Supplementary Information (SI) for Organic Chemistry Frontiers.
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Supplementary Information

# Tricarbazole-based donor–acceptor architectures with diverse acceptors: synthesis, properties and applications

Huabi Xie,[a] Yu Zuo,[a] Chengye Yuan,[a] Peng Xu,[a] Ningwen Sun,[a] Hongxing Jia,[b] Jinjin Ding,[c] Qiang Huang,[c] and Jinling Zhang[a]*

[a]Department of Chemistry and Chemical Engineering, Chongqing University of Science and Technology, Chongqing 401331, China

[b]College of Materials Science and Engineering, National Engineering Research Center for Magnesium Alloys, Chongqing University, Chongqing 400044, China

[c]School of Chemistry and Chemical Engineering, Nantong University, Nantong 226019, China

**E-mail: zhangjinling@cqust.edu.cn*

# Table of Contents

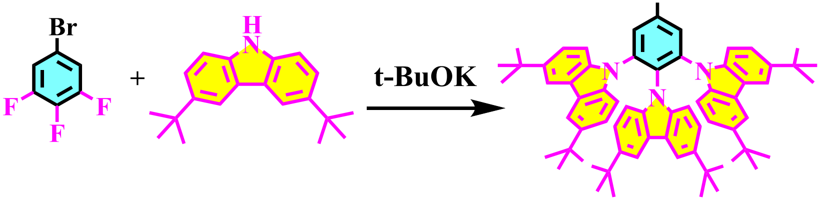
## 1 General Remarks

All solvents for syntheses were purified by distillation under nitrogen prior to use. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Air-sensitive reactions were all carried out under nitrogen. Flash chromatography was performed on silica gel (300−400 mesh) using petroleum ether/dichloromethane (PE/DCM) or petroleum ether/ethyl acetate (PE/EA) as eluent.

Thin layer chromatography (TLC) analysis used silica gel GF254 plates with UV light to visualization. NMR spectra were recorded on a Bruker ADVANCE III ($^1$H 400 MHz, $^{13}$C 100 MHz) spectrometer. Chemical shifts were reported as the delta scale in ppm relative to tetramethylsilane ($\delta$ = 0.00 ppm), $CHCl_3$ ($\delta$ = 7.26 ppm) for $^1$H NMR and $CDCl_3$ ($\delta$ = 77.16 ppm) for $^{13}$C NMR. Data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd, = doublet of doublets, m = multiplet), coupling constant (Hz), and integration. High resolution mass spectrometry (HR-MS) and low resolution electrospray ionization mass spectrometry data were recorded on a Thermo Scientific Q Exactive Focus spectrometer, using APCI-Orbitrap-MS technique.

## 2 Synthetic procedures and characterization data

### Synthesis of compound 5

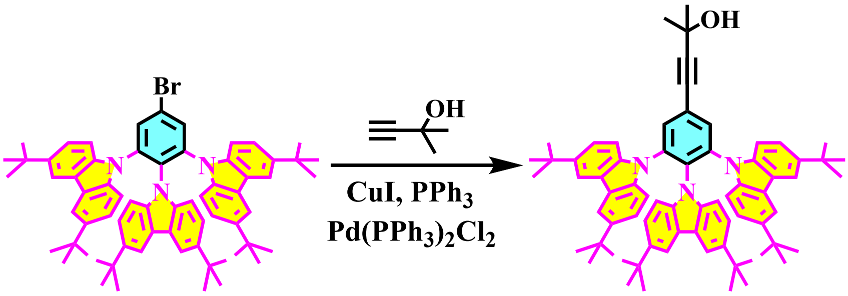


3 4 5

To a solution of **4** (7.1 g, 25.4 mmol) and t-BuOK (3.2 g, 28.5 mmol) in dry DMF (90 mL) under an inert nitrogen atmosphere was added **3** (1.5 g, 7.1 mmol) via a syringe.

The above solution was heated to 60 °C (oil bath) with stirring for 48 h. After cooling to room temperature, the reaction solvent was removed under reduced pressure and vacuum conditions. The residue was purified by silica gel flash chromatography (PE/DCM) to afford bromide **5** (5.2 g, 74% yield) as a white solid. $^1$H NMR (400 MHz, CDCl$_3$): δ 7.97 (s, 2H), 7.77 (d, $J$ = 1.6 Hz 4H), 7.30 (d, $J$ = 1.6 Hz, 2H), 7.10 – 7.04 (m, 8H), 6.74 (d, $J$ = 8.4 Hz, 2H), 6.64 (dd, $J$ = 8.4, 1.6 Hz, 2H), 1.39 (s, 36H), 1.26 (s, 18H). $^{13}$C NMR (100 MHz, CDCl$_3$): δ 143.2, 142.6, 139.0, 138.7, 137.5, 132.2, 132.1, 124.3, 124.1, 123.2, 122.5, 121.1 115.9, 115.1, 109.8, 109.5, 34.7, 34.4, 32.1, 31.9. FT-IR (KBr): $\nu_{\max}$ 3044, 2960, 2900, 2865, 1598, 1550, 1488, 1469, 1361, 1296, 1263, 1232, 871, 811, 739, 656. MS (ESI, m/z) calcd for $C_{66}H_{75}BrN_3$ [M + H]$^+$ 988.51, Found: 988.62. Anal. calcd for $C_{66}H_{74}BrN_3$: C, 80.13; H, 7.54; N, 4.25. Found: C, 80.22; H, 7.45; N, 4.31.

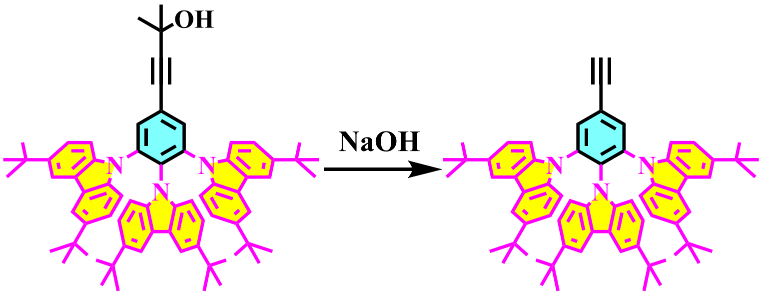
**Synthesis of compound 6**



**5** **6**

To a solution of **5** (5.0 g, 5.1 mmol) and 2-methylbut-3-yn-2-ol (3.5 g, 41.6 mmol) in THF (60 mL) and triethylamine (20 mL) in a 250 mL Schlenk tube with a stir bar was added Pd(PPh$_3$)$_2$Cl$_2$ (101.1 mg, 144.0 μmol), CuI (30.4 mg, 159.6 μmol), and PPh$_3$ (98.7 mg, 376.3 μm) successively. After removal of the oxygen by five consecutive freeze-pump-thaw cycles, the reaction mixture was allowed to warm to room temperature and then heated to 80 °C (oil bath) with stirring for 34 h. Then the reaction solvent was removed under vacuum and the residue was purified by flash chromatography on silica gel (PE/DCM) to afford hydroxy compound **6** (4.3 g, yield 85%) as a light yellow solid. $^1$H NMR (400 MHz, $CDCl_3$): $\delta$ 7.89 (m, 2H), 7.79 (m, 4H), 7.31 (m, 2H), 7.11 –7.07 (m, 8H), 6.78 – 6.75 (m, 2H), 6.66 – 6.63 (m, 2H), 1.68 (m, 6H), 1.40 (m, 36H), 1.27 (m, 18H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 143.0, 142.5, 138.8, 138.0, 137.0, 137.6, 133.0, 132.3, 124.3, 124.0, 123.7, 123.1, 122.4, 115.8, 115.0, 109.8, 109.5, 80.4, 65.8, 34.7, 34.4, 32.1, 31.9, 31.5. FT-IR (KBr): $\nu_{max}$ 3448, 3046, 2958, 2903, 2865, 1595, 1547, 1488, 1473, 1359, 1320, 1294, 1263, 876, 806, 775, 739. MS (ESI, m/z) calcd for $C_{71}H_{82}N_3O$ $[M + H]^+$ 992.65, Found: 992.79. Anal. calcd for $C_{71}H_{81}N_3O$: C, 85.93; H, 8.23; N, 4.23. Found: C, 85.98; H, 8.16; N, 4.29.

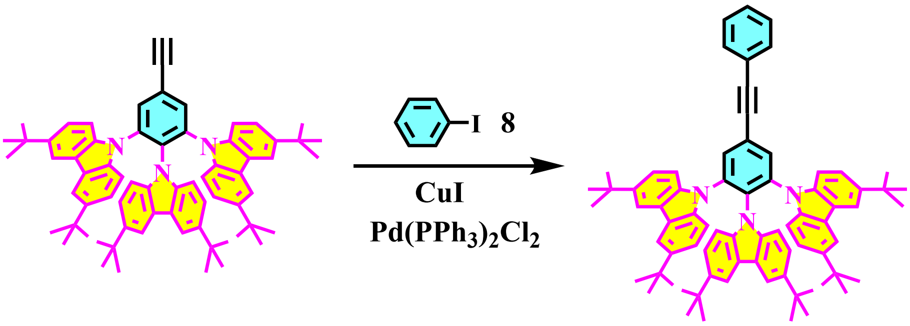
### Synthesis of compound 7



**6** **7**

To a solution of hydroxy compound **6** (2.2 g, 2.2 mmol) in toluene (60 mL) was added NaOH (320.0 mg, 8.0 mmol). Removal of oxygen by five consecutive freeze-pump-thaw cycles was performed. Subsequently, the reaction mixture was heated to vigorous reflux (oil bath) and stirred for 8 h. After being cooled to room temperature, the mixture was placed in an ice bath and quenched by the addition of hydrochloric acid (6.0 M, 10 mL, 60.0 mmol). The organic layer was separated, and the aqueous layer was extracted with EA. The combined organic layers were concentrated under vacuum. The residue was purified by flash chromatography on silica gel (PE/DCM) to afford **7** (1.9 g, 92% yield) as a light yellow solid. $^1$H NMR (400 MHz, $CDCl_3$): δ 7.93 (s, 2H), 7.76 (d, $J$ = 0.8 Hz, 4H), 7.28 (d, $J$ = 1.6 Hz, 2H), 7.08 – 7.02 (m, 8H), 6.73 (d, $J$ = 8.4 Hz, 2H), 6.61 (dd, $J$ = 8.4, 1.6 Hz, 2H), 3.26 (s, 1H), 1.37 (s, 36H), 1.24 (s, 18H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 143.0, 142.6, 138.7, 138.1, 137.5, 133.6, 132.7, 124.4, 124.0, 123.2, 123.0, 122.4, 115.9, 115.0, 109.8, 109.5, 81.7, 80.1, 34.7, 34.4, 32.1, 31.9. FT- IR (KBr): $\nu_{max}$ 3304, 3046, 2953, 2900, 2865, 1595, 1540, 1488, 1466, 1361, 1292, 1263, 1033, 871, 806, 739cm$^{-1}$. MS (ESI, m/z) calcd for $C_{68}H_{76}N_3$ $[M + H]^+$ 934.60, Found: 934.73. Anal. calcd for $C_{68}H_{75}N_3$: C, 87.41; H, 8.09; N, 4.50. Found: C, 87.55; H, 8.03; N, 4.58.

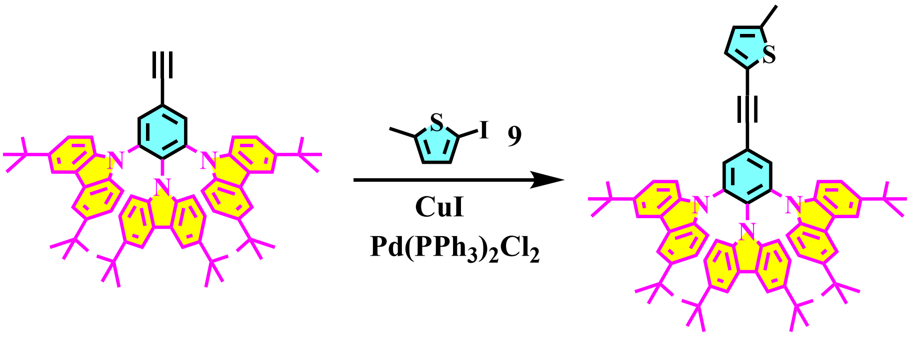
**Synthesis of compound TrCzDAa**



**7** **TrCzDAa**

To a solution of **7** (140.7 mg, 150.6 μmol) and iodide **8** (153.4 mg, 751.9 μmol) in THF (5 mL) and triethylamine (5 mL) in a 50 mL Schlenk tube with a stir bar was added $Pd(PPh_3)_2Cl_2$ (2.8 mg, 4.0 μmol) and CuI (1.0 mg, 5.3 μmol), successively. After removal of the oxygen by five consecutive freeze-pump-thaw cycles, the reaction mixture was allowed to warm to room temperature and then heated to 60 °C (oil bath) with stirring for 2 h. Then the reaction solvent was removed under vacuum and the residue was purified by flash chromatography on silica gel (PE/DCM) to afford **TrCzDAa** (117.6 mg, yield 77%) as a solid. $^1$H NMR (400 MHz, $CDCl_3$): δ 7.98 (m, 2H), 7.78 (s, 4H), 7.59 – 7.57 (m, 2H), 7.39 – 7.36(m, 3H), 7.30 (m, 2H), 7.09 (s, 8H), 6.78 (d, *J* = 8.8 Hz, 2H), 6.64 (dd, *J* = 8.4, 1.6 Hz, 2H), 1.38 (m, 36H), 1.26 (m, 18H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 142.5, 138.8, 138.0, 137.6, 132.9, 132.2, 131.9, 129.0, 128.6, 124.3, 124.0, 123.2, 122.6, 122.4, 115.8, 115.0, 109.9, 109.6, 92.3, 87.5, 34.7, 34.4, 32.1. FT-IR (KBr): $\nu_{max}$ 2961, 2903, 2862, 1630, 1603, 1487, 1474, 1360, 1322, 1295, 1264, 875, 799, 751 cm$^{-1}$. HR MS (APCI) m/z: Calcd for $C_{74}H_{80}N_3$ $[M + H]^+$: 1010.6347, Found: 1010.6339; Calcd for $C_{70}H_{72}N_3$ $[M + H – C_4H_8]^+$: 954.5721, Found: 954.5710.

**Synthesis of compound TrCzDAb**

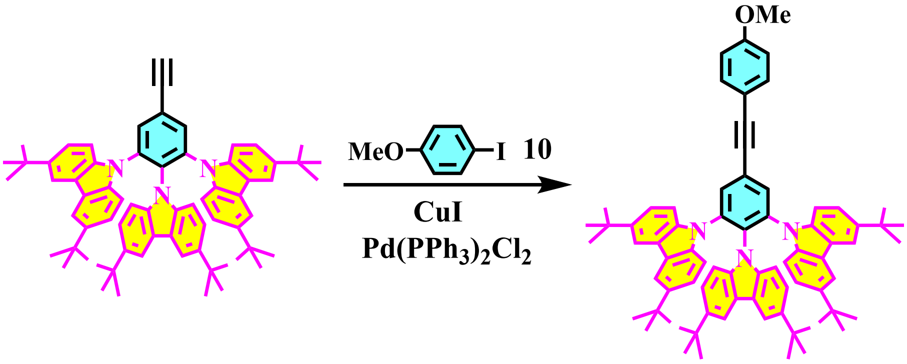


7 **TrCzDAb**

To a solution of **7** (143.7 mg, 153.8 μmol) and iodide **9** (67.9 mg, 303.0 μmol) in THF (5 mL) and triethylamine (5 mL) in a 50 mL Schlenk tube with a stir bar was added $Pd(PPh_3)_2Cl_2$ (2.4 mg, 3.4 μmol) and CuI (1.0 mg, 5.3 μmol), successively. After removal of the oxygen by five consecutive freeze-pump-thaw cycles, the reaction mixture was allowed to warm to room temperature and then heated to 60 °C (oil bath) with stirring for 2 h. The reaction solvent was removed under vacuum and the residue was purified by flash chromatography on silica gel (PE/DCM) to afford **TrCzDAb**

(90.2 mg, 57% yield) as a light yellow solid. $^{1}H$ NMR (400 MHz, $CDCl_3$): δ 7.96 – 7.92 (m, 2H), 7.80 – 7.77 (m, 4H), 7.32 – 7.29 (m, 2H), 7.17 – 7.15 (m, 1H), 7.11 – 7.08 (m, 8H), 6.81 – 6.75 (m, 2H), 6.68 – 6.62 (m, 3H), 2.49 (s, 3H), 1.41 – 1.38 (m, 36H), 1.29 – 1.26(m, 18H). $^{13}C$ NMR (100 MHz, $CDCl_3$): δ 143.3, 143.0, 142.9, 142.5, 138.8, 138.0, 137.64, 137.61, 133.25, 133.23, 131.8, 125.8, 124.4, 124.3, 124.0, 123.98, 123.2, 122.4, 120.0, 115.8, 115.0, 109.9, 109.6, 90.49, 90.47, 34.69, 34.67, 34.42, 34.40, 32.1, 31.9, 15.6. FT-IR (KBr): $\nu_{max}$ 2961, 2903, 2864, 2202, 1630, 1606, 1547, 1487, 1474, 1360, 1295, 1235, 1264, 1033, 875, 799, 739, 606 $cm^{-1}$; HR MS (APCI) m/z: Calcd for $C_{73}H_{80}N_3S$ $[M + H]^+$: 1030.6068, Found: 1030.6055; Calcd for $C_{69}H_{72}N_3S$ $[M + H – C_4H_8]^+$: 974.5442, Found: 974.5448.

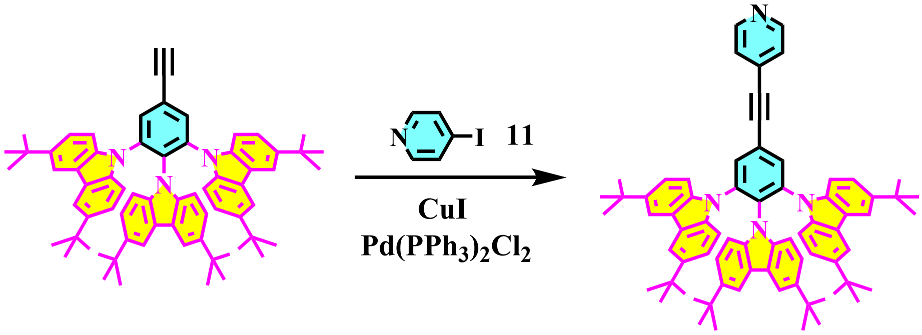
**Synthesis of compound TrCzDAc**



**7** **TrCzDAc**

To a solution of **7** (102.5 mg, 109.7 μmol) and iodide **10** (103.7 mg, 443.1 μmol) in THF (4 mL) and triethylamine (4 mL) in a 50 mL Schlenk tube with a stir bar was added $Pd(PPh_3)_2Cl_2$ (3.0 mg, 4.3 μmol) and CuI (1.3 mg, 6.8 μmol), successively. After removal of the oxygen by five consecutive freeze-pump-thaw cycles, the reaction mixture was allowed to warm to room temperature and then heated to 60 °C (oil bath) with stirring for 2 h. The reaction solvent was removed under vacuum and the residue was purified by flash chromatography on silica gel (PE/DCM) to afford **TrCzDAc** (99.7 mg, 87% yield) as a light yellow solid. $^1$H NMR (400 MHz, $CDCl_3$): δ 7.94 (s, 2H), 7.77 (s, 4H), 7.50 (d, $J$ = 8.8 Hz, 2H), 7.29 (d, $J$ = 1.6 Hz, 2H), 7.08 (s, 8H), 6.89 (d, J = 8.8 Hz, 2H), 6.77 (d, $J$ = 8.8 Hz, 2H), 6.62 (dd, $J$ = 8.4, 1.6 Hz, 2H), 3.81 (s, 3H), 1.38 (s, 36H), 1.25 (s, 18H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 160.3, 143.0, 142.5, 138.9, 138.0, 137.8, 133.5, 132.6, 132.1, 124.8, 124.4, 124.1, 123.2, 122.5, 115.9, 115.1, 114.8, 114.4 110.0, 109.7, 92.6, 86.4, 55.5, 34.8, 34.5, 32.2, 32.0. FT-IR (KBr): $\nu_{\max}$ 2961, 2903, 2864, 2212, 1630, 1607, 1511, 1487, 1474, 1350, 1295, 1251, 1169, 799, 606cm$^{-1}$. HR MS (APCI) m/z: Calcd for $C_{75}H_{82}N_3O$ $[M + H]^+$: 1040.6453, Found: 1040.6426; Calcd for $C_{71}H_{74}N_3O$ $[M + H - C_4H_8]^+$: 984.5827, Found: 984.5785; Calcd for $C_{75}H_{85}N_4O$ $[M + NH_4]^+$: 1057.6718, Found: 1057.6761.

**Synthesis of compound TrCzDAd**



To a solution of **7** (143.1 mg, 153.2 μmol) and iodide **11** (64.0 mg, 312.2 μmol) in THF (5 mL) and triethylamine (5 mL) in a 50 mL Schlenk tube with a stir bar was added $Pd(PPh_3)_2Cl_2$ (3.6 mg, 5.1 μmol) and CuI (1.6 mg, 8.4 μmol), successively. After removal of the oxygen by five consecutive freeze-pump-thaw cycles, the reaction mixture was allowed to warm to room temperature and then heated to 60 °C (oil bath) with stirring for 2 h. The reaction solvent was removed under vacuum and the residue was purified by flash chromatography on silica gel (PE/DCM) to afford **TrCzDAd** (144.2 mg, 93% yield) as a light yellow solid. $^1$H NMR (400 MHz, $CDCl_3$): δ 8.65 (d, *J* = 5.6 Hz, 2H), 8.01 – 8.00 (m, 2H), 7.79 (s, 4H), 7.42 (d, *J* = 6.0 Hz, 2H), 7.31 (d, 1.6 Hz, 2H), 7.09 (s, 8H), 6.77 (dd, *J* = 8.4, 2.0 Hz, 2H), 6.64 (dd, *J* = 8.4 Hz, 1.6 Hz, 2H), 1.39 (m, 36H), 1.26 (m, 18H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ 150.0, 143.1, 142.7, 138.7, 138.2, 137.5, 133.8, 132.4, 130.8, 125.7, 124.4, 124.0, 123.2, 122.9, 122.5, 115.9, 115.1, 109.8, 109.5, 91.8, 89.2, 34.7, 34.4, 32.0, 31.9. FT-IR (KBr): $v_{\text{max}}$ 2961, 2903, 2863, 2218, 1630, 1547, 1487, 1474, 1351, 1322, 1295, 1261, 875, 799, 609 cm$^{-1}$. HR MS (APCI) m/z: Calcd for $C_{73}H_{79}N_4$ $[M + H]^+$: 1011.6300, Found: 1011.6345; Calcd for $C_{69}H_{71}N_4$ $[M + H – C_4H_8]^+$: 955.5674, Found: 955.5648.

## 3 HR MS spectra

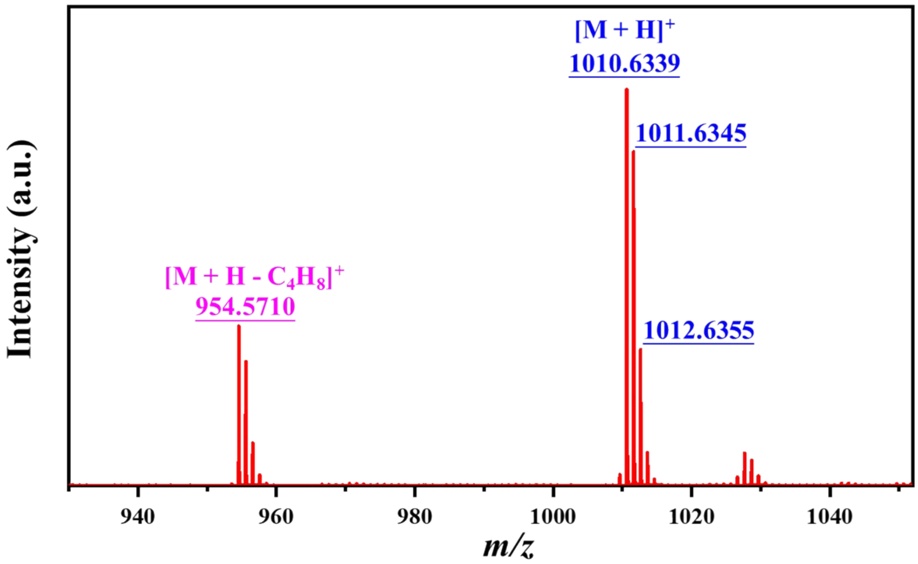


**Fig. S1**. HR APCI mass spectrum and isotopic distribution pattern of **TrCzDAa**.

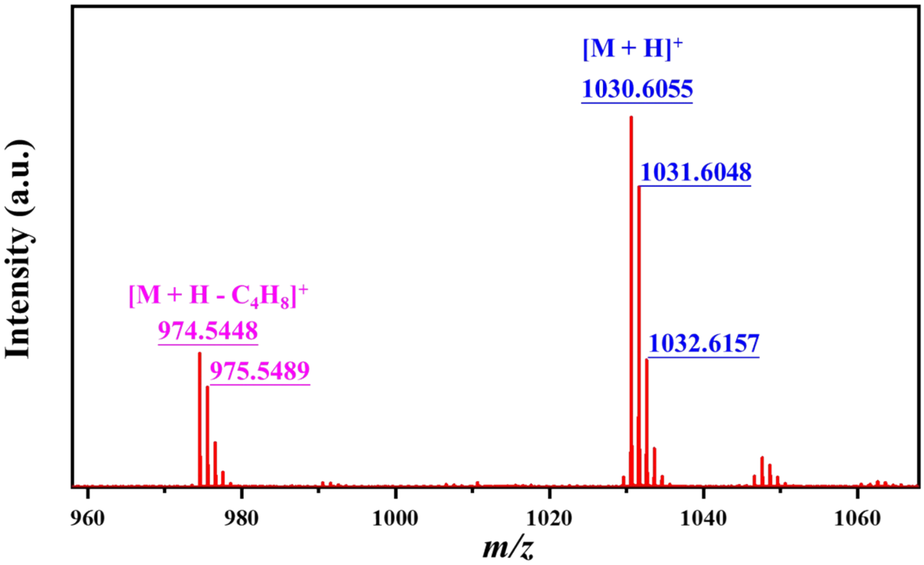


**Fig. S2**. HR APCI mass spectrum and isotopic distribution pattern of **TrCzDAb**.

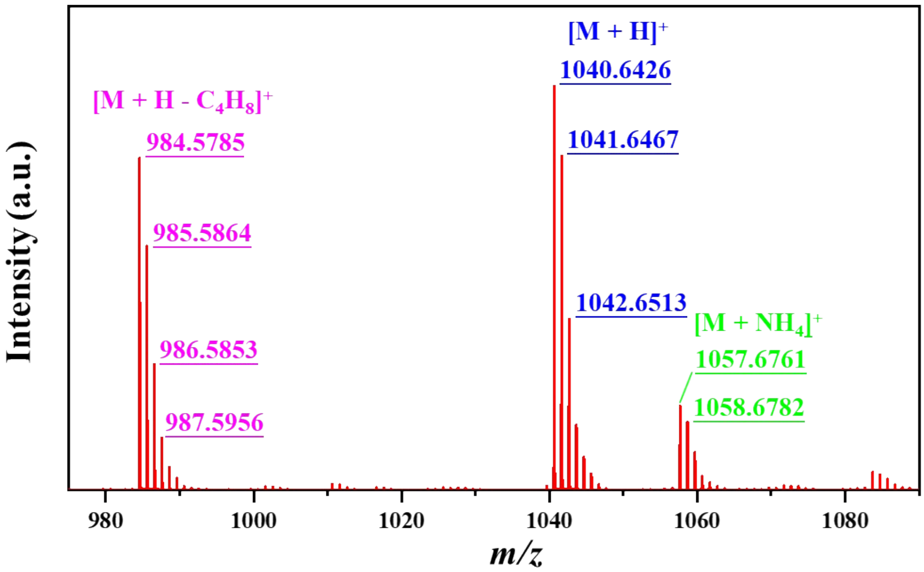


**Fig. S3**. HR APCI mass spectrum and isotopic distribution pattern of **TrCzDAc**.

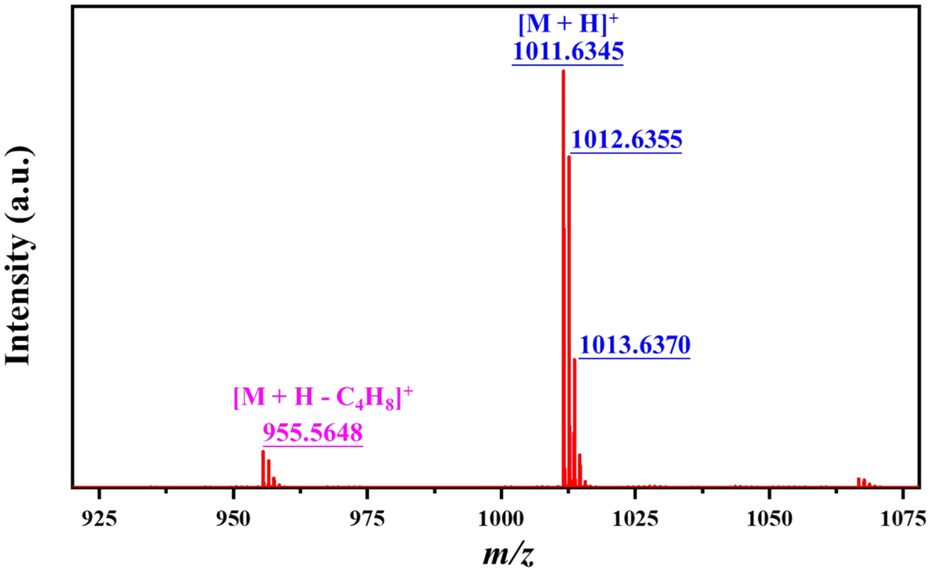


**Fig. S4** HR APCI mass spectrum and isotopic distribution pattern of **TrCzDAd**.

# 4 NMR Spectra

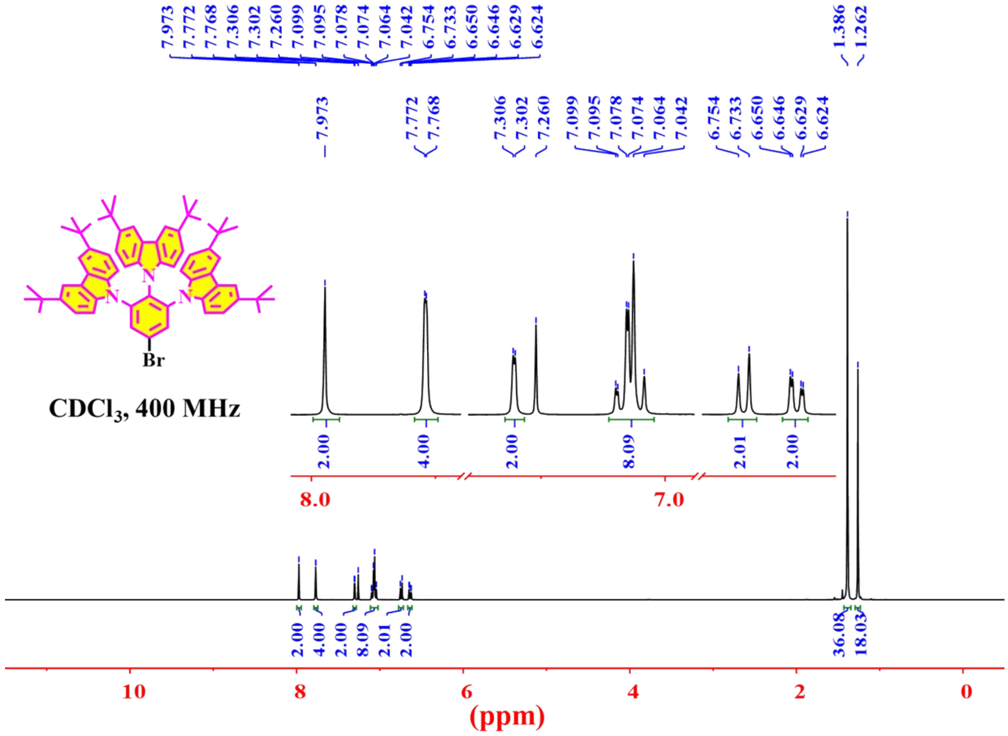


**Fig. S5**. $^1$H NMR spectrum of **5**.

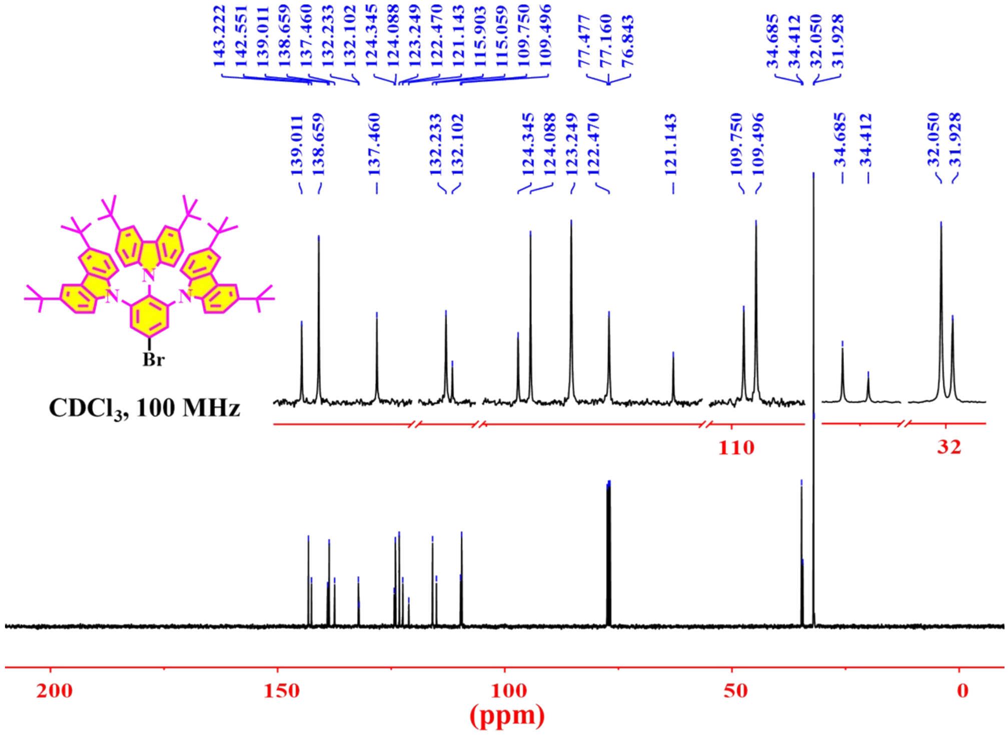


**Fig. S6**. $^{13}$C NMR spectrum of **5**.

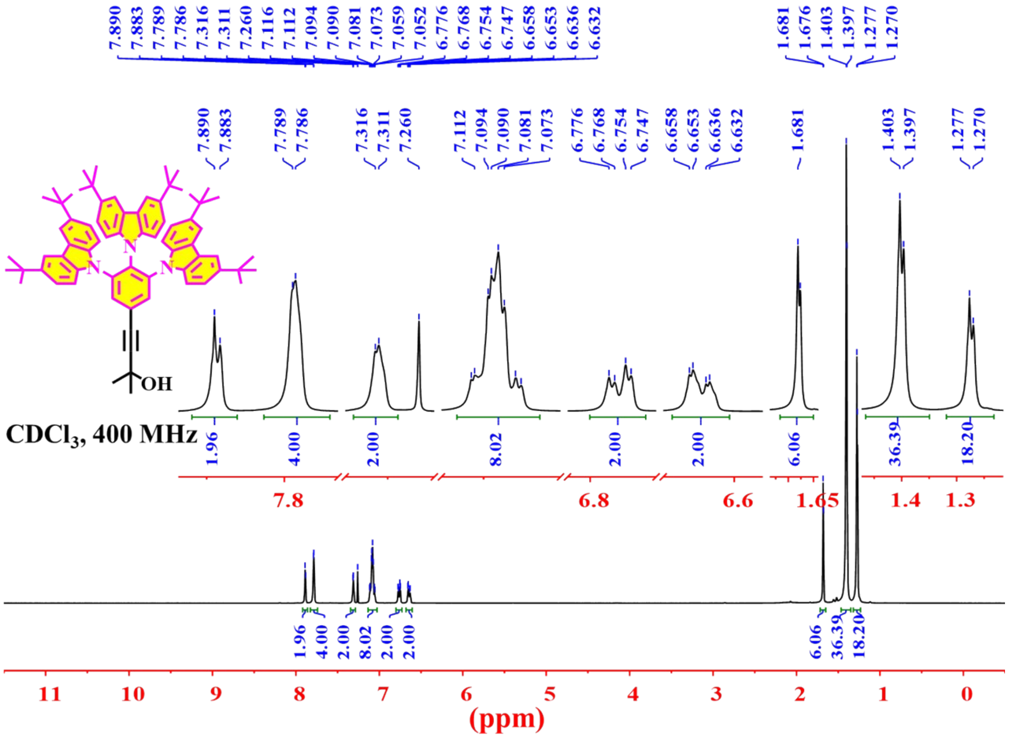


**Fig. S7**. $^1$H NMR spectrum of **6**.

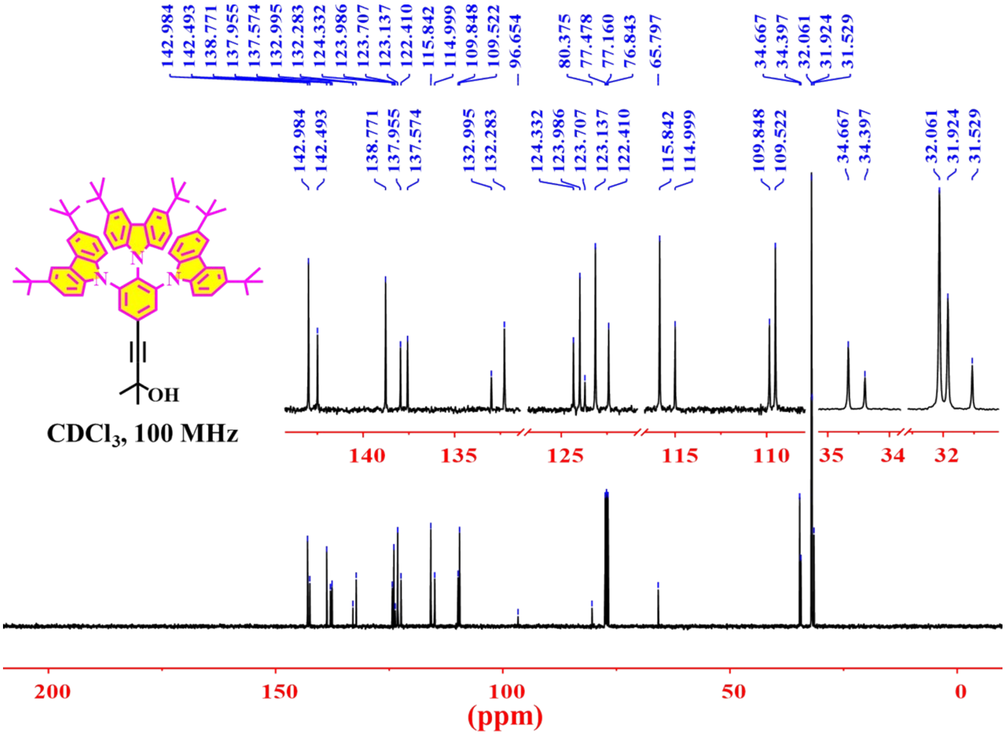


**Fig. S8**. $^{13}$C NMR spectrum of **6**.

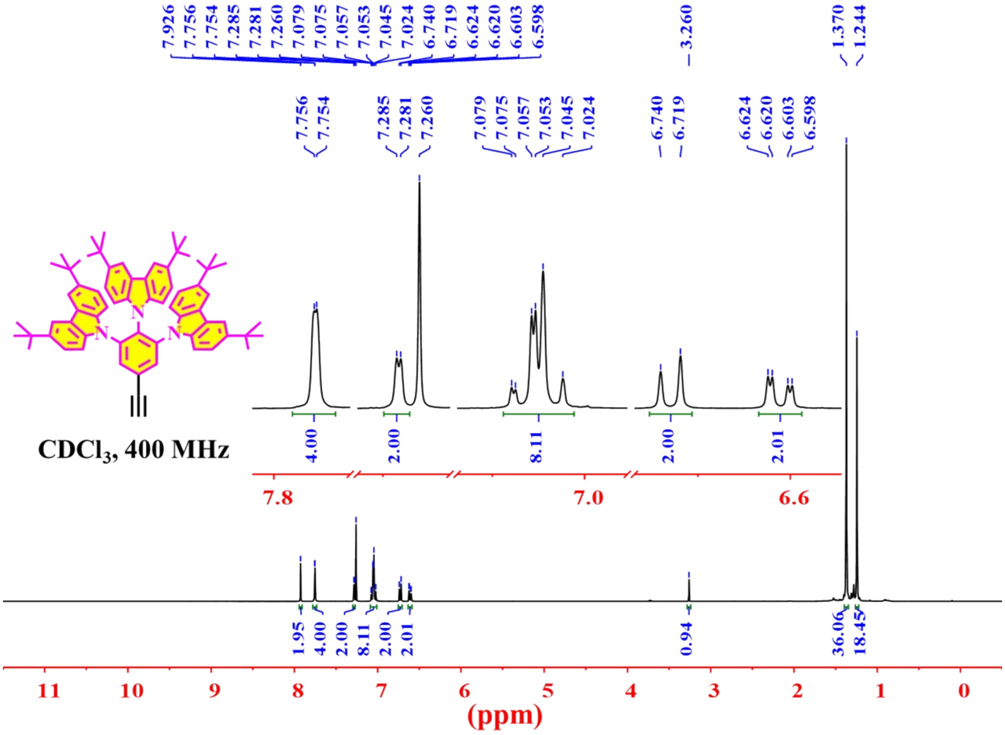


**Fig. S9**. $^1$H NMR spectrum of **7**.

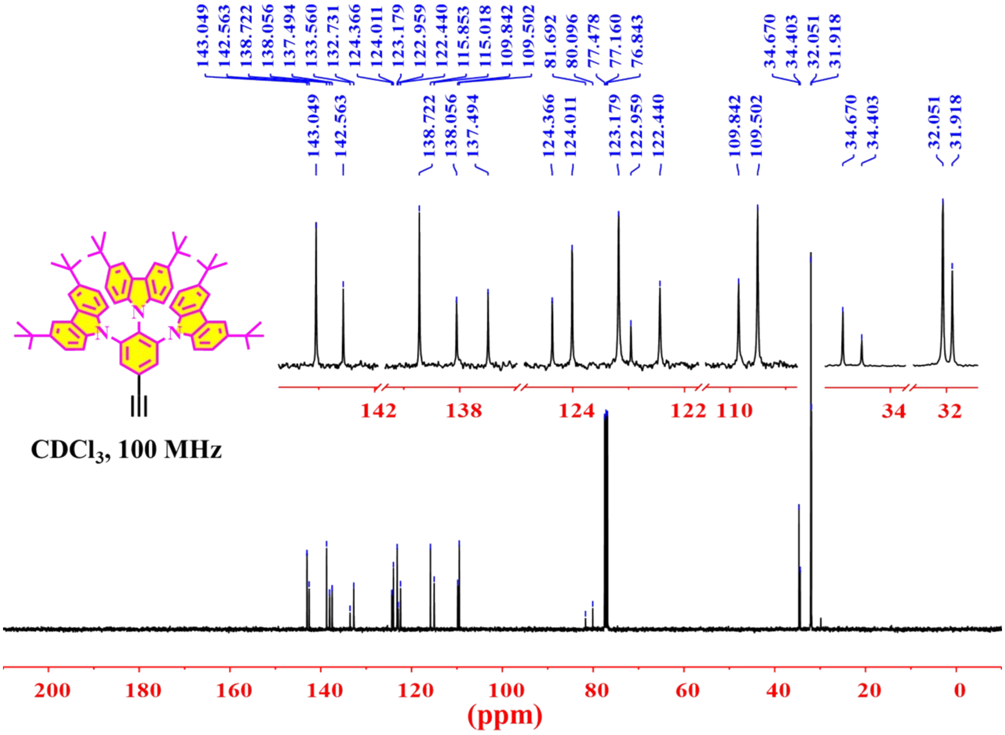


**Fig. S10**. $^{13}$C NMR spectrum of **7**.

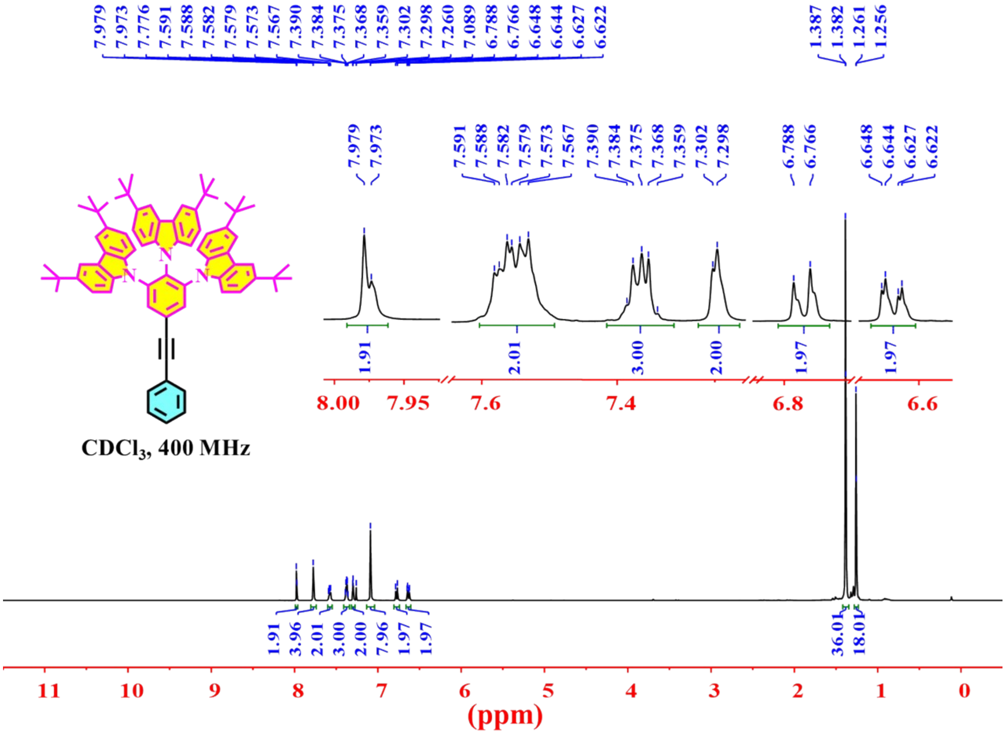


**Fig. S11**. $^{1}$H NMR spectrum of **TrCzDAa**.

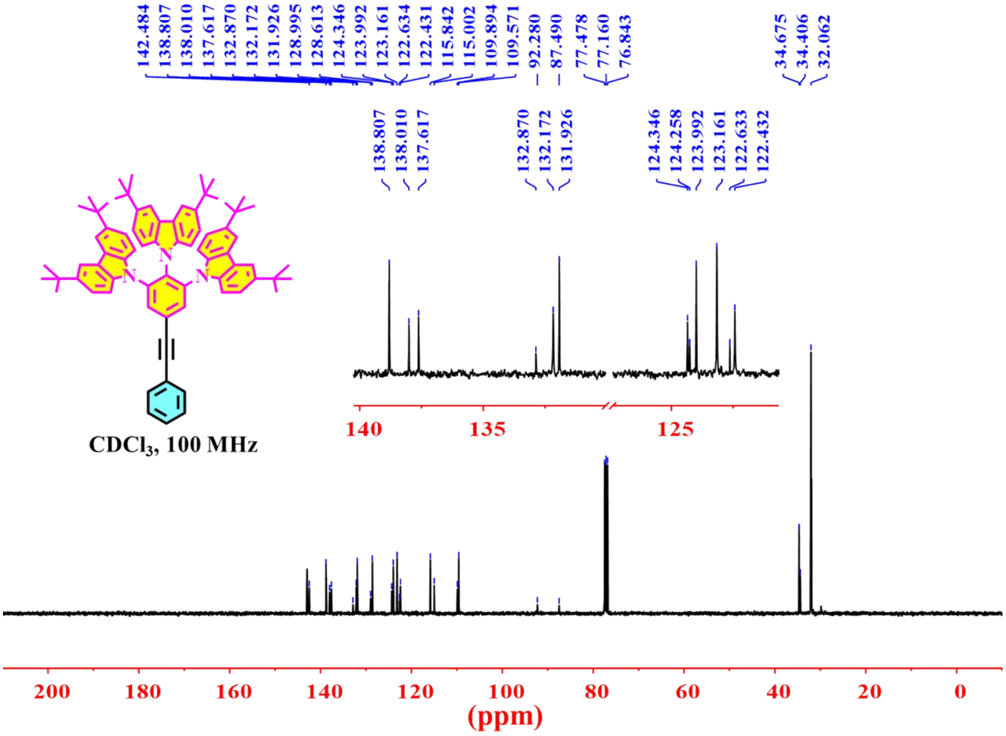


**Fig. S12**. $^{13}$C NMR spectrum of **TrCzDAa**.

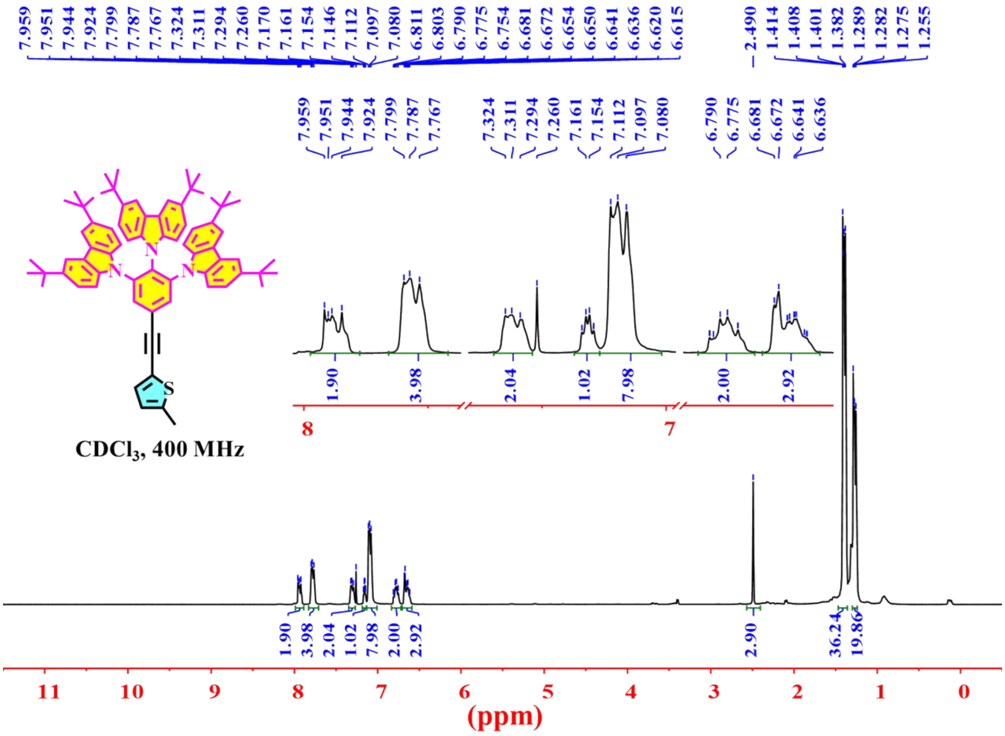


**Fig. S13**. $^1$H NMR spectrum of **TrCzDAb**.

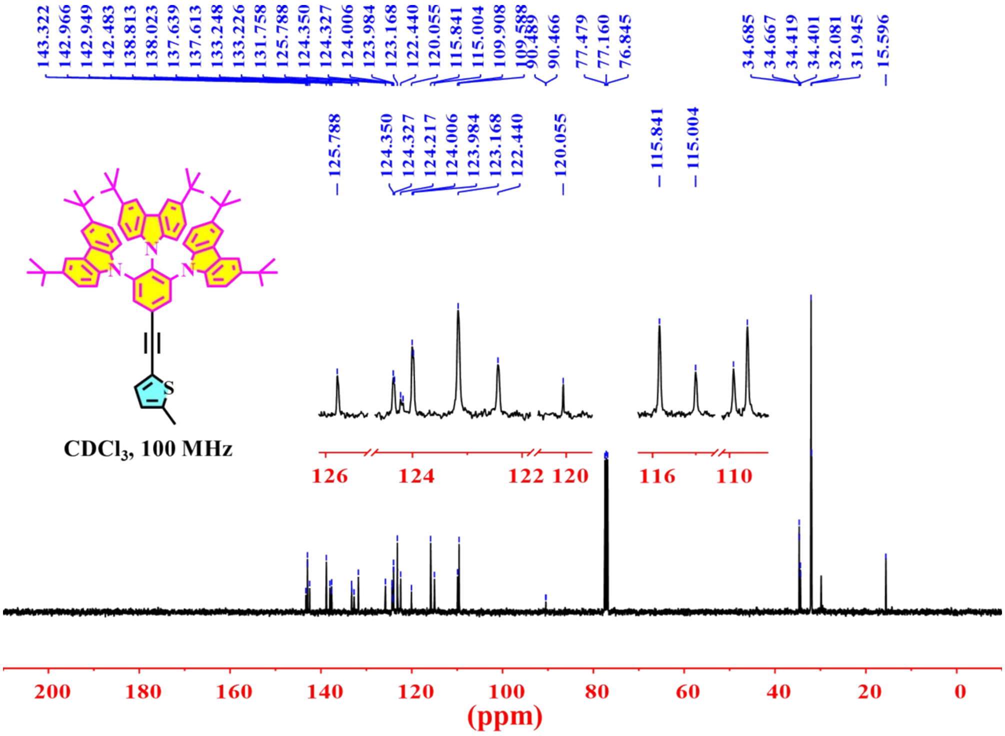


**Fig. S14**. $^{13}$C NMR spectrum of **TrCzDAb**.

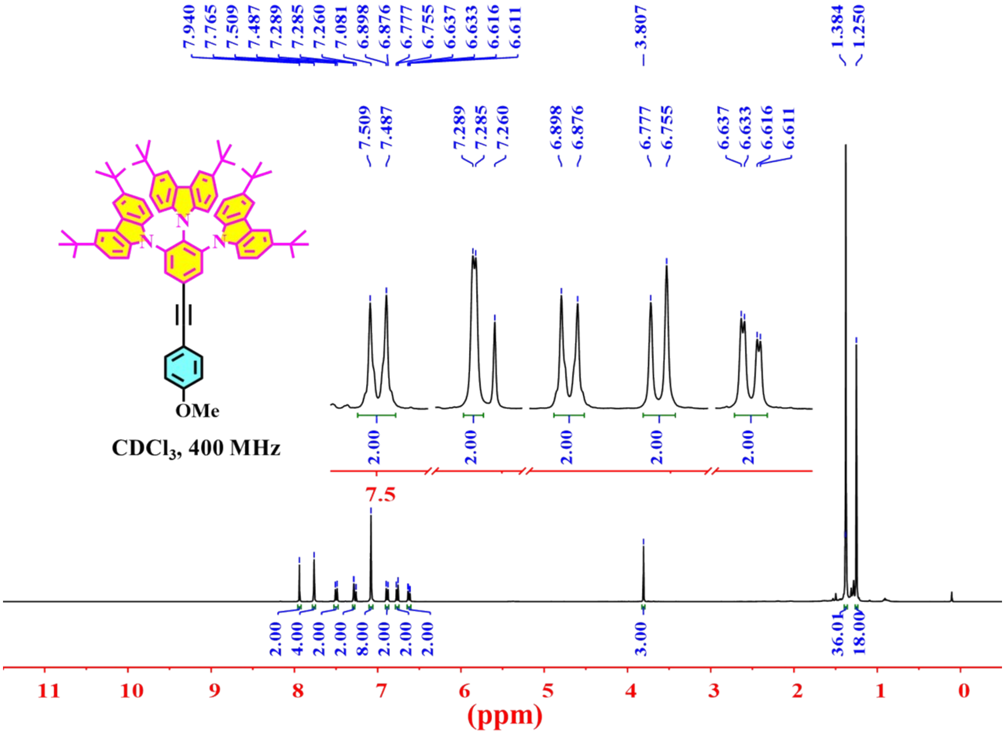


**Fig. S15**. $^1$H NMR spectrum of **TrCzDAc**.

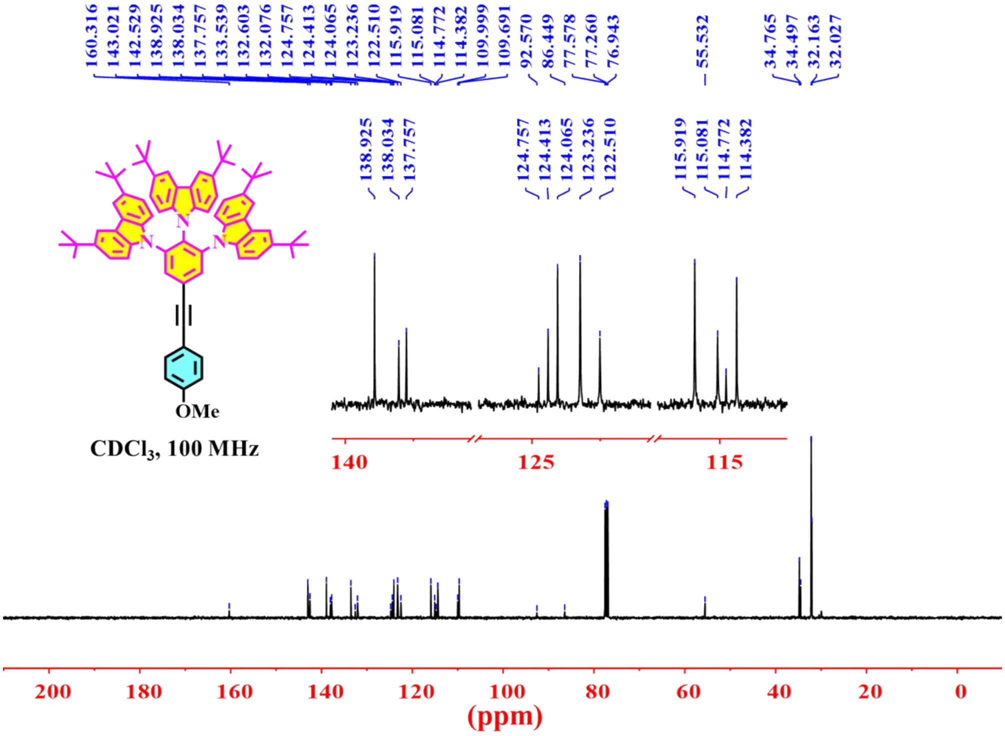


**Fig. S16**. $^{13}$C NMR spectrum of **TrCzDAc**.

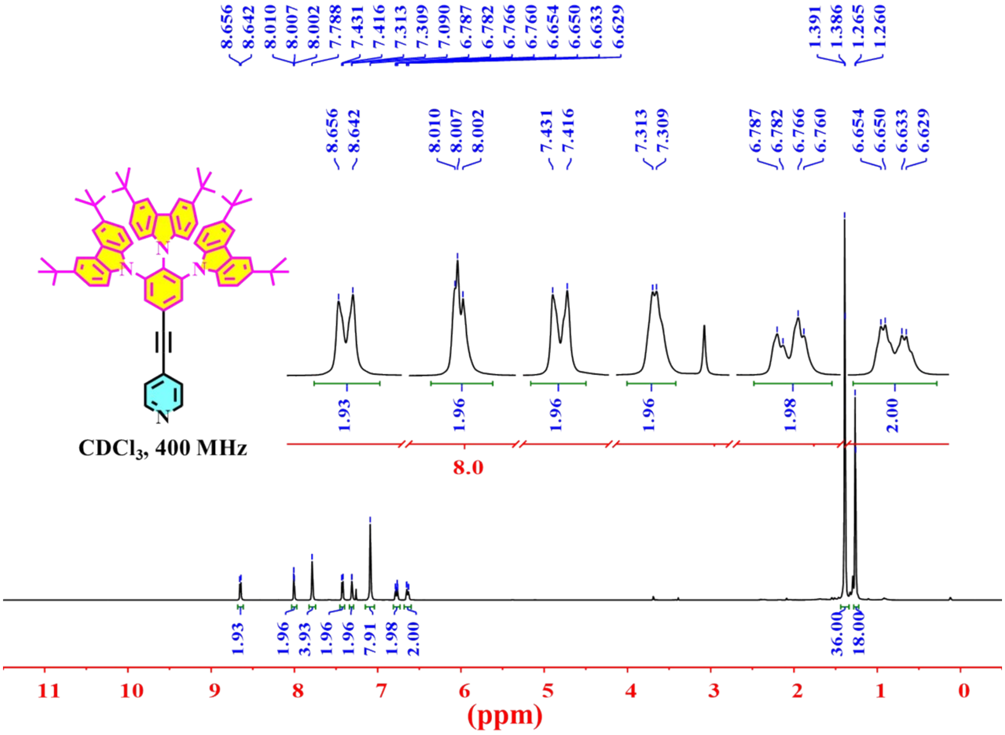


**Fig. S17**. $^{1}$H NMR spectrum of **TrCzDAd**.

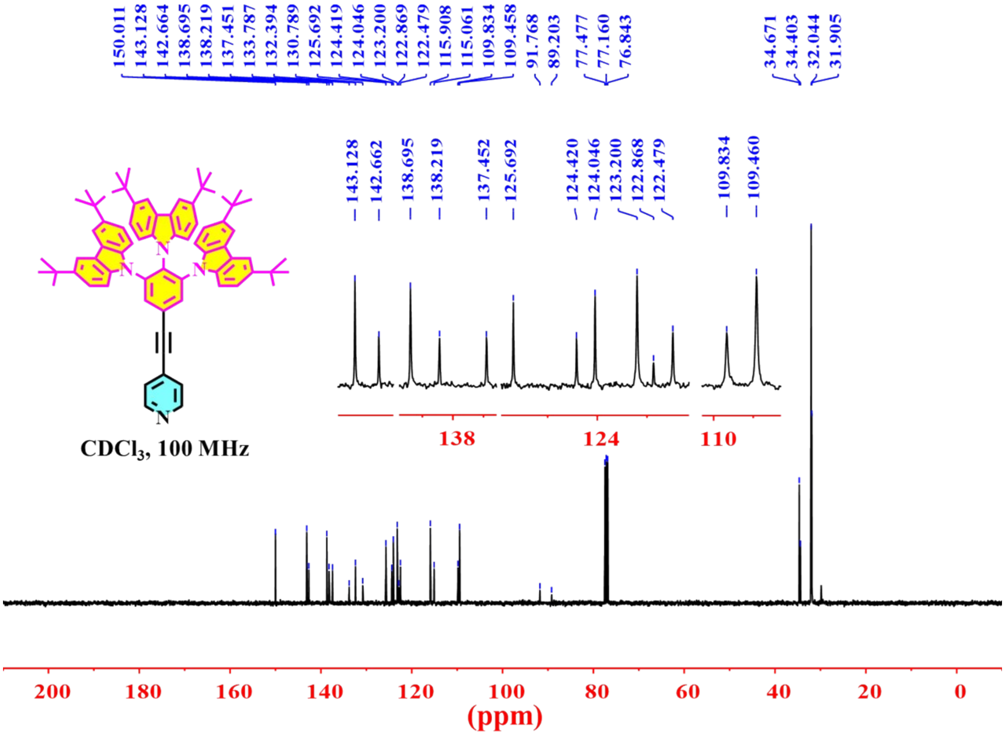


**Fig. S18**. $^{13}$C NMR spectrum of **TrCzDAd**.

## 5 Additional optical physical properties data

**Table 1**. Solvent-dependent data of **TrCzDAs**[a]

| Parameter | | **1,4-Dioxane** | **EA** | **THF** | **ACN** | **DMF** |
|---|---|---|---|---|---|---|
| | | $\lambda_{em}$ (nm) | | | | |
| **compd** | **TrCzDAa** | 430 | 435 | 441 | 463 | 465 |
| | **TrCzDAb** | 432 | 438 | 439 | 462 | 463 |
| | **TrCzDAc** | 419 | 426 | 431 | 449 | 450 |
| | **TrCzDAd** | 455 | 469 | 474 | 502 | 504 |

[a] All Data were obtained at 298 K. $c = 5.0 \times 10^{-7}$ M, $\lambda_{ex} = 295$ nm.

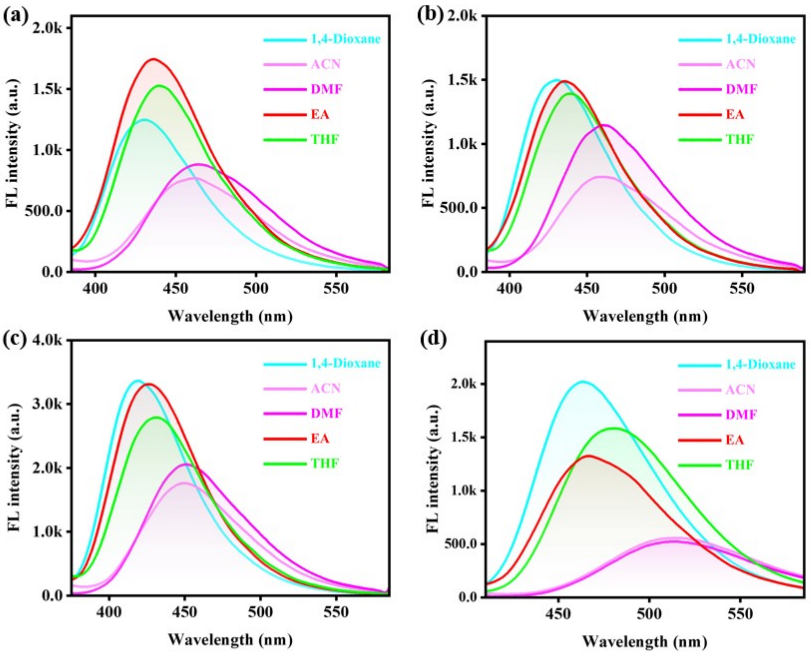


**Fig. S19**. Solvent-dependent fluorescence spectra of (a) **TrCzDAa**, (b) **TrCzDAb**, (c) **TrCzDAc**, and **TrCzDAd** in different solvents. All measurements were performed at 298 K with a concentration of $5.0 \times 10^{-7}$ M ($\lambda_{ex} = 295$ nm).

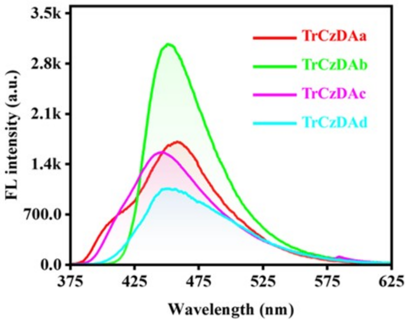


**Fig. S20** Fluorescence spectra of **TrCzDAs** in solid state.

As can be seen from Fig. S20 and Fig. 3e–h, even in the same physical state, the ECL emission wavelength remains redshifted when compared with the fluorescence emission, which may be attributed to the formation of different excimers under the excitation process of electrochemistry.

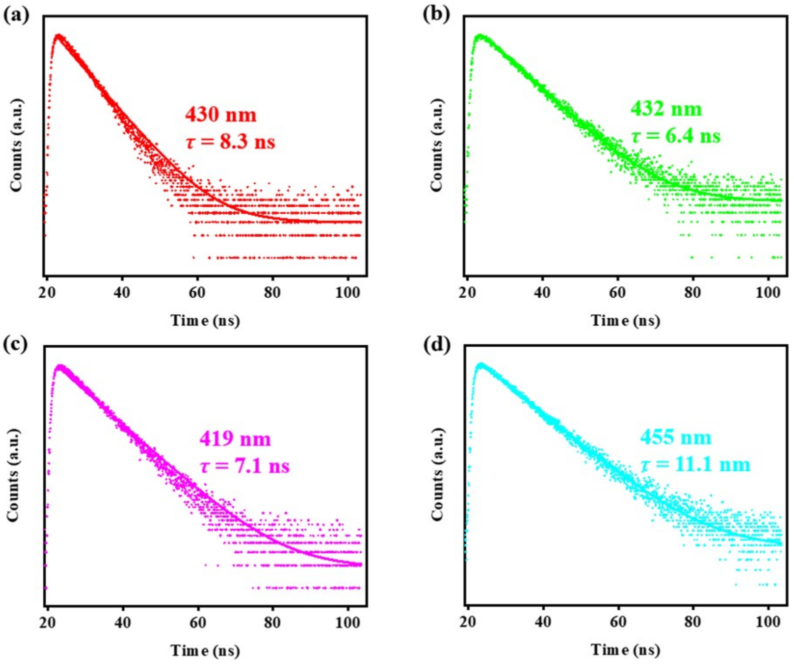


**Fig. S21.** Luminescent decay time ($\tau$) and profile of (a) **TrCzDAa**, (b) **TrCzDAb**, (c) **TrCzDAc**, and **TrCzDAd** with the solid lines as the fitting curves. All measurements were performed in 1,4-Dioxane with a concentration of $5.0 \times 10^{-7}$ M at 298 K, using a nanosecond pulsed laser system.

## 6 ECL measurement procedures

### 6.1 Preparation of the TrCzDAa~d modified electrodes

The samples (**TrCzDAa~d**) were separately dissolved in THF to afford clear solutions ($1.0 \times 10^{-3}$ mol $L^{-1}$). Subsequently, 10 μL of each solution was drop-cast in five 2 μL aliquots onto a glassy carbon electrode (GCE, $\Phi = 4$ mm). After being dried in air at room temperature for 2 hours, the resulting modified electrodes were denoted as, **TrCzDAa-**, **TrCzDAb-**, **TrCzDAc-**, and **TrCzDAd**-modified GCEs.

### 6.2 Measurement of the ECL signals of TrCzDAs

The ECL signals of **TrCzDAa**, **TrCzDAb**, **TrCzDAc**, and **TrCzDAd** were separately gained through testing the aforementioned modified GCE in 0.1M phosphate buffer solution (PBS, pH 7.4) containing 0.018 M $S_2O_8^{2-}$ on an MPI-EII ECL analyzer (Xi’an Remax Electronic Science & Technology Co. Ltd., Xi’an, China). A classic three-electrode system was adopted, the modified GCE was used as the working electrode, the platinum wire was used as the counter electrode, and the Ag/AgCl was used as the reference electrode. The photomultiplier tube voltage was biased at 800 V, the scanning potential was 0 V to −2 V, and the scan rate was 0.3 V/s.

### 6.3 Measurement of the Cathodic 3D ECL Spectra for TrCzDAs

The ECL emission spectra of **TrCzDAa**, **TrCzDAb**, **TrCzDAc**, and **TrCzDAd** were recorded using an RPSE-A spectroscopy detector (Xi'an Remax Electronic Science & Technology Co. Ltd., China). A conventional three-electrode system was employed, with the modified GCE as the working electrode, Ag/AgCl as the reference electrode, and a platinum wire as the counter electrode. The specific procedure was as follows: the aforementioned modified GCEs were immersed in 0.1 M phosphate buffer solution (PBS, pH 7.4) containing 0.018 M $S_2O_8^{2-}$ to acquire their ECL spectra. The instrumental parameters were set as follows: scanning potential was 0 V to −2 V, sampling interval of 500 ms, sensitivity of $1 \times 10^{-3}$, and potential scan rate of 0.05 V/s.

### 6.4 Possible ECL emission mechanism of TrCzDAs

Based on the reports in the literatures[S1] and our previous related works,[S2] a possible ECL mechanism for **TrCzDAs** was proposed, which can explain the observed highest ECL intensity of **TrCzDAb** (equations S1~4). The overall ECL process involves the following key steps: First, under cathodic scanning conditions, the coreactant ($S_2O_8^{2-}$) was reduced to the strongly oxidizing sulfate radical ($SO_4^{\bullet-}$), while **TrCzDAs** were reduced to the corresponding radical anions **TrCzDAs**$^{\bullet-}$ at the electrode surface. Then, **TrCzDAs**$^{\bullet-}$ was oxidized by $SO_4^{\bullet-}$ to generate the excited-state species **TrCzDAs***, and finally, **TrCzDAs*** radiatively relaxed back to the ground state accompanied by an ECL emission.

$$S_2O_8^{2-} + e^- \rightarrow SO_4^{2-} + SO_4^{\bullet-} \qquad \text{(S1)}$$

$$\textbf{TrCzDAs} + e^- \rightarrow \textbf{TrCzDAs}^{\bullet-} \qquad \text{(S2)}$$

$$\textbf{TrCzDAs}^{\bullet-} + SO_4^{\bullet-} \rightarrow \textbf{TrCzDAs*} + SO_4^{2-} \qquad \text{(S3)}$$

$$\textbf{TrCzDAs*} \rightarrow \textbf{TrCzDAs} + h\nu \qquad \text{(S4)}$$

### 6.5 ECL detection of the sensor for EP

The **TrCzDAb**-modified GCE was immersed in 2 mL of PBS containing 0.018 M $S_2O_8^{2-}$ and varying concentrations of EP. The detection of EP was carried out in the potential range of 0 to −2 V with the photomultiplier tube voltage set at 800 V.

To calculate the limit of detection, an ECL measurement with three parallel tests of blank samples was performed, exhibiting average ECL intensity of 15589.9 a.u. with standard deviation (SB) of 233.9. To calculate the LOD, the changes in the ECL intensities ($\Delta I_{\mathrm{ECL}}$) were found to be linearly depended on the logarithm of DA concentration, so the $I_B = 0$, the smallest detectable signal with signal-to-noise ratio value (k1) of 3 could be calculated as:

$$I_L = I_B + \mathrm{k1S_B} = 701.7$$

According to the linear regression equation $\Delta I = 1916.0 \lg c + 19334.2$, the LOD could be calculated as 189 pM.

## 7. References

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