Electronic Supplementary Material (ESI) for Journal of Materials Chemistry C.

Efficient pure near-infrared organic light-emitting diodes based on tris(2,4,6-trichlorophenyl) methyl radical derivatives

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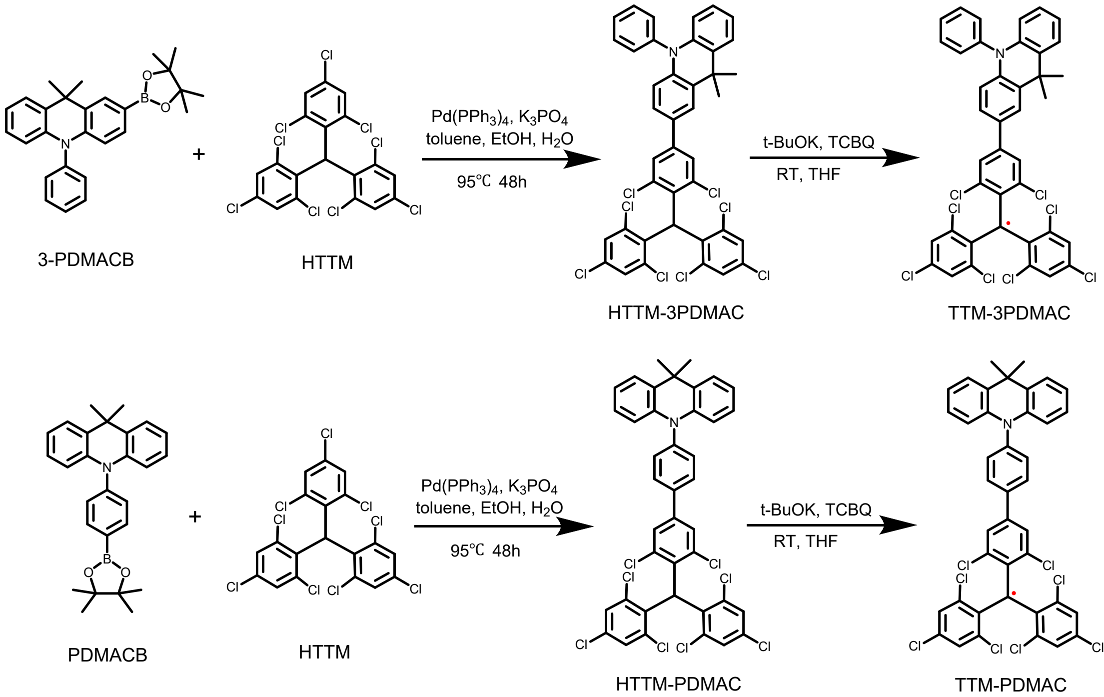
Contents

General information

All reagents and solvents required for synthesis and characterization are purchased from commercial suppliers and used directly without any treatment. A Bruker Avance-III 500 NMR spectrometer was used for the ^{1}H collection in $CDCl_3$ and CH_2Cl_2 solvent with tetramethylsilane as the internal standard. Mass spectra of all compounds were recorded on Thermo Fisher ITQ1100 GC-MS mass detector. A shimadzu UV-2550 spectrophotometer was applied to record the ultraviolet (UV)-visible spectra. Fluorescence spectra were recorded using a RF-5301 PC spectrophotometer and QE pro. All of PLQEs are determined with a calibrated integrating sphere system and using QE pro of Ocean Insight as fluorescence spectrometer. The electrochemical oxidation and reduction potentials were recorded using an electrochemical analyzer (CHI660C, CH Instruments, USA). The fluorescence decay spectra were recorded on an Edinburgh fluorescence spectrometer (FLS980), and the lifetime of the excited states was measured by the time-correlated single photon counting method under the excitation of a laser (375 nm). Thermal gravimetric analysis (TGA) were characterized by a TA INSTRUMENTS Q500 TGA analyzer. Ready-made indium tin oxide (ITO) glass substrates were purchased and cleaned. After dried with N_2, they were treated with UV irradiation for 20 min and next transferred to a vacuum deposition system with the pressure of $4\text{-}6\times10^{-6}$ mbar. The MoO_3 layer was deposited at a rate of 0.3 Å s^{-1}. All the organic layers were deposited at 0.4-0.6 Å s^{-1}. The evaporation rate of cathode LiF and Al metal layer were 0.1 Å s^{-1} and 0.8-1.4 Å s^{-1} respectively. The current-voltage characteristics were measured using a Keithley 2400 programmable electrometer. The EL spectra and EQEs were measured using QE pro spectroradiometer of Ocean Insight together with a calibrated integrating sphere at room temperature in glove box.

1. Synthesis



Scheme 1. Synthetic routes and chemical structures of TTM-3PDMAC and TTM-PDMAC.

The HTTM[1] and 3-PDMACB[2] and PDMACB[3] were prepared as reported.

(1) Synthesis of HTTM-3PDMAC

HTTM (1.0 g, 1.80 mmol) and the pinacol borane of 3-PDMAC (0.67 g, 1.80 mmol) was dissolved in a mixed solvent of toluene (12 ml), K_3PO_4 aqueous solution (8 ml, 2 mol / L) and ethanol (4 ml), and catalyst Pd(PPh$_3$)$_4$ (0.10 g, 0.09 mmol) was added under argon atmosphere. The mixture was stirred at 95℃ for 48 h under argon atmosphere. After the reaction mixture cooling to room temperature, the solution was extracted with dichloromethane, organic layer was collected and dried. The solvent was removed under vacuum and the crude product was purified by silica gel column chromatography (using petroleum ether: dichloromethane = 10:1 v/v). HTTM-3PDMAC was obtained as a white solid. GC-MS (m/z): calculated for $C_{40}H_{25}Cl_8N$, 802.94; found, 803.37; ^{1}H NMR (500 MHz, $CDCl_3$) δ7.68 – 7.62 (m, 3H), 7.56 – 7.46 (m, 3H) , 7.40 – 7.33 (m, 5H), 7.24 (t, J = 2.3 Hz, 2H), 7.19 (dd, J = 8.6, 2.0 Hz, 1H), 7.05 – 6.95 (m, 2H), 6.78 (s, 1H), 6.34 (d, J = 8.6 Hz, 1H), 6.31 (dd, J = 7.9, 1.0 Hz, 1H), 1.78 (s, 3H), 1.77 (s, 3H).

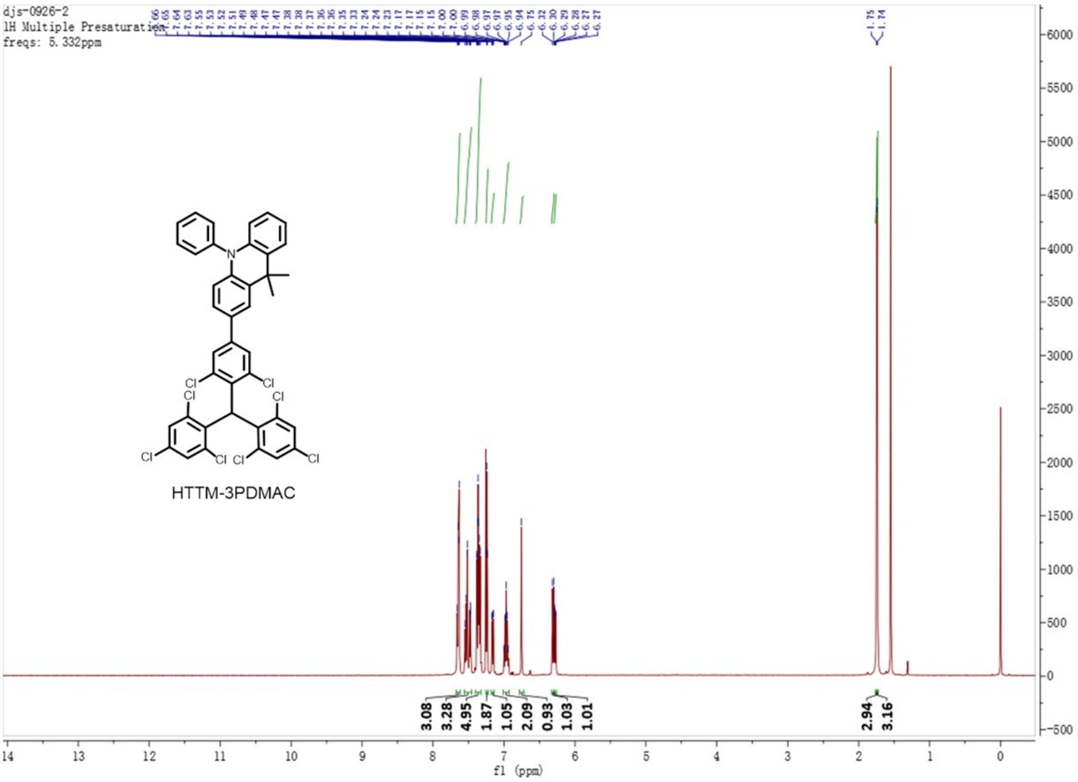


Figure S1. ^{1}H NMR spectrum of HTTM-3PDMAC in $CDCl_3$.

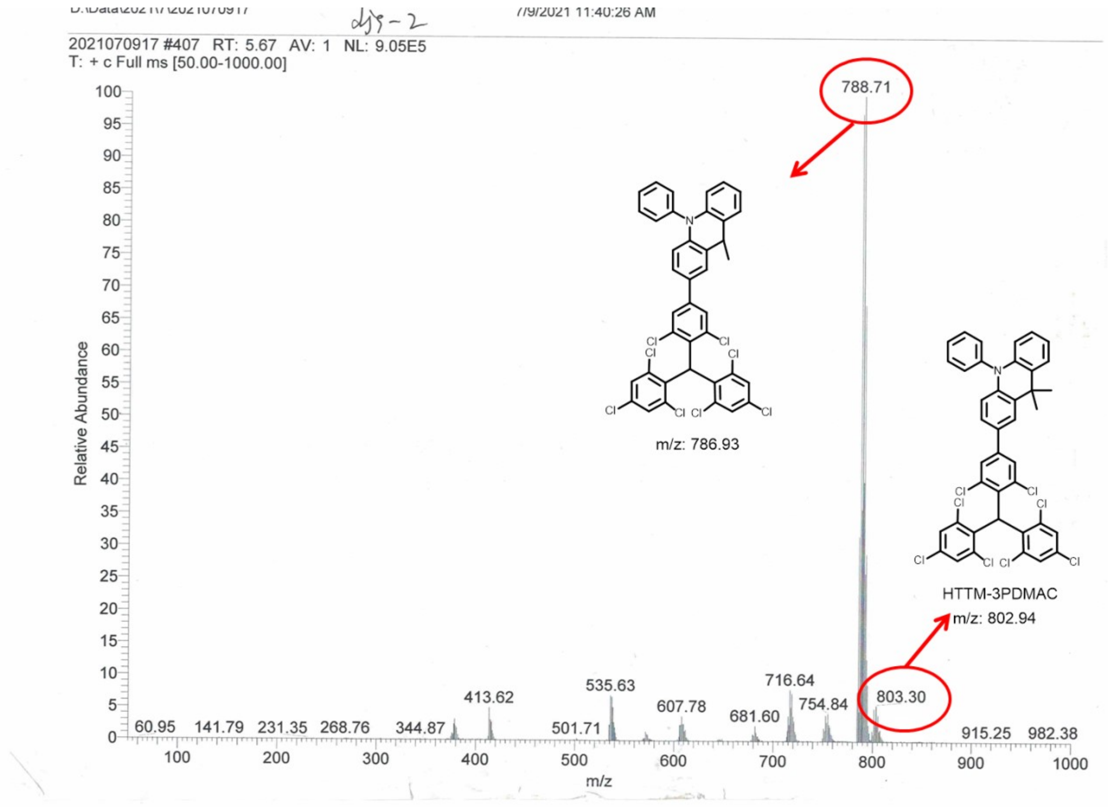


Figure S2. Mass Spectrum of HTTM-3PDMAC.

(2) Synthesis of TTM-3PDMAC

Under argon atmosphere and in the dark, the HTTM-3PDMAC (1.00 equiv) was dissolved in dry THF (40 ml). Then KOtBu (4.00 equiv) was added, the solution become claret-colored immediately. The solution was stirred for 5 h in the dark at room temperature, and then p-Chloranil (5 equiv) was added. The solution was stirred for further 1 h. After the reaction finished, the solvent was removed under vacuum and the crude product was purified by silica gel column chromatography (using petroleum ether: dichloromethane = 10:1v/v). The crude product was recrystallized twice from dichloromethane and methanol and a green solid was obtained. GC-MS (m/z): calculated for $C_{40}H_{25}Cl_8N$, 801.94; found, 802.46;

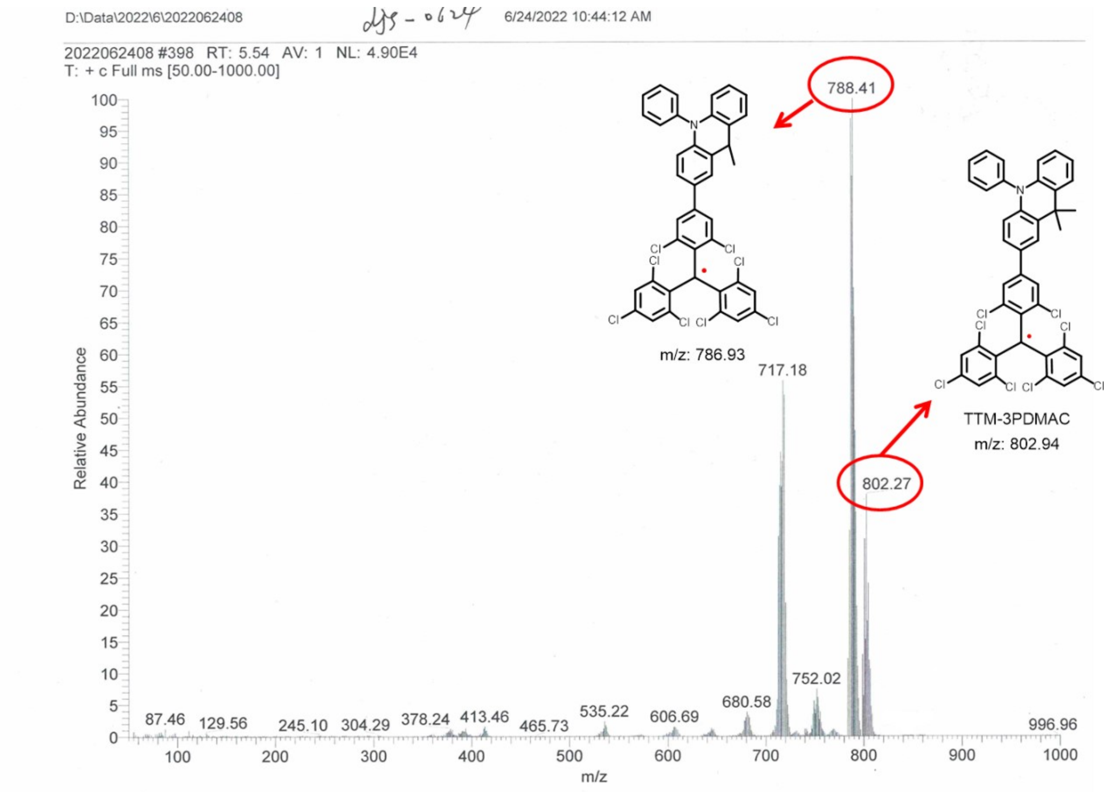


Figure S3. Mass Spectrum of TTM-3PDMAC.

(3) Synthesis of HTTM-PDMAC

HTTM (1.0 g, 1.80 mmol) and the pinacol borane of PDMAC (0.67 g, 1.80 mmol) was dissolved in a mixed solvent of toluene (12 ml), K_3PO_4 aqueous solution (8 ml, 2 M) and ethanol (4 ml), and catalyst $Pd(PPh_3)_4$ (0.10 g, 0.09 mmol) was added under argon atmosphere. The mixture was stirred at 95℃ for 48 h under argon atmosphere and in the dark. After the reaction mixture cooling to room temperature, the solution was extracted with dichloromethane, organic layer was collected and dried. The solvent was removed under vacuum and the crude product was purified by silica gel column chromatography (using petroleum ether: dichloromethane = 10:1 v/v). HTTM-PDMAC was obtained as a white solid. GC-MS (m/z): calculated for $C_{40}H_{25}Cl_8N$, 802.94; found, 803.37; ^1H NMR (500 MHz, CD_2Cl_2) δ 7.91 (d, J = 7.9 Hz, 2H), 7.77 (d, J = 1.8 Hz, 1H), 7.64 (d, J = 1.8 Hz, 1H), 7.54 – 7.44 (m, 5H), 7.34 (d, J = 2.0 Hz, 1H), 7.08 – 6.92

(m, 4H), 6.86 (s, 1H), 6.33 (d, J = 6.0 Hz, 2H), 1.72 (s, 6H).

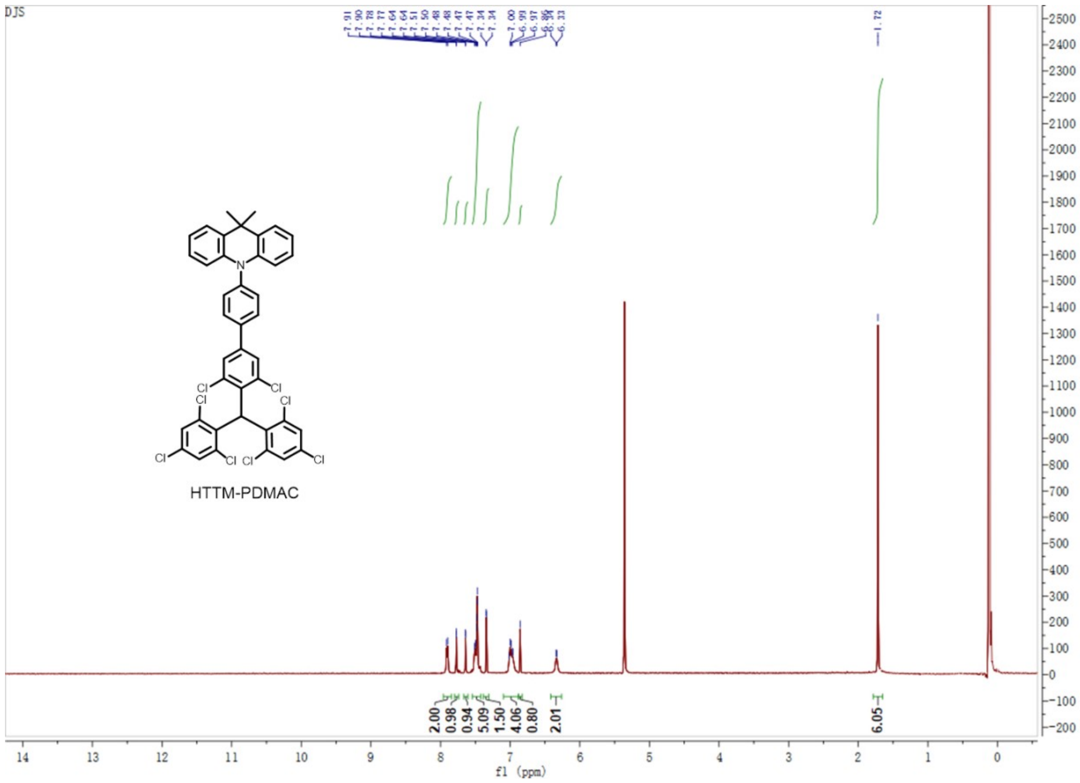


Figure S4. ^{1}H NMR spectrum of HTTM-PDMAC in CD_2Cl_2.

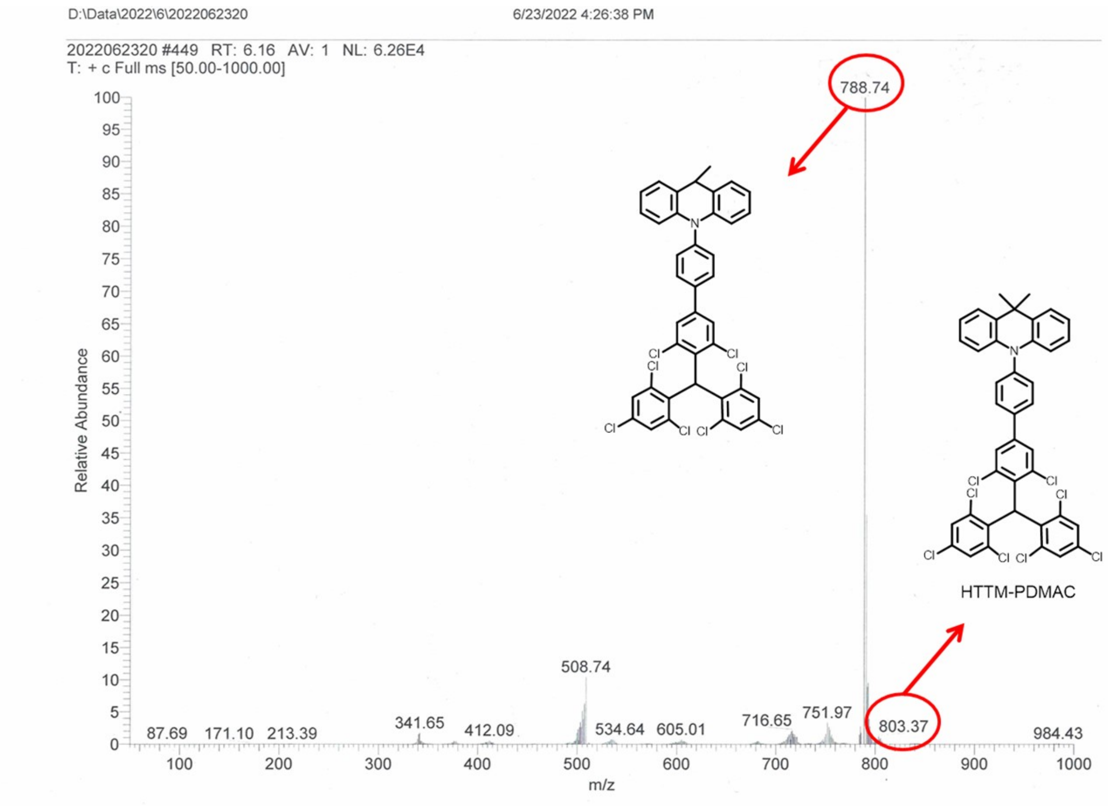


Figure S5. Mass Spectrum of HTTM-PDMAC.

(4) Synthesis of TTM-PDMAC

Under argon atmosphere and in the dark, the TTM-PDMAC (1.00 equiv) was dissolved in dry THF (40 ml). Then KOtBu (10.00 equiv) was added, the solution become claret-colored immediately. The solution was stirred for 5 h in the dark at room temperature, and then p-Chloranil (5 equiv) was added. The solution was stirred for further 1 h. After the reaction finished, the solvent was removed under vacuum and the crude product was purified by silica gel column chromatography (using petroleum ether: dichloromethane = 10:1v/v). The crude product was recrystallized twice from dichloromethane and methanol and a gray solid was obtained. GC-MS (m/z): calculated for $C_{40}H_{24}Cl_8N$, 801.94; found, 802.46.

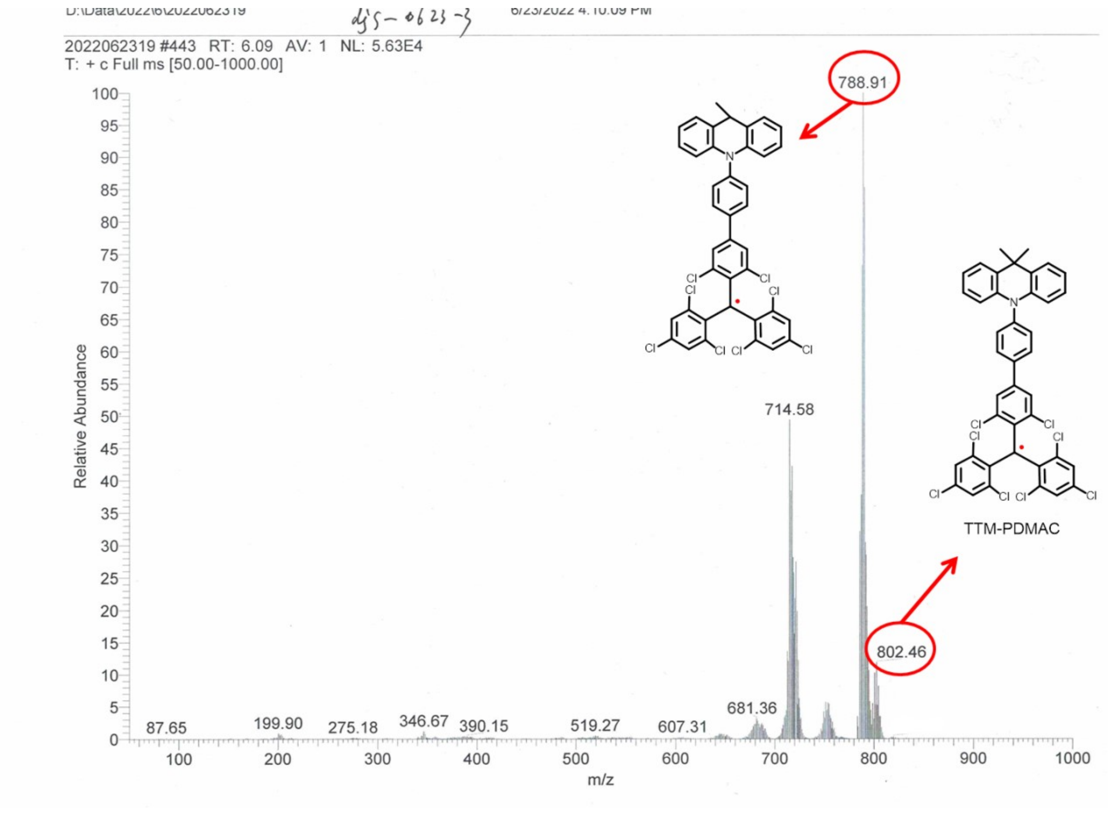


Figure S6. Mass Spectrum of TTM-PDMAC.

2. EPR spectra of TTM-3PDMAC and TTM-PDMAC

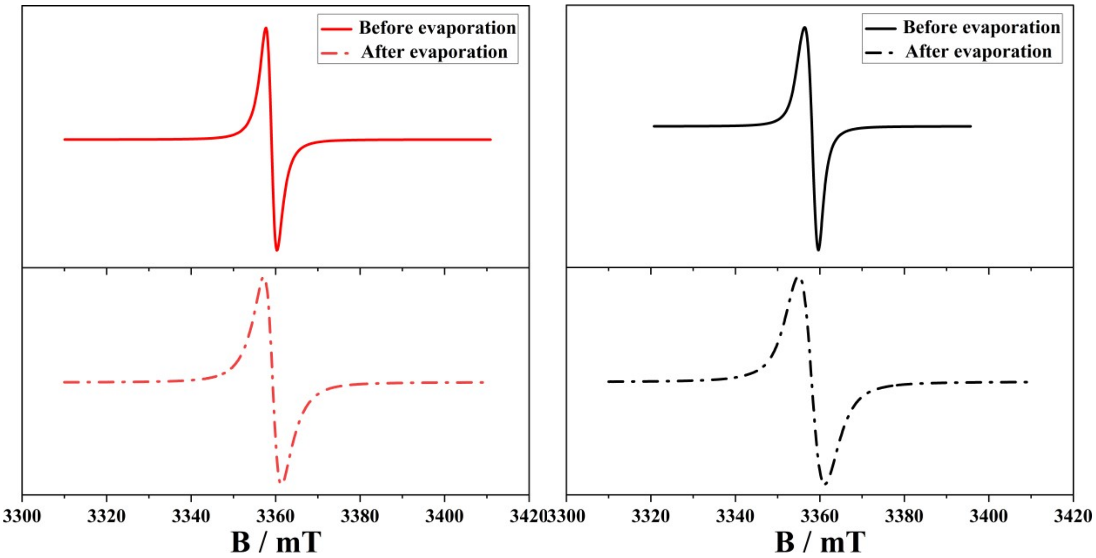
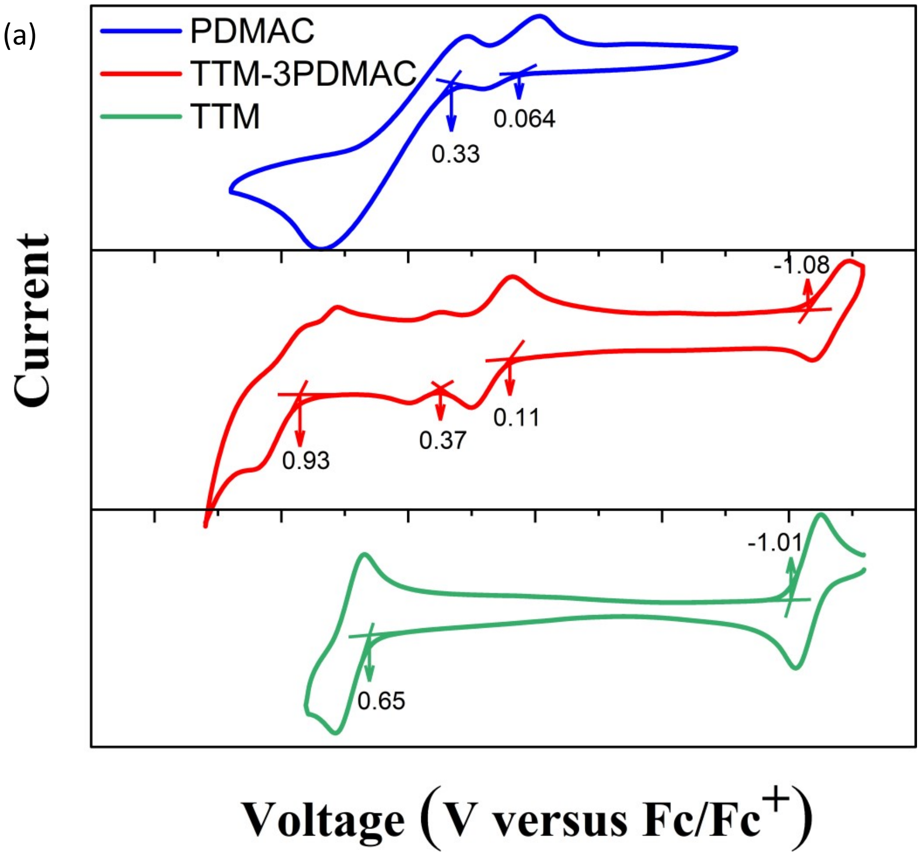


Figure S7. EPR spectra of TTM-3PDMAC (red) and TTM-PDMAC (black).

3. Electrochemical properties



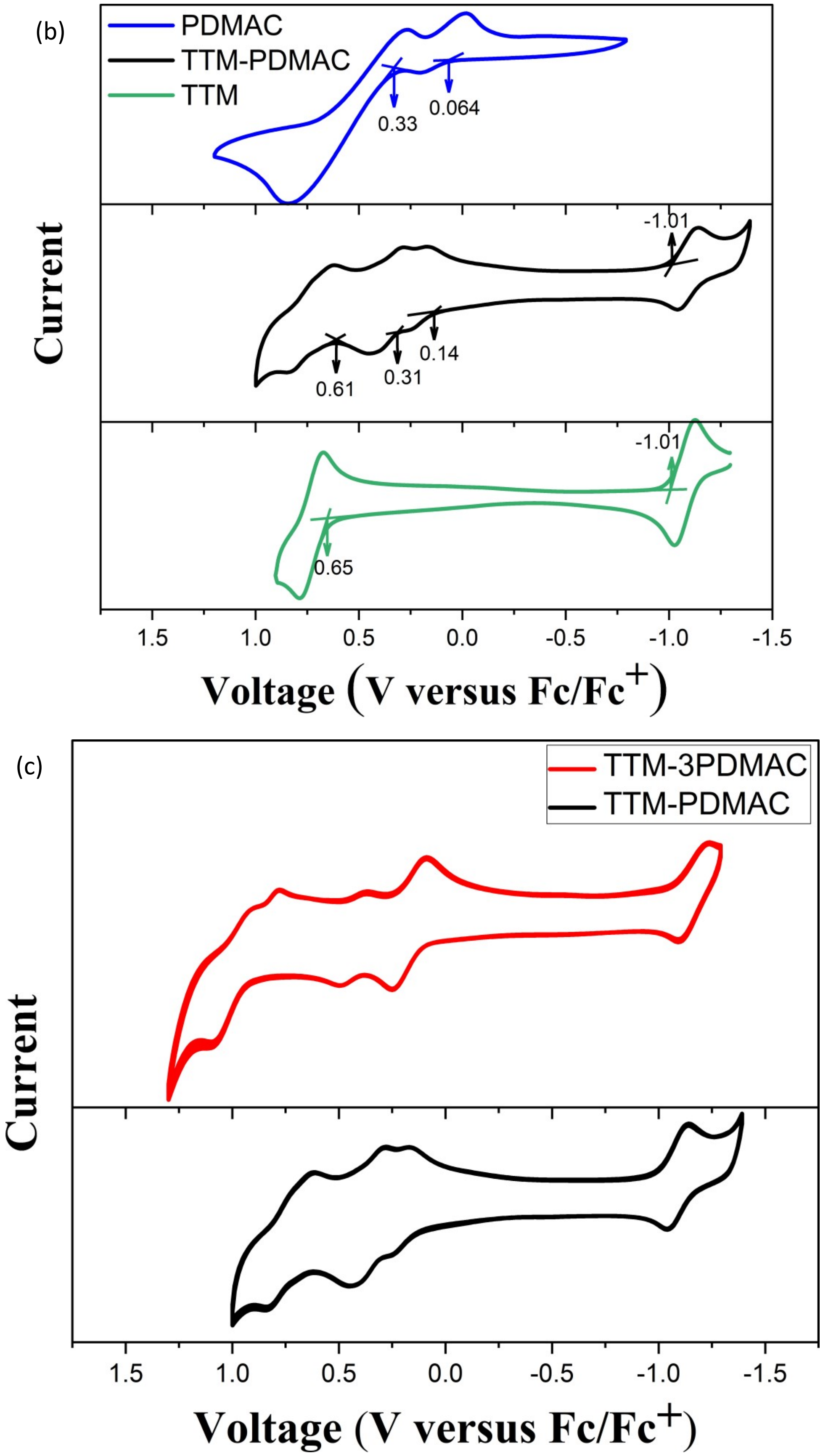


Figure S8. cyclic voltammetry measurements of TTM-3PDMAC (a) and TTM-PDMAC (b); Repeated cyclic voltammetry measurements (20 cycles) of TTM-3PDMAC and TTM-PDMAC (c).

4. Quantum chemical calculations

Table S1. Cartesian coordinates of the optimized ground state geometries by DFT calculation (UB3LYP/6-31G(d, p))

TTM-3PDMAC:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms)		
			X	Y	Z
1	6	0	0.012310	1.220087	-0.080889
2	6	0	-1.373197	1.175807	0.073692
3	6	0	-1.960242	0.146539	0.809655
4	6	0	-1.161769	-0.838416	1.390867
5	6	0	0.223709	-0.794198	1.236214
6	6	0	0.810783	0.235132	0.500324
7	6	0	-3.447244	0.099023	0.975713
8	6	0	-4.067126	1.259658	0.261791
9	6	0	-3.972421	-1.178787	0.399174
10	17	0	-2.357233	2.389624	-0.642748
11	17	0	-1.885281	-2.106856	2.297813
12	6	0	-4.265085	2.466246	0.932944
13	6	0	-4.842788	3.547636	0.267731
14	6	0	-5.222370	3.422404	-1.068583
15	6	0	-5.024331	2.215870	-1.739760
16	6	0	-4.446707	1.134426	-1.074524
17	6	0	-4.959891	-1.895065	1.075443
18	6	0	-5.449127	-3.085755	0.538268
19	6	0	-4.950963	-3.560009	-0.675164
20	6	0	-3.963579	-2.843701	-1.351477
21	6	0	-3.474257	-1.653041	-0.814258
22	17	0	-3.797129	2.620592	2.579806
23	17	0	-4.202676	-0.352628	-1.901614
24	17	0	-2.257296	-0.770210	-1.647670
25	17	0	-5.573923	-1.310475	2.570830
26	17	0	-5.553942	-5.027376	-1.337139
27	17	0	-5.934286	4.755124	-1.888337
28	6	0	2.138861	0.277615	0.352095
29	6	0	2.863023	-0.905169	0.207189
30	6	0	4.256012	-0.867155	0.042447
31	6	0	4.919569	0.369841	0.009810
32	6	0	4.186449	1.548530	0.180542
33	6	0	2.800358	1.503530	0.342282
34	6	0	5.034779	-2.156714	-0.094926
35	6	0	6.521720	-2.002042	-0.326363

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     37          7           0        6.317920    0.407234   -0.102219
     38          6           0        7.321857   -3.138524   -0.518701
     39          6           0        8.696111   -3.006767   -0.715361
     40          6           0        9.280767   -1.742421   -0.712737
     41          6           0        8.490763   -0.607466   -0.520244
     42          6           0        6.898426    1.525194    0.024014
     43          6           0        6.767982    2.249393    1.210153
     44          6           0        8.306732    3.264903   -0.880337
     45          6           0        7.666734    2.032236   -1.019911
     46          6           0        4.455947   -2.929522   -1.272742
     47          6           0        4.893888   -2.872442    1.242016
     48          1           0        0.475293    2.031813   -0.661187
     49          1           0        0.853380   -1.571016    1.694546
     50          1           0       -4.999017    4.499191    0.797009
     51          1           0       -5.323644    2.117159   -2.793642
     52          1           0       -6.227815   -3.650728    1.071606
     53          1           0       -3.570770   -3.217714   -2.308459
     54          1           0        2.339800   -1.872648    0.222095
     55          1           0        4.705690    2.518241    0.187454
     56          1           0        2.230347    2.436669    0.462031
     57          1           0        6.862898   -4.138193   -0.514336
     58          1           0        9.317910   -3.900465   -0.872423
     59          1           0       10.365535   -1.638137   -0.862416
     60          1           0        8.959304    0.387643   -0.505162
     61          1           0        6.159188    1.843047    2.031281
     62          1           0        8.912491    3.660039   -1.709146
     63          1           0        7.769600    1.461717   -1.954751
     64          1           0        4.597667   -2.342731   -2.207815
     65          1           0        4.978284   -3.907852   -1.366558
     66          1           0        3.369417   -3.102659   -1.104677
     67          1           0        4.380329   -3.847932    1.088900
     68          1           0        5.903527   -3.049370    1.675714
     69          1           0        4.295142   -2.243385    1.938118
     70          6           0        8.185968    4.006303    0.306316
     71          1           0        8.681479    4.949922    0.400935
     72          6           0        7.405328    3.491079    1.366963
     73          1           0        7.297727    4.036581    2.281156
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TTM-PDMAC:

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 Center     Atomic      Atomic             Coordinates (Angstroms)
 Number     Number       Type             X           Y           Z
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    1          6           0        4.247801    1.135347    0.516725
    2          6           0        3.667047   -0.160854    0.989585
    3          6           0        4.241756   -1.286491    0.187301
    4          6           0        2.180211   -0.134611    0.817428
    5          6           0        3.419779   -2.030204   -0.659302
    6          6           0        3.955291   -3.079114   -1.406743
    7          6           0        5.312725   -3.384153   -1.307623
    8          6           0        6.134715   -2.640397   -0.461111
    9          6           0        5.599190   -1.591530    0.286419
   10          6           0        4.638850    1.279979   -0.814310
   11          6           0        5.180090    2.487721   -1.254844
   12          6           0        5.330126    3.550757   -0.364368
   13          6           0        4.938998    3.406168    0.966623
   14          6           0        4.397837    2.198382    1.407199
   15          6           0        1.559851    0.994684    0.283259
   16          6           0        0.174441    1.019146    0.122975
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   18          6           0        0.029868   -1.215021    1.030819
   19          6           0        1.415289   -1.239460    1.191200
   20         17           0        2.179910   -2.631212    1.849488
   21         17           0        4.453787   -0.030105   -1.911759
   22         17           0        2.502607    2.356250   -0.177514
   23         17           0        6.612160   -0.674893    1.329793
   24         17           0        5.972633   -4.676795   -2.228797
   25         17           0        5.997113    5.039139   -0.907330
   26          6           0       -1.918413   -0.062226    0.343073
   27         17           0        3.915846    2.020069    3.047580
   28         17           0        1.746879   -1.654142   -0.781544
   29          6           0       -2.566665   -1.125605   -0.285006
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   32          6           0       -4.040873    1.050174    0.650504
   33          6           0       -2.655449    1.025648    0.810843
   34          7           0       -5.946499    0.009120   -0.123242
   35          6           0       -6.530968    1.164541   -0.664285
   36          6           0       -7.928997    1.293197   -0.686538
   37          6           0       -8.808442    0.171969   -0.178861
   38          6           0       -8.082405   -1.074879    0.275805
   39          6           0       -6.679062   -1.122901    0.265271
   40          6           0       -5.735106    2.229150   -1.099184
   41          6           0       -6.321263    3.397414   -1.590111
   42          6           0       -7.708513    3.516609   -1.633394
   43          6           0       -8.511425    2.470808   -1.179505
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     45          6           0       -8.152378   -3.336901    1.151660
     46          6           0       -6.759980   -3.375840    1.162256
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     48          6           0       -9.757942   -0.223026   -1.302230
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     51          1           0        7.205243   -2.880953   -0.383007
     52          1           0        5.488591    2.601806   -2.304516
     53          1           0        5.057285    4.244555    1.668845
     54          1           0       -0.314859    1.909763   -0.298198
     55          1           0       -0.573401   -2.086349    1.325527
     56          1           0       -1.985399   -1.983574   -0.653812
     57          1           0       -4.463335   -1.939572   -0.940826
     58          1           0       -4.622164    1.908071    1.019442
     59          1           0       -2.144170    1.864237    1.306182
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     61          1           0       -5.687252    4.224191   -1.942915
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     64          1           0       -9.912820   -2.158089    0.712968
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     66          1           0       -6.237994   -4.278633    1.512248
     67          1           0       -4.927202   -2.300429    0.752361
     68          1           0       -9.171672   -0.611563   -2.164840
     69          1           0      -10.452029   -1.013970   -0.939711
     70          1           0      -10.343726    0.667354   -1.622932
     71          1           0      -10.609417    0.723079    0.890125
     72          1           0       -9.269614    0.077329    1.934214
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5. Electroluminescence Performances

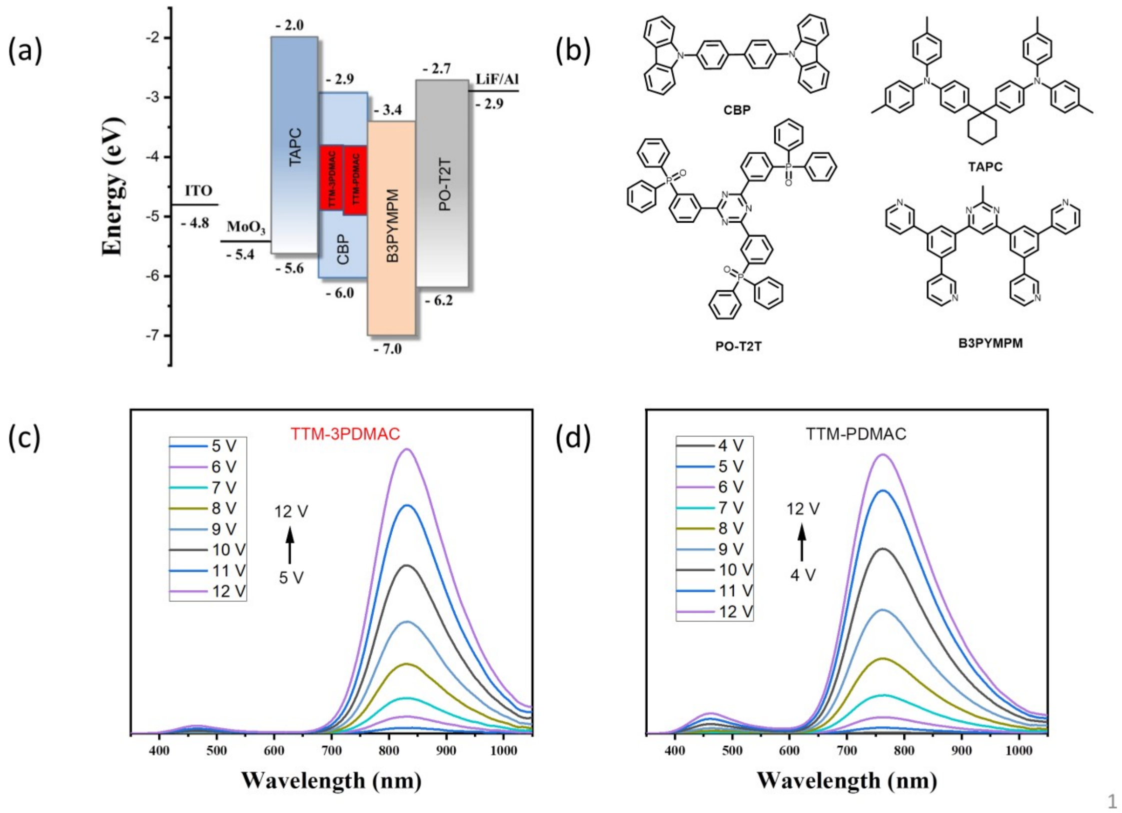

Figure S9. Energy-level diagram and electroluminescent properties of TTM-3PDMAC and TTM-PDMAC. a) Energy-level diagram of device; b) Materials used in this work; c) EL spectra of TTM-3PDMAC from 5-12V; d) EL spectra of TTM-PDMAC from 4-12V.

6. Summary of device performances of near infrared (NIR) OLED published to date

Table S2. Summary of the device performances of NIR OLED published to date with maximum electroluminescent (EL) wavelength over 800 nm.

λ_{EL}^{max} (nm)	EQE (%)	Emitter material	[ref]	λ_{EL}^{max} (nm)	EQE (%)	Emitter material	[ref]
800	1.9	metal free emitter	4	800	1	metal complex emitter	5
802	0.43	metal free emitter	6	803	9.58	metal complex emitter	7
804	2.2	metal free emitter	8	811	0.97	metal complex emitter	9
810	0.51	metal free emitter	10	814	1.5	metal complex emitter	11
814	0.5	metal free emitter	12	826	0.49	metal complex emitter	13
823	0.27	metal free emitter	14	847	0.19	metal complex emitter	15
824	0.16	metal free emitter	16	848	2.8	metal complex emitter	17
828	0.41	metal free emitter	10	846	1.5	metal complex emitter	17
830	2.47	metal free emitter	18	855	1	metal complex emitter	19
830	3.1	metal free emitter	this work	890	3.8	metal complex emitter	20
838	0.58	metal free emitter	21	900	3.8	metal complex emitter	22
840	1.12	metal free emitter	23	920	1.9	metal complex emitter	24
840	3.8	metal free emitter	25	1005	0.2	metal complex emitter	22
850	0.14	metal free emitter	14	1060	0.022	metal complex emitter	26
852	0.3	metal free emitter	10	1060	0.3	metal complex emitter	27
864	0.2	metal free emitter	6				

868	0.09	metal free emitter	[14]					
870	0.02	metal free emitter	[14]					
883	0.1	metal free emitter	[28]					
890	0.015	metal free emitter	[29]					
894	0.23	metal free emitter	[10]					
895	0.091	metal free emitter	[30]					
901	1.1	metal free emitter	[31]					
904	0.019	metal free emitter	[32]					
905	1.32	metal free emitter	[33]					
916	0.07	metal free emitter	[21]					
939	0.006	metal free emitter	[30]					
960	0.009	metal free emitter	[28]					
990	0.018	metal free emitter	[30]					
1010	0.003	metal free emitter	[34]					
1050	0.05	metal free emitter	[35]					
1050	0.16	metal free emitter	[36]					
1050	0.33	metal free emitter	[36]					
1080	0.73	metal free emitter	[36]					

7. References

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