Electronic Supplementary Information

A selective fluorescent sensor for Zn²⁺ based on aggregation-induced emission (AIE) activity and metal chelating ability of bis(2-pyridyl)diphenylethylene

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Experimental:

All commercially available starting materials, reagents, and solvents were used as supplied, unless otherwise stated. Reported yields are isolated yields. Purification of all final products was accomplished by silica gel flash column chromatography. Chloroform : methanol or hexane : ethyl acetate were used as elution solvents. Proton (¹H) and carbon (¹³C) NMR were collected on Bruker NMR spectrometers at 300 MHz or 400 MHz for ¹H and 75 MHz or 100 MHz for ¹³C. Chemical shifts (δ) are reported in parts-per million (ppm) relative to residual undeuterated solvent. Melting points were recorded using a capillary melting point apparatus and are uncorrected. High resolution mass spectra were obtained in positive ion mode using electron spray ionization (ESI) on a double-focusing magnetic sector mass spectrometer. UVvisible spectra were obtained using quartz cuvettes on a Varian Cary 100-Scan dual-beam spectrophotometer. Each measurement was done in duplicate and compared to solvent blank. Blank samples were prepared using HPLC grade acetonitrile. Fluorescence spectra were obtained in air at room temperature using a Horiba Jobin Yvon Fluoromax-4 spectrofluorimeter using 3 mL quartz cuvettes. Samples were prepared using HPLC grade solvents. X-Ray diffraction data were collected on a Nonius Kappa CCD diffractometer equipped with Mo K α radiation with λ = 0.71073 °A. Structures were solved by direct methods and data was refined by full-matrix least squares refinement on F² against all reflections.

2,2'-(2,2-Dibromoethene-1,1-diyl)dipyridine (3)

Di(2-pyridyl) ketone (368 mg, 2.00 mmol) was dissolved in chlorobenzene (50 mL). Carbon tetrabromide (1.33 g, 4.00 mmol) and PPh₃ (2.1 g, 8.00 mmol) were added and the reaction mixture was heated to reflux and maintained for 3 d. After this time the reaction mixture was allowed to cool to rt and insoluble material was removed by filtration. The filtrate was treated with 50 mL of 1 M aq. HCl. The aqueous layer was separated and basified with 1 M aq. NaOH until pH 12. The aqueous phase was extracted with CH₂Cl₂ (2×50 mL), and the combined organic fractions were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography using ethyl acetate/hexane 1:1 as eluent to yield **3** (482 mg, 71%) as off-white solid. Mp 161-163°C. ¹H NMR (300 MHz, CDCl₃) δ 7.15-7.19 (m, 2H), 7.54-7.56 (m, 2H), 7.65-7.70 (m, 2H), 8.55 (d, 2H, *J* = 5.9 Hz). ¹³C NMR (75 MHz, CDCl₃) δ 98.2, 125.5, 127.3, 139.2, 149.1, 152.2, 160.2. HRMS (ESI): calcd for C₁₂H₉N₂Br₂ [M+H]⁺, 338.9132; found, 338.9131.

2,2'-(2,2-Diphenylethene-1,1-diyl)dipyridine (4)

Compound **3** (340 mg, 1.00 mmol) was dissolved in 50 mL of dioxane : water (4 : 1). The flask was charged with Na_2CO_3 (690 mg, 5.00 mmol), $Pd(OAc)_2$ (28 mg, 0.12 mmol), PPh_3 (130 mg, 0.50 mmol) and phenylboronic acid (610 mg, 5.00 mmol). The reaction was heated to reflux under argon overnight. After cooling, the reaction mixture was diluted with water and extracted with ethyl acetate (3 × 50 mL) and the combined organic fractions were dried over anhydrous Na_2SO_4 , filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography using ethyl acetate/hexane 1:1 and 2:1 as eluent to yield **4** (204 mg, 61%) as a yellowish white solid. Mp 185-187°C.

¹H NMR (300 MHz, $CDCl_3$) δ 7.01-7.18 (m, 10H), 7.39-7.58 (m, 4H), 7.69-7.76 (m, 2H), 8.54 (d, 2H, *J* = 5.3 Hz). ¹³C NMR (75 MHz, $CDCl_3$) δ 123.9, 129.4, 129.9, 130.5, 131.3, 133.6, 134.7, 138.4, 145.1, 151.9, 163.9. HRMS (ESI): calcd for C₂₄H₁₉N₂ [M+H]⁺, 335.1548; found, 335.1543.

4·Zn(OAc)₂

To a solution of **4** (20 mg, 0.06 mmol) in methanol (4 ml), $Zn(OAc)_2$ (13 mg, 0.06 mmol) was added and the white precipitate formed was filtered off. The filtrate was allowed to slowly evaporate to give $4 \cdot Zn(OAc)_2$ (26 mg, 85%) as colorless crystals. Mp >200°C.

2,2'-(2,2-Bis(4-methoxyphenyl)ethene-1,1-diyl)dipyridine (5)

Using the procedure given for the preparation of **4**, **3** (340 mg, 1 mmol) and 4-methoxyphenylboronic acid (760 mg, 5.0 mmol) were coupled to give **5** (295 mg, 75%) as orange-yellow solid. Mp 152-153°C. ¹H NMR (400 MHz, $CDCl_3$) δ 3.75 (s, 6H), 6.65 (d, 4H, *J* = 8.1 Hz), 6.93-7.14 (m, 10H), 8.17 (d, 2H, *J* = 7.9 Hz). ¹³C NMR (100 MHz, acetone-d*6*) δ 55.6, 114.0, 114.2, 122.0, 127.8, 133.3, 136.1, 136.5, 136.6, 149.6, 160.1, 162.8. HRMS (ESI): calcd for C₂₆H₂₃N₂O₂ [M+H]⁺, 395.1760; found, 395.1759.

2,2'-(2,2-Bis(dibenzo[b,d]thiophen-4-yl)ethene-1,1-diyl)dipyridine (6)

Using the procedure given for the preparation of **4**, **3** (340 mg, 1 mmol) and dibenzothiophene-4-boronic acid (1.140 g, 5.0 mmol) were coupled to give **6** (366 mg, 67%) as yellow solid. Mp 177-179°C. ¹H NMR (400 MHz, $CDCI_3$) δ 7.26-7.33 (m, 4H), 7.43-7.53 (m, 6H), 7.65-7.70 (m, 6H), 8.02-8.06 (m, 4H), 8.53 (d, 2H, J = 5.3 Hz). ¹³C NMR (100 MHz, $CDCI_3$) δ 121.6, 122.5, 123.6, 125.2, 125.3, 127.2, 127.7, 129.5, 129.6, 133.0, 133.1, 133.2, 134.1, 136.8, 137.5, 140.9, 142.6, 150.4, 161.3. HRMS (ESI): calcd for C₃₆H₂₃N₂S₂ [M+H]⁺, 547.1303; found, 547.1303.

3,3'-(2,2-Dibromoethene-1,1-diyl)dipyridine (7)

Using the procedure given for the preparation of **3**, di(3-pyridyl) ketone¹ (368 mg, 2.00 mmol) was converted into **7** (381 mg, 56%) as pale yellow solid. Mp 140-142°C. ¹H NMR (300 MHz, acetone-d*6*) δ 7.44-7.48 (m, 2H), 7.82-7.86 (m, 2H), 8.62 (d, 2H, J = 5.1 Hz), 8.72 (s, 2H). ¹³C NMR (75 MHz, acetone-d*6*) δ 96.3, 126.2, 138.6, 139.5, 145.1, 151.9, 152.1. HRMS (ESI): calcd for C₁₂H₉N₂Br₂ [M+H]⁺, 338.9132; found, 338.9139.

4,4'-(2,2-Dibromoethene-1,1-diyl)dipyridine (8)

Using the procedure given for the preparation of **3**, di(4-pyridyl) ketone² (368 mg, 2.00 mmol) was converted into **8** (435 mg, 64%) as dark yellow solid. Mp 163-164°C. ¹H NMR (400 MHz, CDCl₃) 7.06 (d, 4H, J = 5.2 Hz), 8.46 (d, 4H, J = 5.2 Hz). ¹³C NMR (75 MHz, CDCl₃) δ 97.0, 125.8, 145.4, 150.1, 152.9. HRMS (ESI): calcd for C₁₂H₉N₂Br₂ [M+H]⁺, 338.9132; found, 338.9136.

1,1,2,2-Tetra(pyridin-3-yl)ethene (9)

Using the procedure given for the preparation of **4**, **7** (340 mg, 1 mmol) and 3-pyridineboronic acid (615 mg, 5.0 mmol) were coupled to give **9** (182 mg, 54%) as yellowish white solid. The crude product was purified by flash column chromatography using 100 % ethyl acetate, followed by chloroform/methanol 3:1 as eluent. Mp 189-191°C. ¹H NMR (400 MHz, methanol-d4) 7.32-7.35 (m, 4H), 7.60-7.63 (m, 4H), 8.30

(s, 4H), 8.38 (d, 4H, J = 4.5 Hz). ¹³C NMR (75 MHz, methanol-d4) δ 126.4, 140.4, 140.5, 141.8, 150.4, 153.4.HRMS (ESI): calcd for C₂₂H₁₇N₄ [M+H]⁺, 337.1453; found, 337.1457.

1,1,2,2-Tetra(pyridin-4-yl)ethene (10)

Using the procedure given for the preparation of **4**, **8** (340 mg, 1 mmol) and 4-pyridineboronic acid (615 mg, 5.0 mmol) were coupled to give **10** (229 mg, 68%) as off-white solid. The crude product was purified by flash column chromatography using 100 % ethyl acetate, followed by chloroform/methanol 3:1 as eluent. Mp 195-196°C. ¹H NMR (300 MHz, methanol-d4) 7.23 (d, 4H, J = 5.1 Hz), 8.47 (d, 4H, J = 5.1 Hz). ¹³C NMR (75 MHz, methanol-d4) δ 128.2, 142.9, 151.6, 151.8. HRMS (ESI): calcd for C₂₂H₁₇N₄ [M+H]⁺, 337.1453; found, 337.1458.



Figure S1. UV-vis absorption spectrum of 4 (CH₃CN, 25 μ M).



Figure S2. UV-vis absorption spectrum of 5 (CH₃CN, 50 μ M).



Figure S3. UV-vis absorption spectrum of 6 (CH₃CN, 50 μ M).



Figure S4. UV-vis absorption spectrum of 9 (CH₃CN, 50 μ M).



Figure S5. UV-vis absorption spectrum of 10 (CH₃CN, 50 μ M).



Figure S6. AIE profile of 5 in CH₃CN/H₂O mixtures. λ_{ex} = 331 nm, [5] = 10 μ M.



Figure S7. AIE profile of 6 in CH₃CN/H₂O mixtures. λ_{ex} = 337 nm, [6] = 10 μ M



Figure S8. Determination of limit of detection (LOD) of 4 for Zn(ClO₄)₂.



Figure S9. Comparison of the fluorescence spectrum of **4** in 9:1 H₂O:CH₃CN and 1 M aqueous HCl. λ_{ex} = 317 nm, [**4**] = 10 μ M.

Job plot

A Job plot was constructed to determine the binding stoichiometry between **4** and $Zn(ClO_4)_2$ using ¹H NMR titrations by monitoring the change in chemical shift ($\Delta\delta$) of the most downfield pyridine hydrogen as a function of **4**/Zn²⁺ mole fraction. Stock solutions (5 mM each) of **4** and $Zn(ClO_4)_2$ were prepared in $D_2O:CD_3CN$ (1:1). NMR samples were prepared with different mole fractions of **4** and $Zn(ClO_4)_2$ while maintaining the total concentration of ([**4**] + [$Zn(ClO_4)_2$]) for each sample at 5 mM.



Figure S10. ¹H NMR spectra of $\mathbf{4}$ + Zn(ClO₄)₂ at different relative mole fractions (x = mole fraction $\mathbf{4}$) in D₂O:CD₃CN (1:1). Total concentration of ([$\mathbf{4}$] + [Zn(ClO₄)₂]) = 5.0 mM in all spectra.



Figure S11. NMR Job plot of **4** with $Zn(ClO_4)_2$ in $D_2O:CD_3CN$ (1:1) showing maximum $\Delta\delta$ at 0.5 mole fraction **4** (1:1 binding stoichiometry).



Figure S12. Fluorescence spectra of **5** in the presence of various metal ions (2 equivalents). 9:1 $H_2O:CH_3CN$, $\lambda_{ex} = 331$ nm, [**5**] = 10 μ M.



Figure S13. Change in fluorescence intensity of **5** (I/I_o) at 427 nm as a function of added Zn²⁺ in 9:1 H₂O:CH₃CN. λ_{ex} = 331 nm, [**5**] = 10 μ M.



Figure S14. Emission of **5** in the presence of various metal ions and Zn^{2+} +metal ion combinations (normalized to emission in the presence of Zn^{2+} alone). 9:1 H₂O:CH₃CN, λ_{ex} = 331 nm, [**5**] = 10 μ M.



Figure S15. Fluorescence spectra of **6** in the presence of various metal ions (2 equivalents). 9:1 $H_2O:CH_3CN$, $\lambda_{ex} = 337$ nm, [**6**] = 10 μ M.



Figure S16. Emission of **6** in the presence of 2 equivalents of metal ions (normalized to emission in the presence of Zn^{2+}). 9:1 H₂O:CH₃CN, λ_{ex} = 337 nm, [**6**] = 10 μ M.



Figure S17. AIE profile of 9 in CH₃CN/H₂O mixtures. λ_{ex} = 305 nm, [9] = 10 μ M.



Figure S18. AIE profile of 10 in CH₃CN/H₂O mixtures. λ_{ex} = 290 nm, [10] = 10 μ M.



Figure S19. Fluorescence spectra of **9** in the presence of various metal ions (4 equivalents) in 9:1 $H_2O:CH_3CN$. $\lambda_{ex} = 305 \text{ nm}$, [**9**] = 10 μ M.



Figure S20. Emission of **9** in the presence of 4 equivalents of metal ions (normalized to emission in the presence of Zn^{2+}) in 9:1 H₂O:CH₃CN. λ_{ex} = 305 nm, [**9**] = 10 μ M.



Figure S21. Fluorescence spectra of **10** in the presence of various metal ions (4 equivalents) in 9:1 $H_2O:CH_3CN$. $\lambda_{ex} = 290$ nm, [**10**] = 10 μ M.



Figure S22. Emission of **10** in the presence of 4 equivalents of metal ions (normalized to emission in the presence of Zn^{2+}) in 9:1 H₂O:CH₃CN. λ_{ex} = 290 nm, [**10**] = 10 μ M.

Formula	$C_{28}H_{24}N_2O_4Zn$
FW	517.86
Crystal System	Monoclinic
Space group	P21/c
a/Å	8.5965(9)
b/Å	18.1619(18)
c/Å	15.3006(15)
α/°	90
β/°	90.404(4)
γ/°	90
V/Å ³	2388.8(4)
Z	4
D _{calc}	1.440
μ (mm ⁻¹)	1.066
Т/К	190(2)
No. of reflections	27531
No. of unique reflections	5451
No. of reflections with $I > 2\sigma(I)$	4688
No. of parameters	318
$R_1 [I > 2\sigma(I)]$	0.0285
wR ₂	0.0770
CCDC No.	1486438

 Table S1. Crystallographic data for 4·Zn(OAc)2.

Table S2. ORTEP Plot and selected bond lengths and angles in 4·Zn(OAc)₂ (hydrogens omitted).



Selected bond distances (Å) and angles (°):

Zn(01)-N(0003)	2.093	N(004)-Zn(01)-N(003)	88.60
Zn(01)-N(004)	2.088	N(004)-Zn(01)-O(002)	99.54
Zn(01)-O(002)	1.937	N(004)-Zn(01)-O(005)	128.93
Zn(01)-O(005)	2.032	N(003)-Zn(01)-O(002)	107.30
Zn(01)-O(006)	2.313	N(003)-Zn(01)-O(005)	92.40
C(00S)-O(005)	1.245	O(002)-Zn(01)-O(005)	128.32
C(00S)-O(006)	1.243	C(00G)-C(00K)-C(00P)-C(00C)	53.80
C(00V)-O(002)	1.266	C(00A)-C(009)-C(00P)-C(00C)	57.25
C(00V)-O(007)	1.221		

References:

- 1. B. S. Park, S. W. Lee, I. T. Kim, J. S. Tae and S. H. Lee, *Heteroatom Chemistry*, 2012, 23, 66-73.
- 2. *n*-Butyllithium (14.2 mL, 23 mmol, 1.6 M solution in THF) was added dropwise to a solution of 4-iodopyridine (3.89 g, 19 mmol) in diethyl ether (40 mL) at -78 °C and stirred for 15 min. A solution of methyl isonicotinate (2.6 g, 19 mmol) in diethyl ether (10 mL) was added dropwise to the reaction mixture at -78 °C and stirred for 4 h at the same temperature. The reaction was quenched with saturated aqueous NH₄Cl solution and extracted with ethyl acetate (3 × 50 mL) and the combined organic fractions were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography using ethyl acetate/hexane 1:1 and 2:1 as eluent to yield pure product (1.32 g, 38%) as a yellow solid.

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Acquisitio	n Time (sec)	2.0447	Comment	1H Suga	r/D2O DRX-400 1H_training	Sep 11 2014 Shim	update- May 28, 2014		
Date		24 Feb 2016 16:43	5.12		Date Stamp	24 Feb 2016 16:	43:12		
File Name		E:\OLD NMR\MG-I	II-87B 1H\1\pdata\1\1r		Frequency (MHz)	400.13	Nucleus	Ŧ	
Number o	f Transients	4	Origin	spect	Original Points Count	16384	Owner	root	
Points Co	unt	131072	Pulse Sequence	zg30	Receiver Gain	71.80	SW(cyclical) (Hz)	8012.82	
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Date		24 Feb 2016 16:58:0	08		Date Stamp	24 Feb 2016 16:5	8:08			
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Numbe	r of Transients	400	Origin	spect	Original Points Count	32768	Owner	root		
Points	Count	131072	Pulse Sequence	zgdc30	Receiver Gain	362.00	SW(cyclical) (Hz)	26246.72		
Tompo	t Taturo (dorneo C)	CHLOROFORM-d	Spectrum Offset (Hz)	7026.2886	Spectrum Type	STANDARD	Sweep Width (Hz)	26246.52		
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Date Stamp 21 Aug 2015 15:43:44 Frequency (MHz) 75.47 Nucleus Original Points Count 32788 Ommer Receiver Gain 2008.00 Swifeyufic Spectrum Type STANDARD Swifeyufic 0.95 0.96 0.97 0.96 0.97 Swifeyufic 0.96 0.86 0.96 0.70 0.75 Swifeyufic 0.86 0.86 0.91 Swifeyufic 0.70 0.75 0.96 0.95 0.70 0.75 0.60 0.60 0.70 0.45 0.60 0.65 0.30 0.45 0.25 0.35 0.30 0.45 0.30 0.35 0.25 0.30 0.35 0.35 0.35 0.20 0.30 0.30 0.30 0.30 0.25 0.35 0.35 0.35 0.35 0.30 0.35 0.35 0.35 0.35 0.35	i 1 lical) (Hz) 1 Midth (Hz) 1 action = 1		1.3 Avance-300 NS = Z	00	Date	27 Aug 2015 15:43:44
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Date	16 Sep 2015 1	11:02:08				Date Stamp	16 Sep 2015 11	:02:08				
-ile Name	E:\OLD NMR\	MG-195 11	H\1\pdata\1\1	-		Frequency (MHz)	400.13	Nucleus	Ħ			
lumber of Transients	4	Origin	-	spe	ot	Original Points Count	16384	Owner	root			
Points Count	131072	Pulse	e Sequence	zg3	0	Receiver Gain	35.90	SW(cyclical) (Hz)	8012.82			
solvent	CHLOROFOR	SM-d				Spectrum Offset (Hz)	3193.2092	Spectrum Type	STANDAF	D		
weep Width (Hz)	8012.76	Temp	perature (deg	rree C) 23.	160							
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quency (MHz) ainal Points Count	16 Sep 2015 1	1:25:36		File Name	E:\OLD NMR 3(00\MG-195 13C ok/2\pdata	\1\1r
ainal Points Count	75.47	Nucleus	13C	Number of Transients	400	Origin	spect
	32768	Owner	root	Points Count	131072	Pulse Sequence	zgdc30
ceiver Gain	46341.00	SW(cyclical) (Hz)	19960.08	Solvent	CHLOROFORM	P-M	
actrum Offset (Hz)	7747.7314	Spectrum Type	STANDARD	Sweep Width (Hz)	19959.93	Temperature (degree C)	24.560
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Date	02 Sep 2015 19:0	06:24		Date Stamp	02 Sep 2015 19	:06:24		
File Name	E:\OLD NMR\MG	-189 1H\1\pdata\1\1r		Frequency (MHz)	400.13	Nucleus	1H	
Number of Transients	4	Origin	spect	Original Points Count	16384	Owner	root	
Points Count	131072	Pulse Sequence	zg30	Receiver Gain	64.00	SW(cyclical) (Hz)	8012.82	
Solvent	METHANOL-d4	Spectrum Offset (Hz)	3193.2092	Spectrum Type	STANDARD	Sweep Width (Hz)	8012.76	
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And Control Cont Control Control <	requency (MHz)	300.13	Nucleus	H	Number of Transients	10	Origin	spect		
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