## Solvent mediated thermodynamically favorable helical supramolecular selfassembly: Recognition behavior towards achiral and chiral analytes

Meenal Kataria<sup>+</sup>, Youngseo Kim<sup>+</sup>, Hong Diem Chau<sup>+</sup>, Na Yeon Kwon<sup>+</sup>. Yongju Hong<sup>+</sup>, Taekyung Kim<sup>+</sup>, Jaewan Ko<sup>‡</sup>, Myung Kook Son<sup>+</sup>, Joona Bang<sup>‡</sup>, Sungnam Park<sup>+</sup>, Hugh I. Kim<sup>+</sup>, Kwangyeol Lee<sup>+</sup> and Dong Hoon Choi<sup>+</sup> \*

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#### **Experimental Procedures**

### General methods and apparatus

Physical measurements UV-Vis absorption spectra were recorded on an Agilent 8453 photodiode array spectrometer ( $\lambda = 190-1100$  nm). Fluorescence spectra were recorded on a HITACHI-700 fluorescence spectrometer. Transmission electron microscopy (TEM) images were obtained using a TECNAI G2 20 S-Twin (200 kV) field-emission microscope. Time-resolved fluorescence spectra were recorded using an LDH-P-520 PicoQuant time-resolved fluorescence spectrometer. <sup>1</sup>H NMR spectra were recorded on a Bruker 500 MHz spectrometer (Ascend 500, Bruker) using CDCl<sub>3</sub> as the solvent and tetramethylsilane (TMS) as the internal standard. The data are reported as follows: chemical shifts in ppm, multiplicity (s = singlet, br = broad signal, d = doublet, t = triplet, m = multiplet), coupling constants (J) in Hz, integration, and interpretation. Silica gel 60 (60–120 mesh) was used for column chromatography. Highresolution TEM (HRTEM) was performed at the FEI Nanoport in the Korea Basic Science Institute (Seoul Center) using a Titan Probe Cs TEM 300 kV microscope. Particle sizes were measured by dynamic light scattering (DLS) using a BI-200SM (Brookhaven Instruments Corporation) with a 616 nm laser. The samples were then diluted in toluene at room temperature. Circular dichroism (CD) spectra were measured using a CD spectrometer (J-815, JASCO). Isothermal titration calorimetry (ITC) was performed using a MicroCal ITC200 USA microcalorimeter, equipped with an instrument-controlled Hamilton syringe with a volume capacity of 40 µL. The titration was performed automatically by diluting with continuous stirring of 700 rpm.

#### UV-vis absorption and fluorescence titrations

For UV-vis absorption and fluorescence titrations, a stock solution ( $10^{-3}$  M) of 4 was freshly prepared in DMSO. For each experiment, titrations were performed with solutions of 4 in H<sub>2</sub>O/DMSO (6/4, v/v), prepared by mixing 30 µL of stock solution with 1,200 µL of DMSO and 1.8 mL of distilled H<sub>2</sub>O. Stock solutions ( $10^{-1}$  to  $10^{-3}$  M) of metal ions (*i.e.*, Cu<sup>2+</sup>, Hg<sup>2+</sup>, Ni<sup>2+</sup>, Cd<sup>2+</sup>, Co<sup>2+</sup>, Pb<sup>2+</sup>, Zn<sup>2+</sup>, Fe<sup>3+</sup>, Al<sup>3+</sup>, Fe<sup>2+</sup>, Ba<sup>2+</sup>, Ag<sup>+</sup>, K<sup>+</sup>, Na<sup>+</sup>, and Li<sup>+</sup> perchlorate salts) and L-amino acids (*i.e.*, glycine, alanine, serine, cysteine, valine, leucine, proline, phenylalanine, tyrosine, tryptophan, aspartic acid, arginine, and histidine) were freshly prepared in water. In each titration experiment, 3 mL of a 5 µM solution of 4 was placed in a quartz cuvette (path length: 1 cm), and nitroaromatic compounds were added to the quartz cuvette via a micropipette.

#### **Calculation of the Stern–Volmer Constant**

The sensitivity of 4 toward various analytes was estimated from their Stern–Volmer constants,  $K_{sv}$ , using the equation  $I_o/I = 1 + K_{sv}$  [Q], where  $I_o$  and I are the fluorescence intensities in the absence and presence

of analytes, respectively. The Stern-Volmer plots were plotted as a function of analyte concentration, [Q]. The  $K_{sv}$  value was calculated from the slope of the Stern–Volmer plot.



Scheme S1. Synthesis of derivative 2.



Scheme S2. Synthesis of derivative 3.



Fig. S1 UV–vis absorption spectra of derivative 4 (5.0  $\mu$ M) in various H<sub>2</sub>O/DMSO mixtures (0–60% volume fraction of water in DMSO).



Fig. S2 Fluorescence spectra of derivative 4 (5.0  $\mu$ M) in various H<sub>2</sub>O/DMSO mixtures (0–60% volume fraction of water in DMSO).



Fig. S3 Images of the vials showing the change in intensity of derivative 4 (5.0  $\mu$ M) in various H<sub>2</sub>O/DMSO mixtures (0–60% volume fraction of water in DMSO) under UV irradiation at 365 nm.

**Table S1:** Fluorescence lifetime of in moving from DMSO to 60 % H2O in DMSO measured at 450 nm, $\lambda_{ex}$ = 375 nm.  $\tau_{avg}$ : average lifetime of derivative 4.

	DMSO	60% H <sub>2</sub> O in DMSO
A <sub>1</sub>	0.25	1.00
$ au_1$	0.06 ns	1.98 ns
A <sub>2</sub>	0.75	
τ <sub>2</sub>	1.85 ns	
$ au_{avg}$	1.40 ns	1.98 ns



Fig. S4 Exponential fluorescence decay of 4 in 100% DMSO and 60% water–DMSO; measured at 450 nm,  $\lambda_{ex}$ = 375 nm.



Fig. S5 Fluorescence spectra of fim of derivative 4.



Fig. S6 DLS studies of derivative 4 (5.0  $\mu$ M) in 60% water–DMSO with a size distribution in the range of 2,000 to 6,000 nm.



**Fig. S7** (a) Size analysis from Transmission electron microscopy (TEM) of derivative **4** (60% water in DMSO); Size distribution for (b) helical pitch (c) diameter.



Fig. S8 Overlay of <sup>1</sup>H NMR spectra of derivative 4 in a) [D<sub>6</sub>]DMSO and b) [D<sub>6</sub>]DMSO/D<sub>2</sub>O (9:1).



Fig. S9 HRTEM analysis of 4 (5.0  $\mu$ M) in 60% water–DMSO.



**Fig. S10** The optimized geometry of the monomer of derivative **4** using B3LYP/6-31G density functional theory calculations (*front view*).



Fig. S11 a) Calculated CD spectra of monomer; Overlay of b) calculated and c) experimental CD spectra of trimeric structure of 4.



Critical Aggregation Concentration (CAC)=0.2mM

**Fig. S12** Isothermal titration calorimetry (ITC) dilution experiment of derivative **4** in 60% water in DMSO at 25 °C.



**Fig. S13** UV-vis absorption spectra of derivative **4** in the presence of Fe<sup>2+</sup> ions (60 equiv.) in 60% water–DMSO.



Fig. S14 UV-vis absorption spectra of derivative 4 in the presence of  $Fe^{2+}$  ions (120 equiv.) in 60% water–DMSO.



**Fig. S15** TEM and AFM images of chiral supramolecular derivative **4** in a solution containing Fe<sup>2+</sup> ions: 60 equiv. (a,c) or 120 equiv. (b,d).



Fig. S16 a) Fluorescence spectra of derivative 4 in the presence of Fe<sup>2+</sup> ions (120 equiv.) in 60% water– DMSO ( $\lambda_{ex} = 350$  nm); b) image of vial before and after the addition of Fe<sup>2+</sup> ions (120 equiv.) under UV irradiation at 365 nm.



Fig. S17 Exponential fluorescence decay of derivative 4 in 60% water–DMSO after the addition of Fe<sup>2+</sup> ions; measured at 450 nm,  $\lambda_{ex}$ = 375 nm.

**Table S2.** Change in fluorescence lifetime of derivative **4** in 60 % H<sub>2</sub>O in DMSO after the addition of Fe<sup>2+</sup> ions, measured at 450 nm,  $\lambda_{ex}$ = 375 nm.  $\tau_{avg}$ : average lifetime.

	60% H <sub>2</sub> O in DMSO	Fe <sup>2+</sup> ions (120 equiv)
<i>A</i> <sub>1</sub>	1.00	0.41
τ <sub>1</sub>	1.98 ns	0.18 ns
<i>A</i> <sub>2</sub>		0.59
τ <sub>2</sub>		1.92 ns
τ <sub>avg</sub>	1.98 ns	1.21 ns



Fig. S18 DLS studies of derivative 4 (5.0  $\mu$ M) in the presence of Fe<sup>2+</sup> ions (120 equiv.) in 60% water– DMSO with a size distribution in the range of 20 to 100 nm.



Fig. S19 Fluorescence response of the chiral supramolecular assembly of derivative 4 to various metal ions (50  $\mu$ M): (1) Chiral supramolecular derivative 4, (2) Fe<sup>2+</sup>, (3) Co<sup>2+</sup>, (4) Fe<sup>3+</sup>, (5) Ba<sup>2+</sup>, (6) Cu<sup>2+</sup>, (7) Nd<sup>3+</sup>, (8) Pb<sup>2+</sup>, (9) Ni<sup>2+</sup>, (10) Al<sup>3+</sup>, (11) Cd<sup>2+</sup>, (12) Zn<sup>2+</sup>, (13) Cs<sup>+</sup>, (14) Ag<sup>+</sup>, and (15) Rb<sup>+</sup>.



**Fig. S20** (a) Plot of fluorescence quenching efficiency of derivative 4 (5  $\mu$ M) in 60% water–DMSO as a function of Fe<sup>2+</sup> concentration. I<sub>0</sub> and I are the initial and final fluorescence intensity, respectively, at 450 nm in the absence and presence of different concentrations of Fe<sup>2+</sup>. (b) Regression curve at lower concentration.



Fig. S21 IR spectra of derivative 4 in the presence of  $Fe^{2+}$  ions.

**Table S3.** Change in IR stretching frequency for derivative 4 and derivative  $4 + Fe^{2+}$  ions.

Derivative 4	3600 cm <sup>-1</sup>	1734 cm <sup>-1</sup>	1644 cm <sup>-1</sup>	1598 cm <sup>-1</sup>	1577cm <sup>-1</sup>
Derivative 4 +	Broadening	1723 cm <sup>-1</sup>	1636 cm <sup>-1</sup>	1552cm <sup>-1</sup>	1544cm <sup>-1</sup>
Fe <sup>2+</sup> ions					



**Fig. S22.** Overlay of <sup>1</sup>H NMR spectra of derivative 4 in a)  $[D_6]DMSO / D_2O.$  (9:1); b) Derivative 4+ Fe<sup>2+</sup>(2 equiv.);  $[D_6]DMSO / D_2O$ ; 9:1; c) Derivative 4+ Fe<sup>2+</sup>(8 equiv.);  $[D_6]DMSO / D_2O$ ; 9:1

**Table S4**. Change in chemical shift ( $\delta$ ) value of <sup>1</sup>H NMR spectra of derivative 4 in [D<sub>6</sub>]DMSO /D<sub>2</sub>O. (9:1) upon the addition of Fe<sup>2+</sup> ions (2 equiv.) and Fe<sup>2+</sup> ions (6 equiv.) (D<sub>2</sub>O).

	Derivative 4 (δ, ppm) [D <sub>6</sub> ]DMSO /D <sub>2</sub> O. (9:1)	Fe <sup>2+</sup> ions (2 equiv.); (δ, ppm)[D <sub>6</sub> ]DMSO /D <sub>2</sub> O. (9:1)	Fe <sup>2+</sup> ions (8 equiv.); (δ, ppm)[D <sub>6</sub> ]DMSO /D <sub>2</sub> O. (9:1)
-NH (x)	8.74	8.78	Disappeared

-NH (y)	6.45	6.48	6.50
Aromatic protons (p)	7.53	7.56	7.55
Aromatic protons (q)	7.35	7.41	7.37
Aromatic protons (r)	7.27	7.31	7.29
Aromatic protons (s)	7.15	7.19	7.17
Aromatic protons (t)	7.09	7.13	7.11
Aromatic protons (u)	7.04	7.08	7.06



**Fig. S23.** UV-vis absorption spectra of derivative **4** in the presence of L-proline (100 equiv.) in 60% volume fraction of water in DMSO.



**Fig. S24.** UV-vis absorption spectra of derivative **4** in the presence of D-proline (100 equiv.) in 60% volume fraction of water in DMSO.



Fig. S25. a) CD spectra; b) TEM image; c) AFM image of chiral supramolecular derivative 4 in the presence of L-proline.



Fig. S26. a) CD spectra; b) TEM image; c) AFM image of chiral supramolecular derivative 4 in the presence of D-proline.



**Fig. S27.** Fluorescence spectra of derivative **4** in the presence of L-proline (100 equiv.) in 60% volume fraction of water in DMSO.



**Fig. S28.** Fluorescence spectra of derivative **4** in the presence of D-proline (100 equiv.) in 60% volume fraction of water in DMSO.



**Fig.S29.** (a) Plot of fluorescence quenching efficiency of derivative 4 (5  $\mu$ M) in 60% water–DMSO as a function of L-proline concentration. I<sub>0</sub> and I are the initial and final fluorescence intensity, respectively, at 450 nm in absence and presence of different concentrations of L-proline. (b) Regression curve at lower concentration.



Fig. S30. (a) Plot of fluorescence quenching efficiency of derivative 4 (5  $\mu$ M) in 60% water–DMSO as a function of D-proline concentration. I<sub>0</sub> and I are the initial and final fluorescence intensity, respectively, at 437 nm in the absence and presence of different concentrations of D-proline. (b) Regression curve at lower concentration.



**Fig. S31.** Comparison of fluorescence quenching of derivative **4** in DMSO after the addition various amino acids: 1) D-Proline; 2) L-proline; 3) Ala; 4) Arg; 5) Val; 6) Cys; 7) Lue; 8) Gly; 9) Phe; 10) His;11) Ser; 12) His; 13) Tyr; and 14) Asp at a wavelength of 437 nm upon excitation at 370 nm.



Fig. S32. Exponential fluorescence decay of 4 in moving from DMSO to 60 % H<sub>2</sub>O in DMSO measured at 450 nm,  $\lambda_{ex}$ = 375 nm.

Table S5.	Change	in f	luorescence	lifetime	of o	derivative	<b>4</b> in	60 %	H <sub>2</sub> O	in	DMSO	after the	addition	n of
aliquots of	ℓL/D-pro	line	, measured	at 450 m	m, λ	$k_{ex} = 375 \text{ m}$	m. τ <sub>a</sub>	vg: ave	erage	life	time			

	60% H <sub>2</sub> O in DMSO	L-proline	D-proline
<i>A</i> <sub>1</sub>	1.00	0.74	0.83
$ au_1$	1.98 ns	0.13 ns	0.12 ns
A <sub>2</sub>		0.23	0.15
τ <sub>2</sub>		0.56 ns	0.59 ns
A <sub>3</sub>		0.03	0.02
τ <sub>3</sub>		1.99 ns	2.17 ns
$ au_{avg}$	1.98 ns	0.28 ns	0.23 ns



Fig. S33. DLS studies of derivative 4 (5.0  $\mu$ M) in the presence of L-proline (100 equiv.) in 60% volume fraction of water in DMSO with size distribution in the range of 200 to 1500 nm.



Fig. S34. DLS studies of derivative 4 (5.0  $\mu$ M) in the presence of D-proline (100 equiv.) in 60% volume fraction of water in DMSO with size distribution in the range of 150 to 1000 nm



Fig. S35. IR spectra of derivative 4 in the presence of L-proline.



Fig. S36. IR spectra of derivative 4 in the presence of D-proline.

Derivative 4	3600 cm <sup>-1</sup>	1734 cm <sup>-1</sup>	1644 cm <sup>-1</sup>	1598 cm <sup>-1</sup>	1577cm <sup>-1</sup>
Derivative 4 + L-proline	Broadening	1730, 1715 cm <sup>-1</sup>	1625 cm <sup>-1</sup>	1570cm <sup>-1</sup>	1540cm <sup>-1</sup>
Derivative 4 + D-proline	Broadening	1711 cm <sup>-1</sup>	1617 cm <sup>-1</sup>	1565cm <sup>-1</sup>	1545cm <sup>-1</sup>

**Table S6.** Change in IR stretching frequency for derivative 4 and derivative 4 + L-proline and D-proline.



**Fig. S37.** Overlay of <sup>1</sup>H NMR spectra of derivative **4** in a) [D<sub>6</sub>]DMSO/D<sub>2</sub>O. (9:1); b) Derivative **4**+ L-proline (2 equiv.); [D<sub>6</sub>]DMSO/D<sub>2</sub>O; 9:1; c) Derivative **4**+ D-proline (8 equiv.); [D<sub>6</sub>]DMSO/D<sub>2</sub>O; 9:1.

**Table S7.** Change in chemical shift ( $\delta$ ) value of <sup>1</sup>H NMR spectra of derivative **4** in a) [D<sub>6</sub>]DMSO/D<sub>2</sub>O. (9:1); b) Derivative **4**+ L-proline (2 equiv.); [D<sub>6</sub>]DMSO/D<sub>2</sub>O; 9:1; c) Derivative **4**+ D-proline (8 equiv.); [D<sub>6</sub>]DMSO/D<sub>2</sub>O; 9:1.

	Derivative 4 (δ1 , ppm) [D <sub>6</sub> ]DMSO /D <sub>2</sub> O. (9:1)	L-proline (8equiv); (δL, ppm)[D <sub>6</sub> ]DMSO /D <sub>2</sub> O. (9:1)	D-proline (8 equiv); (δD, ppm)[D <sub>6</sub> ]DM SO /D <sub>2</sub> O. (9:1)	Change occur In chemical Shift- L-proline (δ1- δL)	Change occur In chemical Shift- D-proline (δ1- δD)
-NH (x)	8.74	8.78	8.83	0.04	0.09
-NH (y)	6.45	6.50	6.51	0.05	0.06
-NH (proline)	-	8.32	8.37	-	-
Aromatic protons (p)	7.53	7.57	7.58	0.04	0.05
Aromatic protons (q)	7.35	7.38	7.41	003	0.06
Aromatic protons (r)	7.27	7.30	7.32	0.03	0.05
Aromatic protons (s)	7.15	7.18	7.20	0.03	0.05
Aromatic protons (t)	7.09	7.12	7.15	0.03	0.06
Aromatic protons (u)	7.04	7.08	7.10	0.04	0.06
New peak 1	-	7.64	766		
New peak 2	-	7.22	7.24		
New peak 3	-	6.95	7.00		



Fig. S38. <sup>1</sup>H NMR spectra of derivative 1 in CDCl<sub>3</sub>.



Fig. S39. <sup>1</sup>H NMR spectra of derivative 4 in CDCl<sub>3</sub>.



Fig. S40. Mass spectrum of derivative 4.



Fig. S41. <sup>1</sup>H NMR spectra of derivative 4 in [D<sub>6</sub>]DMSO.



Fig. S42. <sup>13</sup>C NMR spectra of derivative 4 in [D<sub>6</sub>]DMSO.



Fig. S43. Mass spectrum of derivative 4.



Fig. S44. Thermogravimetric analysis (TGA) thermogram of derivative 4.

# Table S8: Comparison Table

Sr. No.	Manuscript	Organic Molecule	Solvent system	Strategy	Transformation s (Application)	Enantioselec tive detection
1.	Present work	Amide and ester substituted Triphenylamine	Water/ DMSO 6/4 (Green Solvent)	Entropicall y stable supramolec ular helical assemblies by AIEE phenomeno n	Achiral /chiral induced disordering of supramolecular assemblies to lower assemblies along with inversion in mixed aqueous media (Green Solvent)	Yes L/ D-Proline K <sub>D</sub> /K <sub>L</sub> = 2.1
2.	ACS Nano 2019, 13, 7300–7309	diphenylalanine based molecule(FFmoc )	2% DMSO in H <sub>2</sub> O	Self- assembly to beta sheets	Metal induced structural transformation To both superhelix as well as lowered assemblies	-
3.	Macromolec ules 2021, 54, 15, 7291– 7297	C3-symmetric aza-polycyclic aromatic	CHCl <sub>3</sub> / MCH	Chiral Supramole cular polymeriza tion	Trimethylamine induced diassembly	-
4.	Chem. Eur. J. 2021, 27, 326 - 330	chiral bis(urea) amphiphile	Water and ACN	Solvent mediated Chiral inversions	-	-
5.	ACS Nano 2021, 15, 4956–4966	Chiral TPE Derivatives	Water/THF (99 %) mixture	AIE phenomeno n	Liquid crystals (soldier and sergeant principle)	
6.	J. Am. Chem. Soc. 2018, 140, 16275–1628 3	cholesterol- azopyridine conjugate molecule	(i)DMSO (ii)Butanol	Self- assembly	Metal and solvent (Xylene/ DMSO/Butanol ) dependent	-

					helicity inversion	
7.	Angew. Chem. 2019, 131, 6007 – 6011	Pyrene-Histidine	EtOH/H <sub>2</sub> O	Chiral supramolec ular assemblies	Zinc induced inversion	-
8	Chem. Eur. J. 2018, 24, 2826 – 2831	1,3,5- triphenylbenzene tricarboxamides	(MCH/DCE , 95/5)	Self- assembly to form fibrillar bundles	-	-
9.	Chem. Sci., 2019, 10, 4236–4245	three chiral imidazole side arms (ImR and ImS) at the 1,3,5- positions of a central benzene ring	ACN	-	Zinc induced propeller shaped assembly	-
10.	Org. Chem. Front., 2021 (10.1039/d1q o00756d)	dicyanostilbene Based conjugated molecule	MCH and CHCl <sub>3</sub>	Polymeric assembly	-	-
11.	Nat. Commun. (2020) 11:2311 10.1038/s414 67-020- 16127-6	C3 symmetric carboxylic acid	CHCl <sub>3</sub>		Amino alcohol induced chiral supramolecular polymer	Yes, Amino alcohols
12.	J. Am. Chem. Soc. 2019, 141, 18278–1828 5	benzene-1,3,5- tricarboxamide	МСН	Cooperativ e supramolec ular polymeriza tion	-	-
13.	J. Org. Chem. 2019, 84, 14587–1459 2	boronic acid- functionalized PBI	МеОН		Leucine induced chiral supramolecular polymer	Yes, leucine enantiomers ee; 93%
14.	Chem. Sci., 2016, 7, 6689–6694	Triphenylamine based derivative	МСН	sergeants and soldiers principle induced chiral	-	-

				propeller shaped assembly		
15.	<i>Chem.</i> <i>Commun.,</i> 2016, 52, 69076910	C <sub>3</sub> symmetric - trisamides	DHN/CHCl 3, 95/5,	cooperative supramolec ular polymeriza tion	-	-