Supporting Information

Precise Membrane Separation of Nanoparticles Using a Microporous

Conjugated Polymer Containing Radially π -Conjugated Molecular

Carbocycles

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Materials and General Information

All glassware was oven-dried and cooled under an inert atmosphere of nitrogen. Airsensitive reactions were carried out using the standard Schlenk technique under nitrogen. Work-up and purification procedures were carried out with reagent-grade solvents under air. High resolution mass spectrometry (HR-MS) analyses were carried out using MALDI-TOF-MS techniques. Samples were characterized with infrared spectroscopy (IR, FTS-7000, Varian, USA). NMR spectra were recorded on Bruker BioSpin (¹H 400 MHz, ¹³C 100 MHz) spectrometer, and chemical shifts were reported as the delta scale in ppm relative to CDCl₃ (δ = 7.26 ppm) for ¹H NMR and CDCl₃ (δ = 77.0 ppm) for ¹³C NMR. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constant (Hz), and integration. The molecular weight distribution, relative number average and weightaverage molecular weights were determined at 40°C by gel permeation chromatography (GPC, Waters 1515) equipped with a refractive index detector (Waters 2414) and a series of Styragel HR₁, HR₃ and HR₅ (DMF) column with the eluent at 1.0 mL/min. The calibration was built on polystyrene standards. The morphology of the sample was studied using JSM-6700F scanning electron microscope (SEM). Transmission electron microscopy (TEM) images were collected on a JEM-2010 electron microscope, operated at an acceleration voltage of 200 kV. Scanning tunneling microscope (STM) image was collected on a SPECS STM 150 Aarhus with SPECS 260 electronics. Au (111) surface wascleaned by cycles of bombardment with Ar⁺ ions and annealing at 750 K. The steady-state photoluminescence (PL) spectra were recorded using a Perkin-Elmer LS 55 fluorescence spectrometer. Other chemicals were obtained from commercial suppliers (Innochem or Acros). Nylon syringe filters (pore size, 0.22 μ m; filtration area, 0.25 π cm²) were purchased from Sinopharm Chemical Regent Company (SCRC).

Synthesis procedures:

2,7-dibromophenanthrene-9,10-dione (2). Compound **2** was synthesized according to literature reports.^[S1]

1,3-bis(4-chlorophenyl)propan-2-one (3). Compound **3** was synthesized according to literature reports.^[S2]

5,10-dibromo-1,3-bis(4-chlorophenyl)-2H-cyclopenta[l]phenanthren-2-one (4). To a round-bottom flask containing 2,7-dibromophenanthrene-9,10-dione (5.5 g, 15.0 mmol) and 1,3-bis(4-chlorophenyl)propan-2-one (4.2 g, 15.0 mmol) in EtOH (5 mL) was added KOH (560 mg, 10.0 mmol) as a solution in EtOH (10 mL). The reaction was then heated to reflux for 0.5 h and cooled to room temperature. The resulting purple solid was filtered and washed with ice cold MeOH (15 mL) to provide **4** as a

green solid (5.5 g, 60% yield).

6,11-dibromo-2,3-bis(4-(tert-butyl)phenyl)-1,4-bis(4-chlorophenyl)triphenylene

(6). 5,10-dibromo-1,3-bis(4-chlorophenyl)-2H-cyclopenta[1]phenanthren-2-one **4** (3.0 g, 4.9 mmol) and 1,2-bis(4-(*tert*-butyl)phenyl)ethyne **5** (1.5 g, 5 mmol) were refluxed under argon in diphenyl ether (4 mL) for 48 h. After cooling to room temperature, the resulting residue was recrystallized from MeOH to yield the desired product as a white solid **6** (2.8 g, 65% yield). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 8.20 (d, *J* = 8.8 Hz, 2H), 7.62 (d, *J* = 2 Hz, 4H), 7.49 (d, *J* = 8.0 Hz, 2H), 7.12 (d, *J* = 8.4 Hz, 4H), 6.98 (d, *J* = 8.4 Hz, 4H), 6.90 (d, *J* = 8.4 Hz, 4H), 6.53 (d, *J* = 8.4 Hz, 4H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 148.55, 141.80, 140.37, 136.25, 136.22, 133.10, 132.81, 132.79, 131.92, 130.71, 130.09, 129.58, 129.45, 128.39, 124.58, 123.59, 120.20,

34.21, 31.22. HR-MS (MALDI-TOF) *m/z* calcd. for C₅₀H₄₀Br₂Cl₂ [M]⁺: 870.0853, found: 870.0903. IR (KBr) cm⁻¹: 3029, 2955, 2898, 2862, 1899, 1595, 1483, 1394, 1364, 1240, 1091, 1011, 1000, 886, 854, 833, 798, 774, 749, 723.

Compound 7. Compound 7 was synthesized according to literature reports.^[S3]

Compound 8. Dibromide 7 (2.0 g, 2.28 mmol) was dissolved in THF (20 ml) and cooled to -78 °C. To this solution was added a 2.5 M solution of *n*-BuLi in hexanes (2.0 mL, 5 mmol) over 2 min. Immediately, neat isopropyl pinacol borate (1.1 mL, 5.3 mmol) was added rapidly and the solution was stirred for 20 min. Water (10 mL) was then added to the solution and the mixture was allowed to stir for 15 min at room temperature. The aqueous layer was extracted with CH₂Cl₂ and concentrated under reduced pressure. The crude product was purified by column chromatography (hexane/ethyl acetate = 1/1) to afford 8 (1.8 g, 81% yield) as a white solid. ¹H NMR $(CDCl_3, 400 \text{ MHz}): \delta (ppm) 7.75 (d, J = 7.2 \text{ Hz}, 4\text{H}), 7.40 (d, J = 8.4 \text{ Hz}, 4\text{H}), 7.49 (d, J = 8.4 \text{ Hz}, 4\text{Hz}), 7.49 (d, J = 8.4 \text{ Hz}), 7.49 (d, J = 8.4 \text{ Hz$ J = 8.0 Hz, 2H), 7.34 (s, 8H), 3.42 (s, 18H), 1.33 (s, 24H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 146.43, 142.73, 142.59, 134.89, 133.354, 133.254, 133.087, 125.99, 125.25, 83.72, 74.84, 74.59, 74.54, 51.91, 24.82. HR-MS (MALDI-TOF) m/z calcd. for C₆₀H₇₀B₂O₁₀Ag [M]⁺: 1079.4207, found: 1079.7707. IR (KBr) cm⁻¹: 3082, 3029, 2977, 2930, 2899, 2831, 2014, 1921, 1824, 1790, 1754, 1669, 1608, 1504, 1453, 1393, 1352, 1319, 1268, 1137, 1077, 1020, 939, 858, 834, 760.

Compound 9. To a degassed suspension of **8** (195.0 mg, 0.2 mmol), **6** (174.0 mg, 0.2 mmol), and KOH (260.0 mg, 1.88 mmol) in THF/H₂O (200 mL/20 mL) was added $Pd(PPh_3)_4$ (10.0 mg, 0.014 mmol), then the mixture was degassed for 30 min. The

S5

mixture was then heated up to 80 °C for 48 h under nitrogen atmosphere. After cooling down to room temperature, water was added and the mixture was extracted with DCM. The combined organic layer was dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure to afford crude product **9** as a white solid for the next step without further purification.

Compound 10. To a 50-mL round-bottom flask (vessel A) containing a magnetic stirring bar were added SnCl₂·2H₂0 (305 mg, 1.3 mmol), THF (25 mL) and concentrated HCl/H₂O (0.2 mL, 12 mol/L) were added, and the resultant mixture was further stirred at room temperature for 30 min. To another 200-mL round-bottom flask (vessel B) containing a magnetic stirring bar were added the above crude product 9 and dry THF (10 mL). A solution of H₂SnCl₄/THF (18 mL, 0.7 mmol, 0.04 M in THF) in vessel A was added. After stirring the mixture at room temperature for 2 h, the mixture was quenched with aqueous sodium hydroxide, extracted with CH₂Cl₂, dried over Na₂SO₄, and concentrated under reduced pressure. The crude product was purified by column chromatography (hexane/ $CH_2Cl_2 = 4/1$) to afford 10 (52.3 mg, 21% yield over two steps) as a yellow solid. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 8.14 (d, J = 8.8 Hz, 2H), 7.68 (d, J = 8.8 Hz, 2H), 7.54-7.41 (m, 16H), 7.39 (d, J = 8.4 Hz, 4H), 7.31 (d, J = 8.4 Hz, 6H), 7.25-7.20 (m, 4H), 7.17-7.12 (m, 4H), 7.04 (d, J = 8.4 Hz, 6H), 6.91 (d, J = 9.6 Hz, 2H), 6.71 (d, J = 9.6 Hz, 2H), 6.25 (d, J = 8.8 Hz, 2H); ¹³C NMR (CDCl₃, 100 MHz): δ (ppm) 148.20, 142.71, 140.63, 138.29, 138.25, 137.95, 137.87, 137.84, 137.79, 136.99, 136.91, 134.15, 133.78, 132.23, 132.09, 131.61, 131.57, 130.63, 130.19, 129.84, 128.35, 127.43, 127.38, 127.34, 127.29, 127.19, 126.86, 123.91, 123.68, 123.42, 122.78, 34.13, 31.15. HR-MS (MALDI-TOF) *m/z* calcd. for C₉₂H₆₈Cl₂ [M]⁺: 1242.4698, found: 1242.8126. IR (KBr) cm⁻¹: 3071, 3022, 2957, 2917, 2855, 1895, 1589, 1481, 1385, 1365, 1263, 1088, 1014, 810, 729.

Polymer PS2. To a mixture of **10** (121.3 mg, 97.7 μmol), 2,2'-bipyridyl (179.0 mg, 1.14 mmol), and 1,5-cyclooctadiene (140 μL, 1.14 mmol) in a round-bottom flask (25 mL) was added anhydrous DMF/toluene (6 mL/6 mL). The mixture was bubbled with Ar for 0.5 h before bis(1,5-cyclooctadiene)nickel(0) (315.0 mg, 1.1 mmol) was added in one portion. Thereafter, the mixture was heated up to reflux for 120 h to give a deep purple suspension. Upon cooling to room temperature, the solvent was removed by rotary evaporator. The crude product was washed with methanol, filtered and washed with water, 0.5 M HCl solution in water, water, 0.5 M NaOH solution in water, water, methanol, acetone, methanol and hexane, and then the precipitate was collected, giving 83.0 mg (72.4% yield) of polymer **PS2** as a yellow solid: M_n = 8043, M_w = 16430. ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 8.17-8.01 (br), 7.59-7.31 (br), 7.10-6.93 (br), 6.90-6.75 (br), 6.70-6.60 (br), 6.34-6.20 (br), 1.14-0.95(br). IR (KBr) cm⁻¹: 3075, 3024, 2960, 2921, 2863, 1905, 1587, 1486, 1385, 1263, 1106, 1022, 1001, 814, 732.



Figure S1. Synthesis procedure for compound 14.

Compound 11. Compound **11** was synthesized according to the reported method in the literature.^[S4]

Compound 12. To a long neck flask was added 5,10-dibromo-1,3-diphenyl-2Hcyclopenta[1]phenanthren-2-one 3.7 mmol), (2.00)1,2-bis(4-(tertg, butyl)phenyl)ethyne (1.074 g, 3.7 mmol) and diphenyl ether (3 mL). The mixture was degassed 5 times by pumping and backfilling with nitrogen. The reaction was heated to 260 °C for 36 hours and then cooled to 70 °C before excessive MeOH was added. The resulting precipitate was collected by filtration and further purified by silica gel column chromatography (eluent 20% CH₂Cl₂/petroleum ether) to give pure 6,11dibromo-2,3-bis(4-tert phenyl)-1,4-diphenyltriphenylene 11 (1.93 g, 2.4 mmol, 65%). ¹H NMR (400 MHz, CDCl₃): δ (ppm) 8.19 (d, J = 8.7 Hz, 2H), 7.67 (d, J = 2.0 Hz, 2H), 7.47 (dd, J = 8.7, 2.0 Hz, 2H), 7.18-7.11 (m, 6H), 7.07-7.04 (m, 4H), 6.88-6.84 (m, 4H), 6.57-6.53 (m, 4H), 1.15 (s, 18H). ¹³C NMR (100 MHz, CDCl₃): δ(ppm) **S**8 148.09, 141.86, 137.38 (s), 136.78, 132.87, 132.39, 131.86, 130.86, 130.07, 129.39, 128.14 (s), 126.60, 124.44, 123.31, 120.02, 77.32, 77.00, 76.68, 34.13, 31.22. HR-MS (MALDI-TOF) m/z calcd. for: C₅₀H₄₂Br₂[M]⁺: 802.1633, found: 802.1679.

Compound 13. To a round-bottom flask (500 mL) was added compound **8** (181.4 mg, 0.186 mmol), **12** (149.30 mg, 0.186 mmol), THF (250 mL) and H₂O (20 mL), then potassium hydrate (134 mg, 2.38 mmol) and Pd(PPh₃)₄ (30.06 mg, 0.026 mmol) was added after argon bubbling for 25 minutes. Then, the mixture was reacted at 75 °C for 48 hours. After cooling to room temperature, the solvent was removed under vacuum and the residue was extracted with CH₂Cl₂. The organic layer was dried by anhydrous MgSO₄, filtered and concentrated under reduced pressure to afford macrocycle intermediate **13** as a yellow solid for the next step without further purification.

Compound 14. To a 50 mL round-bottom flask (vessel A), sodium metal (274 mg, 11.90 mmol), dry THF (12 mL), and naphthalene (1.00 g, 7.82 mmol) were added under nitrogen and the resultant mixture was stirred at room temperature for 24 hours. To another 250 mL flask (vessel B) containing the intermediate **13** in dry THF (30 mL) was added a solution of sodium napthalenide (2 mL, 2 mmol, 1.0 M in THF) at - 78 °C. This resulting mixture was reacted at -78 °C for 2 hours before quenched with 1.5 mL of I₂ solution (1 M in THF). After warmed up to room temperature, the mixture was added aqueous saturated sodium thiosulfate, extracted with CH₂Cl₂, dried over anhydrous MgSO₄, and concentrated under reduced pressure. Purification by column chromatography with hexane/CH₂Cl₂ as the eluent (v/v, 4:1) afforded pure **14** (43.73 mg, 22%) as yellow solid. ¹H NMR (400 MHz, CDCl₃) δ 8.06 (d, *J* = 8.8 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.06 (s, 4H), 6.95 (d, *J* = 7.9 Hz, 7.21 (d, *J* = 8.2 Hz, 6H), 7.13 (d, *J* = 8.8 Hz, 2H), 7.

8H), 6.60 (d, J = 7.9 Hz, 2H), 6.21 (d, J = 7.9 Hz, 2H), 1.02 (s, 18H). ¹³C NMR (101 MHz, CD₂Cl₂) δ 132.45, 131.52, 130.62, 129.75, 127.91, 127.65,126.86, 126.68, 125.86, 123.35, 123.08, 122.36, 30.95. HR-MS (MALDI-TOF) m/z calcd. for: C₉₂H₇₀[M]⁺: 1174.5477, found: 1174.5469. IR (KBr) cm⁻¹: 3023.72, 2959.35, 2925.28, 2866.35, 1723.40, 1587.35, 1485.39, 1457.41, 1443.55, 1271.30, 1120.25, 1022.72, 813.52, 736.91, 702.13, 603.15, 578.78, 518.47.

Synthesis of Gold nanoparticles (AuNP): Mercaptopropionic acid (MPA)-stabilized gold nanoparticles were prepared according to the reported method.^[S5] A solution of HAuCl₄·3H₂O (57 mg, 0.145 mmol) in 25 ml water was added to 250 ml of refluxing water. Then 25 ml of a mixed solution of MPA-Na (0.435 mmol) and trisodium citrate·2H₂O (500 mg, 1.70 mmol) was added rapidly. The solutions were refluxed for 6 hours. For separation from the excess of salts, the dispersions were treated with HCl (2 M) until the aggregated particles were precipitated. The precipitate was removed from the mother liquor by centrifugation. Then, it was re-dispersed by adding 250 ml water and adjusting the pH to 10 using NaOH (1.0 M).



Figure S2. ¹H NMR spectrum of compound 6 in CDCl₃.



Figure S3. ¹³C NMR spectrum of compound 6 in CDCl₃.



Figure S4. MALDI-TOF-MS spectrum (black) and simulated data (red) for compound 6.



Figure S5. ¹H NMR spectrum of compound 8 in CDCl₃.



Figure S6. ¹³C NMR spectrum of compound 8 in CDCl₃.



Figure S7. MALDI-TOF-MS spectrometry (black) and simulated data (red) for compound $8 + Ag^+$.



Figure S8. ¹H NMR spectrum of compound 10 in CDCl₃.



Figure S9. ¹³C NMR spectrum of compound 10 in CDCl₃.



Figure S10. MALDI-TOF-MS spectrometry (black) and simulated data (red) for compound 10.



Figure S11. Expanded 2D ¹H-¹H COSY NMR spectrum (400 MHz, CDCl₃) of 10.



Figure S12. Expanded 2D (H, C)-HSQC spectrum (400 MHz, CDCl₃) of 10.



Figure S13. Expanded 2D (H, C)-HMBC spectrum (400 MHz, CDCl₃) of 10.



Figure S14. MALDI-TOF-MS spectrometry for PS2.



Figure S15. GPC trace of PS2 using DMF as the solvent.



Figure S16. ¹H NMR spectra of 10 (brown) and PS2 (red) in CDCl₃.



Figure S17. ¹H NMR spectra of PS2 in CDCl₃.



Figure S18. FTIR spectra of PS2 (red) and monomer 10 (black).



Figure S19. TG analyses. TGA data of as-synthesized PS2 confirm the high thermal stability (minimum up to 200 $^{\circ}$ C) under N₂ atmosphere.



Figure S20. Top-view SEM image of the PS2 membrane on the SCRC filter support.



Figure S21. SEM image of the SCRC filter support.



Figure S22. a) A HR-TEM image showing the self-assembled **PS2** in solid state. The experiment was carried out on JEM ARM-200F microscope. b) A high resolution STM image showing the CPP-based nanosheets.



Figure S23. (a) Selected 2D WAXD pattern of PS2 at room temperature. (b) Corresponding 1D WAXD intensity curve of PS2 sample.



Figure S24. ¹H NMR spectrum of 14 in $CS_2/CD_2Cl_2(1:1)$.



Figure S25. ¹³C NMR spectrum of 14 in CD₂Cl₂.



Figure S26. Expanded 2D ¹H-¹H COSY NMR spectrum (400 MHz, CS₂/CD₂Cl₂(1:1)) of 14.



Figure S27. Expanded 2D (H, C)-HSQC NMR spectrum (400 MHz, CS₂/CD₂Cl₂(1:1))

of **14**.



Figure S28. Expanded 2D (H, C)-HMBC NMR spectrum (400 MHz, $CS_2/CD_2Cl_2(1:1)$) of 14.



Figure S29. ¹³C NMR (a), DEPT-135° NMR (b), DEPT-90° NMR (c) spectra of 14 in CD_2Cl_2 .



Figure S30. ¹H NMR spectrum of 6,11-dibromo-2,3-bis(4-(*tert*-butyl) phenyl)-1,4diphenyltriphenylene (**12**) in CDCl₃.



Figure S31. ¹³C NMR spectrum of 6,11-dibromo-2,3-bis(4-(*tert*-butyl) phenyl)-1,4-

diphenyltriphenylene (12) in CDCl₃.



Figure S32. HR-MS (MALDI-TOF) data for 6,11-dibromo-2,3-bis(4-(*tert*-butyl)

phenyl)-1,4-diphenyltriphenylene (12).

Empirical formula	C ₉₂ H ₇₀
Formula weight	1175.48
Temperature/K	100(2)
Crystal system	monoclinic
Space group	Cc
a/Å	12.920
b/Å	65.967
c/Å	18.697
α/°	90
β/°	94.64
γ/°	90
Volume/Å ³	15883.1
Ζ	8
$\rho_{calc}g/cm^3$	0.983
μ/mm ⁻¹	0.055
F(000)	4976.0
Crystal size/mm ³	0.06 imes 0.05 imes 0.02
Radiation	synchrotron ($\lambda = 0.71073$)
2Θ range for data collection/°	2.51 to 50.084
Reflections collected	14071
Independent reflections	14071 [Rsigma = 0.0574]
Data/restraints/parameters	14071/2/1669
Goodness-of-fit on F ²	1.029
Final R indexes [I>= 2σ (I)]	$R_1 = 0.0452, wR_2 = 0.1193$
Final R indexes [all data]	$R_1 = 0.0514, wR_2 = 0.1226$
Largest diff. peak/hole / e Å ⁻³	0.25/-0.24
Flack parameter	-1(5)
CCDC number	2020975

 Table S1. Crystal data, data collection and refinement of compound 14.

References.

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