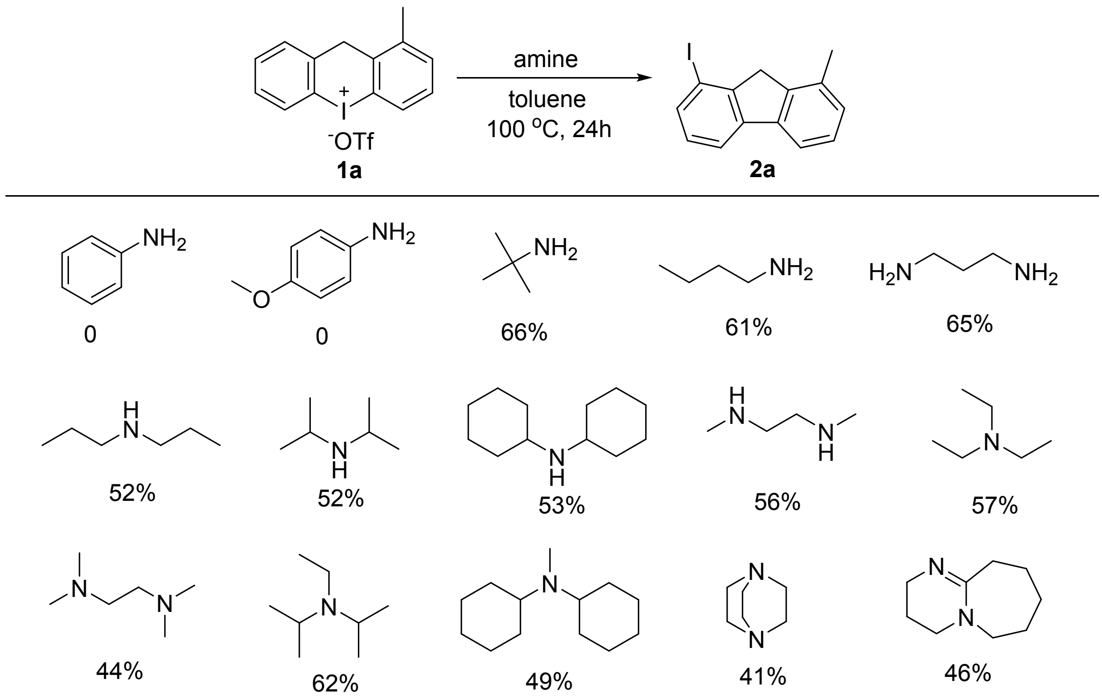
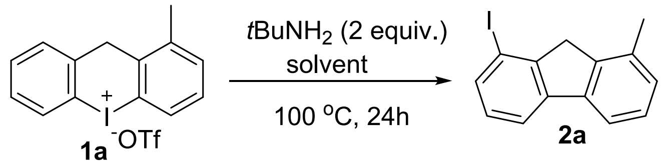
Table S1: Screening of amines [a]



[a]Reaction conditions: all reactions were carried out with **1a** (0.2 mmol), amine (2 equiv.) in 1.0 mL toluene under argon in a sealed tube, 100 °C for 24 h. Yields were determined by NMR.

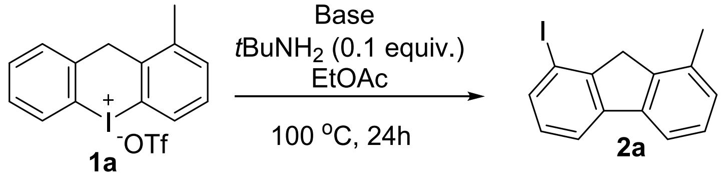
Table S2: Screening of solvent [a]



Entry	Solvent	Yield (%)
1	DMF	48
2	DMSO	30
3	THF	47
4	DCE	34
5	H_2O	12
6	EtOH	45
7	*i*PrOH	51
8	*n*BuOH	52
9	EtOAc	84
10	Anisole	5
11	Sulfolane	80
12	*i*PrOAc	81
13	*n*BuOAc	81

[a] Reaction conditions: all reactions were carried out with **1a** (0.2 mmol), *t*$BuNH_2$ (2 equiv.) in 1.0 mL solvent under argon in a sealed tube, 100 °C for 24 h. Yields were determined by NMR.

Table S3: Screening of base [a]



Entry	Amines	Base	Yield (%)
2	*t*BuNH$_2$	NA	13
3	*t*BuNH$_2$	Na$_2$CO$_3$	88
4	*t*BuNH$_2$	NaHCO$_3$	86
5	*t*BuNH$_2$	K$_2$CO$_3$	83
6	*t*BuNH$_2$	NaOH	41
7	*t*BuNH$_2$	Li$_2$CO$_3$	39
8	*t*BuNH$_2$	(NH$_4$)$_2$CO$_3$	35
9	*t*BuNH$_2$	AcONa	43
10	*t*BuNH$_2$	*t*BuCO$_2$Na	63
11	*t*BuNH$_2$	HCO$_2$Na	46

[a] Reaction conditions: all reactions were carried out with **1a** (0.2 mmol), *t*BuNH$_2$ (0.1 equiv.), base (2.0 equiv.) in 1.0 mL EtOAc under argon in a sealed tube, 100 °C for 24 h. NA: not added. Yields were determined by NMR.

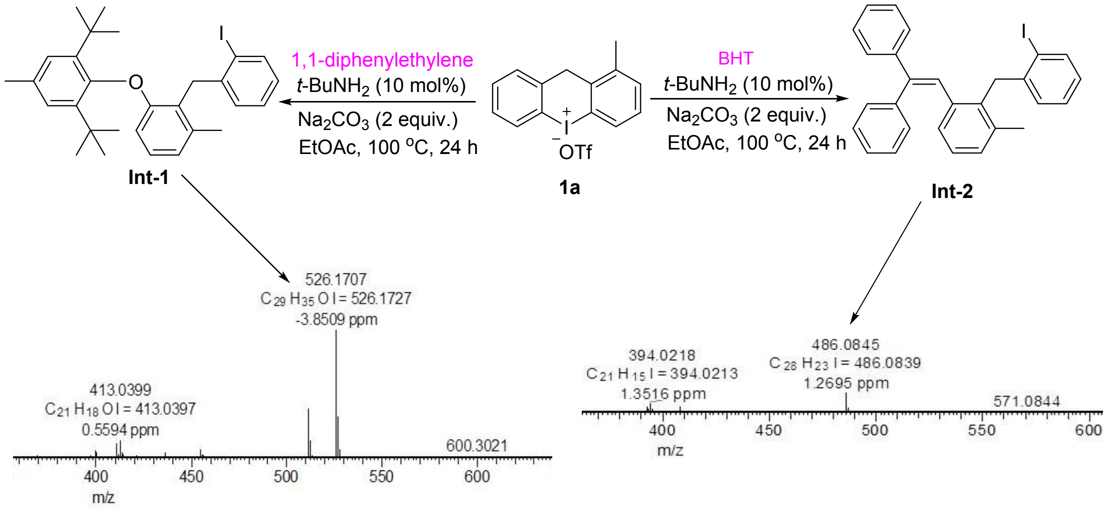
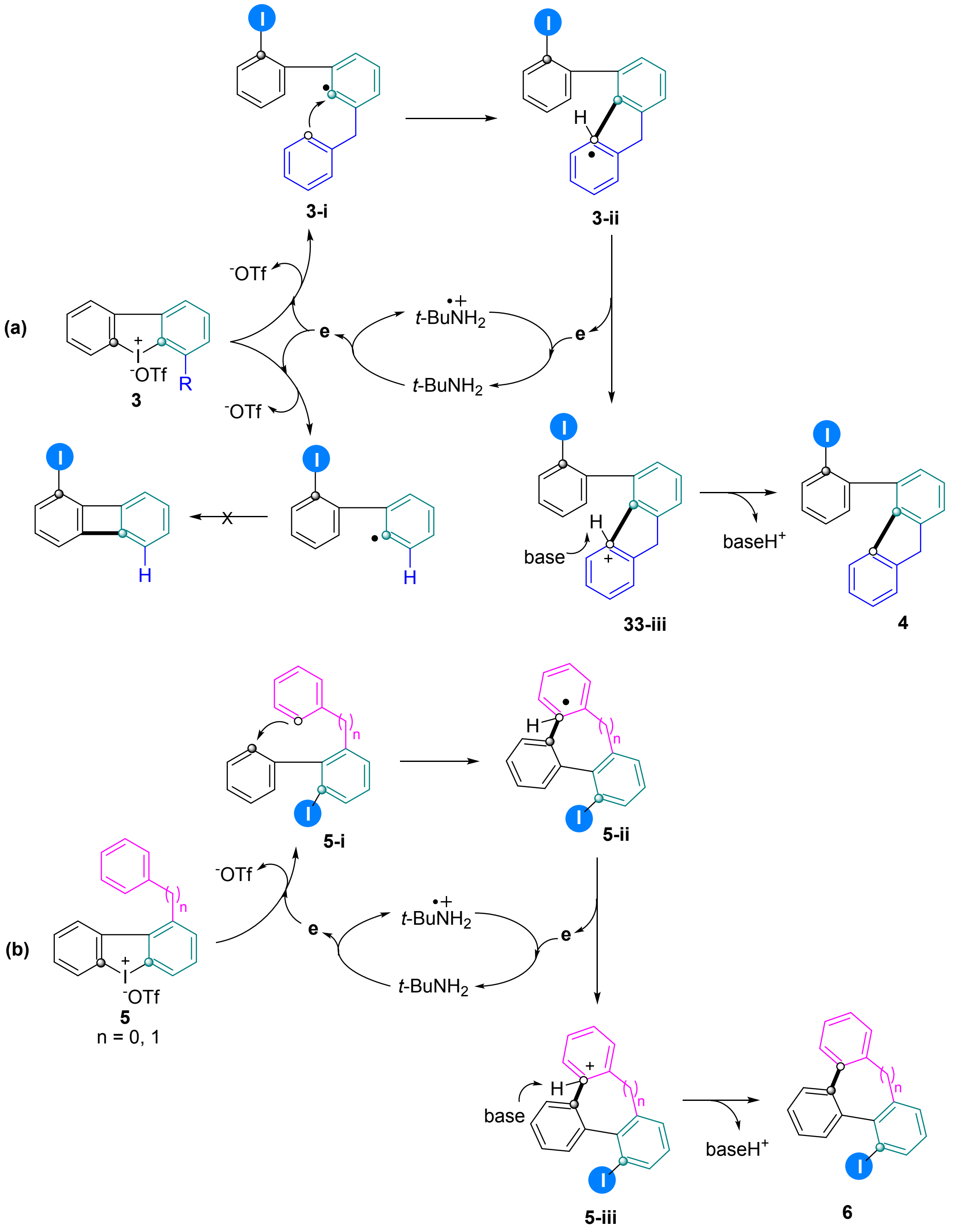


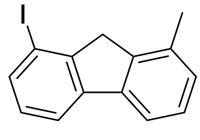
Figure S1. Control experiments with BHT or 1,1-diphenylethylene. Note：One drop of the crude reaction mixture was delivered to EI-HRMS for detection.



Scheme S1. The potential free radical pathways to generate polycyclic aromatic hydrocarbons **4** and **6** from cyclic diaryliodoniums **3** and **5**.

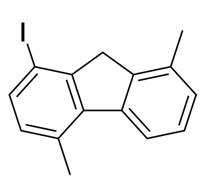
The general information for the synthetic experiments: Synthesis details for key compounds are described here. All solvents were commercially available and were used without a further purification unless stated. The chemicals used were either purchased from commercial sources or prepared according to literature procedures. The ^{1}H and ^{13}C nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance spectrometer 400 at 400 MHz and 100 MHz respectively. Chemical shifts are given in ppm (δ) referenced to $CDCl_3$ with 7.26 for ^{1}H and 77.10 for ^{13}C, and to d_6-*DMSO* with 2.50 for ^{1}H and 39.5 for ^{13}C. In the case of multiplet, the signals are reported as intervals. Signals are abbreviated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Coupling constants are expressed in hertz. High-resolution mass spectra (HRMS) were recorded on Thermo Fisher spectrometer, Orbitrap QExactive GC/MS (EI mode) and Orbitrap QExactive LC/MS(ESI mode) or BRUKER VPEXII spectrometer (ESI mode).The progress of the reactions was monitored by thin-layer chromatography on a glass plate coated with silica gel with fluorescent indicator (GF254). Column chromatography was performed on silica gel (200-300 mesh).

General procedure to synthesize π-extended arenes (2, 4, 6): To a reaction tube was added cyclic diaryliodoniums **1**, **3**, and **5** (0.2 mmol, 1.0 equiv), Na_2CO_3 (2.0 equiv), degassed and recharged with argon, a solution of *tert*-butylamine (0.1 equiv) in EtOAc (1.0 mL) was added by a syringe. Then the tube was sealed, and the reaction proceeded at 100 °C for 24 h under argon atmosphere. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc) to provide products **2**, **4**, **6**.



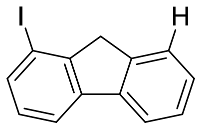
2a

1-Iodo-8-methyl-9*H*-fluorene (2a): The product was isolated as a white solid (52 mg, 85%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 7.5 Hz, 1H), 7.67 (d, J = 7.8 Hz, 1H), 7.59 (d, J = 7.6 Hz, 1H), 7.33 (t, J = 7.5 Hz, 1H), 7.16 (d, J = 7.4 Hz, 1H), 7.11 (t, J = 7.7 Hz, 1H), 3.70 (s, 2H), 2.45 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 142.9, 141.4, 141.0, 135.8, 134.5, 128.7, 128.5, 127.4, 119.8, 118.2, 94.1, 41.5, 19.0 ppm. HRMS (EI, *m/z*) calcd for $C_{14}H_{11}I$ [M]$^+$: 305.9900, found: 305.9900.

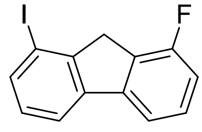


2b

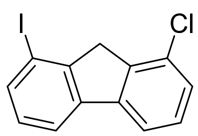
1-Iodo-4,8-dimethyl-9*H*-fluorene (2b): The product was isolated as a white solid (47 mg, 73%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 7.8 Hz, 1H), 7.56 (d, J = 7.9 Hz, 1H), 7.34 (t, J = 7.6 Hz, 1H), 7.17 (d, J = 7.5 Hz, 1H), 6.90 (d, J = 7.9 Hz, 1H), 3.68 (s, 2H), 2.68 (s, 3H), 2.46 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.6, 142.4, 141.3, 141.0, 135.4, 134.2, 133.2, 131.2, 127.8, 127.3, 121.4, 90.7, 41.9, 20.8, 19.1 ppm. HRMS (EI, *m/z*) calcd for $C_{15}H_{13}I$ [M]$^+$: 320.0056, found: 320.0058.


2c

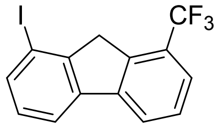
1-Iodo-9*H*-fluorene (2c): The product was isolated as a crystal solid (50 mg, 86%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 7.4, 4.3 Hz, 2H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.57 (d, *J* = 7.4 Hz, 1H), 7.40 (t, *J* = 7.3 Hz, 1H), 7.34 (td, *J* = 7.4, 1.1 Hz, 1H), 7.12 (t, *J* = 7.7 Hz, 1H), 3.82 (s, 2H). ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 142.6, 142.2, 141.8, 135.9, 128.8, 127.5, 127.1, 125.2, 120.7, 119.7, 94.0, 42.6 ppm. HRMS (EI, *m/z*) calcd for $C_{13}H_9I$ [M]$^+$: 291.9743, found: 291.9745.


2d

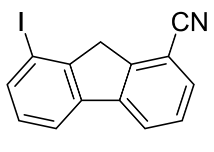
1-Fluoro-8-iodo-9*H*-fluorene (2d): The product was isolated as a white solid (47 mg, 76%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 14.7, 7.7 Hz, 2H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.38 (dt, *J* = 12.9, 6.5 Hz, 1H), 7.13 (t, *J* = 7.7 Hz, 1H), 7.03 (t, *J* = 8.6 Hz, 1H), 3.84 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6 (d, *J* = 247.6 Hz), 147.6, 144.8 (d, *J* = 6.6 Hz), 141.6 (d, *J* = 2.5 Hz), 136.6, 129.2 (d, *J* = 7.1 Hz), 128.9, 127.7 (d, *J* = 18.3 Hz), 120.1, 116.5 (d, *J* = 3.2 Hz), 114.2, 114.0, 94.0, 39.0 ppm. HRMS (EI, *m/z*) calcd for $C_{13}H_8FI$ [M]$^+$: 309.9649, found: 309.9651.


2e

1-Chloro-8-iodo-9*H*-fluorene (2e): The product was isolated as a white solid (53 mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (t, *J* = 8.3 Hz, 2H), 7.63 (d, *J* = 6.7 Hz, 1H), 7.33 (t, *J* = 10.0 Hz, 2H), 7.13 (t, *J* = 7.2 Hz, 1H), 3.81 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.4, 143.4, 141.8, 140.2, 136.6, 131.2, 129.0, 128.8, 127.4, 120.2, 119.0, 93.9, 42.2 ppm. HRMS (EI, *m/z*) calcd for $C_{13}H_8ClI$ [M]$^+$: 325.9354, found: 325.9355.

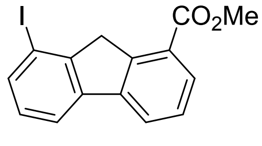

2f

1-Iodo-8-(trifluoromethyl)-9*H*-fluorene (2f): The product was isolated as a white solid (64 mg, 89%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.6 Hz, 1H), 7.75 (dd, *J* = 16.7, 7.7 Hz, 2H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.14 (t, *J* = 7.7 Hz, 1H), 3.97 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 143.4, 140.8, 139.5, 136.9, 128.9, 127.6, 127.5, 127.2, 125.6, 124.1 (q, *J* = 4.5 Hz), 123.9, 123.4, 119.8, 93.7, 41.6. ppm. HRMS (EI, *m/z*) calcd for $C_{14}H_8F_3I$ [M]$^+$: 359.9617, found: 359.9617.



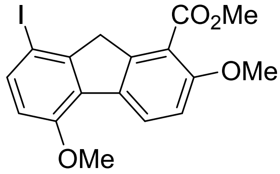
2g

8-Iodo-9*H*-fluorene-1-carbonitrile (2g): The product was isolated as a white solid (43 mg, 68%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94 (d, *J* = 7.3 Hz, 1H), 7.80 – 7.73 (m, 2H), 7.61 (dd, *J* = 7.7, 0.9 Hz, 1H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.17 (t, *J* = 7.7 Hz, 1H), 3.98 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.1, 145.8, 142.9, 140.6, 137.3, 130.4, 129.2, 128.0, 124.8, 120.2, 117.5, 109.5, 93.9, 42.2 ppm. HRMS (EI, *m/z*) calcd for $C_{14}H_8IN$ $[M]^+$: 316.9696, found: 316.9697.



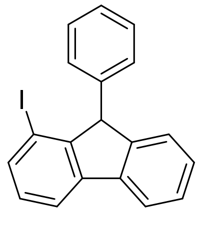
2h

Methyl 8-iodo-9*H*-fluorene-1-carboxylate (2h): The product was isolated as a white solid (55 mg, 79%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 7.8, 0.8 Hz, 1H), 7.92 (d, *J* = 7.6 Hz, 1H), 7.77 (d, *J* = 7.5 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.49 (t, *J* = 7.7 Hz, 1H), 7.13 (t, *J* = 7.7 Hz, 1H), 4.16 (s, 2H), 4.00 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.1, 148.2, 144.1, 143.0, 141.1, 136.6, 129.1, 128.7, 127.4, 127.0, 124.8, 119.6, 93.8, 52.2, 44.1 ppm. HRMS (EI, *m/z*) calcd for $C_{15}H_{11}IO_2$ $[M]^+$: 349.9798, found: 349.9800.



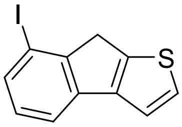
2i

Methyl 8-iodo-2,5-dimethoxy-9H-fluorene-1-carboxylate (2i): The product was isolated as a white solid (66 mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.6 Hz, 1H), 7.54 (d, *J* = 8.6 Hz, 1H), 6.99 (d, *J* = 8.6 Hz, 1H), 6.67 (d, *J* = 8.6 Hz, 1H), 4.00 (s, 3H), 3.98 (s, 3H), 3.93 (s, 3H), 3.92 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.3, 157.2, 155.6, 148.5, 143.4, 135.9, 134.6, 129.8, 127.6, 118.4, 111.5, 110.8, 82.2, 56.6, 55.6, 52.2, 43.2 ppm. HRMS (EI, *m/z*) calcd for $C_{17}H_{15}IO_4$ $[M]^+$: 410.0010, found: 410.0010.



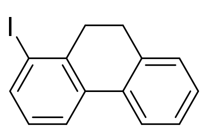
2j

1-Iodo-9-phenyl-9*H*-fluorene (2j): The product was isolated as a white solid (57 mg, 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 7.5, 0.5 Hz, 1H), 7.74 (d, *J* = 7.6 Hz, 1H), 7.70 (dd, *J* = 7.8, 0.7 Hz, 1H), 7.35 (t, *J* = 7.7 Hz, 1H), 7.29 – 7.22 (m, 5H), 7.14 (t, *J* = 7.7 Hz, 1H), 7.10 – 7.05 (m, 2H), 5.00 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 148.1, 143.7, 139.8, 139.7, 137.5, 129.5, 129.0, 128.8, 128.1, 127.5, 126.9, 125.4, 120.4, 119.7, 94.8, 58.6 ppm. HRMS (EI, *m/z*) calcd for $C_{19}H_{13}I$ $[M]^+$: 368.0056, found: 368.0053.



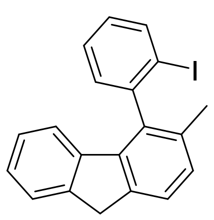
2k

7-Iodo-8H-indeno[2,1-*b*]thiophene (2k): ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.55 (d, *J* = 7.8 Hz, 1H), 7.51 (d, *J* = 7.3 Hz, 1H), 7.36 (d, *J* = 4.2 Hz, 1H), 7.26 (s, 1H), 7.07 (t, *J* = 7.4 Hz, 1H), 3.77 (s, 2H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 151.1, 147.5, 143.7, 140.2, 133.8, 129.0, 128.8, 119.0, 119.0, 93.3, 40.2 ppm. HRMS (EI, *m/z*) calcd for $C_{11}H_7IS$ [M]$^+$: 297.9308, found: 297.9311.



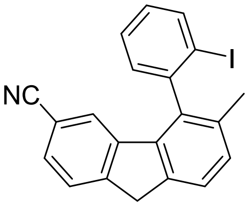
2l

1-Iodo-9,10-dihydrophenanthrene (2l): The product was isolated as a white solid (35 mg, 58%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 7.76 (d, *J* = 7.6 Hz, 2H), 7.34 – 7.28 (m, 2H), 7.25 – 7.22 (m, 3H), 2.88 (s, 4H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 147.5, 143.4, 140.8, 139.5, 136.9, 129.0, 127.6, 127.5, 127.2, 125.8, 124.1, 124.1, 124.0, 124.0, 123.9, 123.1, 93.7, 41.6 ppm. HRMS (EI, *m/z*) calcd for $C_{14}H_{11}I$ [M]$^+$: 305.9900, found: 305.9902.



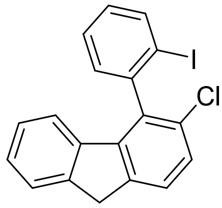
4a

4-(2-Iodophenyl)-3-methyl-9H-fluorene (4a): The product was isolated as a white solid (48 mg, 63%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.07 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.55 – 7.48 (m, 3H), 7.27 (dt, *J* = 12.7, 4.5 Hz, 2H), 7.22 – 7.15 (m, 2H), 7.00 (t, *J* = 7.6 Hz, 1H), 6.24 (d, *J* = 7.8 Hz, 1H), 4.01 – 3.83 (m, 2H), 2.10 (s, 3H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 145.2, 144.0, 141.6, 141.3, 139.6, 139.2, 138.9, 134.5, 130.0, 129.3, 129.1, 128.3, 126.7, 126.3, 124.9, 124.5, 122.2, 100.5, 36.8, 19.8 ppm. HRMS (EI, *m/z*) calcd for $C_{20}H_{15}I$ [M]$^+$: 382.0213, found: 382.0218.



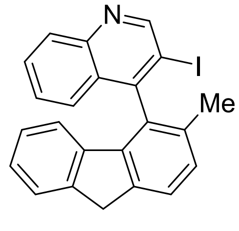
4b

5-(2-Iodophenyl)-6-methyl-9*H*-fluorene-3-carbonitrile (4b): The product was isolated as a white solid (53 mg, 66%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.09 (d, *J* = 7.9 Hz, 1H), 7.60 – 7.55 (m, 2H), 7.53 (dd, *J* = 4.4, 3.2 Hz, 1H), 7.47 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.33 (d, *J* = 8.1 Hz, 1H), 7.27 – 7.21 (m, 2H), 6.39 (d, *J* = 0.7 Hz, 1H), 4.02 – 3.91 (m, 2H), 2.11 (s, 3H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 149.0, 144.2, 142.6, 141.0, 139.9, 139.5, 137.4, 135.2, 130.1, 13.0, 129.8, 129.5, 129.2, 128.2, 125.6, 124.7, 119.7, 110.6, 99.8, 37.2, 19.7 ppm. HRMS (EI, *m/z*) calcd for $C_{21}H_{14}IN$ [M]$^+$: 407.0165, found: 407.0167.



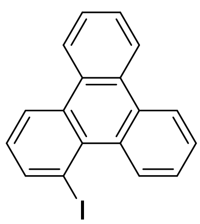
4c

3-Chloro-4-(2-iodophenyl)-9*H*-fluorene (4c): The product was isolated as a white solid (48 mg, 60%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 8.08 (d, *J* = 7.9 Hz, 1H), 7.57 – 7.49 (m, 3H), 7.44 (d, *J* = 8.0 Hz, 1H), 7.31 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.27 – 7.21 (m, 2H), 7.04 (t, *J* = 7.6 Hz, 1H), 6.29 (d, *J* = 7.9 Hz, 1H), 4.01 – 3.87 (m, 2H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 144.1, 143.2, 142.3, 141.2, 140.7, 139.5, 137.4, 132.3, 130.2, 129.9, 129.0, 127.3, 127.2, 127.0, 125.6, 125.0, 122.5, 100.0, 36.8 ppm. HRMS (EI, *m/z*) calcd for $C_{19}H_{12}ClI$ [M]$^+$: 401.9667, found: 401.9672.



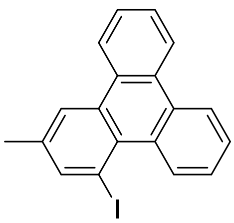
4d

3-Iodo-4-(3-methyl-9*H*-fluoren-4-yl)quinolone (4d): The product was isolated as a colorless oil. ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.27 (s, 1H), 8.18 (d, *J* = 8.4 Hz, 1H), 7.79 – 7.73 (m, 1H), 7.48 (t, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.3 Hz, 3H), 7.28 (d, *J* = 7.9 Hz, 1H), 7.24 (t, *J* = 5.7 Hz, 2H), 7.14 (d, *J* = 7.8 Hz, 1H), 4.22 (s, 2H), 1.89 (s, 3H) ppm. HRMS (EI, *m/z*) calcd for $C_{23}H_{16}NI$ [M]$^+$: 433.0322, found: 433.0322.



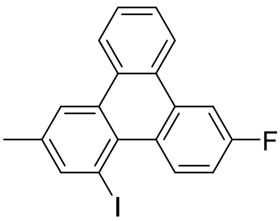
6a

1-iodotriphenylene (6a): The product was isolated as a white solid (35 mg, 49%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.43 (dd, *J* = 8.3, 0.8 Hz, 1H), 8.61 – 8.53 (m, 3H), 8.50 (dd, *J* = 6.7, 2.7 Hz, 1H), 8.29 (dd, *J* = 7.5, 0.9 Hz, 1H), 7.70 – 7.62 (m, 3H), 7.62 – 7.56 (m, 1H), 7.24 (t, *J* = 7.9 Hz, 1H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 142.3, 132.2, 132.2, 131.0, 130.3, 129.9, 129.7, 129.5, 128.4, 128.2, 127.9, 127.8, 127.6, 127.3, 124.8, 123.4, 123.4, 123.3, 123.2, 123.2, 90.9 ppm. HRMS (EI, *m/z*) calcd for $C_{18}H_{11}I$ [M]$^+$: 353.9900, found: 353.9903.



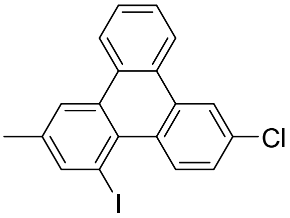
6b

1-Iodo-3-methyltriphenylene (6b): The product was isolated as a white solid (53 mg, 72%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.40 (d, *J* = 8.1 Hz, 1H), 8.62 – 8.46 (m, 3H), 8.37 (s, 1H), 8.16 (s, 1H), 7.72 – 7.51 (m, 4H), 2.52 (s, 3H) ppm. ^{13}C NMR (100 MHz, CDCl$_3$) δ 143.6, 137.7, 131.9, 130.6, 130.4, 129.9, 129.8, 129.4, 128.1, 127.8, 127.7, 127.5, 124.8, 123.6, 123.3, 123.2, 123.1, 90.9, 21.0 ppm. HRMS (EI, *m/z*) calcd for $C_{19}H_{13}I$ [M]$^+$: 368.0056, found: 368.0060.



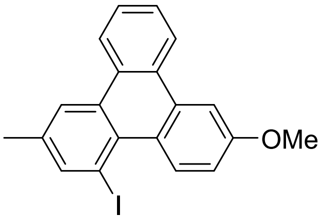
6c

10-Fluoro-1-iodo-3-methyltriphenylene (6c): The product was isolated as a white solid (52 mg, 68%). ^{1}H NMR (400 MHz, CDCl$_3$) δ 9.39 (dd, *J* = 9.2, 5.9 Hz, 1H), 8.49 (dd, *J* = 6.3, 3.1 Hz, 1H), 8.41 (dd, *J* = 6.3, 3.2 Hz, 1H), 8.35 (s, 1H), 8.18 – 8.10 (m, 2H), 7.68 – 7.59 (m, 2H), 7.32 – 7.27 (m, 1H), 2.51 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2 (d, *J* = 247.1 Hz), 143.8, 137.5, 132.9 (d, *J* = 8.1 Hz), 131.4, 130.3, 130.2, 129.9, 129.7 (d, *J* = 3.0 Hz), 129.4, 128.1, 127.9, 126.2 (d, *J* = 2.3 Hz), 123.7, 123.4 (d, *J* = 3.3 Hz), 112.8 (d, *J* = 22.3 Hz), 108.6 (d, *J* = 22.6 Hz), 90.6, 21.0 ppm. HRMS (EI, *m/z*) calcd for $C_{19}H_{12}FI$ $[M]^+$: 385.9962, found: 385.9963.



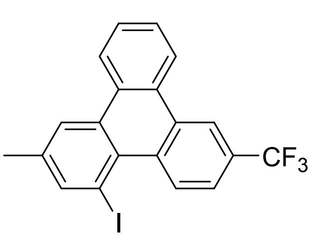
6d

10-Chloro-1-iodo-3-methyltriphenylene (6d): The product was isolated as a white solid (54 mg, 67%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.34 (d, *J* = 8.9 Hz, 1H), 8.52 – 8.42 (m, 3H), 8.35 (s, 1H), 8.15 (s, 1H), 7.63 (dd, *J* = 5.9, 3.5 Hz, 2H), 7.51 (dd, *J* = 8.9, 2.0 Hz, 1H), 2.51 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 137.8, 133.6, 132.1, 131.7, 129.7, 129.3, 129.2, 129.0, 128.0, 128.0, 127.8, 124.9, 123.6, 123.3, 123.2, 122.7, 90.7, 21.0 ppm. HRMS (EI, *m/z*) calcd for $C_{19}H_{12}ClI$ $[M]^+$: 401.9667, found: 401.9665.



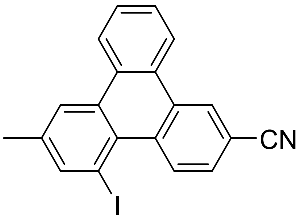
6e

1-Iodo-10-methoxy-3-methyltriphenylene (6e): The product was isolated as a white solid (41 mg, 52%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.36 (d, *J* = 9.2 Hz, 1H), 8.49 (dd, *J* = 9.7, 6.3 Hz, 2H), 8.36 (s, 1H), 8.13 (s, 1H), 7.95 (d, *J* = 2.5 Hz, 1H), 7.62 (dd, *J* = 6.2, 3.3 Hz, 2H), 7.19 (dd, *J* = 9.1, 2.6 Hz, 1H), 4.02 (s, 3H), 2.51 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.1, 143.6, 136.7, 132.4, 130.9, 130.2, 130.0, 129.9, 129.6, 127.6, 127.6, 123.7, 123.6, 123.5, 123.3, 112.6, 106.2, 90.3, 55.6, 21.0 ppm. HRMS (EI, *m/z*) calcd for $C_{20}H_{15}IO$ $[M]^+$: 398.0162, found: 398.0168.



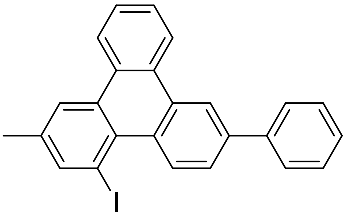
6f

1-Iodo-3-methyl-10-(trifluoromethyl)triphenylene (6f): The product was isolated as a white solid (50 mg, 58%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49 (t, *J* = 7.9 Hz, 1H), 8.75 (d, *J* = 5.0 Hz, 1H), 8.58 – 8.52 (m, 1H), 8.51 – 8.46 (m, 1H), 8.36 (d, *J* = 11.6 Hz, 1H), 8.17 (d, *J* = 4.5 Hz, 1H), 7.76 (d, *J* = 8.7 Hz, 1H), 7.65 (ddd, *J* = 7.0, 5.8, 1.5 Hz, 2H), 2.52 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.0, 138.8, 132.6, 132.2, 130.6, 129.8, 129.6, 129.3, 129.1, 128.9, 128.7, 128.3, 128.2, 125.6, 123.8, 123.5, 123.3, 120.9 (q, *J* = 3.4 Hz), 120.4 (q, *J* = 4.1 Hz), 91.3, 21.1 ppm. HRMS (EI, *m/z*) calcd for $C_{20}H_{12}F_3I$ $[M]^+$: 435.9930, found: 435.9940.



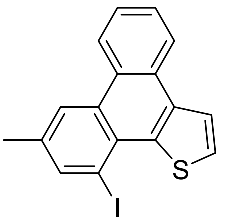
6g

5-Iodo-7-methyltriphenylene-2-carbonitrile (6g): The product was isolated as a white solid (52 mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.46 (d, J = 8.6 Hz, 1H), 8.80 (d, J = 1.4 Hz, 1H), 8.53 – 8.44 (m, 2H), 8.37 (s, 1H), 8.18 (s, 1H), 7.79 – 7.74 (m, 1H), 7.72 – 7.65 (m, 2H), 2.54 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 139.3, 132.9, 132.7, 130.6, 129.7, 128.7, 128.6, 128.6, 128.3, 128.3, 127.8, 126.6, 123.7, 123.4, 123.1, 119.3, 110.7, 91.4, 21.1 ppm. HRMS (EI, *m/z*) calcd for $C_{20}H_{12}IN$ [M]$^+$: 393.0009, found: 393.0016.



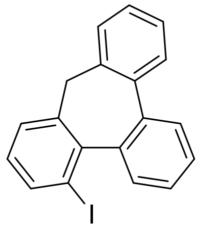
6h

1-Iodo-3-methyl-10-phenyltriphenylene (6h): The product was isolated as a white solid (44 mg, 49%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.49 (d, J = 8.6 Hz, 1H), 8.73 (s, 1H), 8.68 – 8.61 (m, 1H), 8.56 – 8.48 (m, 1H), 8.38 (s, 1H), 8.17 (s, 1H), 7.81 (dd, J = 6.9, 3.8 Hz, 3H), 7.64 (p, J = 7.0 Hz, 2H), 7.53 (t, J = 7.6 Hz, 2H), 7.43 (t, J = 7.3 Hz, 1H), 2.53 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 141.1, 140.2, 137.6, 132.0, 131.0, 130.4, 129.7, 129.0, 128.9, 128.5, 127.8, 127.7, 127.6, 127.5, 123.8, 123.7, 123.4, 123.3, 121.5, 90.9, 21.0 ppm. HRMS (EI, *m/z*) calcd for $C_{25}H_{17}I$ [M]$^+$: 444.0369, found: 444.0374.



6i

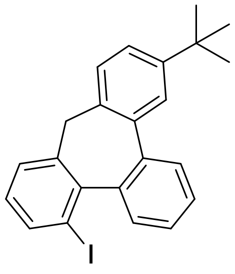
11-Iodo-9-methylphenanthro[9,10-b]thiophene (6i): The product was isolated as a white solid (37 mg, 50%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 – 8.61 (m, 1H), 8.57 (s, 1H), 8.37 – 8.30 (m, 1H), 8.24 (s, 1H), 8.10 (d, J = 5.5 Hz, 1H), 7.69 – 7.61 (m, 2H), 7.60 (d, J = 5.5 Hz, 1H), 2.55 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.2, 136.8, 136.2, 131.3, 129.3, 129.0, 128.9, 127.8, 126.4, 125.8, 124.4, 123.8, 122.4, 93.0, 21.2 ppm. HRMS (EI, *m/z*) calcd for $C_{17}H_{11}IS$ [M]$^+$: 373.9621, found: 373.9626.



6j

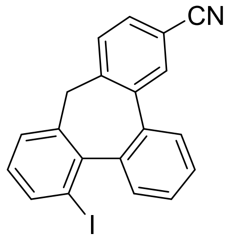
5-Iodo-9*H*-tribenzo[*a, c, e*][7]annulene (6j): The product was isolated as a white solid (32 mg, 44%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 – 7.75 (m, 2H), 7.63 – 7.55 (m, 2H), 7.49 (td, J = 7.5, 1.3 Hz, 1H), 7.41 (td, J = 7.5, 1.4 Hz, 1H), 7.33 – 7.24 (m, 4H), 6.90 (t, J = 7.7 Hz, 1H), 3.60 (d, J = 3.9 Hz, 2H) ppm. ^{13}C NMR

(100 MHz, $CDCl_3$) δ 145.0, 142.5, 140.7, 139.4, 139.2, 139.1, 138.0, 132.6, 129.7, 129.2, 128.7, 128.3, 127.9, 127.1, 126.5, 126.4, 125.8, 99.7, 41.5 ppm. HRMS (EI, *m/z*) calcd for $C_{19}H_{13}I$ $[M]^+$: 368.0056, found: 368.0060.



6k

12-(*tert*-butyl)-5-iodo-9*H*-tribenzo[*a, c, e*][7]annulene (6k): The product was isolated as a white solid (40 mg, 47%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.78 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.62 (d, *J* = 2.0 Hz, 1H), 7.58 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.49 (td, *J* = 7.5, 1.3 Hz, 1H), 7.44 – 7.38 (m, 1H), 7.28 (dd, *J* = 7.8, 2.2 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 1H), 6.90 (t, *J* = 7.7 Hz, 1H), 3.57 (s, 2H), 1.34 (s, 9H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.9, 145.3, 140.9, 139.8, 139.5, 139.4, 139.0, 137.4, 132.7, 129.8, 129.2, 128.2, 126.4, 126.2, 125.7, 125.7, 124.9, 99.8, 41.0, 34.8, 31.6 ppm. HRMS (EI, *m/z*) calcd for $C_{23}H_{21}I$ $[M]^+$: 424.0682, found: 424.0681.

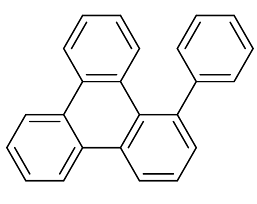


6l

13-Iodo-9*H*-tribenzo[*a, c, e*][7]annulene-6-carbonitrile (6l): The product was isolated as a white solid (34 mg, 43%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 7.5 Hz, 1H), 7.58 – 7.46 (m, 4H), 7.36 (d, *J* = 7.2 Hz, 1H), 7.28 (s, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 3.65 (s, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.2, 143.4, 140.3, 139.7, 139.5, 139.1, 137.0, 132.8, 132.2, 131.4, 129.6, 128.7, 127.4, 127.0, 126.6, 119.0, 111.1, 99.7, 41.6 ppm. HRMS (EI, *m/z*) calcd for $C_{20}H_{12}IN$ $[M]^+$: 393.0009, found: 393.0014.

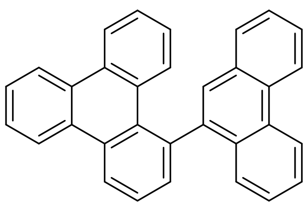
General procedure to synthesize 7a-7c

To **6i** (50 mg, 141.2 μmol) was added arylboronic acid (1.2 equiv.), K_3PO_4 (2.5 equiv.), $Pd(PPh_3)_4$ (0.05 equiv.), DMF (1 mL), degassed and recharged with argon three times. The reaction proceeded at 90 °C for 12 h under argon atmosphere. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel to provide **7a-7c**.



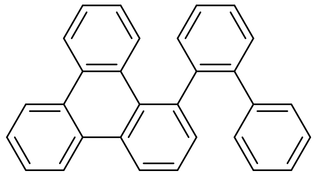
7a

1-phenyltriphenylene (7a): The product was isolated as a white solid (41 mg, 95%). ^1H NMR (400 MHz, $CDCl_3$) δ 8.67 – 8.60 (m, 3H), 8.55 (d, *J* = 8.1 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.69 – 7.63 (m, 3H), 7.53 (dd, *J* = 7.2, 1.0 Hz, 1H), 7.48 – 7.38 (m, 6H), 7.09 – 7.03 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.5, 140.9, 131.7, 131.6, 131.1, 130.4, 130.2, 130.1, 129.8, 129.3, 129.2, 128.8, 127.5, 127.5, 127.4, 127.0, 126.8, 126.5, 125.1, 123.8, 123.4, 123.3, 123.2, 122.4 ppm. HRMS (EI, *m/z*) calcd for $C_{24}H_{16}$ $[M]^+$: 304.1247, found: 304.1245.



7b

1-(Phenanthren-9-yl)triphenylene (7b): The product was isolated as a white solid (50 mg, 93%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (t, J = 8.1 Hz, 3H), 8.77 – 8.74 (m, 1H), 8.67 – 8.61 (m, 1H), 8.54 (d, J = 8.1 Hz, 1H), 7.88 (t, J = 8.5 Hz, 2H), 7.79 (s, 1H), 7.75 – 7.67 (m, 4H), 7.66 – 7.56 (m, 4H), 7.33 (td, J = 7.5, 0.9 Hz, 2H), 6.80 – 6.74 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.3, 138.6, 132.9, 132.2, 131.4, 131.2, 131.0, 130.8, 130.4, 130.3, 130.2, 130.0, 129.9, 128.9, 128.7, 127.6, 127.5, 127.3, 127.2, 127.0, 127.0, 126.9, 126.8, 126.8, 126.6, 126.0, 123.9, 123.4, 123.3, 123.1, 123.0, 122.8 ppm. HRMS (EI, *m/z*) calcd for $C_{32}H_{20}$ $[M]^+$: 404.1560, found: 404.1567.



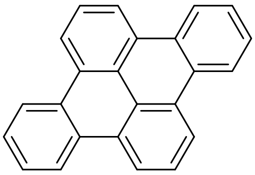
7c

1-([1,1'-Biphenyl]-2-yl)triphenylene (7c): The product was isolated as a white solid (53 mg, 98%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (dd, J = 6.3, 3.3 Hz, 1H), 8.54 (dd, J = 10.7, 5.7 Hz, 2H), 8.50 (d, J = 7.9 Hz, 1H), 7.89 (dd, J = 8.4, 0.7 Hz, 1H), 7.65 – 7.58 (m, 2H), 7.54 – 7.45 (m, 4H), 7.45 – 7.39 (m, 2H), 7.29 (dd, J = 7.3, 1.2 Hz, 1H), 7.11 (ddd, J = 8.3, 7.0, 1.2 Hz, 1H), 6.95 – 6.87 (m, 5H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 141.1, 140.2, 139.9, 132.4, 131.3, 131.1, 130.9, 130.7, 130.2, 130.1, 130.0, 129.5, 129.1, 128.9, 128.2, 127.8, 127.5, 127.3, 127.3, 126.6, 126.3, 126.2, 125.5, 123.6, 123.1, 122.2 ppm. HRMS (EI, *m/z*) calcd for $C_{30}H_{20}$ $[M]^+$: 380.1560, found: 380.1564.

The procedure to synthesize 8a

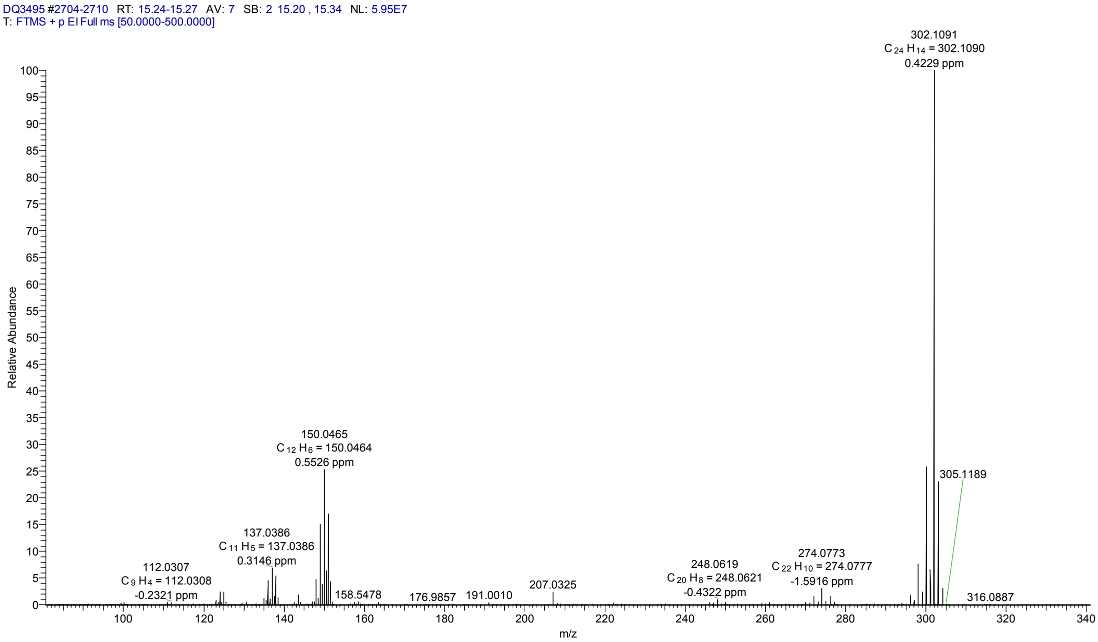
To **7a** (30.00 mg, 98.56 μmol) was added $FeCl_3$ (95.9 mg, 591.34 μmol, 6.0 equiv.), degassed and recharged with argon three times, and DCM (10 mL) was added by a syringe. The reaction proceeded at 0 °C for 12 h under argon atmosphere. The reaction was quenched with methanol (5.0 mL), then the reaction mixture was concentrated by rotary evaporaion. To the crude residue was added HCl (conc. 10 mL) and ultrasonicated for 10 min, the mixture was filtered and washed with water (20 mL), methanol

(20 mL) and DCM (20 mL) successively. After dried in vacuum, **8a** was obtained as a yellow solid (19 mg, 64 %).



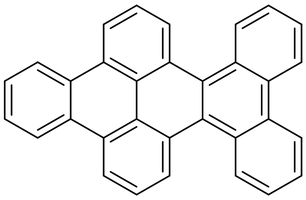
8a

Dibenzo[*fg, op*]tetracene (8a): ^{1}H NMR (400 MHz, $C_2D_4Cl_2$) δ 8.96 (d, *J* = 8.0 Hz, 4H), 8.85 (dd, *J* = 6.2, 3.4 Hz, 4H), 8.11 (t, *J* = 7.9 Hz, 2H), 7.79 (dd, *J* = 6.2, 3.3 Hz, 4H) ppm. HRMS (EI, *m/z*) calcd for $C_{24}H_{14}$ [M]$^+$: 302.1090, found: 302.1091. Data are identical to reported work.[1]



The procedure to synthesize 8b

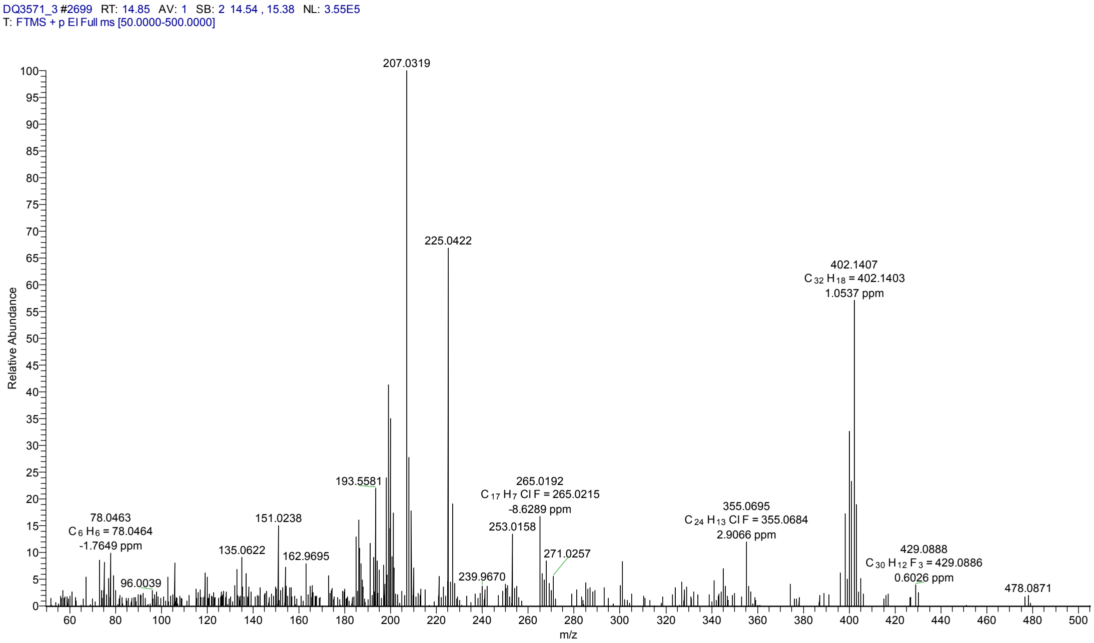
To **7b** (30.00 mg, 74.16 µmol) was added $FeCl_3$ (72.17 mg, 444.98 µmol, 6.0 equiv.), degassed and recharged with argon three times, and DCM (10 mL) was added by a syringe. The reaction proceeded at 0 °C for 12 h under argon atmosphere. The reaction was quenched with methanol (5.0 mL), then the reaction mixture was concentrated by rotary evaporaion. To the crude residue was added HCl (conc. 10 mL) and ultrasonicated for 10 min, the mixture was filtered and washed with water (20 mL), methanol (20 mL) and DCM (20 mL) successively. After dried in vacuum, **8b** was obtained as a yellow solid (17 mg, 57 %).



Tetrabenzo[*a, c, fg, op*]tetracene (8b): ^{1}H NMR (400 MHz, $C_2D_4Cl_2$) δ 8.99 (d, *J* = 7.9 Hz, 2H), 8.94 (d, *J* = 7.8 Hz, 2H), 8.90 – 8.82 (m, 4H), 8.78 (d, *J* = 8.0 Hz, 2H), 8.08 (t, *J* = 7.9 Hz, 2H), 7.80 (dd, *J* = 6.1, 3.1

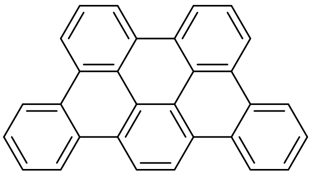
8b

Hz, 2H), 7.76 (d, *J* = 7.6 Hz, 2H), 7.72 (d, *J* = 7.6 Hz, 2H) ppm. HRMS (EI, *m/z*) calcd for $C_{32}H_{18}$ $[M]^+$: 402.1403, found: 402.1407.

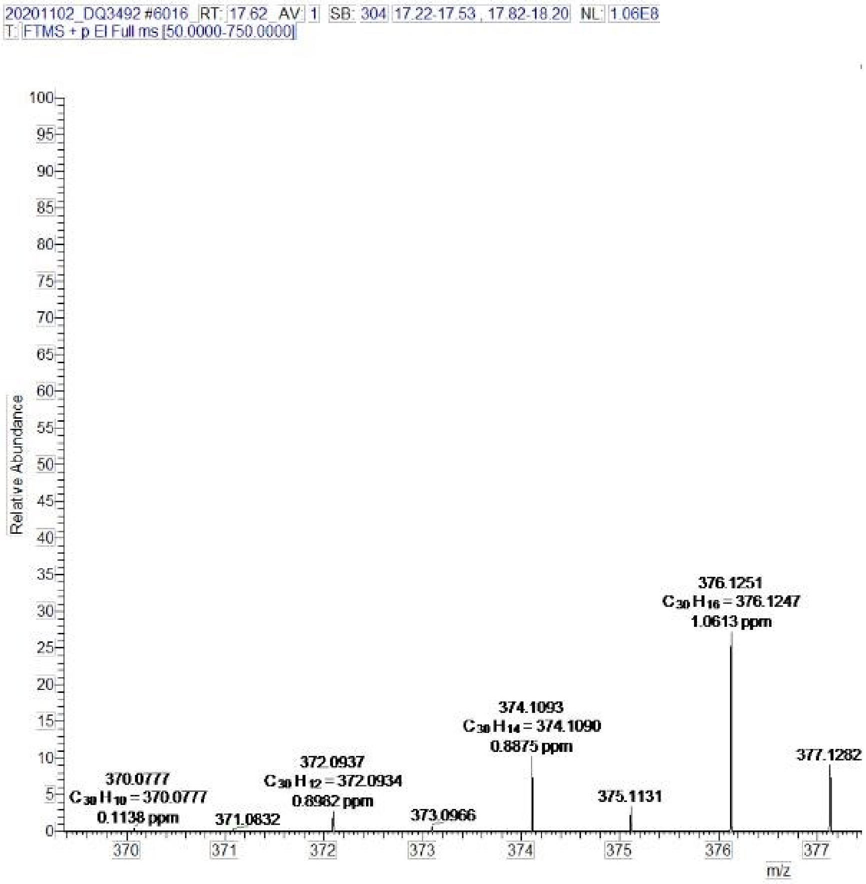


The procedure to synthesize 8c

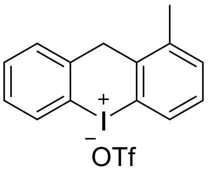
To **7c** (30.00 mg, 78.85 μmol) was added $FeCl_3$ (153.46 mg, 946.15 μmol, 12.0 equiv.), degassed and recharged with argon three times, and DCM (10 mL) was added by a syringe. The reaction proceeded at 0 °C for 12 h under argon atmosphere. The reaction was quenched with methanol (5.0 mL), then the reaction mixture was concentrated by rotary evaporaion. To the crude residue was added HCl (conc. 10 mL) and ultrasonicated for 10 min, the mixture was filtered and washed with water (20 mL), methanol (20 mL) and DCM (20 mL) successively. After dried in vacuum, **8c** was obtained as a dark solid (18 mg, 61 %).


8c

Tribenzo[*fg, ij, rst*]pentaphene (8c). HRMS (EI, *m/z*) calcd for $C_{30}H_{16}$ $[M]^+$: 376.1247, found: 376.1251.

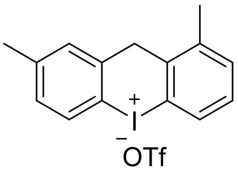


Cyclic diphenyl iodonium salts 1 were prepared according to reported procedures.[2]



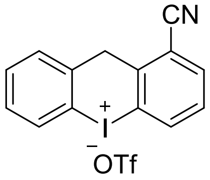
1a

1-Methyl-10*H*-dibenzo[*b, e*]iodinin-5-ium trifluoromethanesulfonate (1a): ^{1}H NMR (400 MHz, DMSO) δ 8.11 – 8.05 (m, 1H), 7.91 (t, *J* = 8.0 Hz, 2H), 7.58 (td, *J* = 7.5, 0.9 Hz, 1H), 7.45 (d, *J* = 7.5 Hz, 1H), 7.43 – 7.38 (m, 1H), 7.27 (t, *J* = 7.8 Hz, 1H), 4.25 (s, 2H), 2.56 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 139.2, 138.9, 137.2, 133.8, 133.4, 132.0, 131.9, 131.0, 129.3, 128.9, 117.6, 116.9, 42.8, 20.6 ppm. HRMS (ESI, *m/z*) calcd for $C_{14}H_{12}I$ [M-OTf]$^+$: 306.9978, found: 306.9974.



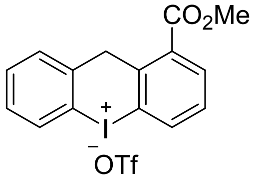
1b

1, 8-Dimethyl-10*H*-dibenzo[*b, e*]iodinin-5-ium trifluoromethanesulfonate (1b): ^{1}H NMR (400 MHz, DMSO) δ 7.95 (d, *J* = 8.2 Hz, 1H), 7.92 (d, *J* = 8.1 Hz, 1H), 7.75 (s, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.24 (d, *J* = 8.3 Hz, 1H), 4.22 (s, 2H), 2.57 (s, 3H), 2.37 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 142.0, 138.8, 138.5, 137.0, 133.2, 133.1, 131.6, 131.3, 129.6, 128.5, 117.4, 112.9, 42.4, 20.6, 20.3 ppm. HRMS (ESI, *m/z*) calcd for $C_{15}H_{14}I$ [M-OTf]$^+$: 321.0135, found: 321.0135.



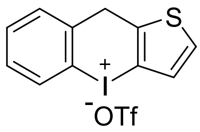
1g

1-Cyano-10*H*-dibenzo[*b, e*]iodinin-5-ium trifluoromethanesulfonate (1g): ^{1}H NMR (400 MHz, DMSO) δ 8.43 – 8.36 (m, 1H), 8.18 – 8.09 (m, 2H), 7.89 (d, *J* = 6.4 Hz, 1H), 7.68 – 7.59 (m, 2H), 7.51 – 7.44 (m, 1H), 4.52 (s, 2H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 141.9, 138.8, 137.3, 136.0, 133.8, 132.1, 131.0, 129.7, 129.5, 117.3, 116.5, 116.5, 112.7, 44.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{14}H_9IN$ [M-OTf]$^+$: 317.9774, found: 317.9769.



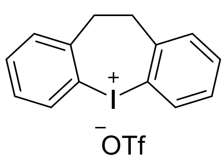
1h

1-(Methoxycarbonyl)-10*H*-dibenzo[*b, e*]iodinin-5-ium trifluoromethanesulfonate (1h): ^{1}H NMR (400 MHz, DMSO) δ 8.31 (dd, *J* = 8.1, 1.1 Hz, 1H), 8.11 (dd, *J* = 8.1, 1.0 Hz, 1H), 8.00 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.81 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.62 (td, *J* = 7.5, 1.1 Hz, 1H), 7.54 (t, *J* = 7.9 Hz, 1H), 7.45 (td, *J* = 8.0, 1.6 Hz, 1H), 4.58 (s, 2H), 3.97 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 166.5, 138.7, 138.6, 137.5, 133.5, 132.7, 132.0, 131.9, 130.8, 129.3, 128.8, 119.3, 117.2, 53.0, 43.2 pmm. HRMS (ESI, *m/z*) calcd for $C_{15}H_{12}IO_2$ [M-OTf]$^+$: 350.9877, found: 350.9868.



1k

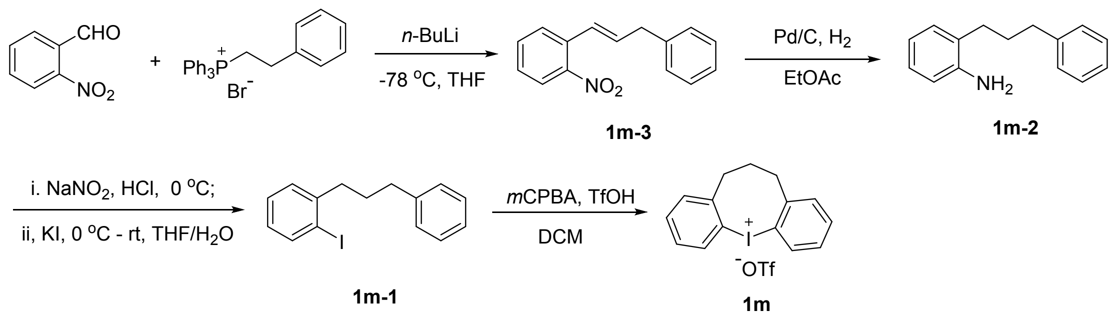
9*H*-Benzo[b]thieno[2,3-*e*]iodinin-4-ium trifluoromethanesulfonate (1k): ^{1}H NMR (400 MHz, DMSO) δ 8.08 (dd, *J* = 8.1, 0.9 Hz, 1H), 7.79 – 7.71 (m, 2H), 7.60 (td, *J* = 7.5, 1.0 Hz, 1H), 7.44 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.40 (d, *J* = 5.4 Hz, 1H), 4.44 (s, 2H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 140.6, 138.2, 133.7, 131.5, 131.3, 129.1, 128.7, 127.8, 115.4, 99.6, 36.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{11}H_8IS$ [M-OTf]$^+$: 298.93859, found: 298.93851.



1l

10, 11-Dihydrodibenzo[*b, f*]iodepin-5-ium trifluoromethanesulfonate (1l): ^{1}H NMR (400 MHz, DMSO) δ 8.04 (dd, *J* = 8.0, 0.9 Hz, 2H), 7.60 – 7.49 (m, 4H), 7.33 – 7.25 (m, 2H), 3.44 (s, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.9, 135.9, 132.8, 132.4, 128.8, 117.7, 33.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{14}H_{12}I$ [M-OTf]$^+$: 306.9978, found: 306.9981.

Procedures for the synthesis of 1m



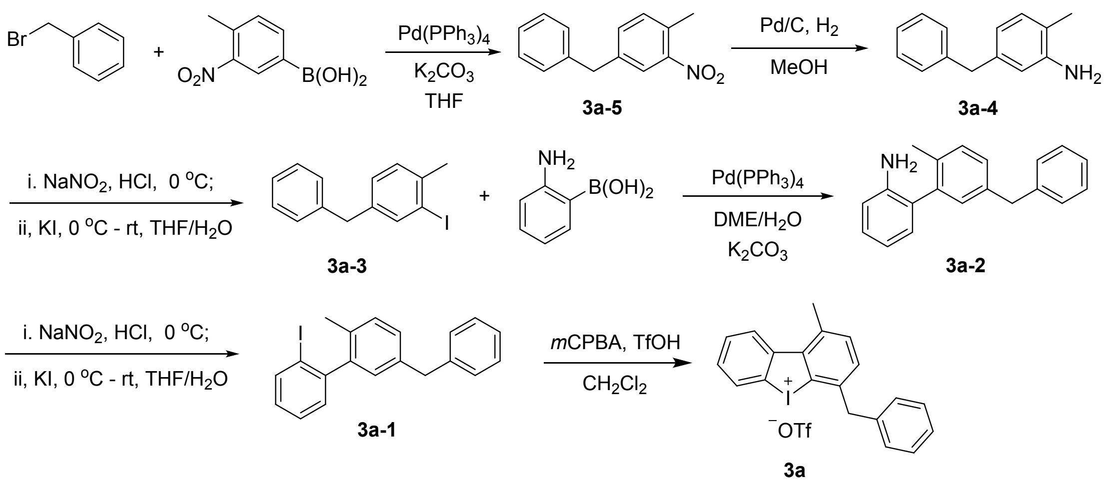
1-Nitro-2-(3-phenylprop-1-en-1-yl)benzene (1m-3): To a solution of the Wittig reagent (1.2 equiv) in dry THF was added n-BuLi (1.6 M in hexanes, 1.2 equiv) dropwise at −78 °C. After 1 h, a solution of 2-nitrobenzaldehyde (1g, 6.62 mmol, 1 equiv) in dry THF was added dropwise. The solution was stirred at −78 °C for 1 h and then at room temperature overnight. The reaction was quenched by saturated NH_4Cl solution. The aqueous phase was extracted with EtOAc (3 × 30 mL), and the combined organic layers were washed by brine, dried over anhydrous Na_2SO_4, and concentrated by rotary evaporation. The crude mixture was purified by column chromatography on a silica gel (PE/EtOAc = 20/1 -10/1) to provide **1m-3** (1.12 g, 71% yield) as a colorless oil.

2-(3-Phenylpropyl)aniline (1m-2): To a solution of **1m-3** (1.1 g, 4.6 mmol) in EtOAc (20 mL) was added Pd/C (110 mg), then this mixture was degassed and recharged by H_2 balloon. The reaction proceeded at room temperature. The reaction was completed after 4 h and filtrated by a pad of celite. The crude was concentrated by rotary evaporation and purified by column chromatography on a silica gel (PE/EtOAc = 10/1) to provide **1m-2** (953 mg, 98% yield) as a yellow oil.

1-Iodo-2-(3-phenylpropyl)benzene (1m-1): To a stirred solution of **1m-2** (950 mg, 4.5 mmol) in THF (20 mL) was added 4M aqueous HCl (11.2 mL), and the solution was cooled in an ice water bath. A solution of $NaNO_2$ (372 mg, 5.39 mmol) in H_2O (5 mL) was added dropwise. After 20 min, a solution of KI (1.87 g, 11.2 mmol) in H_2O (5 mL) was added, and stirred for 10 min in the ice water bath. Then the solution was slowly warmed up to r.t. and stirred for 1 h before 1M aqueous $Na_2S_2O_3$ was added until the color of the mixture didn't change. The phases were separated, and the aqueous phase extracted with EtOAc (20 mL x 3). The combined organic layers were washed with H_2O (5 mL x 2) and brine (5 mL x 1), dried over anhydrous Na_2SO_4, concentrated in a vacuo. The residue was purified by column chromatography on silica gel (PE/EtOAc = 50/1-20/1) to provide **1m-1** (1.16 g, 80% yield) as a yellow liquid.

6,7-Dihydro-5H-dibenzo[b,g]iodocin-12-ium trifluoromethanesulfonate (1m): To a stirred solution of **1m-1** (1.0 g, 3.1 mol) in anhydrous CH_2Cl_2 (15 mL) was added *m*-CPBA (85%, 856 mg, 4.66 mol), TfOH (0.82 mL, 9.31 mmol) at 0 °C, and 30 mins later, the solution was stirred for 1h at r.t. before CH_2Cl_2 was removed by rotary evaporation. Et_2O (15 mL) was added to the remained solid. The mixture was stirred for 30 min, and then filtered. The obtained solid was washed with Et_2O three times, dried in high vacuo to provide **1m** (1.19 g, 81% yield) as a gray solid. ^1H NMR (400 MHz, DMSO) δ 8.30 (d, J = 8.0 Hz, 2H), 7.57 – 7.45 (m, 4H), 7.22 (t, J = 7.4 Hz, 2H), 3.45 – 3.37 (m, 2H), 3.35 – 3.22 (m, 2H), 2.55 – 2.47 (m, 2H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 145.6, 135.8, 132.8, 132.0, 129.1, 124.9, 40.0, 31.1 ppm. ^{13}C-DEPT135 NMR (100 MHz, DMSO) δ 136.3, 133.3, 132.4, 129.6, 40.5, 31.5 ppm. HRMS (ESI, *m/z*) calcd for $C_{15}H_{14}I$ [M-OTf]$^+$: 321.01347, found: 321.01331.

The general procedure for the preparation of cyclic diphenyl iodonium salts 3a-3c was exemplified by the synthesis of 3a.



4-benzyl-1-methyl-2-nitrobenzene (3a-5): To a stirred solution of (4-methyl-3-nitrophenyl)boronic acid (1.90 g, 10.52 mmol) in THF (20 mL) was added (bromomethyl)benzene (1.5 g, 8.77 mmol), K_2CO_3 (4.85 g, 35.08 mmol, 2 M, 17.54 mL), $Pd(PPh_3)_4$ (203 mg, 0.17 mmol). The reaction proceeded at a reflux for 12 h under argon atmosphere before THF was removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 20/1 -10/1) to provide **3a-5** (1.72 g, 86% yield) as a yellow oil.

5-benzyl-2-methylaniline (3a-4): To a stirred solution of **3a-5** (1.7 g, 7.48 mmol) in MeOH (20 mL) was added Pd/C (170 mg), degassed and recharged by H_2. The mixture was stirred vigorously for 4h at r.t. before it was filtered through a pad of celite. The filtrate was concentrated in a vacuo to provide **3a-**

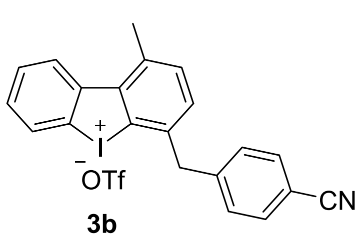
4 (1.5 g, 100% yield) as a yellow solid.

4-benzyl-2-iodo-1-methylbenzene (3a-3): To a stirred solution of **3a-4** (1.5 g, 7.6 mmol) in THF (20 mL) was added 4M aqueous HCl (19.0 mL), and the solution was cooled in an ice water bath. A solution of $NaNO_2$ (630 mg, 13.4 mmol) in H_2O (10 mL) was added dropwise. After 20 min, a solution of KI (3.16 g, 28.0 mmol) in H_2O (10 mL) was added, and stirred for 10 min in the ice water bath. Then the solution was slowly warmed up to r.t. and stirred for 1 h before 1M aqueous $Na_2S_2O_3$ was added until the color of the mixture didn't change. The phases were separated, and the aqueous phase extracted with EtOAc (25 mL x 3). The combined organic layers were washed with H_2O (5 mL x 2) and brine (5 mL x 1), dried over anhydrous Na_2SO_4, concentrated in a vacuo. The residue was purified by column chromatography on silica gel (PE/EtOAc = 40/1-20/1) to provide **3a-3** (1.95 g, 83% yield) as a yellow liquid.

5'-benzyl-2'-methyl-[1,1'-biphenyl]-2-amine (3a-2): To a stirred solution of (2-aminophenyl)boronic acid (1.01 g, 7.40 mmol) in DME (20 mL) and H_2O (20 mL) was added **3a-3** (1.9 g, 6.17 mmol), K_2CO_3 (3.41 g, 24.66 mmol), $Pd(PPh_3)_4$ (143 mg, 0.12 mmol). The reaction proceeded at a reflux for 12 h under argon atmosphere before THF was removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 20/1 -10/1) to provide **3a-2** (1.64 g, 97% yield) as a yellow oil.

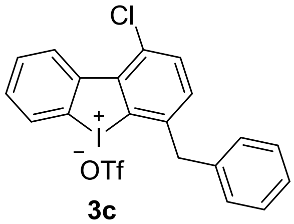
5-benzyl-2'-iodo-2-methyl-1, 1'-biphenyl (3a-1): To a stirred solution of **3a-2** (1.6 g, 5.85 mmol) in THF (20 mL) was added 4M aqueous HCl (14.6 mL), and the solution was cooled in an ice water bath. A solution of $NaNO_2$ (485 mg, 7.02 mmol) in H_2O (10 mL) was added dropwise. After 20 min, a solution of KI (2.43 g, 14.63 mmol) in H_2O (10 mL) was added, and stirred for 10 min in the ice water bath. Then the solution was slowly warmed up to r.t. and stirred for 1 h before 1M aqueous $Na_2S_2O_3$ was added until the color of the mixture didn't change. The phases were separated, and the aqueous phase extracted with EtOAc (25 mL x 3). The combined organic layers were washed with H_2O (5 mL x 2) and brine (5 mL x 1), dried over anhydrous Na_2SO_4, concentrated in a vacuo. The residue was purified by column chromatography on silica gel (PE/EtOAc = 40/1-20/1) to provide **3a-1** (1.7 g, 76% yield) as a yellow solid.

4-Benzyl-1-methyldibenzo[*b, d*]iodol-5-ium trifluoromethanesulfonate (3a): To a stirred solution of **3a-1** (1.7 g, 4.42 mol) in anhydrous CH_2Cl_2 (25 mL) was added *m*-CPBA (85%, 1.35 g, 6.64 mol), TfOH (1.17 mL, 13.27 mmol) at 0 °C, and 30 mins later, the solution was stirred for 1h at r.t. before CH_2Cl_2 was removed by rotary evaporation. Et_2O (20 mL) was added to the remained solid. The mixture was stirred for 30 min, and then filtered. The obtained solid was washed with Et_2O three times, dried in high vacuo to provide **3a** (1.96 g, 83% yield) as a white solid. ^{1}H NMR (400 MHz, DMSO) δ 8.55 (d, *J* = 7.5 Hz, 1H), 8.39 (dd, *J* = 8.3, 0.9 Hz, 1H), 7.91 (t, *J* = 7.2 Hz, 1H), 7.76 – 7.71 (m, 1H), 7.71 – 7.66 (m, 1H), 7.45 – 7.30 (m, 6H), 4.40 (s, 2H), 2.87 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 143.2, 139.8, 139.8, 137.9, 137.8, 135.1, 131.2, 131.0, 130.9, 130.3, 130.2, 129.6, 129.1, 127.5, 126.1, 120.5, 43.9, 22.7 ppm. HRMS (ESI, *m/z*) calcd for $C_{20}H_{16}I$ $[M\text{-}OTf]^+$: 383.02912, found: 383.02890.



3b

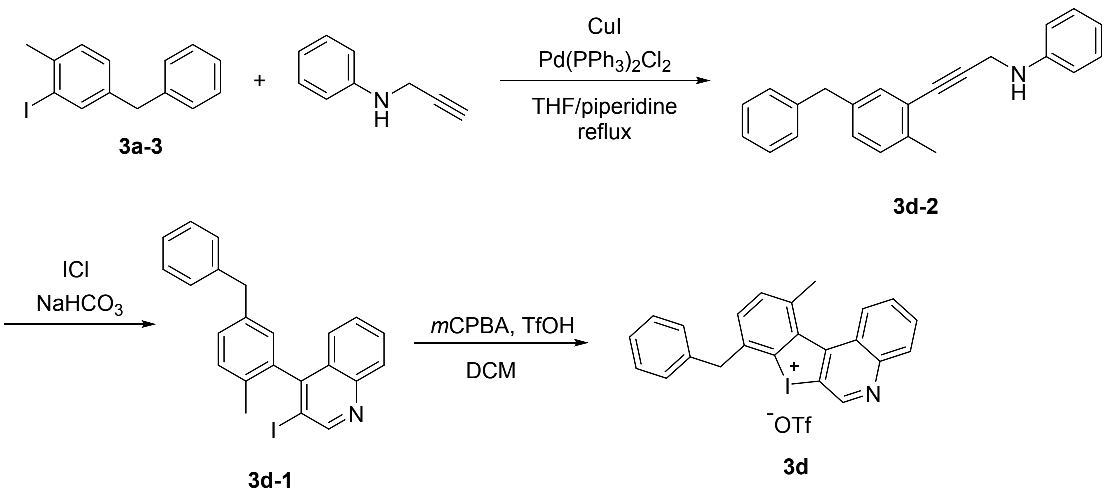
4-(4-Cyanobenzyl)-1-methyldibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (3b): ^{1}H NMR (400 MHz, DMSO) δ 8.55 (d, *J* = 8.1 Hz, 1H), 8.43 (d, *J* = 8.3 Hz, 1H), 7.92 (t, *J* = 7.6 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 2H), 7.75 (t, *J* = 7.7 Hz, 1H), 7.69 (d, *J* = 7.7 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 7.7 Hz, 1H), 4.50 (s, 2H), 2.88 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 144.1, 143.5, 140.0, 138.9, 138.3, 135.2, 132.7, 131.3, 131.0, 131.0, 130.4, 130.2, 130.0, 126.8, 120.6, 118.8, 109.9, 44.1, 22.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{21}H_{15}IN$ $[M\text{-}OTf]^+$: 408.0244, found: 408.0247.



3c

4-Benzyl-1-chlorodibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (3c): ^{1}H NMR (400 MHz, DMSO) δ 9.19 (d, *J* = 7.6 Hz, 1H), 8.41 (d, *J* = 8.1 Hz, 1H), 7.95 (d, *J* = 7.8 Hz, 2H), 7.82 (t, *J* = 6.8 Hz, 1H), 7.38 (dd, *J* = 24.8, 7.8 Hz, 6H), 4.43 (s, 2H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 141.6, 140.9, 137.6, 137.5, 134.3, 131.7, 131.2, 130.9, 130.8, 130.8, 130.8, 129.6, 129.1, 127.6, 127.2, 121.0, 99.5, 43.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{19}H_{13}ClI$ $[M\text{-}OTf]^+$: 402.9745, found: 402.9740.

Procedures for the synthesis of 3d

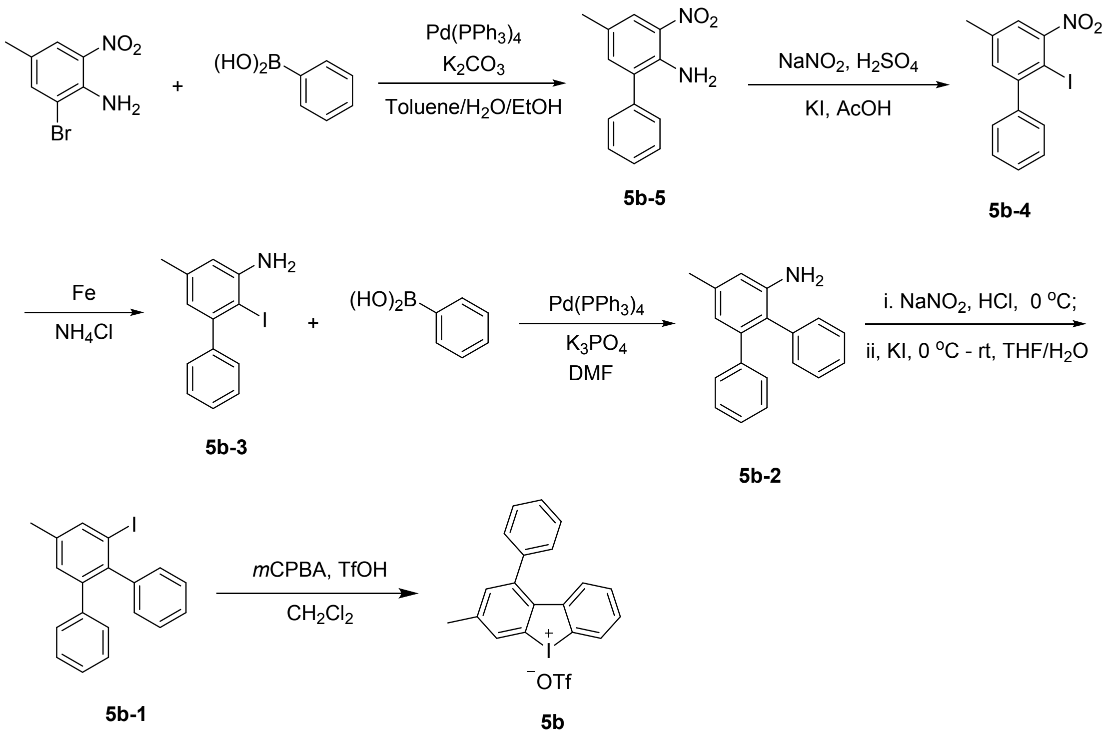


_N_-(3-(5-Benzyl-2-methylphenyl)prop-2-yn-1-yl)aniline (3d-2): To a stirred solution of 3a-3 (800 mg, 2.6 mmol, 1.0 equiv) in THF (5 mL) and piperidine (5 mL) was added **3a-3** (409 g, 3.12 mmol, 1.2 equiv), $Pd(PPh_3)_2Cl_2$ (91 mg, 0.05 equiv), CuI (25 mg, 0.05 equiv). The reaction proceeded at a reflux for 12 h under argon atmosphere before the solvents were removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 30/1 -15/1) to provide **3d-2** (760 mg, 94% yield) as a yellow oil.

4-(5-Benzyl-2-methylphenyl)-3-iodoquinoline (3d-1): To a stirred solution of **3d-2** (750 mg, 2.41 mmol, 1.0 equiv) in MeCN (6 mL) was added $NaHCO_3$ (405 mg, 2.0 equiv), a solutions of ICl (782 mg, 2.0 equiv) in MeCN (4 mL). The reaction proceeded at rt for 2 h. The remained mixture was extracted with EtOAc, the combined organic layers were washed with 1M aqueous $Na_2S_2O_3$ and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 30/1 -10/1) to provide **3d-1** (852 mg, 81% yield) as a yellow oil.

8-Benzyl-11-methylbenzo[4,5]iodolo[2,3-c]quinolin-7-ium trifluoromethanesulfonate (3d): o a stirred solution of **3d-1** (500 mg, 1.15 mol) in anhydrous CH_2Cl_2 (10 mL) was added *m*-CPBA (85%, 350 mg, 1.72 mol), TfOH (0.3 mL, 3.45 mmol) at 0 ^{o}C, and 30 mins later, the solution was stirred for 1h at r.t. before CH_2Cl_2 was removed by rotary evaporation. Et_2O (10 mL) was added to the remained solid. The mixture was stirred for 30 min, and then filtered. The obtained solid was washed with Et_2O three times, dried in high vacuo to provide **3d** (460 mg, 69% yield) as a yellow solid. ^{1}H NMR (400 MHz, DMSO) δ 9.55 (s, 1H), 8.27 (d, J = 7.9 Hz, 1H), 8.14 (d, J = 8.1 Hz, 1H), 8.04 – 7.97 (m, 1H), 7.87 – 7.78 (m, 2H), 7.59 (d, J = 7.8 Hz, 1H), 7.45 – 7.40 (m, 4H), 7.38 – 7.33 (m, 1H), 4.47 (s, 2H), 2.55 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 148.0, 147.9, 147.6, 140.6, 140.1, 139.7, 137.9, 135.2, 132.0, 131.3, 129.7, 129.2, 128.1, 127.6, 127.1, 126.5, 122.3, 119.1, 116.2, 43.7, 24.0 ppm. HRMS (ESI, *m/z*) calcd for $C_{23}H_{17}NI$ $[M-OTf]^+$: 434.04002, found: 434.04001.

The general procedure for the preparation of cyclic diphenyl iodonium salts 5a-5i was exemplified by the synthesis of 5b.



5-methyl-3-nitro-[1,1'-biphenyl]-2-amine (5b-5): To a stirred solution of phenylboronic acid (2.53 g, 20.77 mmol) in toluene (30 mL), H_2O (20 mL), EtOH (10 mL) was added 2-bromo-4-methyl-6-nitroaniline (4.0 g, 17.31 mmol), K_2CO_3 (9.57 g, 69.25 mmol), $Pd(PPh_3)_4$ (400 mg, 0.346 mmol). The reaction proceeded at 100 °C for 12 h under argon atmosphere before solvent was removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 20/1 -10/1) to provide **5b-5** (3.89 g, 98% yield) as a orange oil.

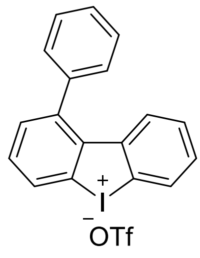
2-iodo-5-methyl-3-nitro-1,1'-biphenyl (5b-4): To $NaNO_2$ (2.12 g, 30.67 mmol) was added concentrated H_2SO_4 slowly until all the solid dissolved at room temperature, and this mixture was added dropwise into a mixture of **5b-5** (3.5 g, 15.33 mmol) in AcOH (30 mL), at room temperature. 20 mins after the addition of $NaNO_2$ mixture was finished, a solution of KI (6.36 g, 38.33 mmol) in H_2O (10 mL) was added slowly, at room temperature. The reaction process was tracked by TLC. After the reaction was finished, the remained mixture was extracted with EtOAc, the combined organic layers were washed with 1M aqueous $Na_2S_2O_3$ and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 40/1 -20/1) to provide **5b-4** (4.36 g, 84%yield) as yellow solid.

2-iodo-5-methyl-[1,1'-biphenyl]-3-amine (5b-3): To **5b-4** (4.3 g, 12.68 mmol) in EtOH (20 mL) and H_2O (20 mL) was added NH_4Cl (1.42 g, 61.92 mmol), iron powder (2.83 g, 50.72 mmol), 60 ^oC, 3 h. After the reaction was finished, the mixture was filtered by a pad of celite, and the filtrate was concentrated by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 20/1 -10/1) to yield **5b-3** (3.58 g, 91% yield) as a yellow liquid.

5'-methyl-[1,1':2',1''-terphenyl]-3'-amine (5b-2): To a stirred solution of phenylboronic acid (1.66 g, 13.59 mmol) in DMF (20 mL), H_2O (20 mL), EtOH (10 mL) was added **5b-3** (3.5 g, 11.32 mmol), K_3PO_4 (6.01 g, 28.30 mmol), $Pd(PPh_3)_4$ (262 mg, 0.226 mmol). The reaction proceeded at 100 ^oC for 12 h under argon atmosphere before solvent was removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 20/1 -10/1) to provide **5b-2** (2.83 g, 96% yield) as an orange oil.

3'-iodo-5'-methyl-1,1':2',1''-terphenyl (5b-1): To a stirred solution of **5b-2** (2.8 g, 10.8 mmol) in THF (25 mL) was added 4M aqueous HCl (27.0 mL), and the solution was cooled in an ice water bath. A solution of $NaNO_2$ (894 mg, 12.96 mmol) in H_2O (10 mL) was added dropwise. After 20 min, a solution of KI (4.48 g, 26.99 mmol) in H_2O (10 mL) was added, and stirred for 10 min in the ice water bath. Then the solution was slowly warmed up to r.t. and stirred for 1 h before 1M aqueous $Na_2S_2O_3$ was added until the color of the mixture didn't change. The phases were separated, and the aqueous phase extracted with EtOAc (25 mL x 3). The combined organic layers were washed with H_2O (5 mL x 2) and brine (5 mL x 1), dried over anhydrous Na_2SO_4, concentrated in a vacuo. The residue was purified by column chromatography on silica gel (PE/EtOAc = 40/1-20/1) to provide **5b-1** (3.55 g, 89% yield) as a yellow liquid.

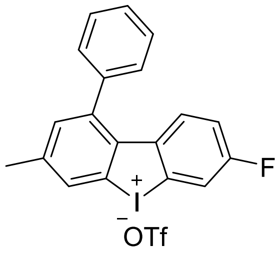
3-Methyl-1-phenyldibenzo[*b, d*]iodol-5-ium trifluoromethanesulfonate (5b): To a stirred solution of **5b-1** (3.0 g, 8.1 mol) in anhydrous CH_2Cl_2 (40 mL) was added *m*-CPBA (85%, 2.47 g, 12.15 mol), TfOH (2.15 mL, 24.31 mmol) at 0 °C, and 30 mins later, the solution was stirred for 1h at r.t. before CH_2Cl_2 was removed by rotary evaporation. Et_2O (20 mL) was added to the remained solid. The mixture was stirred for 30 min, and then filtered. The obtained solid was washed with Et_2O three times, dried in high vacuo to provide **5b** (3.68 g, 88% yield) as a white solid. ^{1}H NMR (400 MHz, DMSO) δ 8.20 (dd, J = 8.2, 0.8 Hz, 1H), 8.10 (d, J = 0.6 Hz, 1H), 7.62 – 7.56 (m, 3H), 7.54 (dd, J = 11.3, 4.1 Hz, 1H), 7.46 (d, J = 0.8 Hz, 1H), 7.42 – 7.35 (m, 3H), 6.83 (dd, J = 8.2, 1.1 Hz, 1H), 2.50 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 143.1, 141.2, 140.1, 139.4, 135.8, 134.5, 130.8, 130.2, 129.8, 129.7, 129.4, 129.2, 128.7, 128.5, 122.6, 121.0, 20.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{19}H_{14}I$ [M-OTf]$^{+}$: 369.0135, found:369.0133.



5a

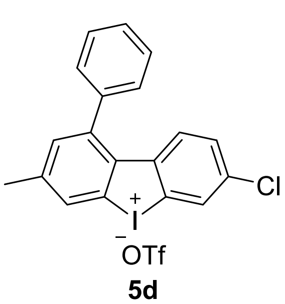
1-Phenyldibenzo[*b, d*]iodol-5-ium trifluoromethanesulfonate (5a):

^{1}H NMR (400 MHz, DMSO) δ 8.33 (d, J = 8.2 Hz, 1H), 8.24 (d, J = 8.2 Hz, 1H), 7.73 (t, J = 7.7 Hz, 1H), 7.68 – 7.53 (m, 5H), 7.41 (s, 3H), 6.89 (d, J = 8.2 Hz, 1H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 143.5, 141.7, 139.5, 138.4, 133.6, 130.9, 130.6, 130.0, 129.7, 129.7, 129.6, 129.5, 128.8, 128.5, 122.6, 122.0, 121.3, 119.4 ppm. HRMS (ESI, *m/z*) calcd for $C_{18}H_{12}I$ [M-OTf]$^{+}$: 354.99782, found: 354.99768.



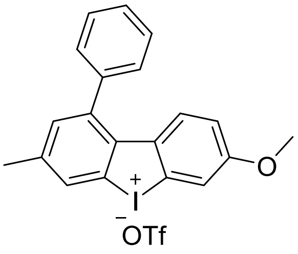
5c

7-Fluoro-3-methyl-1-phenyldibenzo[*b, d*]iodol-5-ium trifluoromethanesulfonate (5c): ^{1}H NMR (400 MHz, DMSO) δ 8.08 (s, 1H), 8.01 (dd, J = 7.7, 2.7 Hz, 1H), 7.63 – 7.54 (m, 3H), 7.46 (s, 1H), 7.37 (ddd, J = 13.2, 7.0, 2.9 Hz, 3H), 6.80 (dd, J = 9.1, 5.2 Hz, 1H), 2.50 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 161.1 (d, J = 253.1 Hz), 142.9, 140.18 (s), 139.19 (s), 138.54 (d, J = 2.8 Hz), 134.75 (d, J = 4.3 Hz), 130.3 (d, J = 8.1 Hz), 129.8, 129.6, 129.0, 128.6, 123.1, 122.4, 121.6 (d, J = 9.4 Hz), 119.2, 118.0 (d, J = 15.6 Hz), 117.8 (d, J = 10.8 Hz), 20.9 ppm. HRMS (ESI, *m/z*) calcd for $C_{19}H_{13}FI$ [M-OTf]$^{+}$: 387.0041, found: 387.0033.



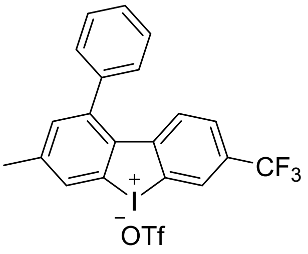
5d

7-Chloro-3-methyl-1-phenyldibenzo[*b, d*]iodol-5-ium trifluoromethanesulfonate (5d): ^{1}H NMR (400 MHz, DMSO) δ 8.21 (d, J = 2.2 Hz, 1H), 8.09 (d, J = 0.8 Hz, 1H), 7.62 – 7.56 (m, 3H), 7.53 (dd, J = 8.9, 2.2 Hz, 1H), 7.48 (d, J = 0.9 Hz, 1H), 7.41 – 7.35 (m, 2H), 6.77 (d, *J* = 8.8 Hz, 1H), 2.51 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 143.2, 140.8, 140.7, 139.1, 134.8, 133.8, 130.1, 129.9, 129.8, 129.6, 129.0, 128.5, 123.2, 121.8, 20.9 ppm. HRMS (ESI, *m/z*) calcd for $C_{19}H_{13}ClI$ [M-OTf]$^+$: 402.97450, found: 402.97458.



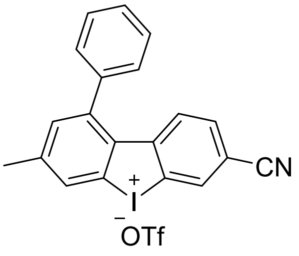
5e

7-Methoxy-3-methyl-1-phenyldibenzo[b, d]iodol-5-ium trifluoromethanesulfonate (5e): ^{1}H NMR (400 MHz, DMSO) δ 8.05 (s, 1H), 7.74 (t, *J* = 8.5 Hz, 1H), 7.56 (t, *J* = 7.0 Hz, 3H), 7.42 (s, 1H), 7.37 (d, *J* = 3.6 Hz, 2H), 7.01 (dd, *J* = 9.1, 2.5 Hz, 1H), 6.69 (d, *J* = 9.1 Hz, 1H), 3.81 (s, 3H), 2.48 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 159.8, 142.0, 139.4, 138.9, 135.6, 134.4, 134.1, 132.8, 130.7, 129.7, 129.4, 128.9, 128.7, 128.5, 128.0, 122.1, 121.9, 116.5, 115.3, 56.0, 20.7 ppm. HRMS (ESI, *m/z*) calcd for $C_{20}H_{16}IO$ [M-OTf]$^+$: 399.0240, found: 399.0242.



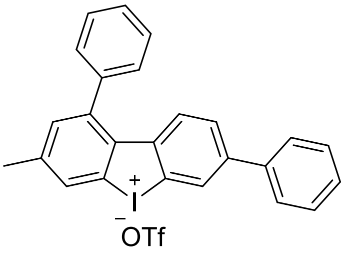
5f

3-Methyl-1-phenyl-7-(trifluoromethyl)dibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (5f): ^{1}H NMR (400 MHz, DMSO) δ 8.55 (s, 1H), 8.15 (s, 1H), 7.85 (d, *J* = 8.6 Hz, 1H), 7.65 – 7.57 (m, 3H), 7.54 (s, 1H), 7.42 (dd, *J* = 6.5, 2.9 Hz, 2H), 7.01 (d, *J* = 8.6 Hz, 1H), 2.54 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 145.6, 143.9, 141.5, 139.0, 134.8, 134.5, 129.8, 129.5, 129.4, 129.0, 128.4, 127.5 (q, *J* = 4.4 Hz), 126.7 (q, *J* = 3.2 Hz), 124.0, 121.6, 99.6, 20.9 ppm. HRMS (ESI, *m/z*) calcd for $C_{20}H_{13}F_3I$ [M-OTf]$^+$: 437.00085, found: 437.00061.



5g

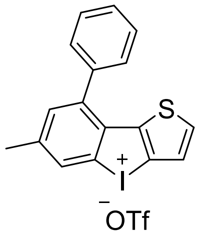
7-Cyano-3-methyl-1-phenyldibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (5g): ^{1}H NMR (400 MHz, DMSO) δ 8.52 (d, *J* = 1.2 Hz, 1H), 8.13 (s, 1H), 7.88 (dd, *J* = 8.6, 1.3 Hz, 1H), 7.64 – 7.56 (m, 3H), 7.53 (s, 1H), 7.44 – 7.35 (m, 2H), 6.91 (d, *J* = 8.6 Hz, 1H), 2.53 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 145.9, 144.0, 141.9, 138.9, 134.8, 134.4, 134.2, 133.2, 129.8, 129.6, 129.0, 129.0, 128.4, 124.4, 121.2, 117.3, 111.5, 21.0 ppm. HRMS (ESI, *m/z*) calcd for $C_{20}H_{13}IN$ [M-OTf]$^+$: 394.00872, found: 394.00858.



5h

3-Methyl-1,7-diphenyldibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (5h): ^{1}H NMR (400 MHz, DMSO) δ

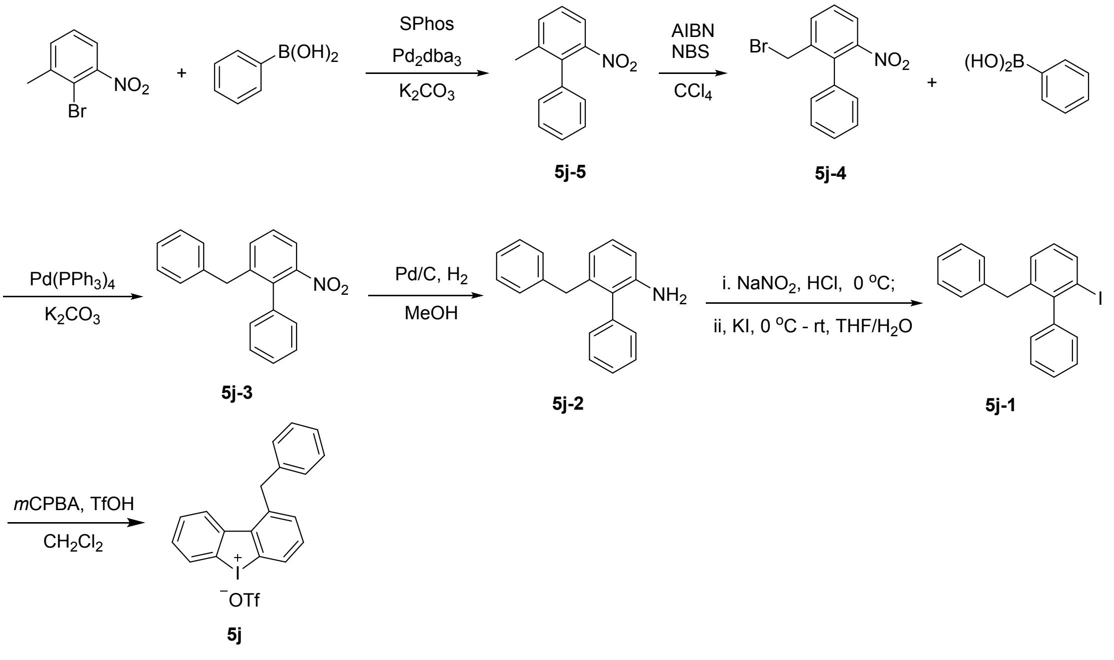
8.48 (d, J = 1.7 Hz, 1H), 8.10 (s, 1H), 7.73 (dd, J = 8.6, 1.7 Hz, 1H), 7.68 (d, J = 7.3 Hz, 2H), 7.64 – 7.56 (m, 3H), 7.50 (dd, J = 14.2, 6.4 Hz, 3H), 7.46 – 7.38 (m, 3H), 6.88 (d, J = 8.6 Hz, 1H), 2.51 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 143.0, 141.4, 140.5, 140.2, 139.4, 137.5, 135.4, 134.6, 129.8, 129.4, 129.3, 128.8, 128.8, 128.5, 128.1, 127.9, 126.6, 122.8, 122.0, 20.8 ppm. HRMS (ESI, *m/z*) calcd for $C_{25}H_{18}I$ [M-OTf]$^+$: 445.04477, found: 445.04312.



5i

6-Methyl-8-phenylbenzo[*b*]thieno[2, 3-*d*]iodol-4-ium trifluoromethanesulfonate (5i): ^{1}H NMR (400 MHz, DMSO) δ 8.09 (s, 1H), 7.66 (d, J = 5.4 Hz, 1H), 7.61 – 7.55 (m, 3H), 7.46 (s, 1H), 7.45 (d, J = 5.4 Hz, 1H), 7.40 (dd, J = 6.2, 2.8 Hz, 2H), 2.50 (s, 3H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 144.4, 140.7, 139.1, 138.0, 132.8, 132.5, 131.0, 130.0, 129.2, 129.1, 129.1, 125.8, 125.5, 122.0, 119.4, 111.2, 20.9 ppm. HRMS (ESI, *m/z*) calcd for $C_{17}H_{12}IS$ [M-OTf]$^+$: 374.96989, found: 374.96988.

The general procedure for the preparation of cyclic diphenyl iodonium salts 5j-5l was exemplified by the synthesis of 5j.



2-Methyl-6-nitro-1,1'-biphenyl (5j-5): To a stirred solution of phenylboronic acid (1.69 g, 13.89 mmol) in toluene (15 mL), H_2O (10 mL), EtOH (5 mL) was added 2-bromo-1-methyl-3-nitrobenzene (2.0 g, 9.26 mmol), K_2CO_3 (5.12 g, 37.03 mmol), Pd$_2$dba$_3$ (84.78 mg, 92.58 μmol), SPhos (152.03 mg, 370.31μmol). The reaction proceeded at 100 °C for 24 h under argon atmosphere before solvent was removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 30/1 -15/1) to provide **5j-5** (1.83 g, 93% yield) as a white solid.

2-(bromomethyl)-6-nitro-1, 1'-biphenyl (5j-4): to a solution of **5j-5** (1.53 g , 7.18 mmol) in CCl_4 (15 mL) was added AIBN (117.82 mg , 717.51 μmol), NBS (1.4 g, 7.89 mmol). The reaction proceeded at a reflux for 6h, and the reaction was monitored by TLC plate. When the reaction was finished, the solvent was removed by rotary evaporation, and the residue was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 30/1 -15/1) to yield **5j-4** (1.79 g, 85% yield) as a yellow lsolid.

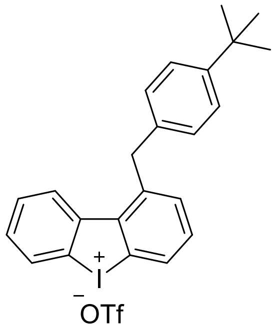
2-benzyl-6-nitro-1, 1'-biphenyl (5j-3): To a stirred solution of phenylboronic acid (851.45 mg, 6.98 mmol) in toluene (15 mL), H_2O (10 mL), EtOH (5 mL) was added **5j-4** (1.70 g, 5.82 mmol), K_2CO_3 (3.22 g, 23.28 mmol, 2M, 11.64 mL H_2O), $Pd(PPh_3)_4$ (201.74 mg, 174.58 μmol). The reaction proceeded at 100 ^oC for 12 h under argon atmosphere before solvent was removed by rotary evaporation. The remained mixture was extracted with EtOAc, the combined organic layers were washed with H_2O and brine and dried over anhydrous Na_2SO_4, evaporated in a vacuo. The residue was purified by column chromatography on a silica gel (PE/EtOAc = 30/1 -15/1) to provide **5j-3** (1.65 g, 98% yield) as a white solid.

6-benzyl-[1, 1'-biphenyl]-2-amine (5j-2): To a stirred solution of **5j-3** (1.65 g, 5.70 mmol) in MeOH (20 mL) was added Pd/C (165 mg), degassed and recharged by H_2. The mixture was stirred vigorously for 4h at r.t. before it was filtered through a pad of celite. The filtrate was concentrated in a vacuo to provide **5j-2** (1.27 g, 86% yield) as a white solid.

2-benzyl-6-iodo-1, 1'-biphenyl (5j-1):

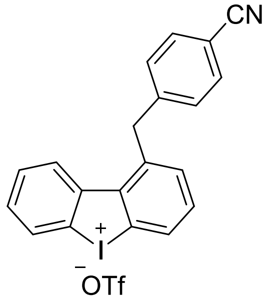
To a stirred solution of **5j-2** (1.2 g, 4.63 mmol) in THF (15 mL) was added 4M aqueous HCl (11.57 mL), and the solution was cooled in an ice water bath. A solution of $NaNO_2$ (383.08 mg, 5.55 mmol) in H_2O (5 mL) was added dropwise. After 20 min, a solution of KI (1.92 g, 11.57 mmol) in H_2O (5 mL) was added, and stirred for 10 min in the ice water bath. Then the solution was slowly warmed up to r.t. and stirred for 1 h before 1M aqueous $Na_2S_2O_3$ was added until the color of the mixture didn't change. The phases were separated, and the aqueous phase extracted with EtOAc (20 mL x 3). The combined organic layers were washed with H_2O (5 mL x 2) and brine (5 mL x 1), dried over anhydrous Na_2SO_4, concentrated in a vacuo. The residue was purified by column chromatography on silica gel (PE/EtOAc = 40/1-20/1) to provide **5j-1** (1.49 g, 87% yield) as a colorless oil.

1-Benzyldibenzo[b, d]iodol-5-ium trifluoromethanesulfonate (5j): To a stirred solution of **5j-1** (1.45 g, 3.92 mol) in anhydrous CH_2Cl_2 (20 mL) was added *m*-CPBA (85%, 1.19 g, 5.87 mol), TfOH (1.04 mL, 11.75 mmol) at 0 °C, and 30 mins later, the solution was stirred for 1h at r.t. before CH_2Cl_2 was removed by rotary evaporation. Et_2O (20 mL) was added to the remained solid. The mixture was stirred for 30 min, and then filtered. The obtained solid was washed with Et_2O three times, dried in high vacuo to provide **5j** (1.82 g, 90% yield) as a white solid. ^{1}H NMR (400 MHz, DMSO) δ 8.35 – 8.24 (m, 3H), 7.76 – 7.62 (m, 4H), 7.31 (t, *J* = 7.4 Hz, 2H), 7.23 (d, *J* = 7.3 Hz, 1H), 7.15 (d, *J* = 7.2 Hz, 2H), 4.73 (s, 2H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 141.9, 141.6, 139.8, 138.6, 135.1, 130.9, 130.6, 130.6, 130.5, 130.2, 129.4, 128.9, 128.4, 126.7, 122.7, 120.6, 40.2 ppm. HRMS (ESI, *m/z*) calcd for $C_{19}H_{14}I$ [M-OTf]$^+$: 369.01347, found: 369.01334.



5k

1-(4-(*tert*-Butyl)benzyl)dibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (5k): ^{1}H NMR (400 MHz, DMSO) δ 8.35 (d, *J* = 8.1 Hz, 1H), 8.27 (dd, *J* = 12.7, 7.7 Hz, 2H), 7.74 (t, *J* = 7.4 Hz, 1H), 7.65 (t, *J* = 7.2 Hz, 3H), 7.32 (d, *J* = 7.6 Hz, 2H), 7.07 (d, *J* = 7.7 Hz, 2H), 4.67 (s, 2H), 1.23 (s, 9H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 149.0, 142.0, 141.8, 139.7, 135.5, 135.0, 130.9, 130.7, 130.5, 130.4, 130.2, 129.4, 128.2, 125.6, 122.6, 122.1, 120.6, 119.5, 40.0, 34.2, 31.2 ppm. HRMS (ESI, *m/z*) calcd for $C_{23}H_{22}I$ [M-OTf]$^+$: 425.07607, found: 425.07587.



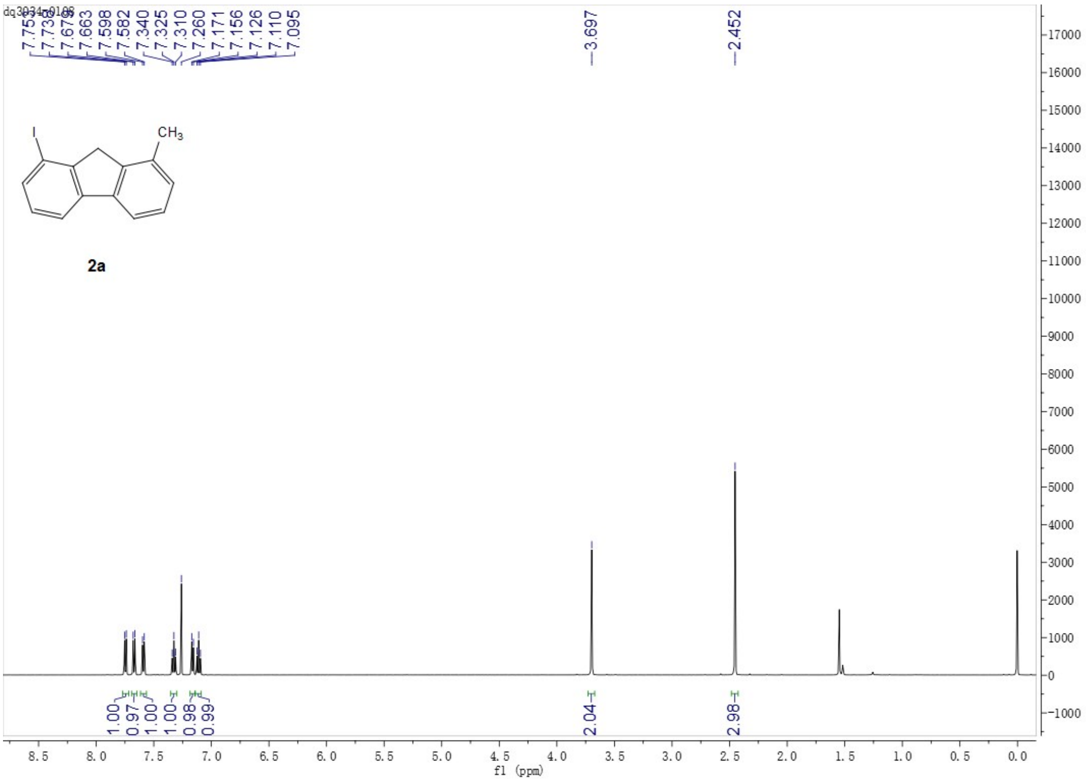
5l

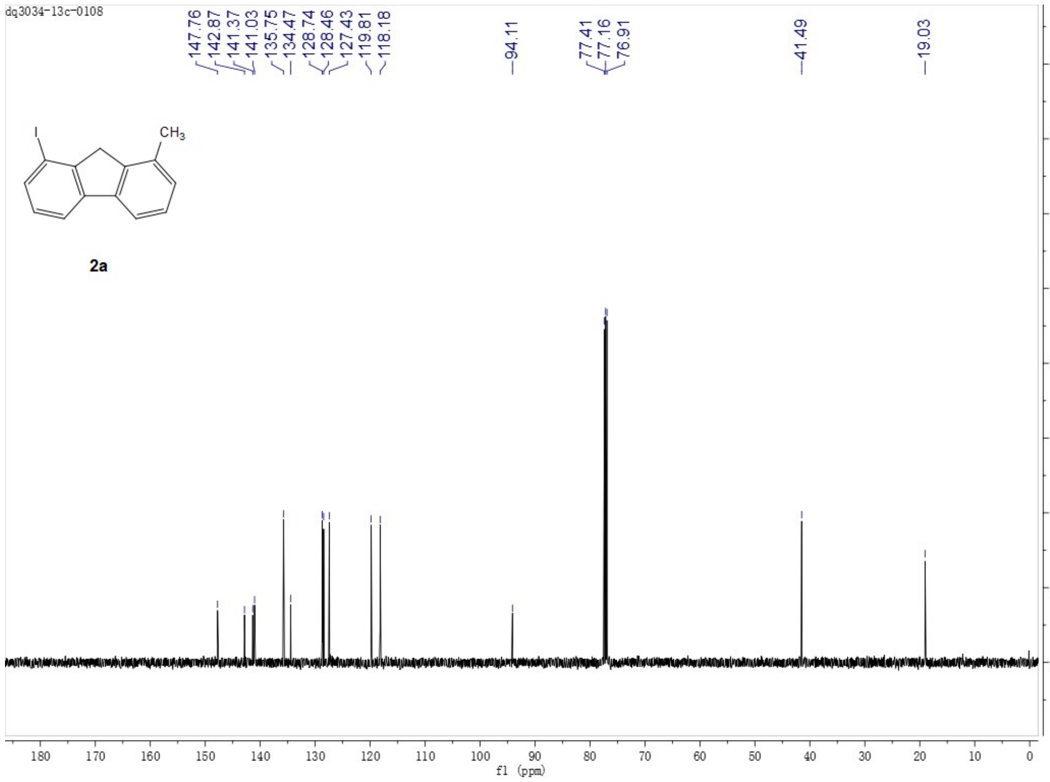
1-(4-Cyanobenzyl)dibenzo[*b*, *d*]iodol-5-ium trifluoromethanesulfonate (5l): ^{1}H NMR (400 MHz, DMSO) δ 8.28 (d, *J* = 6.4 Hz, 2H), 8.22 (d, *J* = 7.9 Hz, 1H), 7.78 (d, *J* = 7.4 Hz, 2H), 7.73 – 7.63 (m, 4H), 7.34 (d, *J* = 7.5 Hz, 2H), 4.84 (s, 2H) ppm. ^{13}C NMR (100 MHz, DMSO) δ 144.8, 141.8, 140.2, 139.9, 135.3, 132.8, 131.0, 130.8, 130.6, 130.4, 130.4, 129.9, 129.6, 122.9, 122.4, 120.7, 119.2, 119.0, 109.6, 40.2 ppm. HRMS (ESI, *m/z*) calcd for $C_{20}H_{13}IN$ [M-OTf]$^+$: 394.00872, found: 394.00858.

References

[1] C. Zhu, D. Wang, D. Wang, Y. Zhao, W. Sun, and Z. Shi. *Angew. Chem. Int. Ed*. **2018**, *57*, 8848-8853

[2] D. Zhu, M. Li, Z. Wu, Y. Du, B. Luo, P. Huang, S. Wen. *Eur. J. Org. Chem*. **2019**, 4566–4571.





2a

dq3034-13c-0108
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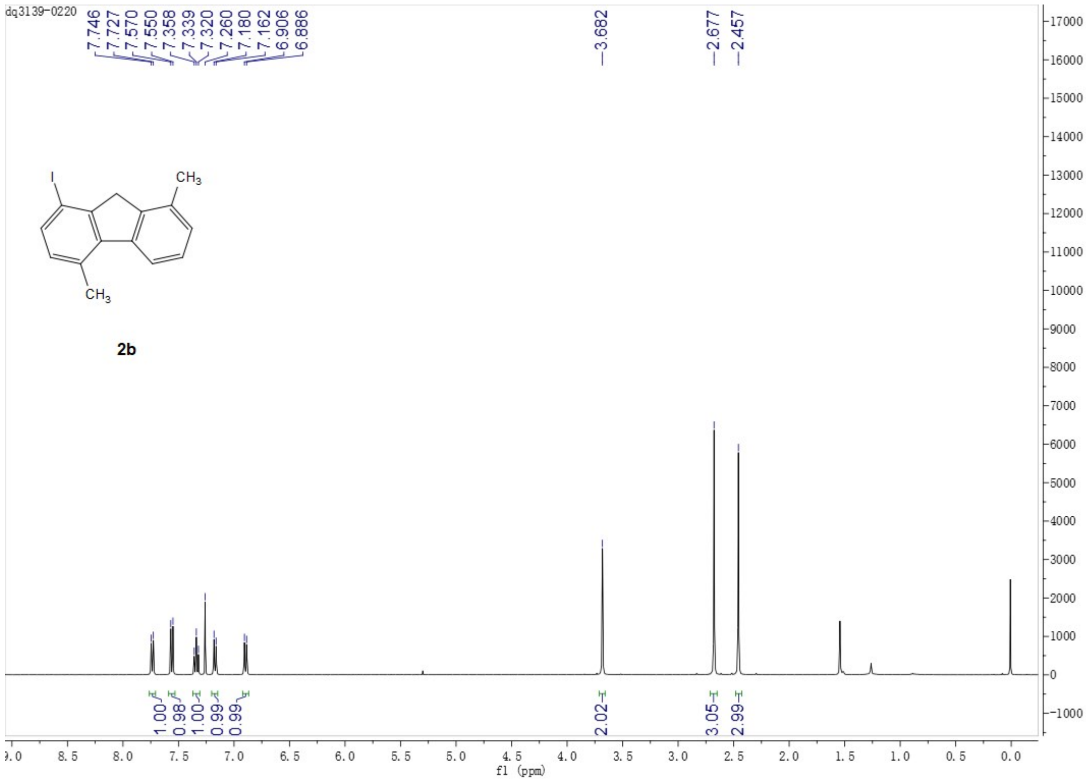
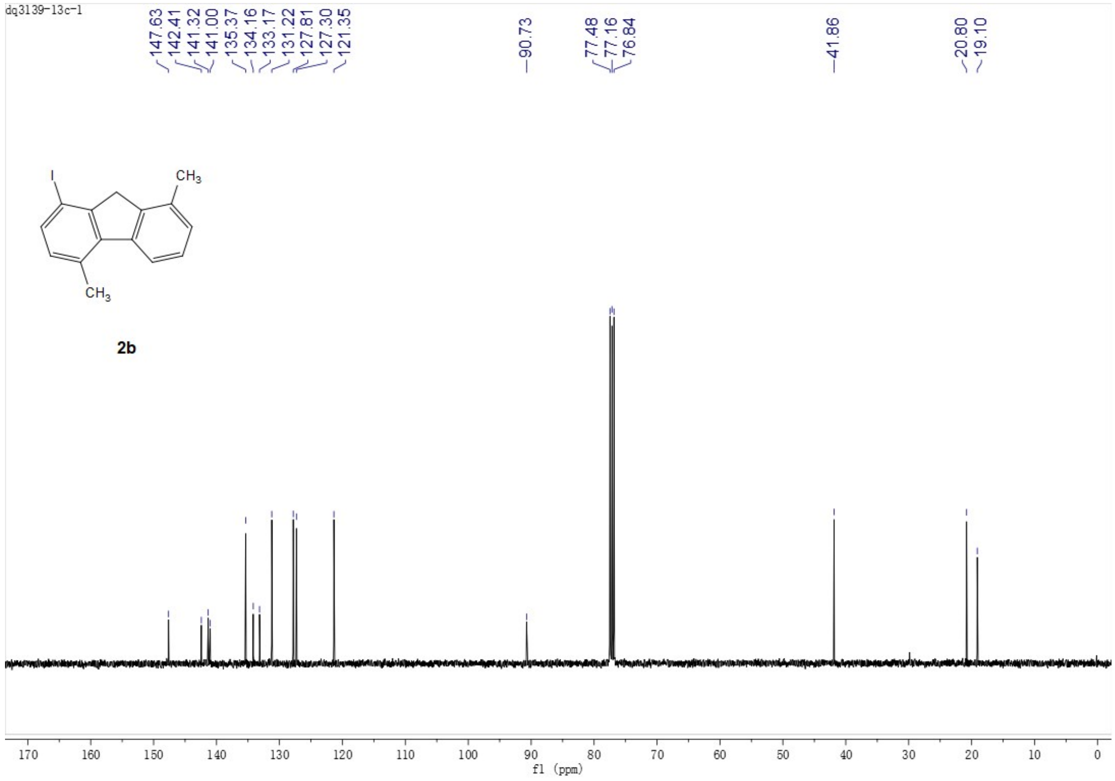
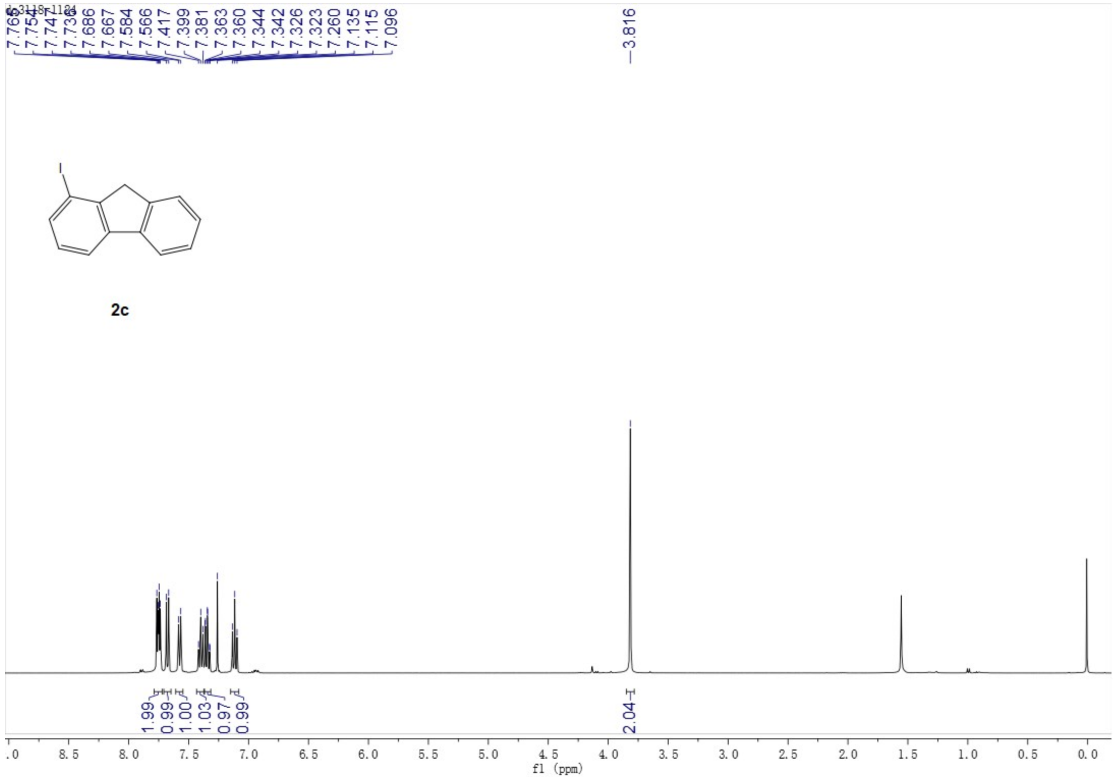
127.43
119.81
118.18
94.11
77.41
77.16
76.91
41.49
19.03
I
CH3
400
350
300
250
200
150
100
50
0
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

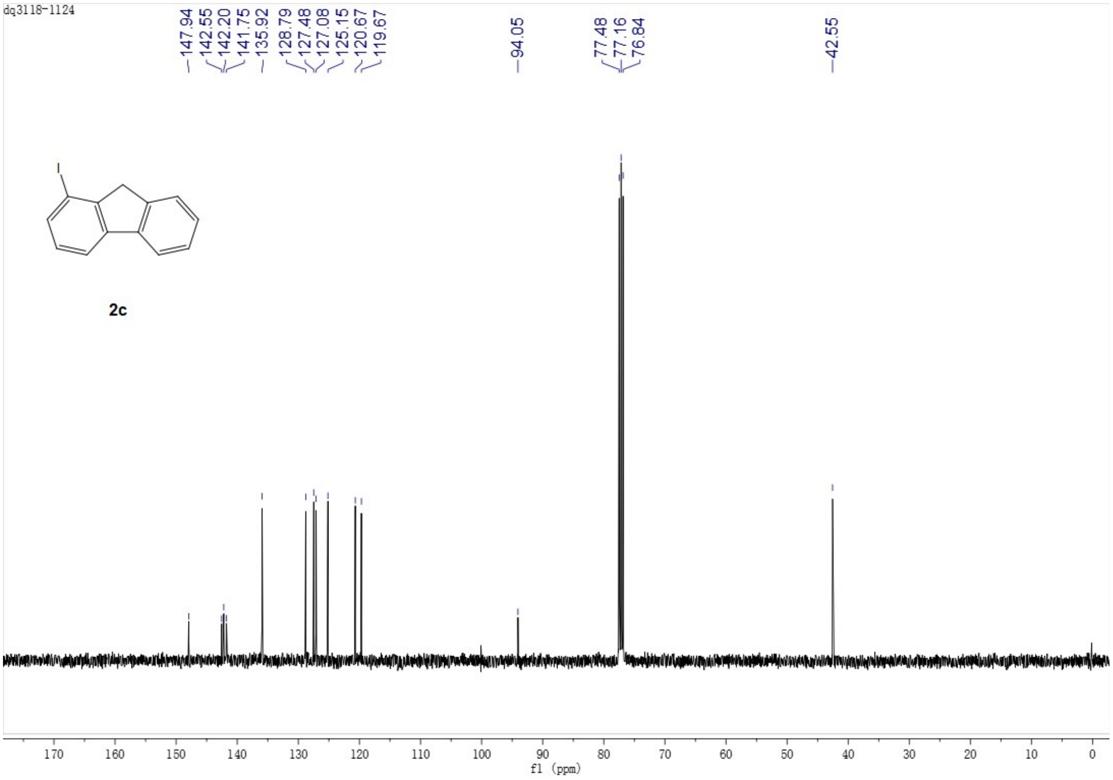


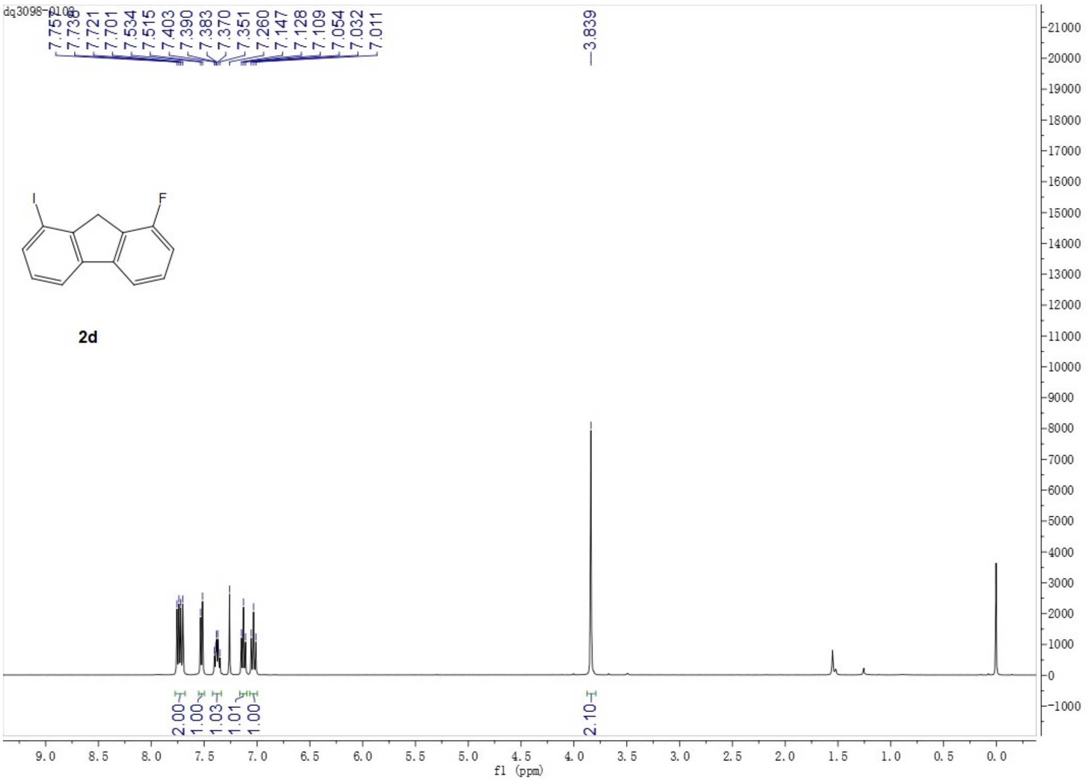
2b

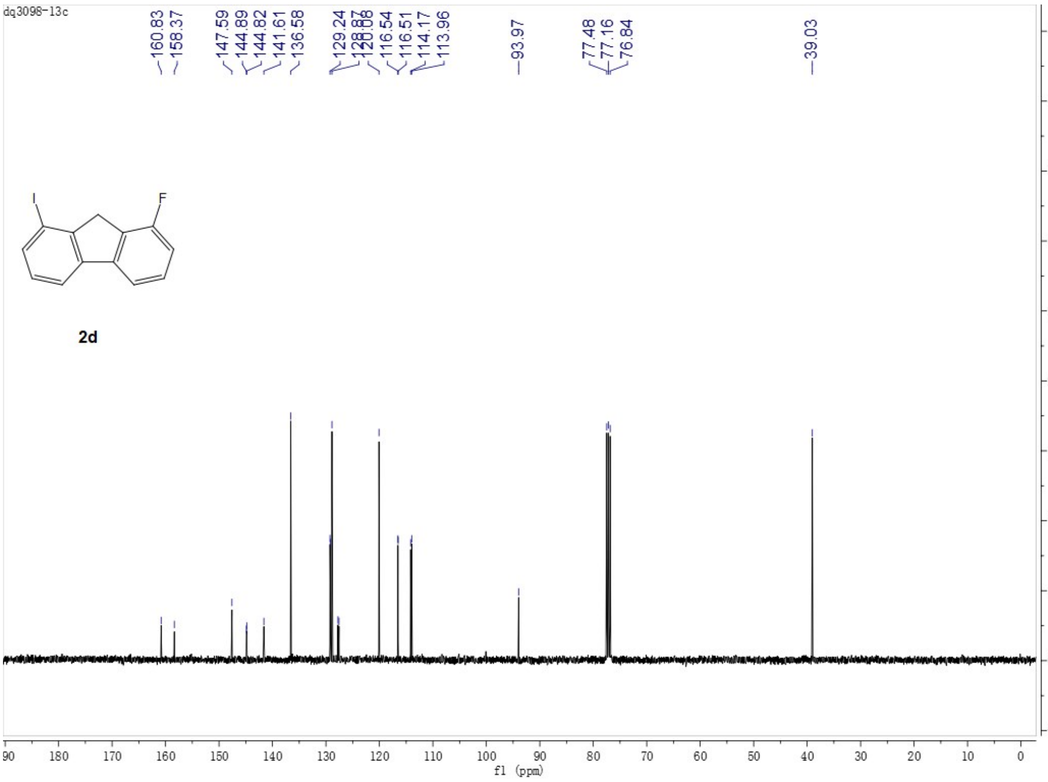
dq3139-0220
7.746
7.727
7.570
7.550
7.358
7.339
7.320
7.260
7.180
7.162
6.906
6.886
3.682
2.677
2.457
I
CH3
CH3
1.00
0.98
1.00
0.99
0.99
2.02
3.05
2.99
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

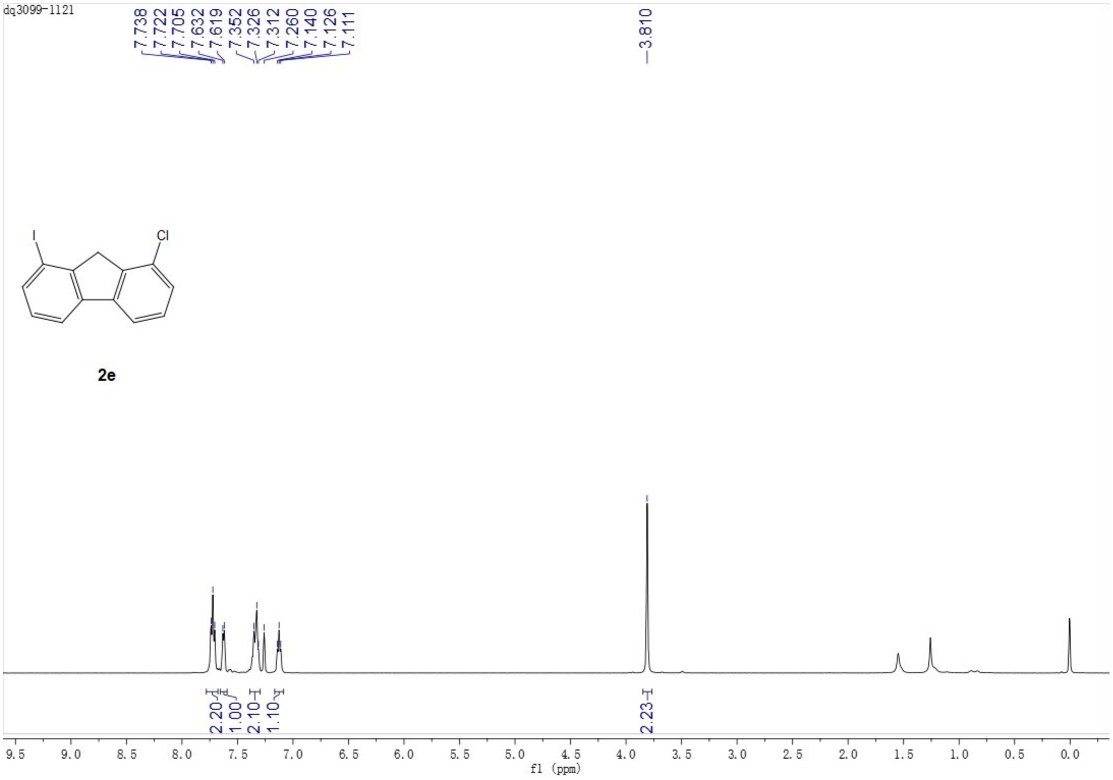

dq3139-13c-1
147.63
142.41
141.32
141.00
135.37
134.16
133.17
131.22
127.81
127.30
121.35
90.73
77.48
77.16
76.84
41.86
20.80
19.10
I
CH3
CH3
2b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

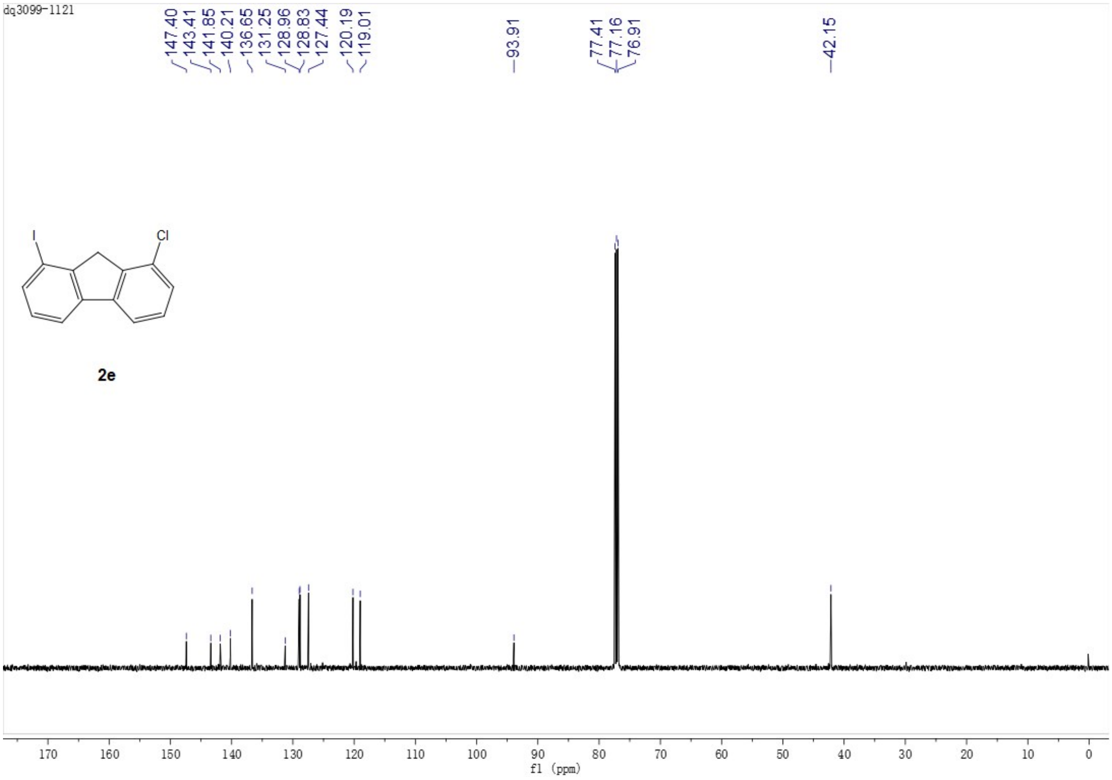

dq3118-1124
7.765
7.754
7.747
7.736
7.686
7.667
7.584
7.566
7.417
7.399
7.381
7.363
7.360
7.344
7.342
7.326
7.323
7.260
7.135
7.115
7.096
3.816
I
2c
1.99
0.99
1.00
1.03
0.97
0.99
2.04
.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

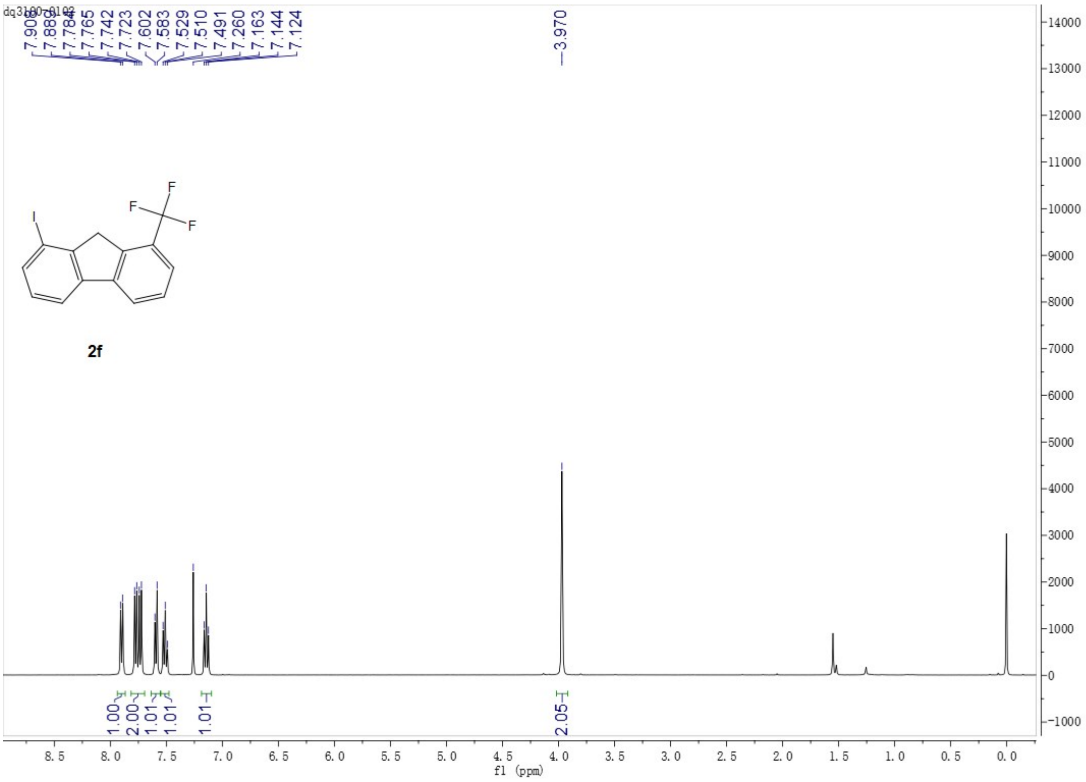

dq3118-1124
147.94
142.55
142.20
141.75
135.92
128.79
127.48
127.08
125.15
120.67
119.67
94.05
77.48
77.16
76.84
42.55
I
2c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

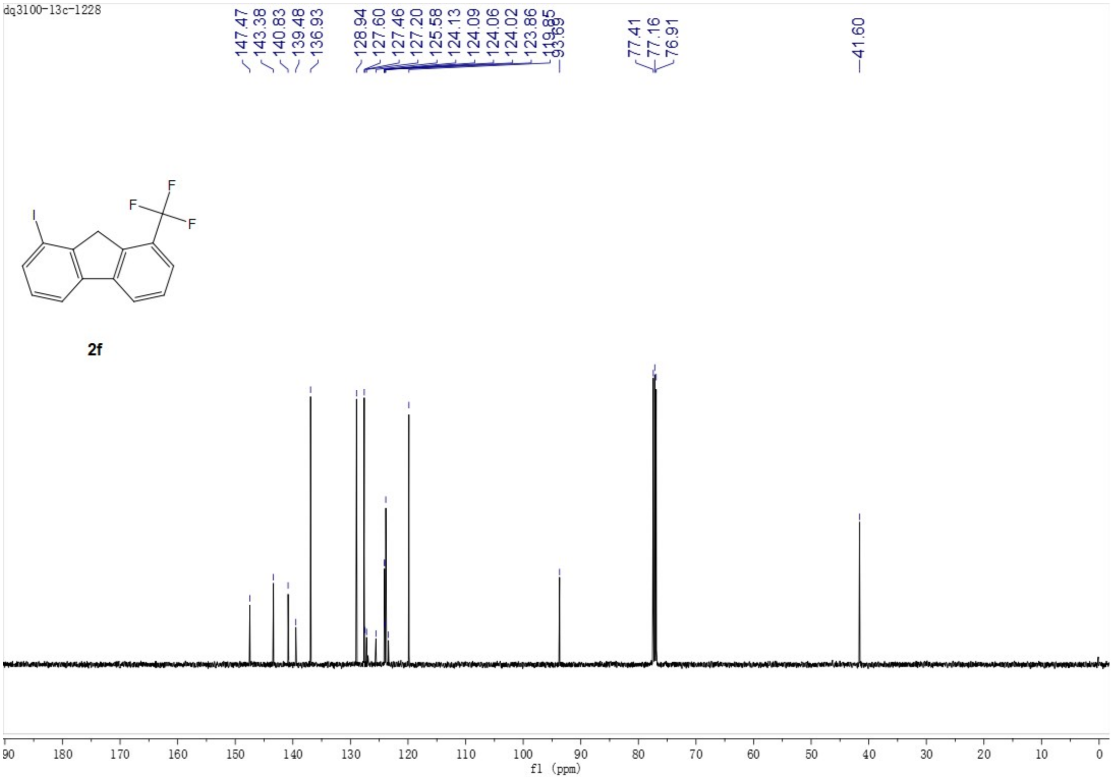

dq3098-0109
7.751
7.738
7.721
7.701
7.534
7.515
7.403
7.390
7.383
7.370
7.351
7.260
7.147
7.128
7.109
7.054
7.032
7.011
3.839
I
F
2d
2.00
1.00
1.03
1.01
1.00
2.10
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000

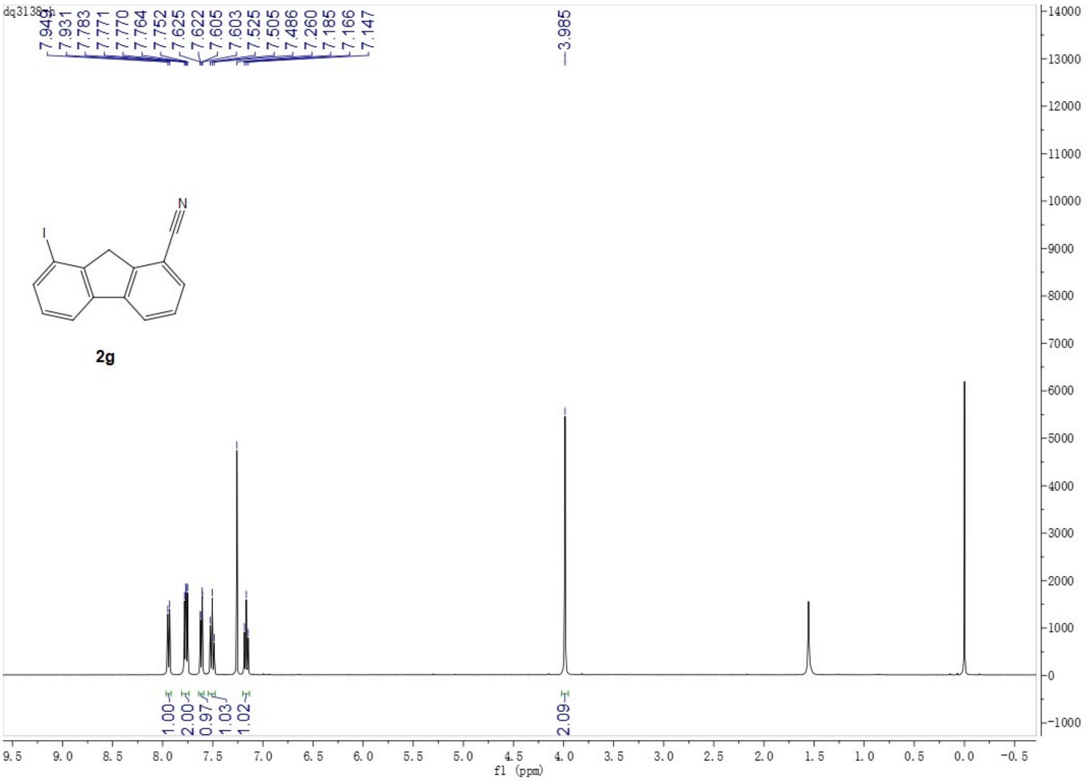

dq3098-13c
160.83
158.37
147.59
144.89
144.82
141.61
136.58
129.24
128.87
128.08
116.54
116.51
114.17
113.96
93.97
77.48
77.16
76.84
39.03
I
F
2d
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

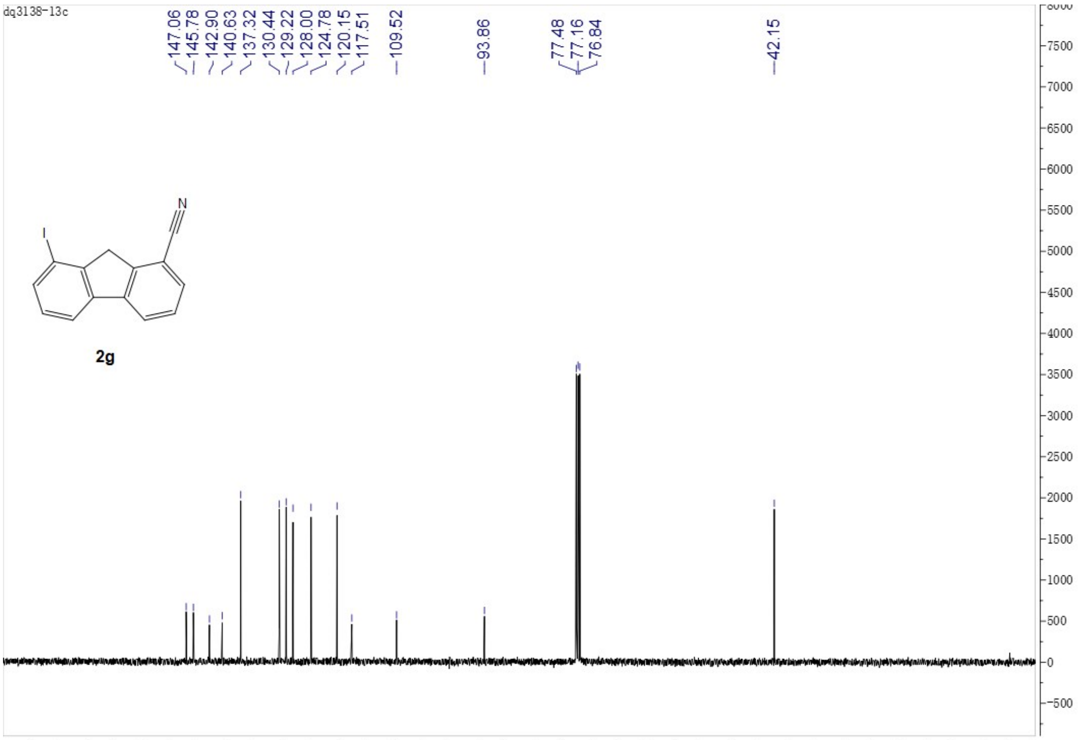

dq3099-1121
7.738
7.722
7.705
7.632
7.619
7.352
7.326
7.312
7.260
7.140
7.126
7.111
3.810
I
Cl
2e
2.20
1.00
2.10
1.10
2.23
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

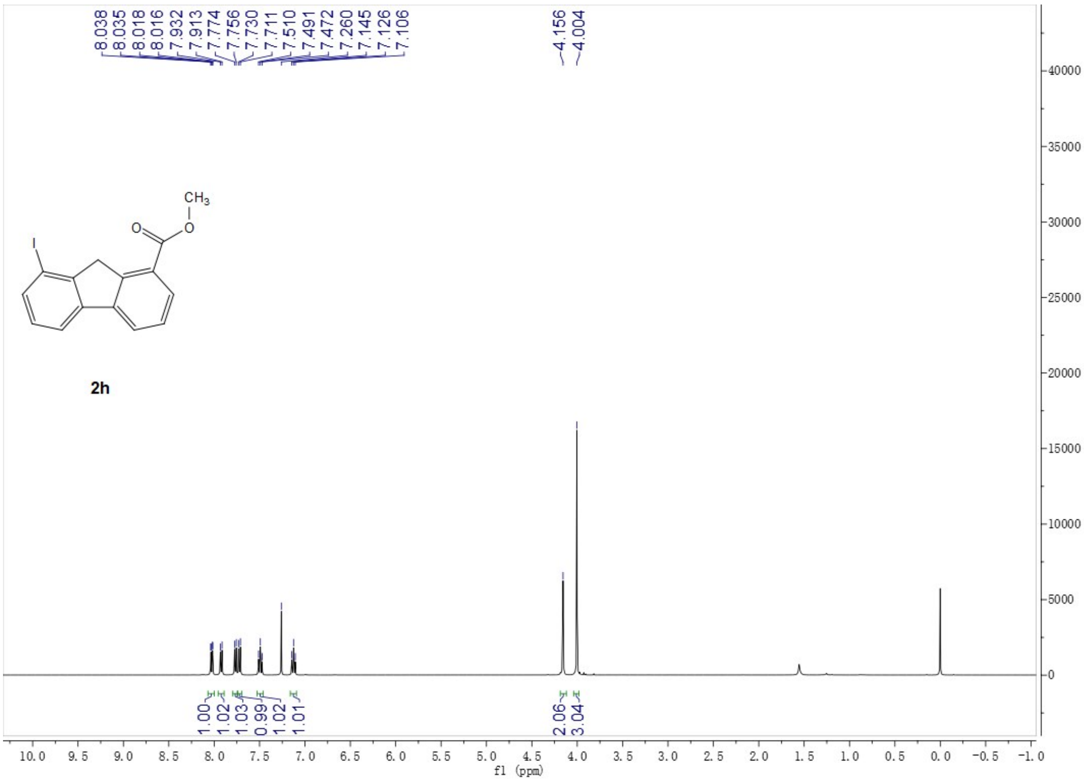

dq3099-1121
147.40
143.41
141.85
140.21
136.65
131.25
128.96
128.83
127.44
120.19
119.01
93.91
77.41
77.16
76.91
42.15
I
Cl
2e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

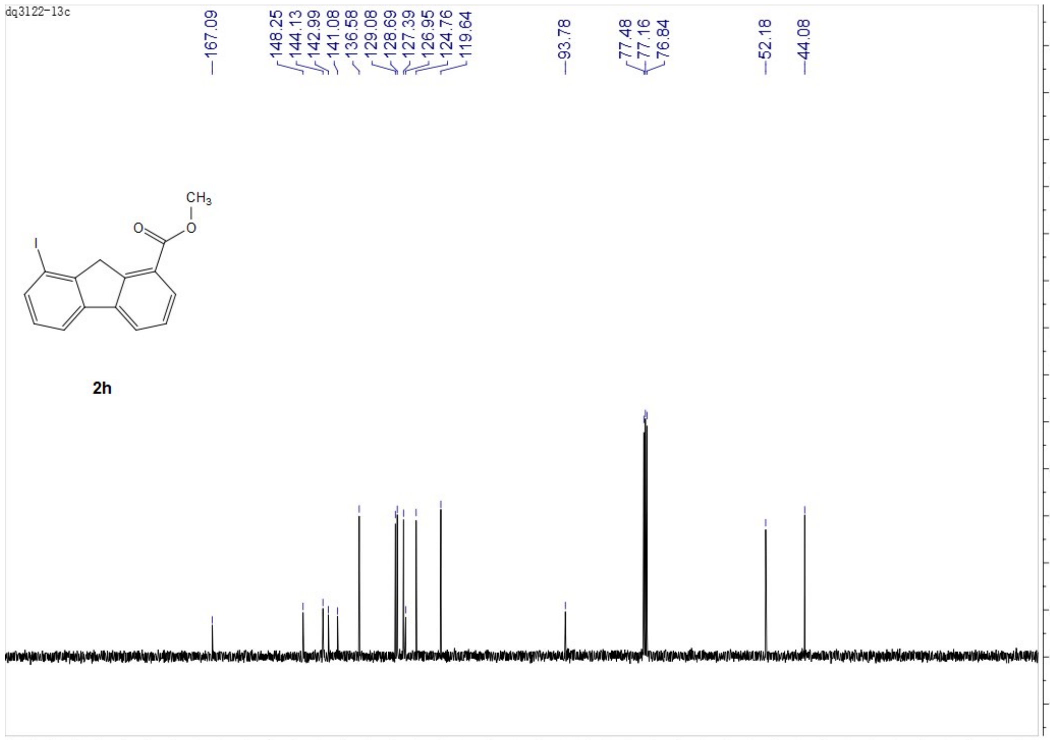

dq3100-0102
7.908
7.889
7.784
7.765
7.742
7.723
7.602
7.583
7.529
7.510
7.491
7.260
7.163
7.144
7.124
3.970
I
F
F
F
2f
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
1.00
2.00
1.01
1.01
1.01
2.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

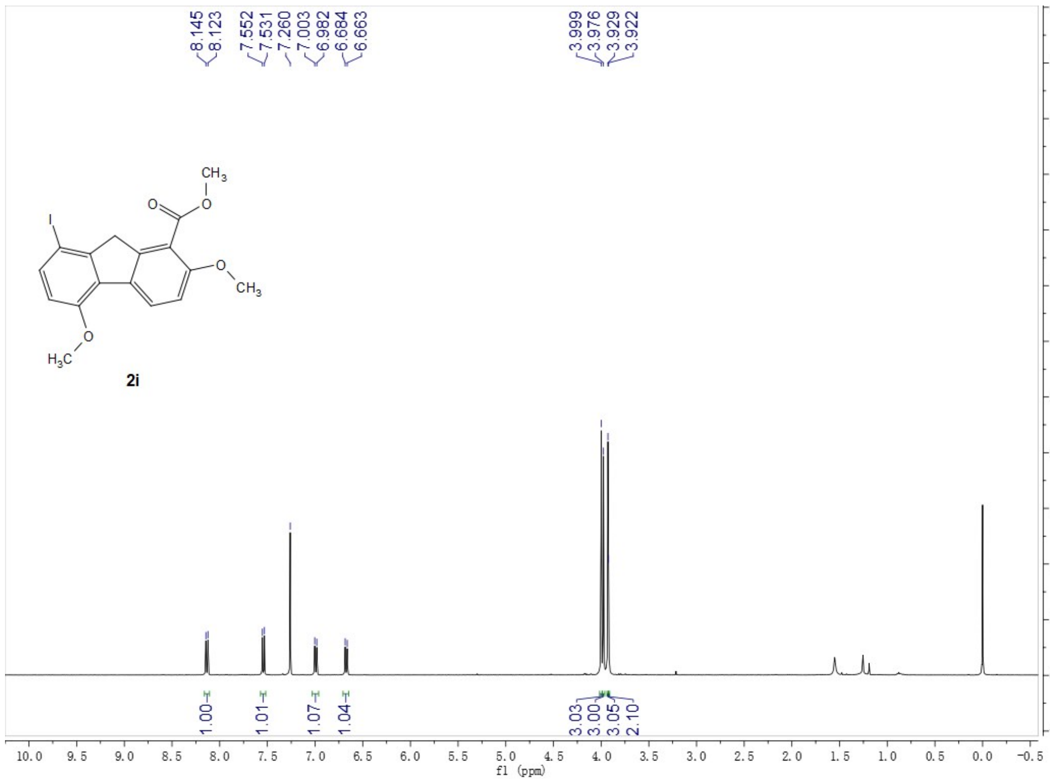

dq3100-13c-1228
147.47
143.38
140.83
139.48
136.93
128.94
127.60
127.46
127.20
125.58
124.13
124.09
124.06
124.02
123.86
119.85
93.69
77.41
77.16
76.91
41.60
I
F
F
F
2f
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

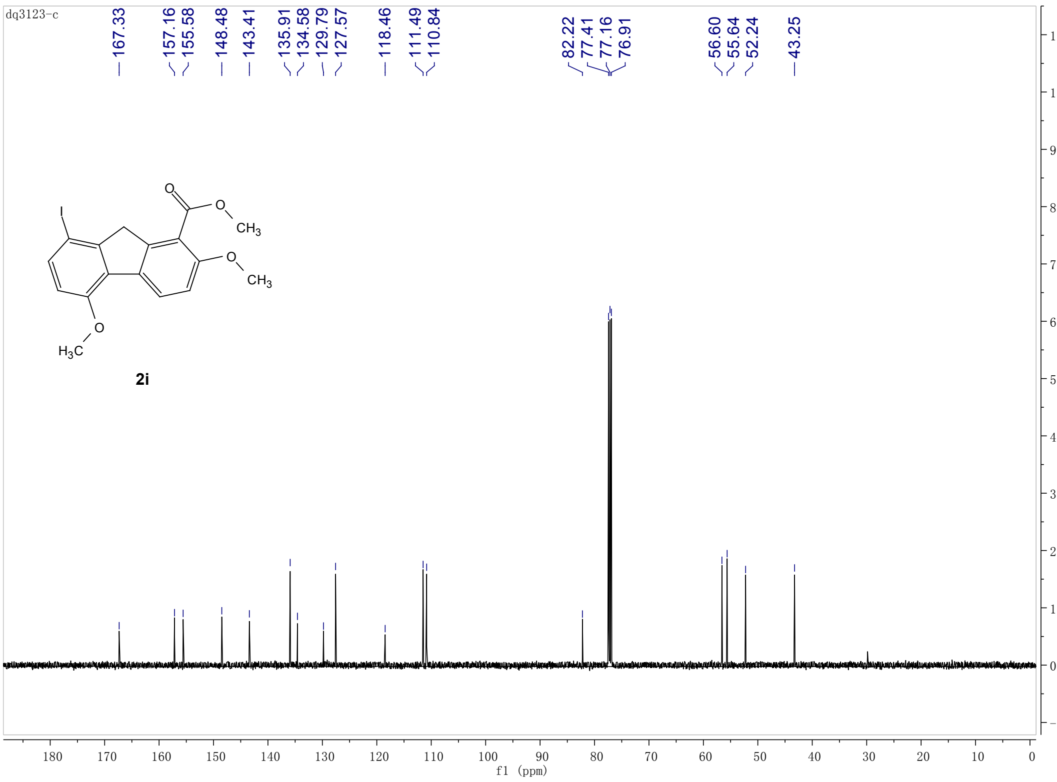

dq3138-h
7.949
7.931
7.783
7.771
7.770
7.764
7.752
7.625
7.622
7.605
7.603
7.525
7.505
7.486
7.260
7.185
7.166
7.147
3.985
I
N
2g
1.00
2.00
0.97
1.03
1.02
2.09
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


dq3138-13c
147.06
145.78
142.90
140.63
137.32
130.44
129.22
128.00
124.78
120.15
117.51
109.52
93.86
77.48
77.16
76.84
42.15
I
N
2g
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

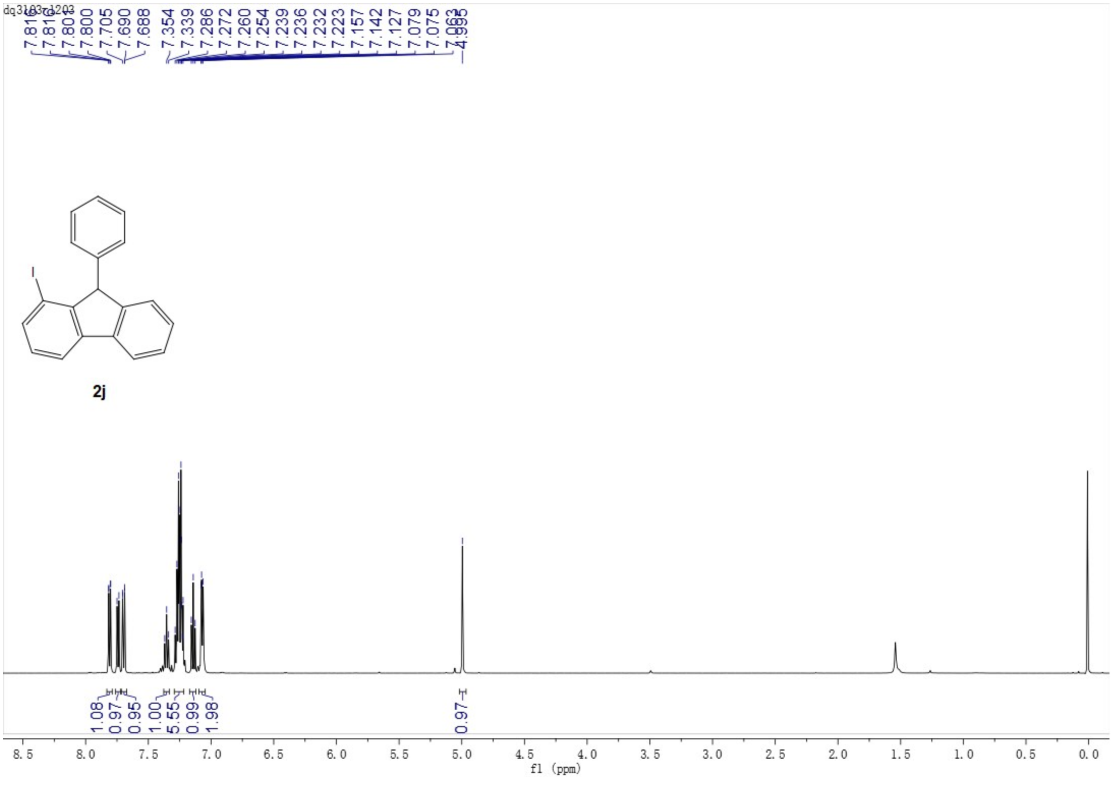

8.038
8.035
8.018
8.016
7.932
7.913
7.774
7.756
7.730
7.711
7.510
7.491
7.472
7.260
7.145
7.126
7.106
4.156
4.004
CH3
O
O
I
2h
1.00
1.02
1.03
0.99
1.02
1.01
2.06
3.04
40000
35000
30000
25000
20000
15000
10000
5000
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


dq3122-13c
167.09
148.25
144.13
142.99
141.08
136.58
129.08
128.69
127.39
126.95
124.76
119.64
93.78
77.48
77.16
76.84
52.18
44.08
CH3
O
O
I
2h
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

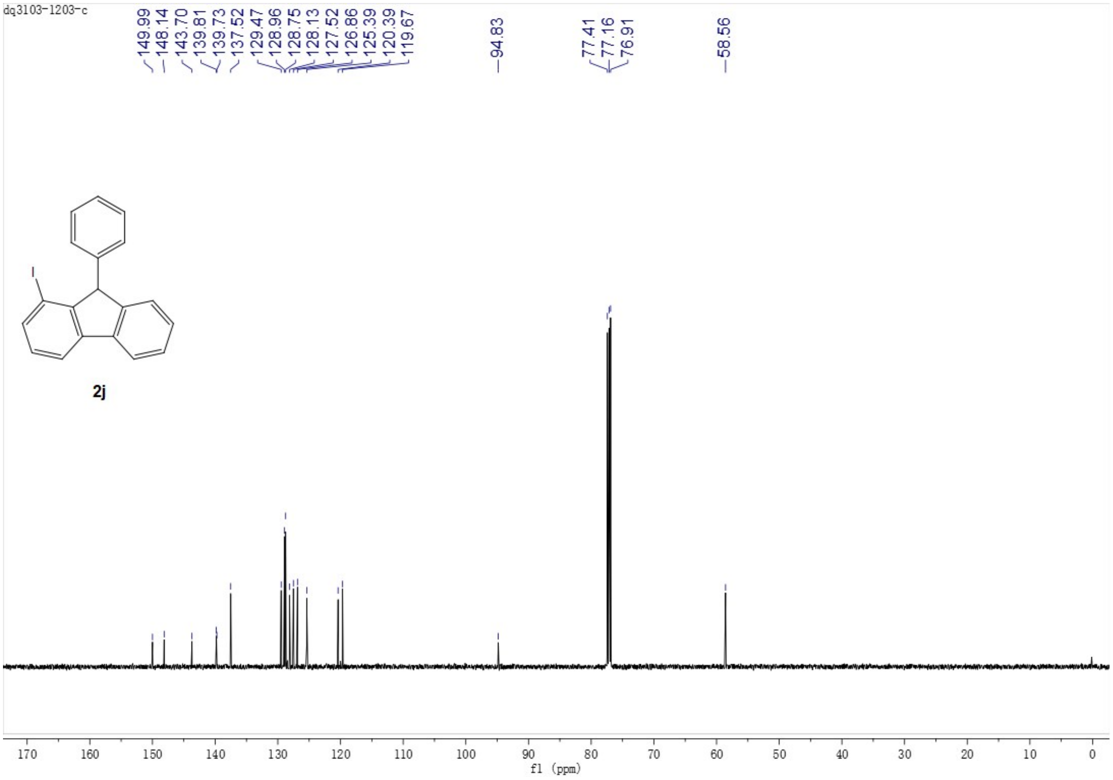

8.145
8.123
7.552
7.531
7.260
7.003
6.982
6.684
6.663
3.999
3.976
3.929
3.922
CH3
O
O
I
O
CH3
O
H3C
2i
1.00
1.01
1.07
1.04
3.03
3.00
3.05
2.10
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


dq3123-c
167.33
157.16
155.58
148.48
143.41
135.91
134.58
129.79
127.57
118.46
111.49
110.84
82.22
77.41
77.16
76.91
56.60
55.64
52.24
43.25
I
O
O
CH3
O
CH3
O
H3C
1100
1000
900
800
700
600
500
400
300
200
100
0
−100
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

2i

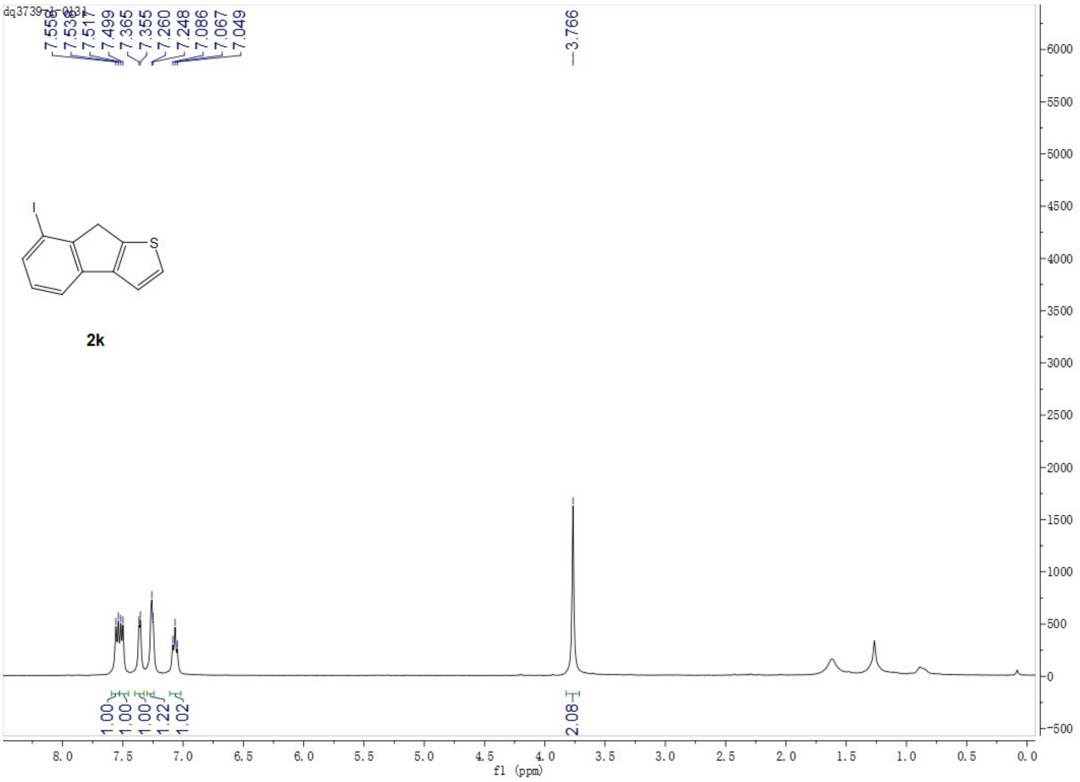

dq3103-1203
7.816
7.816
7.801
7.800
7.705
7.690
7.688
7.354
7.339
7.286
7.272
7.260
7.254
7.239
7.236
7.232
7.223
7.157
7.142
7.127
7.079
7.075
7.063
4.995
I
1.08
0.97
0.95
1.00
5.55
0.99
1.98
0.97
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

2j



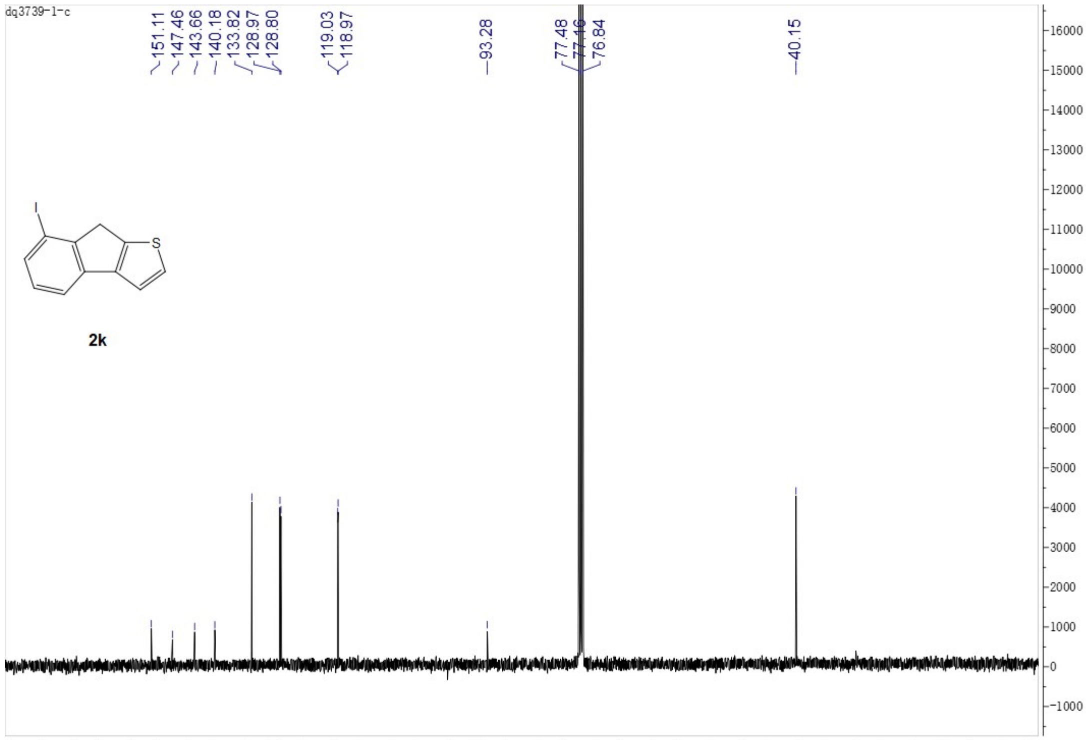
2j

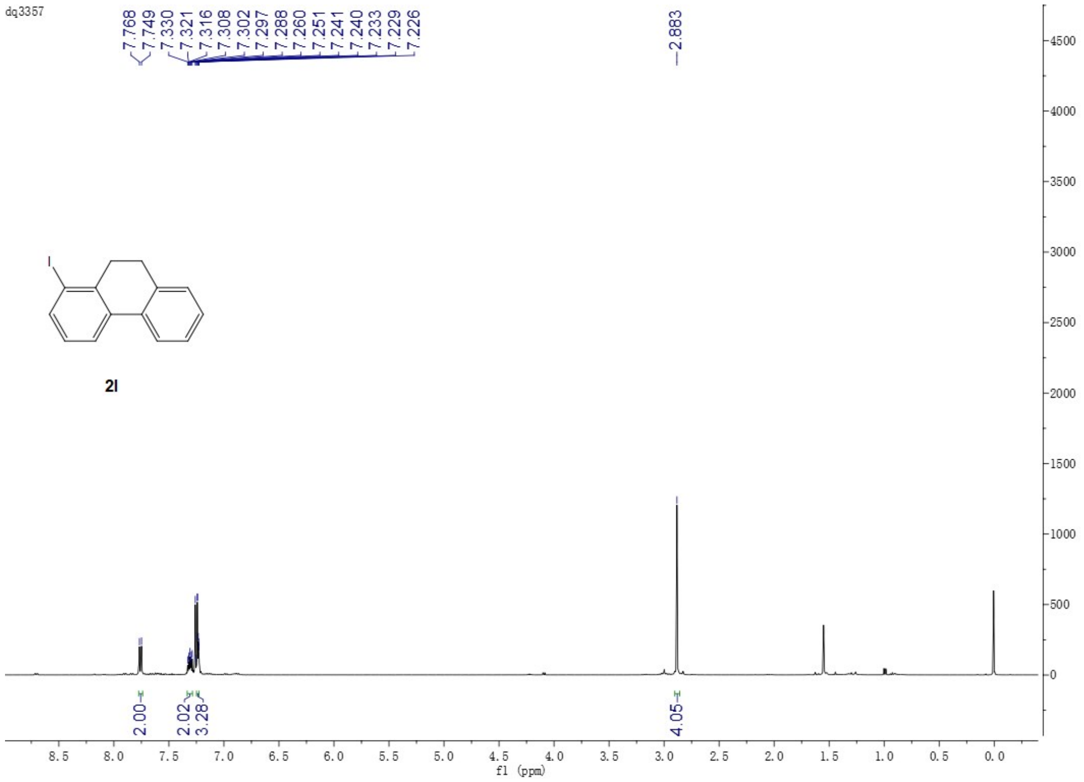
dq3103-1203-c
149.99
148.14
143.70
139.81
139.73
137.52
129.47
128.96
128.75
128.13
127.52
126.86
125.39
120.39
119.67
94.83
77.41
77.16
76.91
58.56
I
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

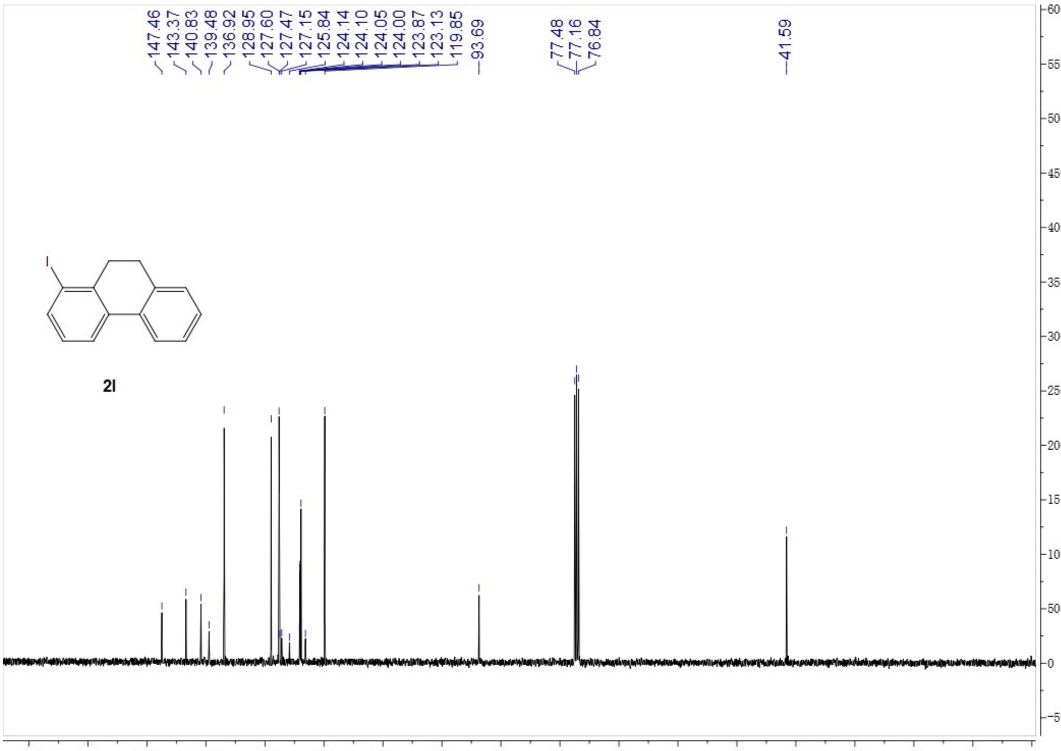


2k

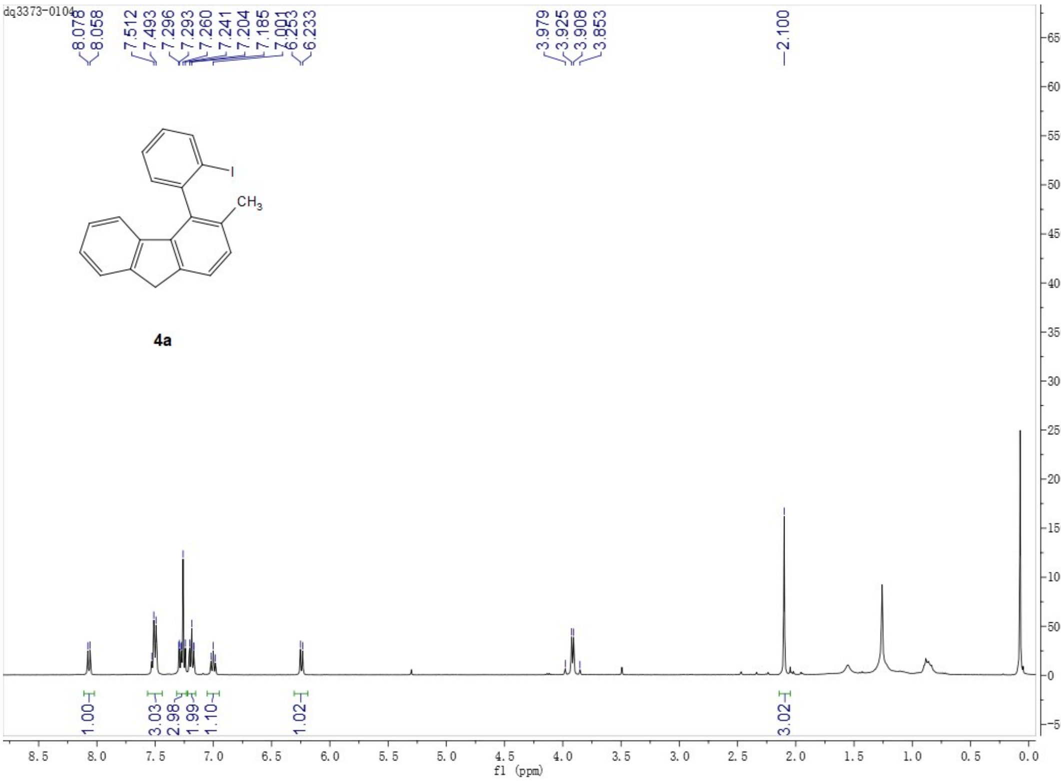
dq3739-1-0131
7.558
7.538
7.517
7.499
7.365
7.355
7.260
7.248
7.086
7.067
7.049
3.766
I
S
1.00
1.00
1.00
1.22
1.02
2.08
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500


dq3739-1-c
151.11
147.46
143.66
140.18
133.82
128.97
128.80
119.03
118.97
93.28
77.48
77.16
76.84
40.15
I
S
2k
fl (ppm)

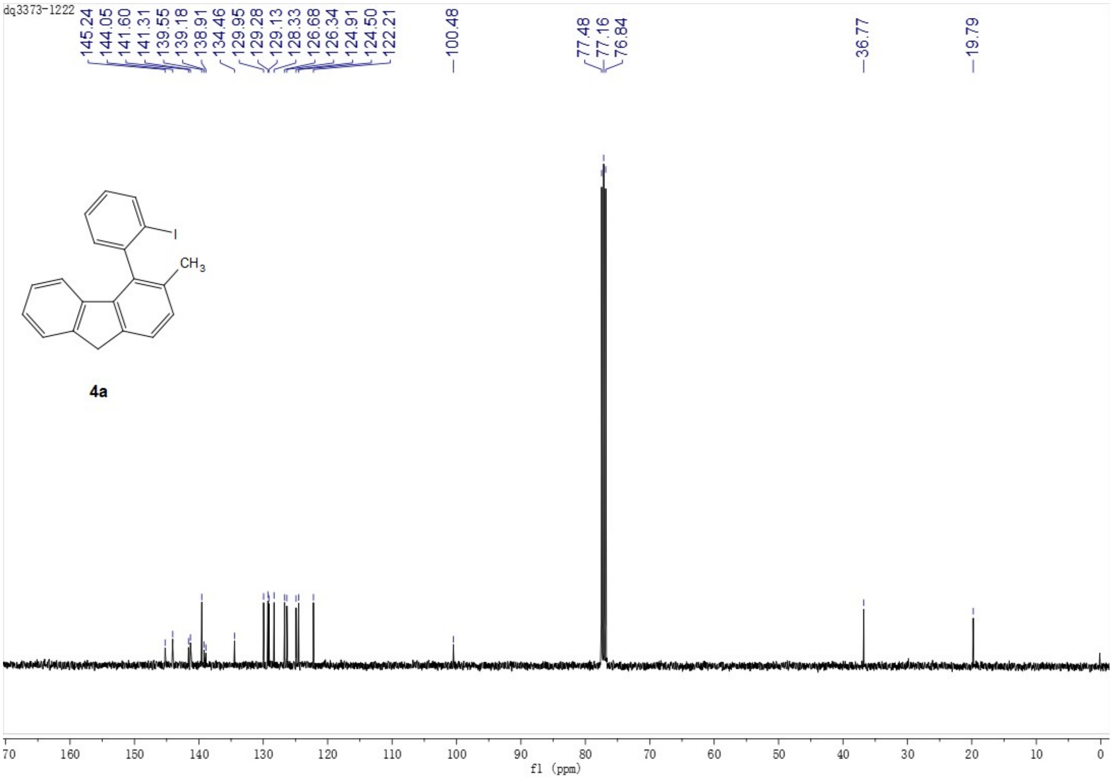

dq3357
7.768
7.749
7.330
7.321
7.316
7.308
7.302
7.297
7.288
7.260
7.251
7.241
7.240
7.233
7.229
7.226
2.883
I
2l
2.00
2.02
3.28
4.05
fl (ppm)

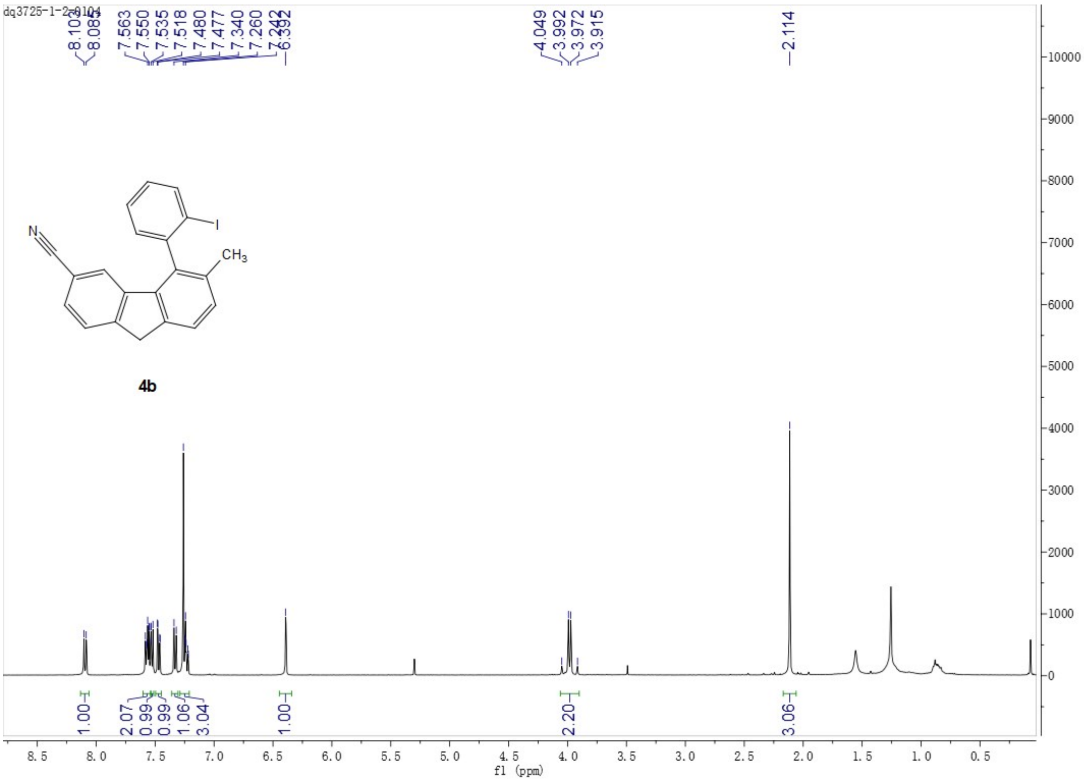

147.46
143.37
140.83
139.48
136.92
128.95
127.60
127.47
127.15
125.84
124.14
124.10
124.05
124.00
123.87
123.13
119.85
93.69
77.48
77.16
76.84
41.59
I
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

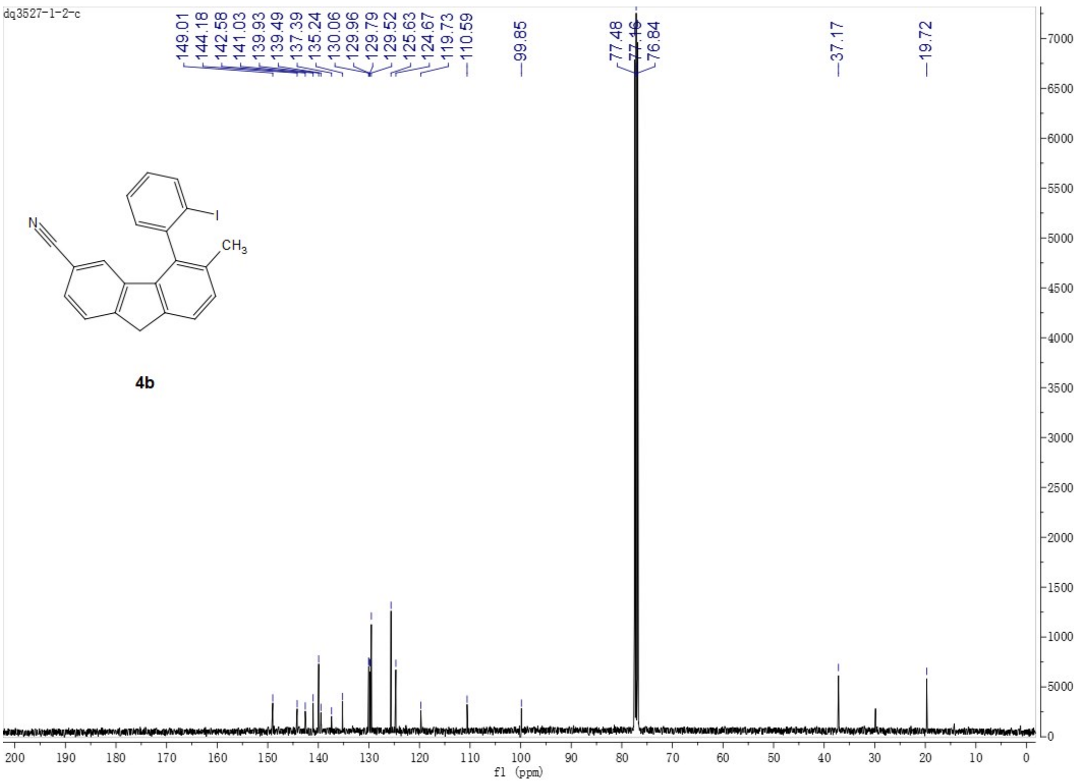
2l

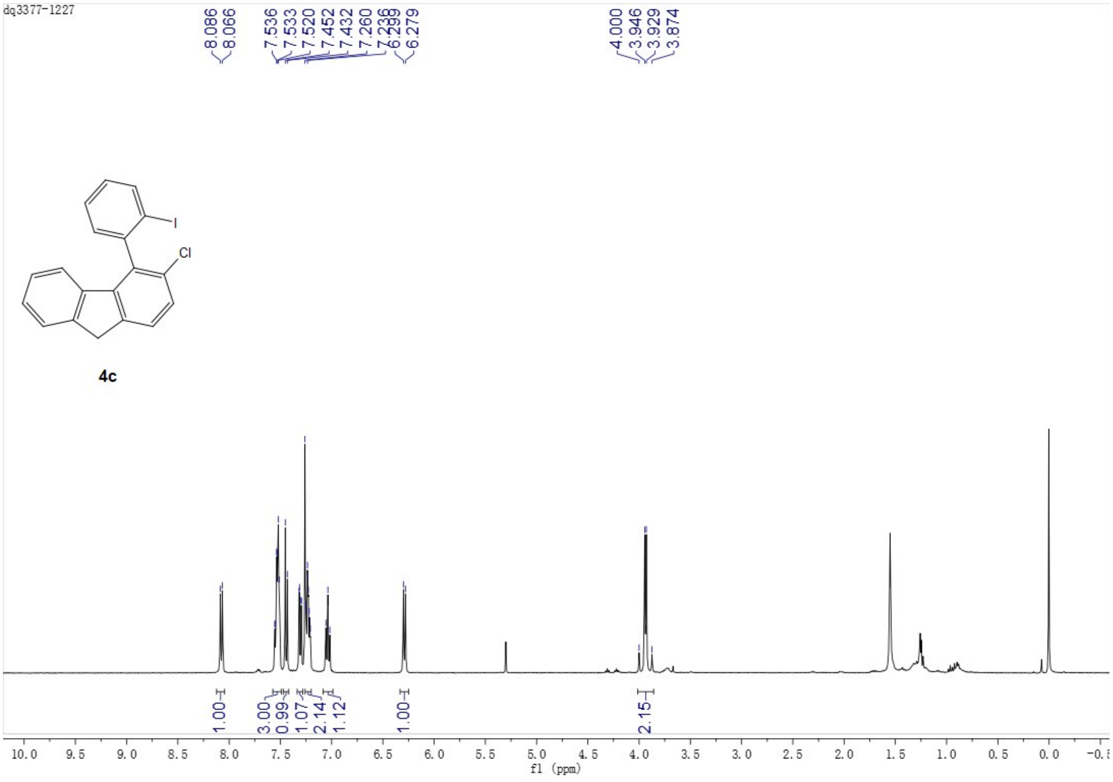

dq3373-0104
8.078
8.058
7.512
7.493
7.296
7.293
7.260
7.241
7.204
7.185
7.001
6.253
6.233
3.979
3.925
3.908
3.853
2.100
I
CH3
1.00
3.03
2.98
1.99
1.10
1.02
3.02
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

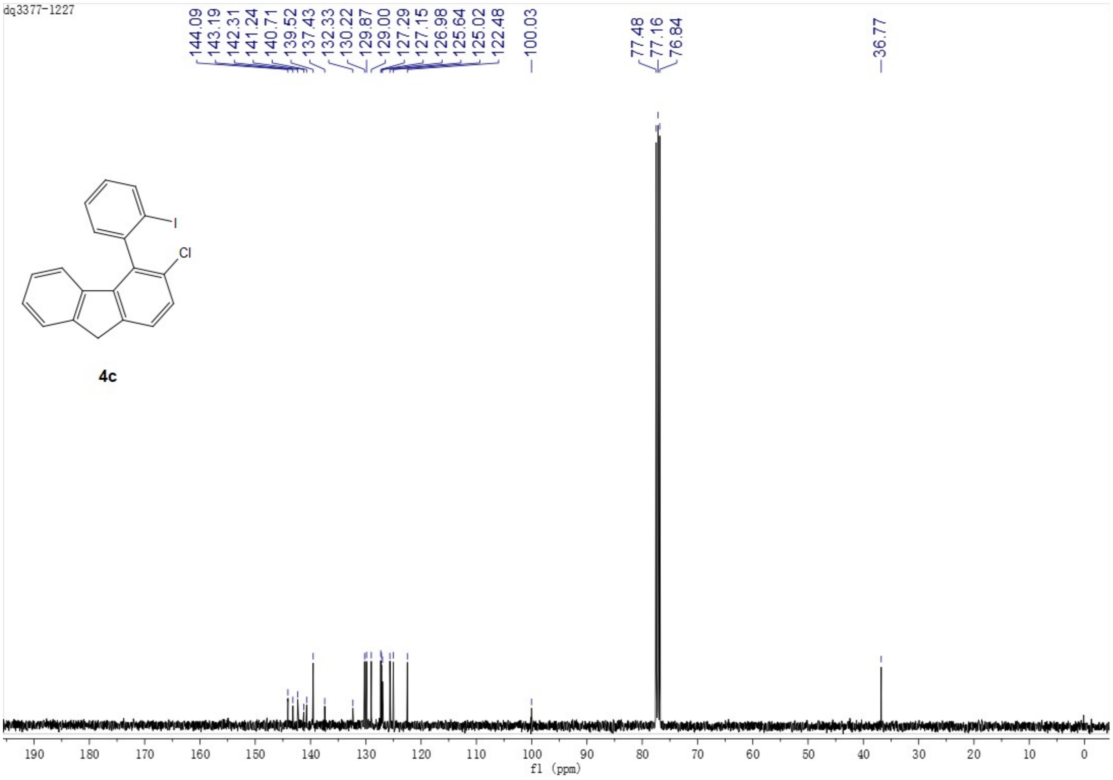
4a


dq3373-1222
145.24
144.05
141.60
141.31
139.55
139.18
138.91
134.46
129.95
129.28
129.13
128.33
126.68
126.34
124.91
124.50
122.21
100.48
77.48
77.16
76.84
36.77
19.79
I
CH3
4a
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


dq3725-1-2-0104
8.103
8.085
7.563
7.550
7.535
7.518
7.480
7.477
7.340
7.260
7.242
6.392
4.049
3.992
3.972
3.915
2.114
N
I
CH3
4b
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
1.00
2.07
0.99
0.99
1.06
3.04
1.00
2.20
3.06
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
f1 (ppm)

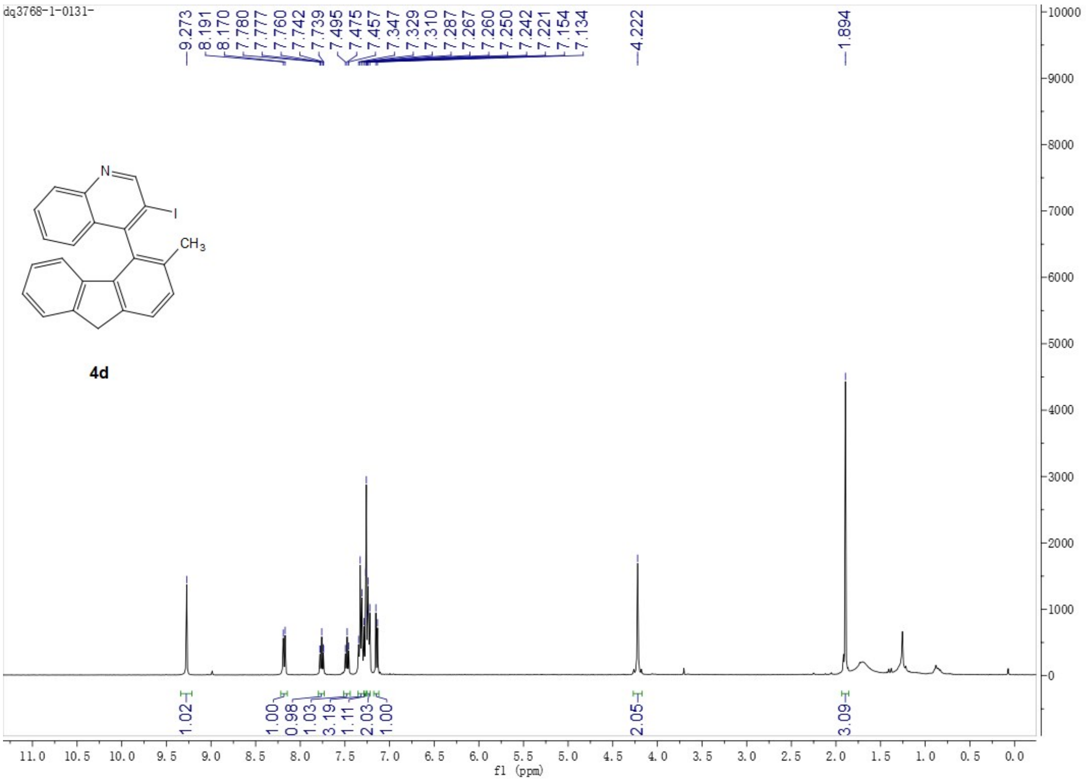

dq3527-1-2-c
149.01
144.18
142.58
141.03
139.93
139.49
137.39
135.24
130.06
129.96
129.79
129.52
125.63
124.67
119.73
110.59
99.85
77.48
77.16
76.84
37.17
19.72
N
I
CH3
4b
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


dq3377-1227
8.086
8.066
7.536
7.533
7.520
7.452
7.432
7.260
7.236
6.299
6.279
4.000
3.946
3.929
3.874
I
Cl
4c
1.00
3.00
0.99
1.07
2.14
1.12
1.00
2.15
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)



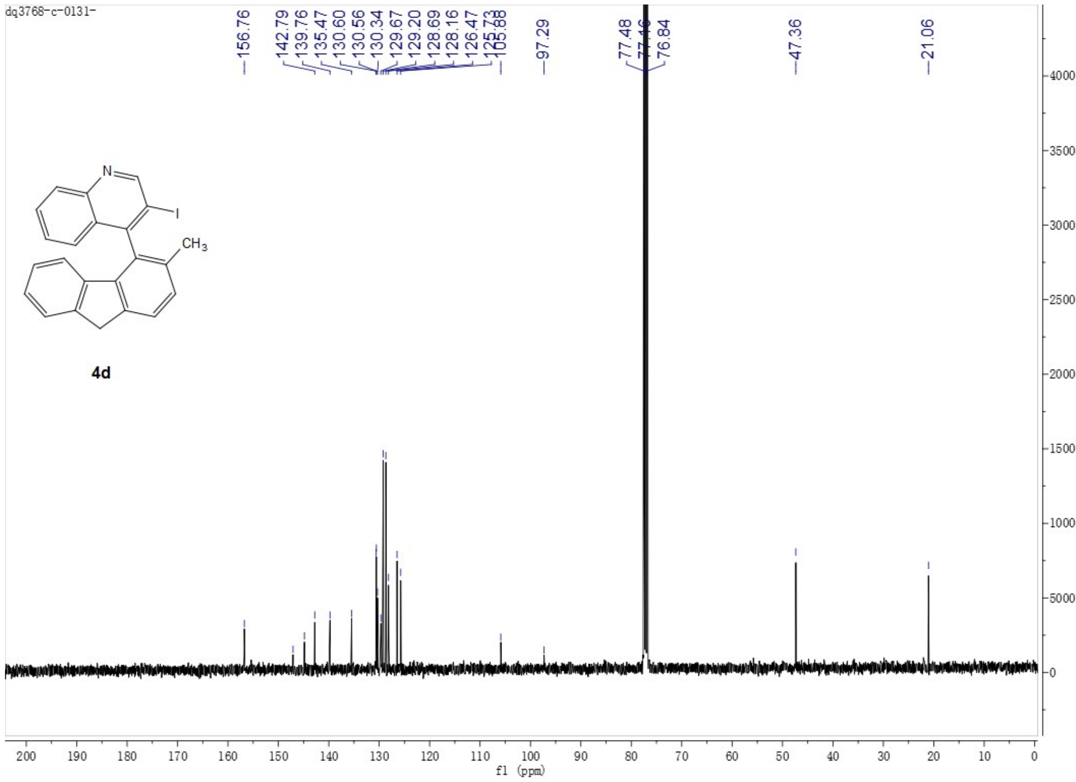
4c

dq3377-1227
144.09
143.19
142.31
141.24
140.71
139.52
137.43
132.33
130.22
129.87
129.00
127.29
127.15
126.98
125.64
125.02
122.48
100.03
77.48
77.16
76.84
36.77
Cl
I
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

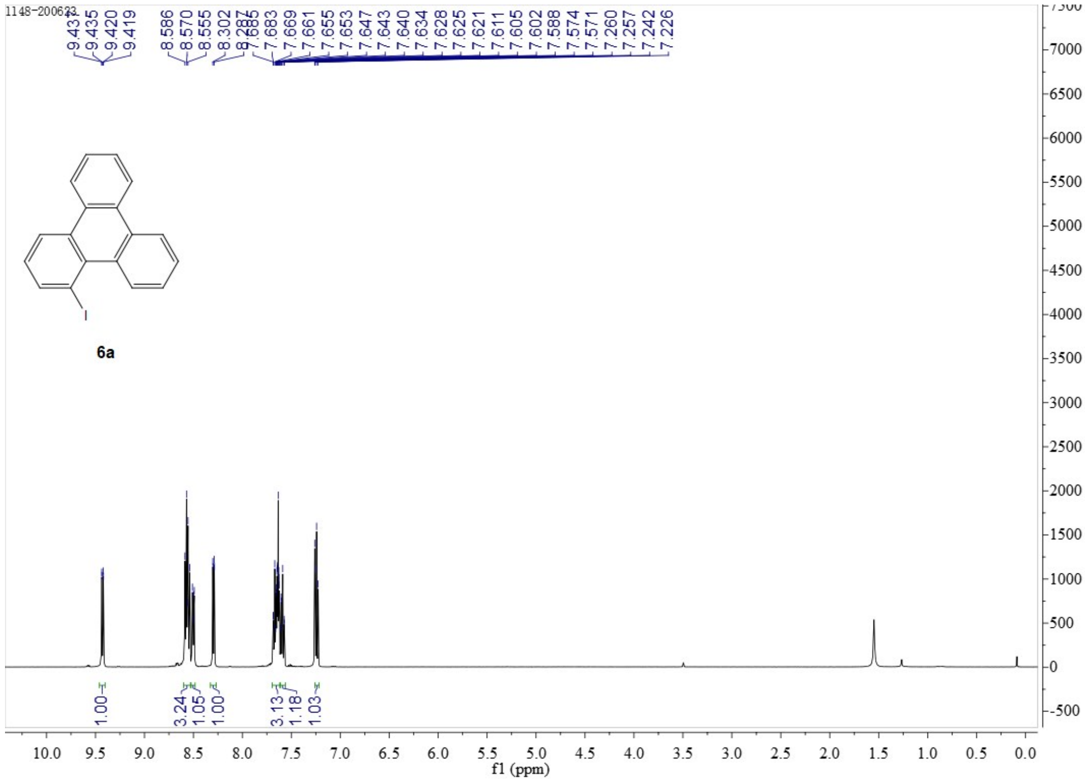


4d

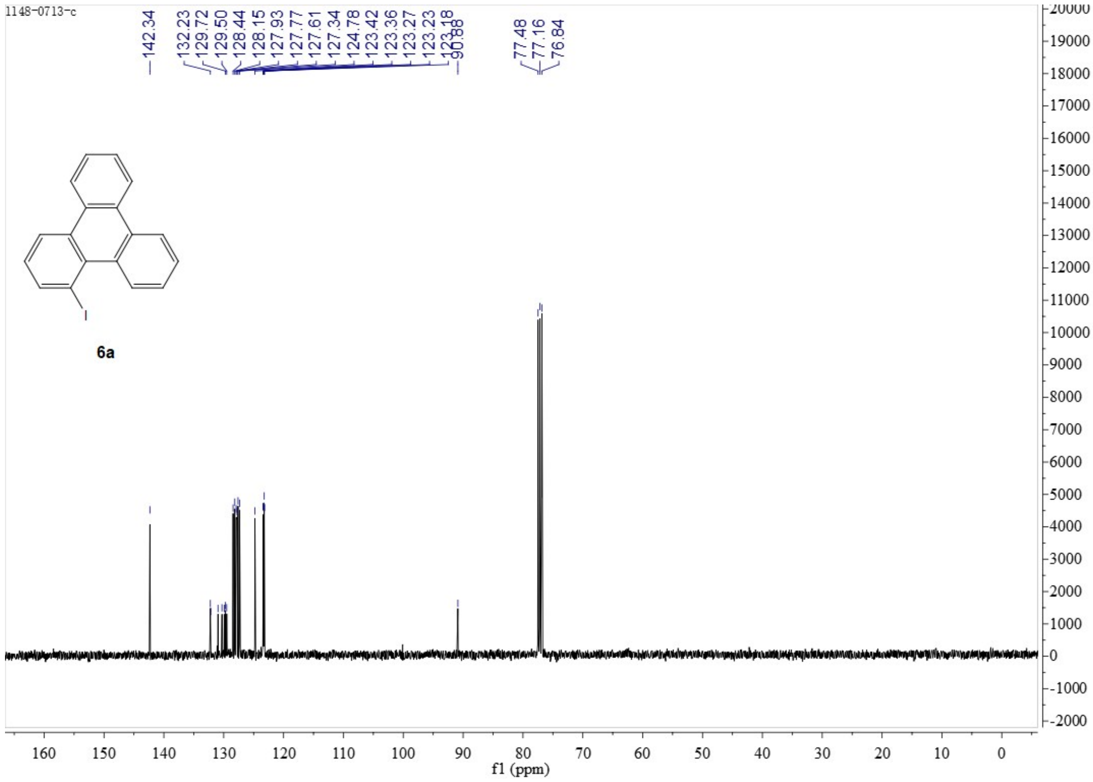
dq3768-1-0131-
9.273
8.191
8.170
7.780
7.777
7.760
7.742
7.739
7.495
7.475
7.457
7.347
7.329
7.310
7.287
7.267
7.260
7.250
7.242
7.221
7.154
7.134
4.222
1.894
N
I
CH3
1.02
1.00
0.98
1.03
3.19
1.11
2.03
1.00
2.05
3.09
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
11.0 10.5 10.0 9.5 9.0 8.5 8.0 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
f1 (ppm)


dq3768-c-0131-
156.76
142.79
139.76
135.47
130.60
130.56
130.34
129.67
129.20
128.69
128.16
126.47
125.73
105.88
97.29
77.48
77.16
76.84
47.36
21.06
N
I
CH3
40000
35000
30000
25000
20000
15000
10000
5000
0
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

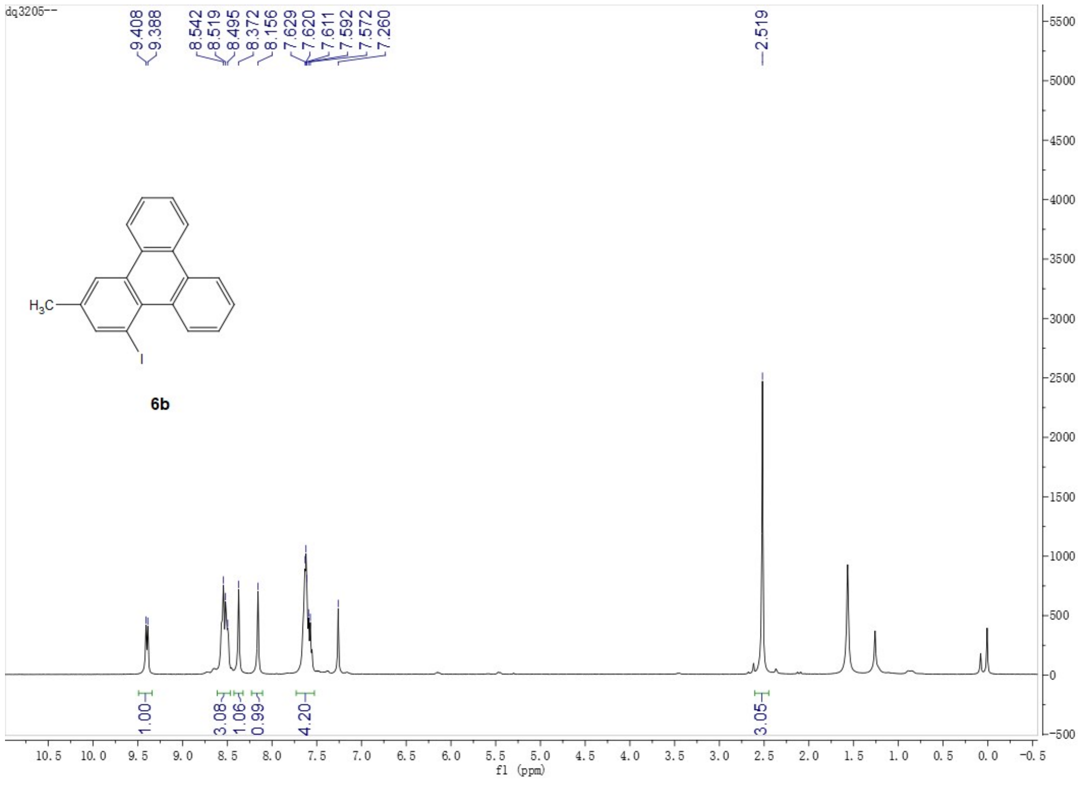
4d


1148-200623
9.437
9.435
9.420
9.419
8.586
8.570
8.555
8.302
8.287
7.685
7.683
7.669
7.661
7.655
7.653
7.647
7.643
7.640
7.634
7.628
7.625
7.621
7.611
7.605
7.602
7.588
7.574
7.571
7.260
7.257
7.242
7.226
I
1.00
3.24
1.05
1.00
3.13
1.18
1.03
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
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
f1 (ppm)

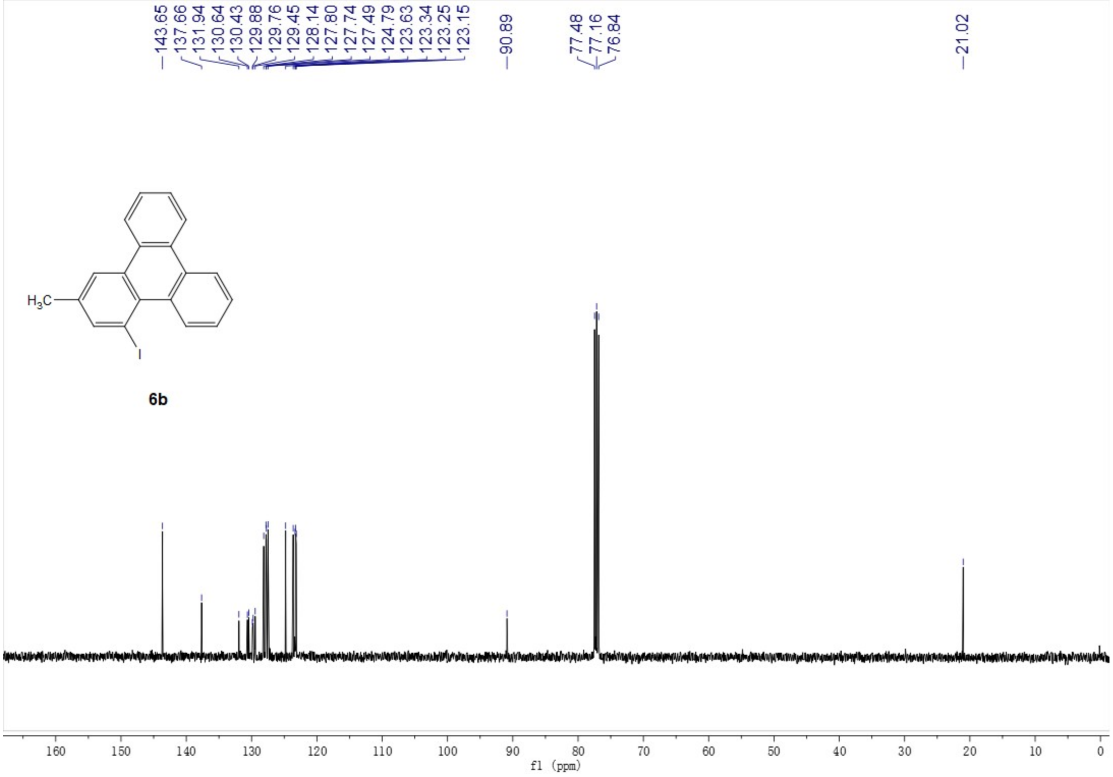
6a


6a

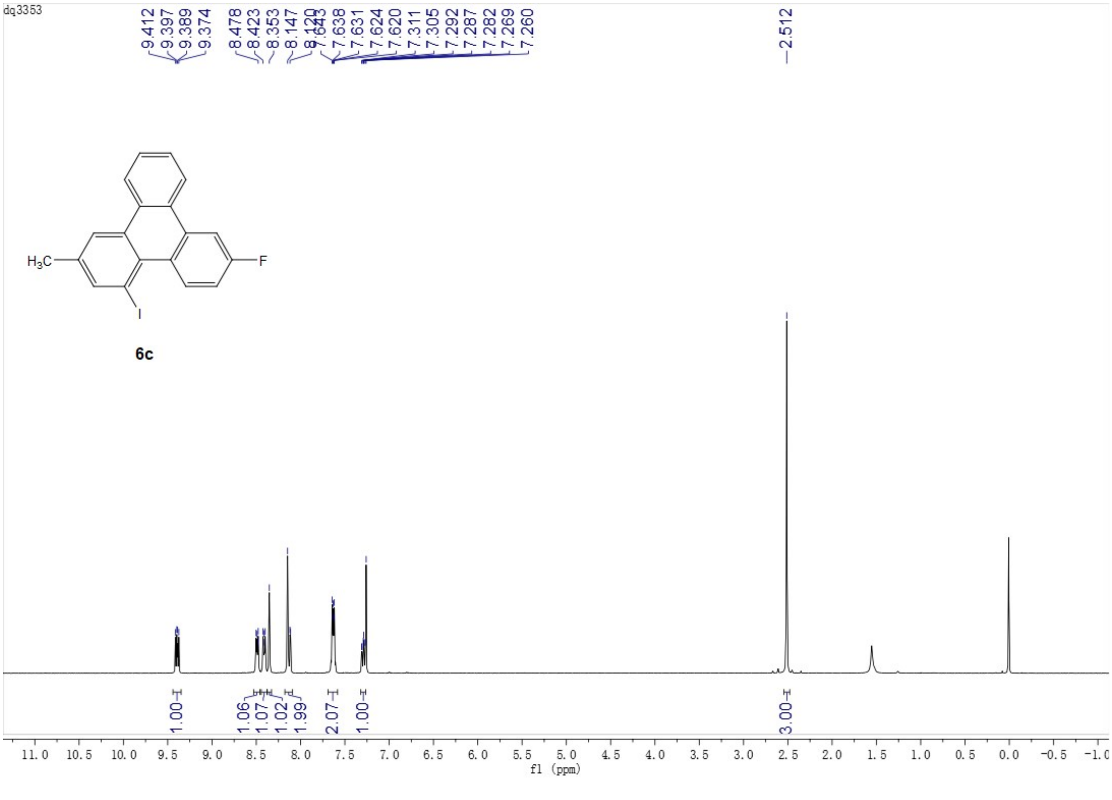
1148-0713-c
142.34
132.23
129.72
129.50
128.44
128.15
127.93
127.77
127.61
127.34
124.78
123.42
123.36
123.27
123.23
123.18
90.88
77.48
77.16
76.84
I
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
20000 19000 18000 17000 16000 15000 14000 13000 12000 11000 10000 9000 8000 7000 6000 5000 4000 3000 2000 1000 0 -1000 -2000


6b

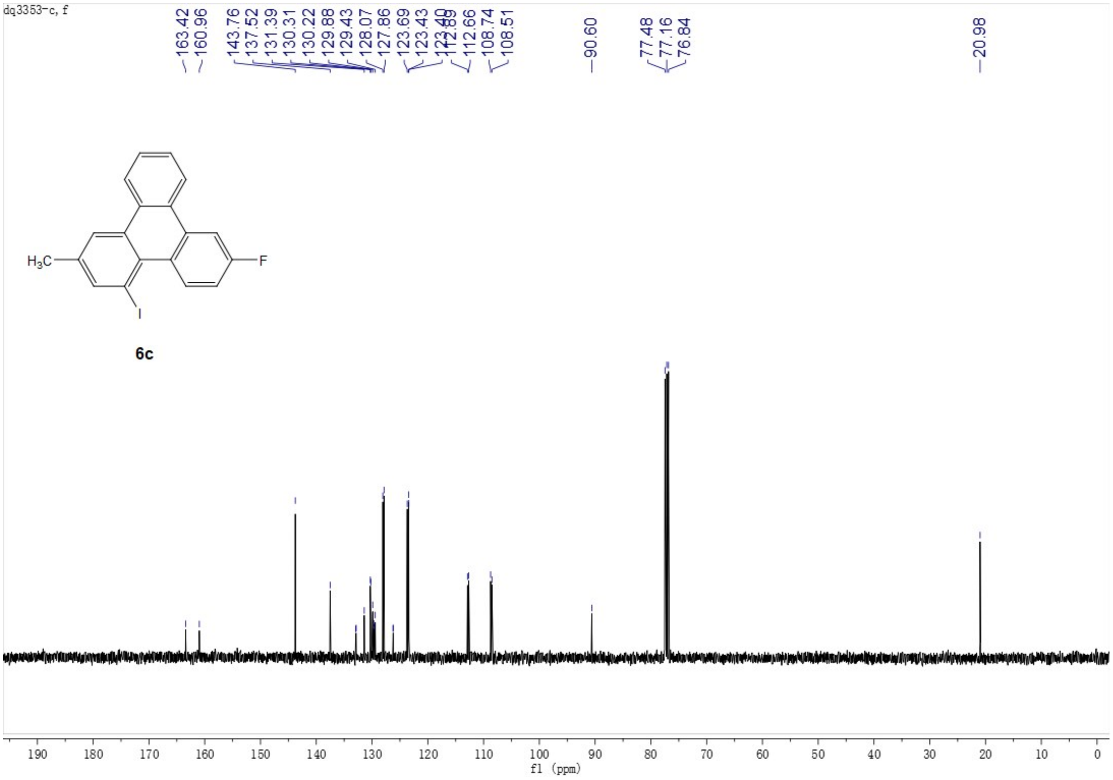
dq3205--
9.408
9.388
8.542
8.519
8.495
8.372
8.156
7.629
7.620
7.611
7.592
7.572
7.260
2.519
H_3C
I
1.00
3.08
1.06
0.99
4.20
3.05
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500


143.65
137.66
131.94
130.64
130.43
129.88
129.76
129.45
128.14
127.80
127.74
127.49
124.79
123.63
123.34
123.25
123.15
90.89
77.48
77.16
76.84
21.02
H$_3$C
I
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

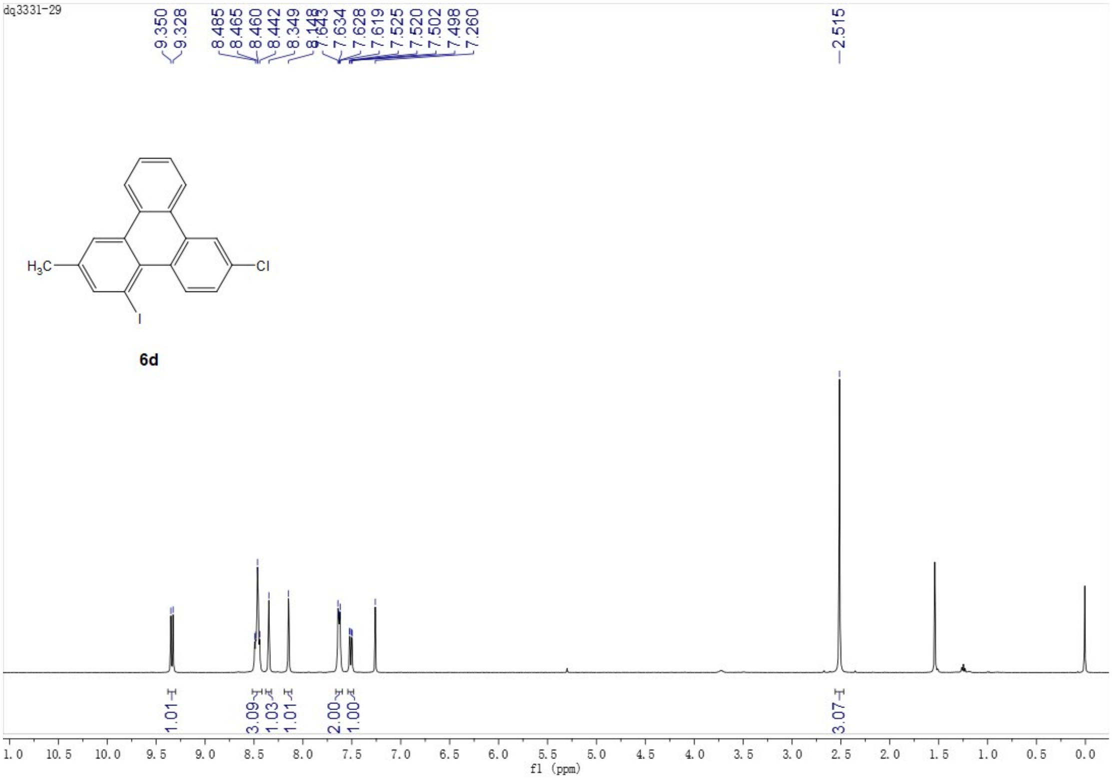
6b


dq3353
9.412
9.397
9.389
9.374
8.478
8.423
8.353
8.147
8.120
7.643
7.638
7.631
7.624
7.620
7.311
7.305
7.292
7.287
7.282
7.269
7.260
2.512
H$_3$C
F
I
1.00
1.06
1.07
1.02
1.99
2.07
1.00
3.00
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)

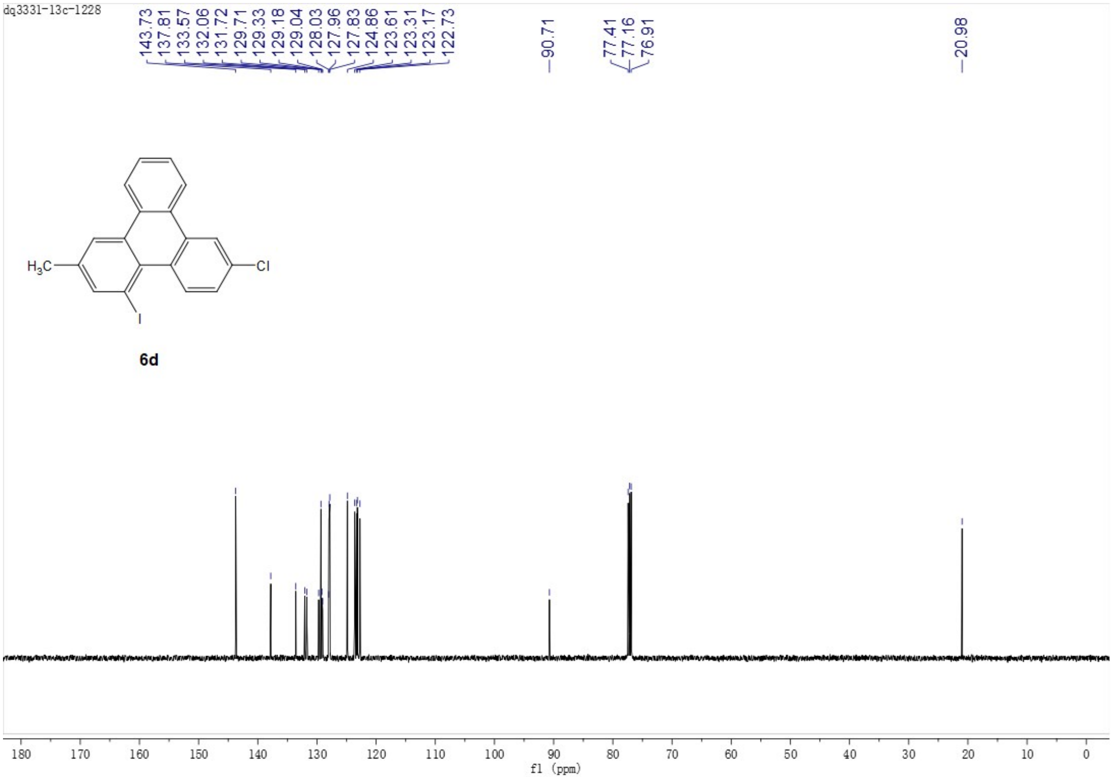
6c

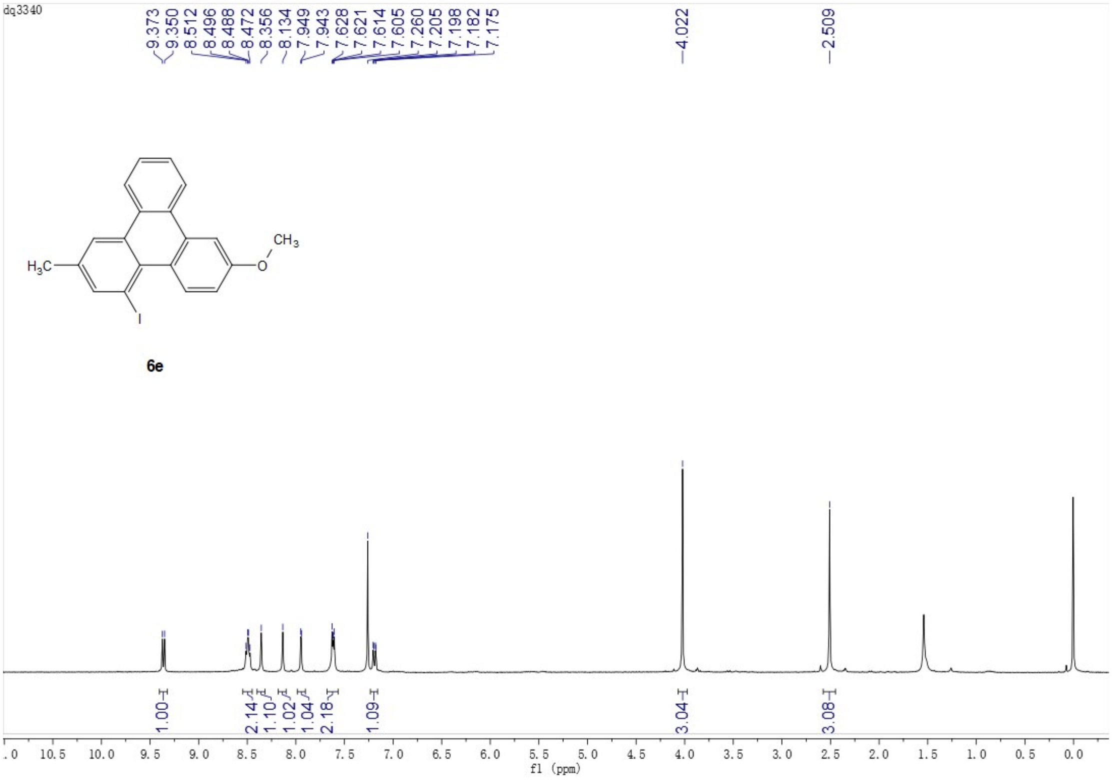

dq3353-c, f
163.42
160.96
143.76
137.52
131.39
130.31
130.22
129.88
129.43
128.07
127.86
123.69
123.43
123.40
112.89
112.66
108.74
108.51
90.60
77.48
77.16
76.84
20.98
H$_3$C
F
I
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

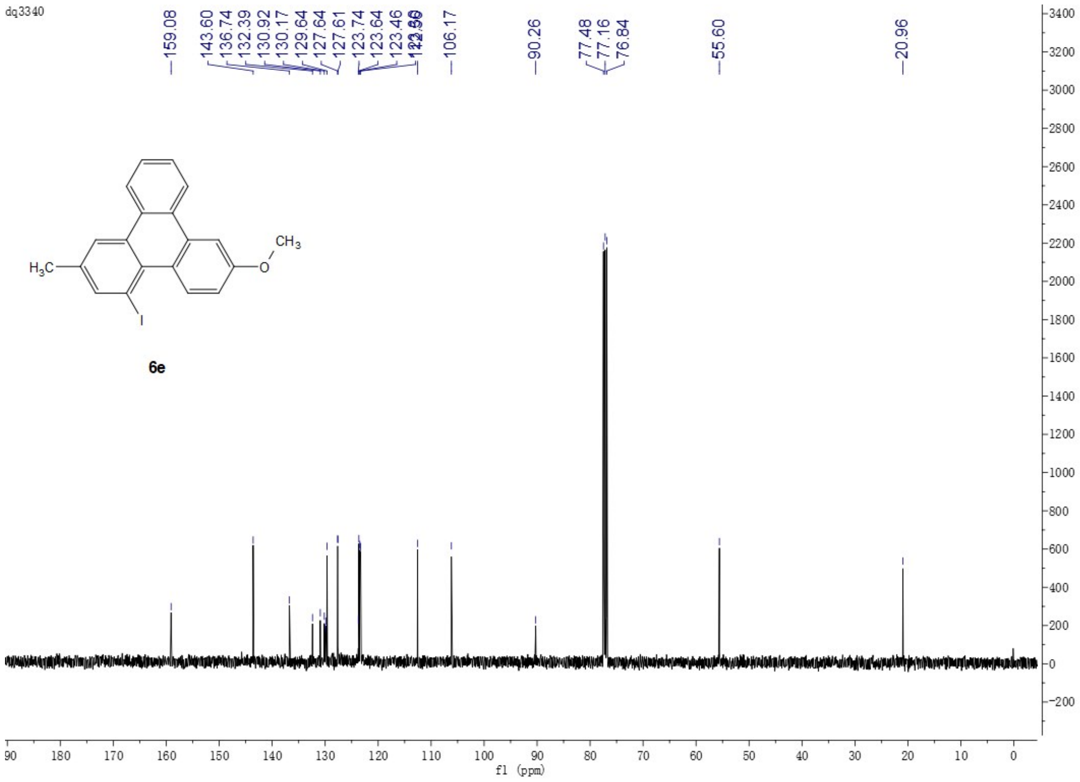
6c

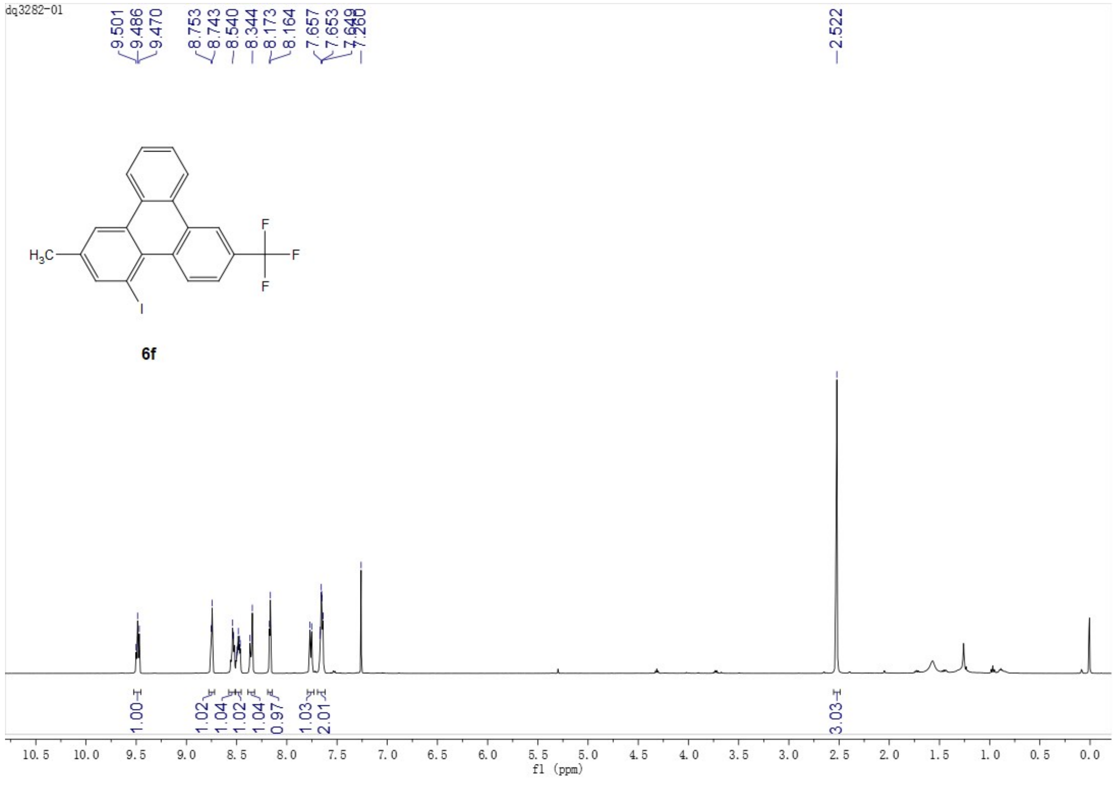

dq3331-29
9.350
9.328
8.485
8.465
8.460
8.442
8.349
8.148
7.643
7.634
7.628
7.619
7.525
7.520
7.502
7.498
7.260
2.515
H$_3$C
Cl
I
1.01
3.09
1.03
1.01
2.00
1.00
3.07
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

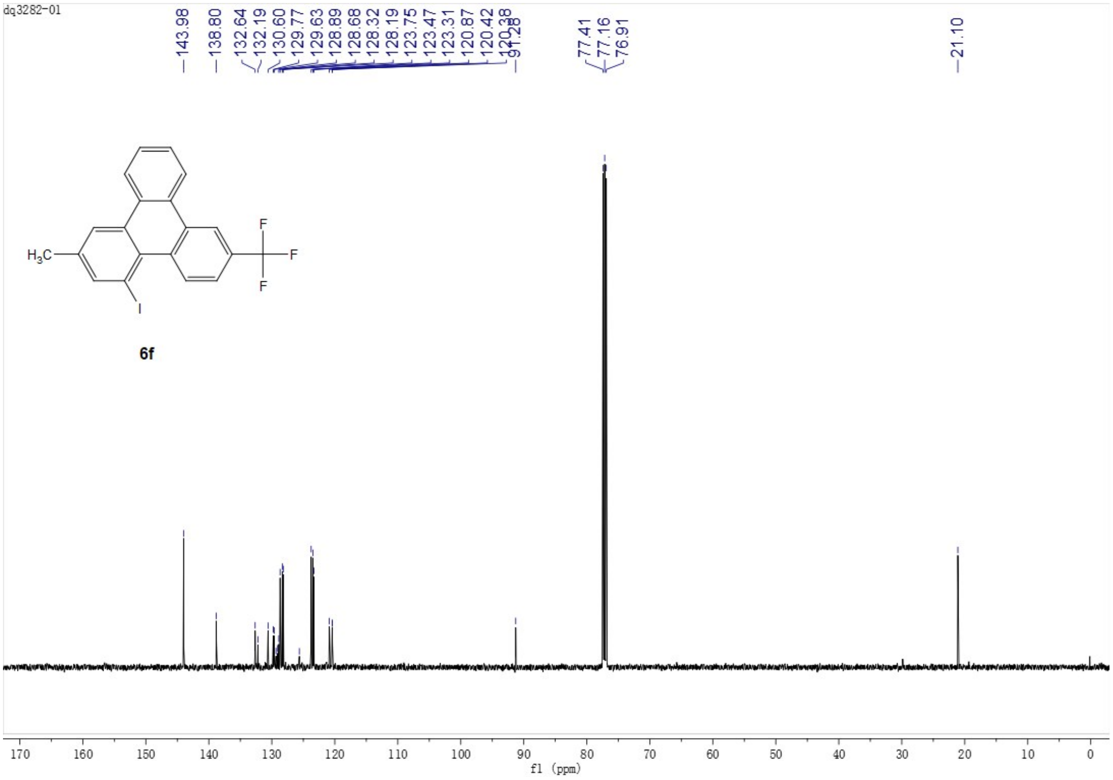
6d

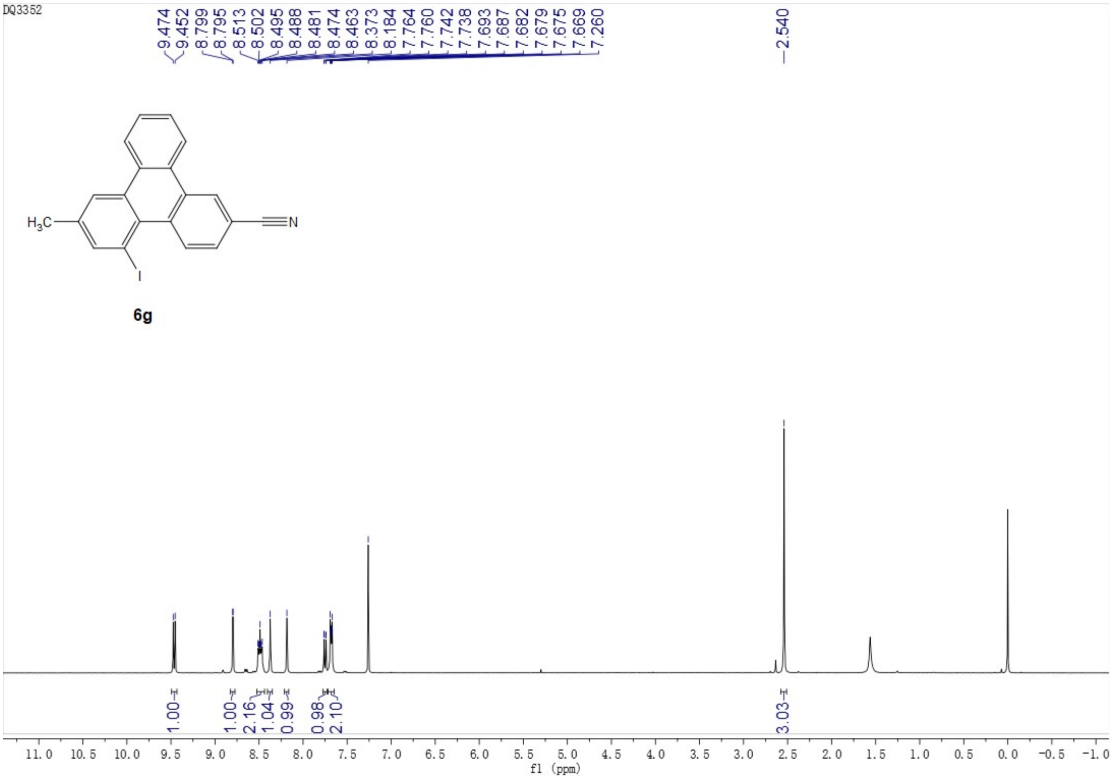

dq3331-13c-1228
143.73
137.81
133.57
132.06
131.72
129.71
129.33
129.18
129.04
128.03
127.96
127.83
124.86
123.61
123.31
123.17
122.73
90.71
77.41
77.16
76.91
20.98
H3C
Cl
I
6d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

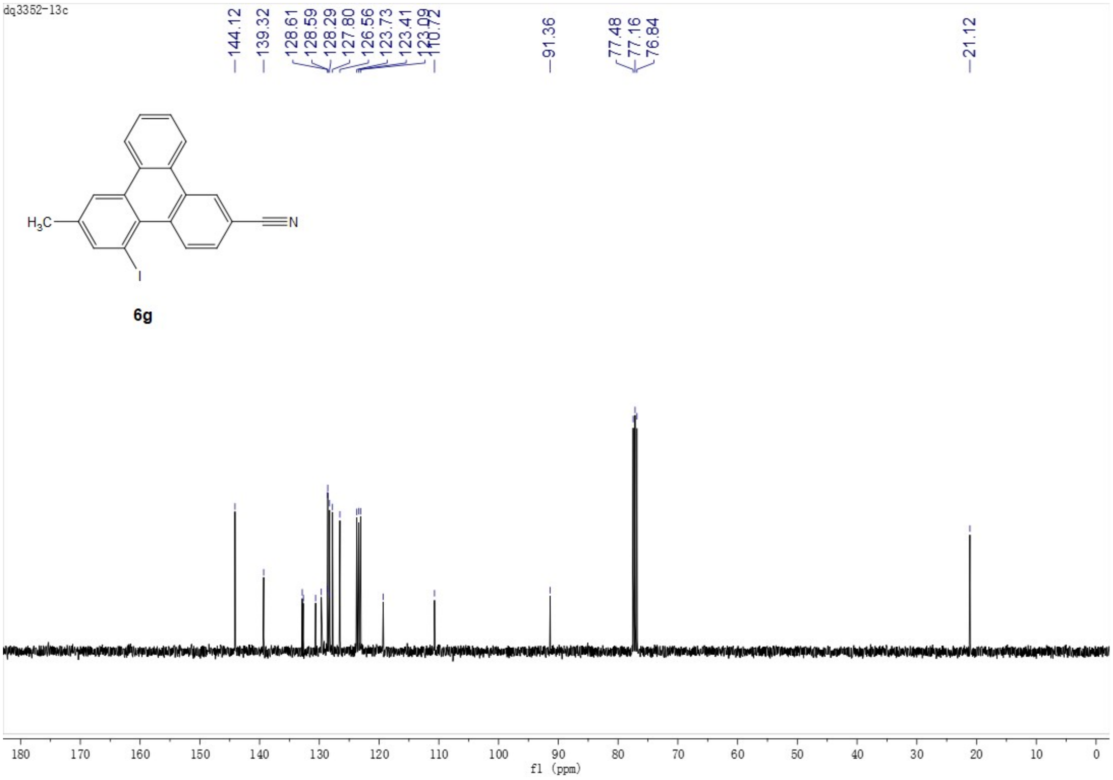

dq3340
9.373
9.350
8.512
8.496
8.488
8.472
8.356
8.134
7.949
7.943
7.628
7.621
7.614
7.605
7.260
7.205
7.198
7.182
7.175
4.022
2.509
CH3
O
H3C
I
6e
1.00
2.14
1.10
1.02
1.04
2.18
1.09
3.04
3.08
.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

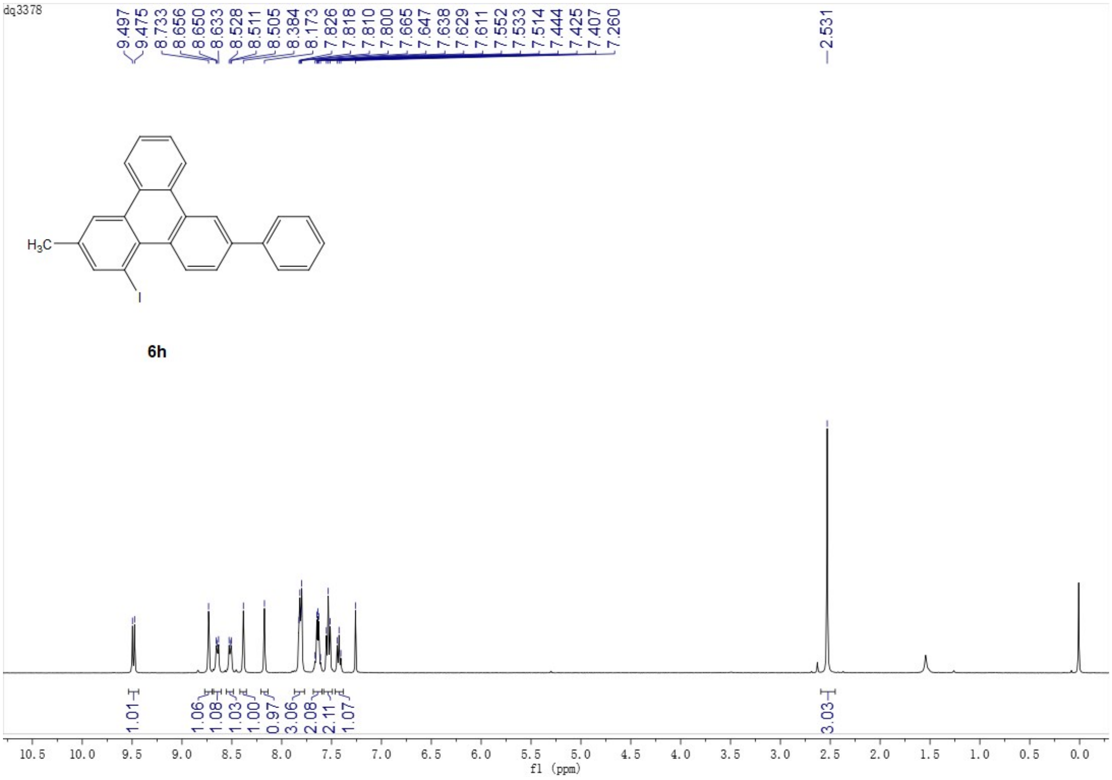

dq3340
159.08
143.60
136.74
132.39
130.92
130.17
129.64
127.64
127.61
123.74
123.64
123.46
112.56
112.50
106.17
90.26
77.48
77.16
76.84
55.60
20.96
CH3
H3C
O
I
6e
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
−200
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

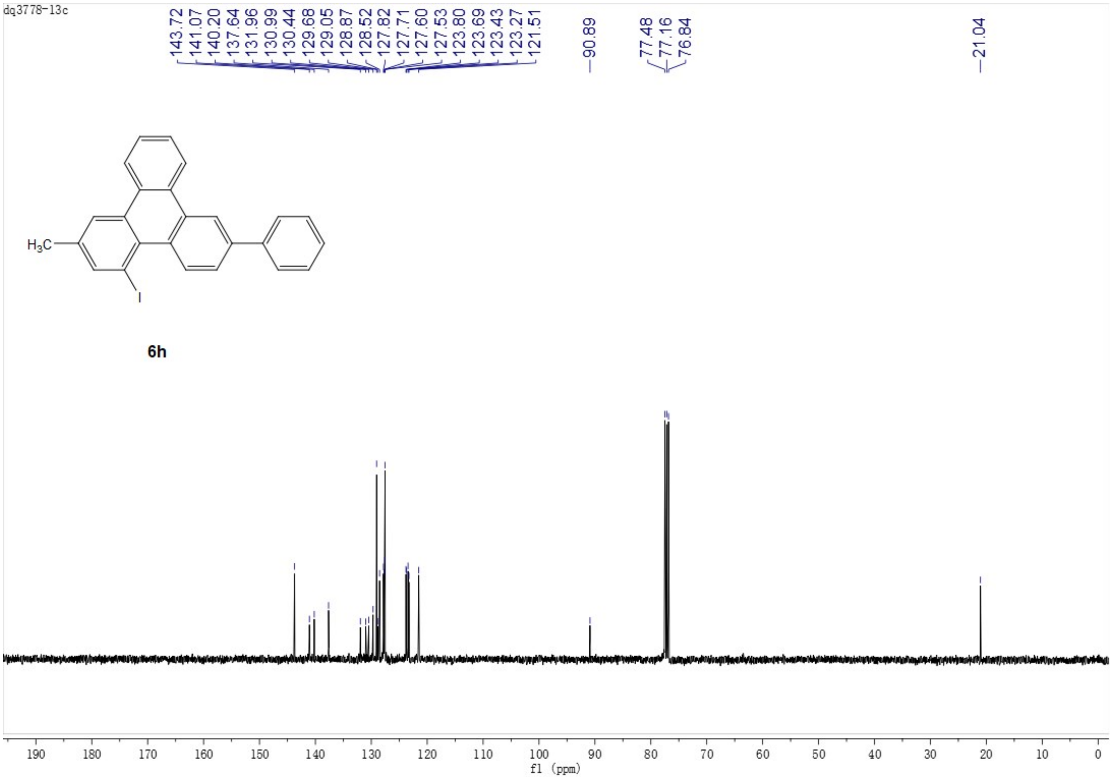

dq3282-01
9.501
9.486
9.470
8.753
8.743
8.540
8.344
8.173
8.164
7.657
7.653
7.649
7.260
2.522
H3C
F
F
F
I
6f
1.00
1.02
1.04
1.02
1.04
0.97
1.03
2.01
3.03
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


dq3282-01
143.98
138.80
132.64
132.19
130.60
129.77
129.63
128.89
128.68
128.32
128.19
123.75
123.47
123.31
120.87
120.42
120.38
91.28
77.41
77.16
76.91
21.10
H$_3$C
F
F
F
I
6f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

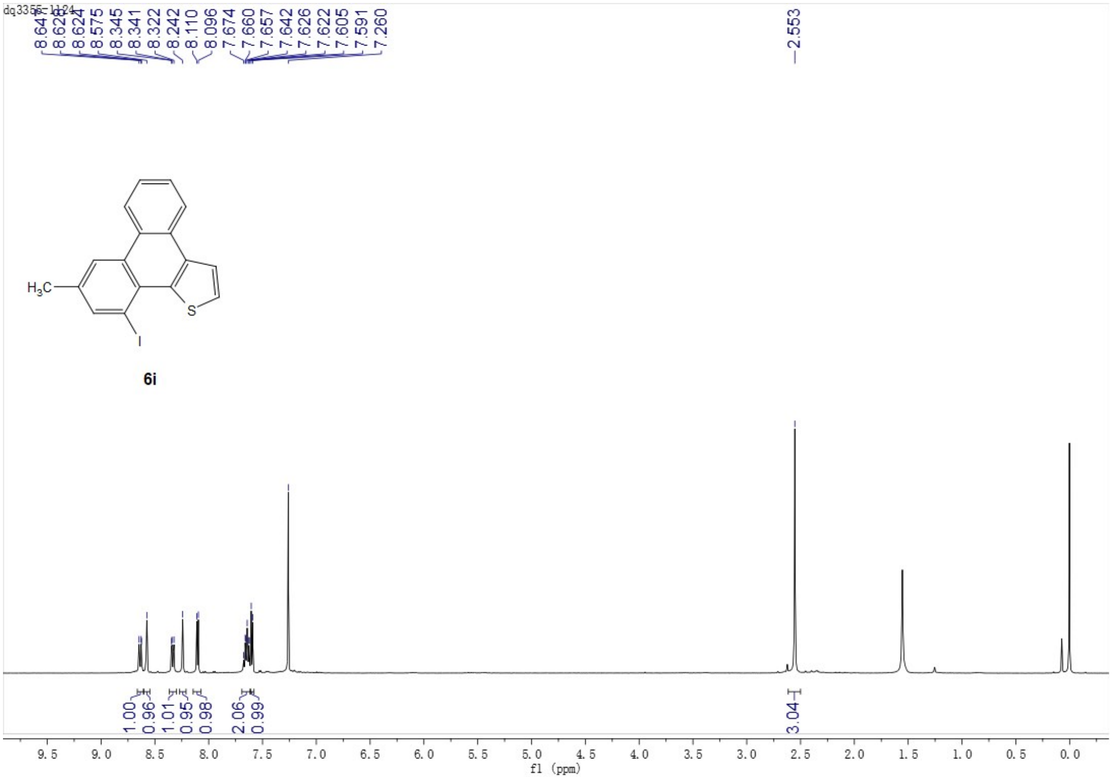

DQ3352
9.474
9.452
8.799
8.795
8.513
8.502
8.495
8.488
8.481
8.474
8.463
8.373
8.184
7.764
7.760
7.742
7.738
7.693
7.687
7.682
7.679
7.675
7.669
7.260
2.540
H$_3$C
N
I
6g
1.00
1.00
2.16
1.04
0.99
0.98
2.10
3.03
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


dq3352-13c
144.12
139.32
128.61
128.59
128.29
127.80
126.56
123.73
123.41
123.09
110.72
91.36
77.48
77.16
76.84
21.12
H3C
N
I
6g
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

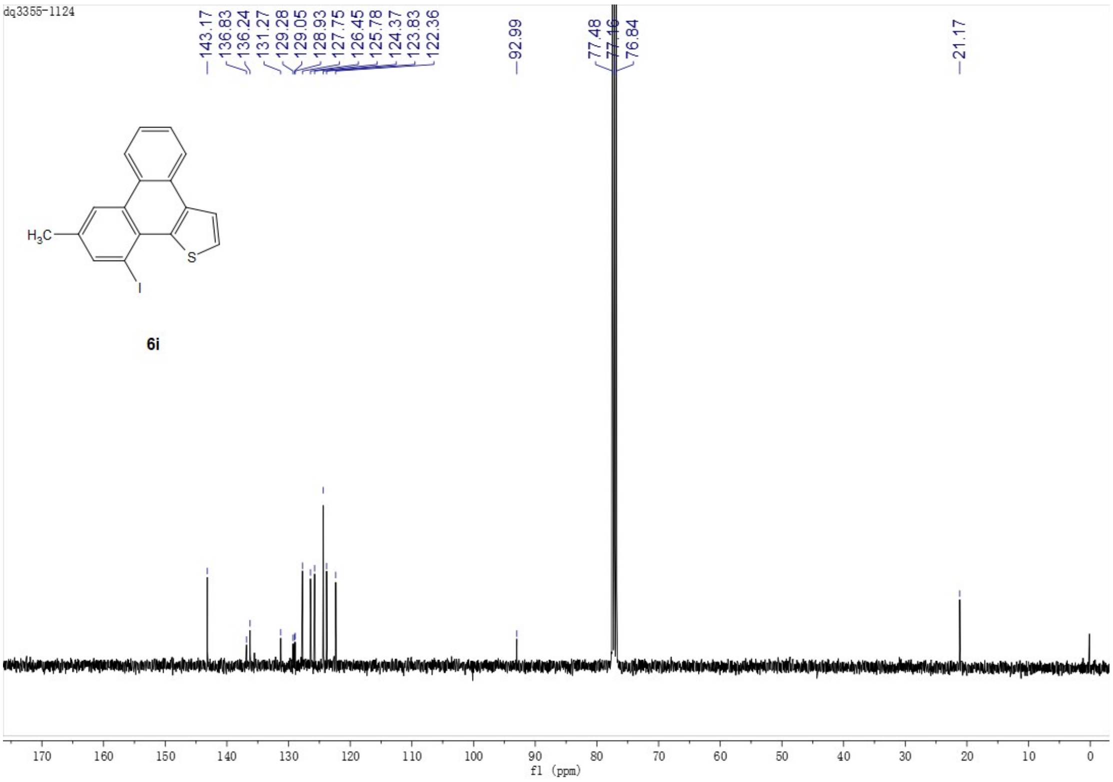

dq3378
9.497
9.475
8.733
8.656
8.650
8.633
8.528
8.511
8.505
8.384
8.173
7.826
7.818
7.810
7.800
7.665
7.647
7.638
7.629
7.611
7.552
7.533
7.514
7.444
7.425
7.407
7.260
2.531
H3C
I
6h
1.01
1.06
1.08
1.03
1.00
0.97
3.06
2.08
2.11
1.07
3.03
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


dq3778-13c
143.72
141.07
140.20
137.64
131.96
130.99
130.44
129.68
129.05
128.87
128.52
127.82
127.71
127.60
127.53
123.80
123.69
123.43
123.27
121.51
90.89
77.48
77.16
76.84
21.04
H3C
I
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

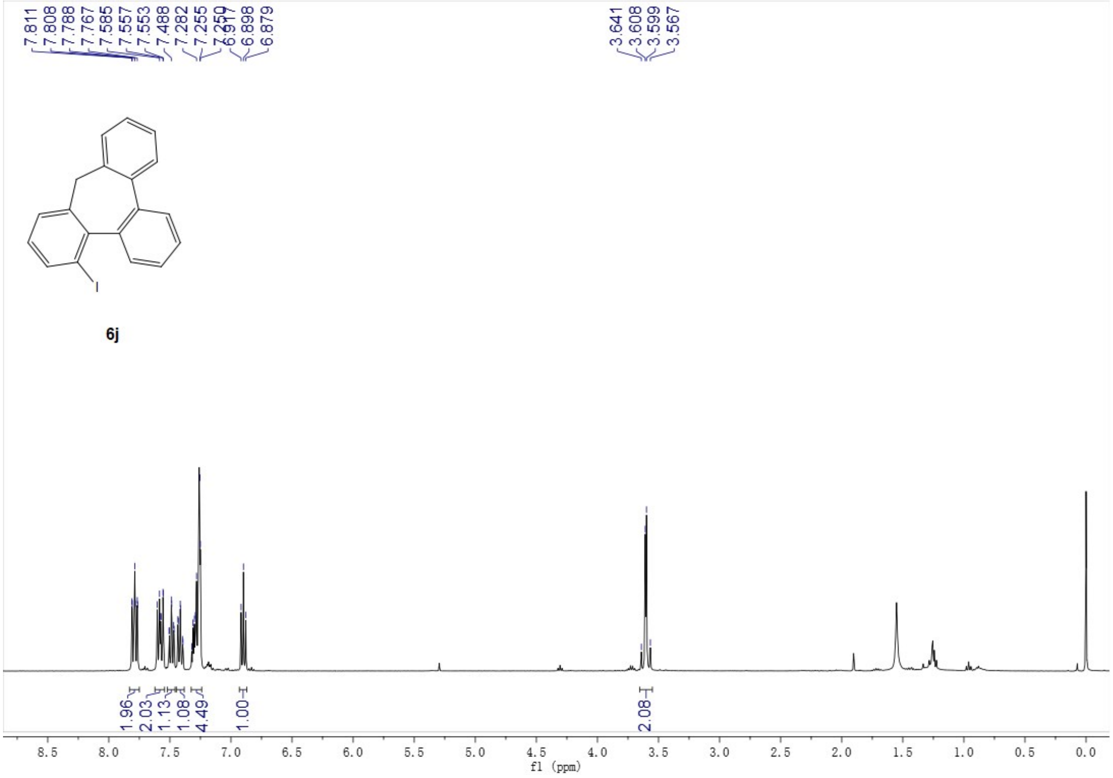
6h


dq3355-1124
8.641
8.626
8.624
8.575
8.345
8.341
8.322
8.242
8.110
8.096
7.674
7.660
7.657
7.642
7.626
7.622
7.605
7.591
7.260
2.553
H3C
S
I
1.00
0.96
1.01
0.95
0.98
2.06
0.99
3.04
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
f1 (ppm)

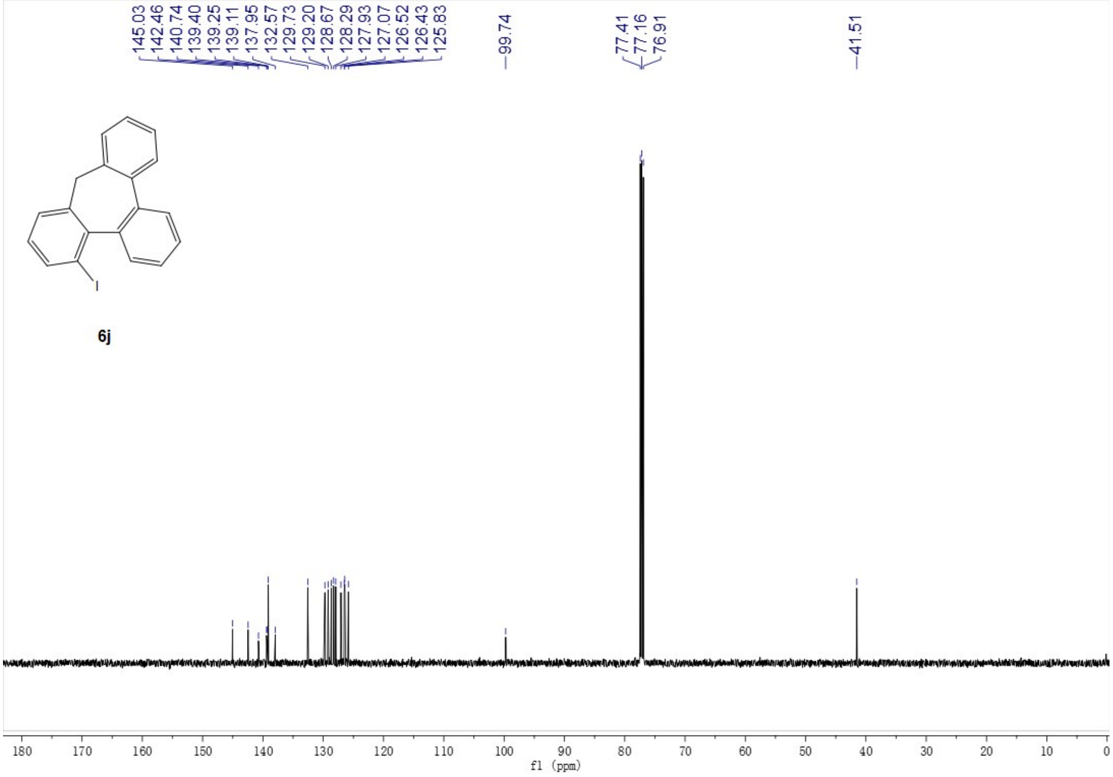
6i


dq3355-1124
143.17
136.83
136.24
131.27
129.28
129.05
128.93
127.75
126.45
125.78
124.37
123.83
122.36
92.99
77.48
77.16
76.84
21.17
H$_3$C
S
I
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

6i

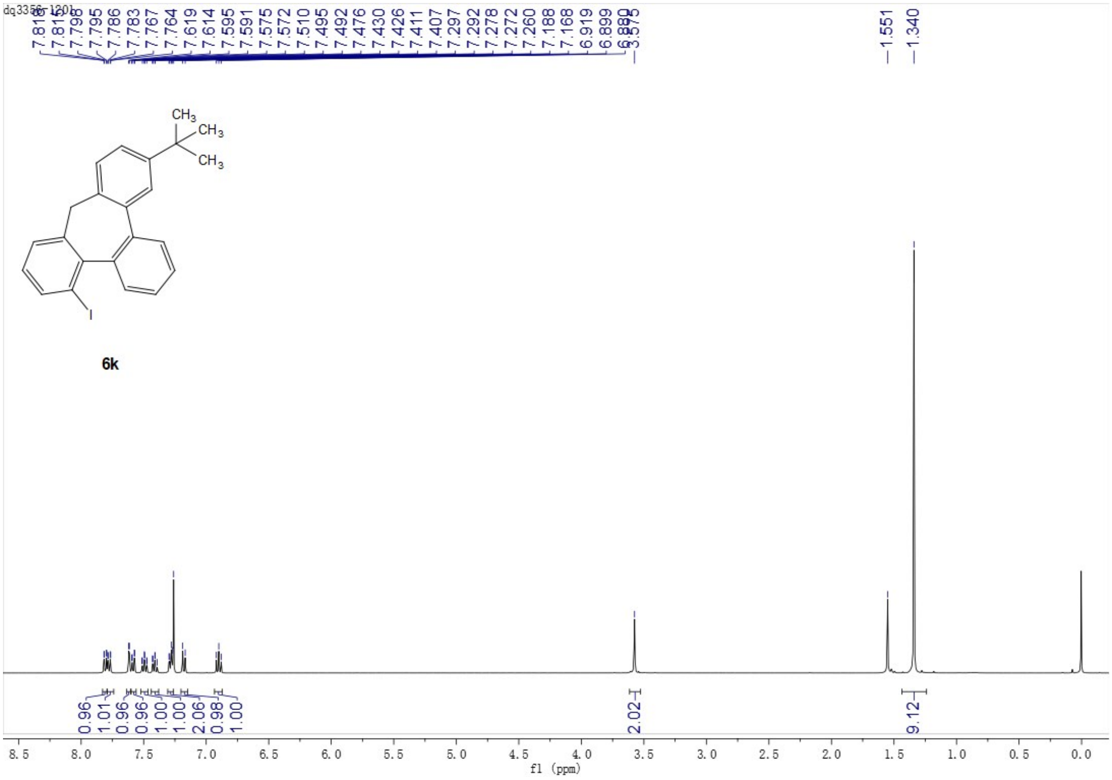

7.811
7.808
7.788
7.767
7.585
7.557
7.553
7.488
7.282
7.255
7.250
6.917
6.898
6.879
3.641
3.608
3.599
3.567
I
1.96
2.03
1.13
1.08
4.49
1.00
2.08
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

6j



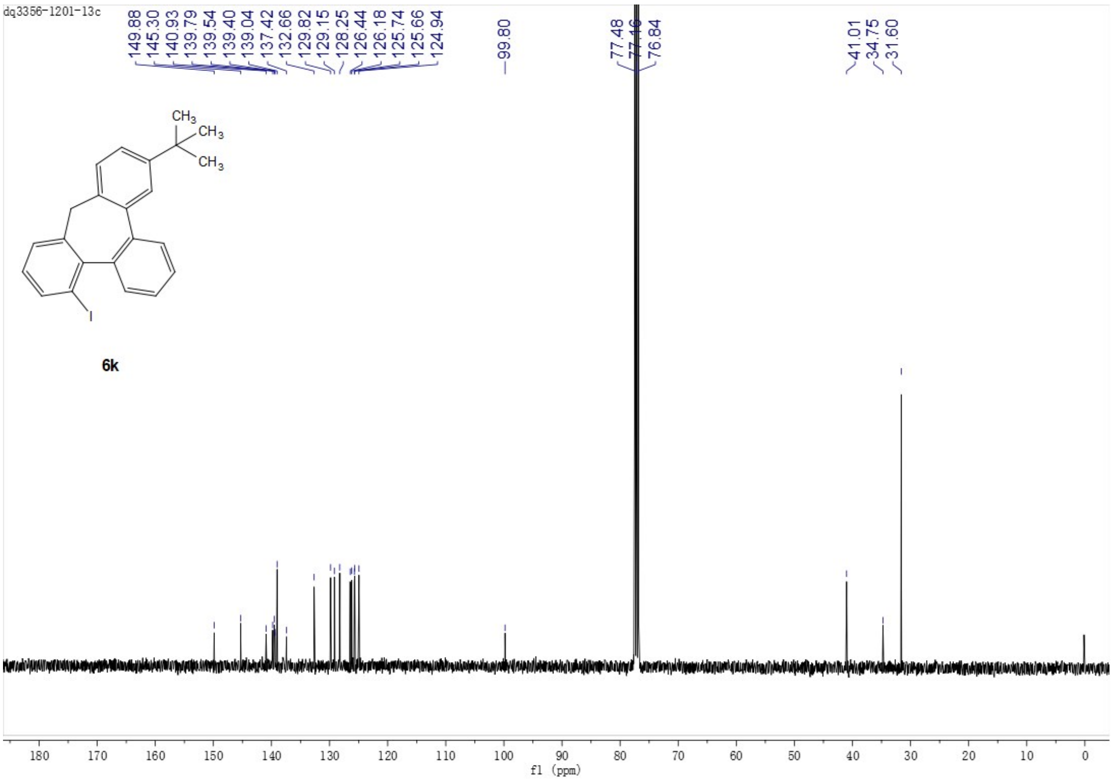
6j

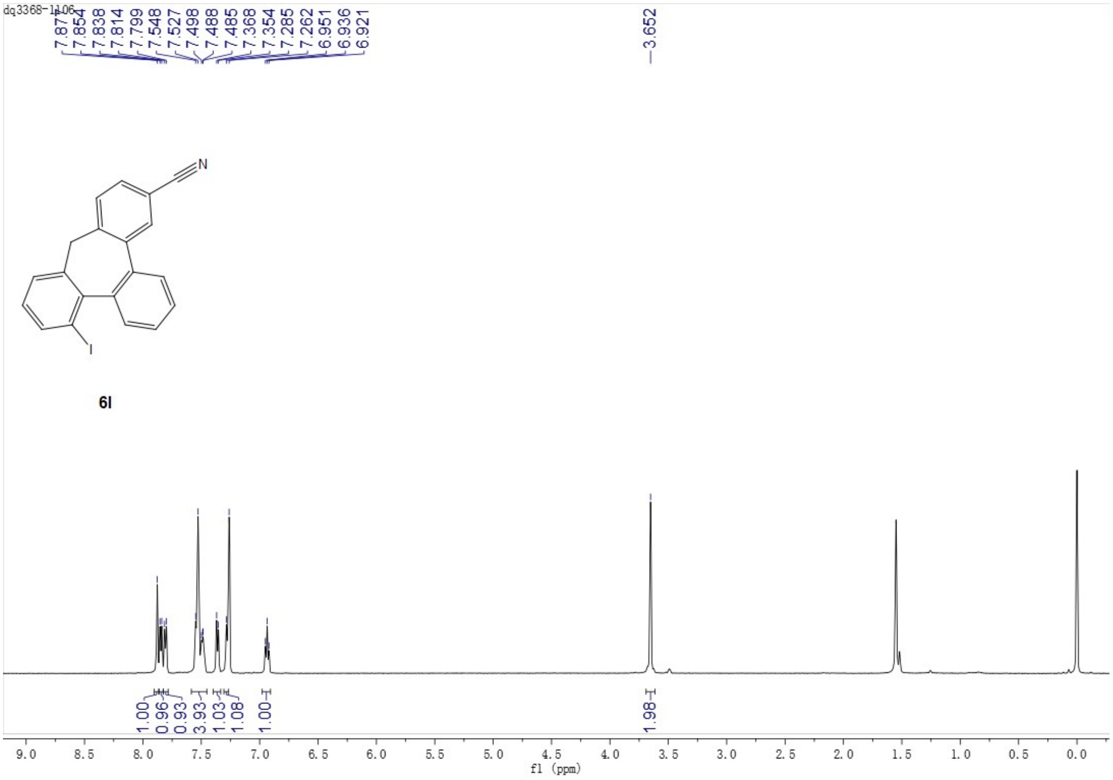
145.03
142.46
140.74
139.40
139.25
139.11
137.95
132.57
129.73
129.20
128.67
128.29
127.93
127.07
126.52
126.43
125.83
99.74
77.41
77.16
76.91
41.51
I
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

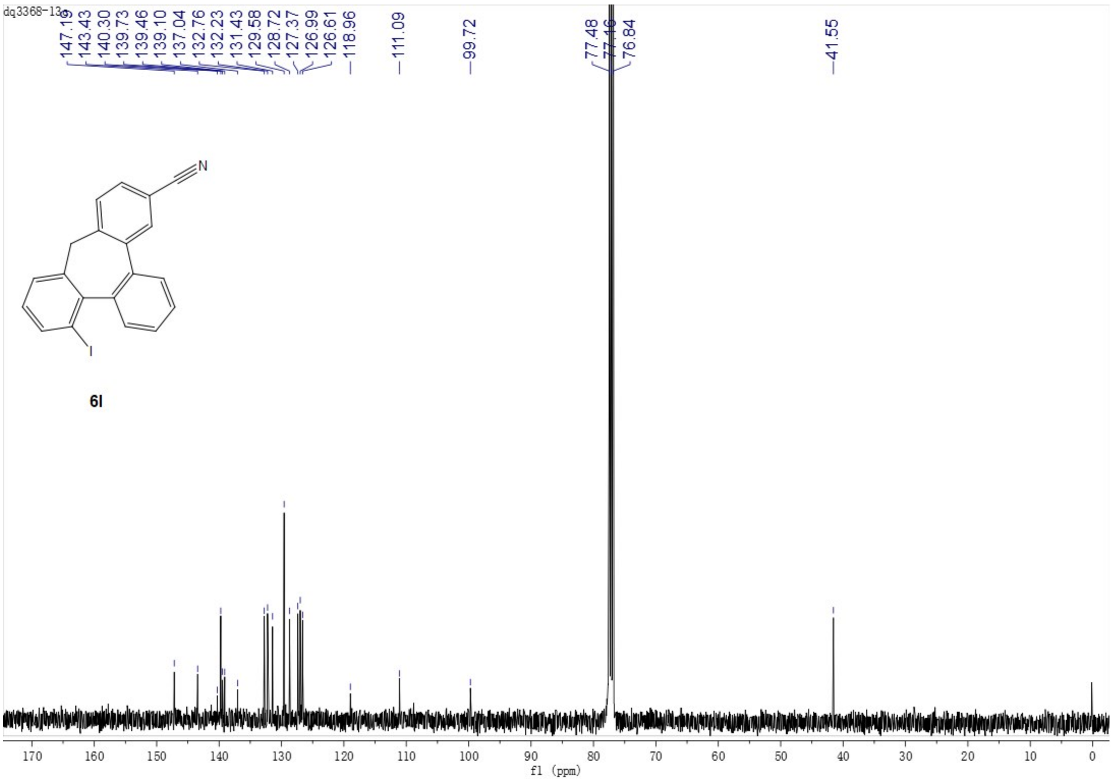


6k

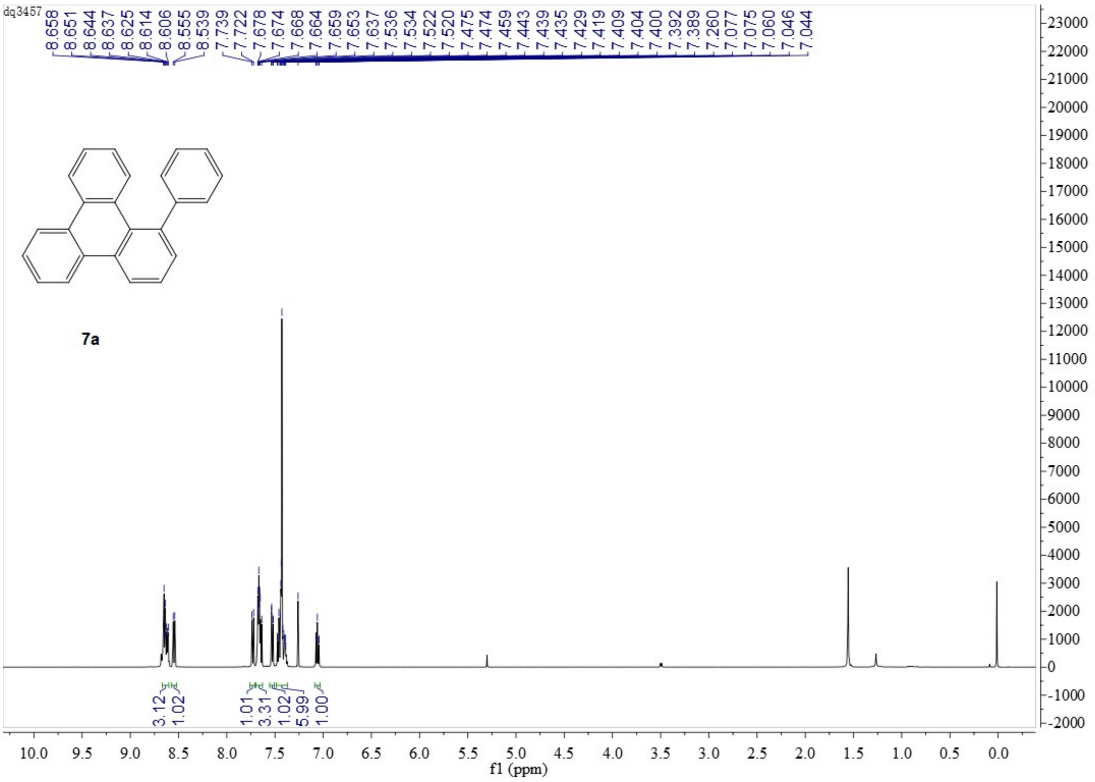
dq3356-1201
7.816
7.81[illegible]
7.798
7.795
7.786
7.783
7.767
7.764
7.619
7.614
7.595
7.591
7.575
7.572
7.510
7.495
7.492
7.476
7.430
7.426
7.411
7.407
7.297
7.292
7.278
7.272
7.260
7.188
7.168
6.919
6.899
6.880
3.575
1.551
1.340
CH3
CH3
CH3
I
0.96
1.01
0.96
0.96
1.00
1.00
2.06
0.98
1.00
2.02
9.12
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
f1 (ppm)


dq3356-1201-13c
149.88
145.30
140.93
139.79
139.54
139.40
139.04
137.42
132.66
129.82
129.15
128.25
126.44
126.18
125.74
125.66
124.94
99.80
77.48
77.16
76.84
41.01
34.75
31.60
CH3
CH3
CH3
I
6k
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

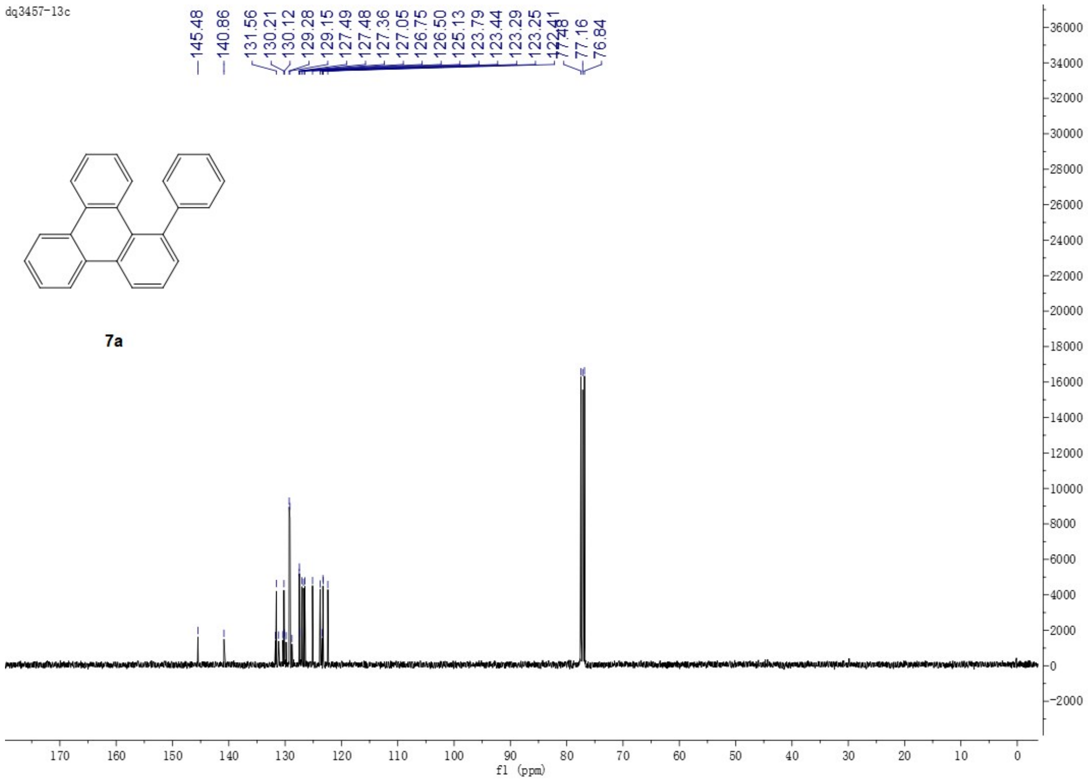

dq3368-1106
7.877
7.854
7.838
7.814
7.799
7.548
7.527
7.498
7.488
7.485
7.368
7.354
7.285
7.262
6.951
6.936
6.921
3.652
N
I
6l
1.00
0.96
0.93
3.93
1.03
1.08
1.00
1.98
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

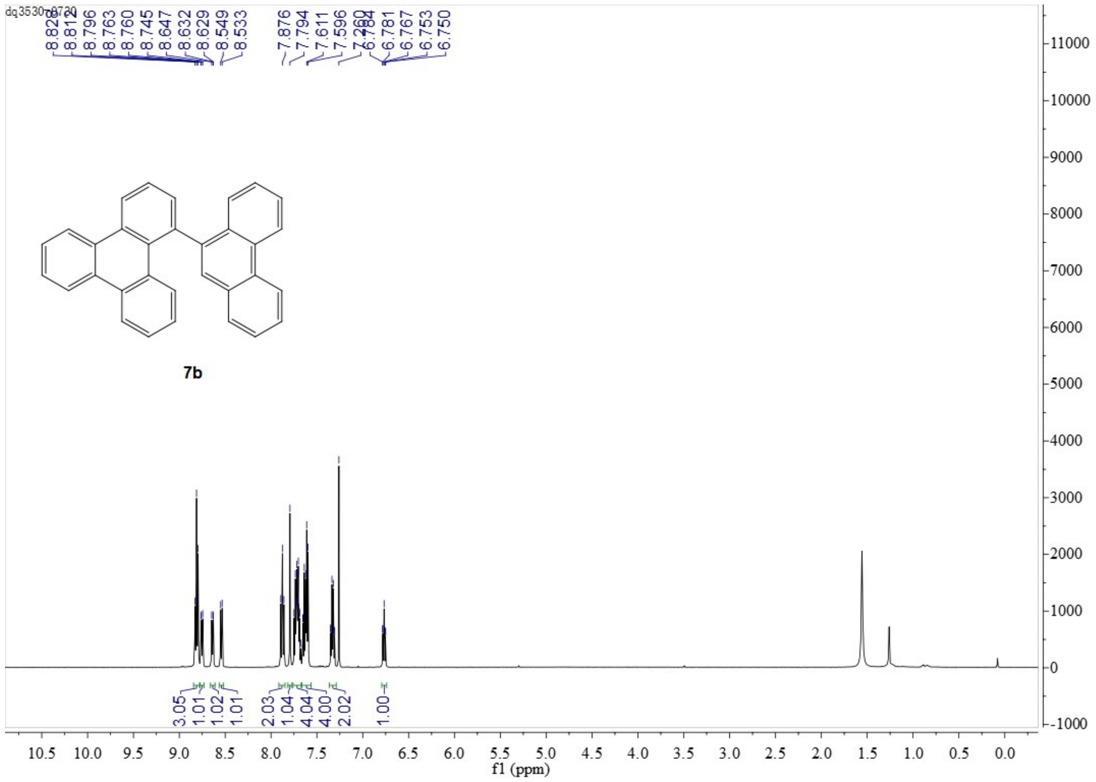

dq3368-13c
147.19
143.43
140.30
139.73
139.46
139.10
137.04
132.76
132.23
131.43
129.58
128.72
127.37
126.99
126.61
118.96
111.09
99.72
77.48
77.16
76.84
41.55
N
I
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

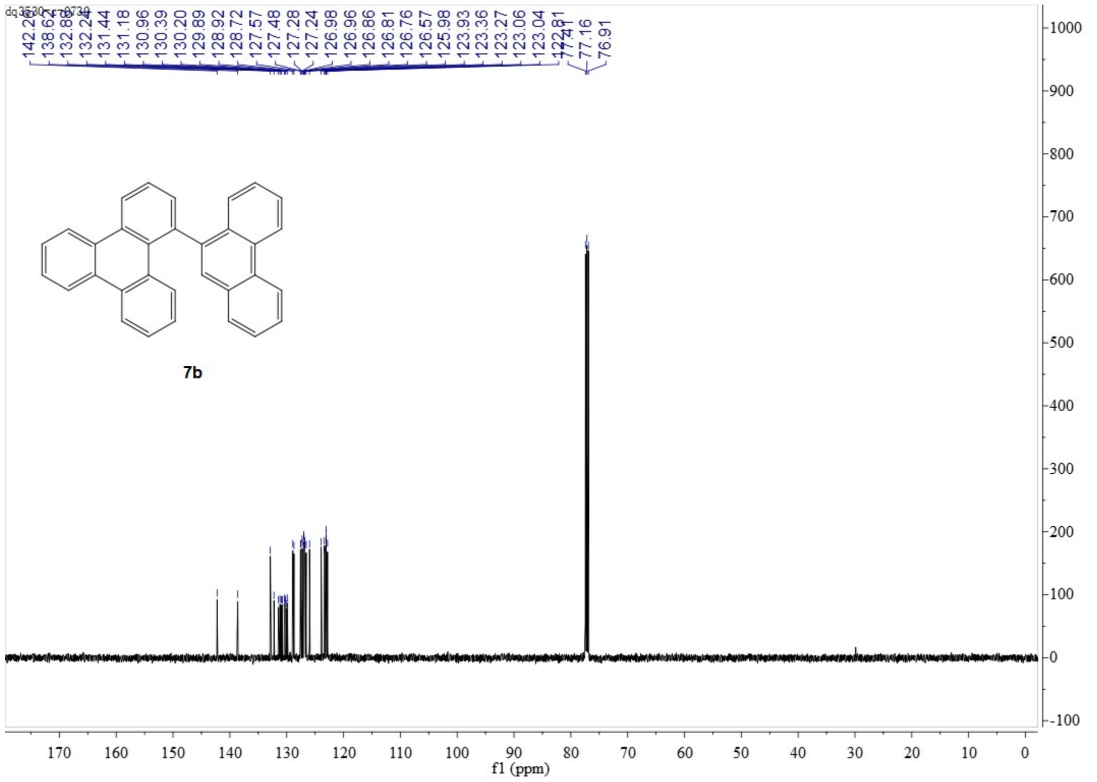
6l


dq3457
8.658
8.651
8.644
8.637
8.625
8.614
8.606
8.555
8.539
7.739
7.722
7.678
7.674
7.668
7.664
7.659
7.653
7.637
7.536
7.534
7.522
7.520
7.475
7.474
7.459
7.443
7.439
7.435
7.429
7.419
7.409
7.404
7.400
7.392
7.389
7.260
7.077
7.075
7.060
7.046
7.044
3.12
1.02
1.01
3.31
1.02
5.99
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
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

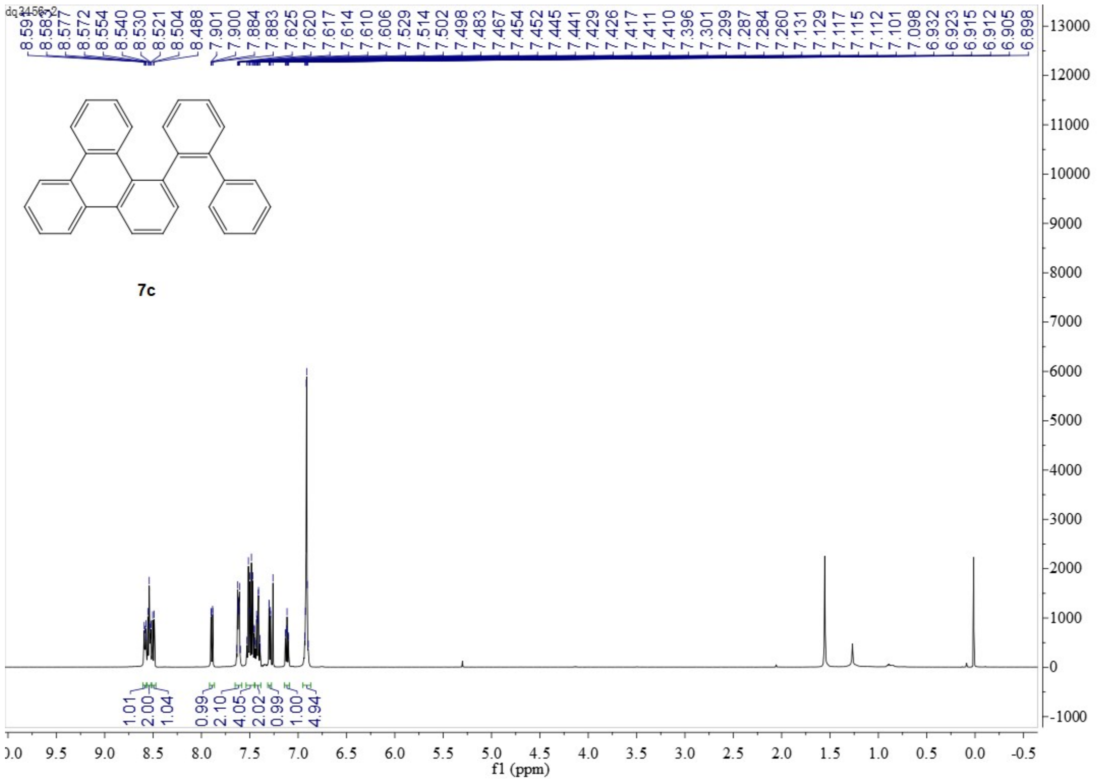
7a


dq3457-13c
145.48
140.86
131.56
130.21
130.12
129.28
129.15
127.49
127.48
127.36
127.05
126.75
126.50
125.13
123.79
123.44
123.29
123.25
122.41
77.48
77.16
76.84
7a
36000
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

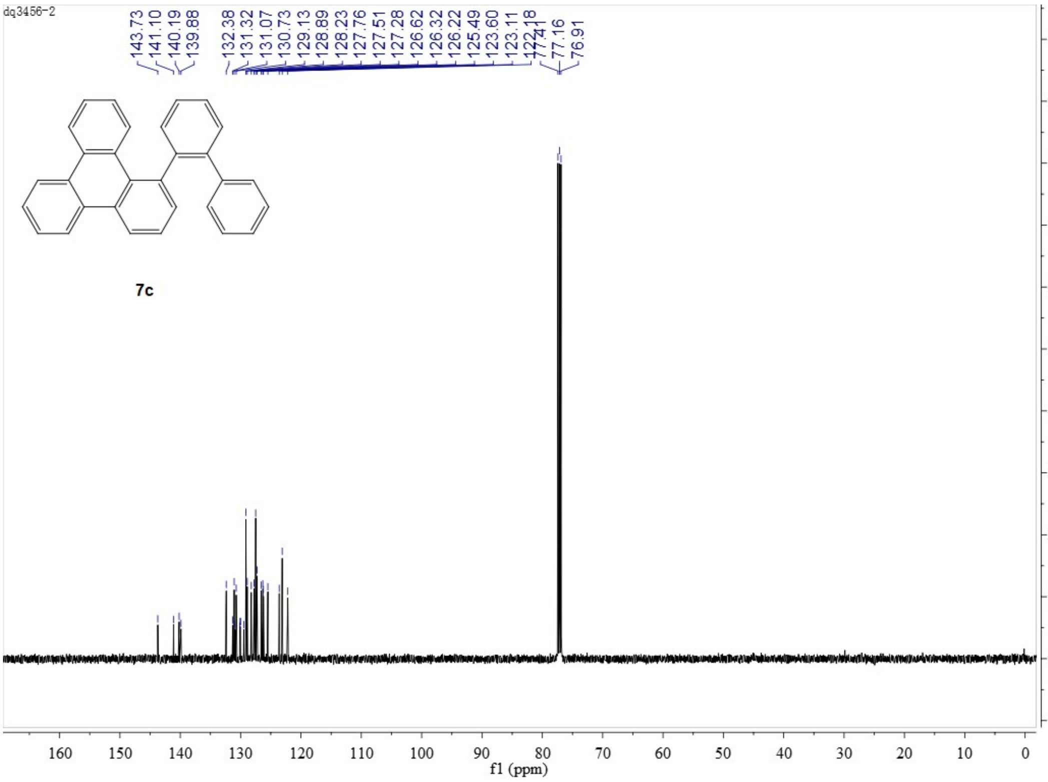

dq3530-0720
8.828
8.812
8.796
8.763
8.760
8.745
8.647
8.632
8.629
8.549
8.533
7.876
7.794
7.611
7.596
7.260
6.784
6.781
6.767
6.753
6.750
7b
3.05
1.01
1.02
1.01
2.03
1.04
4.04
4.00
2.02
1.00
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


dq3530-c-0730
142.26
138.62
132.86
132.24
131.44
131.18
130.96
130.39
130.20
129.89
128.92
128.72
127.57
127.48
127.28
127.24
126.98
126.96
126.86
126.81
126.76
126.57
125.98
123.93
123.36
123.27
123.06
123.04
122.81
77.41
77.16
76.91
1000
900
800
700
600
500
400
300
200
100
0
-100
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

7b

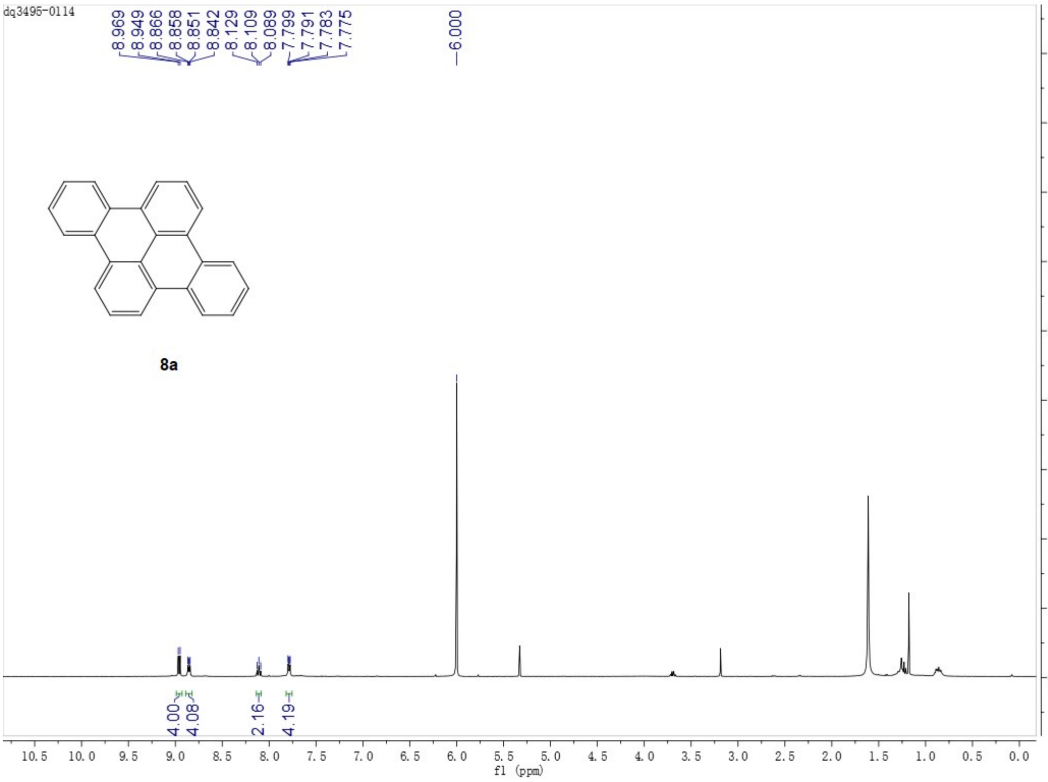

dq3456-2
8.591
8.587
8.577
8.572
8.554
8.540
8.530
8.521
8.504
8.488
7.901
7.900
7.884
7.883
7.625
7.620
7.617
7.614
7.610
7.606
7.529
7.514
7.502
7.498
7.483
7.467
7.454
7.452
7.445
7.441
7.429
7.426
7.417
7.411
7.410
7.396
7.301
7.299
7.287
7.284
7.260
7.131
7.129
7.117
7.115
7.112
7.101
7.098
6.932
6.923
6.915
6.912
6.905
6.898
1.01
2.00
1.04
0.99
2.10
4.05
2.02
0.99
1.00
4.94
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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)

7c



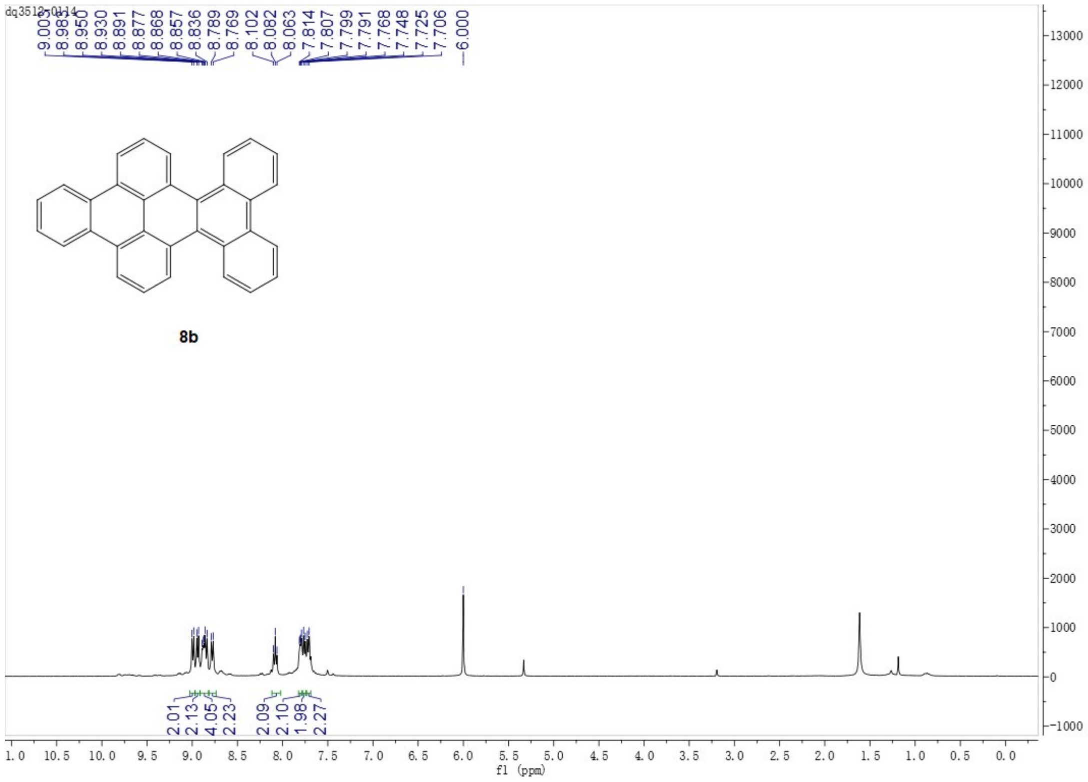
7c

dq3456-2
143.73
141.10
140.19
139.88
132.38
131.32
131.07
130.73
129.13
128.89
128.23
127.76
127.51
127.28
126.62
126.32
126.22
125.49
123.60
123.11
122.18
77.41
77.16
76.91
1000
900
800
700
600
500
400
300
200
100
0
-100
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

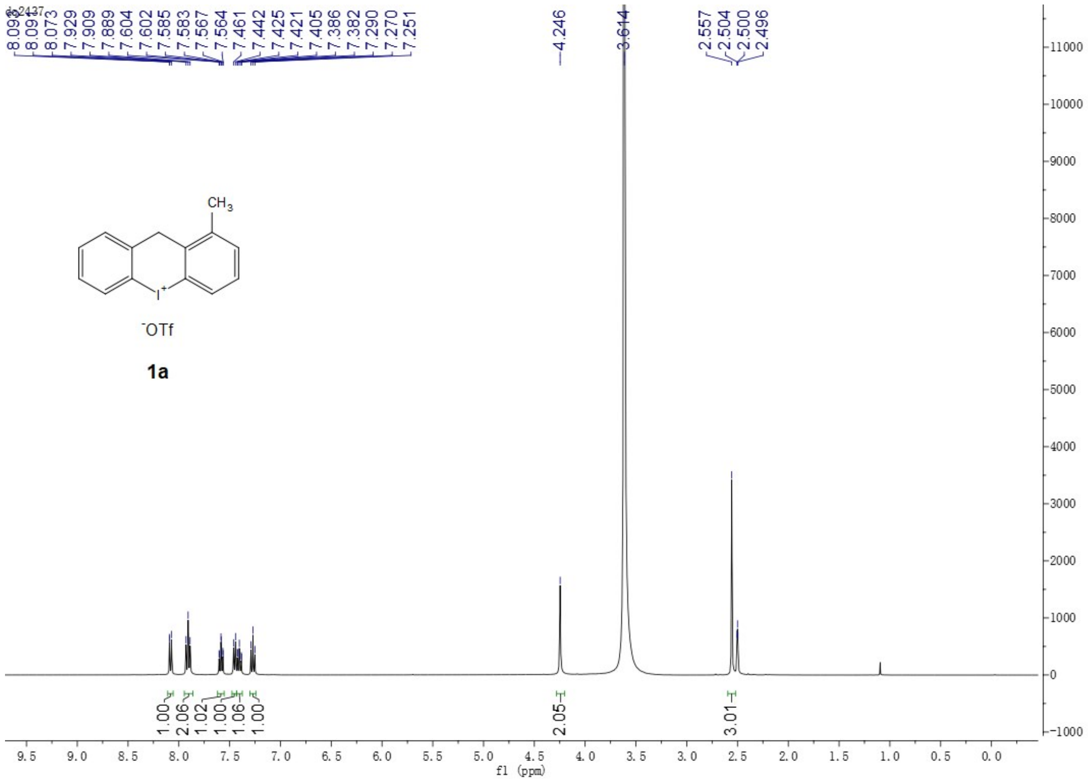


8a

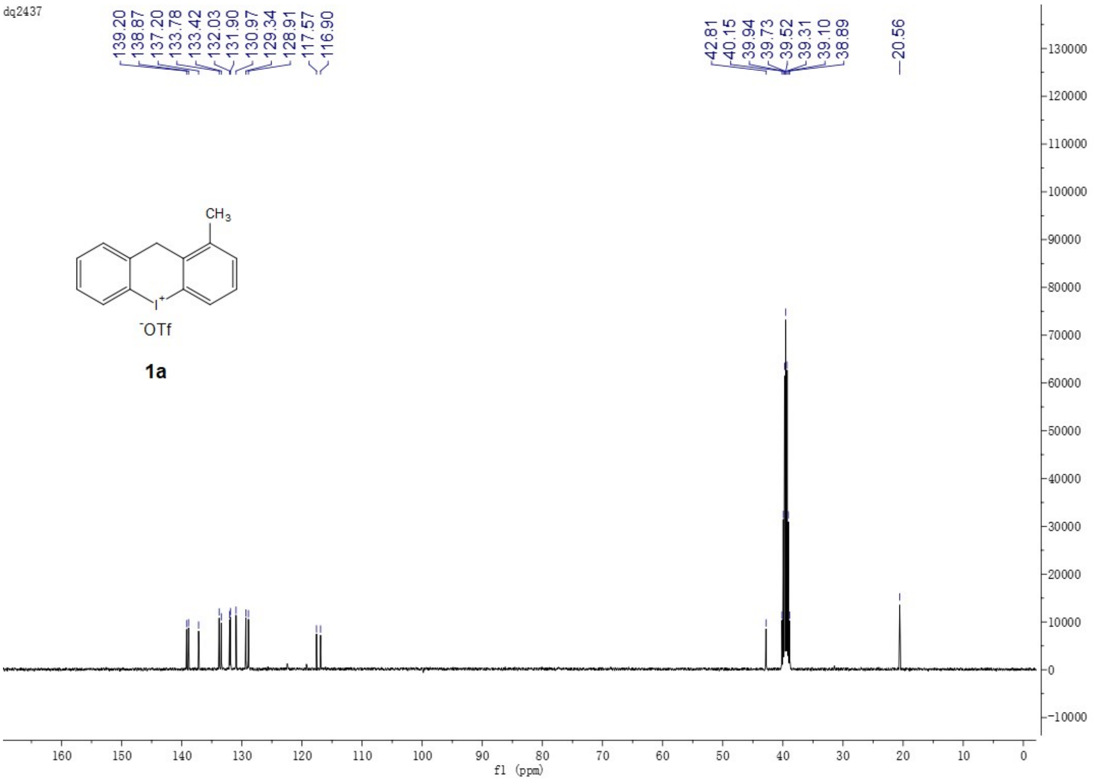
dq3495-0114
8.969
8.949
8.866
8.858
8.851
8.842
8.129
8.109
8.089
7.799
7.791
7.783
7.775
6.000
4.00
4.08
2.16
4.19
4500
4000
3500
3000
2500
2000
1500
1000
500
0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


8b

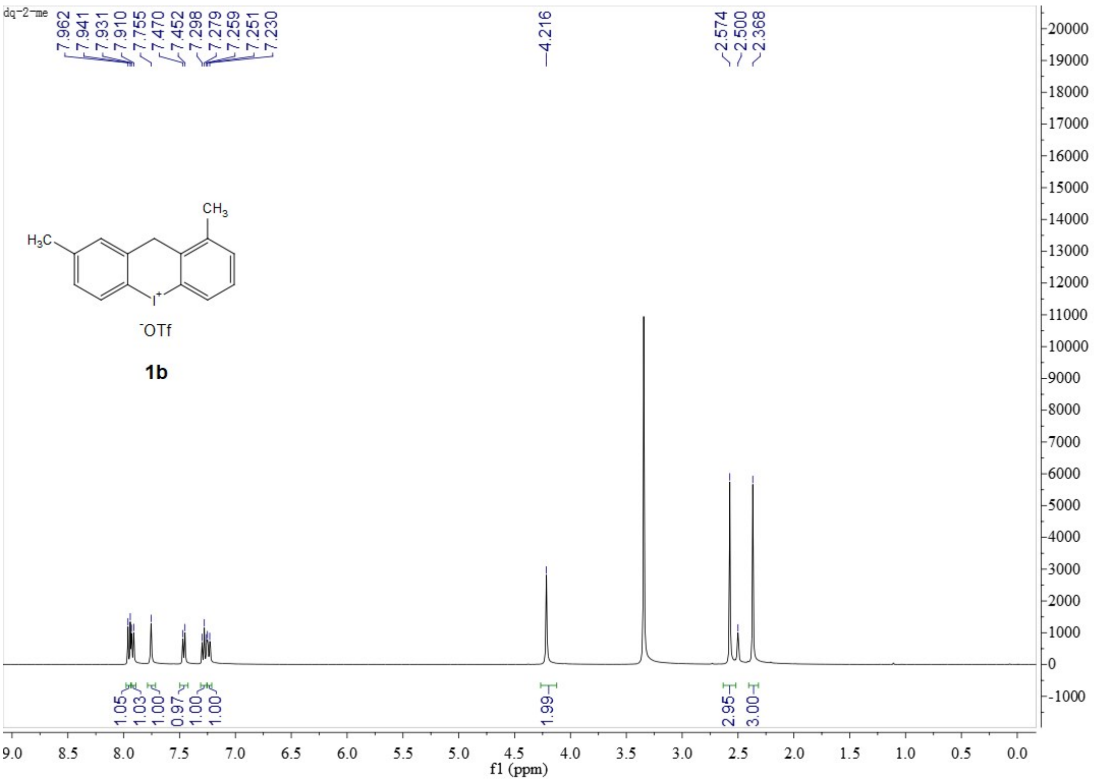
dq3512-0114
9.003
8.983
8.950
8.930
8.891
8.877
8.868
8.857
8.836
8.789
8.769
8.102
8.082
8.063
7.814
7.807
7.799
7.791
7.768
7.748
7.725
7.706
6.000
2.01
2.13
4.05
2.23
2.09
2.10
1.98
2.27
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
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
f1 (ppm)


1a

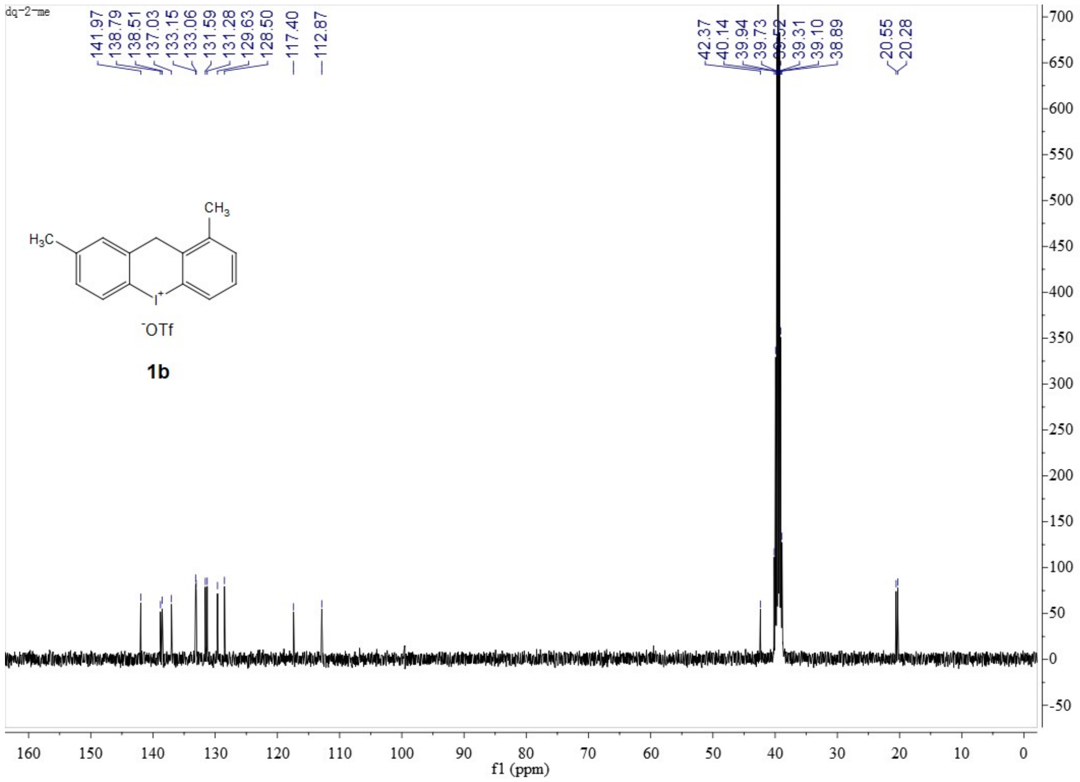
dq2437
8.093
8.091
8.073
7.929
7.909
7.889
7.604
7.602
7.585
7.583
7.567
7.564
7.461
7.442
7.425
7.421
7.405
7.386
7.382
7.290
7.270
7.251
4.246
3.614
2.557
2.504
2.500
2.496
CH3
I+
−OTf
1.00
2.06
1.02
1.00
1.06
1.00
2.05
3.01
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
9.5
9.0
8.5
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
f1 (ppm)

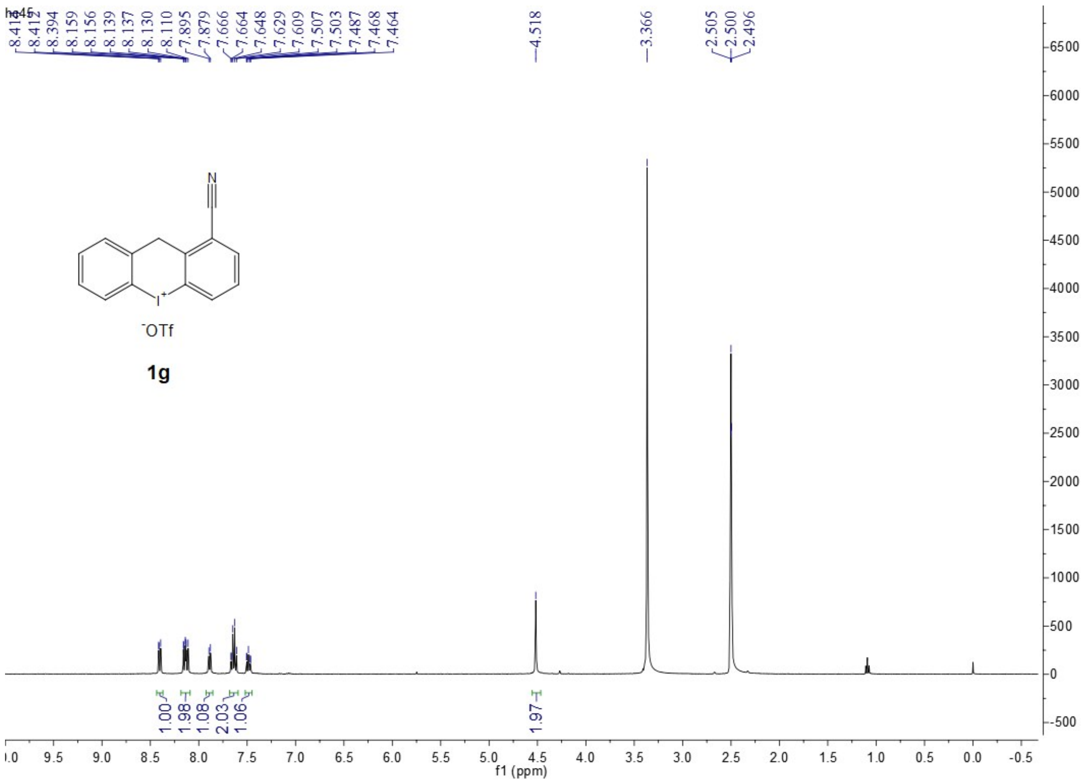

dq2437
139.20
138.87
137.20
133.78
133.42
132.03
131.90
130.97
129.34
128.91
117.57
116.90
42.81
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.56
CH3
I+
-OTf
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

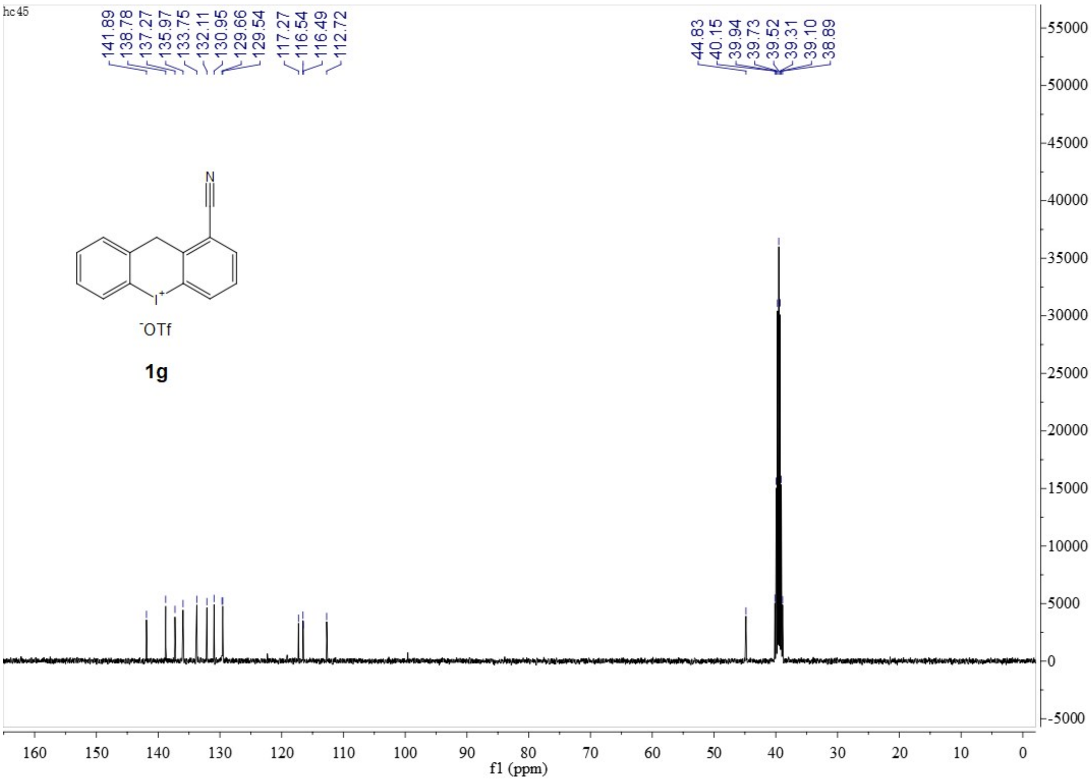
1a


dq-2-me
7.962
7.941
7.931
7.910
7.755
7.470
7.452
7.298
7.279
7.259
7.251
7.230
4.216
2.574
2.500
2.368
H3C
CH3
I+
-OTf
1.05
1.03
1.00
0.97
1.00
1.00
1.99
2.95
3.00
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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
f1 (ppm)

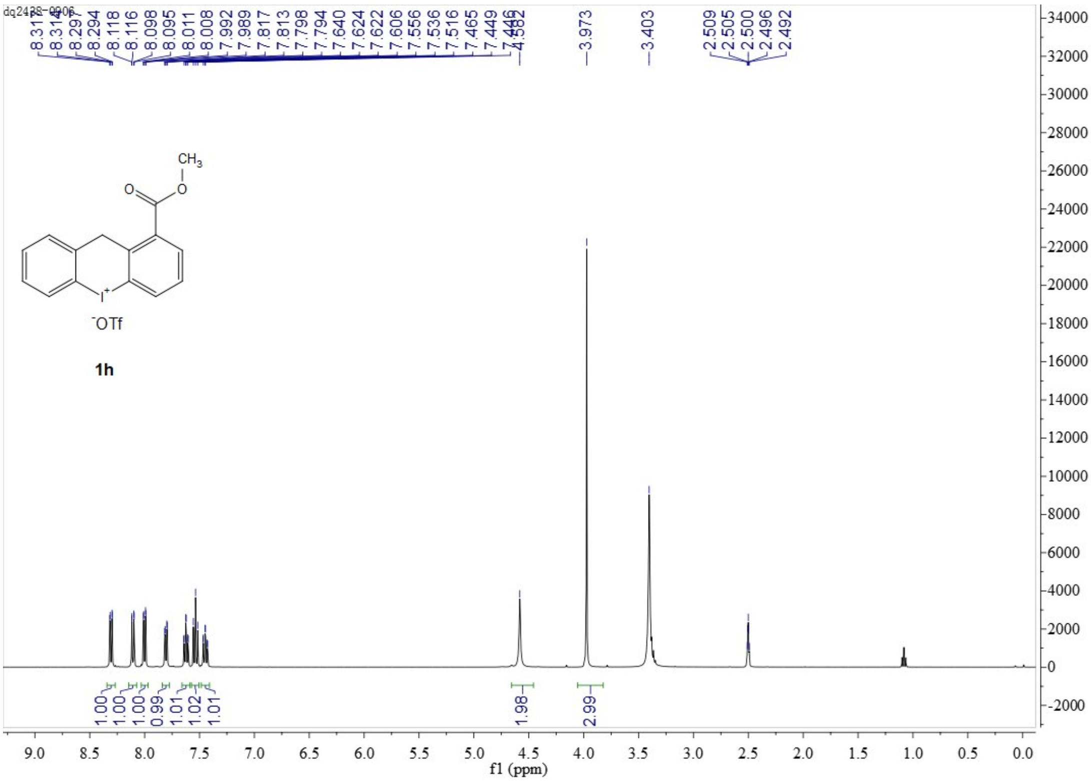
1b


dq-2-me
141.97
138.79
138.51
137.03
133.15
133.06
131.59
131.28
129.63
128.50
117.40
112.87
42.37
40.14
39.94
39.73
39.52
39.31
39.10
38.89
20.55
20.28
CH3
H3C
I+
-OTf
1b
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
700 650 600 550 500 450 400 350 300 250 200 150 100 50 0 -50

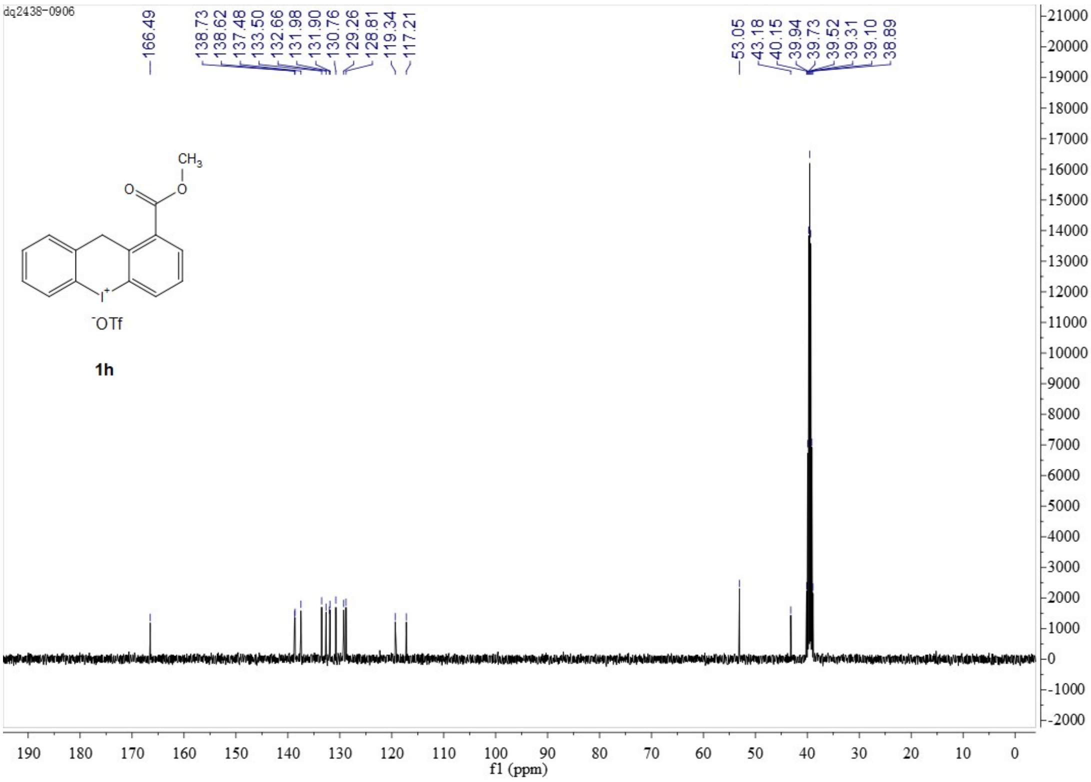

he45
8.415
8.412
8.394
8.159
8.156
8.139
8.137
8.130
8.110
7.895
7.879
7.666
7.664
7.648
7.629
7.609
7.507
7.503
7.487
7.468
7.464
4.518
3.366
2.505
2.500
2.496
N
I+
-OTf
1g
1.00
1.98
1.08
2.03
1.06
1.97
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)
6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

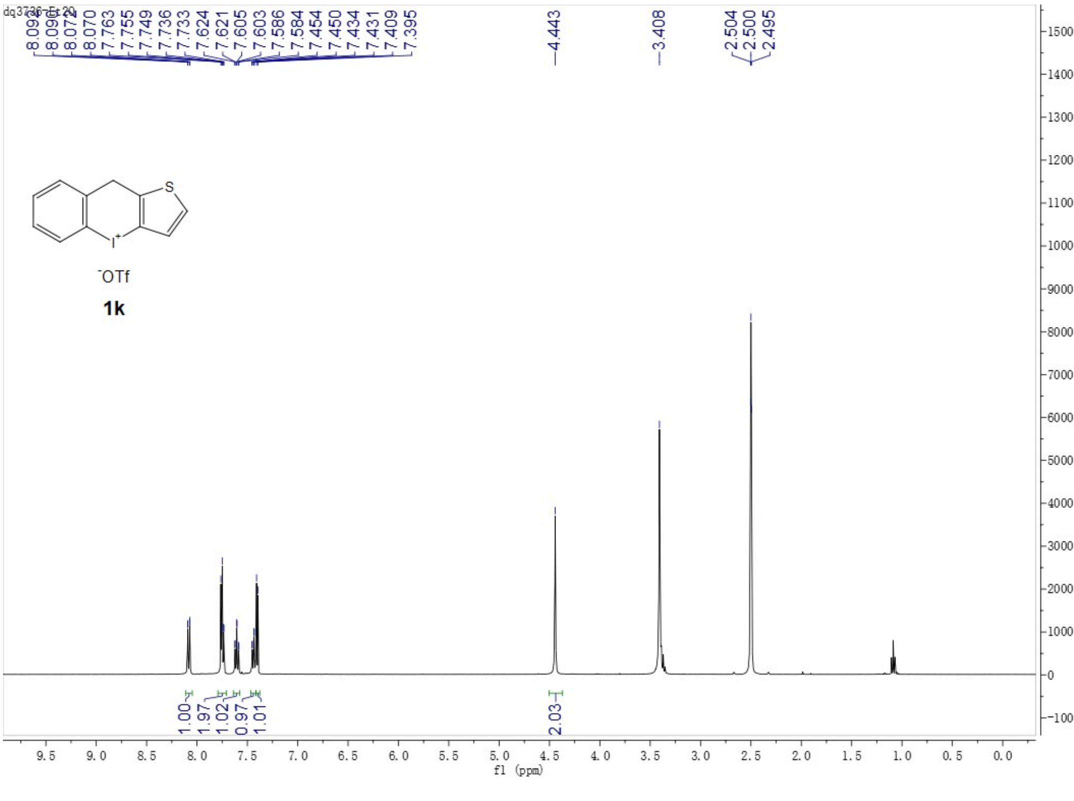

hc45
141.89
138.78
137.27
135.97
133.75
132.11
130.95
129.66
129.54
117.27
116.54
116.49
112.72
44.83
40.15
39.94
39.73
39.52
39.31
39.10
38.89
N
I+
-OTf
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

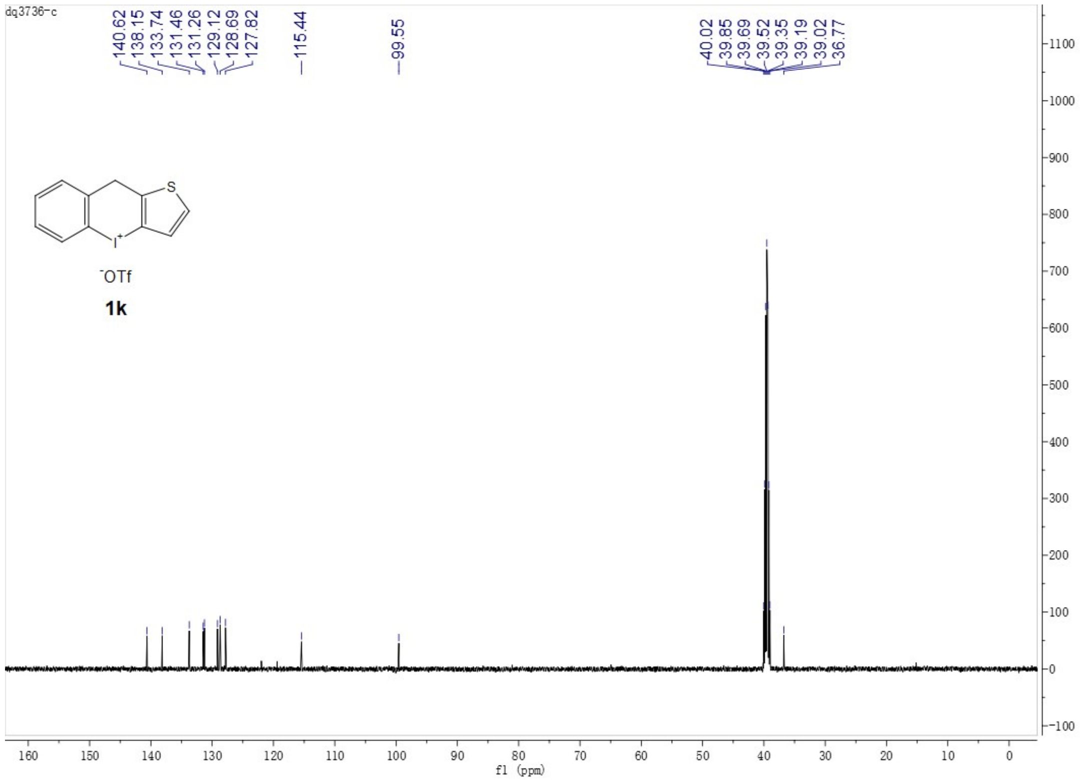
1g

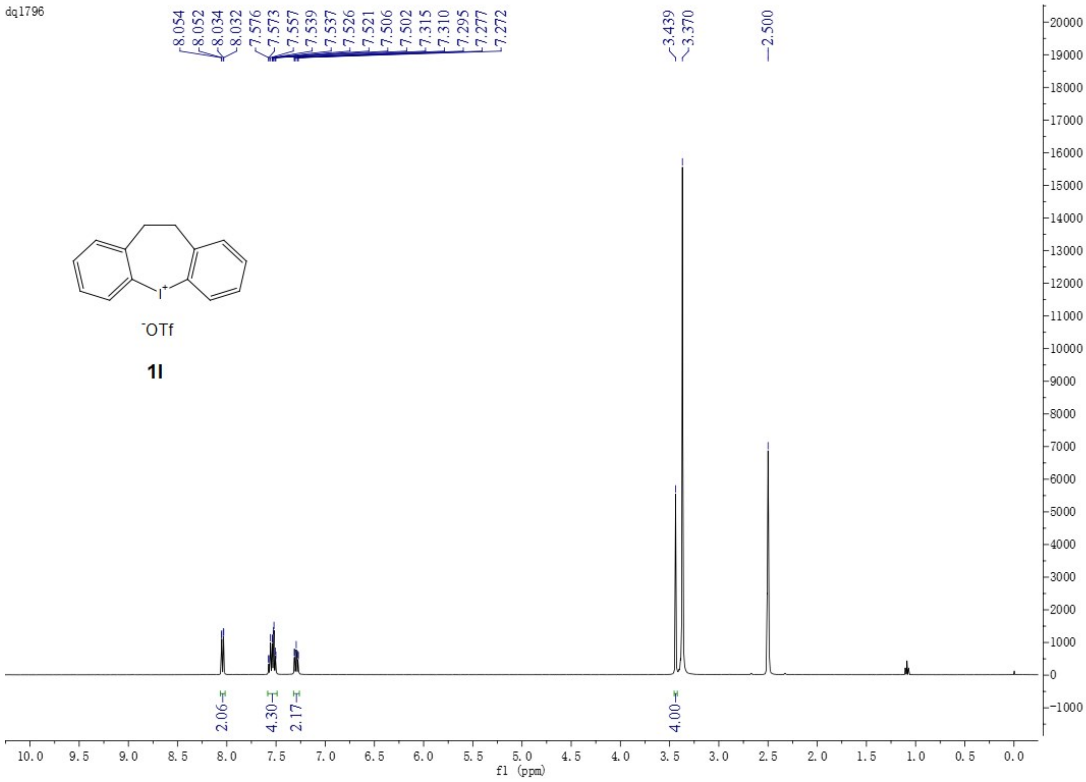

dq2428-0906
8.314
8.311
8.297
8.294
8.118
8.116
8.098
8.095
8.011
8.008
7.992
7.989
7.817
7.813
7.798
7.794
7.640
7.624
7.622
7.606
7.556
7.536
7.516
7.465
7.449
7.446
4.582
3.973
3.403
2.509
2.505
2.500
2.496
2.492
CH3
O
O
I+
-OTf
1.00
1.00
1.00
0.99
1.01
1.02
1.01
1.98
2.99
34000
32000
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
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
f1 (ppm)

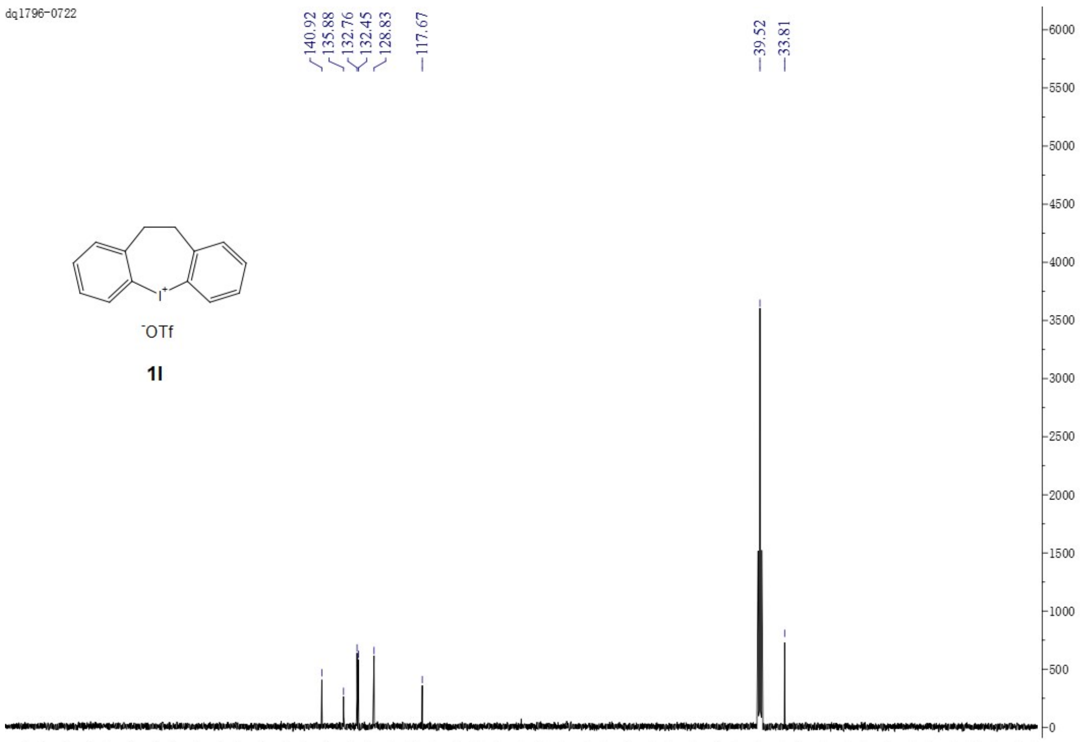
1h

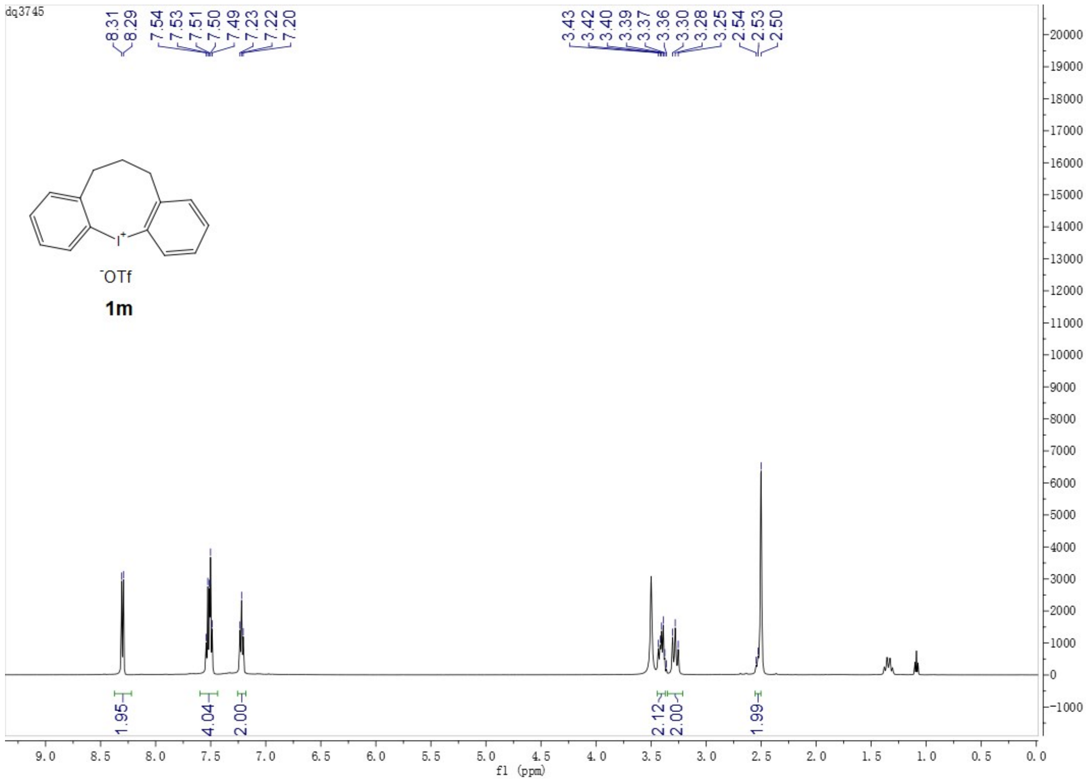

dq2438-0906
166.49
138.73
138.62
137.48
133.50
132.66
131.98
131.90
130.76
129.26
128.81
119.34
117.21
53.05
43.18
40.15
39.94
39.73
39.52
39.31
39.10
38.89
CH3
O
O
I+
-OTf
1h
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

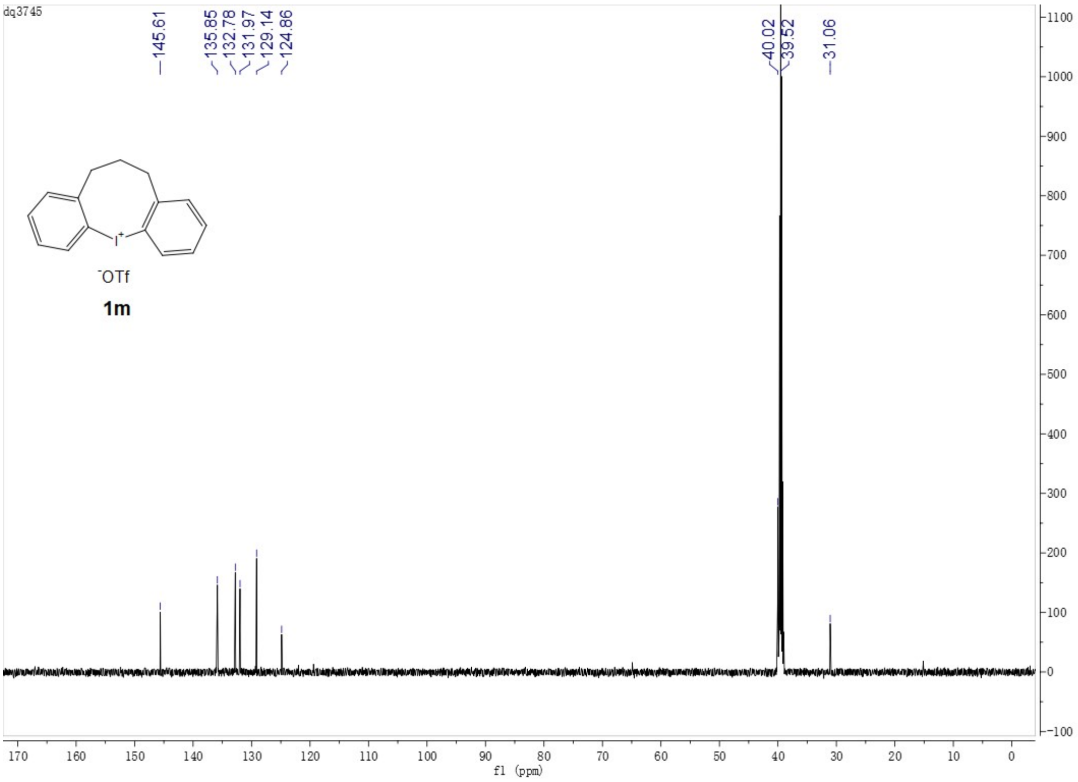

dq3736-Et20
8.094
8.092
8.072
8.070
7.763
7.755
7.749
7.736
7.733
7.624
7.621
7.605
7.603
7.586
7.584
7.454
7.450
7.434
7.431
7.409
7.395
4.443
3.408
2.504
2.500
2.495
S
I+
-OTf
1k
1.00
1.97
1.02
0.97
1.01
2.03
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
9.5
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
f1 (ppm)

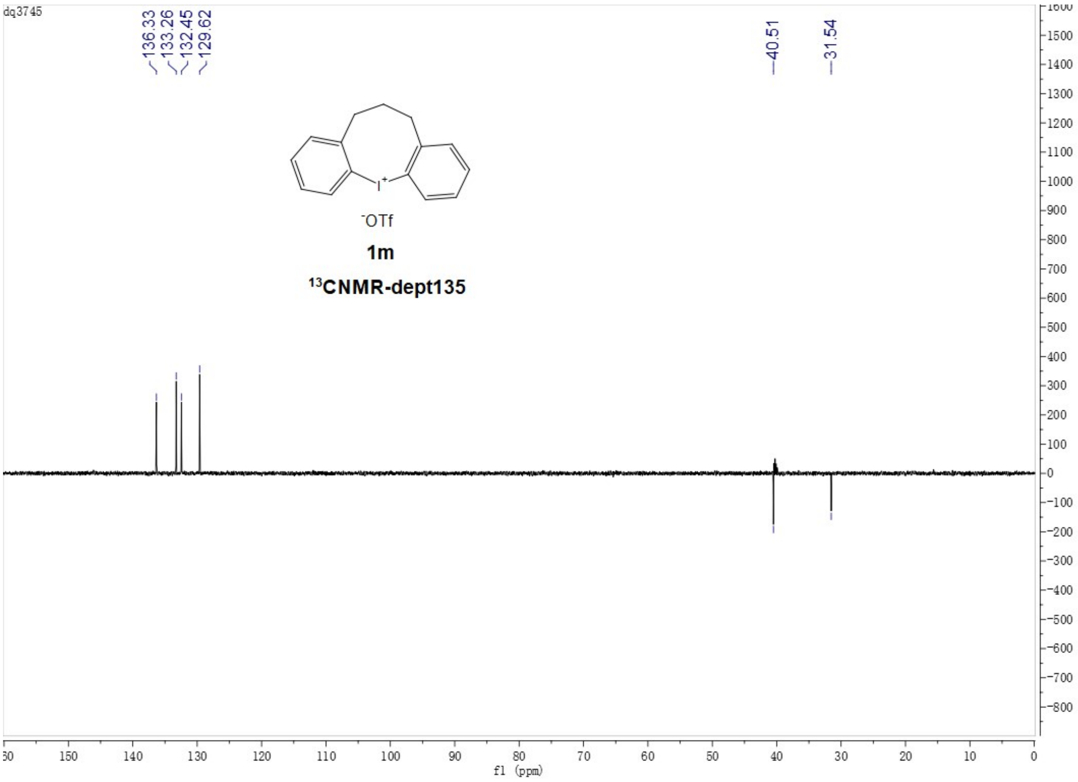

dq3736-c
140.62
138.15
133.74
131.46
131.26
129.12
128.69
127.82
115.44
99.55
40.02
39.85
39.69
39.52
39.35
39.19
39.02
36.77
S
I+
-OTf
1k
1100
1000
900
800
700
600
500
400
300
200
100
0
−100
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

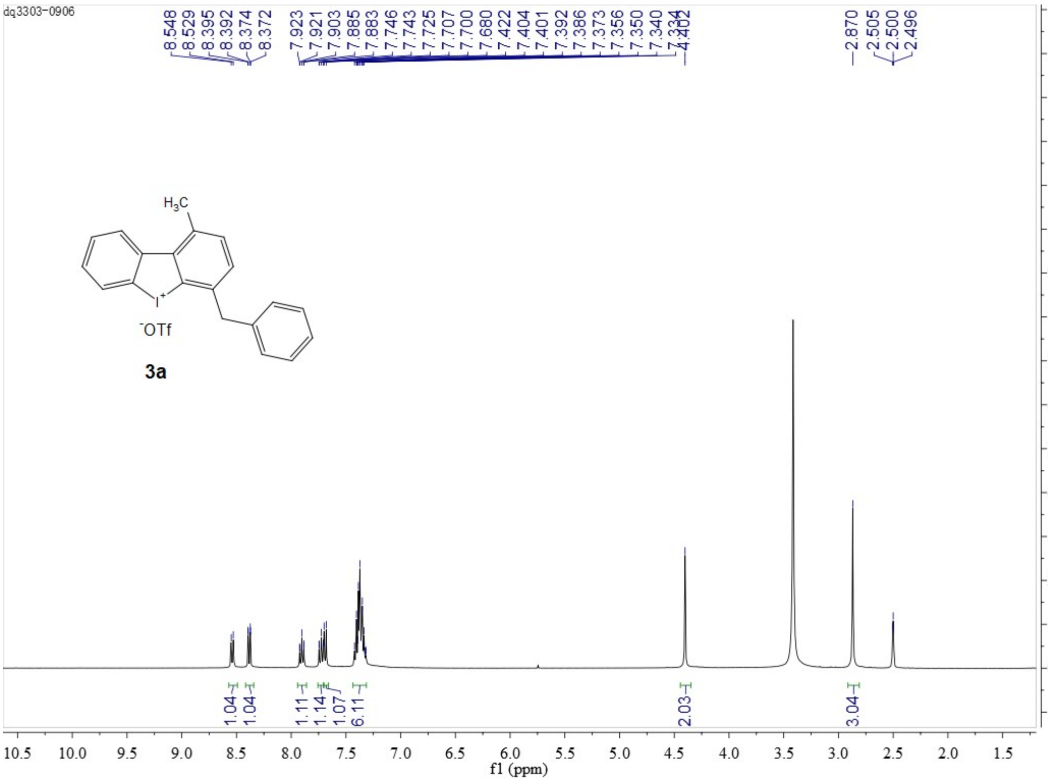

dq1796
8.054
8.052
8.034
8.032
7.576
7.573
7.557
7.539
7.537
7.526
7.521
7.506
7.502
7.315
7.310
7.295
7.277
7.272
3.439
3.370
2.500
I+
-OTf
1l
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
−1000
2.06
4.30
2.17
4.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

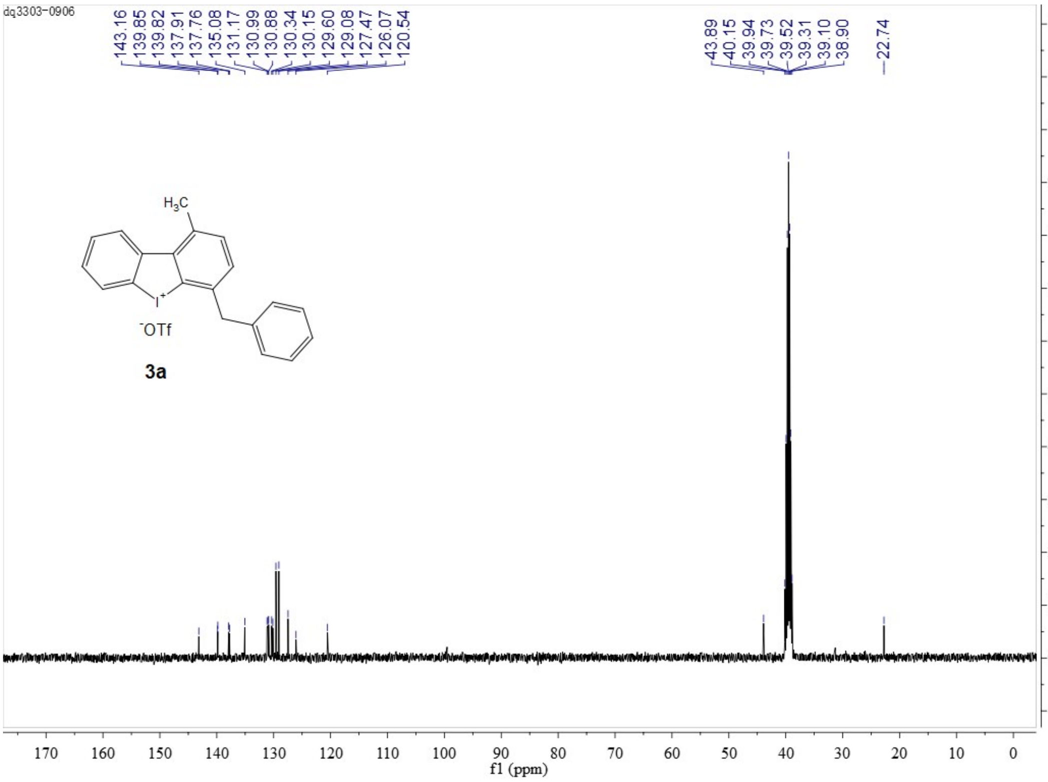

dq1796-0722
140.92
135.88
132.76
132.45
128.83
117.67
39.52
33.81
I+
-OTf
1l
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

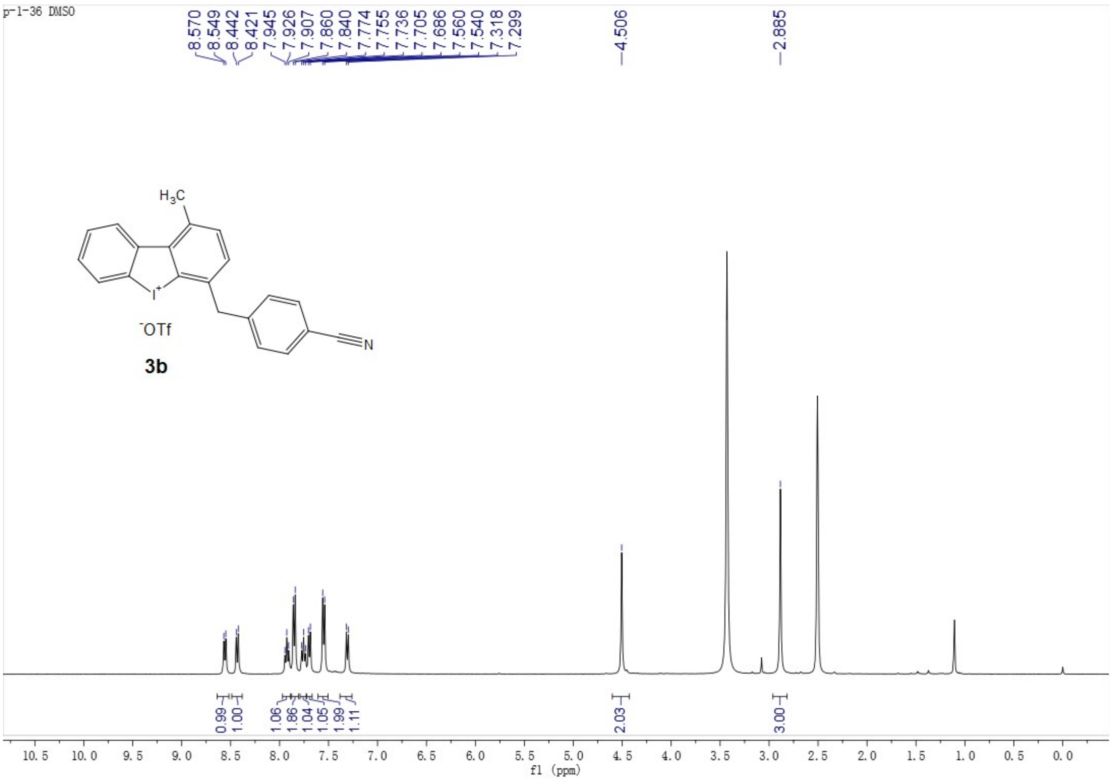

dq3745
8.31
8.29
7.54
7.53
7.51
7.50
7.49
7.23
7.22
7.20
3.43
3.42
3.40
3.39
3.37
3.36
3.30
3.28
3.25
2.54
2.53
2.50
I+
-OTf
1m
1.95
4.04
2.00
2.12
2.00
1.99
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
9.0 8.5 8.0 7.5 7.0 6.5 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
f1 (ppm)

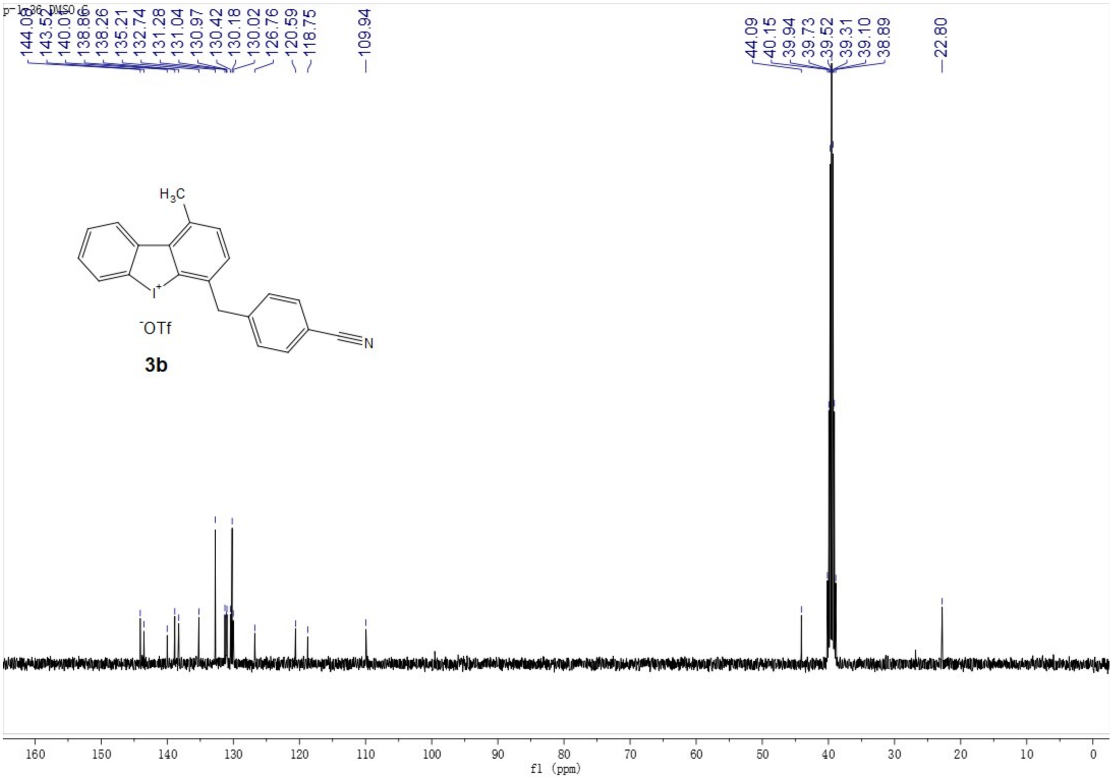

dq3745
145.61
135.85
132.78
131.97
129.14
124.86
40.02
39.52
31.06
I+
-OTf
1m
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
1100 1000 900 800 700 600 500 400 300 200 100 0 −100

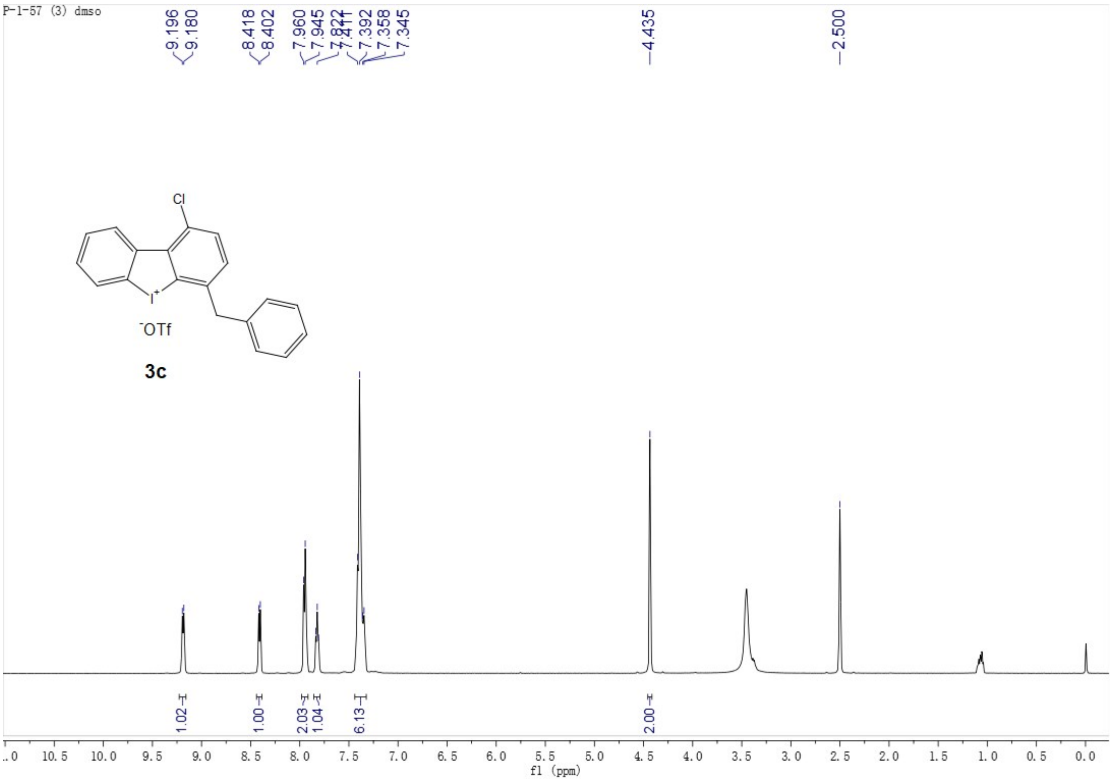

dq3745
136.33
133.26
132.45
129.62
40.51
31.54
I+
-OTf
1m
13CNMR-dept135
f1 (ppm)
50 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
1600 1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 −100 −200 −300 −400 −500 −600 −700 −800

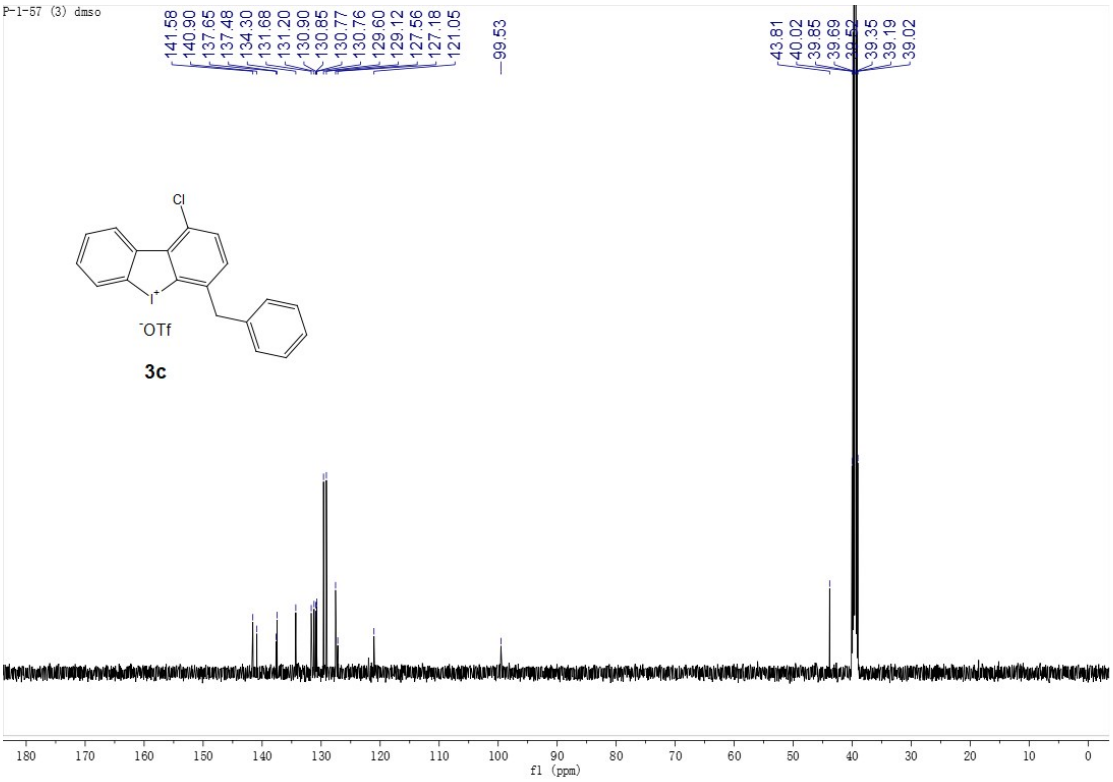

dq3303-0906
8.548
8.529
8.395
8.392
8.374
8.372
7.923
7.921
7.903
7.885
7.883
7.746
7.743
7.725
7.707
7.700
7.680
7.422
7.404
7.401
7.392
7.386
7.373
7.356
7.350
7.340
7.334
4.402
2.870
2.505
2.500
2.496
H$_3$C
I$^+$
$^-$OTf
3a
1.04
1.04
1.11
1.14
1.07
6.11
2.03
3.04
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
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
f1 (ppm)

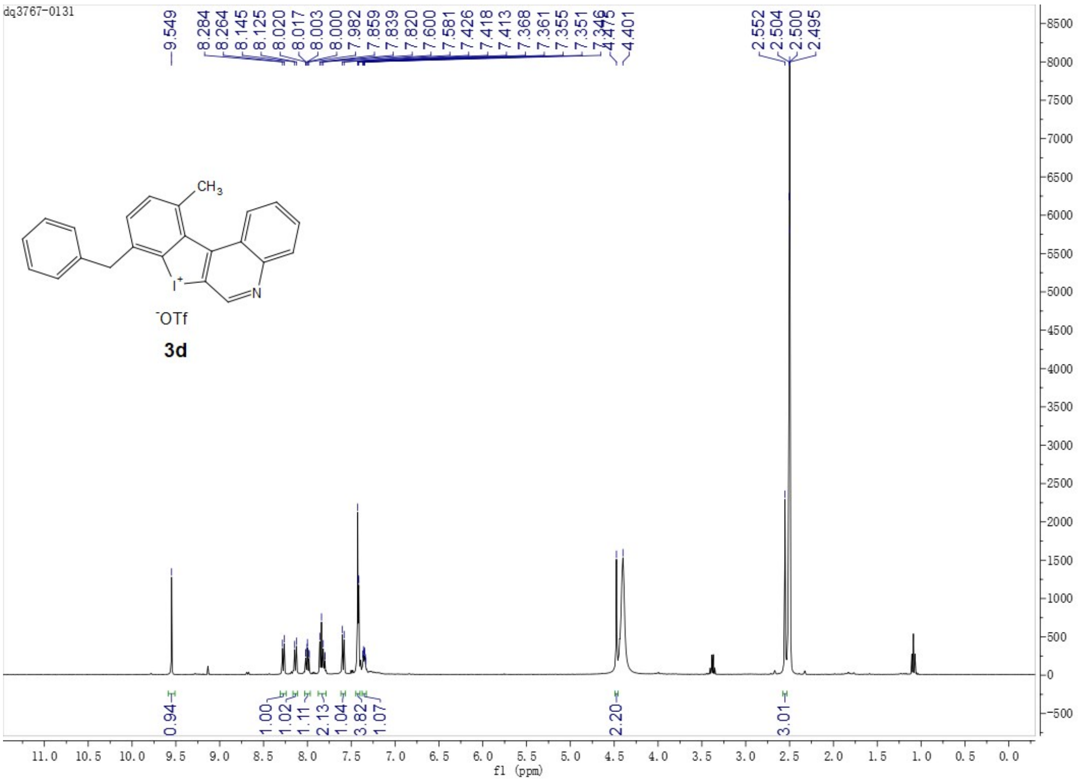

dq3303-0906
143.16
139.85
139.82
137.91
137.76
135.08
131.17
130.99
130.88
130.34
130.15
129.60
129.08
127.47
126.07
120.54
43.89
40.15
39.94
39.73
39.52
39.31
39.10
38.90
22.74
H$_3$C
I$^+$
$^-$OTf
3a
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

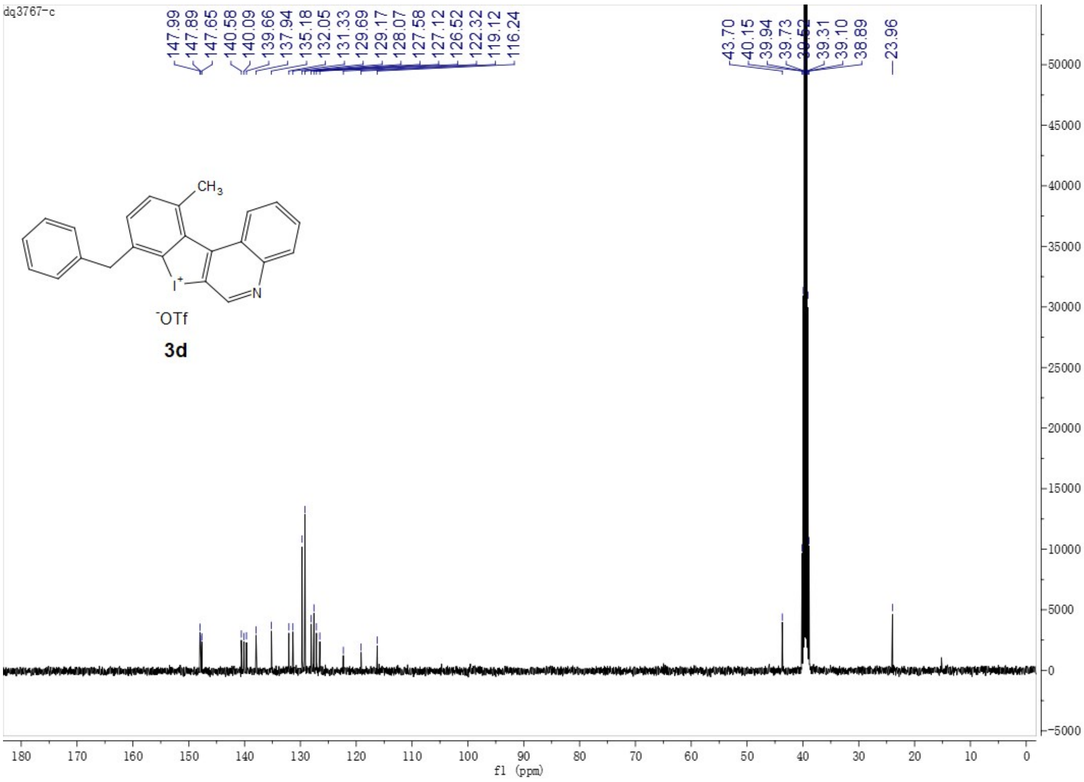

p-1-36 DMSO
8.570
8.549
8.442
8.421
7.945
7.926
7.907
7.860
7.840
7.774
7.755
7.736
7.705
7.686
7.560
7.540
7.318
7.299
4.506
2.885
H_3C
I^+
^-OTf
N
3b
0.99
1.00
1.06
1.86
1.04
1.05
1.99
1.11
2.03
3.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

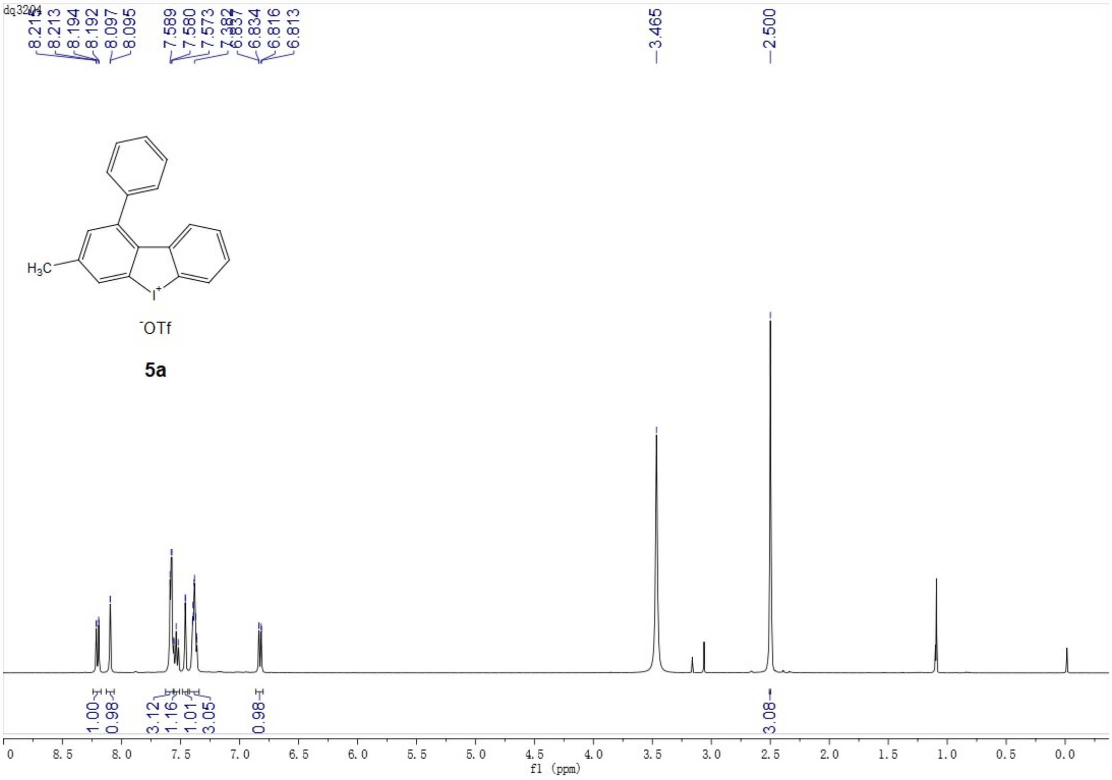

p-1-36 DMSO 6
144.08
143.52
140.0
138.86
138.26
135.21
132.74
131.28
131.04
130.97
130.42
130.18
130.02
126.76
120.59
118.75
109.94
44.09
40.15
39.94
39.73
39.52
39.31
39.10
38.89
22.80
H_3C
I^+
^-OTf
N
3b
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

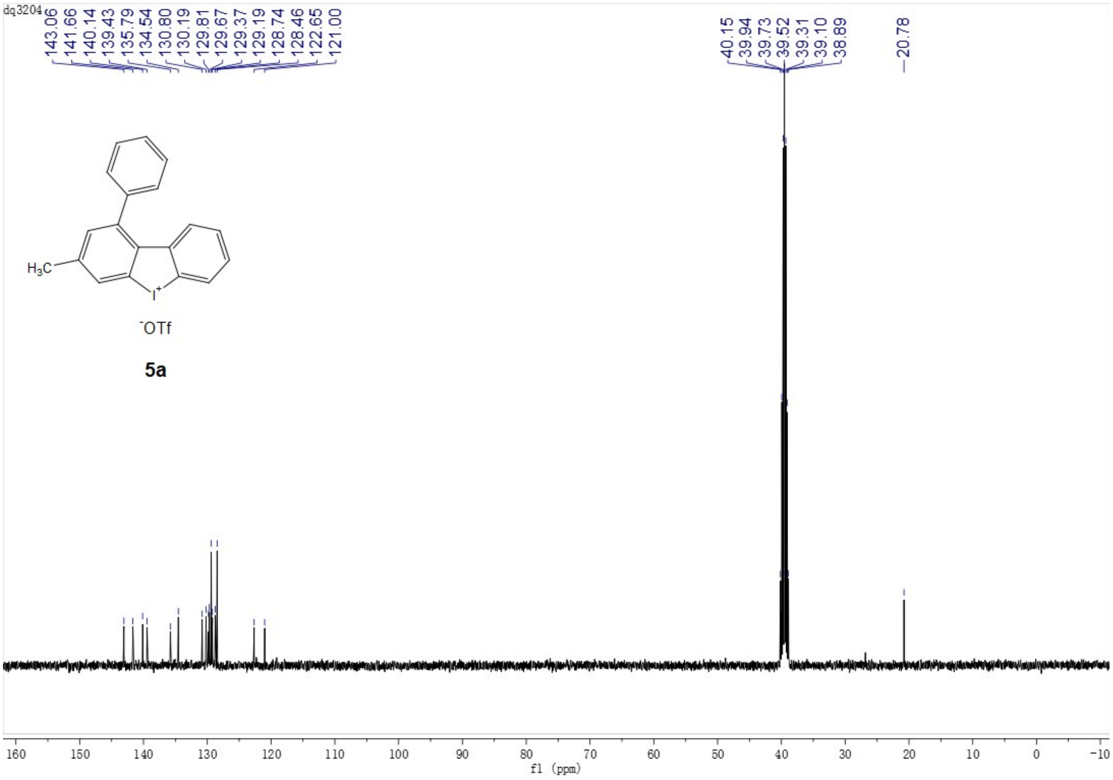

P-1-57 (3) dmso
9.196
9.180
8.418
8.402
7.960
7.945
7.822
7.411
7.392
7.358
7.345
4.435
2.500
Cl
I+
-OTf
3c
1.02
1.00
2.03
1.04
6.13
2.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

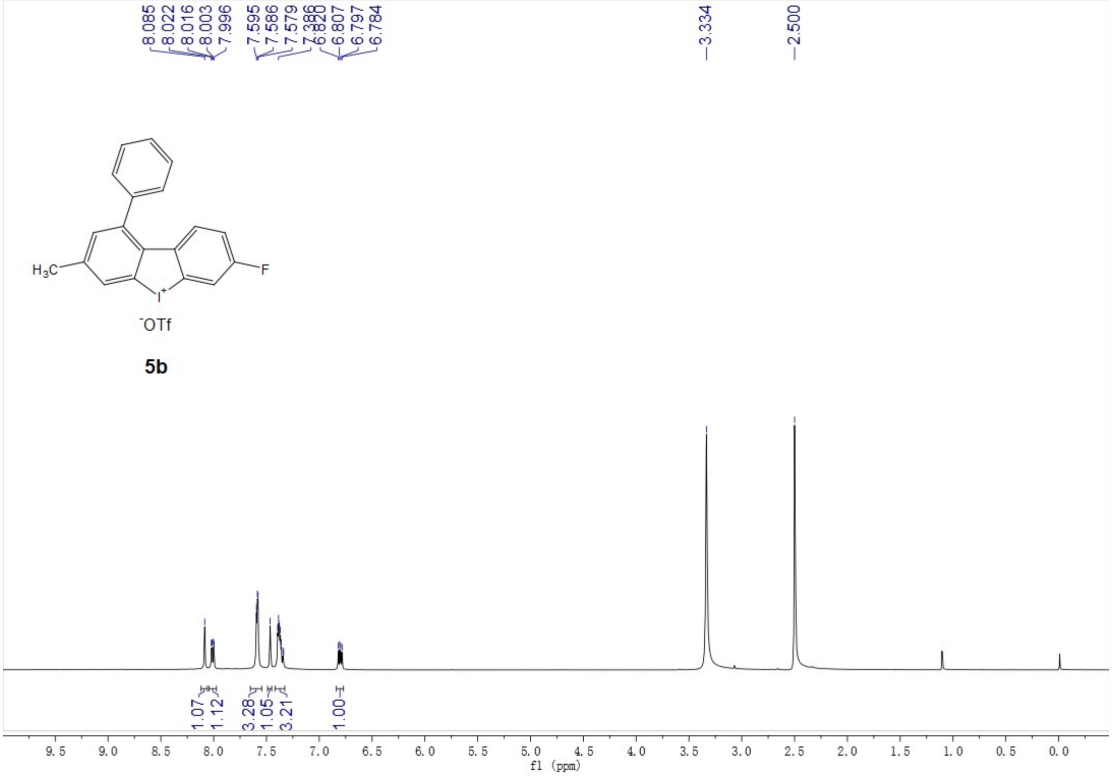

P-1-57 (3) dmso
141.58
140.90
137.65
137.48
134.30
131.68
131.20
130.90
130.85
130.77
130.76
129.60
129.12
127.56
127.18
121.05
99.53
43.81
40.02
39.85
39.69
39.52
39.35
39.19
39.02
Cl
I+
-OTf
3c
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

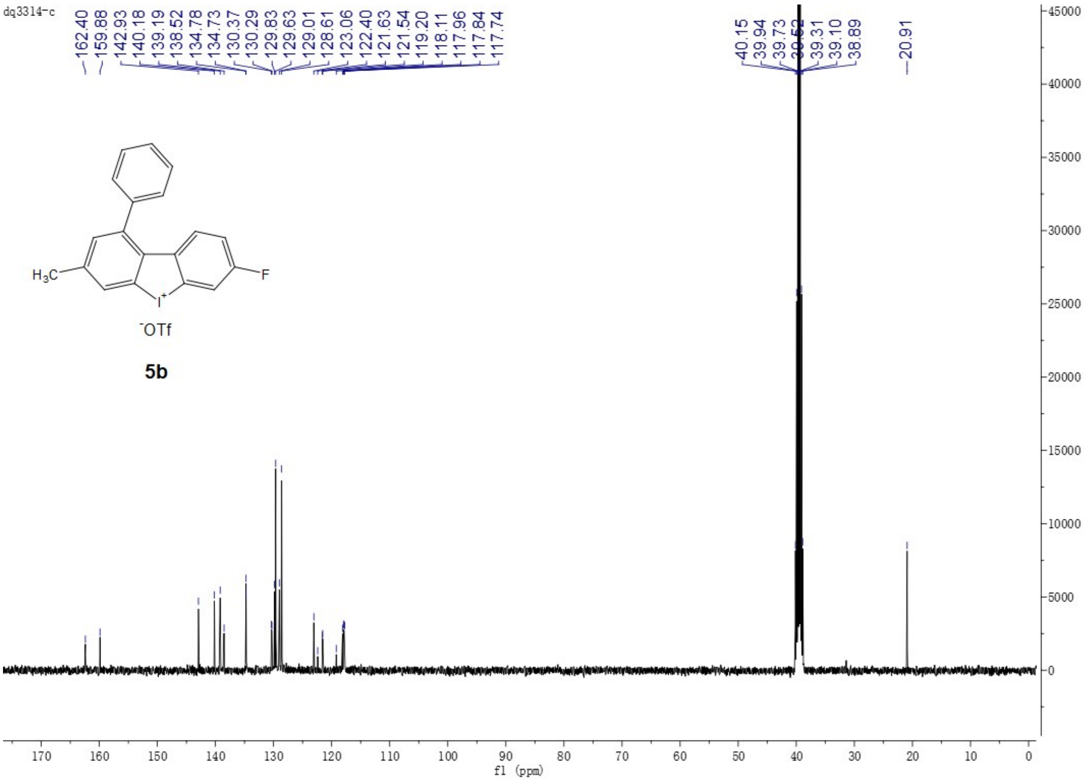

dq3767-0131
9.549
8.284
8.264
8.145
8.125
8.020
8.017
8.003
8.000
7.982
7.859
7.839
7.820
7.600
7.581
7.426
7.418
7.413
7.368
7.361
7.355
7.351
7.346
4.415
4.401
2.552
2.504
2.500
2.495
CH3
I+
N
-OTf
3d
0.94
1.00
1.02
1.11
2.13
1.04
3.82
1.07
2.20
3.01
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
8500 8000 7500 7000 6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

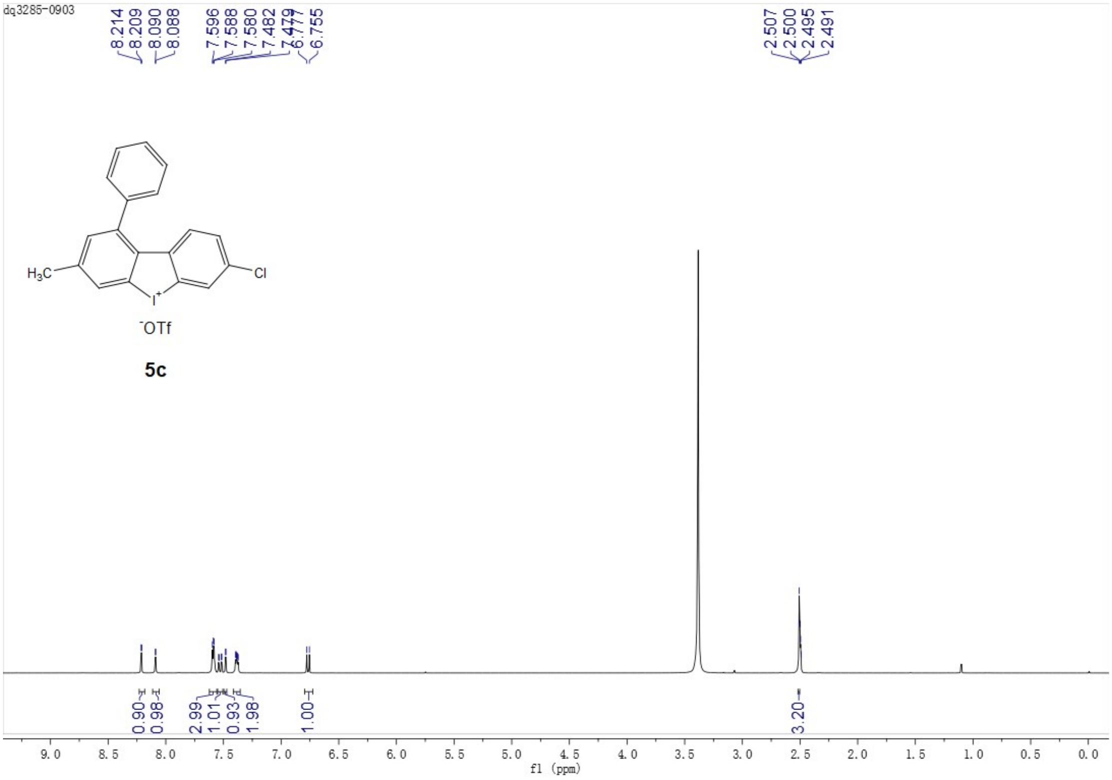

dq3767-c
147.99
147.89
147.65
140.58
140.09
139.66
137.94
135.18
132.05
131.33
129.69
129.17
128.07
127.58
127.12
126.52
122.32
119.12
116.24
43.70
40.15
39.94
39.73
39.52
39.31
39.10
38.89
23.96
CH3
I+
N
-OTf
3d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
50000 45000 40000 35000 30000 25000 20000 15000 10000 5000 0 -5000

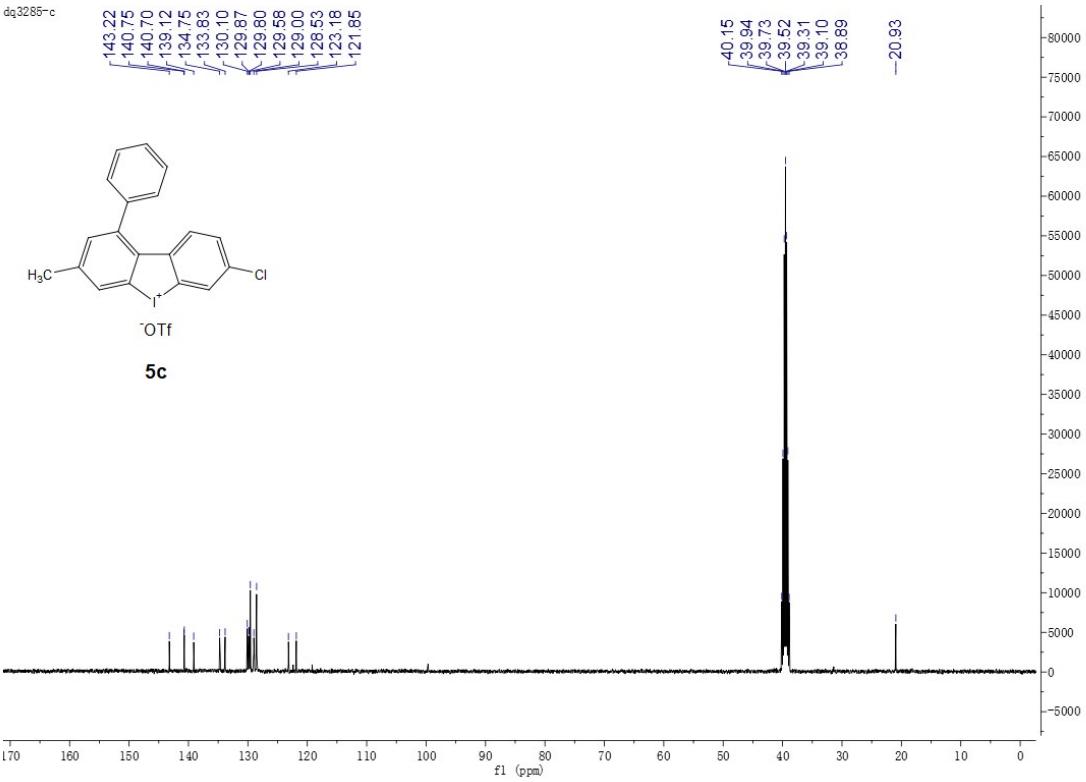

dq3204
8.213
8.194
8.192
8.097
8.095
7.589
7.580
7.573
7.382
6.837
6.834
6.816
6.813
3.465
2.500
H$_3$C
I$^+$
$^-$OTf
5a
1.00
0.98
3.12
1.16
1.01
3.05
0.98
3.08
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

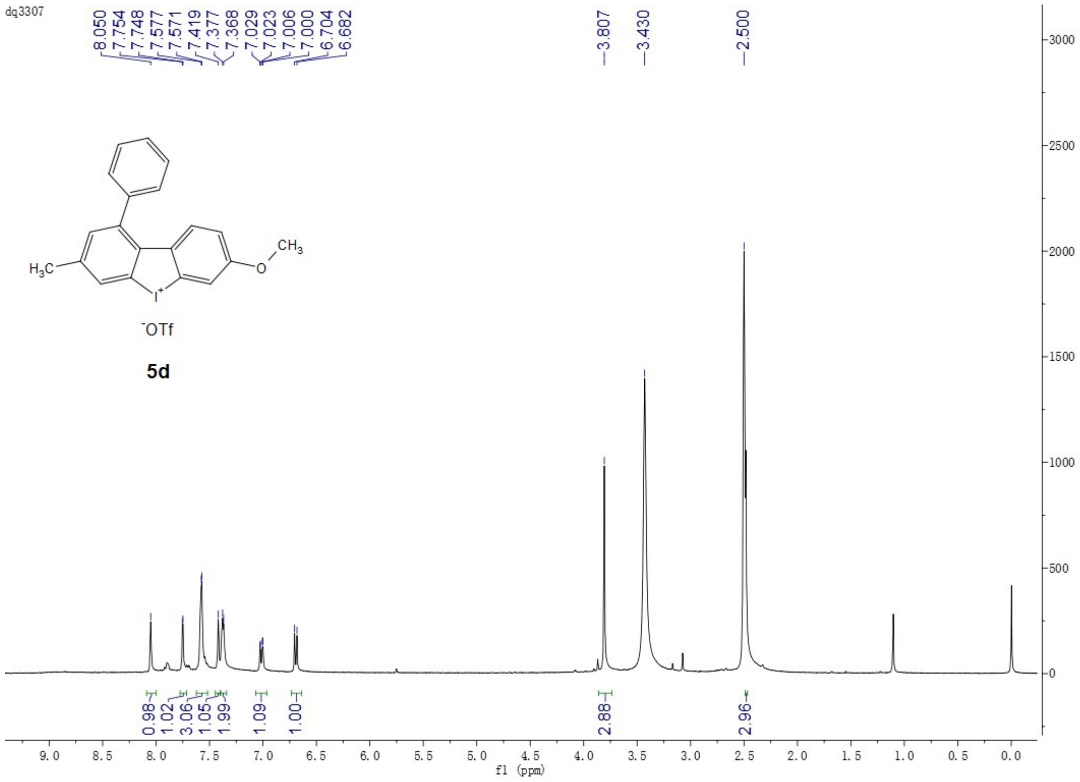

dq3204
143.06
141.66
140.14
139.43
135.79
134.54
130.80
130.19
129.81
129.67
129.37
129.19
128.74
128.46
122.65
121.00
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.78
H$_3$C
I$^+$
$^-$OTf
5a
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

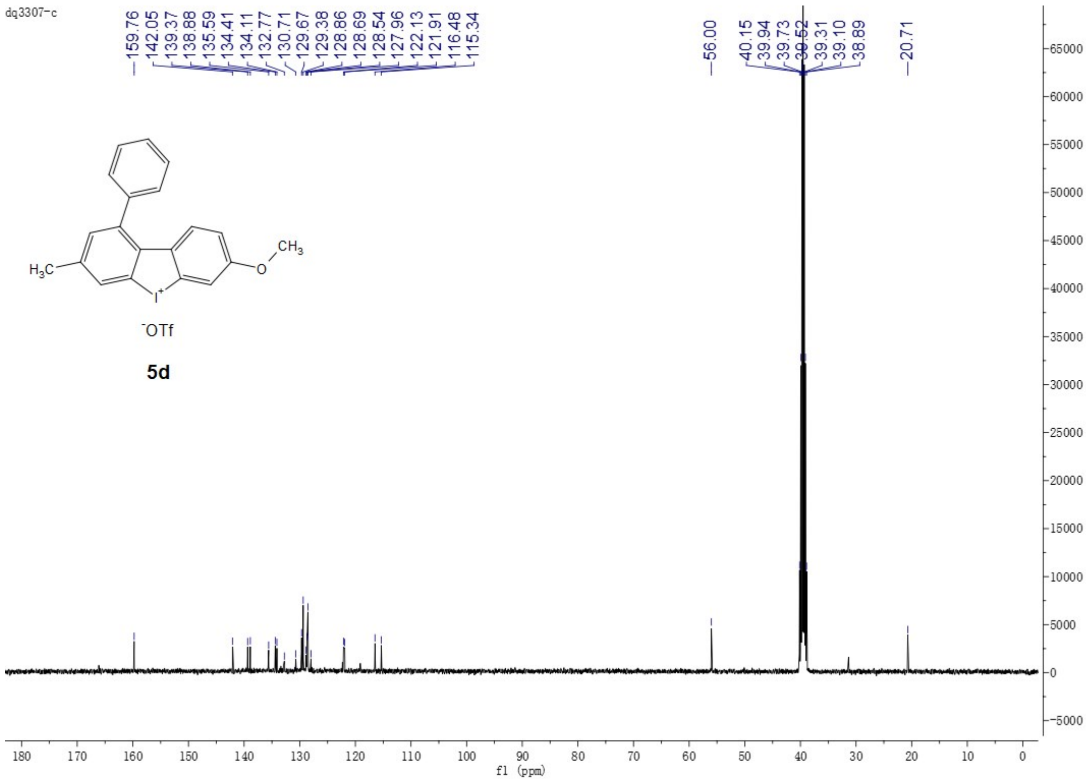

8.085
8.022
8.016
8.003
7.996
7.595
7.586
7.579
7.386
6.820
6.807
6.797
6.784
3.334
2.500
H3C
I+
F
-OTf
5b
1.07
1.12
3.28
1.05
3.21
1.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

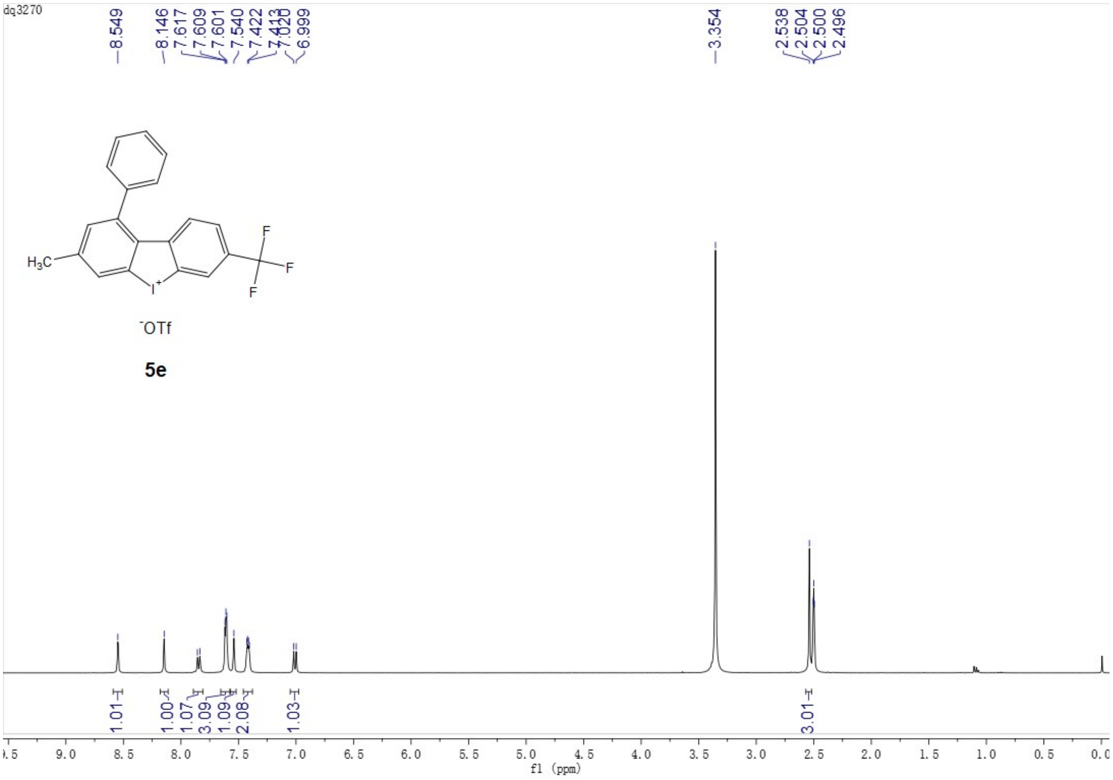

dq3314-c
162.40
159.88
142.93
140.18
139.19
138.52
134.78
134.73
130.37
130.29
129.83
129.63
129.01
128.61
123.06
122.40
121.63
121.54
119.20
118.11
117.96
117.84
117.74
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.91
H3C
I+
F
-OTf
5b
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

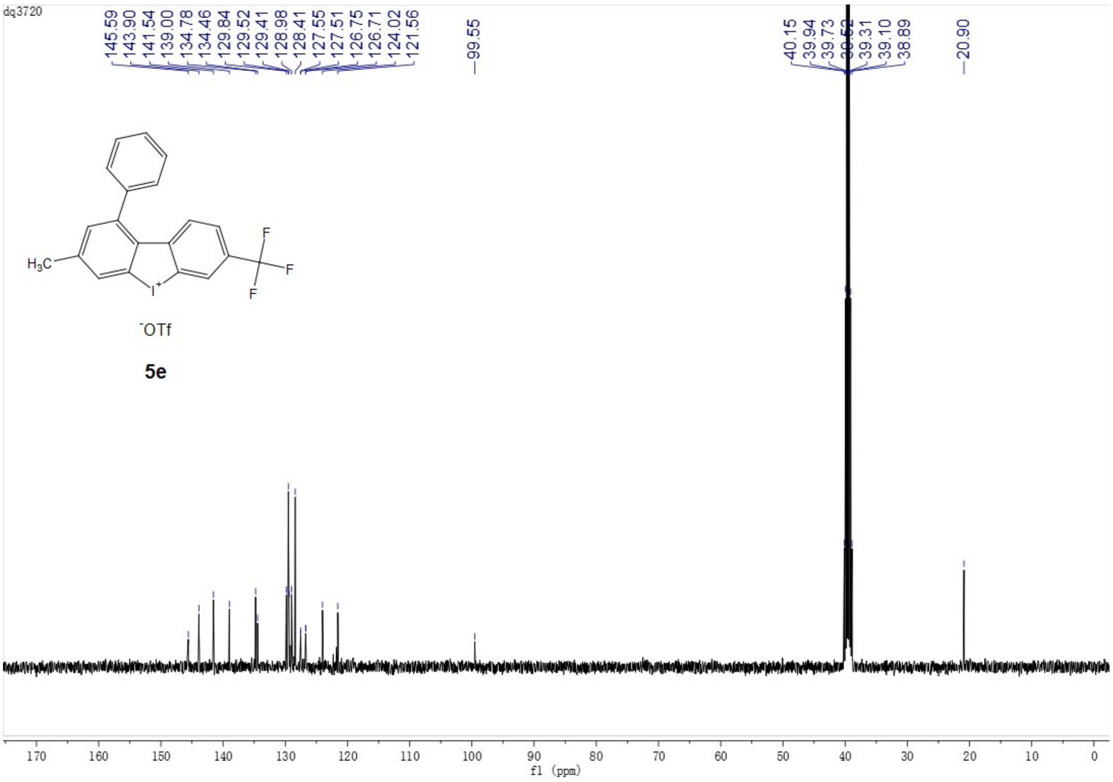

dq3285-0903
8.214
8.209
8.090
8.088
7.596
7.588
7.580
7.482
7.479
6.777
6.755
2.507
2.500
2.495
2.491
H3C
Cl
I+
-OTf
5c
0.90
0.98
2.99
1.01
0.93
1.98
1.00
3.20
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

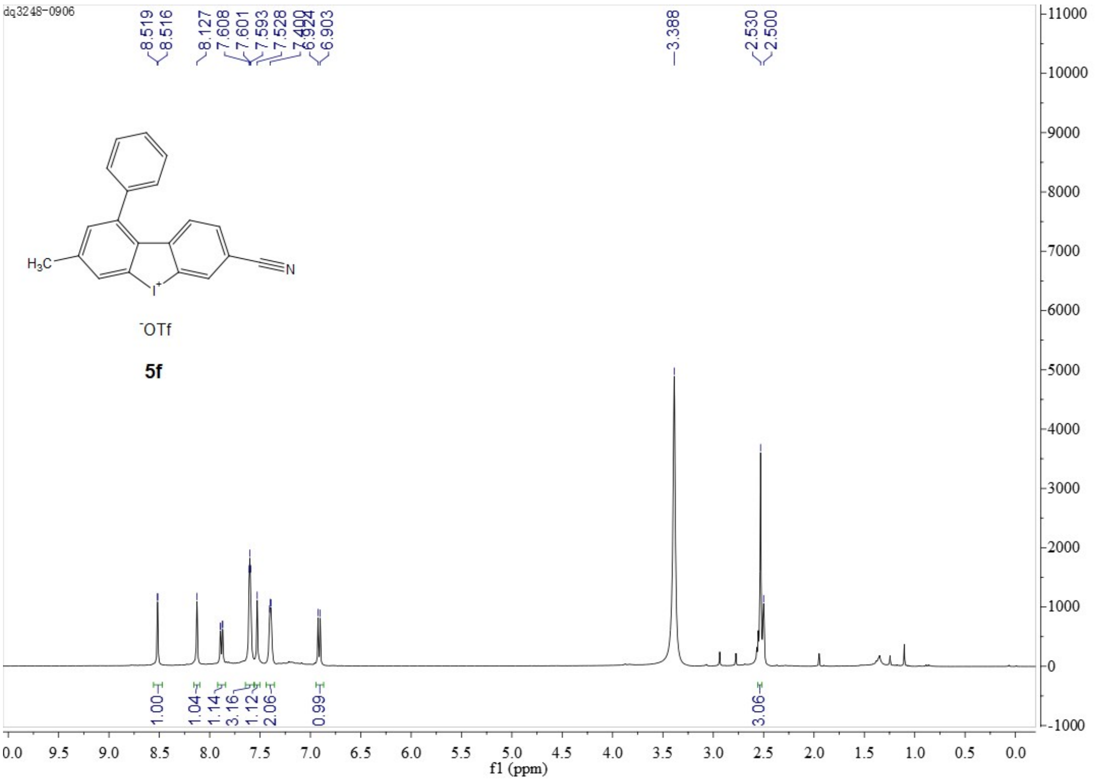

dq3285-c
143.22
140.75
140.70
139.12
134.75
133.83
130.10
129.87
129.80
129.58
129.00
128.53
123.18
121.85
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.93
H3C
Cl
I+
-OTf
5c
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

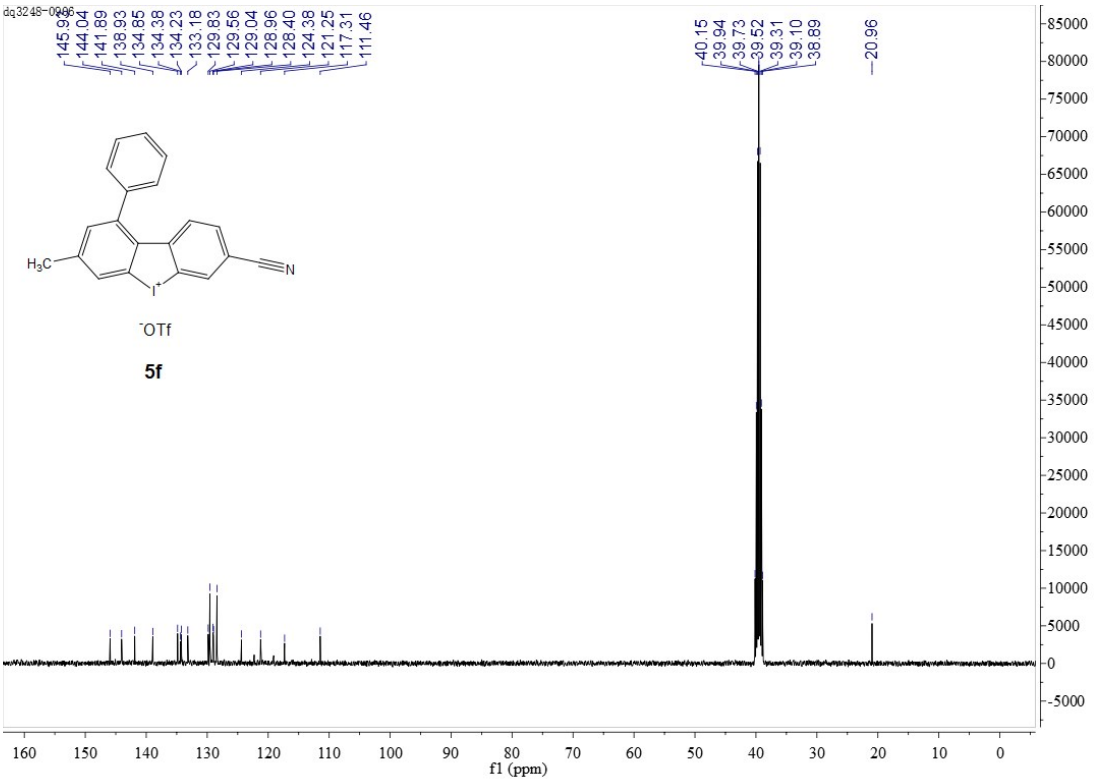

dq3307
8.050
7.754
7.748
7.577
7.571
7.419
7.377
7.368
7.029
7.023
7.006
7.000
6.704
6.682
3.807
3.430
2.500
H3C
O
CH3
I+
-OTf
5d
0.98
1.02
3.06
1.05
1.99
1.09
1.00
2.88
2.96
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
3000
2500
2000
1500
1000
500
0

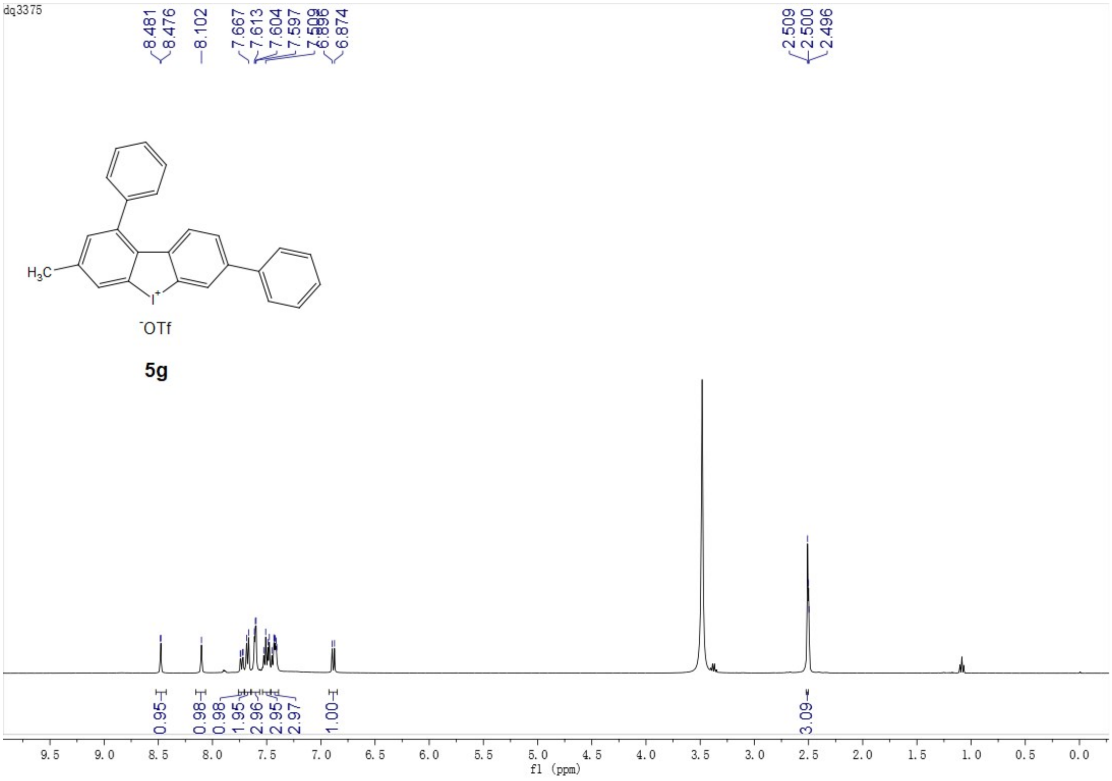

dq3307-c
159.76
142.05
139.37
138.88
135.59
134.41
134.11
132.77
130.71
129.67
129.38
128.86
128.69
128.54
127.96
122.13
121.91
116.48
115.34
56.00
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.71
H3C
O
CH3
I+
-OTf
5d
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000

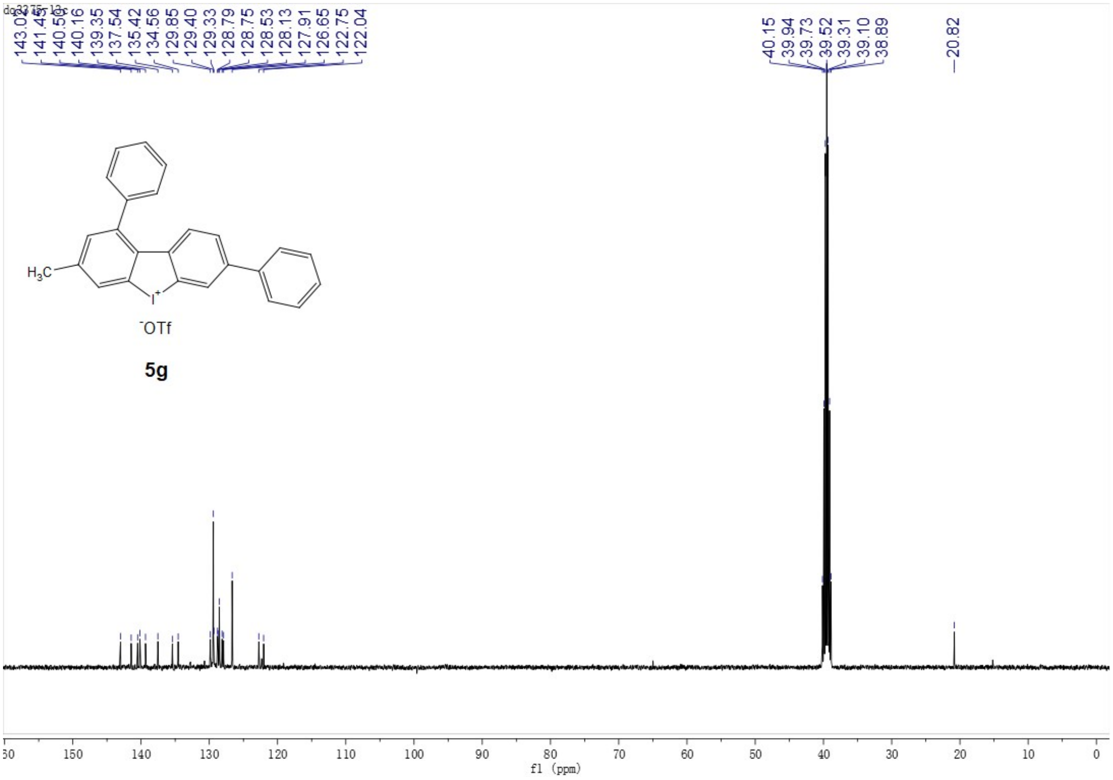

dq3270
8.549
8.146
7.617
7.609
7.601
7.540
7.422
7.413
7.020
6.999
3.354
2.538
2.504
2.500
2.496
H3C
F
F
F
I+
-OTf
5e
1.01
1.00
1.07
3.09
1.09
2.08
1.03
3.01
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

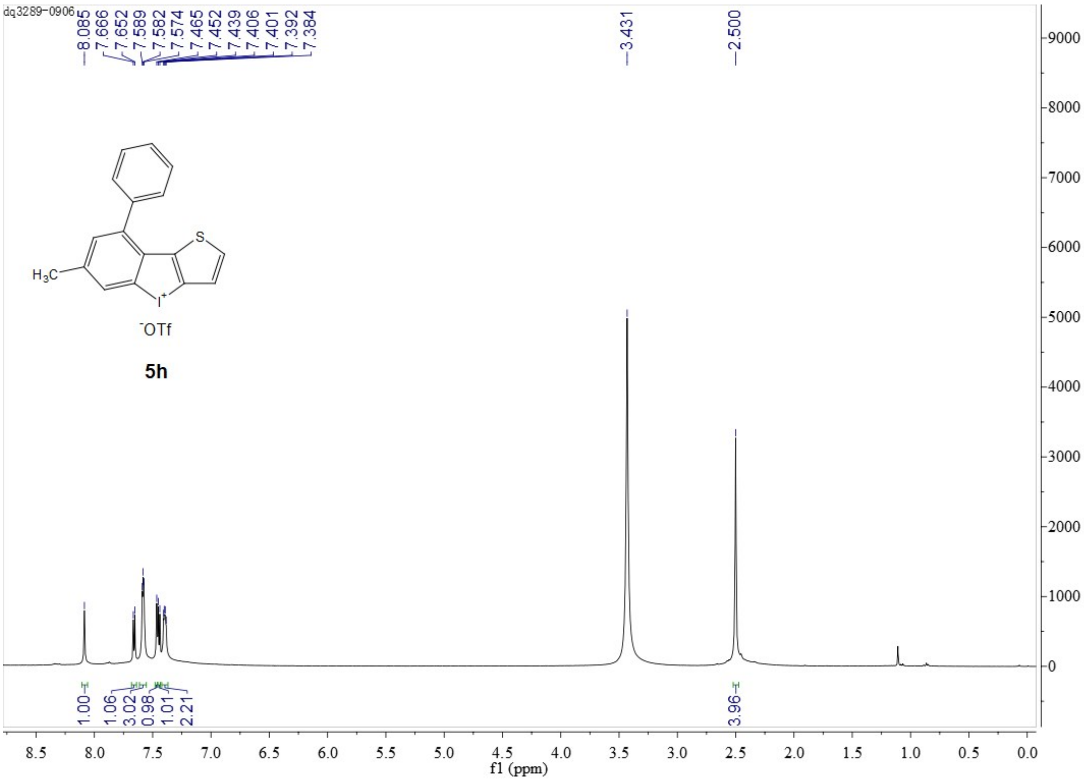

dq3720
145.59
143.90
141.54
139.00
134.78
134.46
129.84
129.52
129.41
128.98
128.41
127.55
127.51
126.75
126.71
124.02
121.56
99.55
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.90
H3C
F
F
F
I+
-OTf
5e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

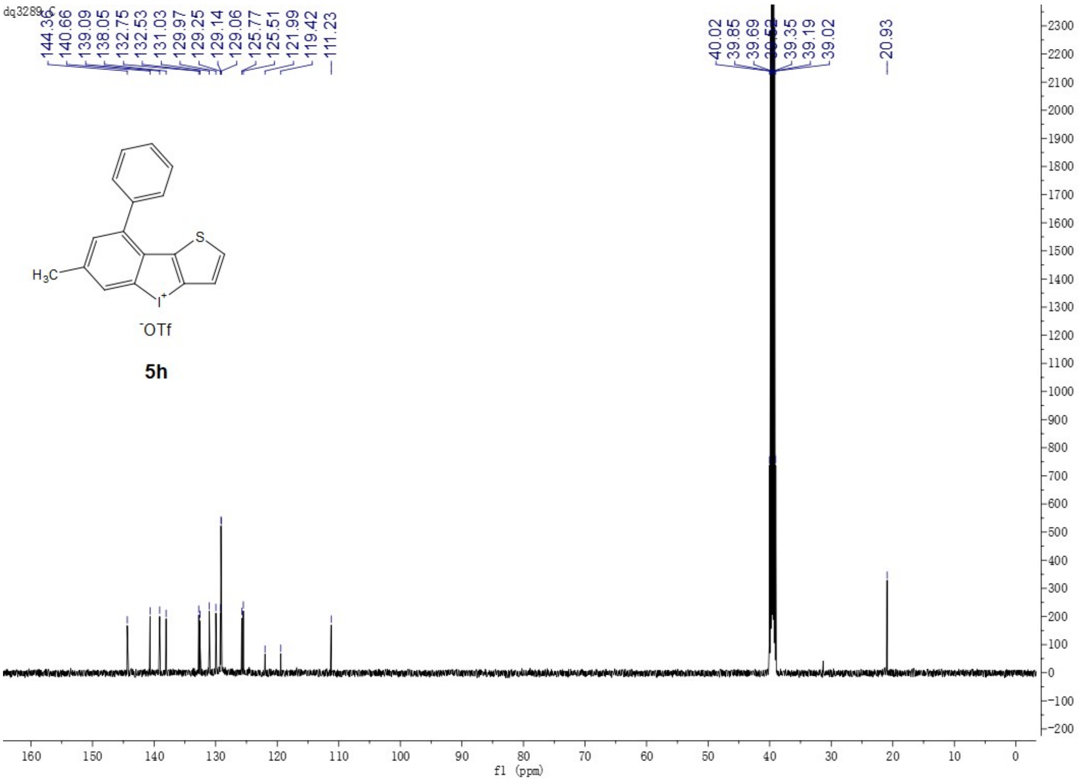

dq3248-0906
8.519
8.516
8.127
7.608
7.601
7.593
7.528
7.400
6.924
6.903
3.388
2.530
2.500
H3C
I+
N
-OTf
5f
1.00
1.04
1.14
3.16
1.12
2.06
0.99
3.06
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

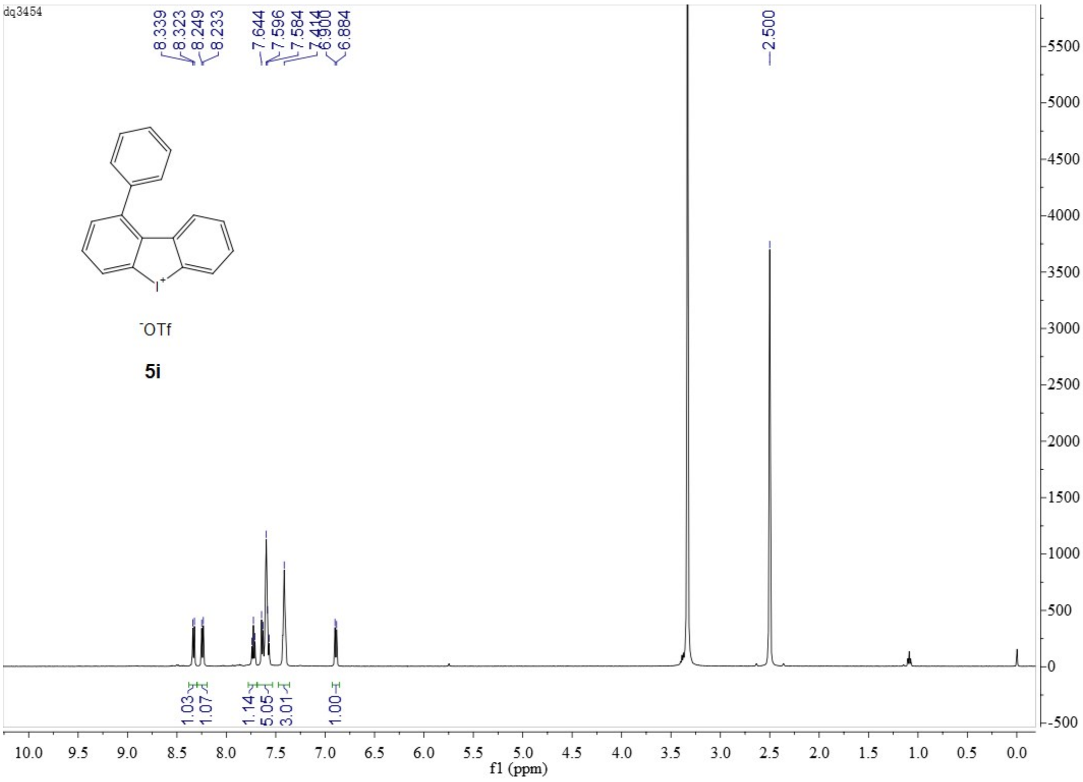

dq3248-0906
145.93
144.04
141.89
138.93
134.85
134.38
134.23
133.18
129.83
129.56
129.04
128.96
128.40
124.38
121.25
117.31
111.46
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.96
H3C
I+
N
-OTf
5f
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

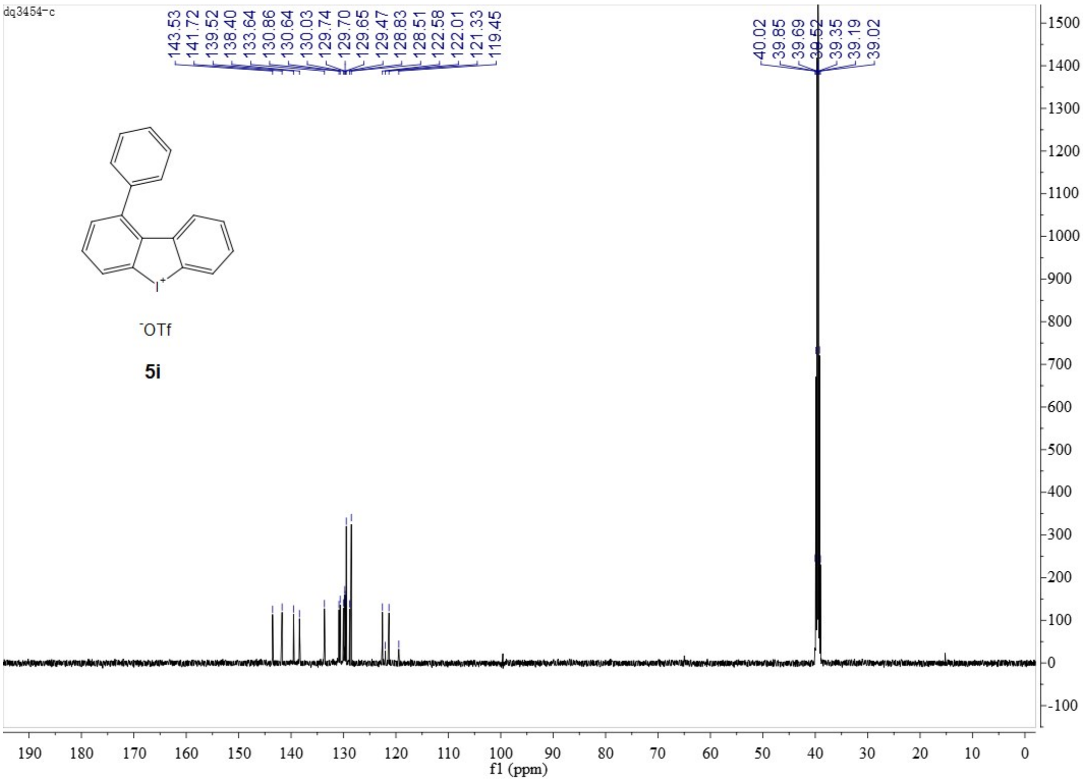

dq3375
8.481
8.476
8.102
7.667
7.613
7.604
7.597
7.509
6.896
6.874
2.509
2.500
2.496
H_3C
I^+
^-OTf
5g
0.95
0.98
0.98
1.95
2.96
2.95
2.97
1.00
3.09
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

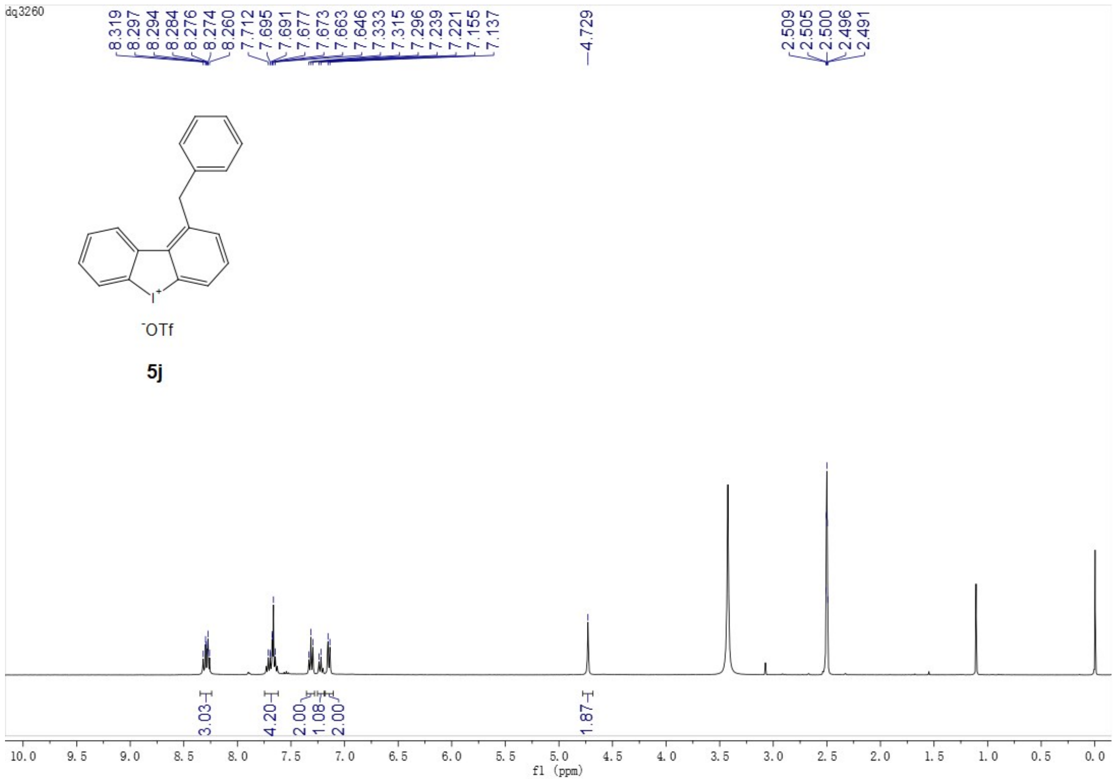

dq3375-13c
143.04
141.45
140.50
140.16
139.35
137.54
135.42
134.56
129.85
129.40
129.33
128.79
128.75
128.53
128.13
127.91
126.65
122.75
122.04
40.15
39.94
39.73
39.52
39.31
39.10
38.89
20.82
H_3C
I^+
^-OTf
5g
50
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

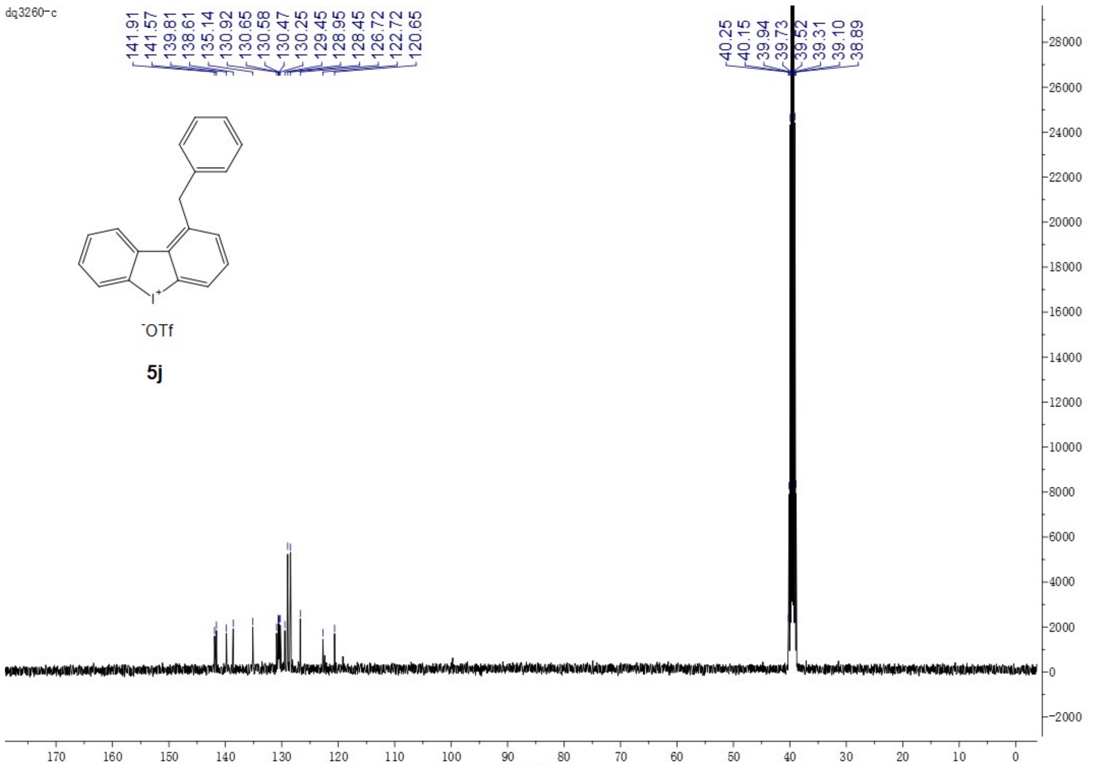

dq3289-0906
8.085
7.666
7.652
7.589
7.582
7.574
7.465
7.452
7.439
7.406
7.401
7.392
7.384
3.431
2.500
H_3C
S
I+
-OTf
5h
1.00
1.06
3.02
0.98
1.01
2.21
3.96
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

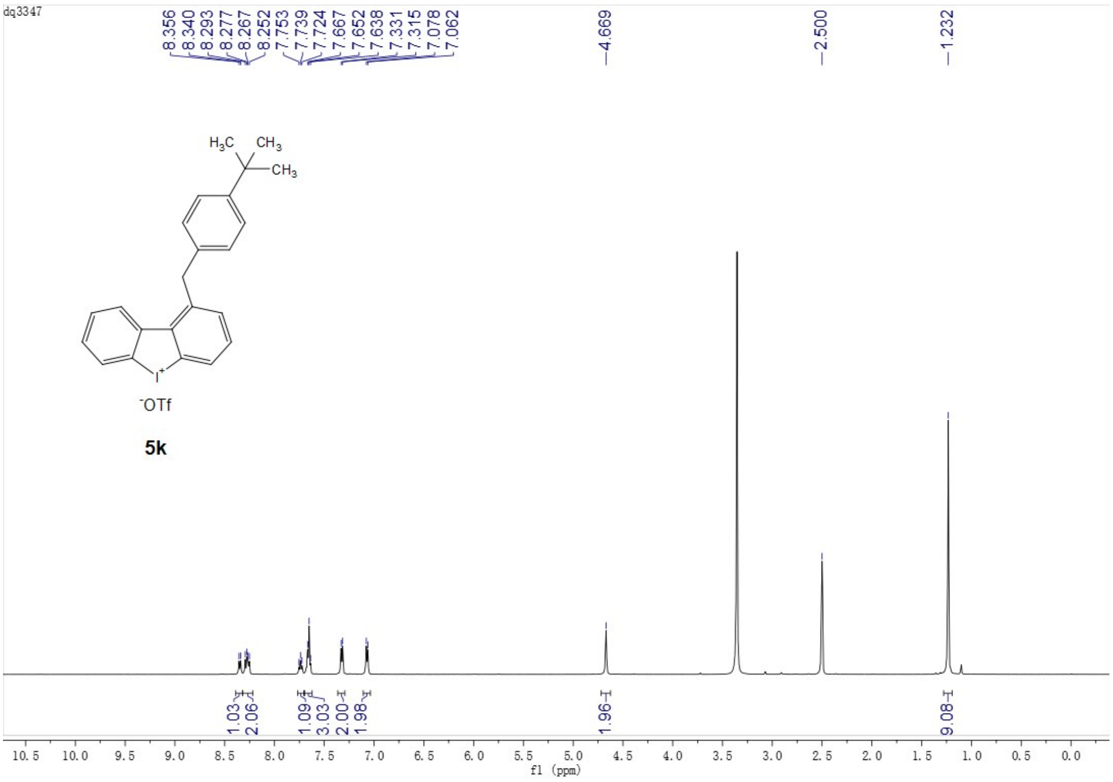

dq3289-C
144.36
140.66
139.09
138.05
132.75
132.53
131.03
129.97
129.25
129.14
129.06
125.77
125.51
121.99
119.42
111.23
40.02
39.85
39.69
39.52
39.35
39.19
39.02
20.93
H_3C
S
I+
-OTf
5h
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
−100
−200
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

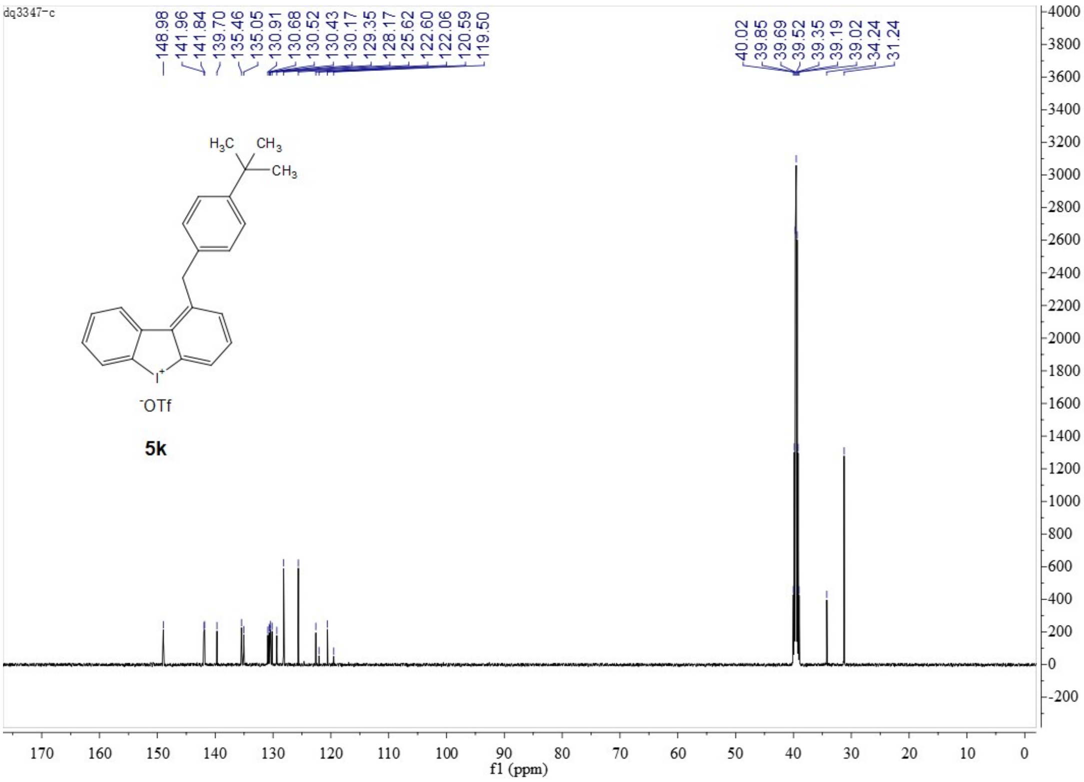

dq3454
8.339
8.323
8.249
8.233
7.644
7.596
7.584
7.414
6.900
6.884
2.500
I+
-OTf
5i
1.03
1.07
1.14
5.05
3.01
1.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500

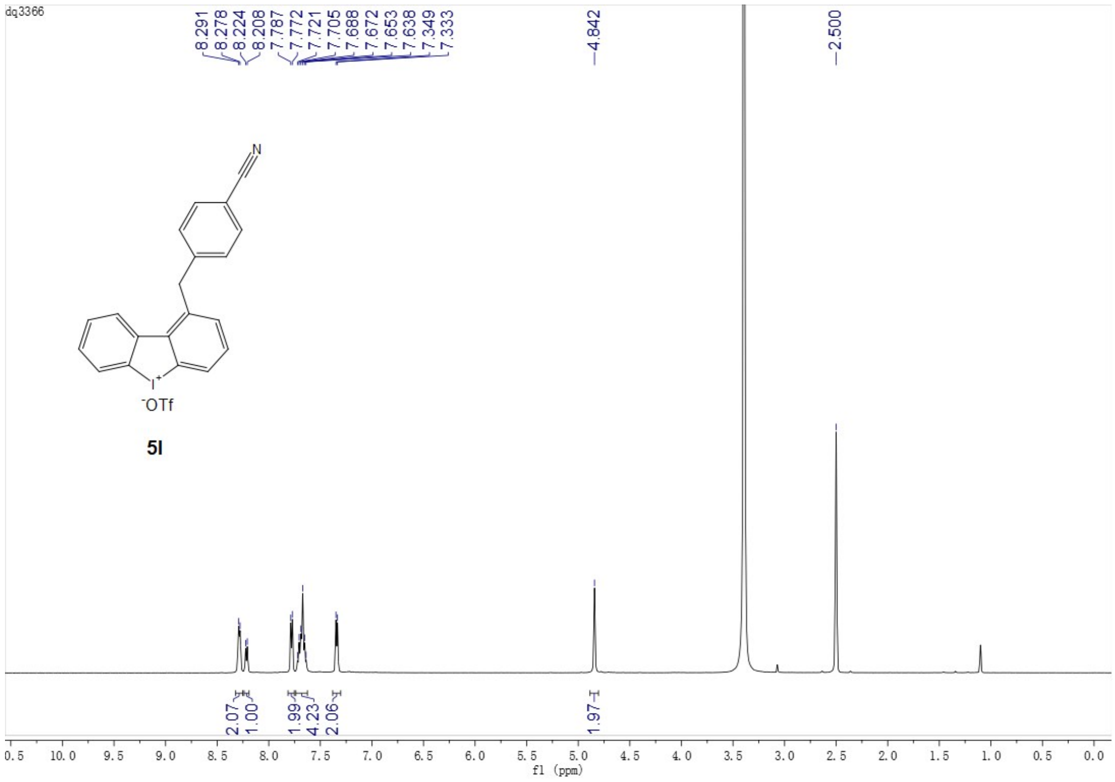

dq3454-c
143.53
141.72
139.52
138.40
133.64
130.86
130.64
130.03
129.74
129.70
129.65
129.47
128.83
128.51
122.58
122.01
121.33
119.45
40.02
39.85
39.69
39.52
39.35
39.19
39.02
I+
-OTf
5i
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)
1500 1400 1300 1200 1100 1000 900 800 700 600 500 400 300 200 100 0 -100

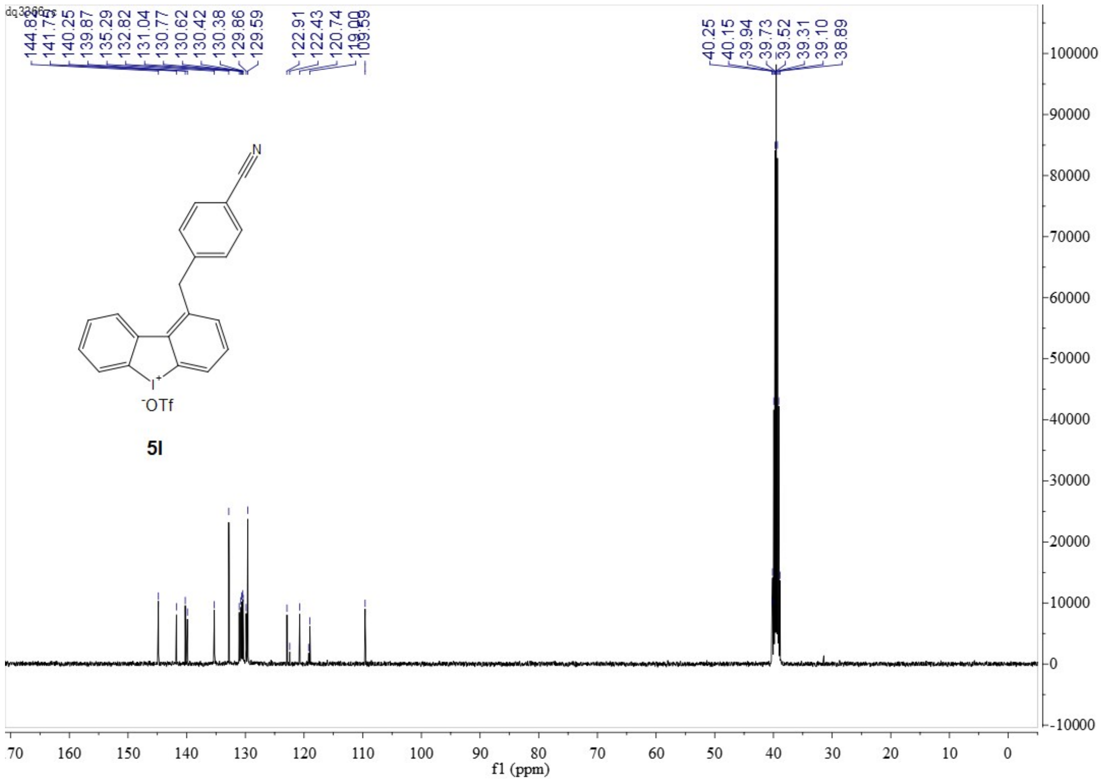

dq3260
8.319
8.297
8.294
8.284
8.276
8.274
8.260
7.712
7.695
7.691
7.677
7.673
7.663
7.646
7.333
7.315
7.296
7.239
7.221
7.155
7.137
4.729
2.509
2.505
2.500
2.496
2.491
-OTf
5j
3.03
4.20
2.00
1.08
2.00
1.87
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


dq3260-c
141.91
141.57
139.81
138.61
135.14
130.92
130.65
130.58
130.47
130.25
129.45
128.95
128.45
126.72
122.72
120.65
40.25
40.15
39.94
39.73
39.52
39.31
39.10
38.89
-OTf
5j
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


dq3347
8.356
8.340
8.293
8.277
8.267
8.252
7.753
7.739
7.724
7.667
7.652
7.638
7.331
7.315
7.078
7.062
4.669
2.500
1.232
H_3C
CH_3
CH_3
I+
-OTf
5k
1.03
2.06
1.09
3.03
2.00
1.98
1.96
9.08
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
f1 (ppm)


dq3347-c
148.98
141.96
141.84
139.70
135.46
135.05
130.91
130.68
130.52
130.43
130.17
129.35
128.17
125.62
122.60
122.06
120.59
119.50
40.02
39.85
39.69
39.52
39.35
39.19
39.02
34.24
31.24
H_3C
CH_3
CH_3
I+
-OTf
5k
4000 3800 3600 3400 3200 3000 2800 2600 2400 2200 2000 1800 1600 1400 1200 1000 800 600 400 200 0 -200
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


dq3366
8.291
8.278
8.224
8.208
7.787
7.772
7.721
7.705
7.688
7.672
7.653
7.638
7.349
7.333
4.842
2.500
N
I+
-OTf
5l
2.07
1.00
1.99
4.23
2.06
1.97
0.5
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
f1 (ppm)


dq3366.cs
144.82
141.75
140.25
139.87
135.29
132.82
131.04
130.77
130.62
130.42
130.38
129.86
129.59
122.91
122.43
120.74
119.00
109.59
40.25
40.15
39.94
39.73
39.52
39.31
39.10
38.89
N
I+
-OTf
5l
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
70
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)