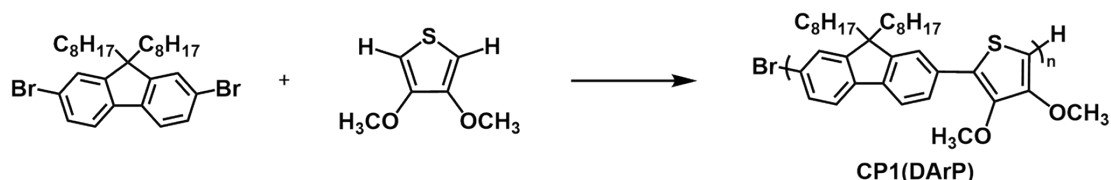


## Supporting Information

### Synthesis of CPs

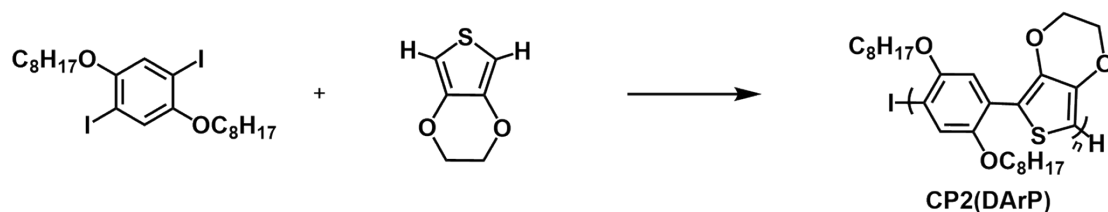
#### CP1



A mixture of 9,9-dioctyl-2,7-dibromofluorene (109.7 mg, 0.20 mmol),  $K_2CO_3$  (166 mg, 1.2 mmol), pivalic acid (20.4 mg, 0.20 mmol), Pd@DMSNs (35.4 mg), and N,N-dimethylacetamide (DMAc, 2 mL) was added to a 10 ml sealed tube. The system was freeze-pump-thaw for three times to remove residual water and air. Then, using a microsyringe, 3,4-dimethoxythiophene (28.8 mg, 0.20 mmol) was carefully added to the tube under a nitrogen atmosphere and the tube was tightly sealed under nitrogen. The system was then heated at 120 °C for 96 hours. After the reaction, tetrahydrofuran (THF) was added to the system to fully dissolve the product, and the supernatant (precipitate is base and catalyst) was collected after centrifugation. THF was then removed by rotary evaporation, and DMAc was removed by vacuum distillation. The resulting solid was dissolved in a small amount of THF (approximately 1 mL) and reprecipitated in anhydrous methanol. The precipitate was collected by centrifugation and washed for three times and then vacuum-dried for 12 hours to obtain 84 mg of solid product.

For CP3 and CP5, thiophene monomers were added together with other raw materials without the need for freeze-pump-thawing. The operating steps for other CPs were the same as CP1, with only the feeding amount and reaction conditions specified later.

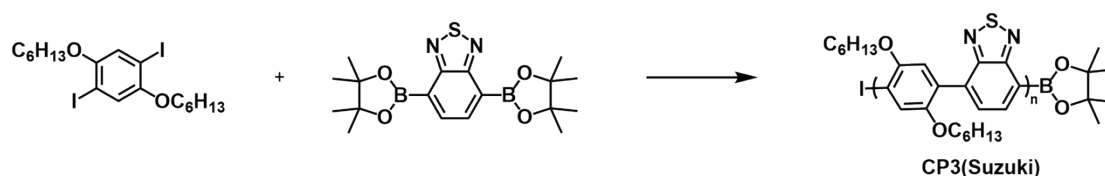
#### CP2



1,4-Diiodo-2,5-bis(2-ethylhexyloxy)benzene (175.9 mg, 0.30 mmol), 3,4-ethylenedioxythiophene (2.6 mg, 0.30 mmol),  $K_2CO_3$  (248.6 mg, 1.8 mmol), pivalic

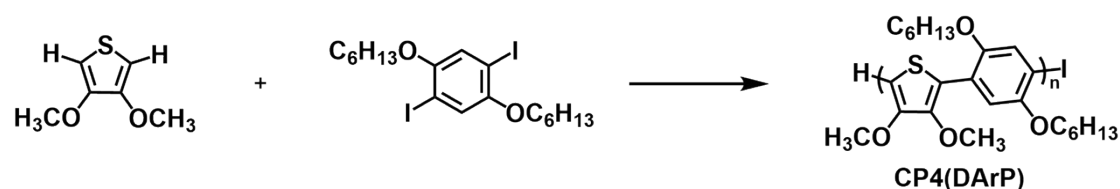
acid (30.6 mg, 0.30 mmol), Pd@DMSNs (35.4 mg) and DMAc (2 mL) were used during polymerization. The polymerization was carried out at 100 °C for 72 h to yield the product (127 mg).

### CP3



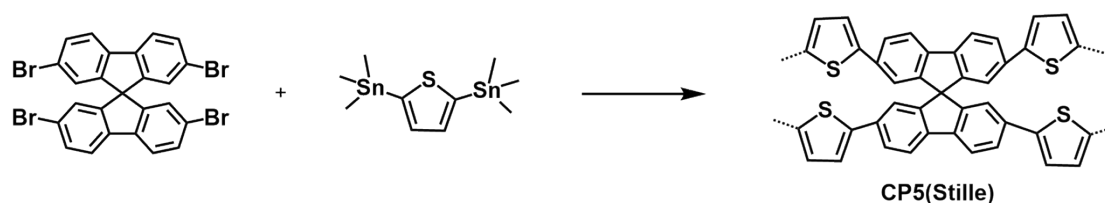
1,4-Diiodo-2,5-bis(hexyloxy)benzene (106.0 mg, 0.20 mmol), 4,7-bis(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-2,1,3-benzothiadiazole (77.6 mg, 0.20 mmol), K<sub>2</sub>CO<sub>3</sub> (96.7 mg, 0.70 mmol), Pd@DMSNs (35.4 mg) and DMAc (2 mL) were used during polymerization. The polymerization was carried out at 80 °C for 48 h to yield the product (46 mg).

### CP4



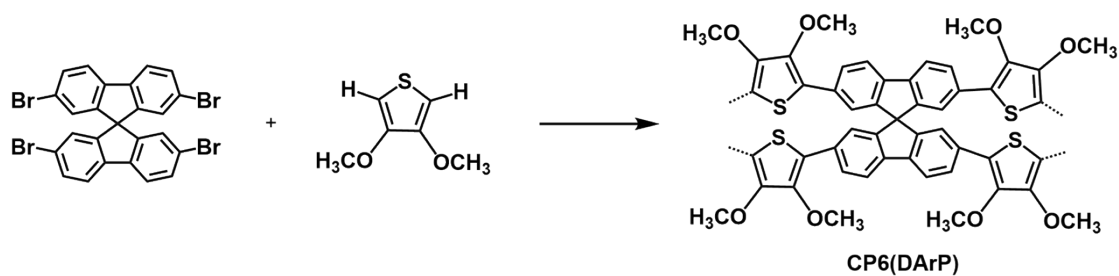
1,4-Diiodo-2,5-bis(hexyloxy)benzene (159.0 mg, 0.30 mmol), 3,4-dimethoxythiophene (43.3 mg, 0.30 mmol), K<sub>2</sub>CO<sub>3</sub> (290.0 mg, 2.1 mmol), pivalic acid (15 mg, 0.15 mmol), Pd@DMSNs (100.0 mg) and DMAc (1.5 mL) were used during polymerization. The polymerization was carried out at 100 °C for 96 h to yield the product (118 mg).

### CP5



2,2',7,7'-Tetrabromo-9,9'-spirobifluorene (94.8 mg, 0.15 mmol), 2,5-bis(trimethylstannyl)thiophene (123.0 mg, 0.30 mmol), Pd@DMSNs (20.0 mg) and DMAc (2 mL) were used during polymerization. The polymerization was carried out at 100 °C for 120 h to yield the product (49 mg).

## CP6



2,2',7,7'-Tetrabromo-9,9'-spirobifluorene (158.0 mg, 0.25 mmol), 3,4-dimethoxythiophene (72.0 mg, 0.50 mmol), potassium acetate (343.4 mg, 3.5 mmol), pivalic acid (25.0 mg, 0.25 mmol), Pd@DMSNs (20.0 mg) and DMAc (2 mL) were used during polymerization. The polymerization was carried out at 100 °C for 120 h to yield the product (82 mg).

Table S1 Summary of structural properties of 6 CPs

CPs	$M_n$	PDI
CP1	7008	1.55
CP2	4136	1.76
CP3	1394	1.15
CP4	7842	1.90
CP5	3911	1.39
CP6	5205	1.37

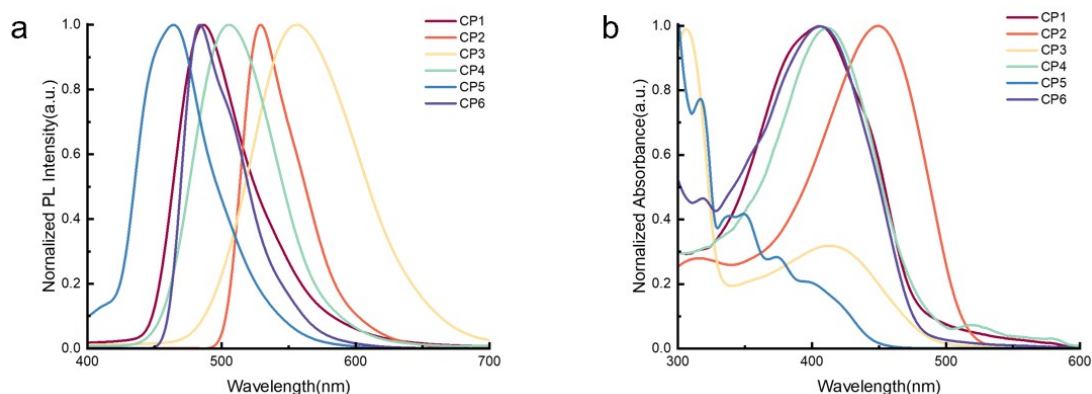


Fig. S1 (a) Normalized fluorescent emission spectra of 6 CPs. (b) Normalized UV-Vis absorption spectra of 6 CPs

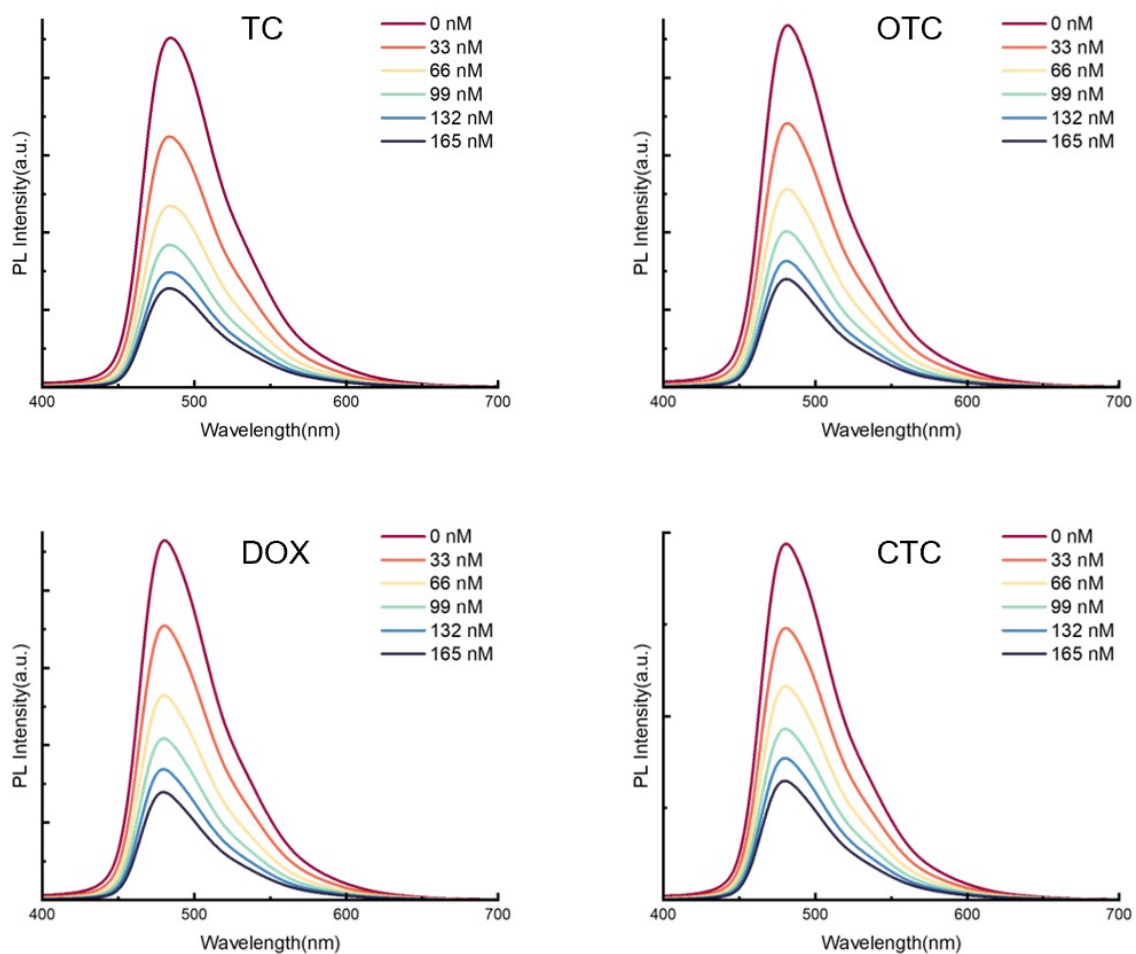


Fig. S2 Fluorescence quenching titration experiments for CP1 with TCs

Table S2 Fluorescent response pattern  $(I-I_0)/I_0$

Analytes	c ( $\mu$ M)	CP1	CP2	CP3	CP4	CP5	CP6
TC	33	-0.2876	-0.3706	-0.4408	-0.3789	-0.3468	-0.1992
TC	33	-0.2982	-0.3456	-0.4423	-0.3650	-0.4056	-0.1676
TC	33	-0.2928	-0.3550	-0.4199	-0.3615	-0.3866	-0.1616
TC	33	-0.3088	-0.3532	-0.4244	-0.3726	-0.3746	-0.1556
TC	33	-0.2787	-0.3624	-0.4157	-0.3655	-0.3659	-0.1637
TC	66	-0.4820	-0.6032	-0.6754	-0.5992	-0.6214	-0.3398
TC	66	-0.4902	-0.5746	-0.6850	-0.5807	-0.6148	-0.3332
TC	66	-0.4761	-0.6000	-0.6696	-0.5912	-0.6220	-0.3168
TC	66	-0.4839	-0.5973	-0.6711	-0.5983	-0.5983	-0.3303
TC	66	-0.4756	-0.5920	-0.6638	-0.5844	-0.5970	-0.2951
TC	99	-0.5933	-0.7341	-0.8092	-0.7364	-0.7475	-0.4166
TC	99	-0.5904	-0.7136	-0.8147	-0.7243	-0.7371	-0.4225
TC	99	-0.5950	-0.7302	-0.8042	-0.7332	-0.7468	-0.4140

TC	99	-0.6045	-0.7241	-0.8045	-0.7324	-0.7419	-0.4294
TC	99	-0.5914	-0.7271	-0.7980	-0.7206	-0.7370	-0.4086
TC	132	-0.6730	-0.8152	-0.8811	-0.8207	-0.8232	-0.5189
TC	132	-0.6696	-0.7990	-0.8890	-0.8092	-0.8244	-0.5142
TC	132	-0.6684	-0.8095	-0.8763	-0.8160	-0.8231	-0.4978
TC	132	-0.6814	-0.8064	-0.8790	-0.8161	-0.8109	-0.5074
TC	132	-0.6625	-0.8117	-0.8759	-0.8053	-0.8126	-0.4592
TC	165	-0.7189	-0.8655	-0.9246	-0.8712	-0.8707	-0.5571
TC	165	-0.7137	-0.8530	-0.9280	-0.8634	-0.8689	-0.5496
TC	165	-0.7146	-0.8545	-0.9227	-0.8685	-0.8692	-0.5527
TC	165	-0.7233	-0.8615	-0.9237	-0.8687	-0.8633	-0.5719
TC	165	-0.7068	-0.8607	-0.9192	-0.8614	-0.8613	-0.5363
OTC	33	-0.2693	-0.3606	-0.3877	-0.3791	-0.3707	-0.1919
OTC	33	-0.2714	-0.3396	-0.3662	-0.3859	-0.3586	-0.1970
OTC	33	-0.2654	-0.3360	-0.3741	-0.3883	-0.3610	-0.1890
OTC	33	-0.2689	-0.3419	-0.3732	-0.3855	-0.3617	-0.1911
OTC	33	-0.2599	-0.3407	-0.3588	-0.3773	-0.3606	-0.1972
OTC	66	-0.4498	-0.5422	-0.6151	-0.6109	-0.5849	-0.3120
OTC	66	-0.4435	-0.5433	-0.6157	-0.6120	-0.5613	-0.3072
OTC	66	-0.4426	-0.5504	-0.6229	-0.6161	-0.5717	-0.2987
OTC	66	-0.4505	-0.5551	-0.6196	-0.6083	-0.5713	-0.3144
OTC	66	-0.4458	-0.5492	-0.6137	-0.6097	-0.5767	-0.3123
OTC	99	-0.5709	-0.6889	-0.7763	-0.7508	-0.7161	-0.3995
OTC	99	-0.5663	-0.6827	-0.7638	-0.7449	-0.6940	-0.3899
OTC	99	-0.5597	-0.6875	-0.7876	-0.7526	-0.7026	-0.3879
OTC	99	-0.5672	-0.6859	-0.7687	-0.7435	-0.6928	-0.3914
OTC	99	-0.5624	-0.6861	-0.7668	-0.7501	-0.6953	-0.4030
OTC	132	-0.6515	-0.7768	-0.8577	-0.8327	-0.7854	-0.4646
OTC	132	-0.6415	-0.7677	-0.8547	-0.8284	-0.7725	-0.4561
OTC	132	-0.6395	-0.7746	-0.8679	-0.8293	-0.7761	-0.4561
OTC	132	-0.6403	-0.7768	-0.8538	-0.8280	-0.7805	-0.4501
OTC	132	-0.6419	-0.7747	-0.8553	-0.8313	-0.7919	-0.4647
OTC	165	-0.7022	-0.8261	-0.9034	-0.8842	-0.8442	-0.5147
OTC	165	-0.6938	-0.8269	-0.9007	-0.8799	-0.8368	-0.5039
OTC	165	-0.6930	-0.8317	-0.9128	-0.8816	-0.8394	-0.5080
OTC	165	-0.6949	-0.8333	-0.9029	-0.8821	-0.8382	-0.5056
DOX	165	-0.6954	-0.8357	-0.9035	-0.8845	-0.8440	-0.5123
DOX	33	-0.2352	-0.3833	-0.4153	-0.3945	-0.3676	-0.1923
DOX	33	-0.2561	-0.3528	-0.3844	-0.3997	-0.3635	-0.2059
DOX	33	-0.2652	-0.3454	-0.3837	-0.3972	-0.3619	-0.1653
DOX	33	-0.2728	-0.3636	-0.3895	-0.3674	-0.3662	-0.1232
DOX	33	-0.2636	-0.3225	-0.3764	-0.3599	-0.3679	-0.1289

DOX	66	-0.4336	-0.5788	-0.6487	-0.6195	-0.5721	-0.3163
DOX	66	-0.4516	-0.5670	-0.6381	-0.6217	-0.5799	-0.3064
DOX	66	-0.4458	-0.5739	-0.6293	-0.6204	-0.5739	-0.3092
DOX	66	-0.4532	-0.5719	-0.6330	-0.6022	-0.5685	-0.2870
DOX	66	-0.4405	-0.5628	-0.6270	-0.5979	-0.5757	-0.2773
DOX	99	-0.5493	-0.7154	-0.7829	-0.7497	-0.7084	-0.3910
DOX	99	-0.5628	-0.7123	-0.7736	-0.7467	-0.7090	-0.3884
DOX	99	-0.5703	-0.7102	-0.7643	-0.7465	-0.7049	-0.4219
DOX	99	-0.5751	-0.7032	-0.7671	-0.7435	-0.7033	-0.3766
DOX	99	-0.5668	-0.6919	-0.7548	-0.7413	-0.7109	-0.3723
DOX	132	-0.6360	-0.7926	-0.8551	-0.8336	-0.7941	-0.4667
DOX	132	-0.6427	-0.7934	-0.8475	-0.8278	-0.7924	-0.4542
DOX	132	-0.6491	-0.7875	-0.8497	-0.8293	-0.7966	-0.4626
DOX	132	-0.6599	-0.7947	-0.8491	-0.8276	-0.7932	-0.4390
DOX	132	-0.6499	-0.7829	-0.8399	-0.8149	-0.7952	-0.4298
DOX	165	-0.7004	-0.8434	-0.8988	-0.8781	-0.8459	-0.5102
DOX	165	-0.7027	-0.8459	-0.8986	-0.8751	-0.8465	-0.5043
DOX	165	-0.6965	-0.8411	-0.8970	-0.8778	-0.8431	-0.5178
DOX	165	-0.7077	-0.8432	-0.9011	-0.8750	-0.8470	-0.4890
DOX	165	-0.7021	-0.8354	-0.8930	-0.8681	-0.8478	-0.4844
CTC	33	-0.2446	-0.3002	-0.3542	-0.3381	-0.3515	-0.1484
CTC	33	-0.2407	-0.2885	-0.3407	-0.3807	-0.3427	-0.1360
CTC	33	-0.2330	-0.3181	-0.3168	-0.3513	-0.3382	-0.1404
CTC	33	-0.2258	-0.2742	-0.3361	-0.3485	-0.3374	-0.1536
CTC	33	-0.2346	-0.3126	-0.3530	-0.3129	-0.3339	-0.1623
CTC	66	-0.4048	-0.5282	-0.5919	-0.5456	-0.5559	-0.2712
CTC	66	-0.3989	-0.4991	-0.5620	-0.5835	-0.5469	-0.2543
CTC	66	-0.3876	-0.5161	-0.5466	-0.5549	-0.5420	-0.2478
CTC	66	-0.3989	-0.4877	-0.5698	-0.5769	-0.5710	-0.2521
CTC	66	-0.4026	-0.5069	-0.5498	-0.5548	-0.5502	-0.2636
CTC	99	-0.5233	-0.6527	-0.7185	-0.6923	-0.6892	-0.3532
CTC	99	-0.5318	-0.6396	-0.7063	-0.7224	-0.6779	-0.3449
CTC	99	-0.5024	-0.6466	-0.6914	-0.7080	-0.6903	-0.3368
CTC	99	-0.5139	-0.6313	-0.7124	-0.7090	-0.7037	-0.3376
CTC	99	-0.5284	-0.6463	-0.6926	-0.6913	-0.6826	-0.3401
CTC	132	-0.6066	-0.7490	-0.8135	-0.7818	-0.7804	-0.4137
CTC	132	-0.6096	-0.7422	-0.7974	-0.7983	-0.7803	-0.4036
CTC	132	-0.6017	-0.7427	-0.7881	-0.7894	-0.7828	-0.4087
CTC	132	-0.6088	-0.7438	-0.7988	-0.7942	-0.7960	-0.4002
CTC	132	-0.6182	-0.7511	-0.7674	-0.7787	-0.7755	-0.4042
CTC	165	-0.6663	-0.8175	-0.8625	-0.8370	-0.8314	-0.4681
CTC	165	-0.6592	-0.8041	-0.8570	-0.8526	-0.8295	-0.4709

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CTC	165	-0.6573	-0.8117	-0.8504	-0.8365	-0.8337	-0.4738
CTC	165	-0.6538	-0.8005	-0.8558	-0.8552	-0.8483	-0.4619
CTC	165	-0.6717	-0.8063	-0.8259	-0.8376	-0.8339	-0.4540