

Supporting Information for

"High-performance transmissive-to-black electrochromism derived from diphenylamine-based polyimides with tetraphenylethylene as bridged units"

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Supplementary Methods

Materials

4-Bromobenzophenone ($\geq 98\%$), 4-benzoyl-4'-bromobiphenyl (HPLC, $\geq 98\%$), p-anisidine (99%), 4-hydroxy benzophenone (98%), 4-fluoronitrobenzene (98%), TiCl_4 (AR, 99%), 80% hydrazine monohydrate, 10% palladium on charcoal (Pd/C), tri-tert-butyl phosphine (TPP, 1.0 M) were all purchased from Aladdin. Zinc (AR, 99%) and potassium carbonate (AR, 99%) were purchased from Tokyo Chemical Industry (TCL). 1,2,4,5-Cyclohexanetetracarboxylic dianhydride (HPMDA, T, $\geq 98\%$) was purchased from Aladdin and dried before use. N-methyl-2-pyrrolidinone (NMP, AR, $>99\%$), acetic anhydride (AR, 99%), and pyridine (Py, AR, $>99\%$) were purchased from Aladdin and treated using 4 Å molecular sieves before use. All other chemicals were directly used as received from commercial sources.

Measurements

Nuclear magnetic resonance (NMR) spectra were recorded on a BRUKER-300 using deuterated DMSO as the solvent. Mass spectra were recorded on a MALDI-TOF-ESI. Film thicknesses were recorded on a profilometer (DEKTAK 150). Fourier transforms infrared (FTIR) spectra were recorded on a Bruker Vector 22 spectrometer. The polymers' glass-transition temperature (T_g) was measured using differential scanning calorimetry (DSC) on a METTLER 821e. Thermogravimetric analysis (TGA) was carried out in the temperature range of 100-800 °C on a Pyris TGA under a nitrogen flow. Electrochemistry data were acquired with an electrochemical analyzer (CHI 660e)

using a three-electrode cell. The UV-vis spectra were recorded with a Shimadzu UV 3101-PC spectrophotometer. The polymers' number and weight averaged molar mass was measured using a high-temperature gel permeation chromatographer system with DMAC under 80 °C (PL-GPC-220).

Methods

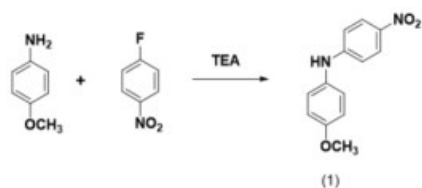
The CV and electrochromic experiments were performed in the liquid electrolyte which is anhydrous acetonitrile (CH_3CN) solution containing 0.1 M tetrabutylammonium perchlorate (TBAP). The 0.01 M Ag/AgCl electrode and a platinum wire were used as reference and auxiliary electrodes, respectively. All the thin films described in this study were prepared in the following procedure: 50 mg (or 20 mg) polyimides were dissolved in 1 mL DMAc; the solution was stirred at 85 °C for 1 h. 100 μL of the above solution was spin-casted (details see **Table S4**) on an ITO-coated substrate. After annealing at 85 °C for 1 h and 130 ° C for another 1 h in sequence, the film electrodes were saved under an argon atmosphere. All the procedures are carried out under a dry argon atmosphere.

Synthesis of monomers and polyimides

Three monomers (TPE-2DPANH₂, 2DPA-TPE-2NH₂, and TPE-4DPANH₂) and corresponding polyimides (ST-TPE-PI, PG-TPE-PI, and HB-TPE-PI) were synthesized by the following steps. Monomer TPE-2DPANH₂, TPE-2DPANH₄, and polyimide ST-TPE-PI and HB-TPE-PI were synthesized following our previous work, the others are

new.^{1,2}

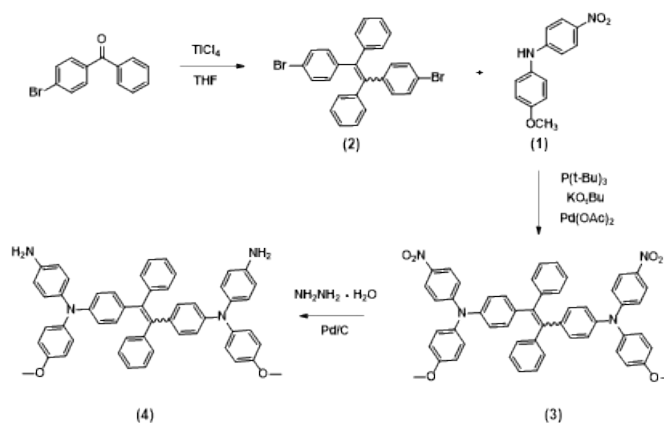
1.1. Synthesis of 4-methoxy-4'-nitrodiphenylamine (Compound 1)



Scheme 1. Synthetic route to compound 1.

Compound 1 was obtained following a reported method in previous work.² 3-Fluoronitrobenzene (28.2 g, 200 mmol), p-anisidine (36.9 g, 300 mmol), triethyl-amine (54 mL), and DMF (360 mL) were added in a 500 ml two-neck flask. The reaction mixture was stirred under 85°C. After 72 h and cooling to room temperature, the solution was filtered and poured into water. The resulting mixture was filtered and dried. The crude product was recrystallized from anhydrous methanol to obtain orange-yellow fine needle-like crystals. ¹H NMR (300 MHz, DMSO-d₆, δ, ppm): 9.1(s, 1H), 8.05 (d, J= 6 Hz, 2H), 7.18 (d, J= 7.5 Hz, 2H), 6.98 (d, J= 9.0 Hz, 2H), 6.88 (d, J= 9.0 Hz, 2H), 3.76 (s, 3H).

Synthesis of TPE-2DPANH₂:



Scheme 2. Synthetic route to compound 4 (TPE-2DPANH₂)

2.1. Synthesis of 1,2-bis (4-bromophenyl)-1,2-diphenylmethane (Compound 2)

Compound 2 was synthesized through the McMurry reaction.³ An ice-cooled (-5°C) suspension of zinc powder (10.0 g, 152.91 mmol) in anhydrous THF (450 ml) was prepared in a three-neck flask under argon atmosphere, and TiCl₄ (14.4 g, 75.92 mmol) was added slowly to the suspension with a syringe. After refluxing the resulting mixture for 3h, the mixture was cooled to room temperature and added a solution of 4-bromobenzophenone (20.00 g, 76.60 mmol) was in THF (50 ml). After refluxing for 12 h, the mixture was cooled to room temperature and quenched with 10% K₂CO₃ aqueous solution until no bubbles appeared. The resulting mixture was filtered and extracted with chloroform three times. Then the combined organic layer was dried over sodium sulfate. After the solvent was evaporated, the resulting white crude product was purified by chromatography (mixture of Z/E isomers). Compound 2 was obtained as a white powder (11.1 g, yield= 55.5%). ¹H NMR (300 MHz, CDCl₃ δ, ppm): 7.25 (m, 4H), 7.13 (m, 4H), 6.98 (m, 4H), 6.87 (m, 4H).

2.2. Synthesis of 4-methoxy-N-{4-[2-{4-[(4-methoxyphenyl)(4-nitrophenyl)amino]phenyl}-1,2-diphenylethenyl]phenyl}-N-(4-nitrophenyl)aniline (Compound 3) (TPE-2DPANO₂)

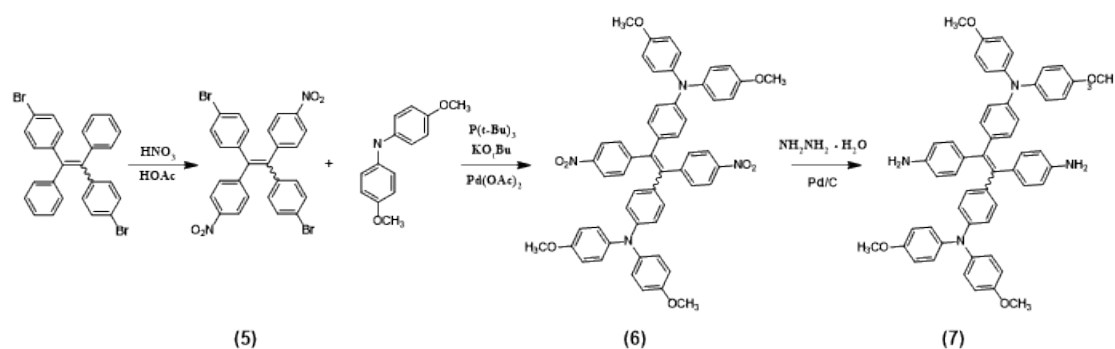
Compound 1 (6.22 g, 25.5 mmol), compound 2 (5.0 g, 10.2 mmol), KO_tBu (2.855 g, 25.5 mmol), Pd (OAc)₂ (0.255 g, 1.13 mmol), 0.1M TPP (7 g, 3.42 mmol) and toluene (250 ml) were added in a 500 ml two-neck flask. The reaction mixture was refluxed for 24 h under a nitrogen atmosphere. After cooling to room temperature, the

solution was filtered and poured into petroleum ether. The resulting mixture was filtered and dried. Then the crude product was purified by chromatography. Compound 3 was obtained as an orange powder (5.16 g, yield= 62%). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 8.05 (d, J= 9.0Hz, 2H), 7.92 (d, J= 9.0Hz, 2H), 7.09 (Br, 26H), 6.65 (dd, J= 8.6Hz, 4H), 3.75 (m, 6H, -OCH₃). MS (ESI) m/z calculated. FTIR (KBr), 1590 and 1335 cm⁻¹ (-NO₂ group). For (C₅₂H₄₀N₄O₆): 816.2948, found: 816.4382.

2.3. Synthesis of N¹-{4-[2-{4-[(4-aminophenyl)(4-methoxyphenyl)amino]phenyl}-1,2-diphenylethenyl]phenyl}-N¹-(4-methoxyphenyl)benzene-1,4-diamine (Compound 4) (TPE-2DPANH₂)

Compound 3 (5 g, 6.1 mmol), Pd/C (0.34 g), and 1,4-dioxane (70 ml) were added in a 250 ml two-neck flask. After heating the mixture to reflux, add 80% hydrazine monohydrate slowly. The reaction mixture was refluxed for 10 h under a nitrogen atmosphere. Then filtrated and poured the filtrate into water. Collected the crude product and purified it by chromatography. Compound 4 was obtained as an orange-brown powder (4.0 g, yield= 80%). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 7.08 (m, 6H), 6.96 (ddd, 8H), 6.83 (m, 8H), 6.70 (d, J= 9.0Hz, 4H), 6.52 (m, 8H), 5.12 (s, 4H, -NH₂), 3.72 (m, 6H, -OCH₃). FTIR (KBr), 3455 and 3370 cm⁻¹ (-NH₂ group). MS (ESI) m/z calculated. For (C₅₂H₄₄N₄O₂): 756.3464, found: 756.3477.

Synthesis of 2DPA-TPE-2NH₂:



Scheme 3. Synthetic route to compound 7 (2DPA-TPE-2NH₂).

3.1. Synthesis of 1,2-bis(4-bromophenyl)-1,2-bis(4-nitrophenyl)ethane (Compound 5)

Compound 5 was obtained following a reported method in previous work.⁴ Compound 2 (3 g, 6.12 mmol) was added to a solution of acetic acid (HOAc) (12 mL) in dichloromethane (DCM, 180 mL) under vigorous stirring. The solution was cooled to 0 °C. Concentrated nitric acid (15 mL) was slowly added to the solution. The reaction mixture was allowed to warm to room temperature; stirring was continued overnight. The reaction mixture was quenched with water before it was extracted with DCM. The combined organic layers were washed with brine and dried over anhydrous MgSO₄. After filtration, the solvent was removed under a vacuum to afford the crude product. The residue was purified by column chromatography (PE: EA = 3:1). Compound 5 was obtained as an orange-brown powder (2.58 g, yield= 78%). ¹H NMR (300 MHz, CDCl₃, δ ppm), 8.02 (m, 4 H), 7.32 (m, 4 H), 7.18 (m, 4 H), 6.85 (m, 4 H).

3.2. Synthesis of N-{4-[2-{4-[bis(4-methoxyphenyl)amino]phenyl}-1,2-bis(4-

nitrophenyl)ethenyl]phenyl}-4-methoxy-N-(4-methoxyphenyl)aniline (Compound 6)
(2DPA-TPE-2NO₂)

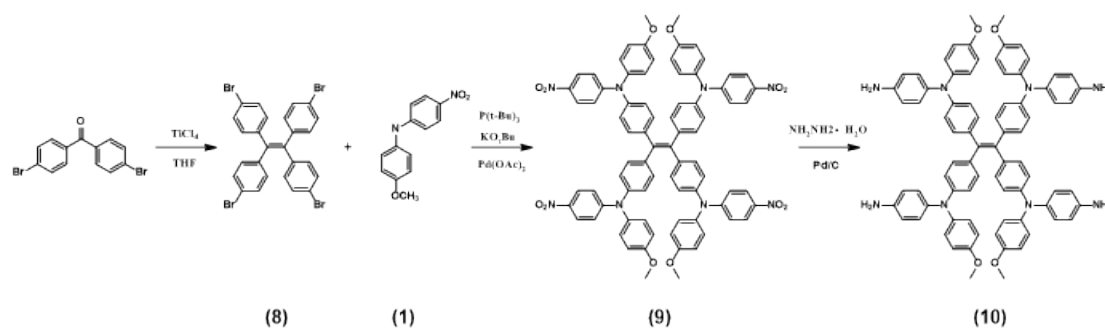
Compound 5 (1.154 g, 2 mmol), bis(4-methoxyphenyl)amine (0.57 g, 2.5 mmol), KO_tBu (0.224 g, 2 mmol), Pd(OAc)₂ (0.017 g, 0.044 mmol), 0.1 M TPP (0.45 g, 0.27 mmol) and toluene (10 ml) were added in a 25 ml two-neck flask. The reaction mixture was refluxed for 48 h under a nitrogen atmosphere. After cooling to room temperature, the solution was filtered and poured into petroleum ether. Filtered and collected the crude orange product. After drying under a vacuum, the residue was purified by chromatography. Compound 6 was obtained as a dark orange powder (1.4 g, yield= 80 %). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 8.13-7.99 (d, J= 9.0 Hz, 4H), 7.32-7.25 (d, J= 9.0 Hz, 4H), 7.05-6.67 (m, 16 H), 6.76-6.69 (d, J= 9.0 Hz, 4H), 6.55-6.48 (d, J= 9.0 Hz, 4H), 3.82-3.55 (m, 12H, -OCH₃ group). FTIR (KBr), 1593 and 1341 cm⁻¹ (-NO₂ group). MS (ESI) m/z calculated. For (C₅₅H₄₄N₄O₈): 876.315, found: 876.323.

3.3. Synthesis of N-{4-[1,2-bis(4-aminophenyl)-2-{4-[bis(4-methoxyphenyl)amino]phenyl}ethenyl]phenyl}-4-methoxy-N-(4-methoxyphenyl)aniline (Compound 7)
(2DPA-TPE-2NH₂)

Compound 6 (1.31 g, 1.5 mmol), Pd/C (0.2 g), and 1,4-dioxane (15 ml) were added in a 25 ml two-neck flask. After heating, the mixture to reflux added 3 ml 80% hydrazine monohydrate slowly. The reaction mixture was refluxed for 12 h under a nitrogen atmosphere. After cooling to room temperature, filtrated and poured the filtrate into water. Collected the crude product and purified it by chromatography. Compound

7 was obtained as a brown powder (1.11 g, yield= 93%). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 6.98-6.88 (m, 16H), 6.77-6.69 (d, J= 3, 4H), 6.67-6.60 (d, J= 3, 4H), 6.59-6.53 (d, J= 3, 4H), 6.36-6.26 (d, J= 3, 4H), 4.96 (s, 4H, -NH₂ group), 3.73 (m, 12H, -OCH₃ group). FTIR (KBr), 3472 and 3379 cm⁻¹ (-NH₂ group). MS (ESI) m/z calculated. For (C₅₄H₄₈N₄O₄): 816.368, found: 816.368.

Synthesis of TPE-4DPANH₂:



Scheme 4. Synthetic route to compound 10 (TPE-4DPANH₂).

4.1. Synthesis of 1,1',1'',1'''-(1,2-ethenediylidene)tetrakis[4-bromobenzene] (Compound 8)

Compound 8 was obtained through the McMurry reaction. An ice-cooled (-75°C) suspension of zinc powder (10.0 g, 152.91 mmol) in anhydrous THF (450 ml) was prepared in a two-neck flask under an argon atmosphere. TiCl₄ (14.4 g, 75.92 mmol) was slowly added to the suspension with a syringe and quickly stirred. After refluxing for 3 h, the resulting mixture was cooled to room temperature and added a solution of 4,4'-dibromobenzophenone (20.00 g, 58.82 mmol) in THF (50 ml). After refluxing for 12 h, the mixture was cooled to room temperature, quenched with 10 % K₂CO₃ aqueous

solution, and stirred overnight. The resulting mixture was filtered and extracted with chloroform three times. Then the combined organic layer was dried over sodium sulfate, evaporating the solvent under reduced pressure. The resulting white crude product was purified by chromatography. Compound 8 was obtained as a white powder (16.2 g, yield= 85 %). ¹H NMR (300 MHz, CDCl₃, δ, ppm): 7.26 (d, J= 9.0 Hz, 8H), 6.85 (m, 8H).

4.2. Synthesis of 4-methoxy-N-(4-nitrophenyl)-N-{4-[tris({4-[(4-methoxyphenyl) (4-nitrophenyl)amino]phenyl})ethenyl]phenyl}aniline (Compound 9) (TPE-4DPANO₂)

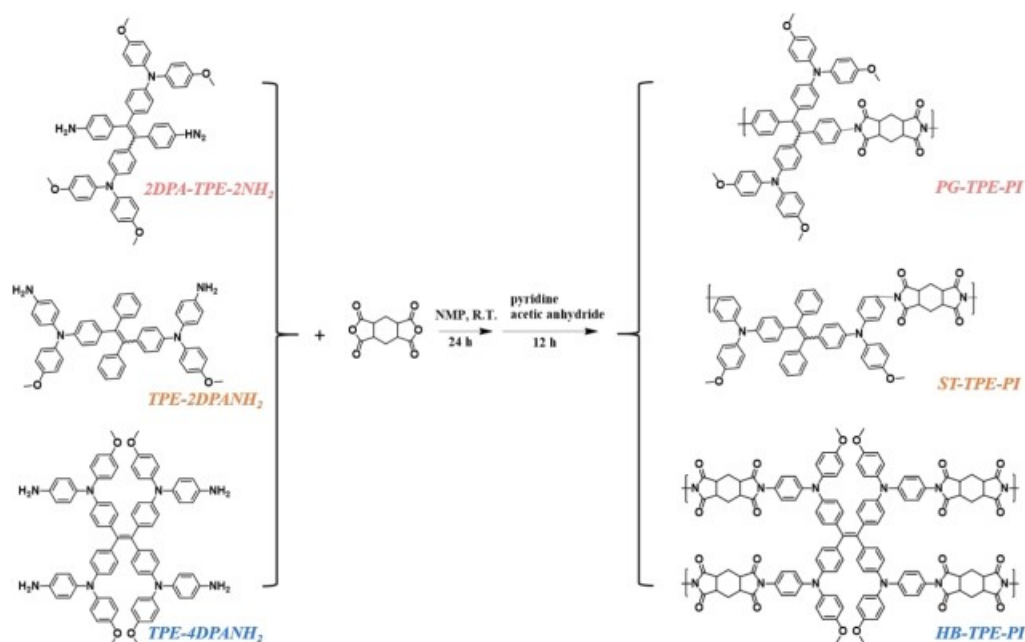
Compound 1 (6.0 g, 24.6 mmol), compound 9 (5.3 g, 8.9 mmol), KO_tBu (2.97 g, 26.53 mmol), Pd (OAc)₂ (0.255 g, 1.13 mmol), 0.1 M TPP (6.75 g, 3.32 mmol) and toluene (250 ml) were added in a 500 ml two-neck flask. The reaction mixture was refluxed for 24 h under a nitrogen atmosphere. After cooling to room temperature, the solution was filtered and poured into petroleum ether. Filtered and collected the crude orange product. After being filtered and dried under vacuum, the residue was purified by column chromatography to yield compound 9 as an orange solid (10.04 g, yield= 56.1%). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 8.17-8.00 (m, 4H), 7.97-7.87 (d, J= 3, 2H), 7.81-7.72 (d, J= 6, 2H), 7.32-6.93 (m, 32H), 6.72-6.61 (m, 8H), 3.84-3.69 (m, 12H, -OCH₃ group). FTIR (KBr), 1588 and 1317 cm⁻¹ (-NO₂ group). MS (ESI) m/z calculated. For (C₇₈H₆₀N₈O₁₂): 1300.433, found: 1300.312.

4.3. Synthesis of N¹-(4-methoxyphenyl)-N¹-{4-[tris({4-[(4-aminophenyl) (4-

methoxyphenyl)amino]phenyl}ethenyl]phenyl}benzene-1,4-diamine (Compound 10)
(TPE-4DPANH₂)

Compound 9 (5 g, 3.84 mmol), Pd/C (0.5 g), and 1,4-dioxane (65 ml) were added in a 250 ml two-neck flask under a nitrogen atmosphere. After heating, the mixture to reflux added 10 ml 80% hydrazine monohydrate slowly. The reaction mixture was refluxed for 24 h. Then the solution was filtered and poured into water. Collected the crude product and purified it by chromatography. Compound 11 was obtained as a light brown powder (4.3 g, yield= 89.3%). ¹H NMR (300 MHz, CDCl₃, δ ppm), 7.04-6.69 (d, J= 9.0 Hz, 8H), 9.63-9.86 (d, J= 9.0 Hz, 8H), 7.04-6.69 (d, J= 9.0 Hz, 8H), 6.80-6.74 (d, J= 9.0 Hz, 8H), 6.69-6.62 (d, J= 6.0 Hz, 8H), 6.62-6.55 (d, J= 9.0 Hz, 8H), 3.75 (s, 12 H, -OCH₃ group), 3.56 (s, 8 H, -NH₂ group). FTIR (KBr), 3454 and 3369 cm⁻¹ (-NH₂ group). MS (ESI) m/z calculated. For (C₇₈H₆₈N₈O₄): 1180.537, found: 1180.536.

Synthesis of polyimides:



Scheme 5. Synthetic routes to polyimides.

All polyimides were polymerized by the same method. The 2DPA-TPE-2NH₂ (1.0 mmol) or TPE-2DPANH₂ (1.0 mmol) or TPE-4DPANH₂ (0.333 mmol), anhydride (HPMDA, 1 mmol), DMAc (10 ml) were added in a 25 ml two neck flask. The reaction mixture was stirred at room temperature 24 h under a nitrogen atmosphere. After warming to room temperature, pyridine (2 ml) and acetic anhydride (4 ml) were added into the solution. Then the reaction mixture was heated for 12 h at 85 °C and cooled. The resultant polyimide solid was filtrated and washed with ethyl alcohol and hot water. We named the polyimides as follows:

ST-TPE-PI: Polymerized by TPE-2DPANH₂ and HPMDA. FTIR (KBr), 1774, 1718 and 1382 cm⁻¹ (imide group). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 7.76-6.6 (Br, 32H), 3.71 (s, 6H, -OCH₃), 1.95 (s, 4H), 1.71 (s, 2H), 1.58 (s, 2H).

PG-TPE-PI: Polymerized by 2DPA-TPE-2NH₂ and HPMDA. FTIR (KBr), 1779, 1725 and 1382 cm⁻¹ (imide group). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 7.75-6.45

(Br, 32H), 3.79-3.51 (m, 12H, -OCH₃ group), 3.17 (s, 1H), 2.83 (s, 1H), 2.14 (s, 4H).
1.17 (s, 2H).

HB-TPE-PI: Polymerized by TPE-4DPANH₂ and HPMDA. FTIR (KBr), 1783, 1712 and 1380 cm⁻¹ (imide group). ¹H NMR (300 MHz, DMSO-d₆, δ ppm), 7.30-6.61 (Br, 48H), 3.72 (s, 12H, -OCH₃ group), 3.16 (s, 6H), 2.82 (s, 6H), 2.17 (s, 14H). 1.17 (s, 6H).

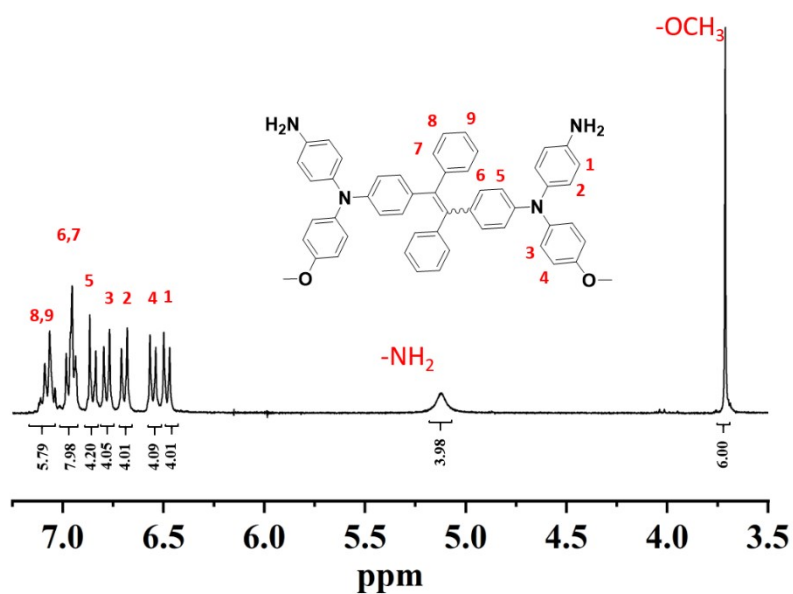


Figure S1. ¹H NMR spectrum of compound 4 (TPE-2DPANH₂).

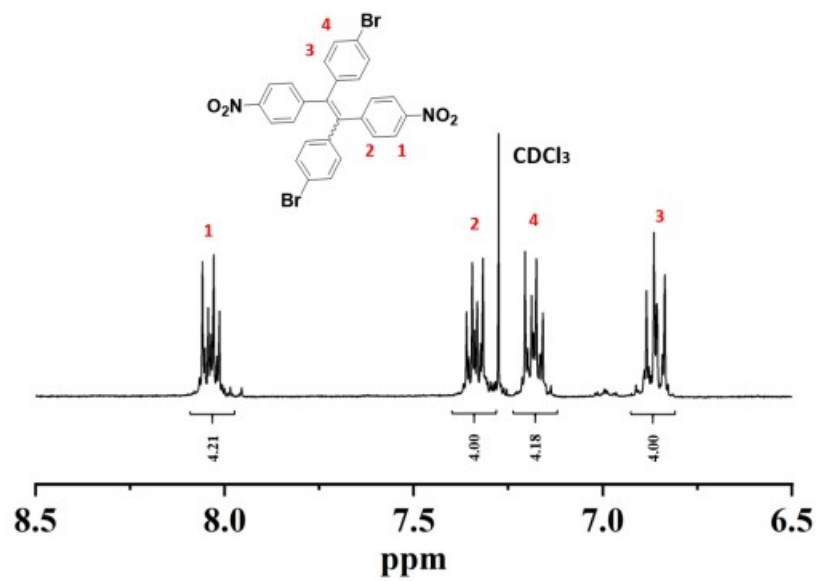


Figure S2. ¹H NMR spectrum of compound 5.

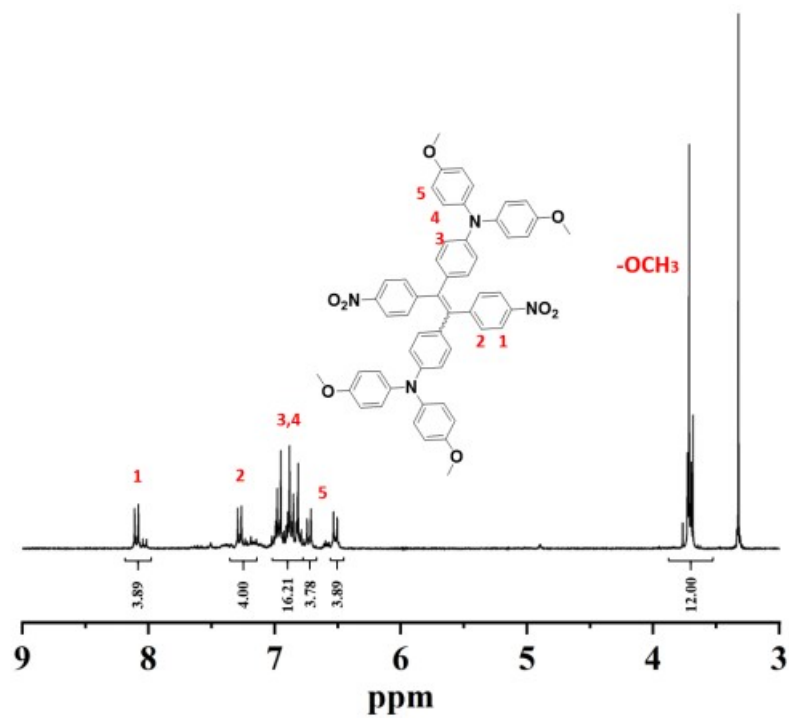


Figure S3. ¹H NMR spectrum of compound 6 (2DPA-TPE-2NO₂).

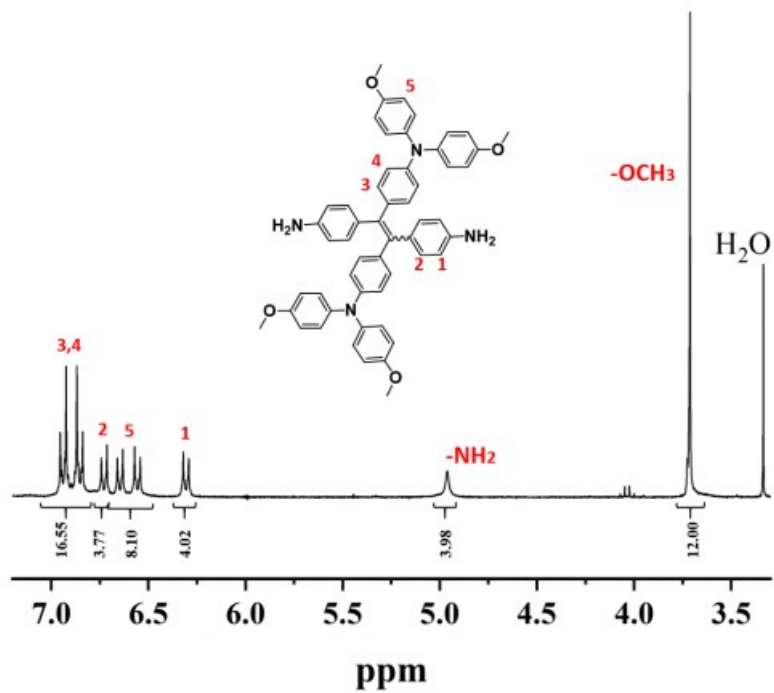


Figure S4. ¹H NMR spectrum of diamine monomer 7 (2DPA-TPE-2NH₂).

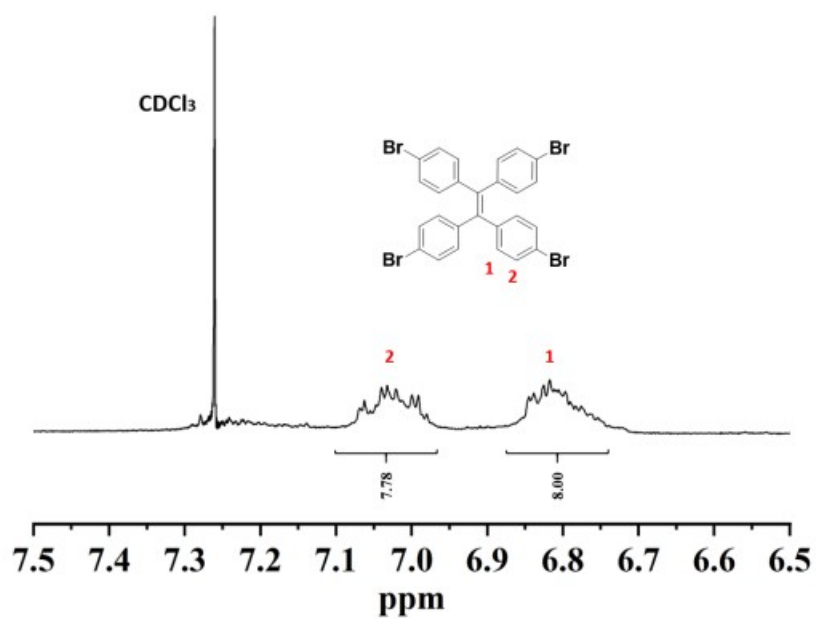


Figure S5. ¹H NMR spectrum of compound 8.

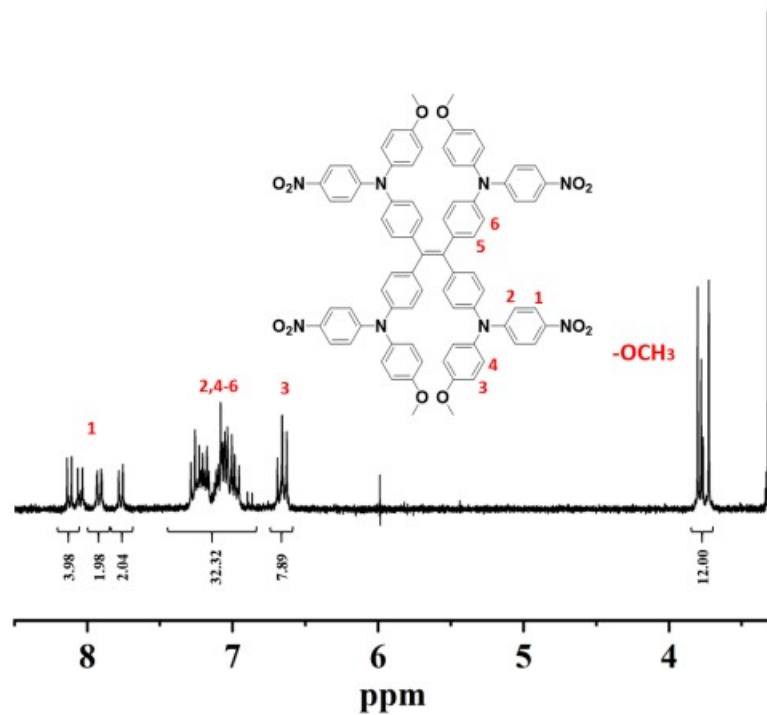


Figure S6. ¹H NMR spectrum of compound 9 (TPE-4DPANO₂)

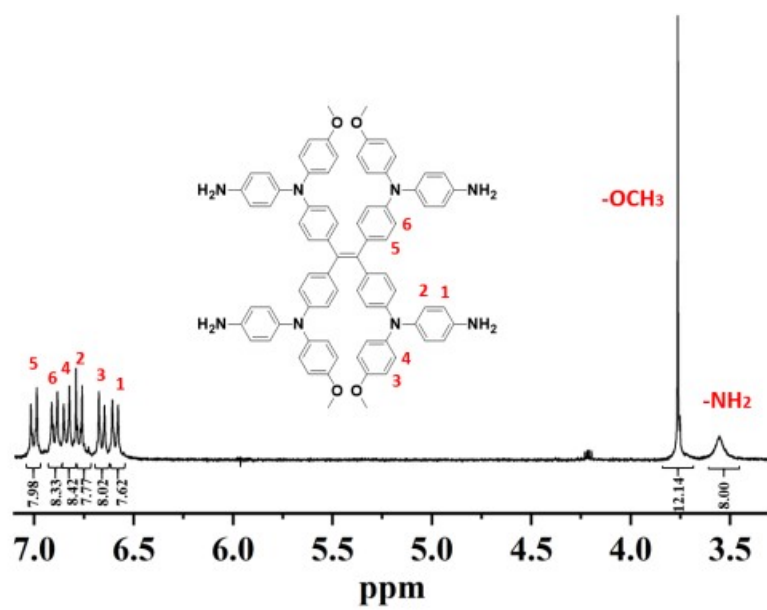


Figure S7. ¹H NMR spectrum of monomer 10 (TPE-4DPANH₂).

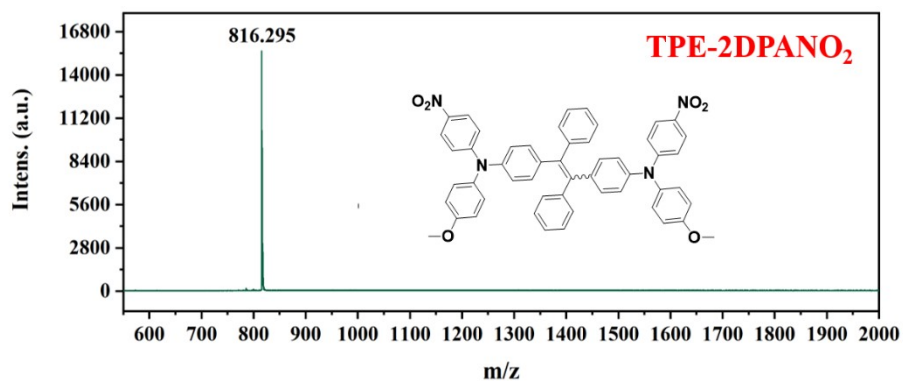


Figure S8. HRMS (ESI) m/z of compound 3 (TPE-2DPANO₂).

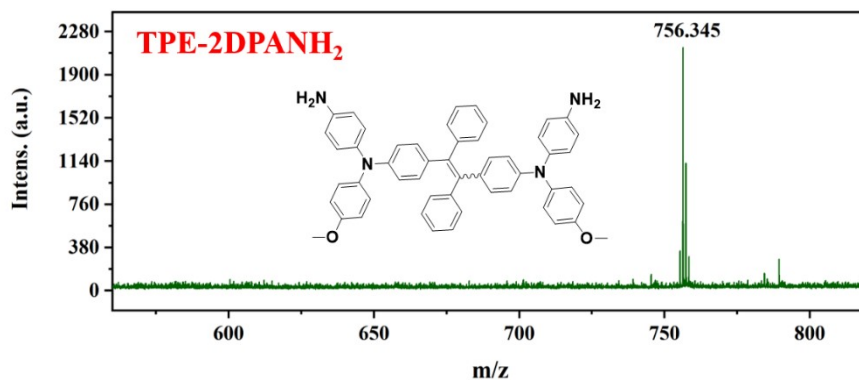


Figure S9. HRMS (ESI) m/z of compound 4 (TPE-2DPANH₂).

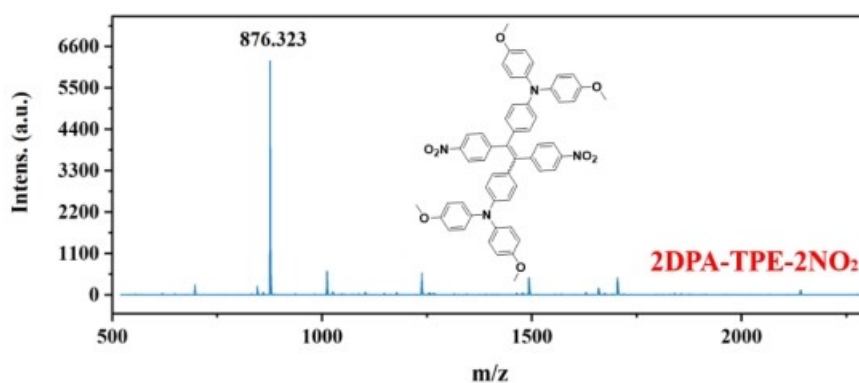


Figure S10. HRMS (ESI) m/z of compound 6 (2DPA-TPE-2NO₂).

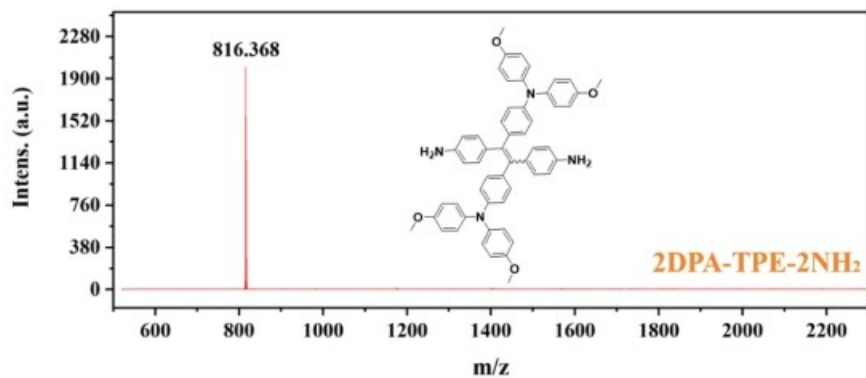


Figure S11. HRMS (ESI) m/z of monomer 7 (2DPA-TPE-2NH₂).

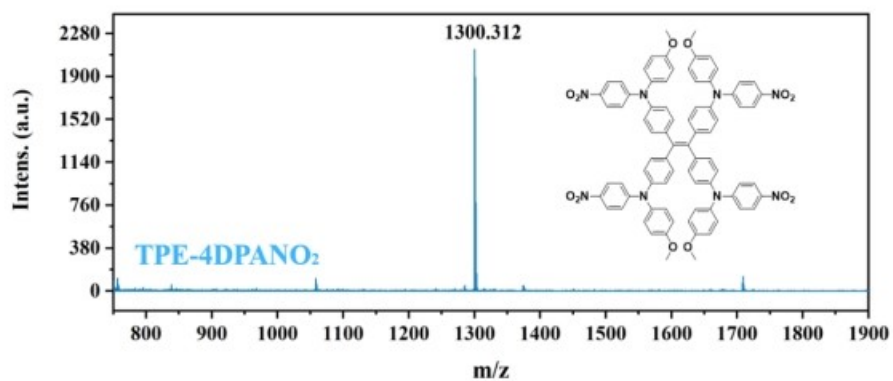


Figure S12. HRMS (ESI) m/z of compound 9 (TPE-4DPANO₂).

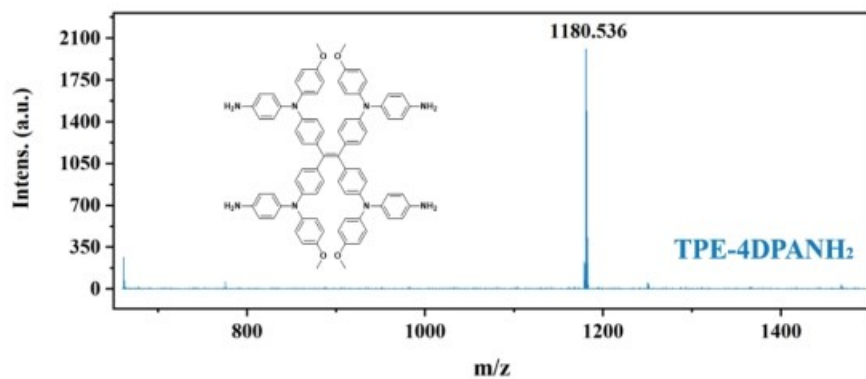


Figure S13. HRMS (ESI) m/z of diamine compound 10 (TPE-4DPANH₂).

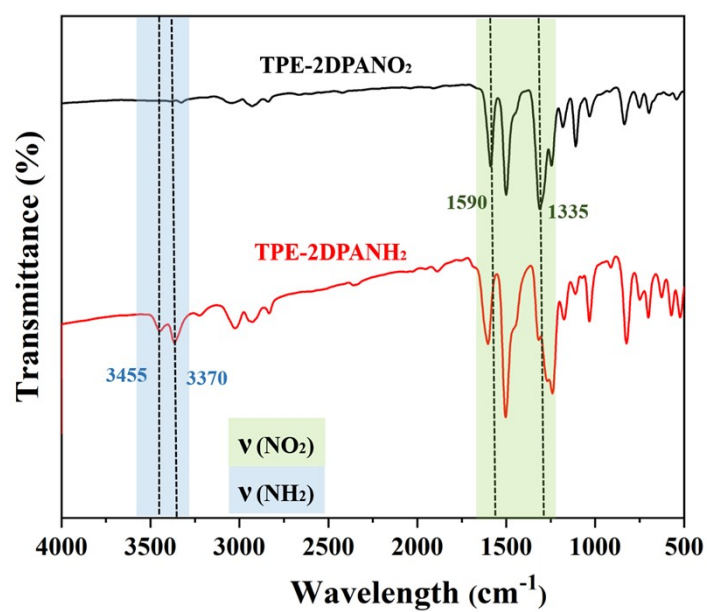


Figure S14. FTIR spectra of compound 6 (TPE-2DPANO₂) and monomer 7 (TPE-2DPANH₂).

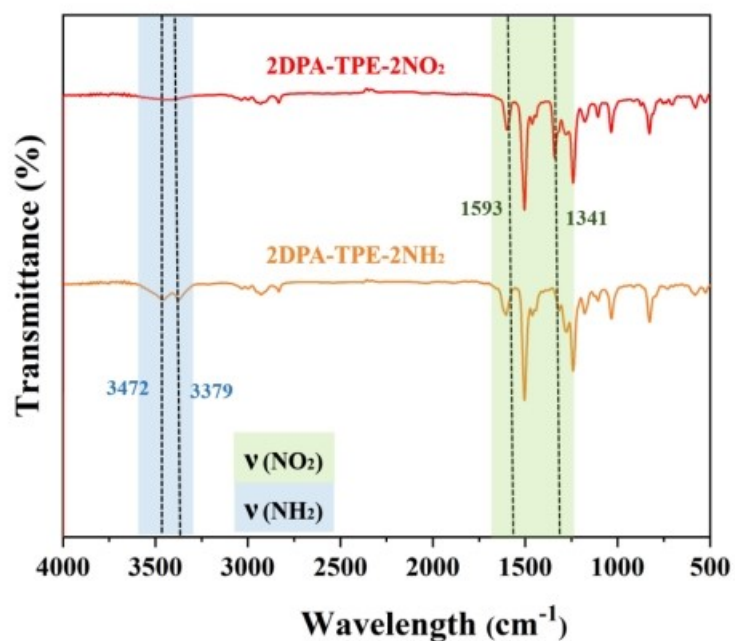


Figure S15. FTIR spectra of compound 6 (2DPA-TPE-2NO₂) and monomer 7 (2DPA-TPE-2NH₂).

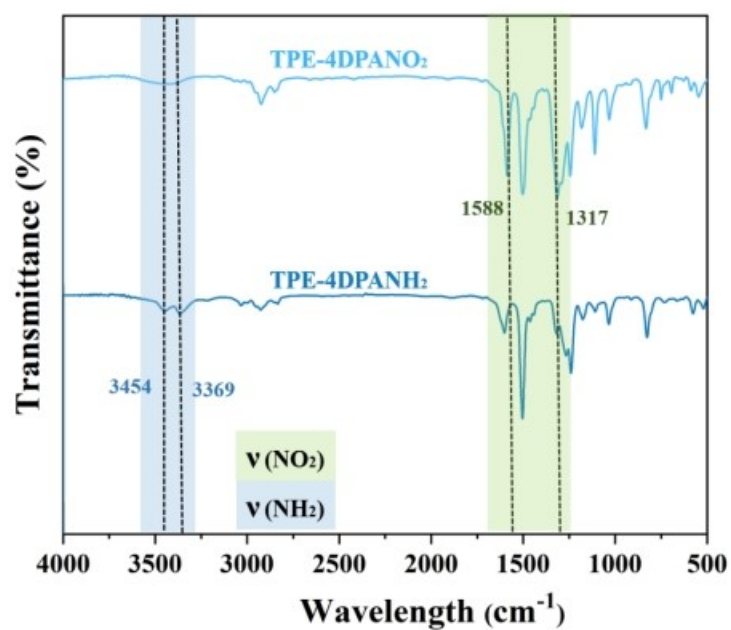


Figure S16. FTIR spectra of compound 9 (TPE-4DPANO₂) and compound 10 (TPE-4DPANH₂).

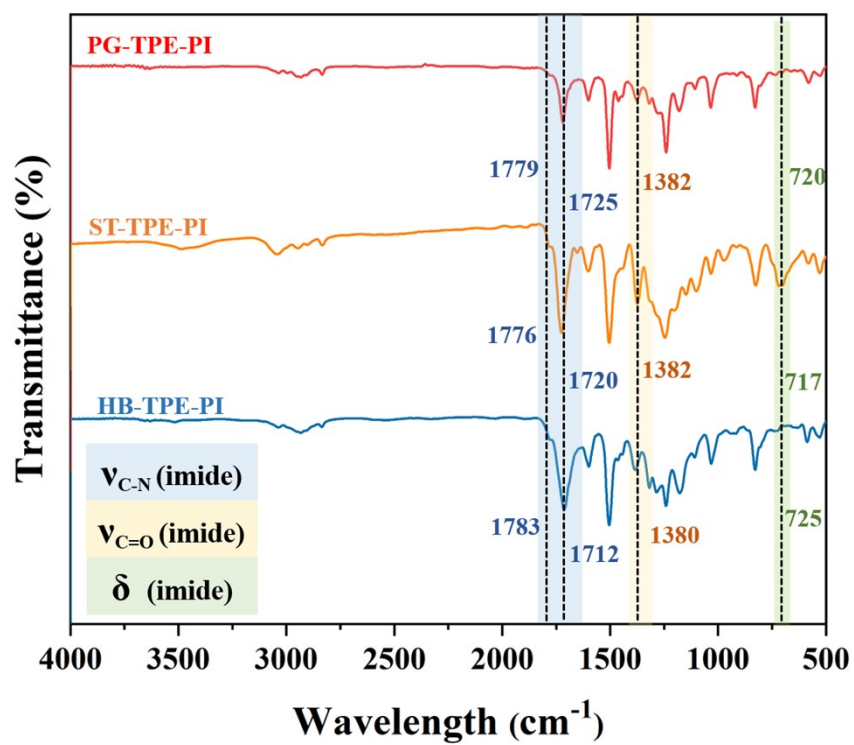


Figure S17. FTIR spectra of polyimides.

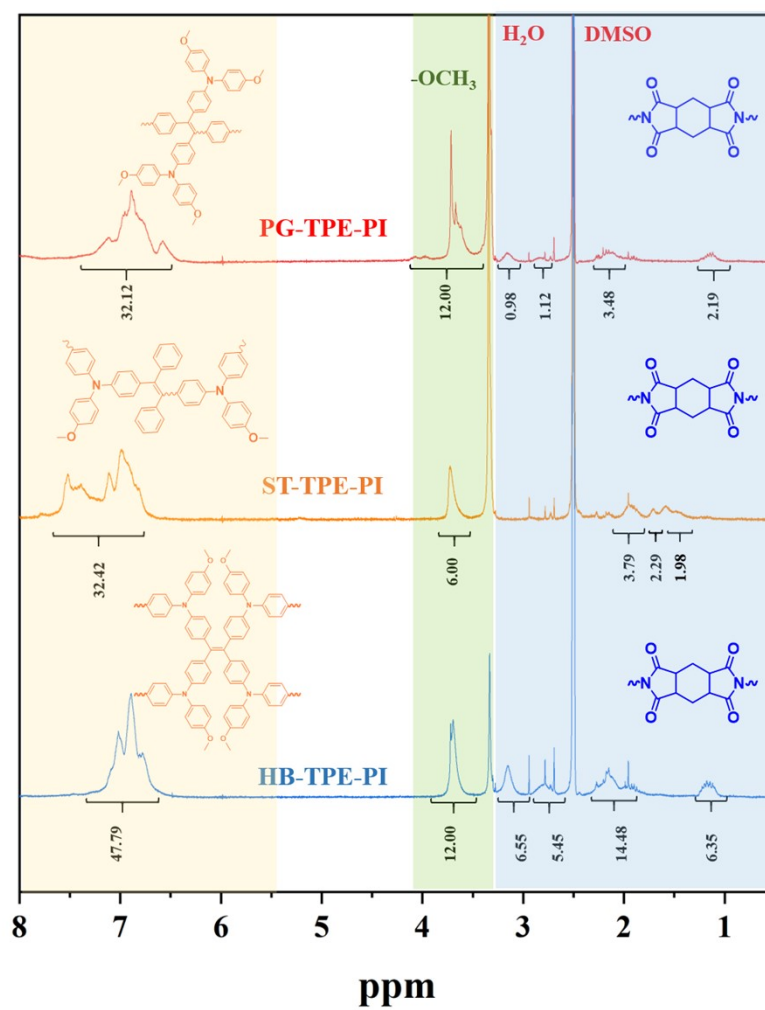


Figure S18. ¹H NMR spectra of polyimides.

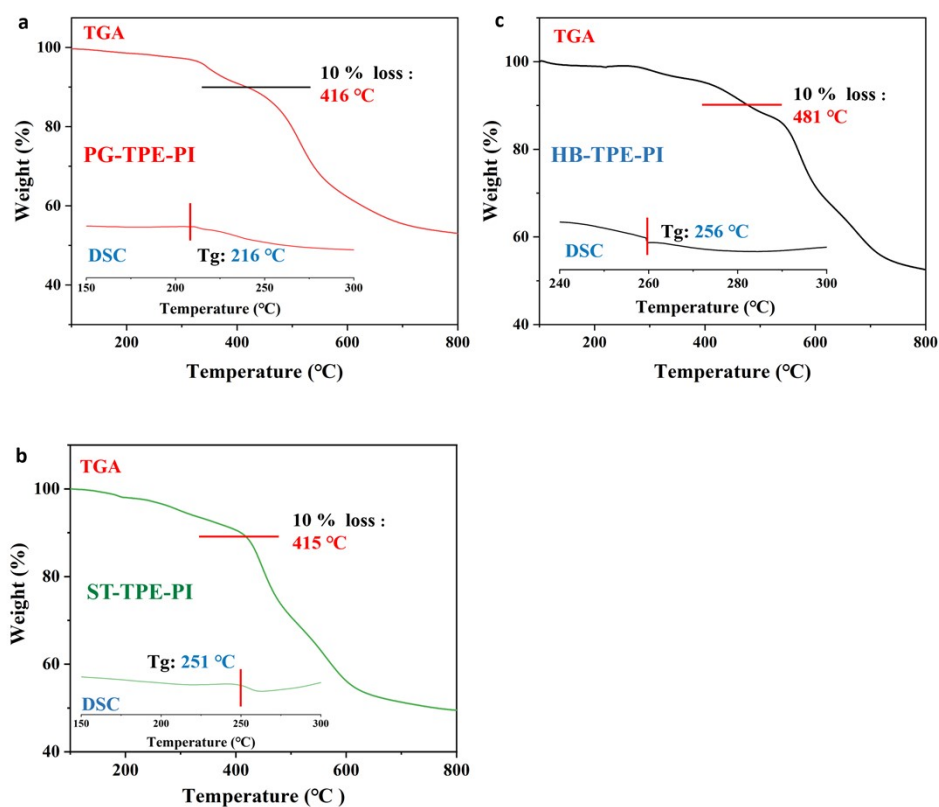


Figure S19. Thermal properties of the (a) PG-TPE-PI, (b) ST-TPE-PI and (c) HB-TPE-PI (tested in 10 °C/min under N₂).

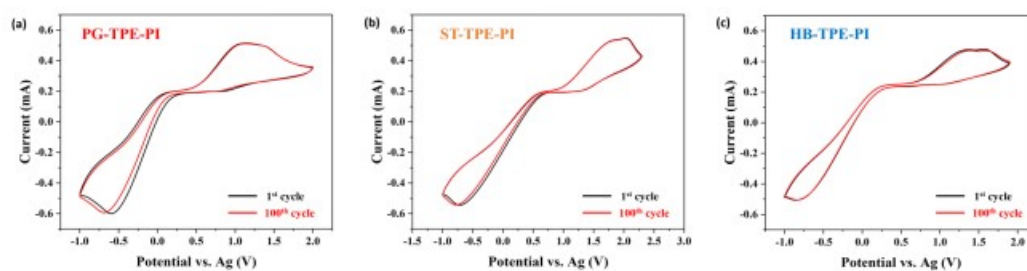


Figure S20. Cyclic voltammetric diagrams of (a) PG-TPE-PI (1×10^{-3} mol L⁻¹), (b) ST-TPE-PI (1×10^{-3} mol L⁻¹) and (c) HB-TPE-PI (1×10^{-3} mol L⁻¹) in 0.1 M TBAP/THF at a scan rate of 50 mV s⁻¹ (vs. Ag).

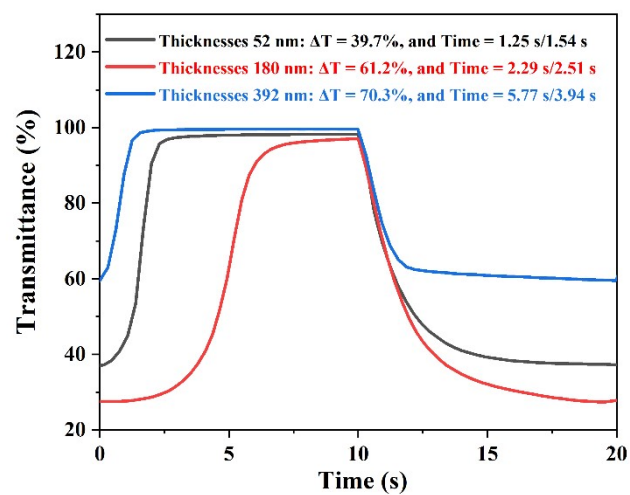


Figure S21. Electrochromic switching of PI film electrodes with different thicknesses. under a pulse of 20 s

Table S1. Solubility of Polyimides.

Polymer	Solvent ^a					
	NMP	DMAc	DMF	DMSO	THF	CHCl ₃
PG-TPE-PI	++	++	++	++	++	+ -
ST-TPE-PI	++	++	++	++	++	+
HB-TPE-PI	++	++	++	++	++	+

a. Solubility: ++, soluble at room temperature; +, soluble on heating; +-, partially soluble or swelling; -, insoluble even on heating. The solubility was determined by adding a 10 mg sample in 1 mL solvent.

Table S2. The density of Polyimides (average value of 5 pieces).

Polymer	Weight (mg) ^a	Thickness (nm) ^b	Area (cm ²) ^c	Density (g/cm ²) ^d
PG-TPE-PI	3.49	736	4	1.185
ST-TPE-PI	2.97	701	4	1.059
HB-TPE-PI	2.56	650	4	0.985

a. Average weight of five pieces of film.

b. The average thickness of the middle area of five films.

c. Average area of five films.

d. $\text{Density}_{\text{Polyimide}} = \text{Average Weight} / \text{Average Volume} = \text{Average Weight} / \text{Average (Thickness} \times \text{Area)}$

Table S3. Molecular weights^a of the polyimides.

Polyimide	$M_n \times 10^4$	$M_w \times 10^4$	PDI ^b
ST-TPE-PI	4.99	6.33	1.27
PG-TPE-PI	4.98	5.78	1.16
HB-TPE-PI	5.89	6.62	1.12

a DMF as the eluent at a flow rate of 1.0 mL/min and the test temperature of 80 °C.

b Polydispersity Index (M_w / M_n)

Table S4. Spin-coating processes for polymer films.

Thicknesses (nm)	Concentration (mg/mL)	Spin-speed (rpm)/Coating-time (s)	
		Low-speed	High-speed
50-70	20	150/20	1000/30
150-200	50	150/20	1000/30
370-420	50	100/10	500/20

Table S5. Electrochromic properties of typical transparent-to-black polymers

	Response speed (blanch/color) (s)	Applied potential (V)	CE (cm ² C ⁻¹)	Cycling stability	
				Cycling time	Decay (%)
PTPA-HDI ³	4.1/8.2	-1.3-1.3	214	100	--
TPOST-X ⁴	8.1/4.2	-2.8-2.8	--	1500	6.0
Blending-O ⁵	--	-1.1-1.1	184.63	100	6.09
SWNT/P2/ECP black ⁶	1.5/1.8	0.0-1.1	148	200	13
NTPB-PA ⁷	--	-0.2-0.48	163.7	500	4.4
PI-1a ⁸	1.3/1.1	0.0-1.3	99	5000	--
ECD-3 ⁹	7.1/31.5	-0.5-1.4	301.3	500	1.7
				1000	3.6
ECD-4 ⁹	6.5/21.7	-0.5-1.4	368.4	500	1.5
				1000	2.9
E-12TPE-PA ¹⁰	1.1/0.6	0-0.75	--	300	5.6
PDTT ¹¹	1.6/2.6	0-1.7	126	100	--
ECD 3 ¹²	8.5/68.7	-0.6-1.5	225.7	500	4.4
				1000	9.7
PG-TPE-PI (this work)	2.29/2.51	0.0-0.43	566	1000	0.0

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