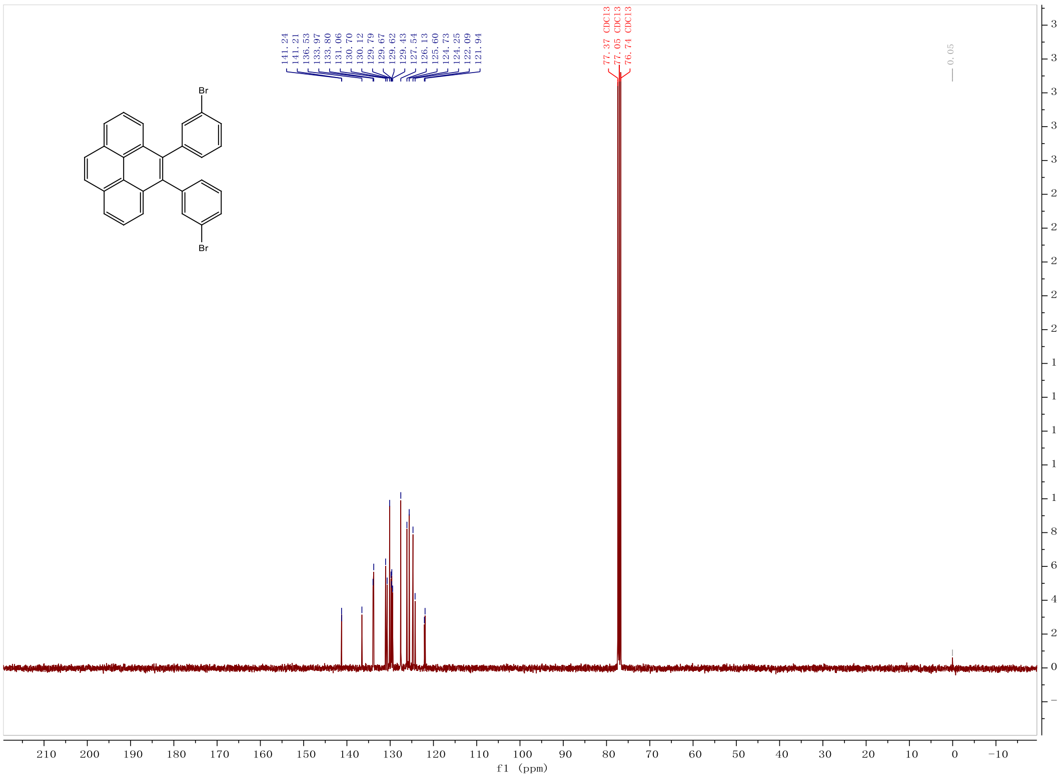
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

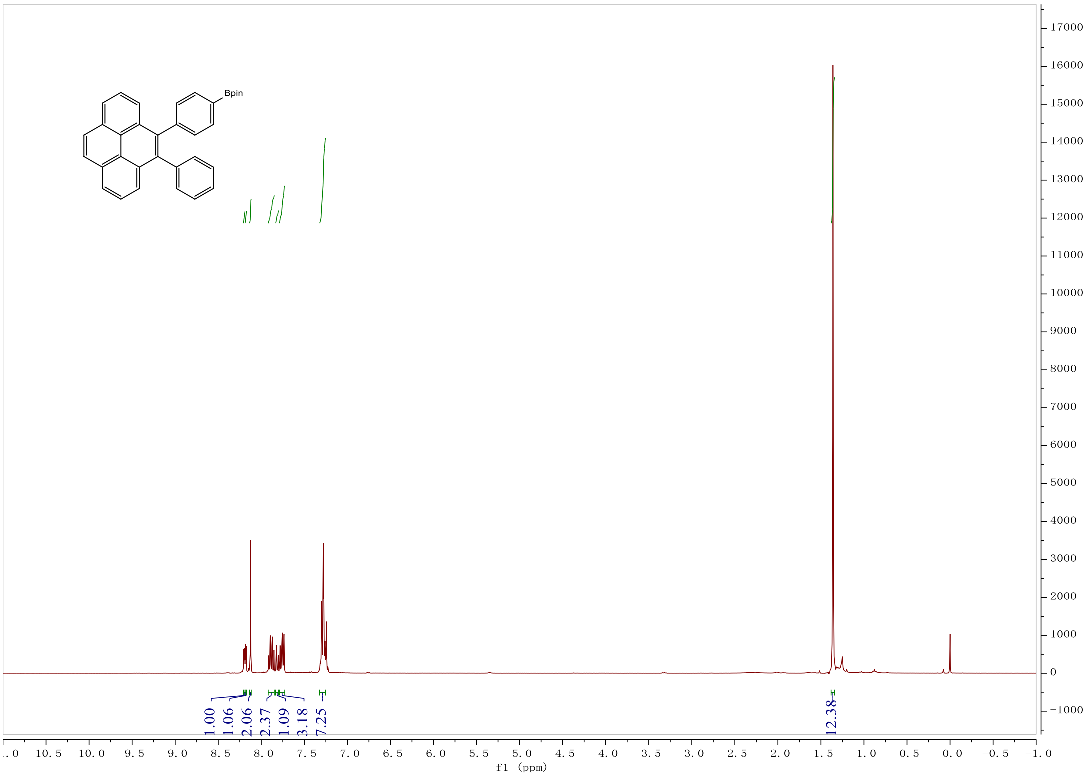

F
F
F
F
F
F
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
2.00
2.00
2.08
2.06
4.15
4.10
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

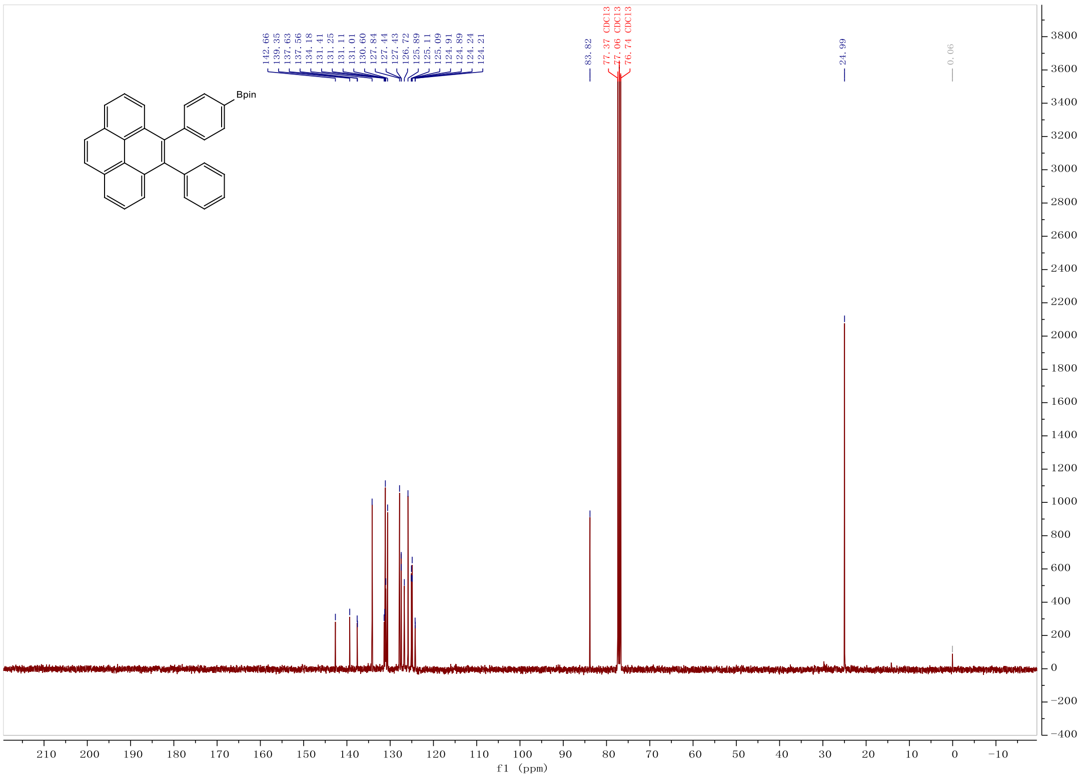

142.94
142.92
136.56
131.39
131.14
130.57
129.74
129.42
129.10
128.77
128.21
127.61
126.20
125.79
125.50
125.06
125.02
124.98
124.94
124.60
124.31
122.80
120.10
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

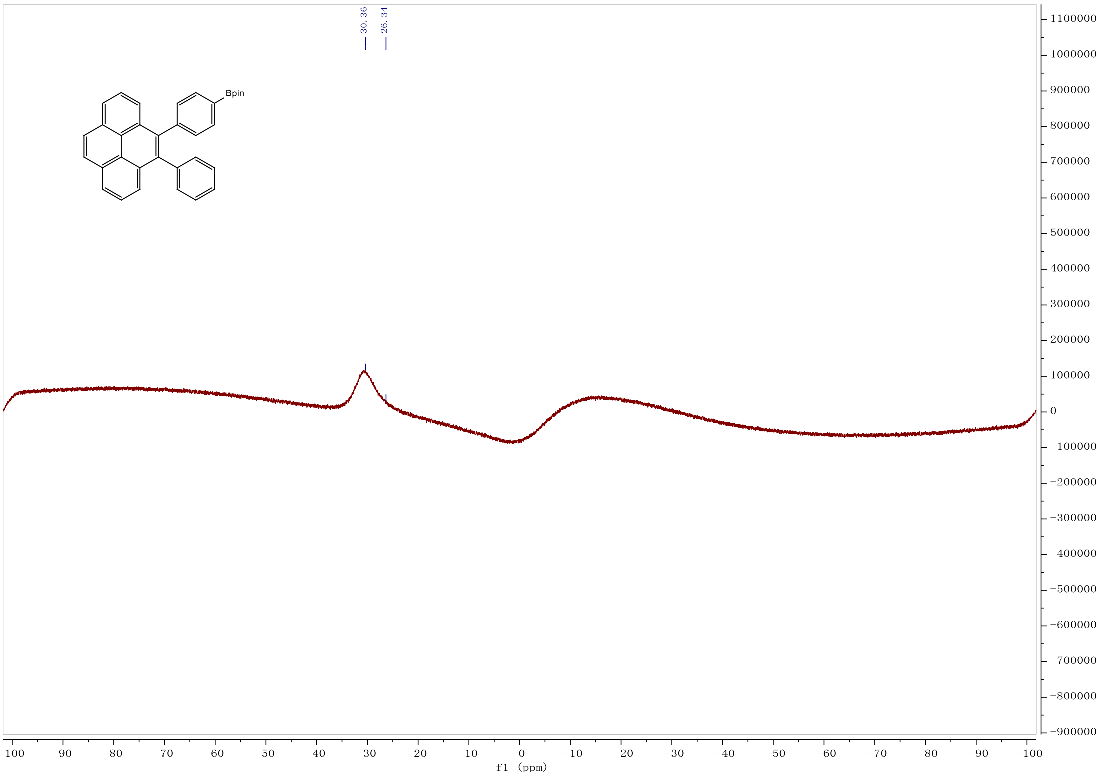

−62.46
F
F
F
F
F
F
9.0E+07
8.0E+07
7.0E+07
6.0E+07
5.0E+07
4.0E+07
3.0E+07
2.0E+07
1.0E+07
0.0E+00
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210 −220
f1 (ppm)

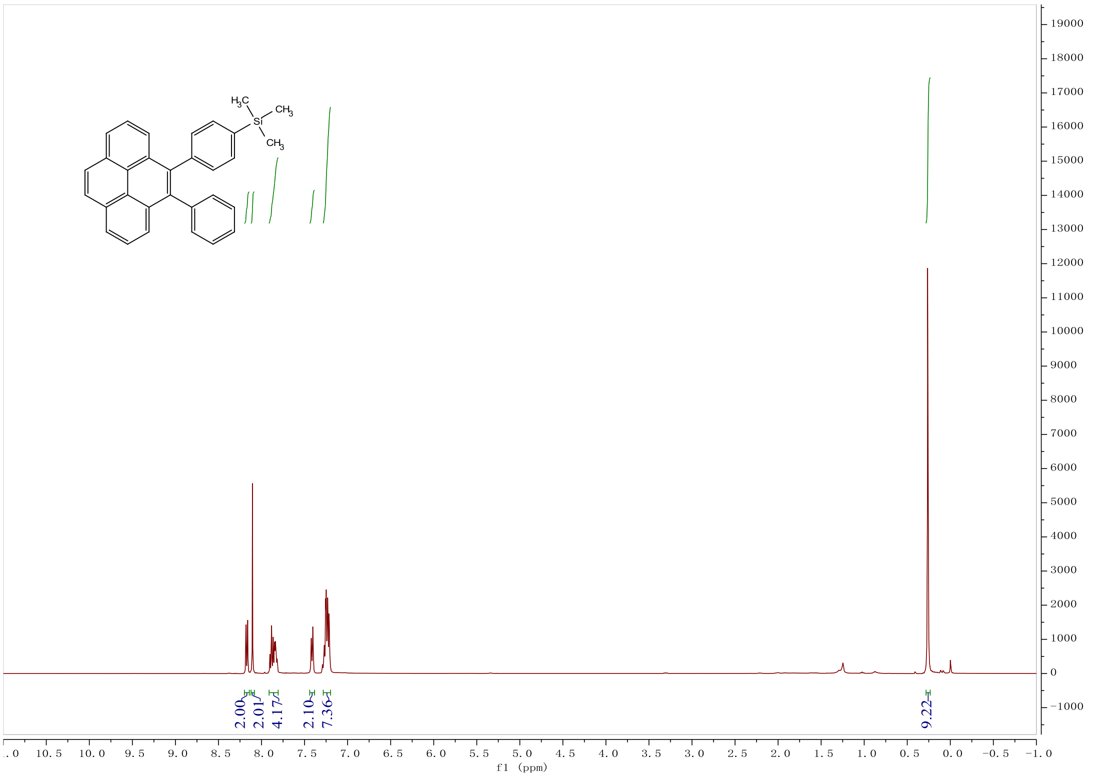

Br
Br
2.00
1.95
2.14
2.07
4.25
4.51
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

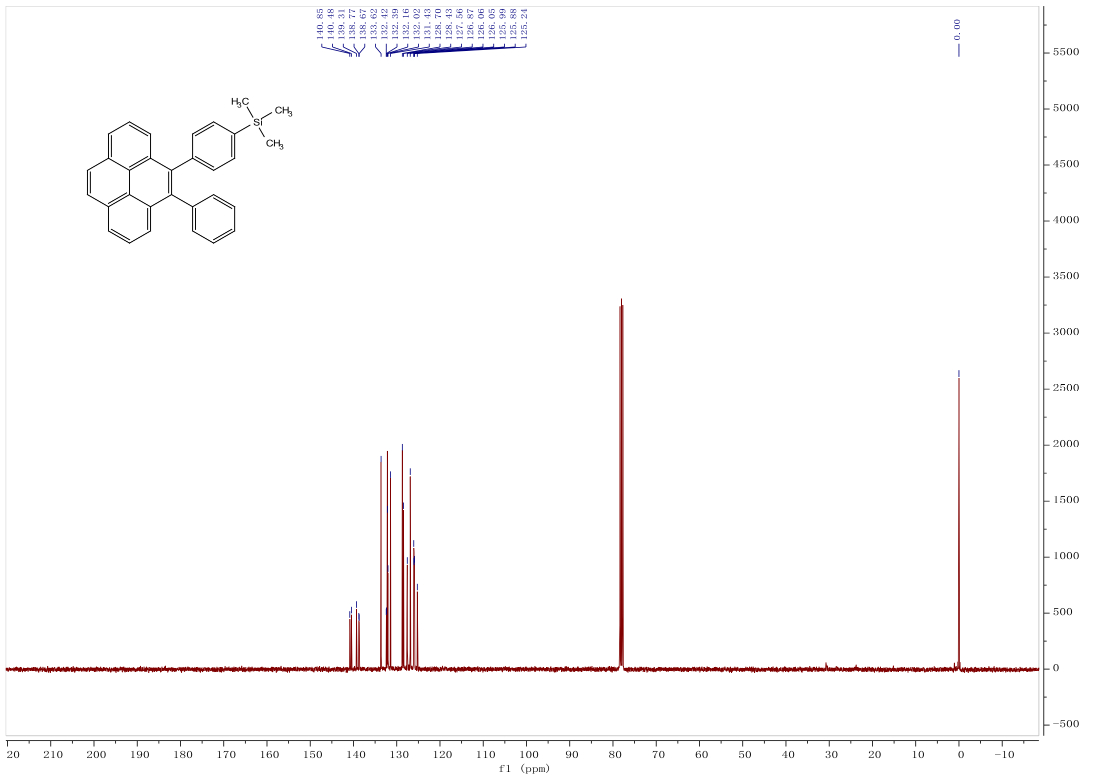

141.24
141.21
136.53
133.97
133.80
131.06
130.70
130.12
129.79
129.67
129.62
129.43
127.54
126.13
125.60
124.73
124.25
122.09
121.94
77.37 CDCl3
77.05 CDCl3
76.74 CDCl3
0.05
Br
Br
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

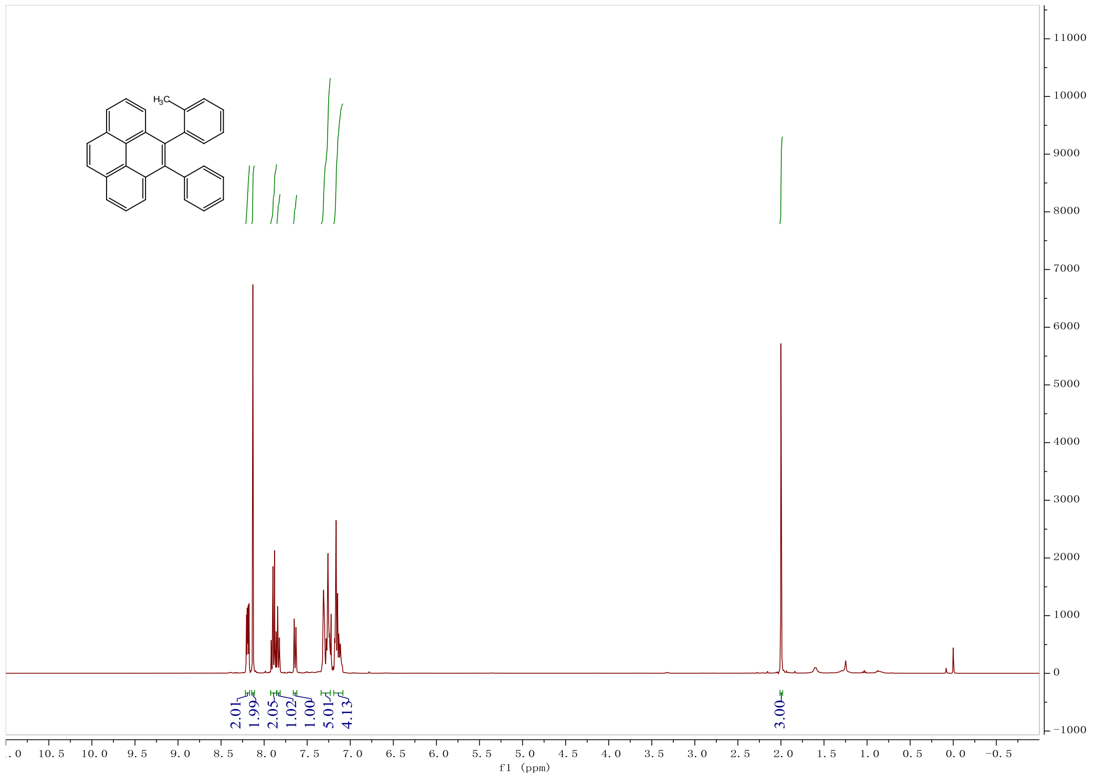

Bpin
1.00
1.06
2.06
2.37
1.09
3.18
7.25
12.38
f1 (ppm)

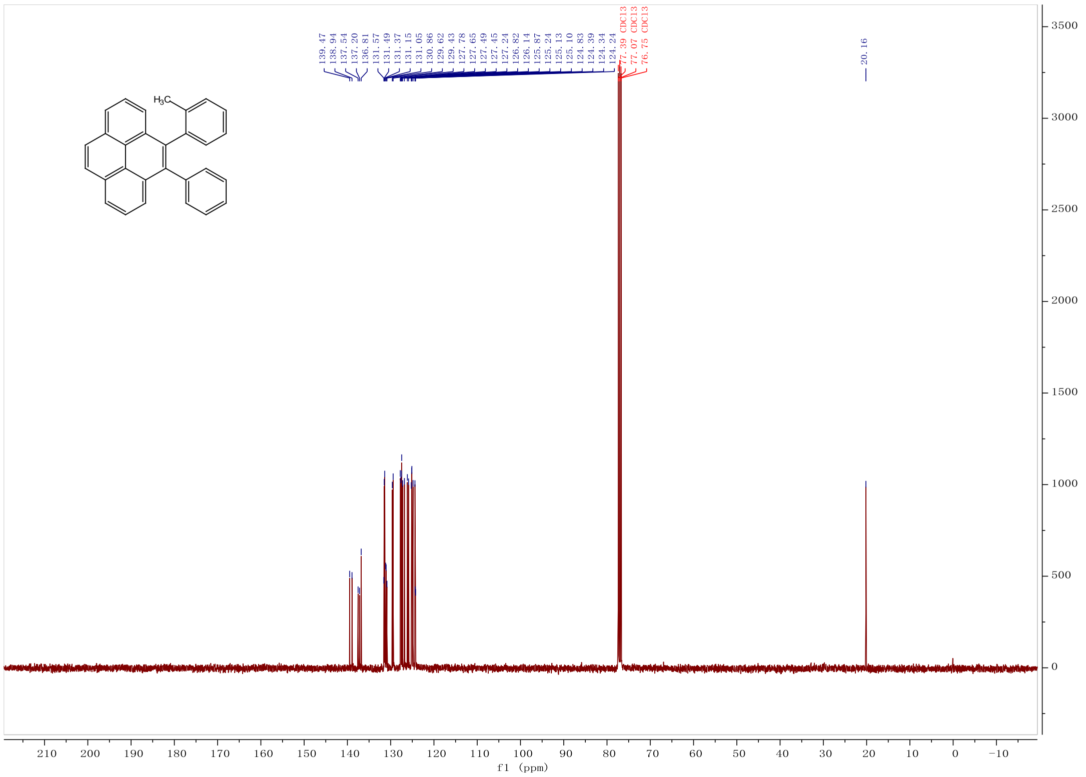

Bpin
142.66
139.35
137.63
137.56
134.18
131.41
131.25
131.11
131.01
130.60
127.84
127.44
127.43
126.72
125.89
125.11
125.09
124.91
124.89
124.24
124.21
83.82
77.37 CDCl3
77.06 CDCl3
76.74 CDCl3
24.99
0.06
f1 (ppm)

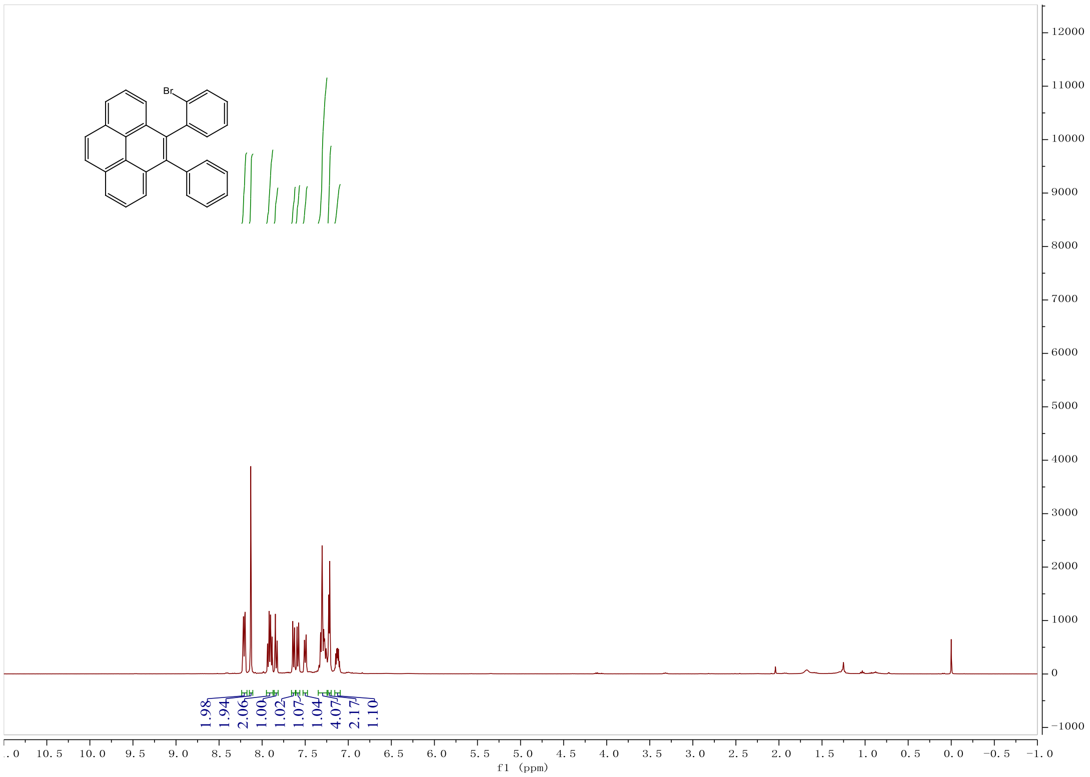

30.36
26.34
Bpin
1100000
1000000
900000
800000
700000
600000
500000
400000
300000
200000
100000
0
−100000
−200000
−300000
−400000
−500000
−600000
−700000
−800000
−900000
100
90
80
70
60
50
40
30
20
10
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
f1 (ppm)

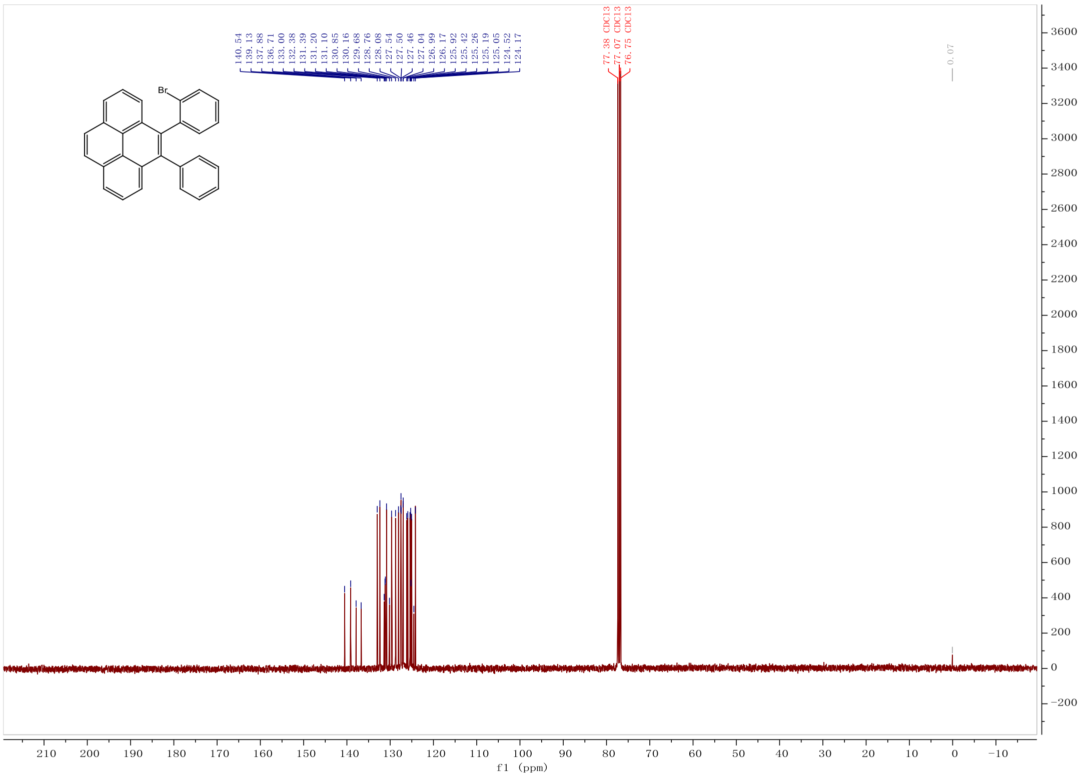

H3C
CH3
Si
CH3
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
2.00
2.01
4.17
2.10
7.36
9.22
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

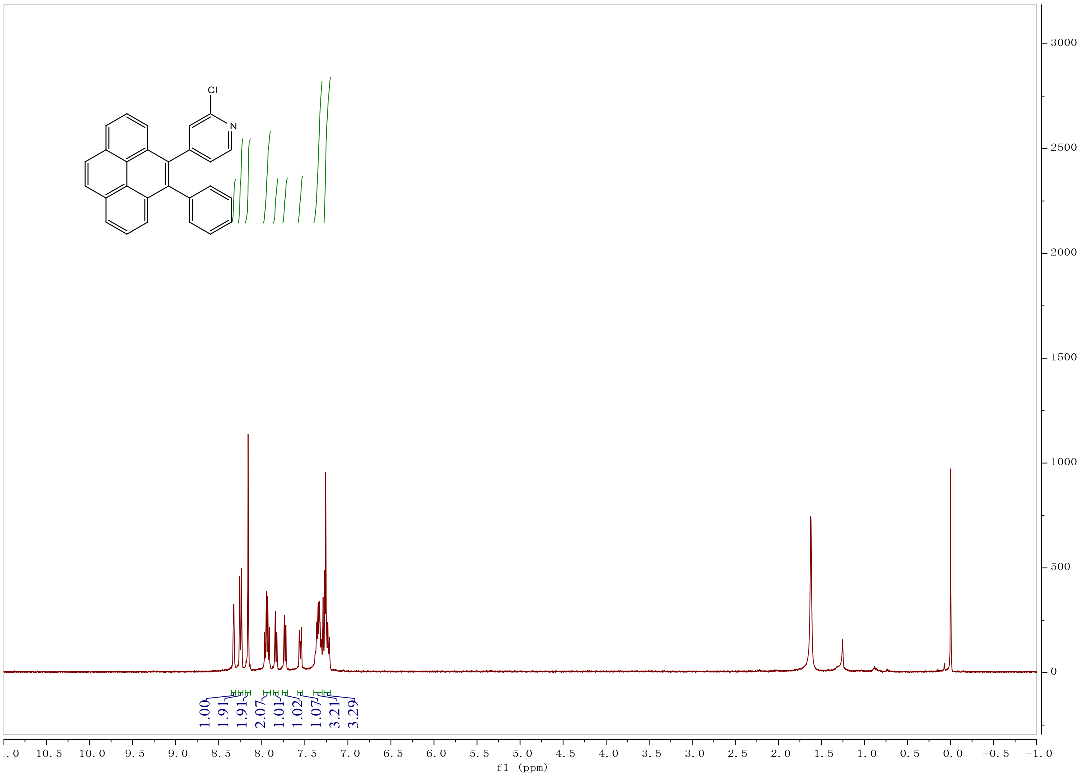

140.85
140.48
139.31
138.77
138.67
133.62
132.42
132.39
132.16
132.02
131.43
128.70
128.43
127.56
126.87
126.06
126.05
125.99
125.88
125.24
0.00
H3C
Si
CH3
CH3
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

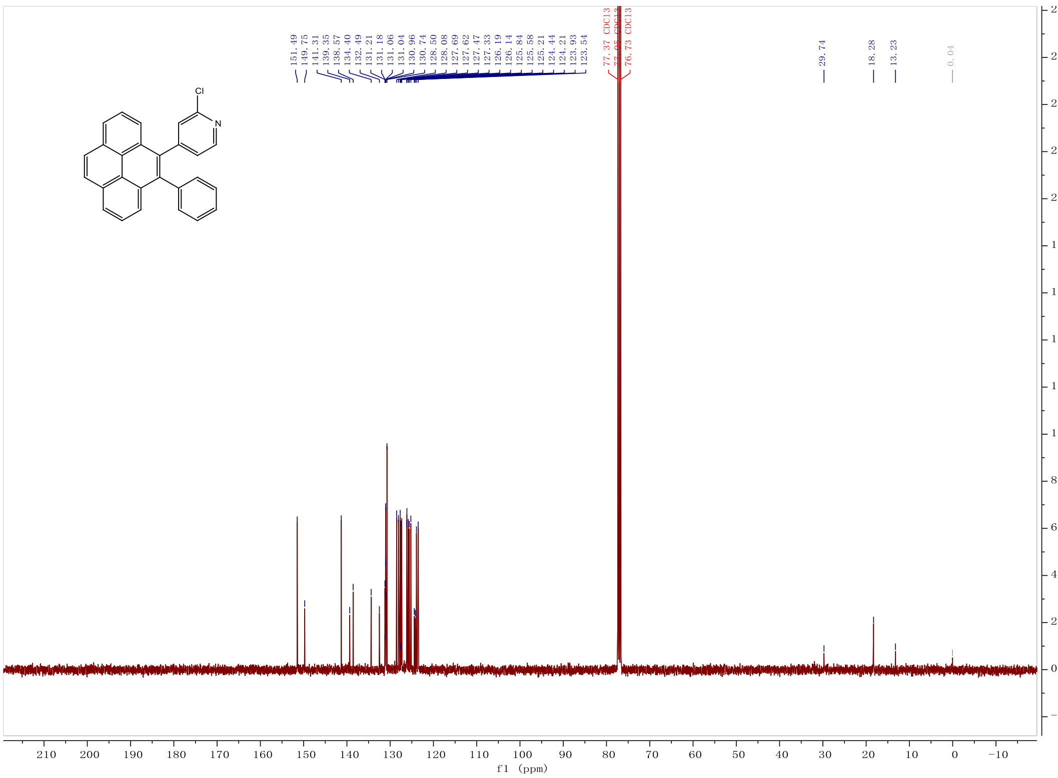

H3C
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
2.01
1.99
2.05
1.02
1.00
5.01
4.13
3.00
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

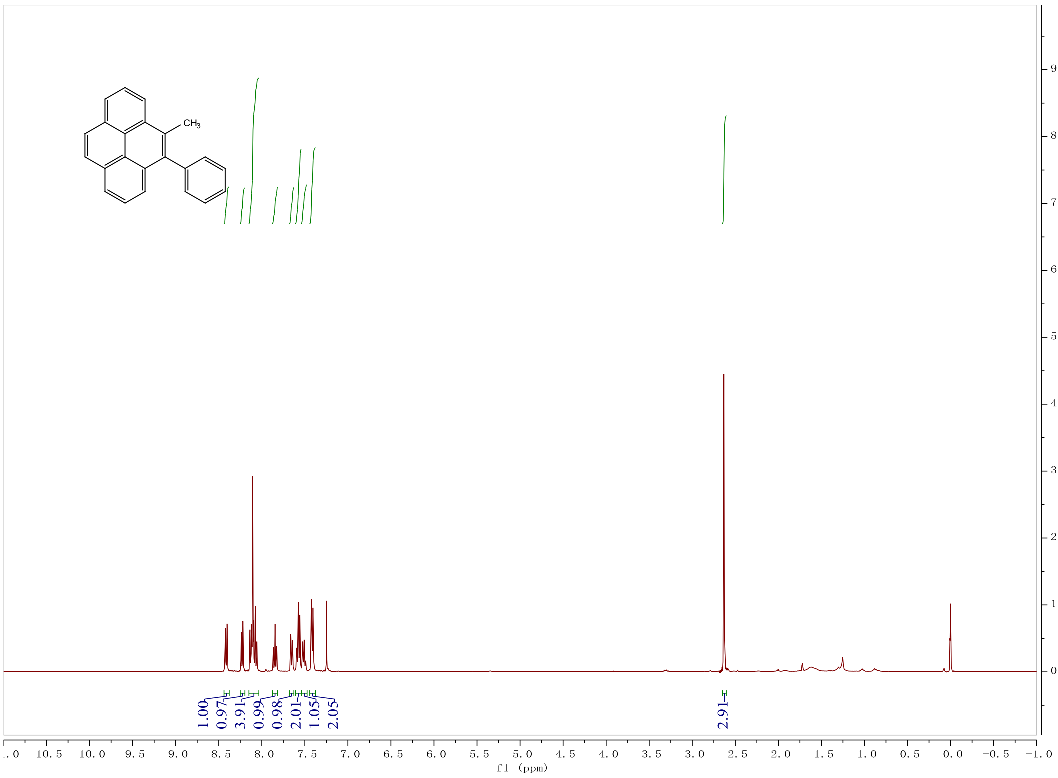

139.47
138.94
137.54
137.20
136.81
131.57
131.49
131.37
131.15
131.05
130.86
129.62
129.43
127.78
127.65
127.49
127.45
127.24
126.82
126.14
125.87
125.24
125.13
125.10
124.83
124.39
124.34
124.21
77.39 CDCl3
77.07 CDCl3
76.75 CDCl3
20.16
$H_3C$
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

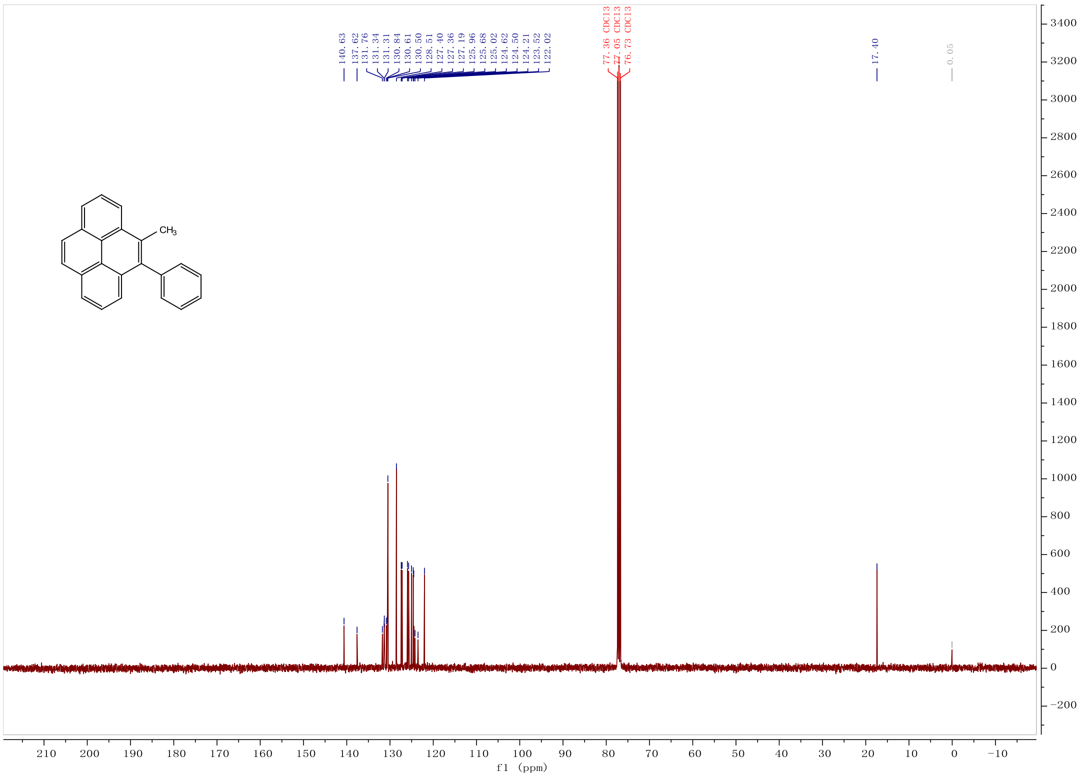

Br
1.98
1.94
2.06
1.00
1.02
1.07
1.04
4.07
2.17
1.10
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

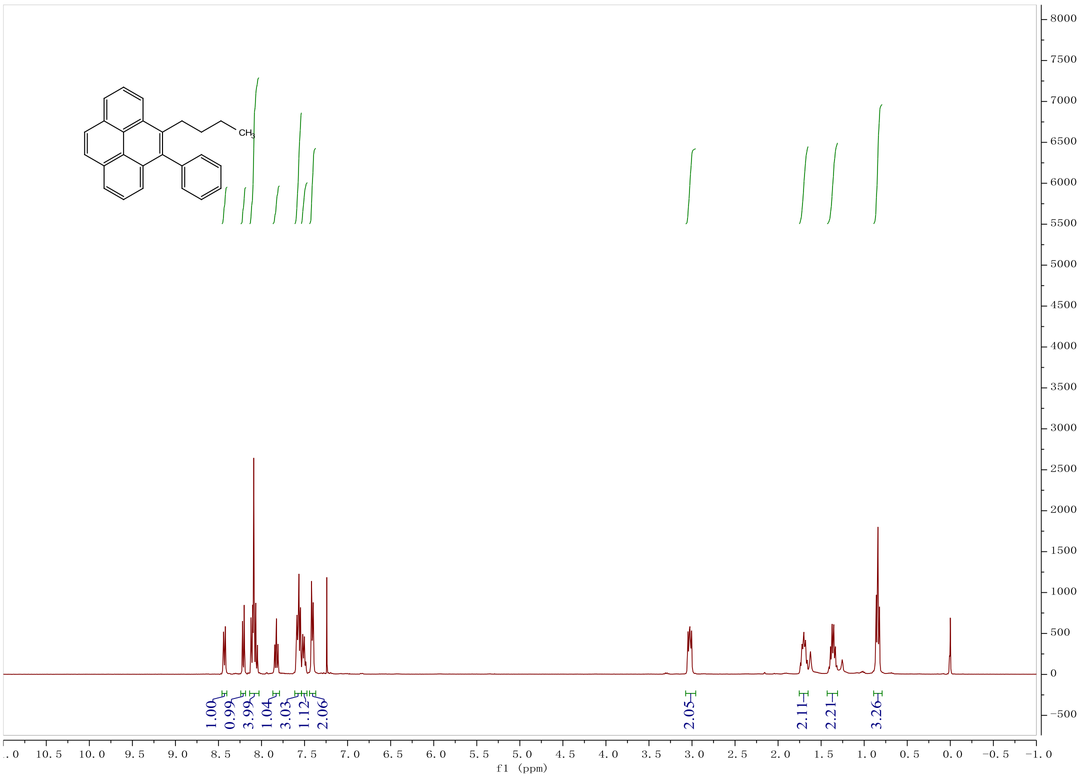

Br
140.54
139.13
137.88
136.71
133.00
132.38
131.39
131.20
131.10
130.85
130.16
129.68
128.76
128.08
127.54
127.50
127.46
127.04
126.99
126.17
125.92
125.42
125.26
125.19
125.05
124.52
124.17
77.38 CDCl3
77.07 CDCl3
76.75 CDCl3
0.07
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

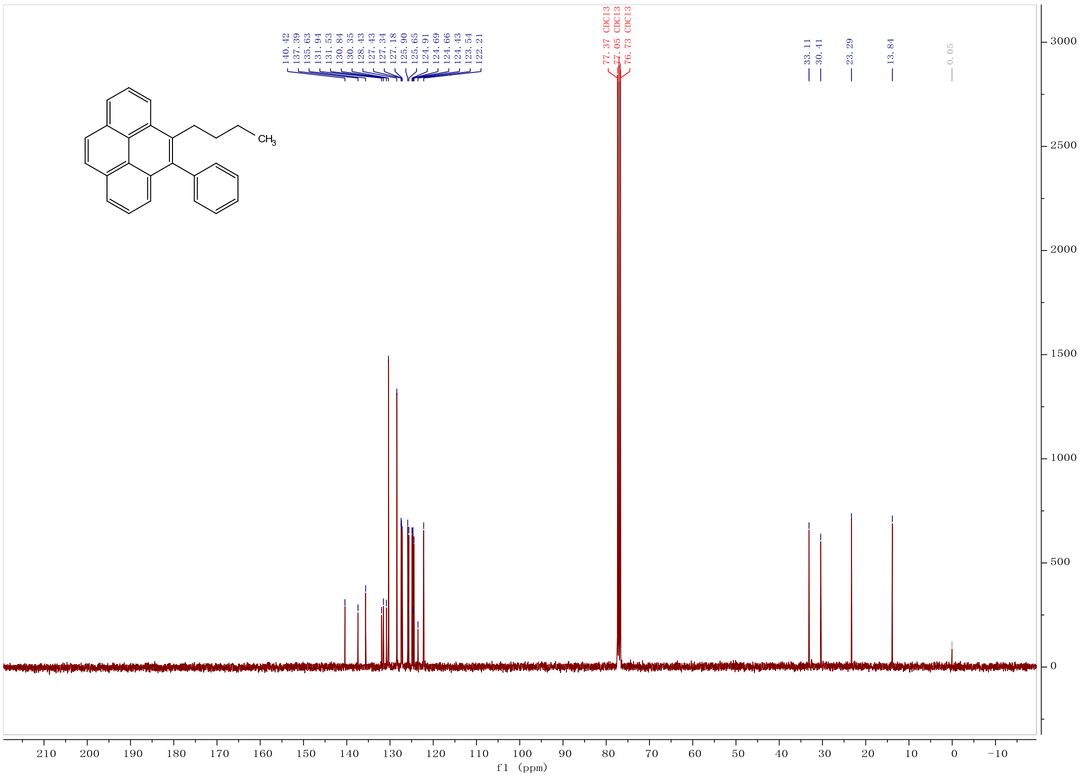

Cl
N
1.00
1.91
1.91
2.07
1.01
1.02
1.07
3.21
3.29
3000
2500
2000
1500
1000
500
0
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

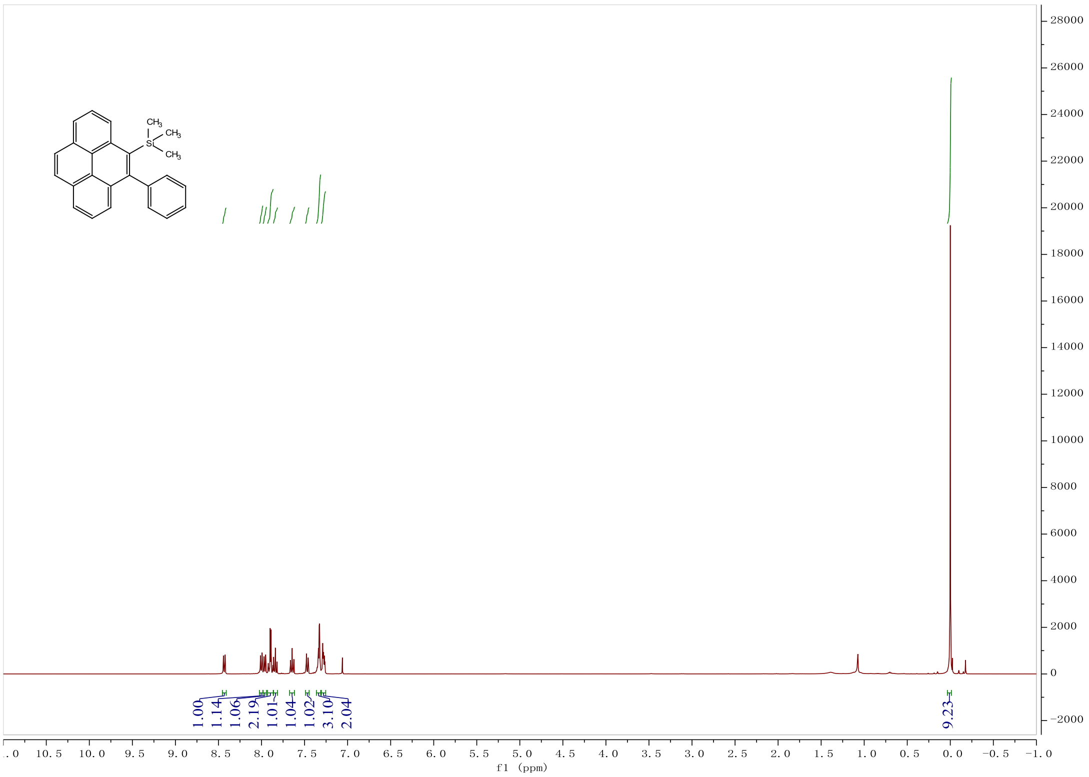

Cl
N
151.49
149.75
141.31
139.35
138.57
134.40
132.49
131.21
131.18
131.06
131.04
130.96
130.74
128.50
128.08
127.69
127.62
127.47
127.33
126.19
126.14
125.84
125.58
125.21
124.44
124.21
123.93
123.54
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
29.74
18.28
13.23
0.04
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

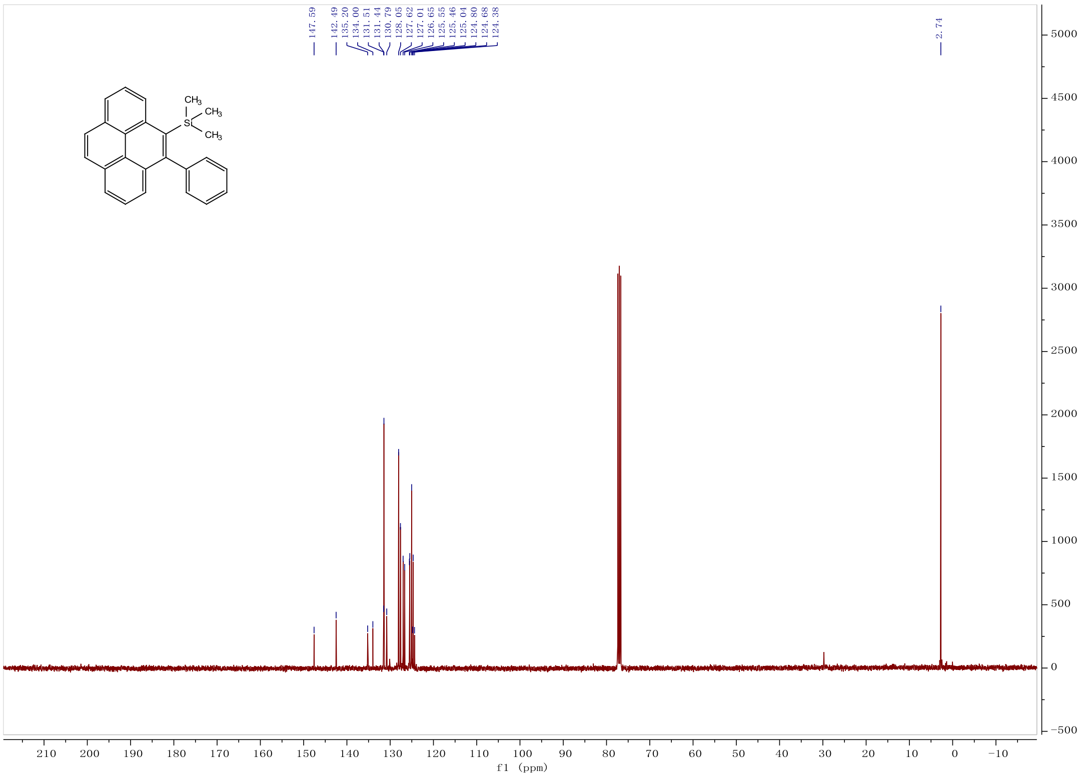

CH3
1.00
0.97
3.91
0.99
0.98
2.01
1.05
2.05
2.91
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

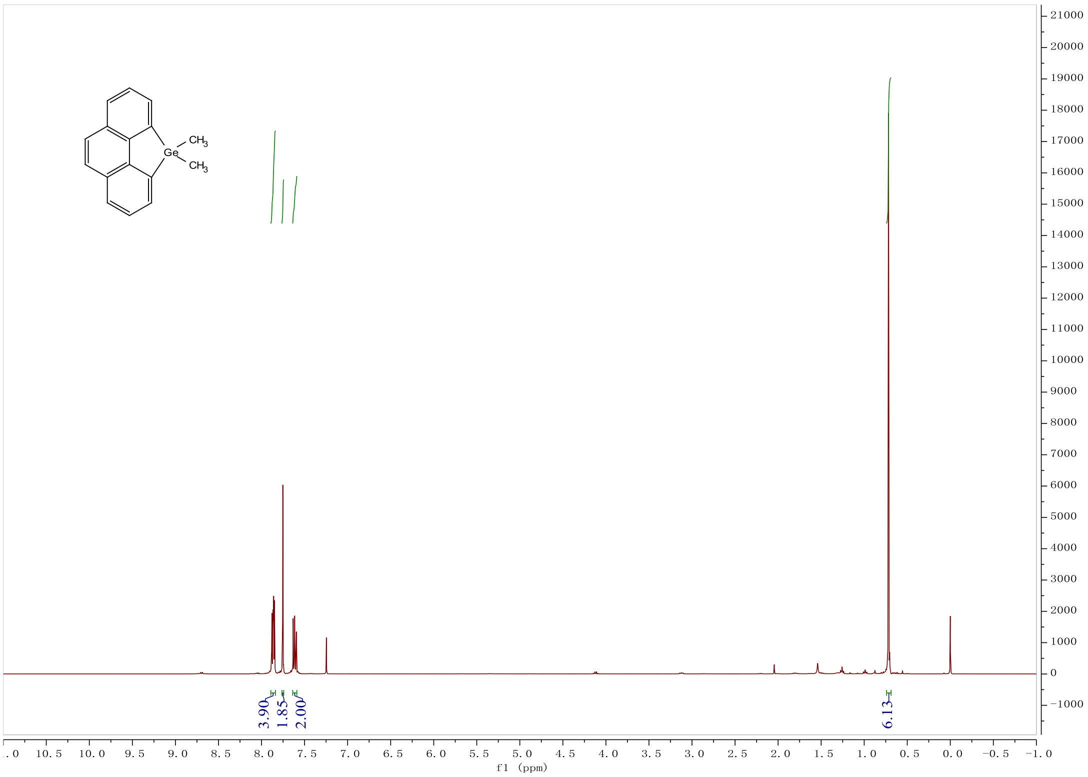

140.63
137.62
131.76
131.34
131.31
130.84
130.61
130.50
128.51
127.40
127.36
127.19
125.96
125.68
125.02
124.62
124.50
124.21
123.52
122.02
77.36 CDCl3
77.05 CDCl3
76.73 CDCl3
17.40
0.05
$CH_3$
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

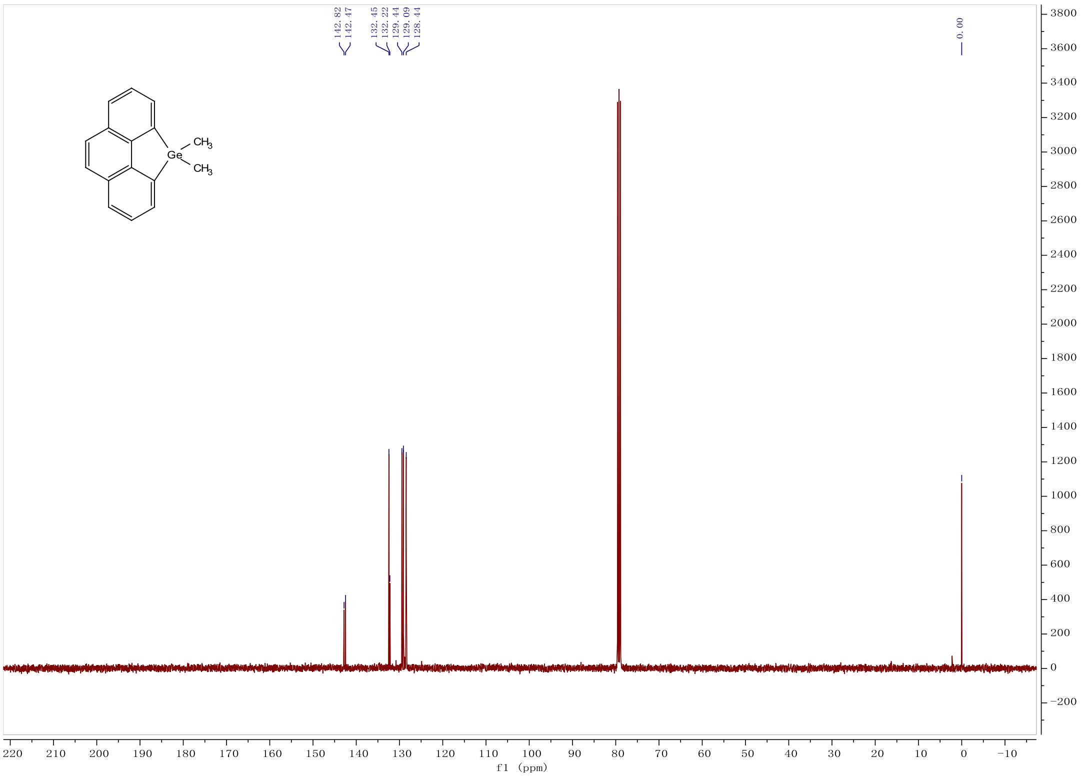

$CH_3$
1.00
0.99
3.99
1.04
3.03
1.12
2.06
2.05
2.11
2.21
3.26
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

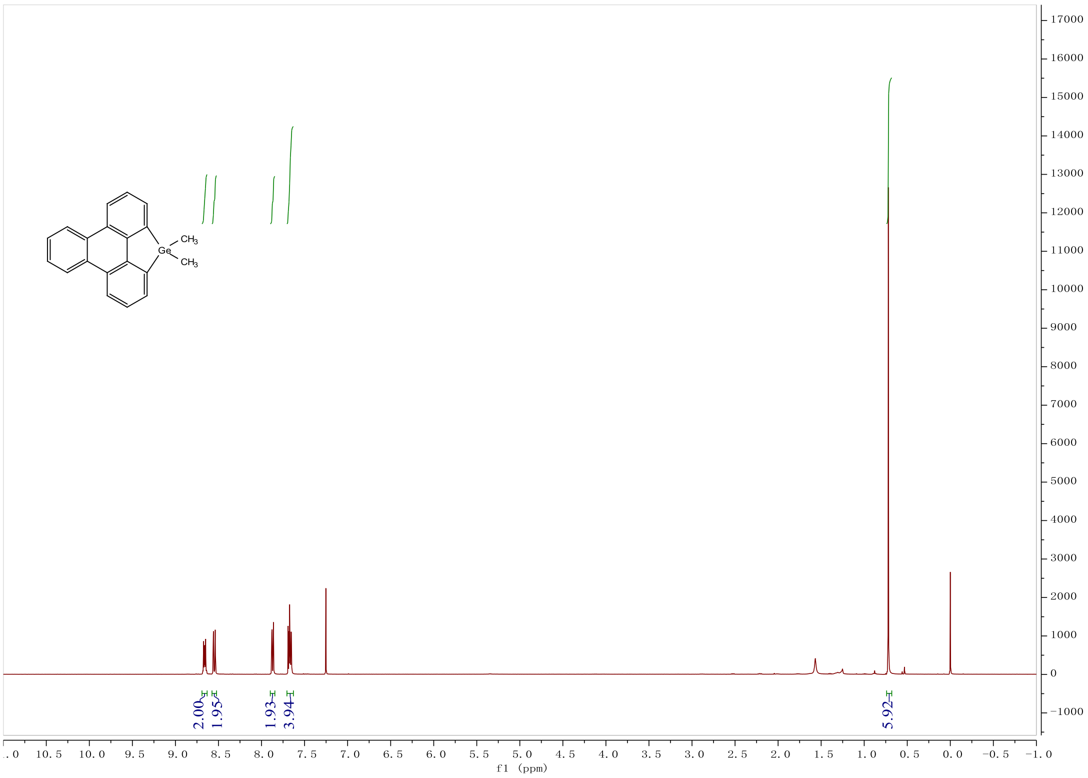

CH3
140.42
137.39
135.63
131.94
131.53
130.84
130.35
128.43
127.43
127.34
127.18
125.90
125.65
124.91
124.69
124.66
124.43
123.54
122.21
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
33.11
30.41
23.29
13.84
0.05
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

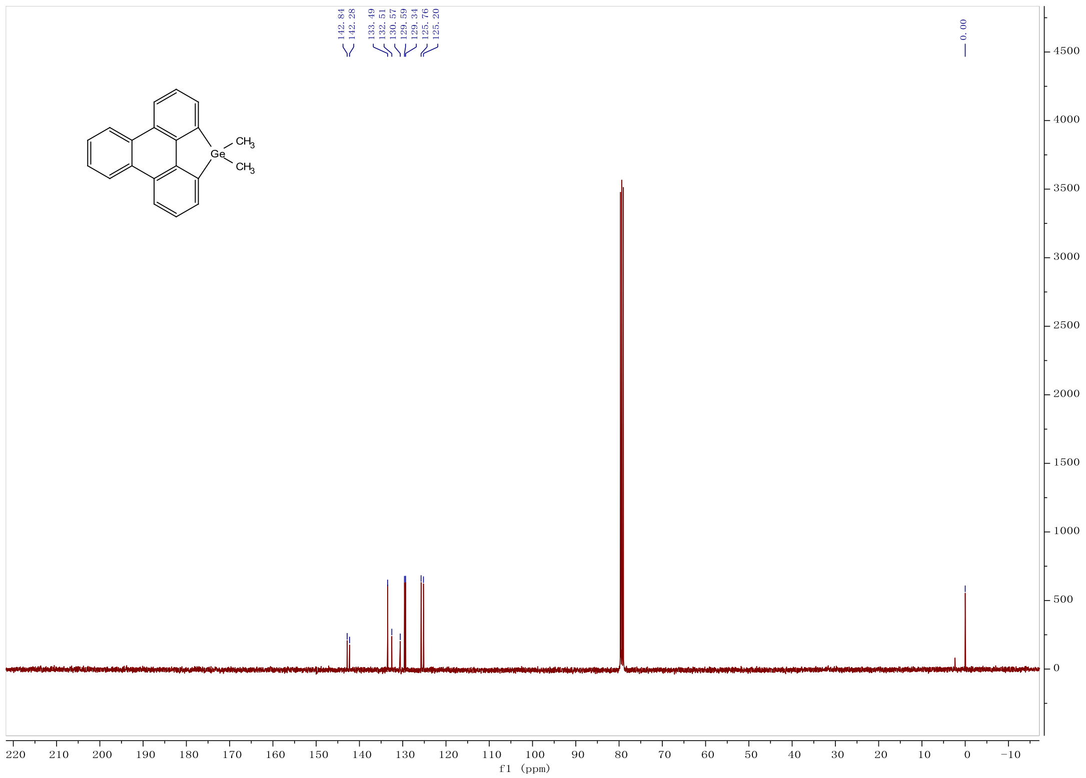

CH3
Si
CH3
CH3
1.00
1.14
1.06
2.19
1.01
1.04
1.02
3.10
2.04
9.23
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

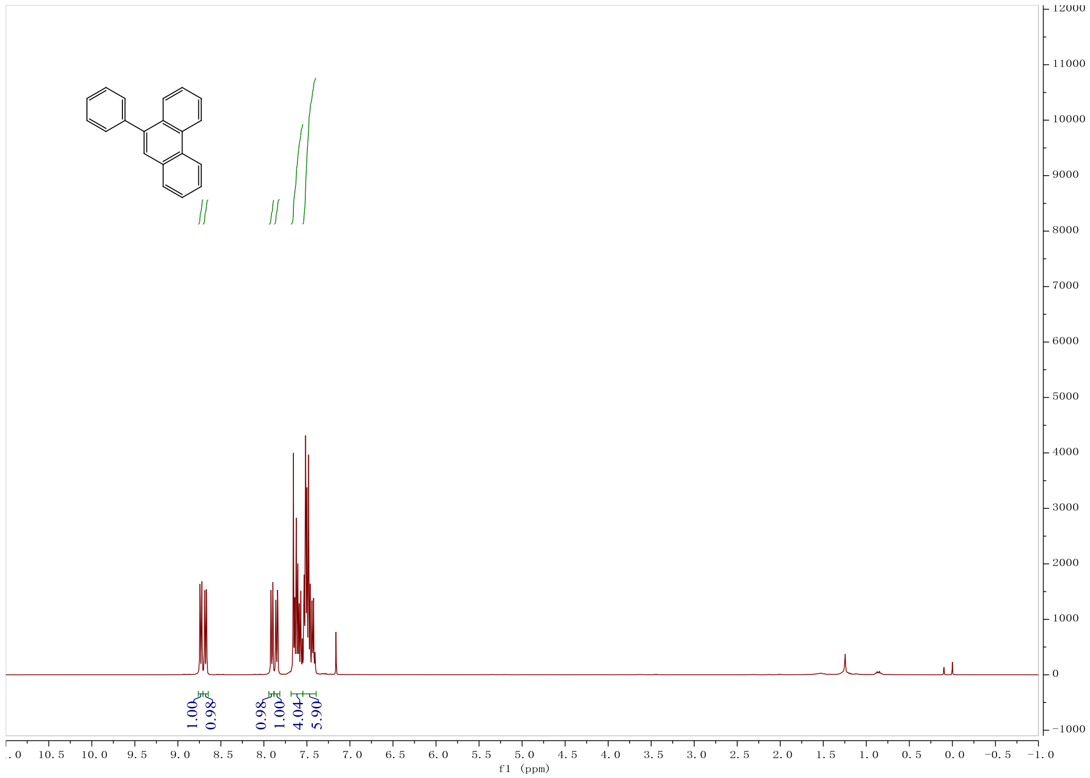

147.59
142.49
135.20
134.00
131.51
131.44
130.79
128.05
127.62
127.01
126.65
125.55
125.46
125.04
124.80
124.68
124.38
2.74
CH3
CH3
CH3
Si
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

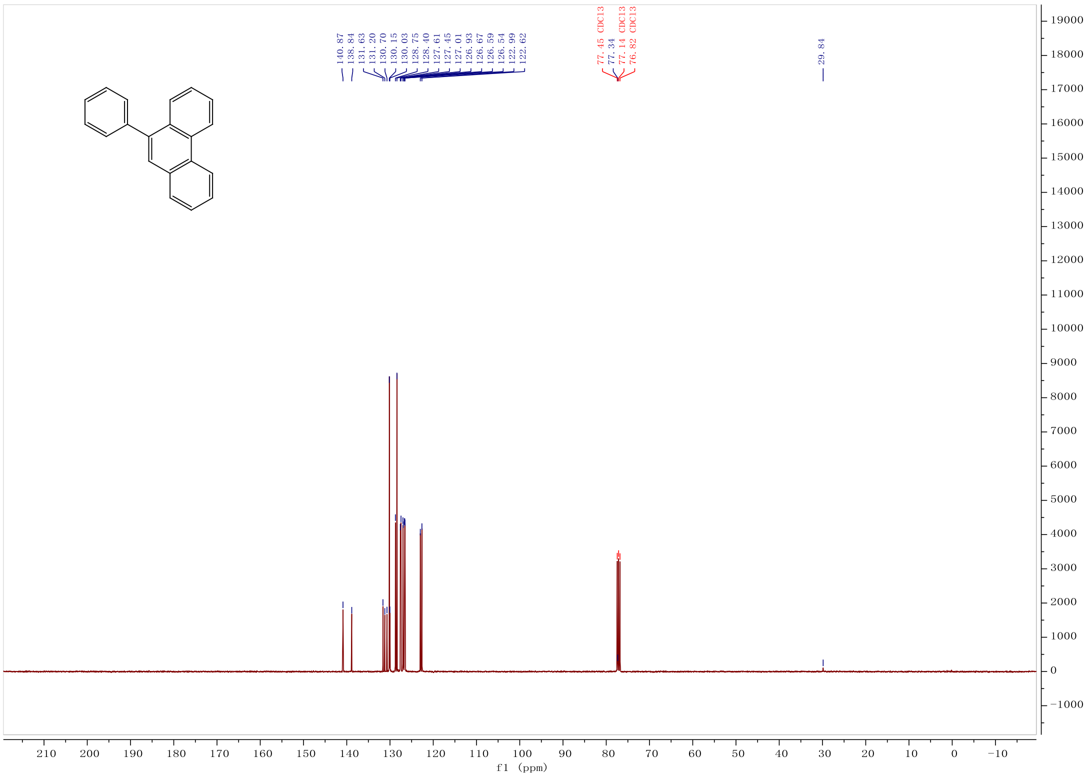

Ge
CH3
CH3
3.90
1.85
2.00
6.13
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
21000 20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000

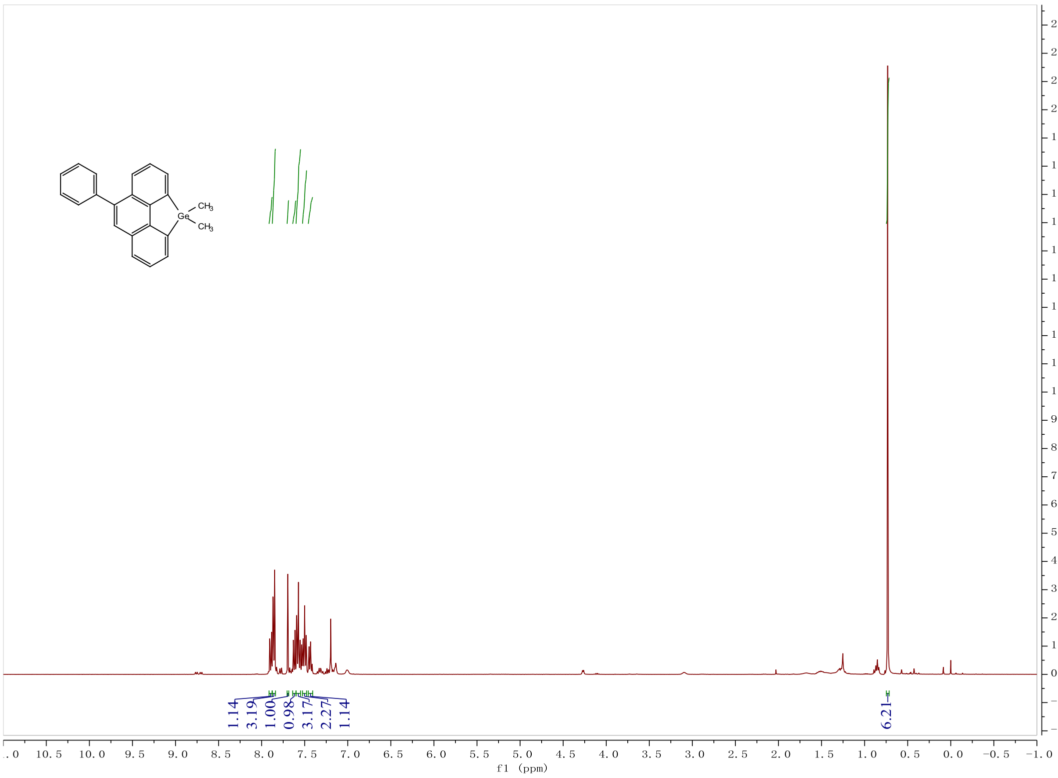

142.82
142.47
132.45
132.22
129.44
129.09
128.44
0.00
Ge
CH3
CH3
f1 (ppm)

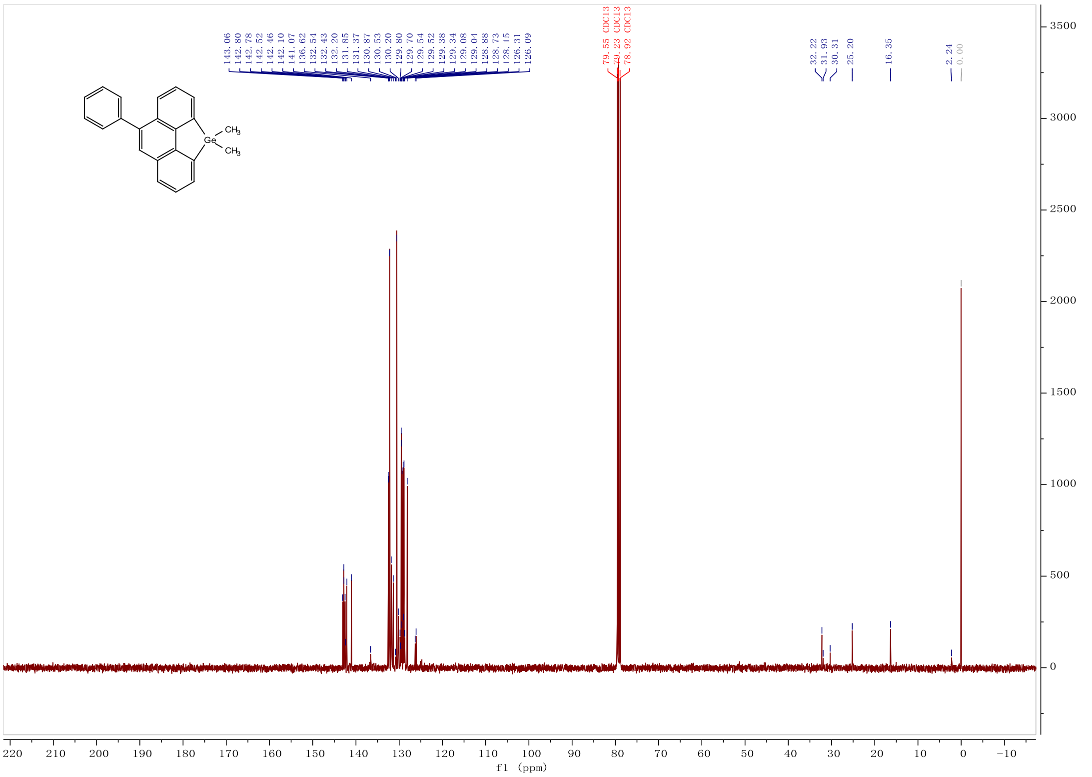

Ge
CH3
CH3
2.00
1.95
1.93
3.94
5.92
f1 (ppm)


142.84
142.28
133.49
132.51
130.57
129.59
129.34
125.76
125.20
0.00
Ge
CH3
CH3
f1 (ppm)

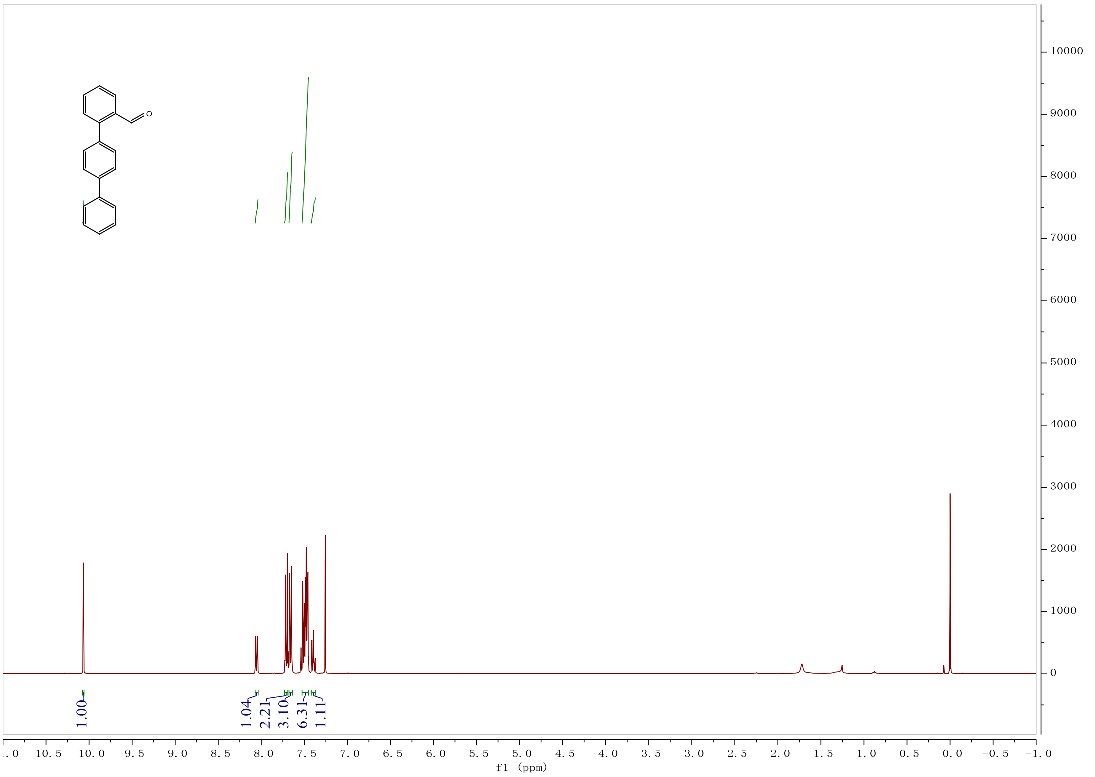

1.00
0.98
0.98
1.00
4.04
5.90
f1 (ppm)


140.87
138.84
131.63
131.20
130.70
130.15
130.03
128.75
128.40
127.61
127.45
127.01
126.93
126.67
126.59
126.54
122.99
122.62
77.45 CDCl3
77.34
77.14 CDCl3
76.82 CDCl3
29.84
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

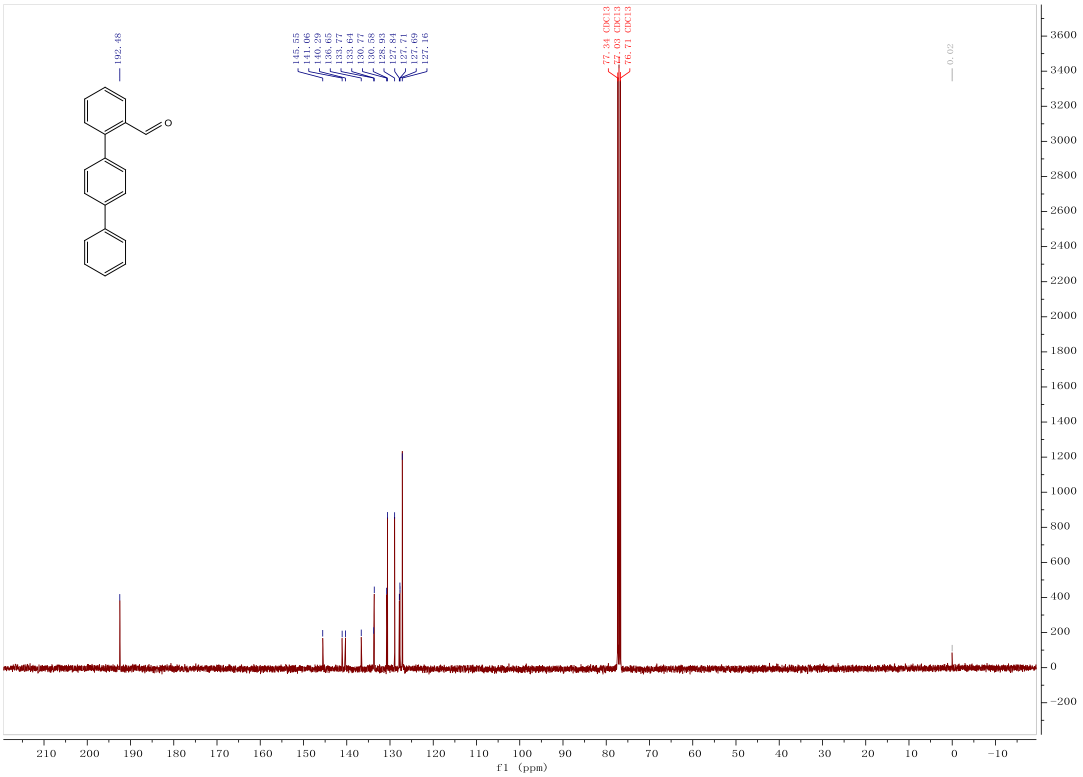

Ge
CH3
CH3
1.14
3.19
1.00
0.98
3.17
2.27
1.14
6.21
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
−2000
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)



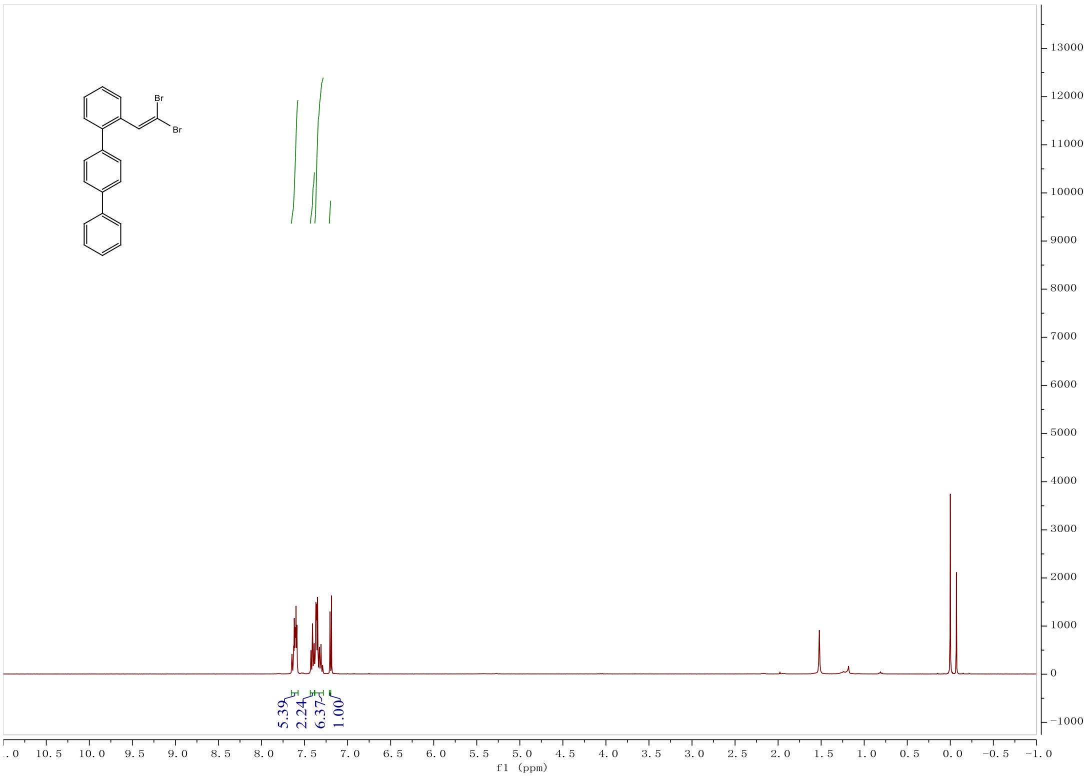
143.06
142.80
142.78
142.52
142.46
142.10
141.07
136.62
132.54
132.43
132.20
131.85
131.37
130.87
130.53
130.20
129.80
129.70
129.54
129.52
129.38
129.34
129.08
129.04
128.88
128.73
128.15
126.31
126.09
79.55 CDCl3
79.23 CDCl3
78.92 CDCl3
32.22
31.93
30.31
25.20
16.35
2.24
0.00
Ge
CH3
CH3
3500
3000
2500
2000
1500
1000
500
0
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)



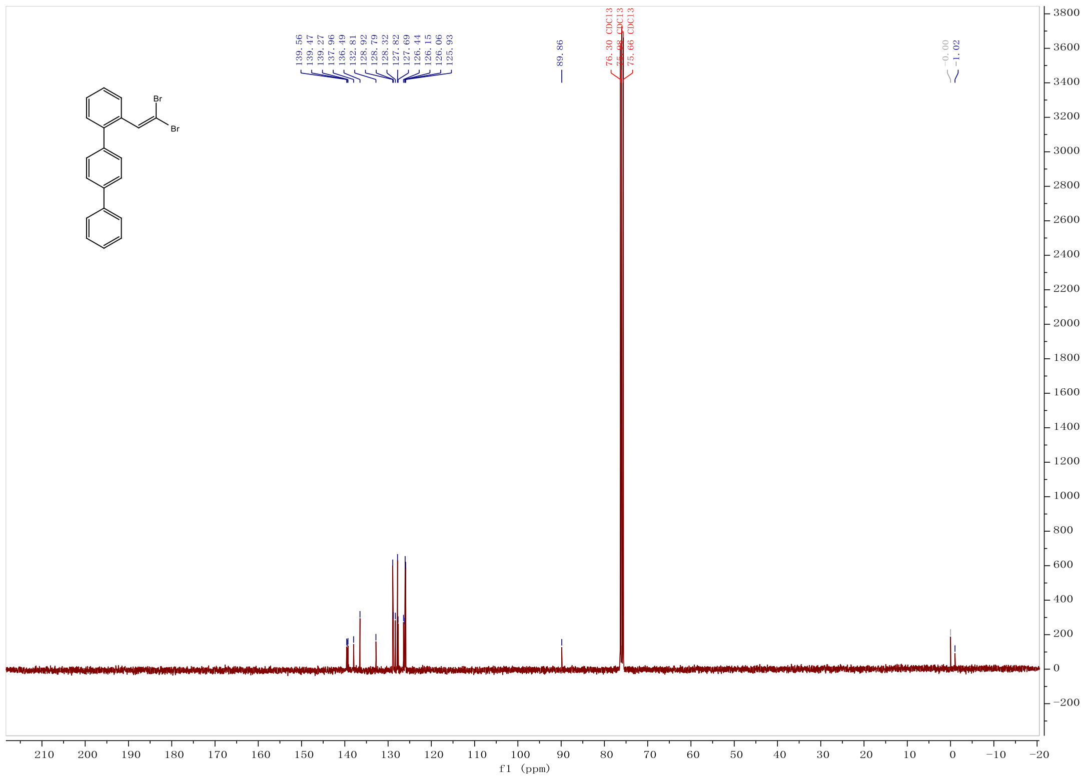
O
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
1.00
1.04
2.21
3.10
6.31
1.11
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)



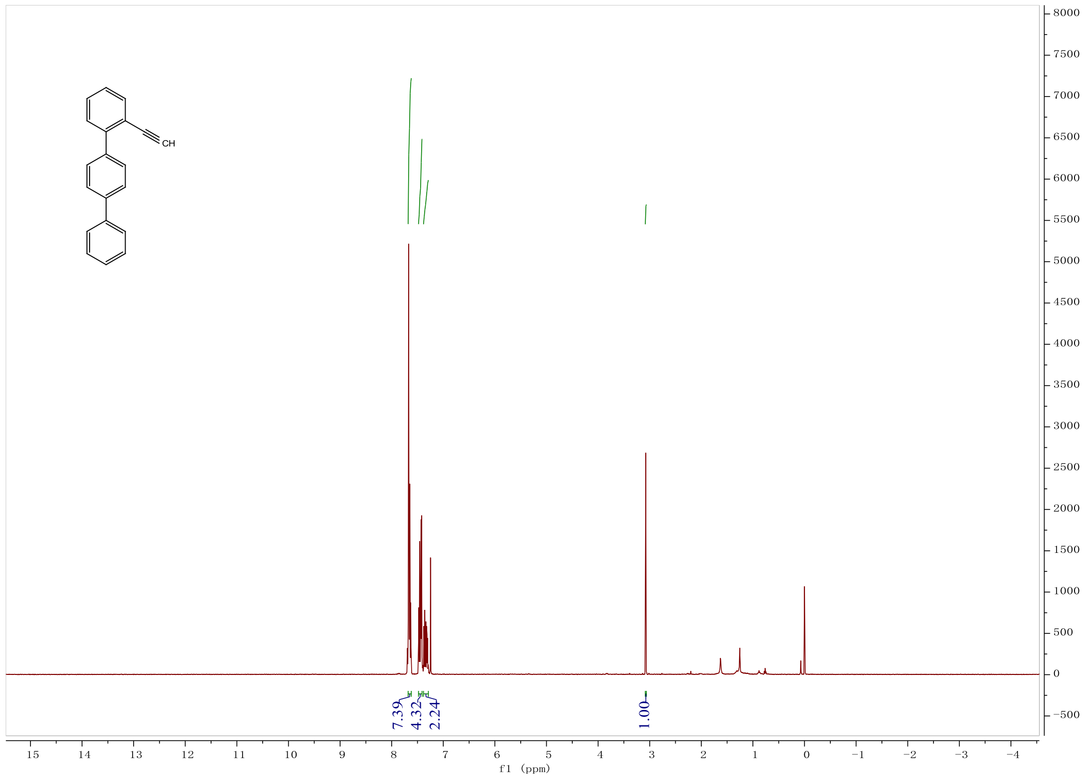
192.48
145.55
141.06
140.29
136.65
133.77
133.64
130.77
130.58
128.93
127.84
127.71
127.69
127.16
77.34 CDCl3
77.03 CDCl3
76.71 CDCl3
0.02
O
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)



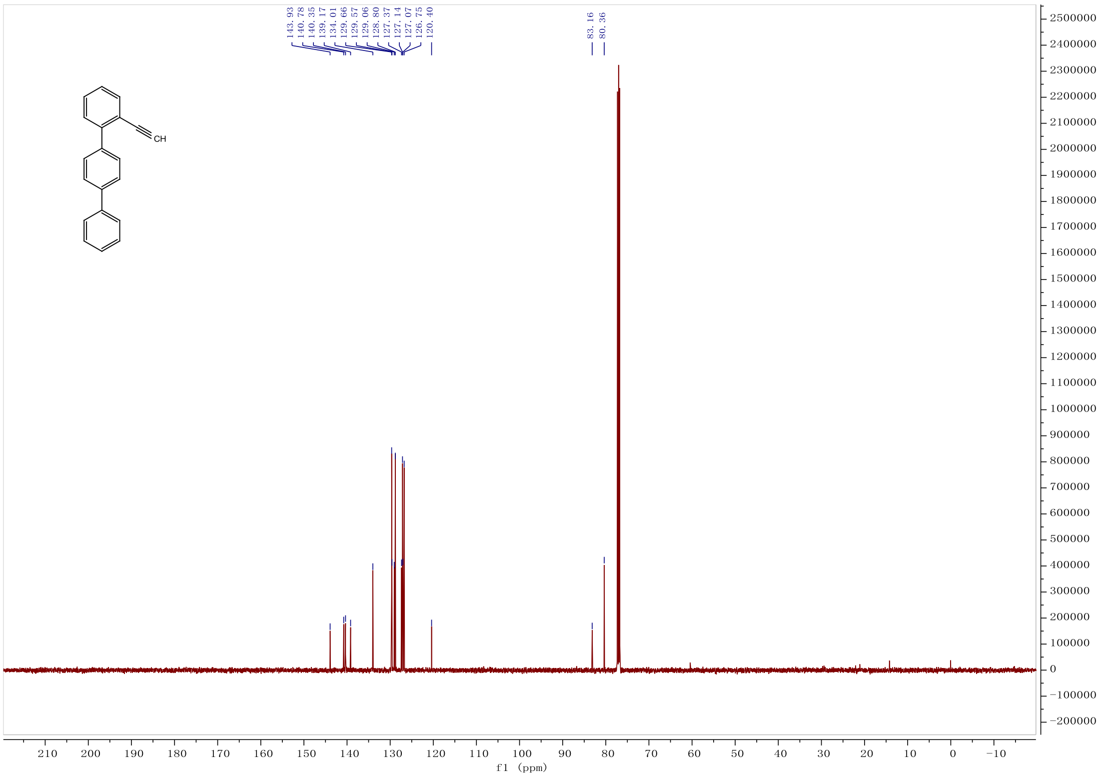
Br
Br
5.39
2.24
6.37
1.00
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)



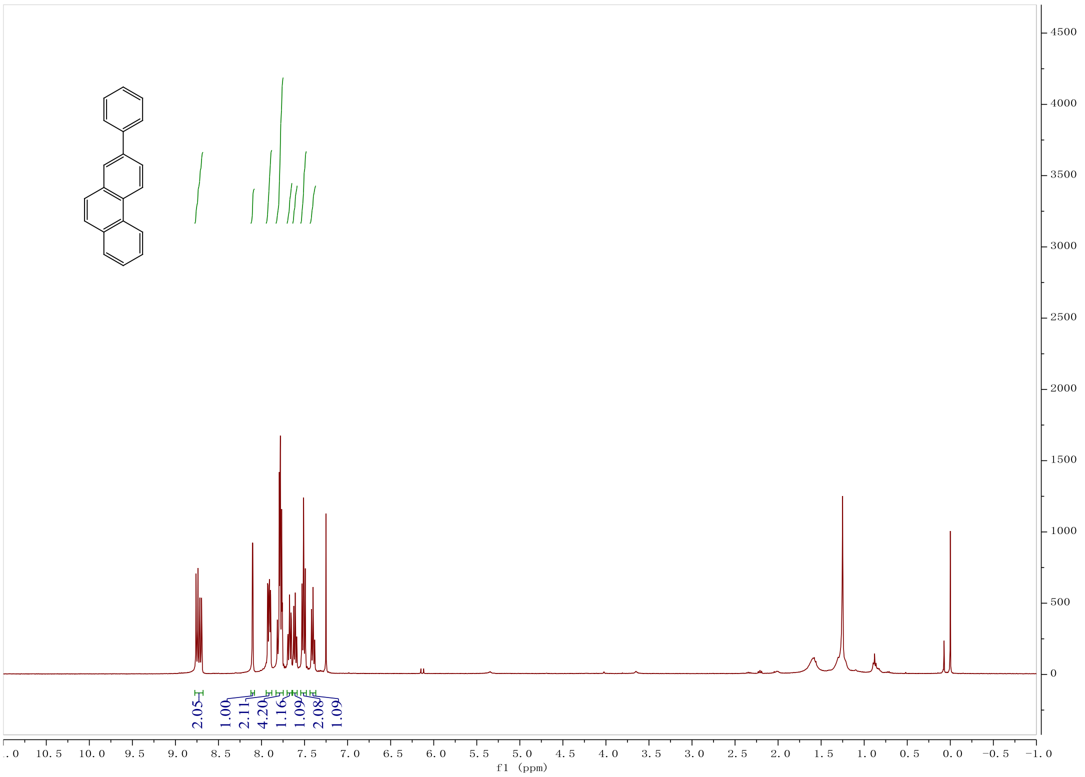
Br
Br
139.56
139.47
139.27
137.96
136.49
132.81
128.92
128.79
128.32
127.82
127.69
126.44
126.15
126.06
125.93
89.86
76.30 CDCl3
75.98 CDCl3
75.66 CDCl3
0.00
-1.02
f1 (ppm)



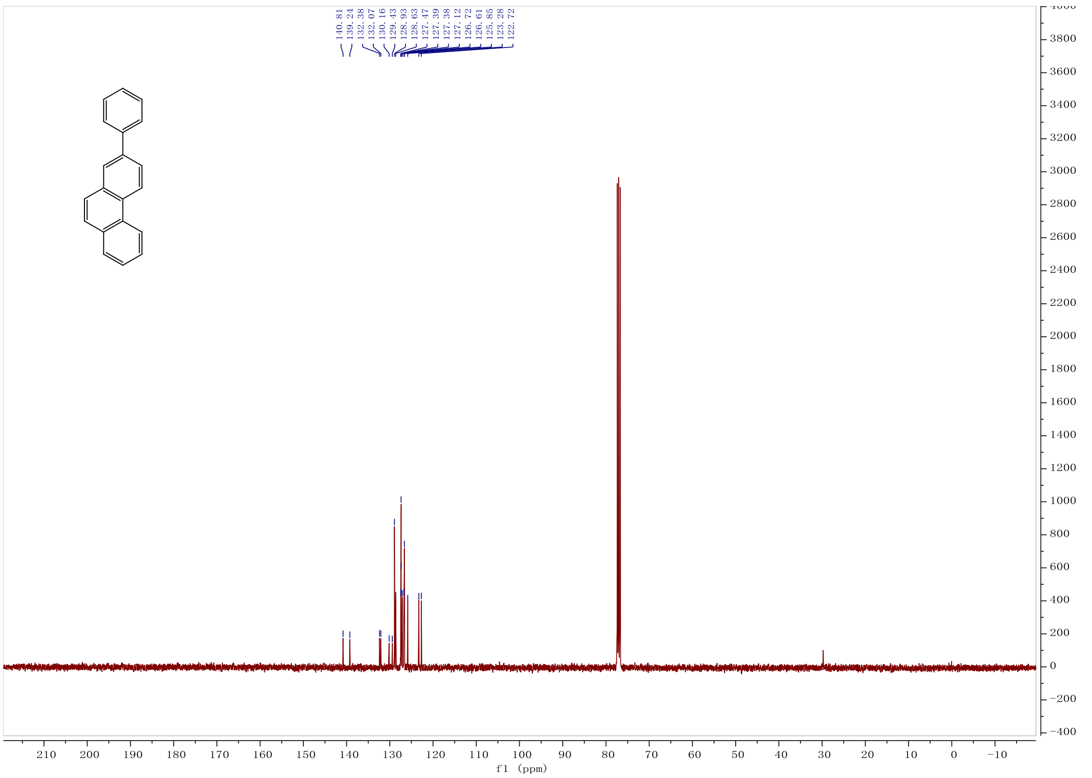
CH
7.39
4.32
2.24
1.00
f1 (ppm)

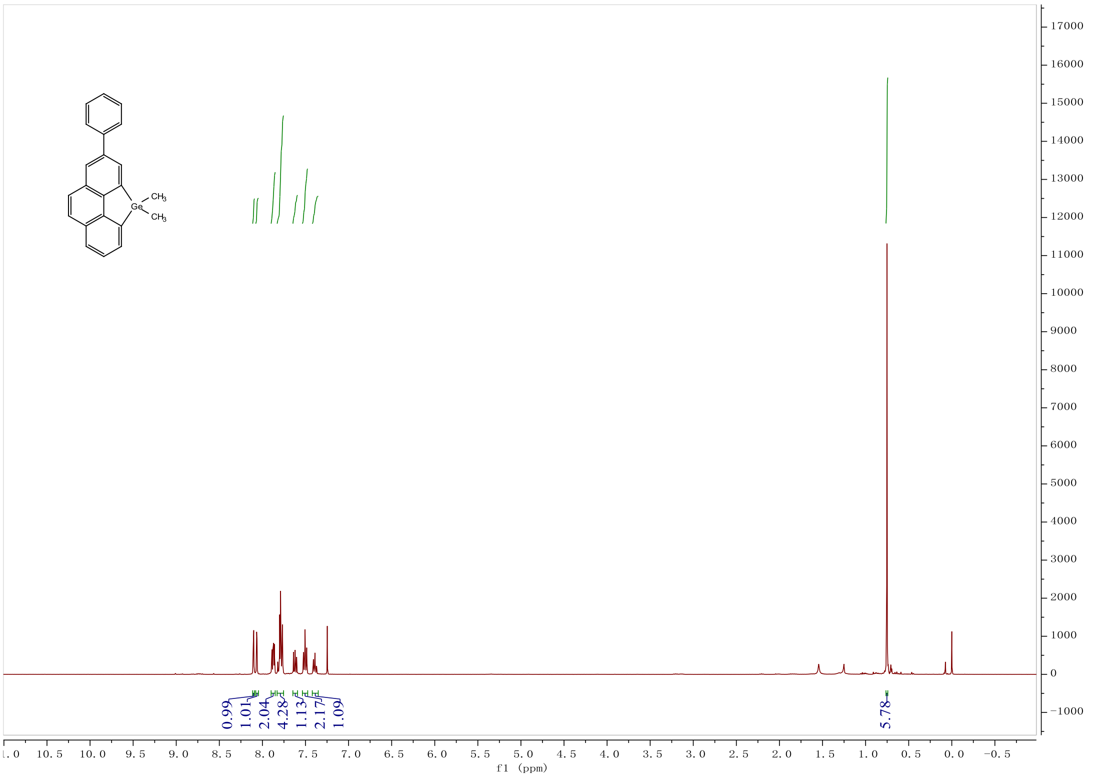


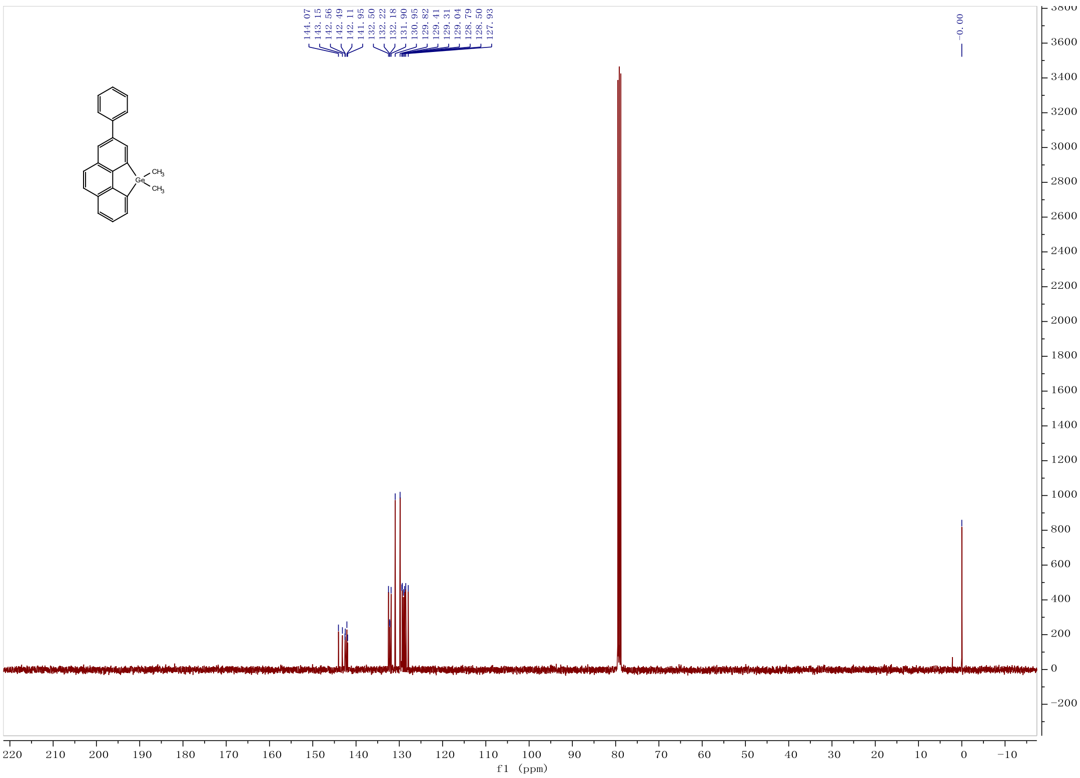
143.93
140.78
140.35
139.17
134.01
129.66
129.57
129.06
128.80
127.37
127.14
127.07
126.75
120.40
83.16
80.36
CH
f1 (ppm)

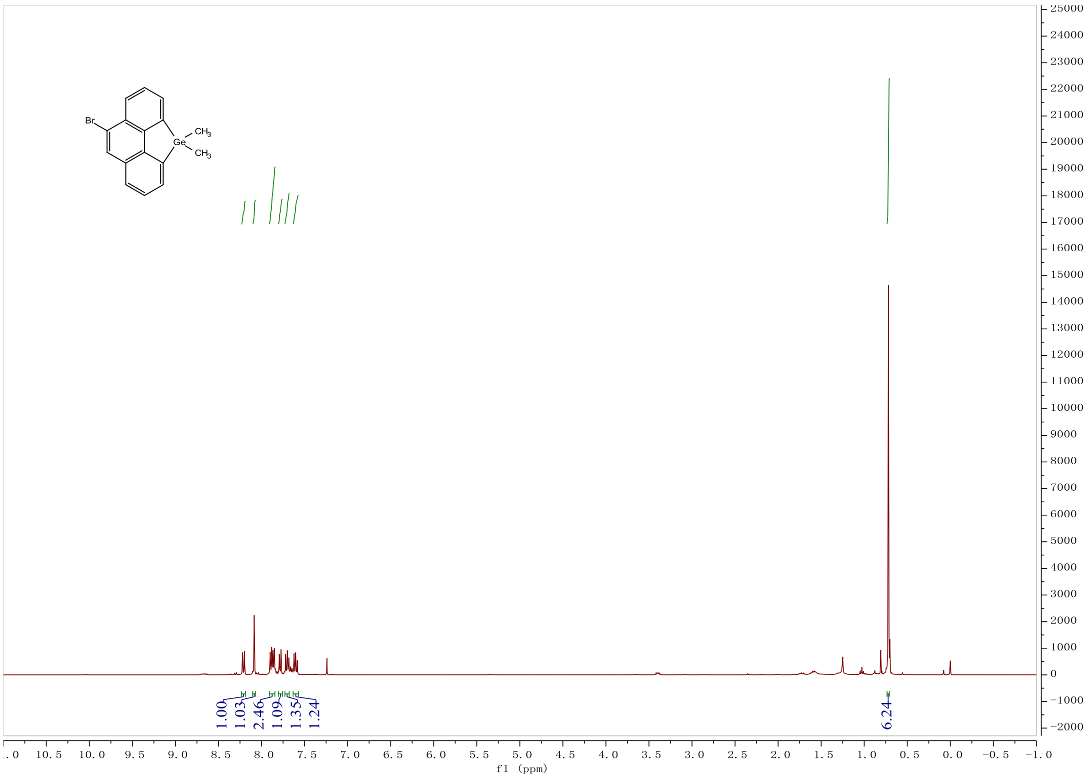


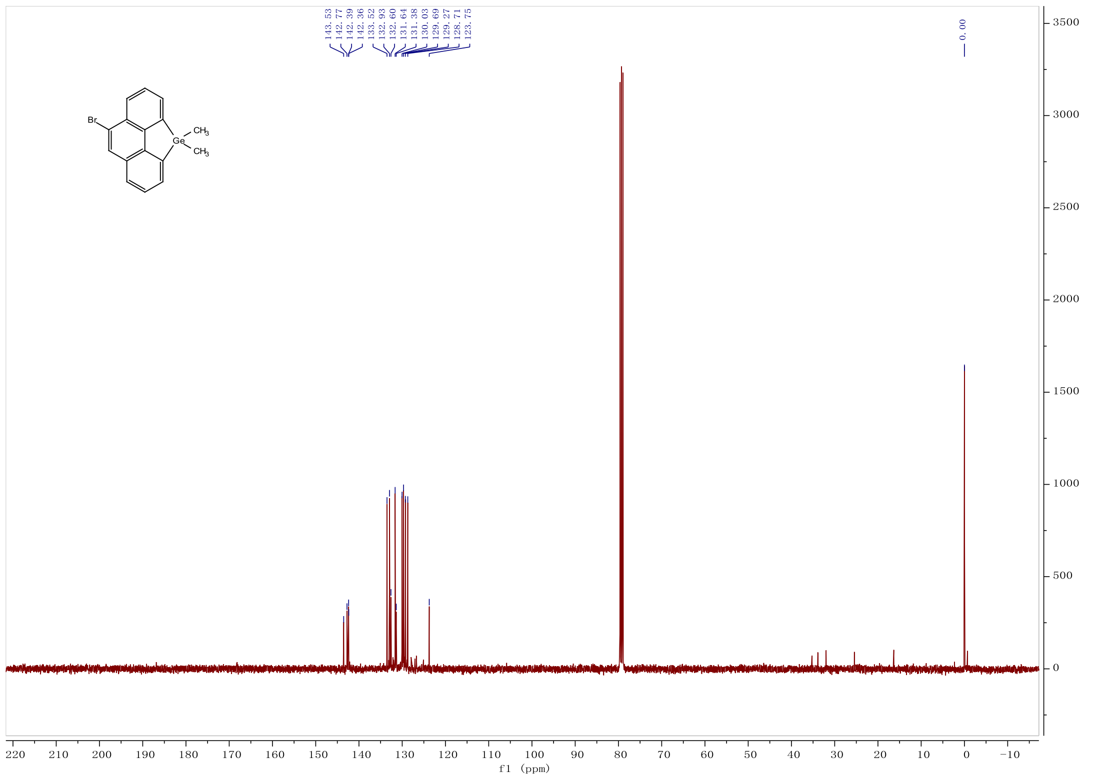
2.05
1.00
2.11
4.20
1.16
1.09
2.08
1.09
f1 (ppm)


140.81
139.24
132.38
132.07
130.16
129.43
128.93
128.63
127.47
127.39
127.38
127.12
126.72
126.61
125.85
123.28
122.72
4000
3800
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
−400
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

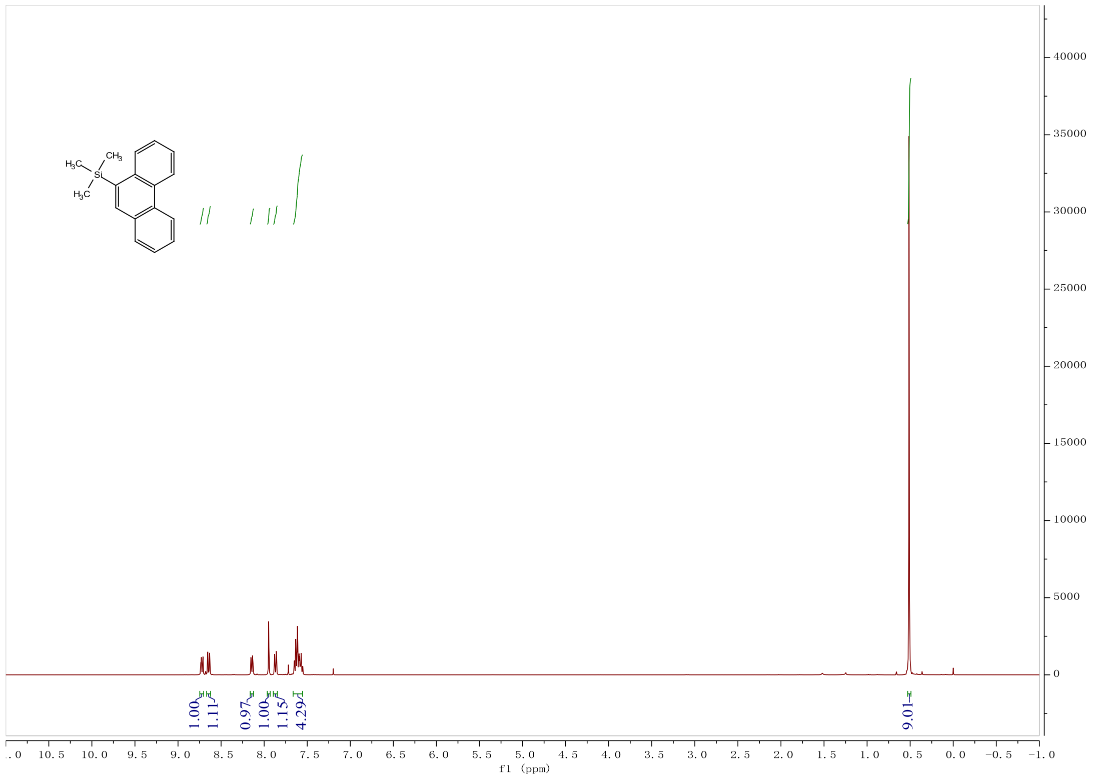

Ge
CH3
CH3
0.99
1.01
2.04
4.28
1.13
2.17
1.09
5.78
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)


144.07
143.15
142.56
142.49
142.11
141.95
132.50
132.22
132.18
131.90
130.95
129.82
129.41
129.31
129.04
128.79
128.50
127.93
-0.00
Ge
CH3
CH3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)
3800 3600 3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200

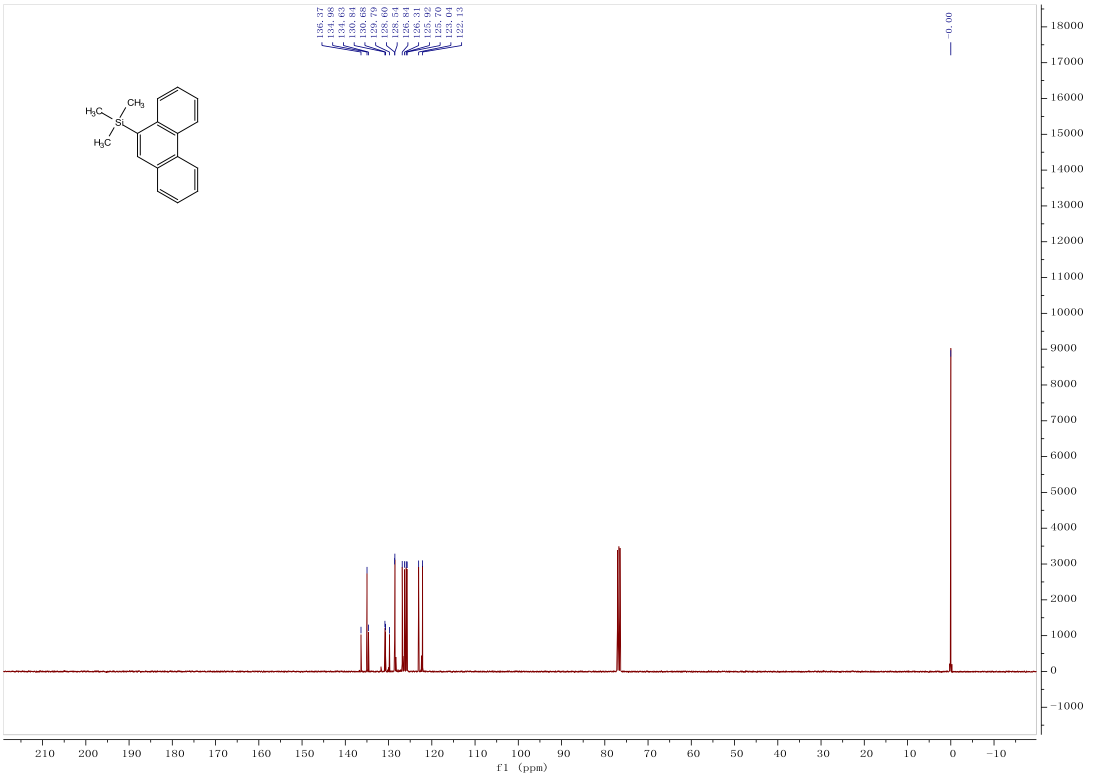

Br
Ge
CH3
CH3
1.00
1.03
2.46
1.09
1.35
1.24
6.24
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
25000 24000 23000 22000 21000 20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000

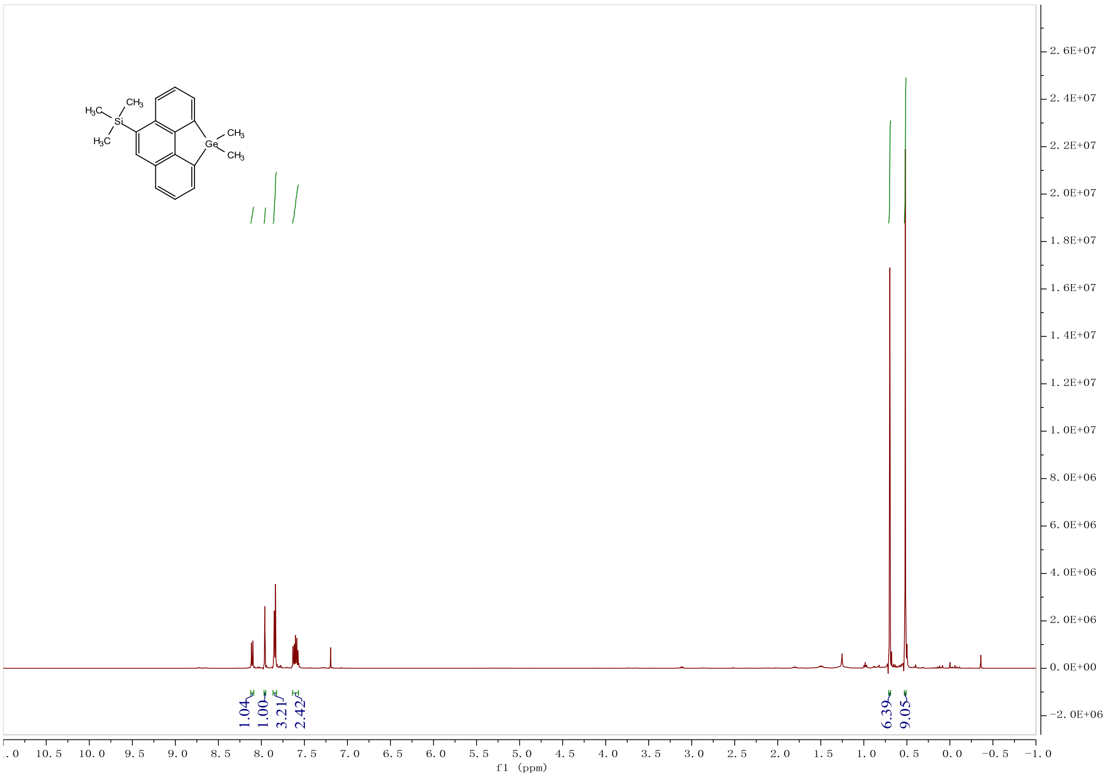


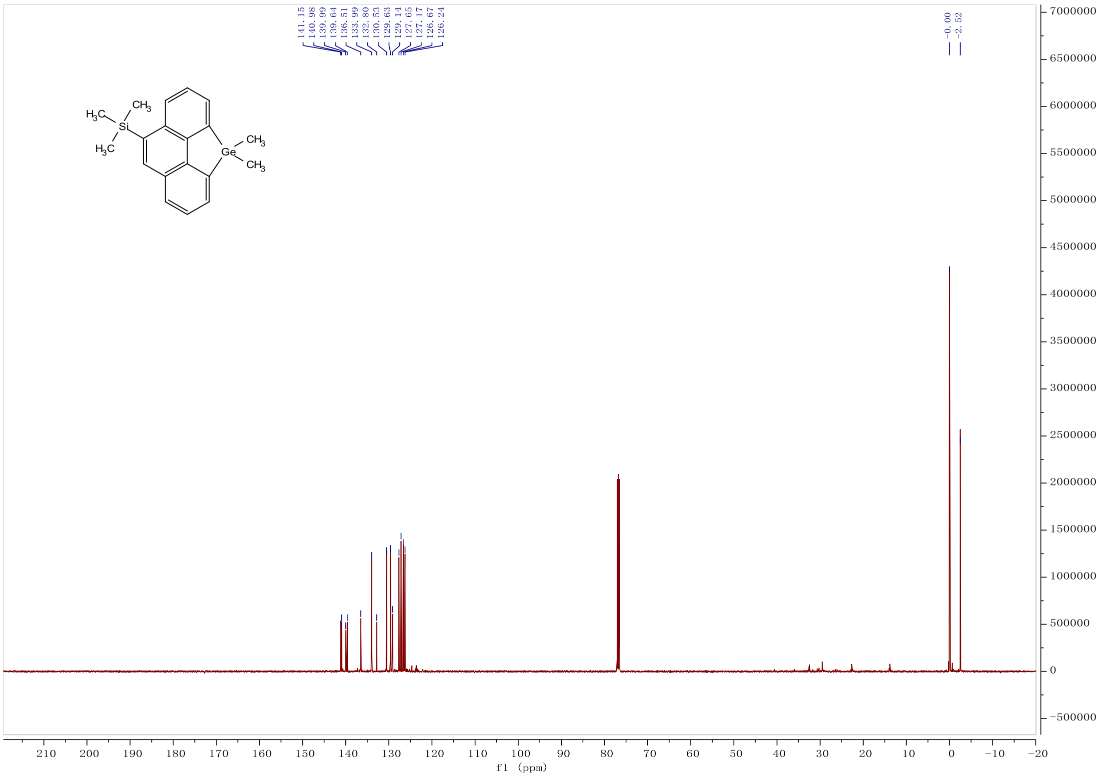
143.53
142.77
142.39
142.36
133.52
132.93
132.60
131.64
131.38
130.03
129.69
129.27
128.71
123.75
0.00
Br
Ge
CH3
CH3
3500
3000
2500
2000
1500
1000
500
0
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)



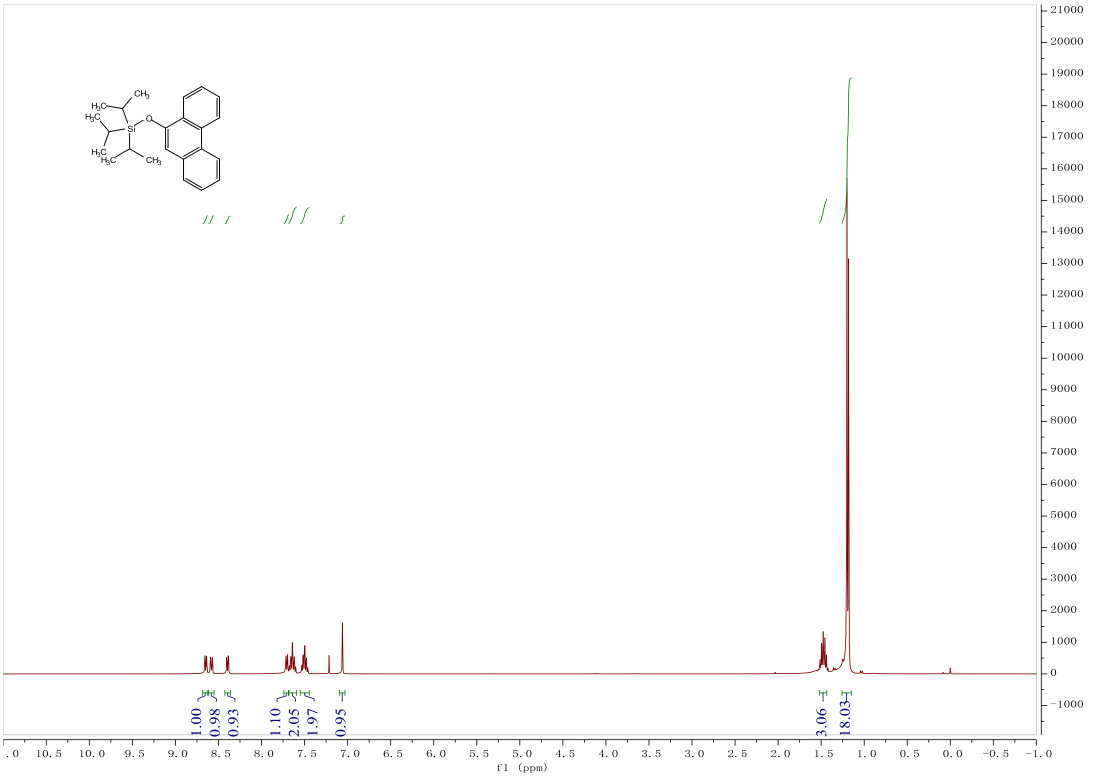
H3C
CH3
Si
H3C
1.00
1.11
0.97
1.00
1.15
4.29
9.01
40000
35000
30000
25000
20000
15000
10000
5000
0
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)


136.37
134.98
134.63
130.84
130.68
129.79
128.60
128.54
126.84
126.31
125.92
125.70
123.04
122.13
-0.00
$H_3C$
$CH_3$
Si
$H_3C$
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

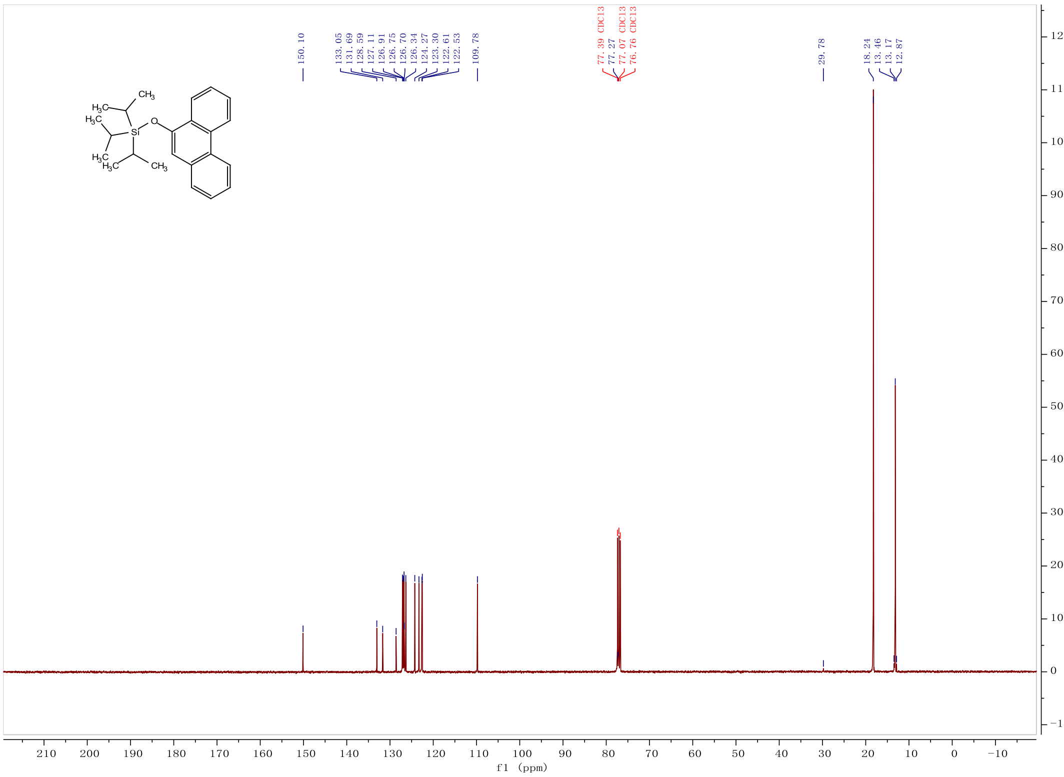

$H_3C$
$CH_3$
Si
$H_3C$
Ge
$CH_3$
$CH_3$
2.6E+07
2.4E+07
2.2E+07
2.0E+07
1.8E+07
1.6E+07
1.4E+07
1.2E+07
1.0E+07
8.0E+06
6.0E+06
4.0E+06
2.0E+06
0.0E+00
-2.0E+06
1.04
1.00
3.21
2.42
6.39
9.05
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

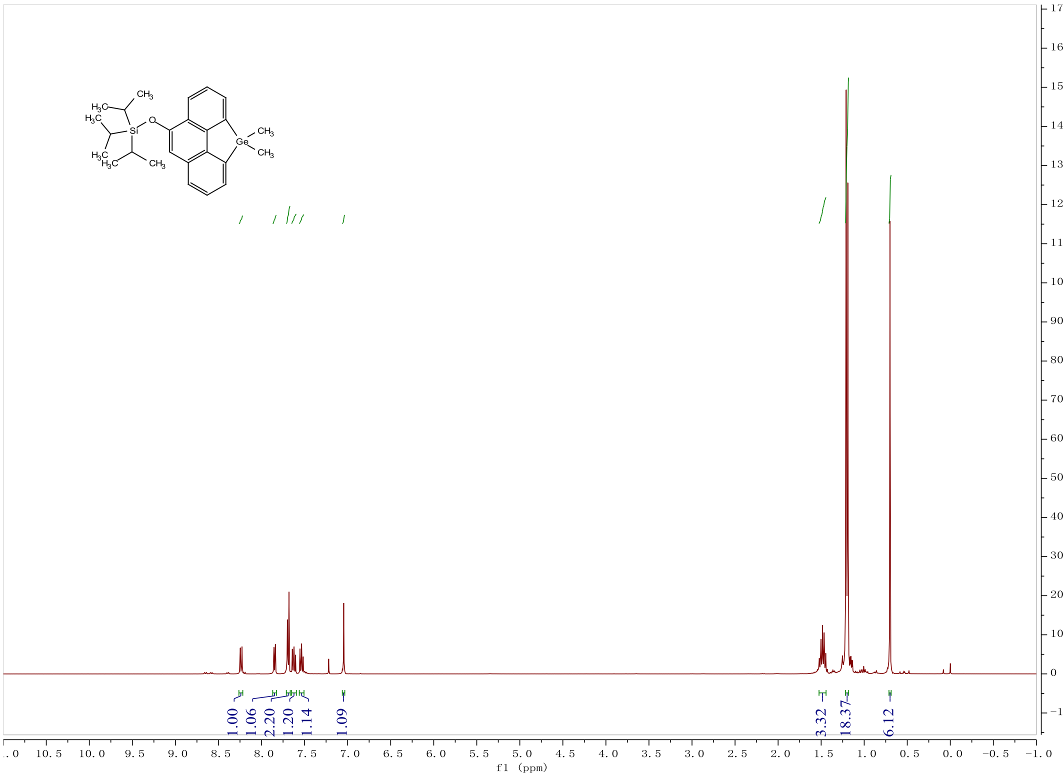


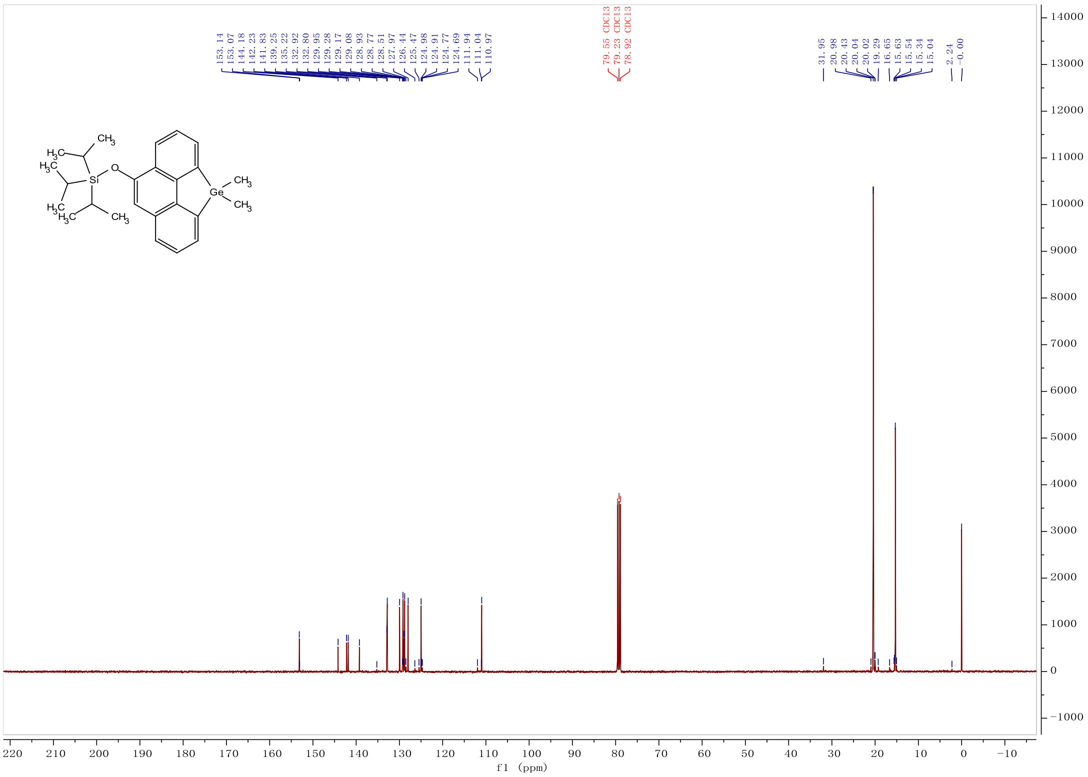
141.15
140.98
139.99
139.64
136.51
133.99
132.80
130.53
129.63
129.14
127.65
127.17
126.67
126.24
-0.00
-2.52
H3C
CH3
Si
H3C
Ge
CH3
CH3
7000000
6500000
6000000
5500000
5000000
4500000
4000000
3500000
3000000
2500000
2000000
1500000
1000000
500000
0
-500000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

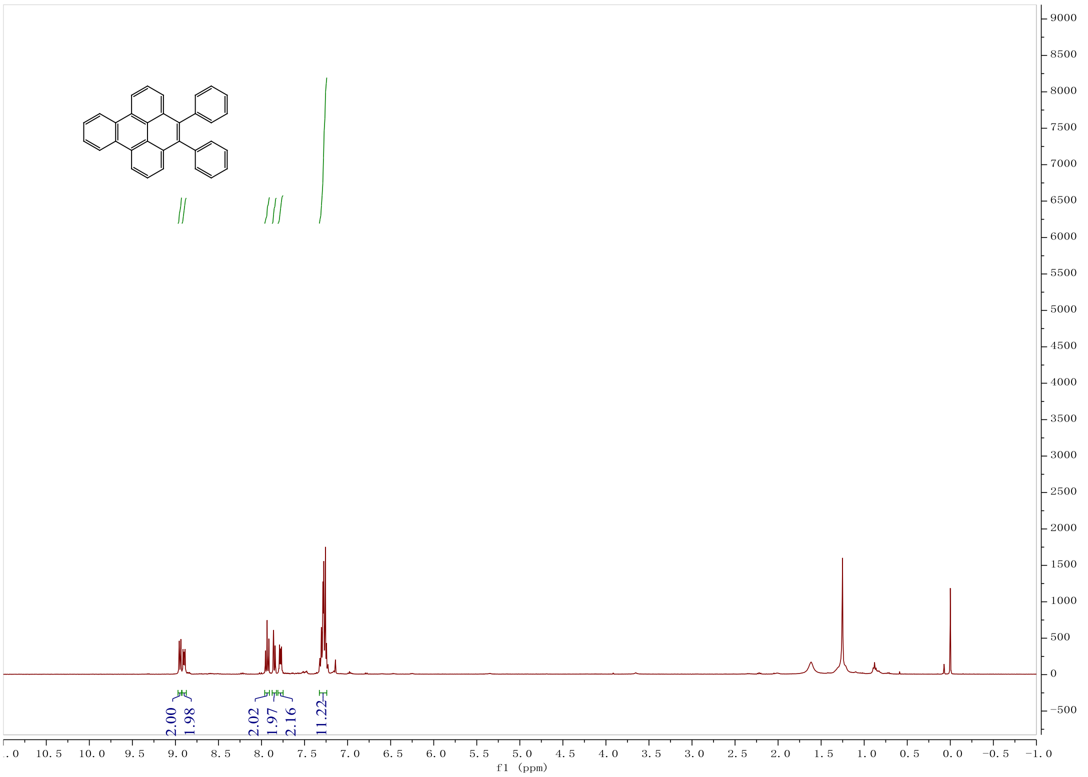


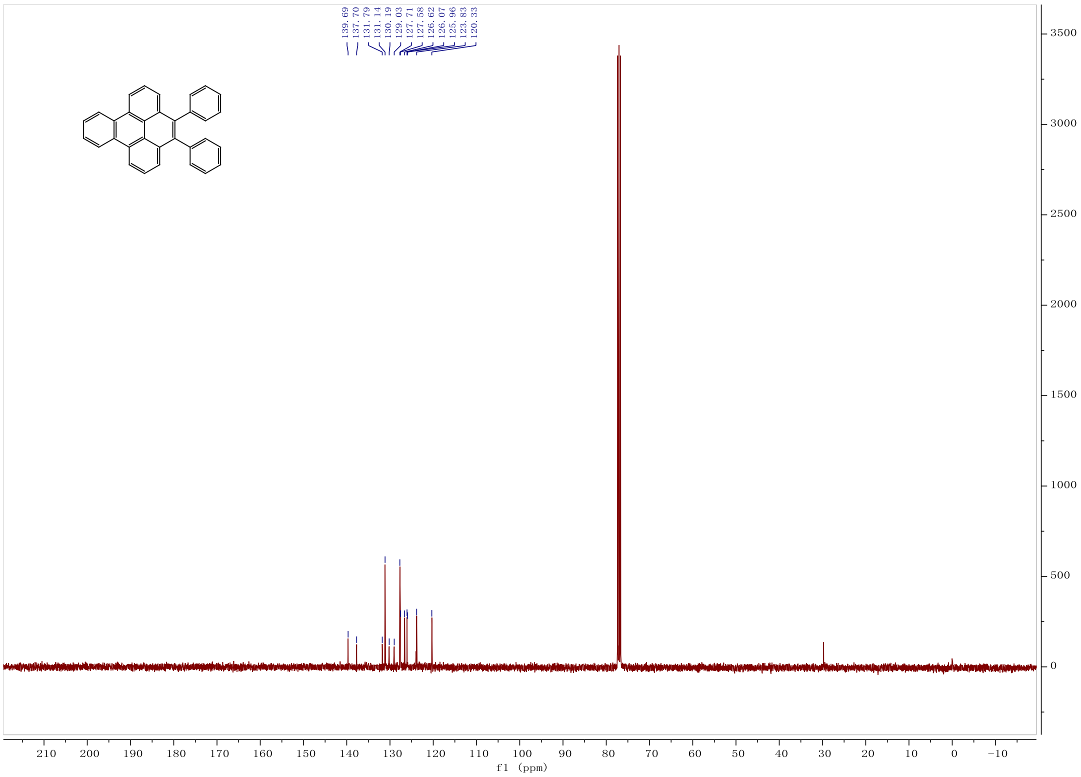
CH3
H3C
H3C
Si
O
H3C
H3C
CH3
1.00
0.98
0.93
1.10
2.05
1.97
0.95
3.06
18.03
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

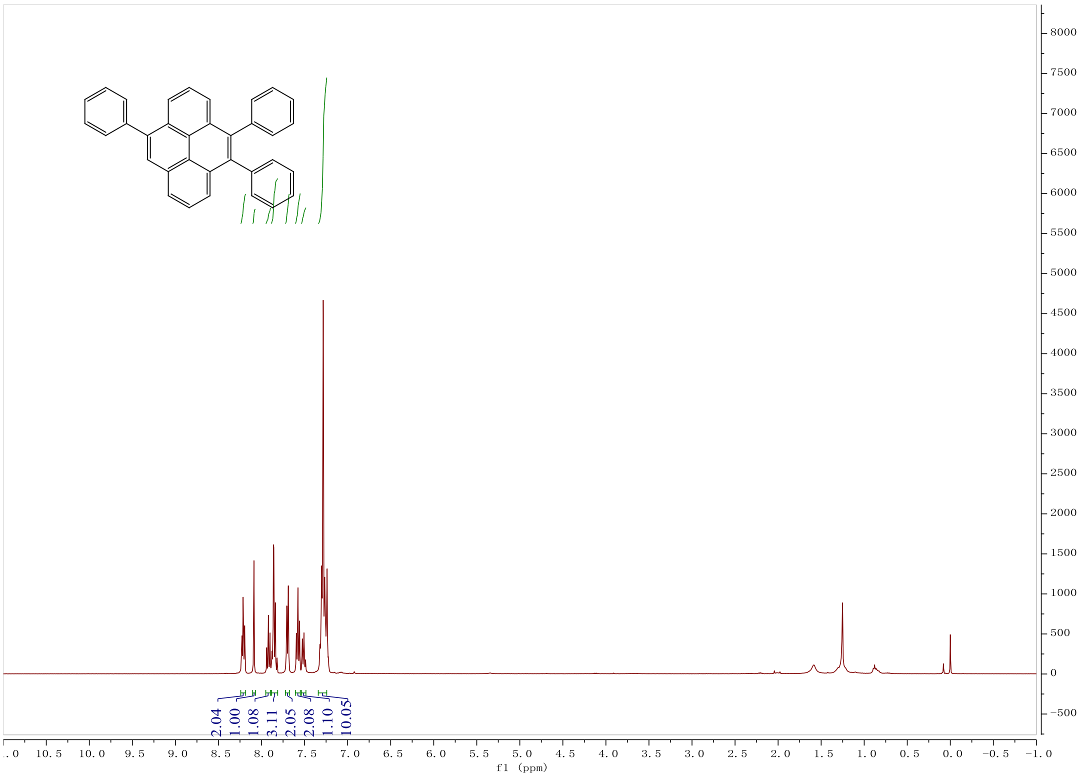

150.10
133.05
131.69
128.59
127.11
126.91
126.75
126.70
126.34
124.27
123.30
122.61
122.53
109.78
77.39 CDCl3
77.27 CDCl3
77.07 CDCl3
76.76 CDCl3
29.78
18.24
13.46
13.17
12.87
H3C
CH3
H3C
Si
O
H3C
H3C
CH3
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

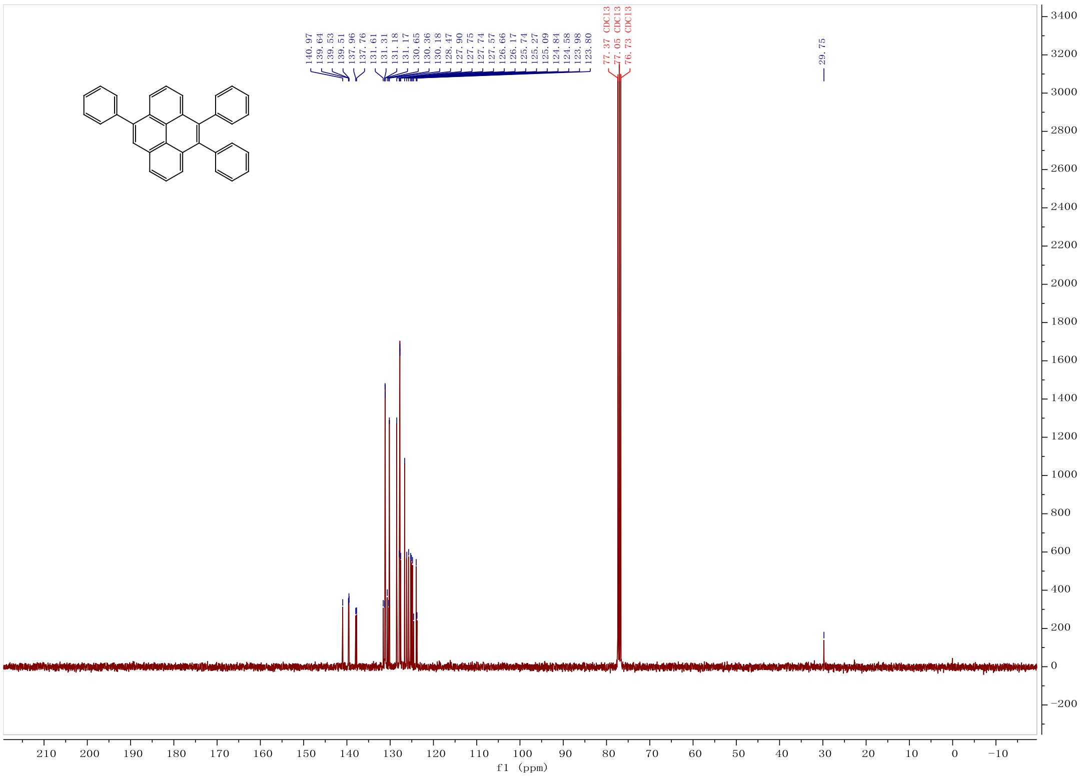

H3C
CH3
H3C
Si
O
H3C
H3C
CH3
Ge
CH3
CH3
1.00
1.06
2.20
1.20
1.14
1.09
3.32
18.37
6.12
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)

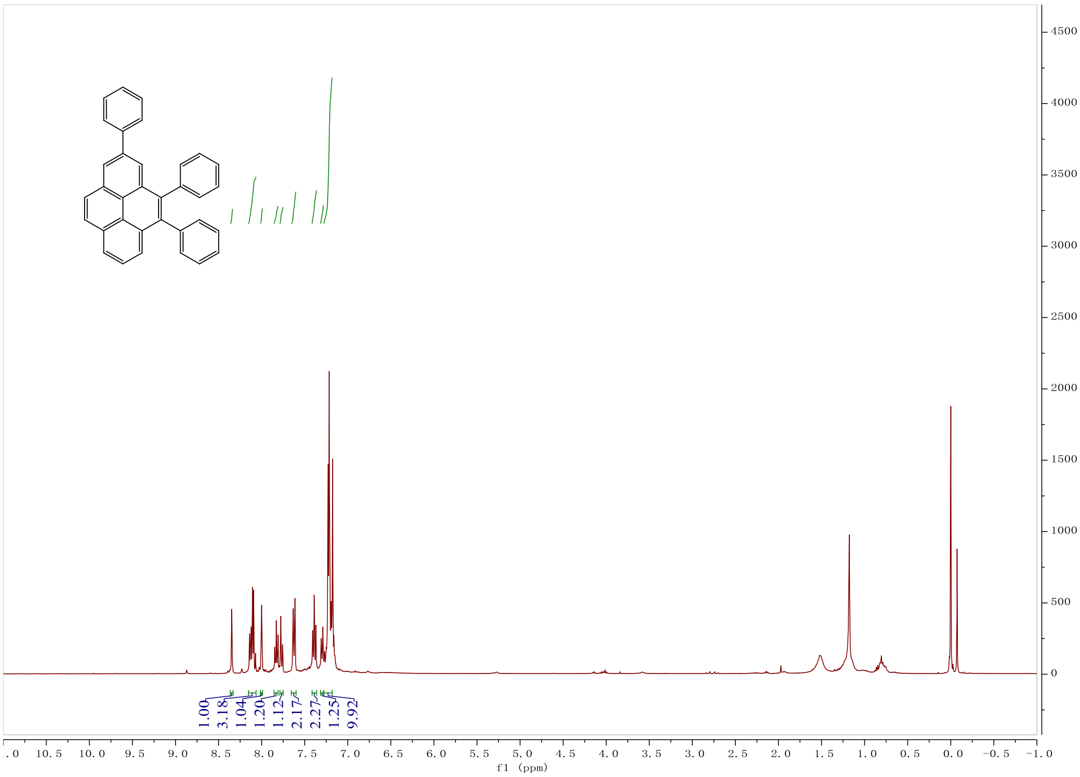

153.14
153.07
144.18
142.23
141.83
139.25
135.22
132.92
132.80
129.95
129.28
129.17
129.08
128.93
128.77
128.51
127.97
126.44
125.47
124.98
124.91
124.77
124.69
111.94
111.04
110.97
79.55 CDCl3
79.23 CDCl3
78.92 CDCl3
31.95
20.98
20.43
20.04
20.02
19.29
16.65
15.63
15.54
15.34
15.04
2.24
-0.00
H3C
CH3
H3C
Si
O
H3C
H3C
CH3
Ge
CH3
CH3
f1 (ppm)

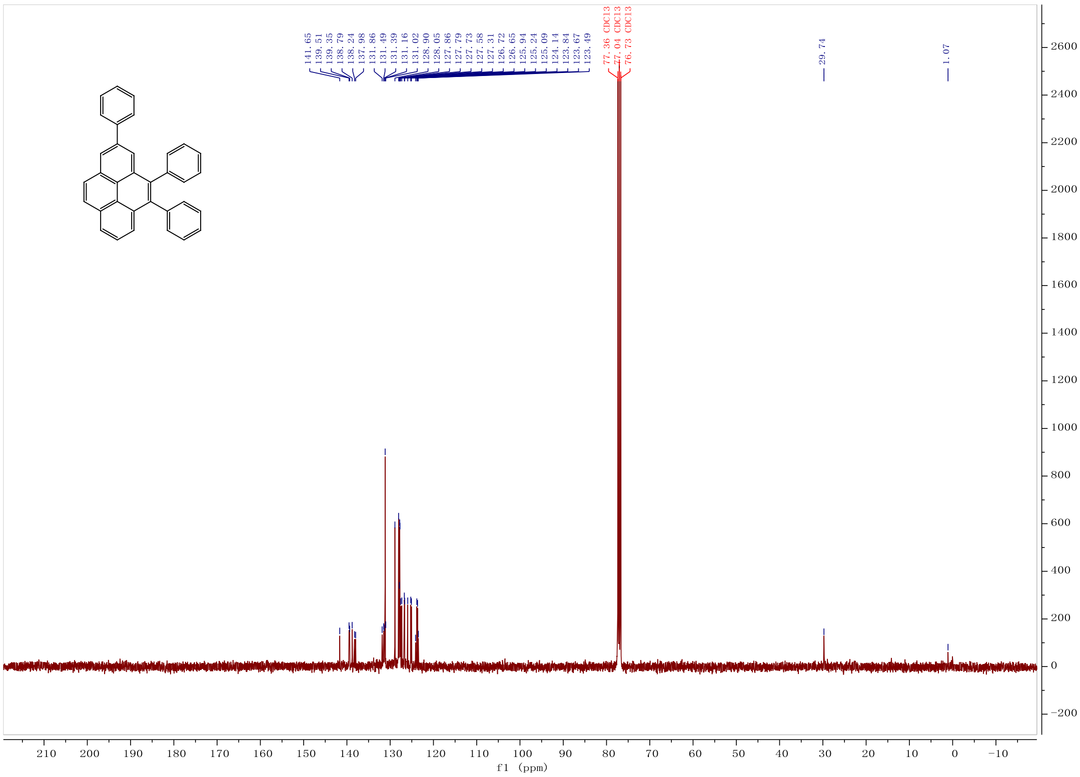

2.00
1.98
2.02
1.97
2.16
11.22
f1 (ppm)

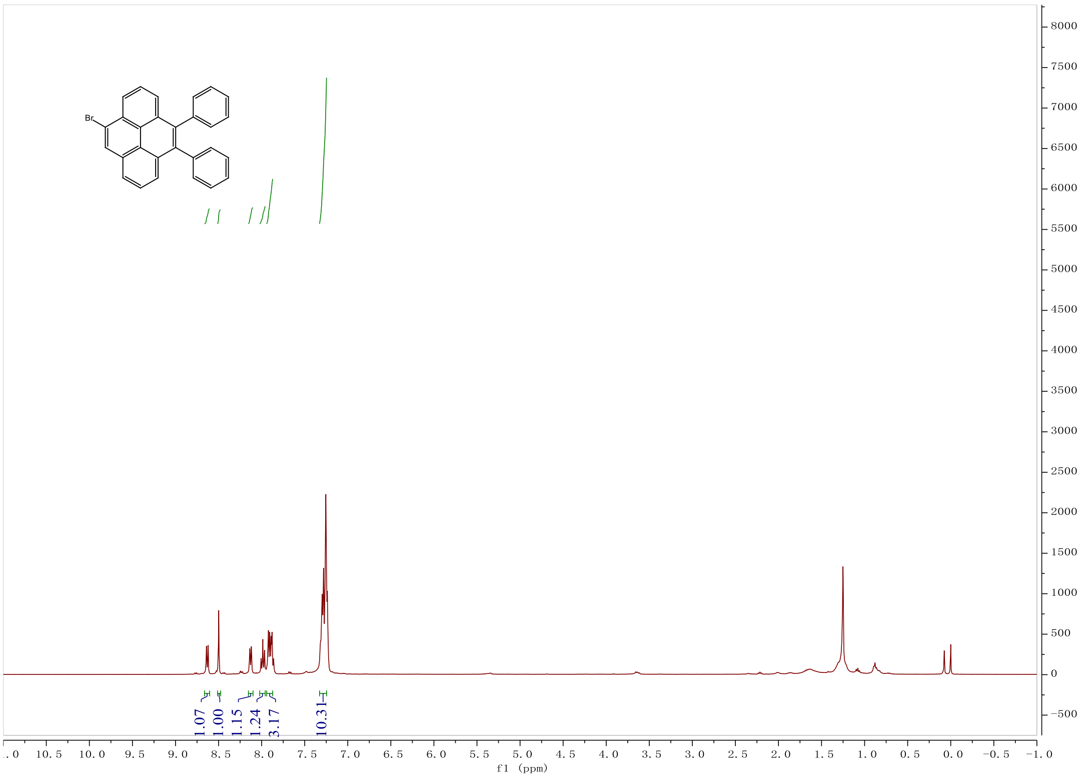

139.69
137.70
131.79
131.14
130.19
129.03
127.71
127.58
126.62
126.07
125.96
123.83
120.33
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

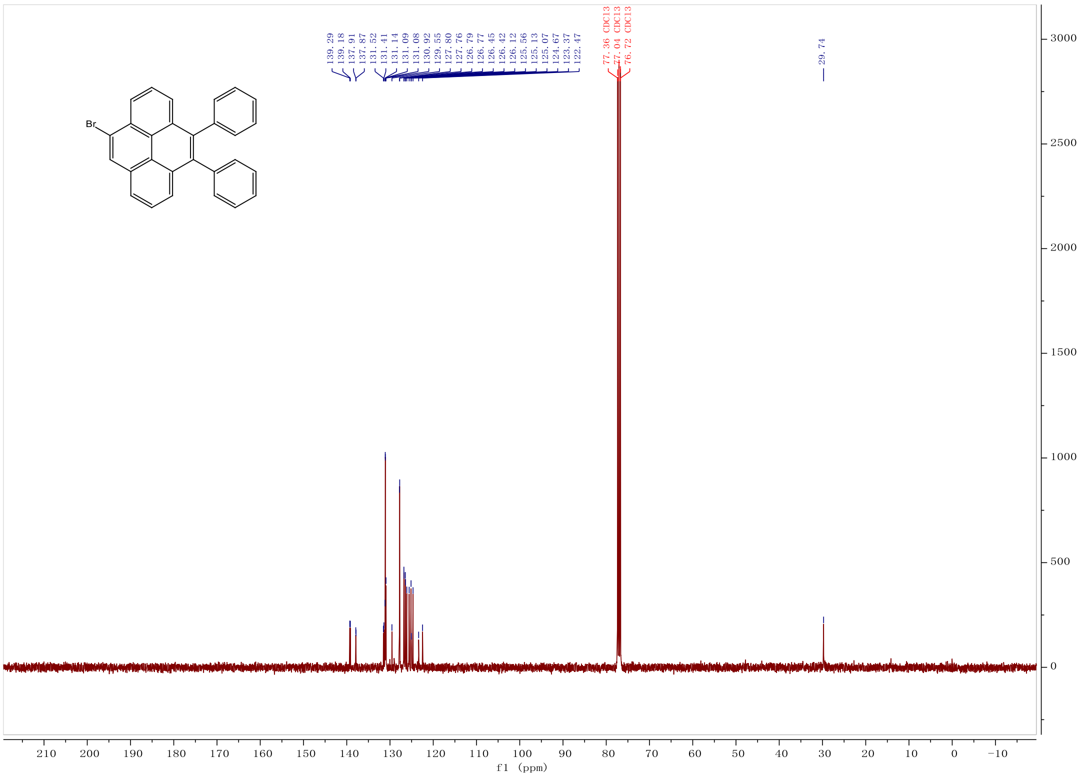

8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
2.04
1.00
1.08
3.11
2.05
2.08
1.10
10.05
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)

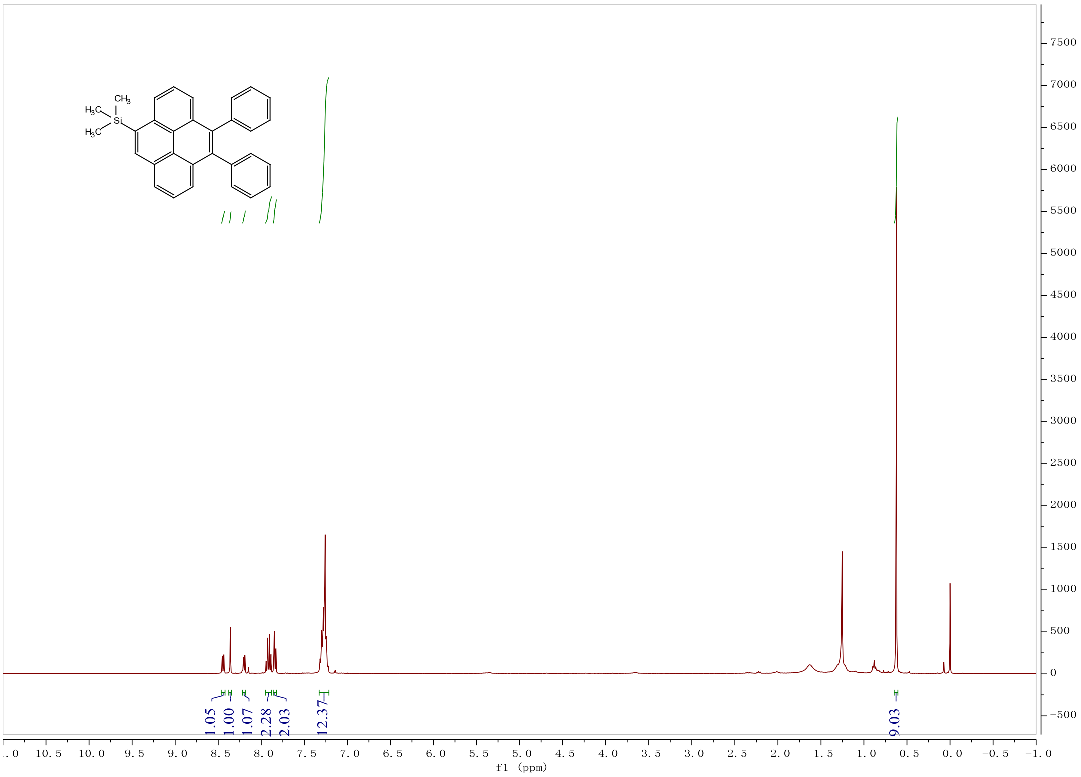

140.97
139.64
139.53
139.51
137.96
137.76
131.61
131.31
131.18
131.17
130.65
130.36
130.18
128.47
127.90
127.75
127.74
127.57
126.66
126.17
125.74
125.27
125.09
124.84
124.58
123.98
123.80
77.37 CDCl3
77.05 CDCl3
76.73 CDCl3
29.75
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

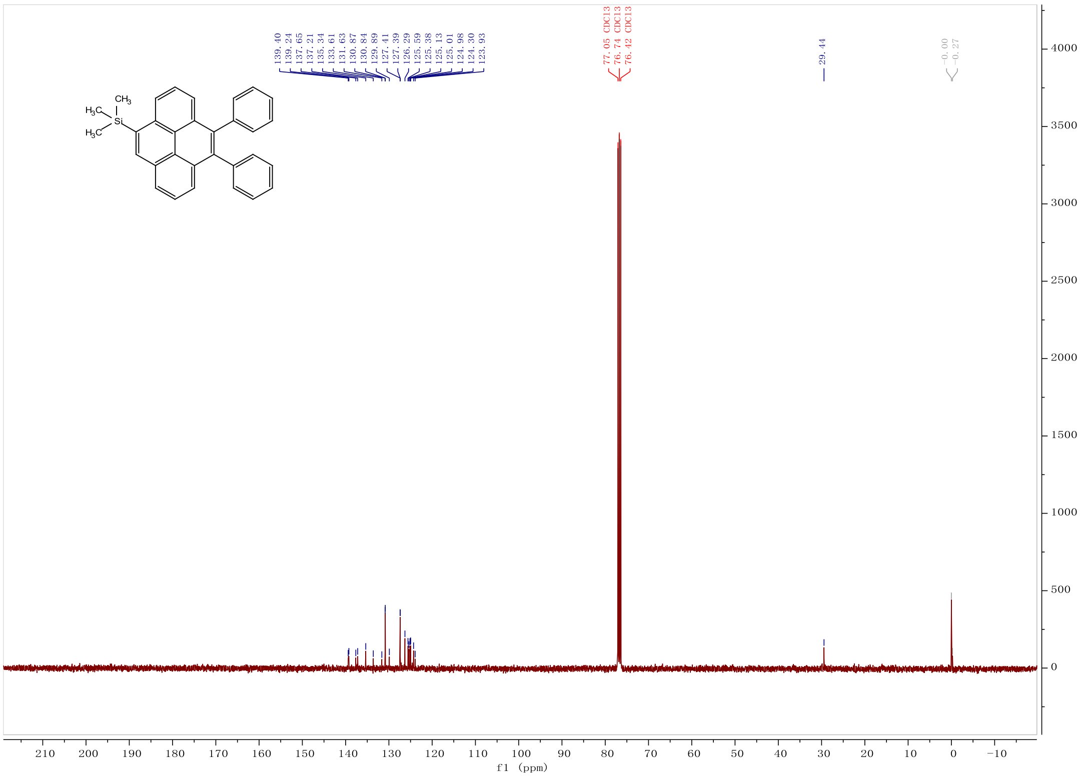

1.00
3.18
1.04
1.20
1.12
2.17
2.27
1.25
9.92
4500
4000
3500
3000
2500
2000
1500
1000
500
0
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)


141.65
139.51
139.35
138.79
138.24
137.98
131.86
131.49
131.39
131.16
131.02
128.90
128.05
127.86
127.79
127.73
127.58
127.31
126.72
126.65
125.94
125.24
125.09
124.14
123.84
123.67
123.49
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
29.74
1.07
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

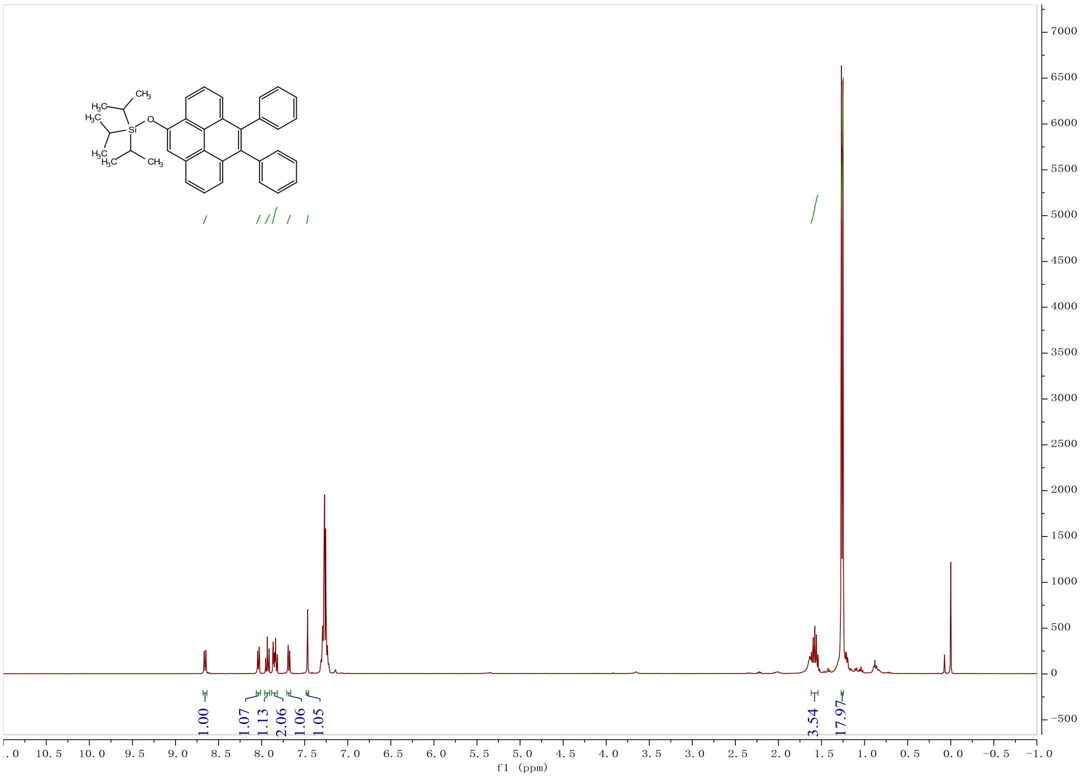

Br
1.07
1.00
1.15
1.24
3.17
10.31
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


139.29
139.18
137.91
137.87
131.52
131.41
131.14
131.09
131.08
130.92
129.55
127.80
127.76
126.79
126.77
126.45
126.42
126.12
125.56
125.13
125.07
124.67
123.37
122.47
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
29.74
Br
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

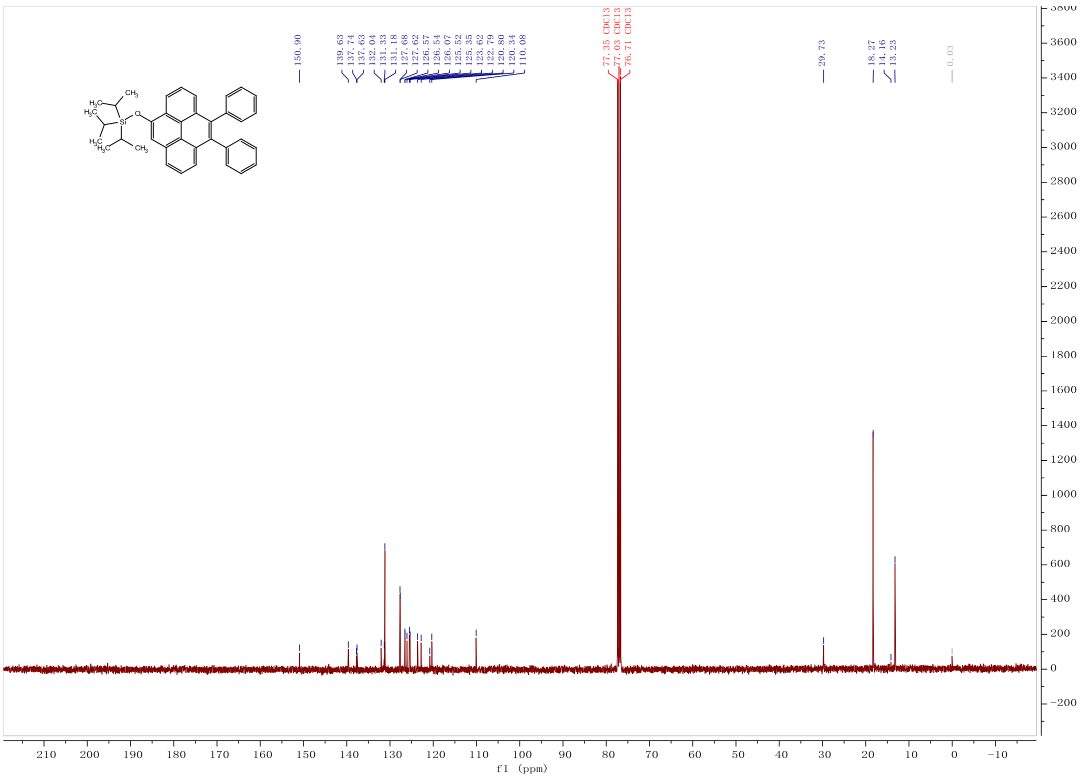

CH3
H3C
Si
H3C
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
−500
1.05
1.00
1.07
2.28
2.03
12.37
9.03
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)



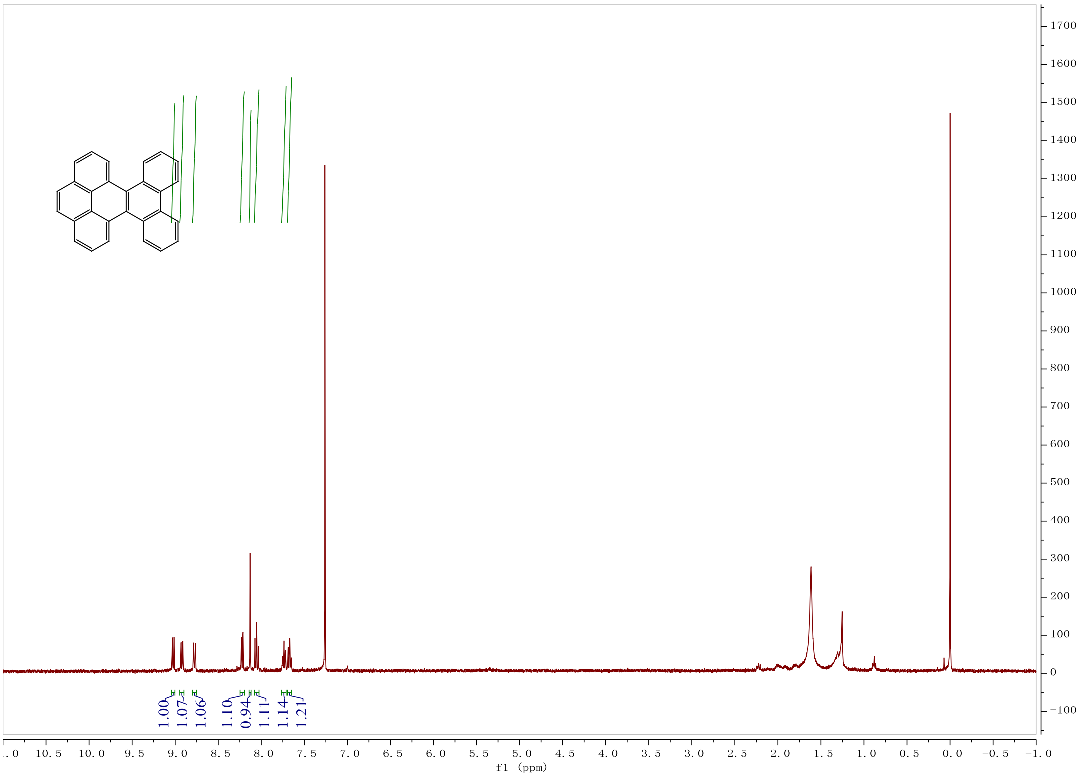
CH3
H3C
Si
H3C
139.40
139.24
137.65
137.21
135.34
133.61
131.63
130.87
130.84
129.89
127.41
127.39
126.29
125.59
125.38
125.13
125.01
124.98
124.30
123.93
77.05 CDCl3
76.74 CDCl3
76.42 CDCl3
29.44
-0.00
-0.27
4000
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



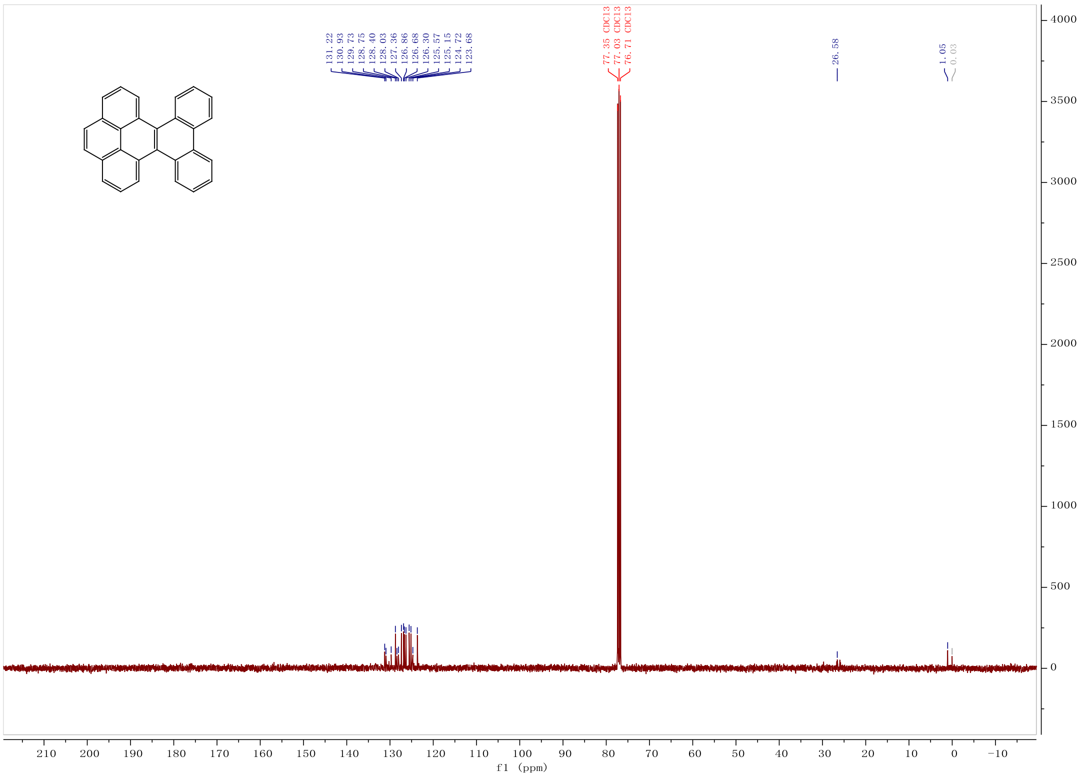
CH3
H3C
H3C
Si
O
H3C
H3C
CH3
1.00
1.07
1.13
2.06
1.06
1.05
3.54
17.97
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

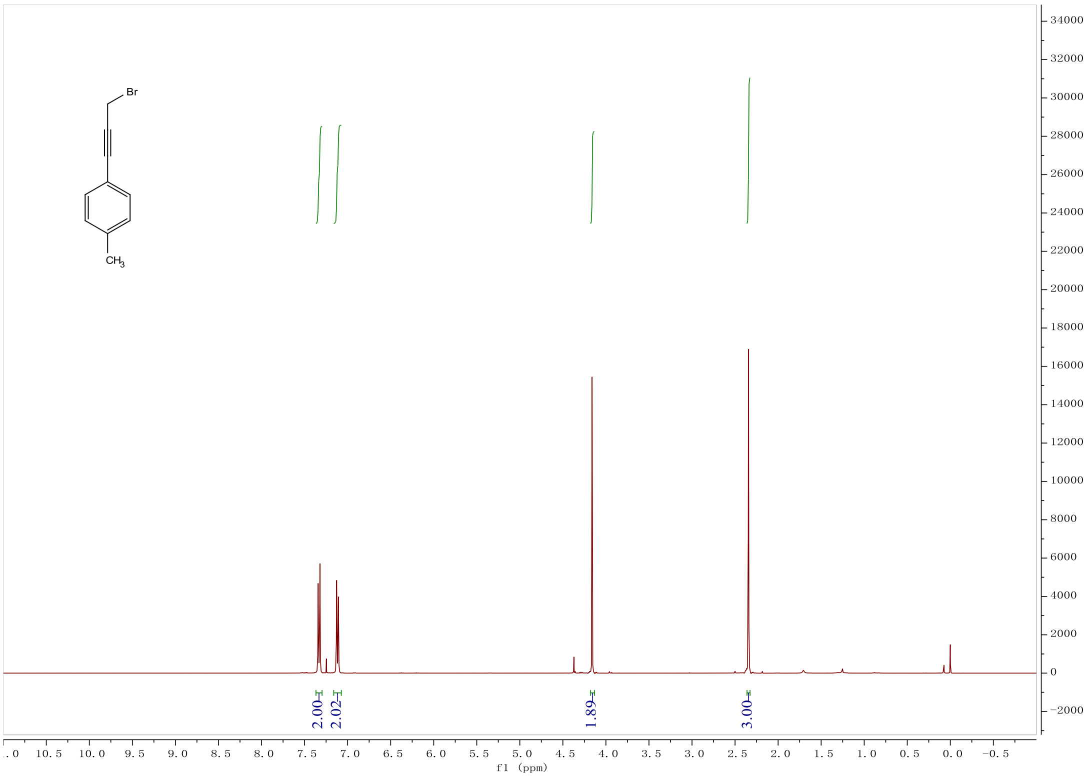


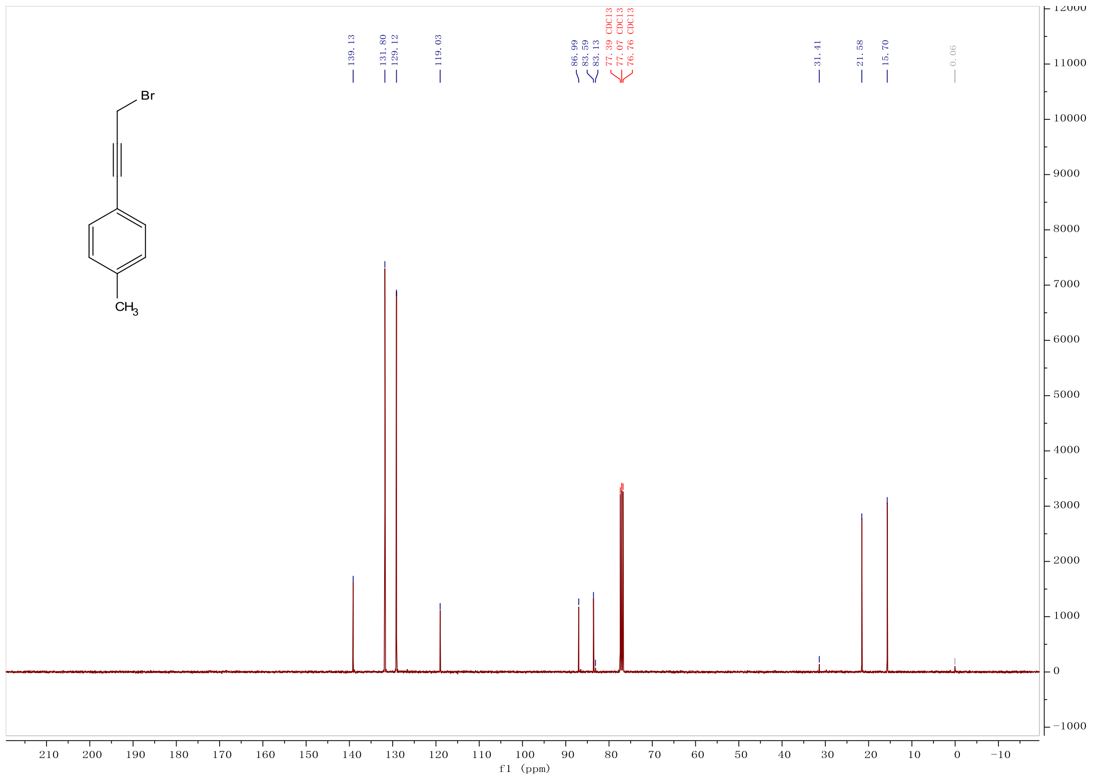
150.90
139.63
137.74
137.63
132.04
131.33
131.18
127.68
127.62
126.57
126.54
126.07
125.52
125.35
123.62
122.79
120.80
120.34
110.08
77.35 CDCl3
77.03 CDCl3
76.71 CDCl3
29.73
18.27
14.16
13.23
0.03
H3C
CH3
H3C
Si
O
H3C
H3C
CH3
f1 (ppm)

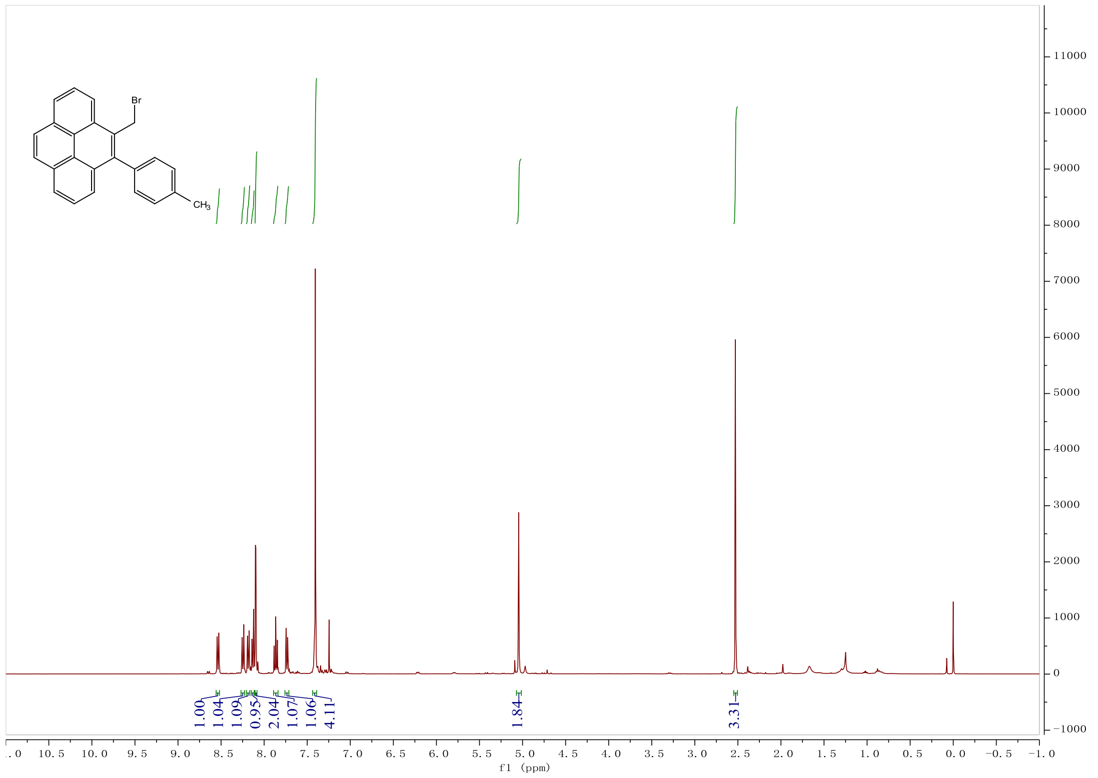


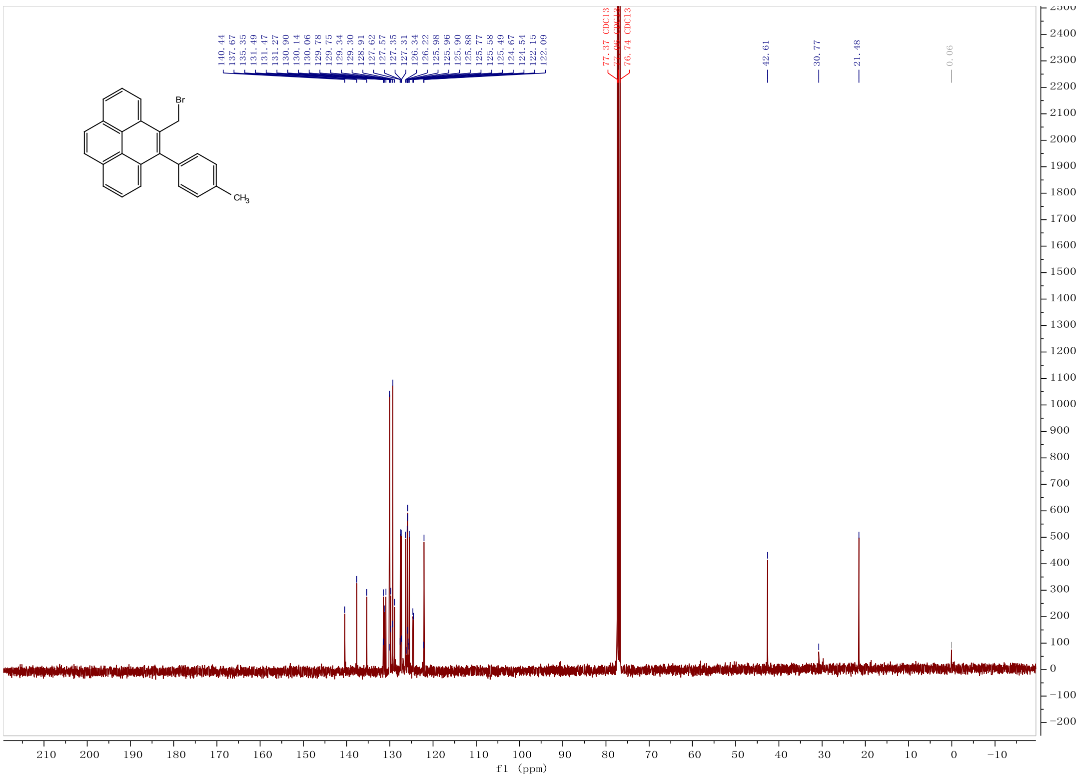
1.00
1.07
1.06
1.10
0.94
1.11
1.14
1.21
f1 (ppm)

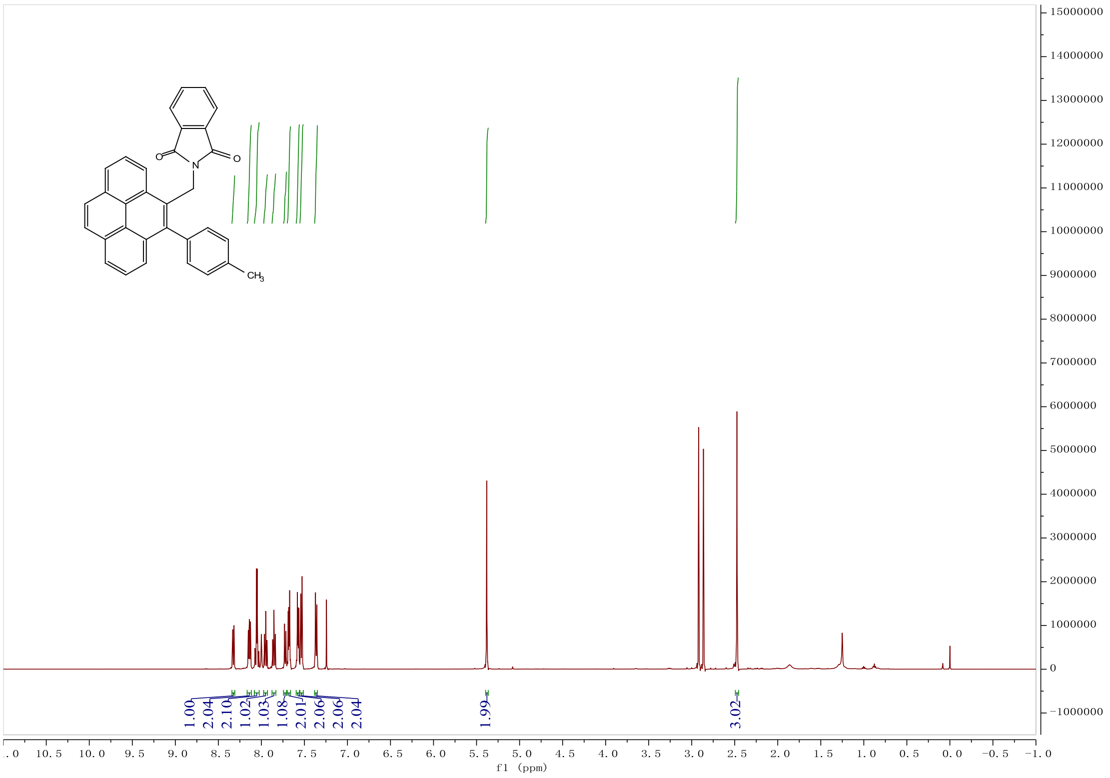

131.22
130.93
129.73
128.75
128.40
128.03
127.36
126.86
126.68
126.30
125.57
125.15
124.72
123.68
77.35 CDCl3
77.03 CDCl3
76.71 CDCl3
26.58
1.05
0.03
4000
3500
3000
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

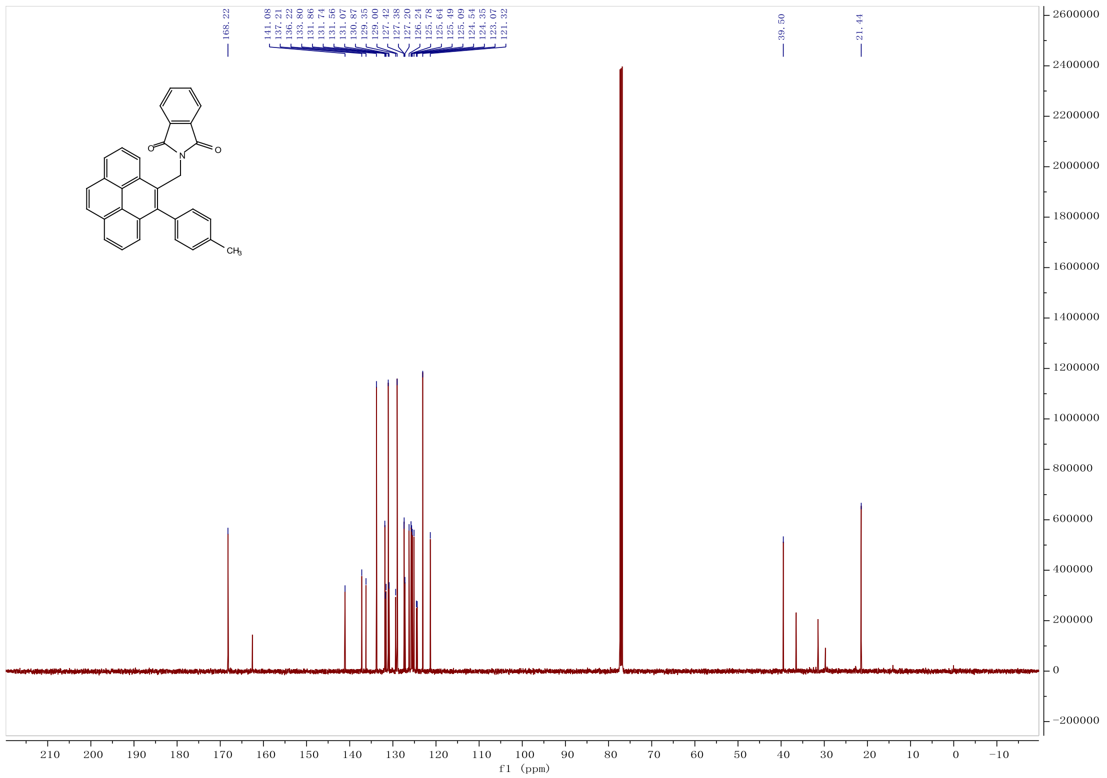

Br
CH3
2.00
2.02
1.89
3.00
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
−2000
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
f1 (ppm)

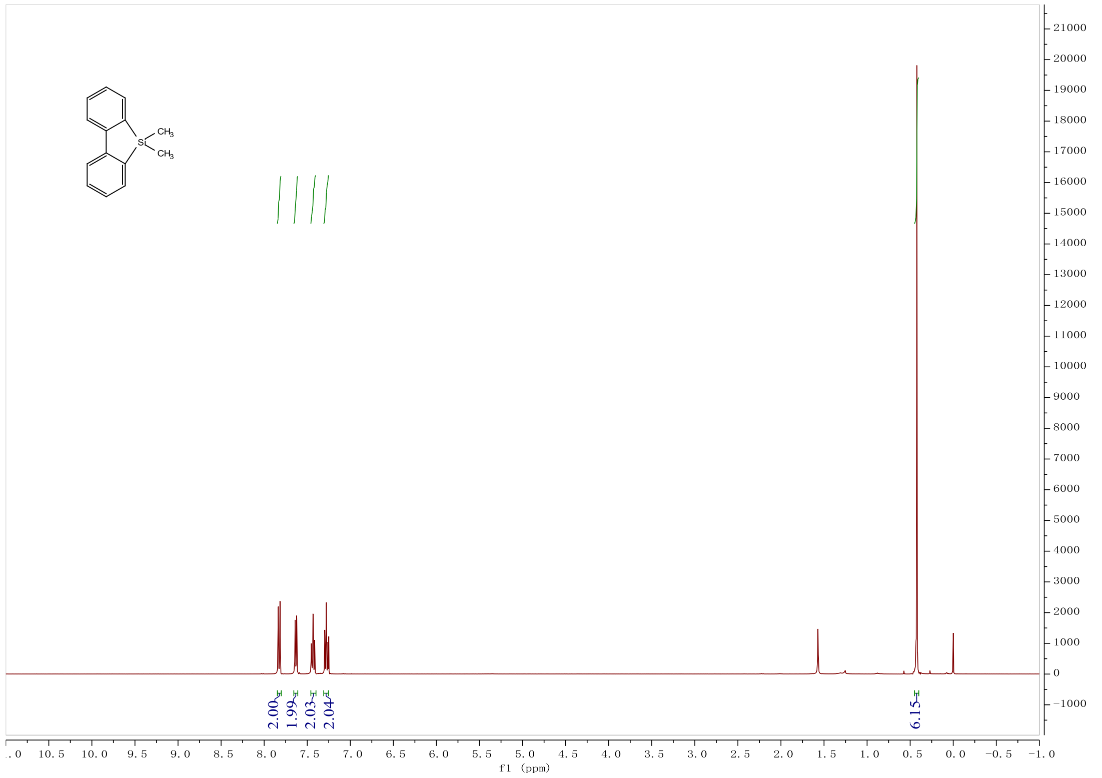

Br
CH3
139.13
131.80
129.12
119.03
86.99
83.59
83.13
77.39 CDCl3
77.07 CDCl3
76.76 CDCl3
31.41
21.58
15.70
0.06
f1 (ppm)

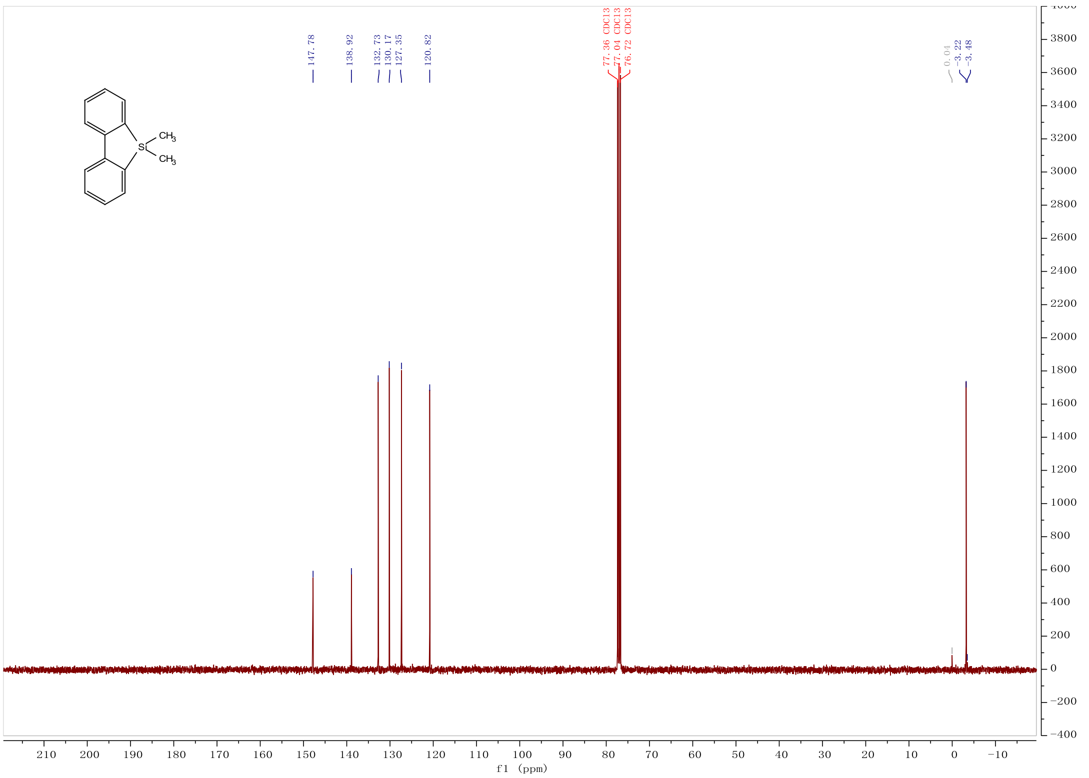

Br
CH3
1.00
1.04
1.09
0.95
2.04
1.07
1.06
4.11
1.84
3.31
f1 (ppm)

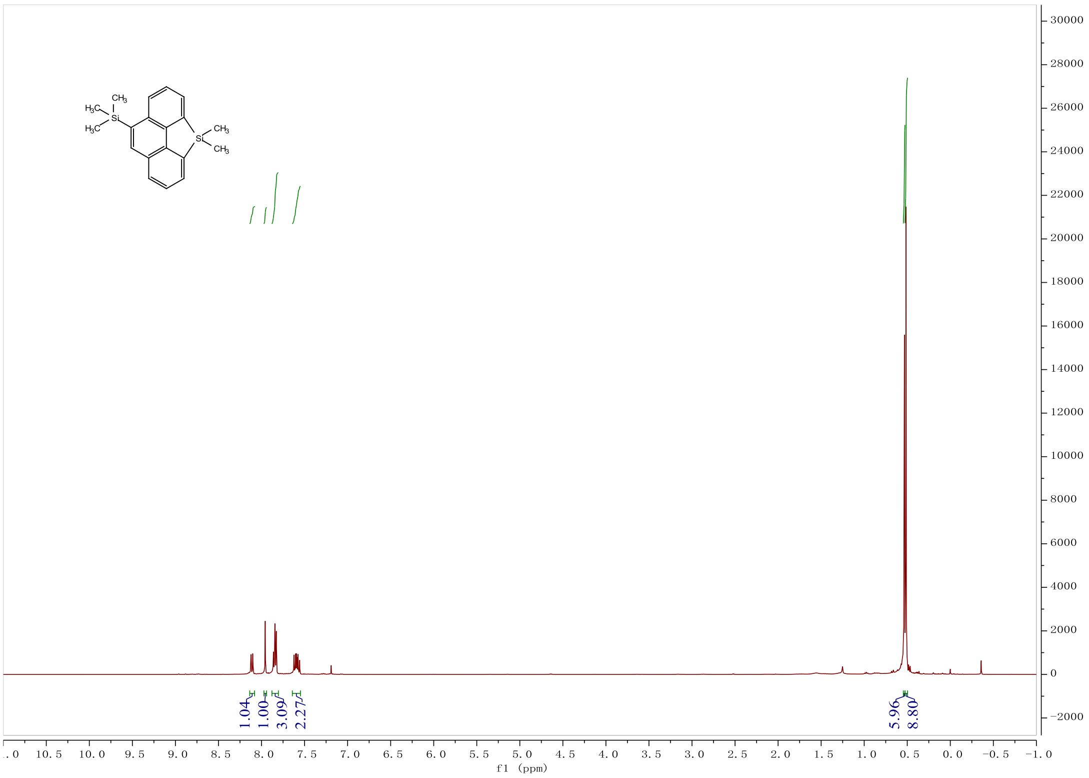

Br
CH3
140.44
137.67
135.35
131.49
131.47
131.27
130.90
130.14
130.06
129.78
129.75
129.34
129.30
128.91
127.62
127.57
127.35
127.31
126.34
126.22
125.98
125.96
125.90
125.88
125.77
125.58
125.49
124.67
124.54
122.15
122.09
77.37 CDCl3
77.05 CDCl3
76.74 CDCl3
42.61
30.77
21.48
0.06
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)
2500 2400 2300 2200 2100 2000 1900 1800 1700 1600 1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 −100 −200

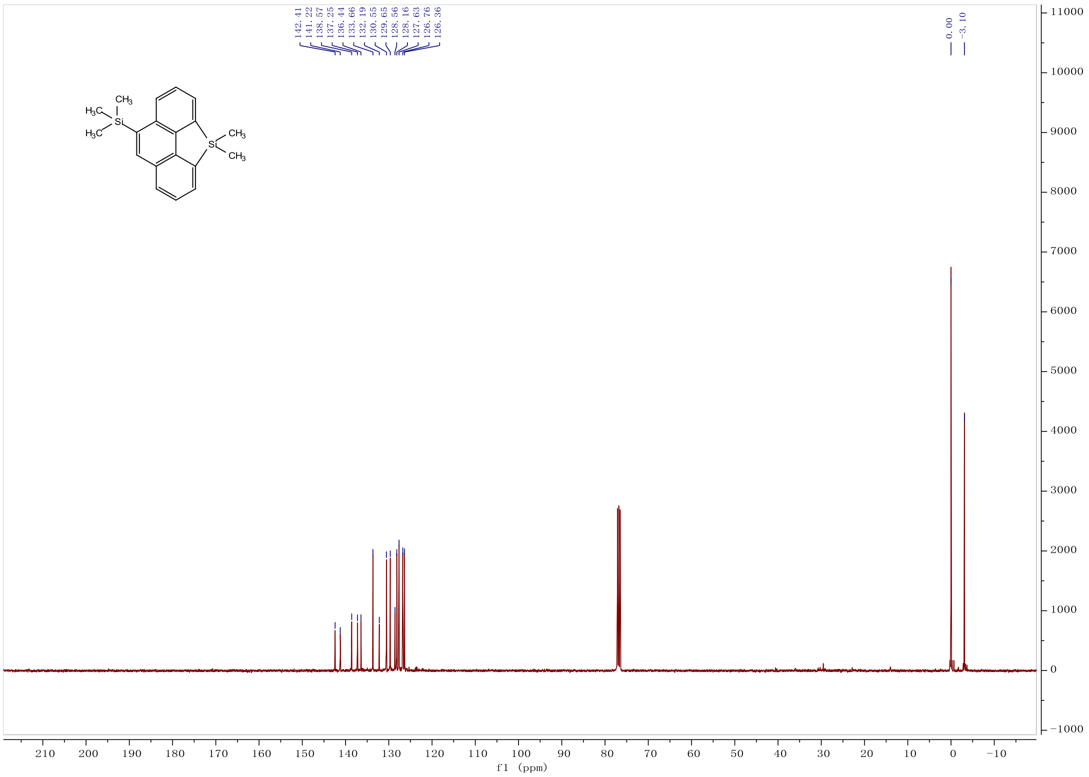

O
N
O
CH3
1.00
2.04
2.10
1.02
1.03
1.08
2.01
2.06
2.06
2.04
1.99
3.02
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1.0
f1 (ppm)
15000000 14000000 13000000 12000000 11000000 10000000 9000000 8000000 7000000 6000000 5000000 4000000 3000000 2000000 1000000 0 −1000000


168.22
141.08
137.21
136.22
133.80
131.86
131.74
131.56
131.07
130.87
129.35
129.00
127.42
127.38
127.20
126.24
125.78
125.64
125.49
125.09
124.54
124.35
123.07
121.32
39.50
21.44
O
N
O
CH3
2600000
2400000
2200000
2000000
1800000
1600000
1400000
1200000
1000000
800000
600000
400000
200000
0
−200000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


Si
CH3
CH3
2.00
1.99
2.03
2.04
6.15
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
−0.5
−1.0
f1 (ppm)


147.78
138.92
132.73
130.17
127.35
120.82
77.36 CDCl3
77.04 CDCl3
76.72 CDCl3
0.04
-3.22
-3.48
Si
CH3
CH3
f1 (ppm)


H3C
CH3
H3C
Si
Si
CH3
CH3
1.04
1.00
3.09
2.27
5.96
8.80
f1 (ppm)


142.41
141.22
138.57
137.25
136.44
133.66
132.19
130.55
129.65
128.56
128.16
127.63
126.76
126.36
0.00
-3.10
CH3
H3C
Si
H3C
Si
CH3
CH3
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)