Electronic Supporting Information

Syntheses, Optical Properties, and Bioapplications of the Aggregation-Induced Emission of 2, 3, 4, 5-Tetraphenylcyclopenta-2, 4-dienyl Benzene Derivatives

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Synthesis of Intermediate Compounds

1-Chloro-8-iodooctane. 1-Chloro-8-iodooctane was prepared from the corresponding alcohol using a standard literature procedure.[23] Under an argon atmosphere, a solution of 8-chloro-1-octanol (6.83 g, 41.48 mmol) in anhydrous THF (204 mL) was cooled to 0 ^{o}C with an ice/water bath. Triphenylphosphine (13.07 g, 49.83 mmol), imidazole (8.48 g, 124.56 mmol) and iodine (11.59 g, 45.66 mmol) were added and the brownish reaction mixture was stirred at 0 ^{o}C for 30 min. The reaction mixture was warmed to room temperature and stirred at room temperature for 30 min. The brownish reaction mixture was poured into deionized water (134 mL) and extracted with Et_2O (2 × 149 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, hexanes) provided 1-chloro-8-iodooctane as a pale yellow liquid (9.20 g, 81 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 3.55 (t, J = 6.72 Hz, 2H), 3.20 (t, J = 7.02 Hz, 2H), 1.87-1.75 (m, 4H), 1.46-1.32 (m, 8H).

1-Chloro-10-iododecane. 1-Chloro-10-iododecane was prepared from the corresponding alcohol using a standard literature procedure.[24] Under an argon atmosphere, a solution of 10-chloro-1-decanol (1.61 g, 8.35 mmol) in anhydrous THF (41 mL) was cooled to 0 ^{o}C with an ice/water bath. Triphenylphosphine (2.26 g, 8.62 mmol), imidazole (1.70 g, 24.97 mmol) and iodine (2.33 g, 9.18 mmol) were added and the brownish reaction mixture was stirred at 0 ^{o}C for 30 min. The reaction mixture was warmed to room temperature and stirred at room temperature for 30 min. The brownish reaction mixture was poured into deionized water (27 mL) and extracted with Et_2O (2 × 30 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, hexanes) provided 1-chloro-10-iododecane as a colorless liquid (2.13 g, 84 %). ^{1}H

NMR (400 MHz, $CDCl_3$, 25 °C): δ = 3.55 (t, *J* = 6.76 Hz, 2H), 3.20 (t, *J* = 7.02 Hz, 2H), 1.87-1.74 (m, 4H), 1.45-1.31 (m, 12H).

1-(2-Iodoethoxy)-2-chloroethane. 1-(2-Iodoethoxy)-2-chloroethane was prepared from the corresponding alcohol using a standard literature procedure.[24] Under an argon atmosphere, a solution of 2-(2-chloroethoxy)ethanol (5.17 g, 41.50 mmol) in anhydrous THF (204 mL) was cooled to 0 °C with an ice/water bath. Triphenylphosphine (13.06 g, 49.80 mmol), imidazole (8.48 g, 124.56 mmol) and iodine (11.59 g, 45.67 mmol) were added and the brownish reaction mixture was stirred at 0 °C for 30 min. The reaction mixture was warmed to room temperature and stirred at room temperature for 30 min. The brownish reaction mixture was poured into deionized water (134 mL) and extracted with Et_2O (2 × 149 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/1 hexanes/dichloromethane) provided 1-(2-iodoethoxy)-2-chloroethane as a yellow liquid (3.99 g, 41 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ = 3.82-3.77 (m, 4H), 3.66 (t, *J* = 5.92 Hz, 2H), 3.28 (t, *J* = 6.80 Hz, 2H).

1-(2-(2-Iodoethoxy)ethoxy)-2-chloroethane. 1-(2-(2-Iodoethoxy)ethoxy)-2-chloroethane was prepared from the corresponding alcohol using a standard literature procedure.[24] Under an argon atmosphere, a solution of 2-[2-(2-chloroethoxy)-ethoxy]-ethanol (7.00 g, 41.51 mmol) in anhydrous THF (204 mL) was cooled to 0 °C with an ice/water bath. Triphenylphosphine (13.07 g, 49.83 mmol), imidazole (8.48 g, 124.56 mmol) and iodine (11.59 g, 45.66 mmol) were added and the brownish reaction mixture was stirred at 0 °C for 30 min. The reaction mixture was warmed to room temperature and stirred at room temperature for 30 min. The brownish reaction mixture was poured into deionized water (134 mL) and extracted with Et_2O (2 × 149 mL). The combined organic layers were dried over Na_2SO_4 and the solvent

was removed by rotary evaporation. Purification by column chromatography (silica gel, dichloromethane) provided 1-(2-(2-iodoethoxy)ethoxy)-2-chloroethane as a colorless liquid (5.31 g, 46 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 3.81-3.77 (m, 4H), 3.71-3.64 (m, 6H), 3.28 (t, *J* = 6.86 Hz, 2H).

1-(1-(3-Chloropropoxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (2a). Under an argon atmosphere, a solution of **1** (1.00 g, 2.16 mmol) in anhydrous THF (7.5 mL) was added to a suspension of NaH (0.10 g, 4.17 mmol) in anhydrous THF (5 mL) dropwise at room temperature. The yellow suspension was stirred at room temperature for 30 min. 1-Chloro-3-iodopropane (0.49 g, 2.40 mmol) was added dropwise and the yellow suspension was stirred at room temperature for 40 min. Tetrabutylammonium iodide (0.088 g, 0.24 mmol) was added and the yellow suspension was stirred at room temperature for 20.5 h. The solvent from the yellow-brown reaction mixture was removed by rotary evaporation. Saturated aqueous NaCl (13 mL) was added and the product was extracted with dichloromethane (3 × 13 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/1 dichloromethane/hexanes) provided **2a** as a yellow solid (0.41 g, 35 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.66-7.64 (m, 2H), 7.27-7.13 (m, 9H), 7.02-6.99 (m, 15H), 5.96-5.86 (m, 1H), 5.33 (dq, *J* = 17.23 and 1.77 Hz, 1H), 5.14 (dq, *J* = 10.47 and 1.59 Hz, 1H), 4.21 (dt, *J* = 5.24 and 1.56 Hz, 2H).

1-(1-(5-Chloropentyloxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (2b). Under an argon atmosphere, a solution of **1** (1.00 g, 2.16 mmol) in anhydrous THF (7.5 mL) was added to a suspension of NaH (0.10 g, 4.17 mmol) in anhydrous THF (5 mL) dropwise at room temperature. The yellow suspension was stirred at room temperature for 30 min. 1-Chloro-5-iodopentane (0.55 g, 2.37 mmol) was added dropwise and the yellow suspension was stirred at

room temperature for 3.5 h. Tetrabutylammonium iodide (0.088 g, 0.24 mmol) was added and the orange suspension was stirred at room temperature for 18.5 h. The solvent from the yellow-orange reaction mixture was removed by rotary evaporation. Saturated aqueous NaCl (13 mL) was added and the product was extracted with dichloromethane (3 × 13 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/2 dichloromethane/hexanes) provided **2b** as a yellow solid (0.56 g, 46 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.64 (d, *J* = 7.33 Hz, 2H), 7.24 (d, *J* = 8.33 Hz, 2H), 7.21-7.13 (m, 7H), 7.02-7.00 (m, 14H), 3.68 (t, *J* = 6.04 Hz, 2H), 3.37 (t, *J* = 6.76 Hz, 2H), 1.64-1.52 (m, 4H), 1.38-1.31 (m, 2H).

1-(1-(6-Chlorohexyloxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (2c). Under an argon atmosphere, a solution of **1** (0.60 g, 1.30 mmol) in anhydrous THF (4.5 mL) was added to a suspension of NaH (0.060 g, 2.50 mmol) in anhydrous THF (3 mL) dropwise at room temperature. The dark yellow suspension was stirred at room temperature for 30 min. 1-Chloro-6-iodohexane (0.35 g, 1.42 mmol) was added dropwise and the dark yellow suspension was stirred at room temperature for 7 h. Tetrabutylammonium iodide (0.053 g, 0.14 mmol) was then added and the orange suspension was stirred at room temperature for 17 h. The solvent from the yellow-orange suspension was removed by rotary evaporation. Saturated aqueous NaCl (8 mL) was added and the product was extracted with dichloromethane (3 × 8 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/1 dichloromethane/hexanes) provided **2c** as a yellow solid (0.44 g, 58 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.64 (d, *J* = 7.28 Hz, 2H), 7.25-7.15 (m, 9H), 7.03-6.99 (m, 14H), 3.67 (t, *J* = 6.10 Hz, 2H), 3.43 (t, *J* = 6.84 Hz, 2H), 1.68-1.58 (m, 4H), 1.26-1.22 (m, 4H).

1-(1-(8-Chlorooctyloxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (2d). Under an argon atmosphere, a solution of **1** (1.00 g, 2.16 mmol) in anhydrous THF (7.5 mL) was added to a suspension of NaH (0.10 g, 4.17 mmol) in anhydrous THF (5 mL) dropwise at room temperature. The yellow suspension was stirred at room temperature for 30 min. 1-Chloro-8-iodooctane (0.65 g, 2.37 mmol) was added dropwise and the yellow suspension was stirred at room temperature for 3.5 h. Tetrabutylammonium iodide (0.088 g, 0.24 mmol) was added and the orange suspension was stirred at room temperature for 18 h. The solvent from the yellow-orange reaction mixture was removed by rotary evaporation. Saturated aqueous NaCl (13 mL) was added and the product was extracted with dichloromethane (3 × 13 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/2 dichloromethane/hexanes) provided **2d** as a yellow solid (0.67 g, 51 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.64 (d, J = 7.29 Hz, 2H), 7.25-7.15 (m, 9H), 7.03-7.00 (m, 14H), 3.66 (t, J = 6.24 Hz, 2H), 3.52 (t, J = 6.76 Hz, 2H), 1.77-1.70 (m, 2H), 1.61-1.54 (m, 2H), 1.38-1.12 (m, 8H).

1-(1-(10-Chlorodecyloxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (2e). Under an argon atmosphere, a solution of **1** (0.60 g, 1.30 mmol) in anhydrous THF (4.5 mL) was added to a suspension of NaH (0.060 g, 2.50 mmol) in anhydrous THF (3 mL) dropwise at room temperature. The dark yellow suspension was stirred at room temperature for 30 min. 1-Chloro-10-iododecane (0.43 g, 1.42 mmol) was added dropwise and the dark yellow suspension was stirred at room temperature for 7 h. Tetrabutylammonium iodide (0.053 g, 0.14 mmol) was added and the orange suspension was stirred at room temperature for 17 h. The solvent from the yellow-orange suspension was removed by rotary evaporation. Saturated aqueous NaCl (8 mL) was added and the product was extracted with dichloromethane (3 × 8 mL). The combined

organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/1 dichloromethane/hexanes) provided **2e** as a yellow liquid (0.55 g, 67 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.64 (d, *J* = 7.28 Hz, 2H), 7.26-7.14 (m, 9H), 7.03-6.99 (m, 14H), 3.66 (t, *J* = 6.30 Hz, 2H), 3.54 (t, *J* = 6.76 Hz, 2H), 1.79-1.75 (m, 2H), 1.45-1.15 (m, 14H).

1-(1-(2-(2-Chloroethoxy)ethoxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (4a). Under an argon atmosphere, a solution of **1** (1.00 g, 2.16 mmol) in anhydrous THF (7.5 mL) was added to a suspension of NaH (0.10 g, 4.17 mmol) in anhydrous THF (5 mL) dropwise at room temperature. The yellow suspension was stirred at room temperature for 30 min. 1-(2-Iodoethoxy)-2-chloroethane (0.56 g, 2.39 mmol) was added dropwise and the yellow suspension was stirred at room temperature for 3.5 h. Tetrabutylammonium iodide (0.088 g, 0.24 mmol) was added and the orange suspension was stirred at room temperature for 18.5 h. The solvent from the yellow-orange reaction mixture was removed by rotary evaporation. Saturated aqueous NaCl (13 mL) was added and the product was extracted with dichloromethane (3 × 13 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 1/1 dichloromethane/hexanes) provided **4a** as a yellow solid (0.42 g, 34 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.63 (d, *J* = 7.28 Hz, 2H), 7.25-7.14 (m, 9H), 7.04-6.99 (m, 14H), 3.85 (t, *J* = 4.90 Hz, 2H), 3.68 (t, *J* = 4.88 Hz, 2H), 3.63 (t, *J* = 5.77 Hz, 2H), 3.51 (t, *J* = 5.85 Hz, 2H).

1-(1-(2-(2-(2-Chloroethoxy)ethoxy)ethoxy)-2,3,4,5-tetraphenylcyclopenta-2,4-dienyl)benzene (4b). Under an argon atmosphere, a solution of **1** (1.00 g, 2.16 mmol) in anhydrous THF (7.5 mL) was added to a suspension of NaH (0.10 g, 4.17 mmol) in anhydrous THF (5 mL) dropwise at room temperature. The yellow suspension was stirred at room

temperature for 30 min. 1-(2-(2-Iodoethoxy)ethoxy)-2-chloroethane (0.66 g, 2.37 mmol) was added dropwise and the yellow suspension was stirred at room temperature for 4.5 h. Tetrabutylammonium iodide (0.088 g, 0.24 mmol) was added and the orange suspension was stirred at room temperature for 17 h. The solvent from the yellow-orange reaction mixture was removed by rotary evaporation. Saturated aqueous NaCl (13 mL) was added and the product was extracted with dichloromethane (3 × 13 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was removed by rotary evaporation. Purification by column chromatography (silica gel, 4/1 dichloromethane/hexanes) provided **4b** as a yellow solid (0.55 g, 42 %). ^{1}H NMR (400 MHz, $CDCl_3$, 25 ^{o}C): δ = 7.63 (d, J = 7.24 Hz, 2H), 7.25-7.13 (m, 9H), 7.04-6.98 (m, 14H), 3.85 (t, J = 5.20 Hz, 2H), 3.75 (dt, J = 5.94 and 0.60 Hz, 2H), 3.66 (t, J = 5.18 Hz, 2H), 3.62 (dt, J = 5.95 and 0.56 Hz, 2H), 3.60-3.55 (m, 4H).

Tables

Table S1 UV-Vis absorbance and fluorescence studies of **3a**, **3c**, and **3e**.

Solvent	Dielectric Constant [a]	λ_{abs} / nm	ε [b]	λ_{em} / nm	% Φ_f / 23 °C [c]
		3a			
Toluene	2.4	367	7320	457	6.2
Acetone	21	359	12000	453	1.5
Ethanol	30	360	7150	451	1.6
Acetonitrile	37	356	4425	453	1.6
DMF	38	361	11300	458	2.8
DMSO	47	362	11643	458	6.0
H_2O	80	—[d]	—[d]	—[d]	—[d]
PBS	—[e]	359	1195	460	3.5
Tris-HCl	—[e]	—[d]	—[d]	—[d]	—[d]
		3c			
Toluene	2.4	360	8520	456	11.0
Acetone	21	360	8235	452	2.8
Ethanol	30	360	8105	451	1.0
Acetonitrile	37	349	4370	455	1.6
DMF	38	361	10985	458	3.1
DMSO	47	362	11120	462	5.9
H_2O	80	360	8440	453	5.5
PBS	—[e]	360	4415	455	10.0
Tris-HCl	—[e]	386	902	456	4.3
		3e			
Toluene	2.4	360	4445	456	9.5
Acetone	21	360	6660	456	1.8
Ethanol	30	360	5715	444	1.7
Acetonitrile	37	353	7220	453	1.6
DMF	38	362	10885	457	3.3
DMSO	47	363	11469	462	5.2
H_2O	80	360	1145	459	14.0
PBS	—[e]	359	1475	459	7.4
Tris-HCl	—[e]	386	1782	459	6.0

a. Based on refs 1 and 2.
b. Based on final absorbance of 0.1 units, given in $M^{-1}cm^{-1}$.
c. Quantum yield is relative to 1.54×10^{-5} M quinine in 0.05 M H_2SO_4 solution. λ_{ex} = 345 nm and λ_{em} = 452 nm.
d. No analysis was carried out as material was insoluble.
e. No dielectric constant values are reported.

Table S2 UV-Vis absorbance and fluorescence studies of **3b**, **5a, 3d**, and **5b.**

Solvent	Dielectric Constant [a]	λ_{abs} / nm	ε [b]	λ_{em} / nm	% Φ_f / 23 °C [c]
		3b			
Toluene	2.4	372	8500	457	24.0
Acetone	21	360	10080	451	2.4
Ethanol	30	362	10280	457	2.3
Acetonitrile	37	356	10400	454	1.5
DMF	38	361	11920	459	2.8
DMSO	47	363	12180	448	5.4
H_2O	80	367	8640	454	1.1
PBS	—[d]	357	4120	458	3.0
Tris-HCl	—[d]	—[e]	—[e]	—[e]	—[e]
		5a			
Toluene	2.4	364	4880	458	69.0
Acetone	21	360	13840	451	7.6
Ethanol	30	361	10920	460	2.3
Acetonitrile	37	358	11640	457	2.1
DMF	38	362	20060	460	2.8
DMSO	47	363	123200	455	4.9
H_2O	80	360	8000	457	1.2
PBS	—[d]	362	1940	454	2.4
Tris-HCl	—[d]	365	3380	449	0.4
		3d			
Toluene	2.4	361	8480	455	11.0
Acetone	21	351	9760	451	3.2
Ethanol	30	361	9200	460	2.2
Acetonitrile	37	347	10260	453	2.0
DMF	38	362	10500	460	2.9
DMSO	47	362	10840	454	5.1
H_2O	80	357	9100	457	2.2
PBS	—[d]	365	3200	464	2.5
Tris-HCl	—[d]	—[e]	—[e]	—[e]	—[e]
		5b			
Toluene	2.4	363	7140	458	52.0
Acetone	21	360	10040	450	6.9
Ethanol	30	361	9720	458	2.4
Acetonitrile	37	357	10060	454	2.4
DMF	38	361	11360	460	2.7
DMSO	47	363	12080	451	5.4
H_2O	80	362	9900	455	12.2
PBS	—[d]	371	6100	453	2.5
Tris-HCl	—[d]	365	2960	455	4.0

a. Based on refs 1 and 2.
b. Based on final absorbance of 0.1 units, given in $M^{-1}cm^{-1}$.
c. Quantum yield is relative to 1.54×10^{-5} M quinine in 0.05 M H_2SO_4 solution. λ_{ex} = 345 nm and λ_{em} = 452 nm.
d. No dielectric constant values are reported.
e. No analysis was carried out as material was insoluble.

Table S3 Effect of temperature on quantum yields for **3a**, **3c**, and **3e.**

Solvent	% Φ_f / 6 °C [a]	% Φ_f / 23 °C [a]	% Φ_f / 50 °C [a]
		3a	
Toluene	5.1	6.2	5.3
Acetone	0.6	1.5	0.6
Ethanol	0.6	1.6	0.6
Acetonitrile	0.7	1.6	0.6
DMF	2.7	2.8	2.3
DMSO	—[b]	6.0	5.7
H_2O	—[c]	—[c]	—[c]
PBS	2.4	3.5	3.4
Tris-HCl	—[c]	—[c]	—[c]
		3c	
Toluene	6.9	11.0	5.4
Acetone	0.7	1.7	0.8
Ethanol	0.9	1.0	0.9
Acetonitrile	0.6	1.6	0.6
DMF	3.1	3.1	2.5
DMSO	—[b]	5.9	5.3
H_2O	13.0	5.5	6.4
PBS	18.0	10.0	6.2
Tris-HCl	10.0	4.3	8.5
		3e	
Toluene	5.0	9.5	5.6
Acetone	0.8	1.8	0.8
Ethanol	0.6	1.7	0.6
Acetonitrile	0.6	1.6	0.7
DMF	2.9	3.3	2.5
DMSO	—[b]	5.2	4.3
H_2O	9.4	14.0	19.0
PBS	8.1	7.4	6.8
Tris-HCl	7.7	6.0	3.4

a. Quantum yield is relative to 1.54×10^{-5} M quinine in 0.05 M H_2SO_4 solution. λ_{ex} = 345 nm and λ_{em} = 452 nm.
b. DMSO freezes before reaching 6˚C.
c. No analysis was carried out as material was insoluble.

Figures

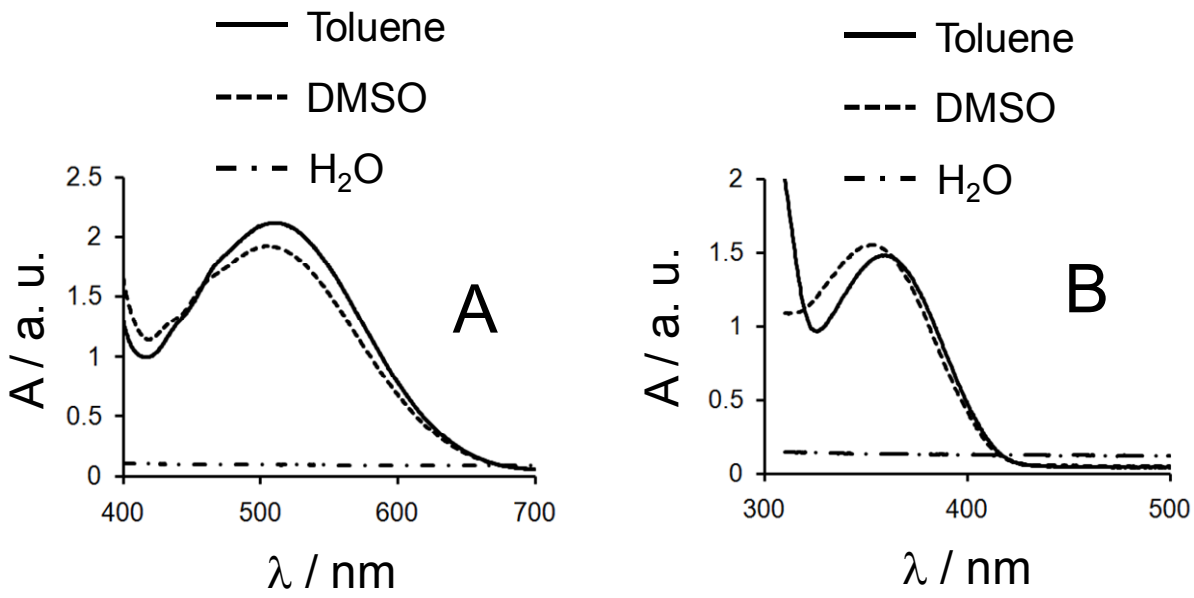

Fig. S1 (A) UV-Vis spectra of starting material compound **1,** and (B) intermediate compound **4a** under various solvents. Note that both compounds are highly insoluble in H_2O. Concentration for both compounds was ~ 2×10^{-4} M.

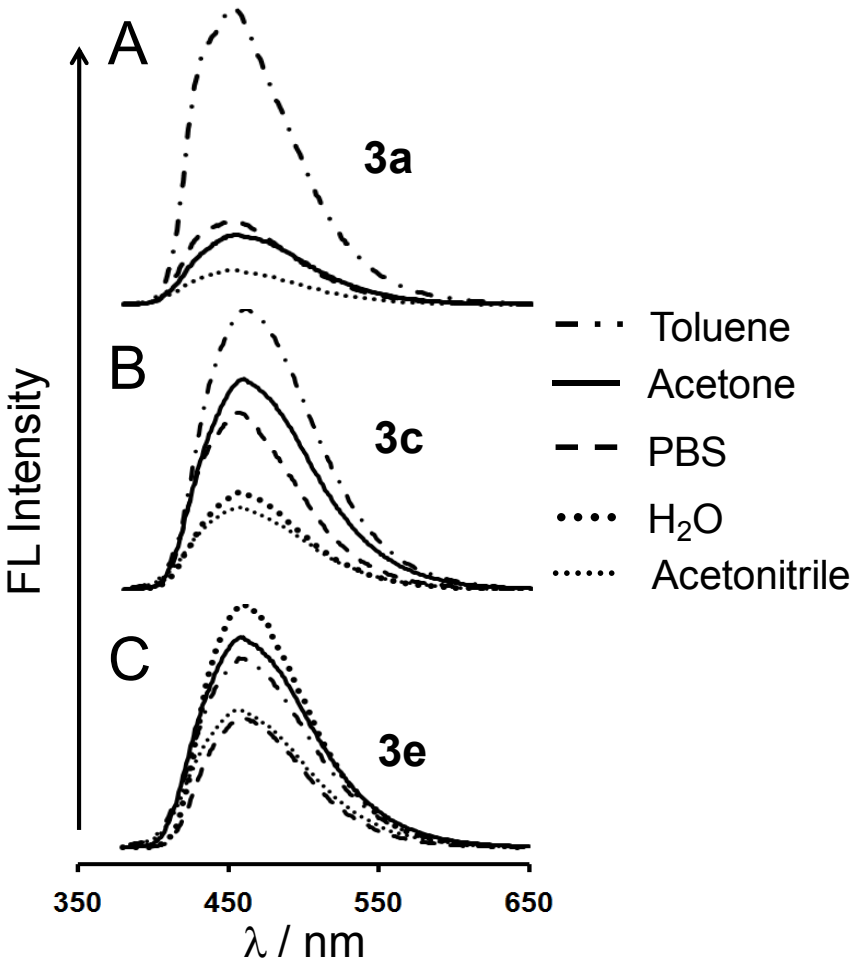

Fig. S2 Fluorescence spectra of **3a**, **3b**, and **3c** in various solvents. λ_{ex} = 360 nm.

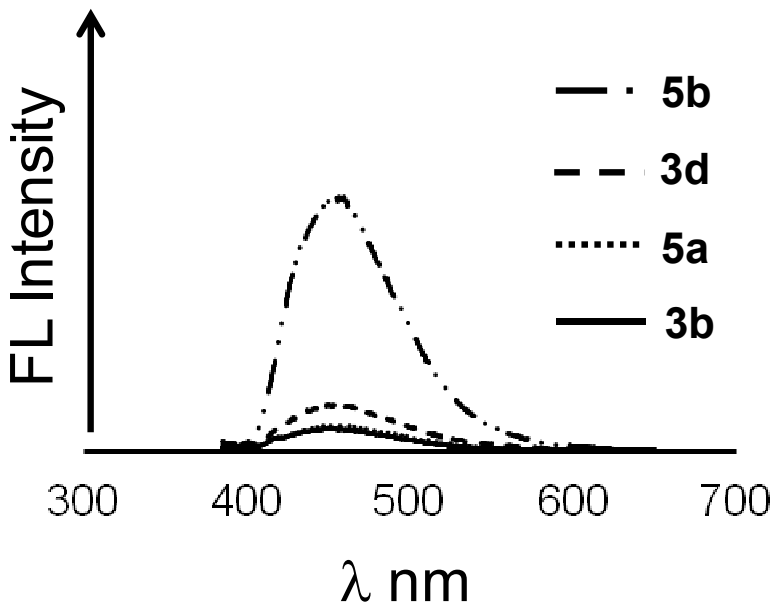

Fig. S3 Fluorescent dyes **3b, 3d, 5a**, and **5b** dissolved in H_2O, all with a concentration of 5.0×10^{-5} M. λ_{ex} = 360 nm. Note the higher fluorescence response for **5b**.

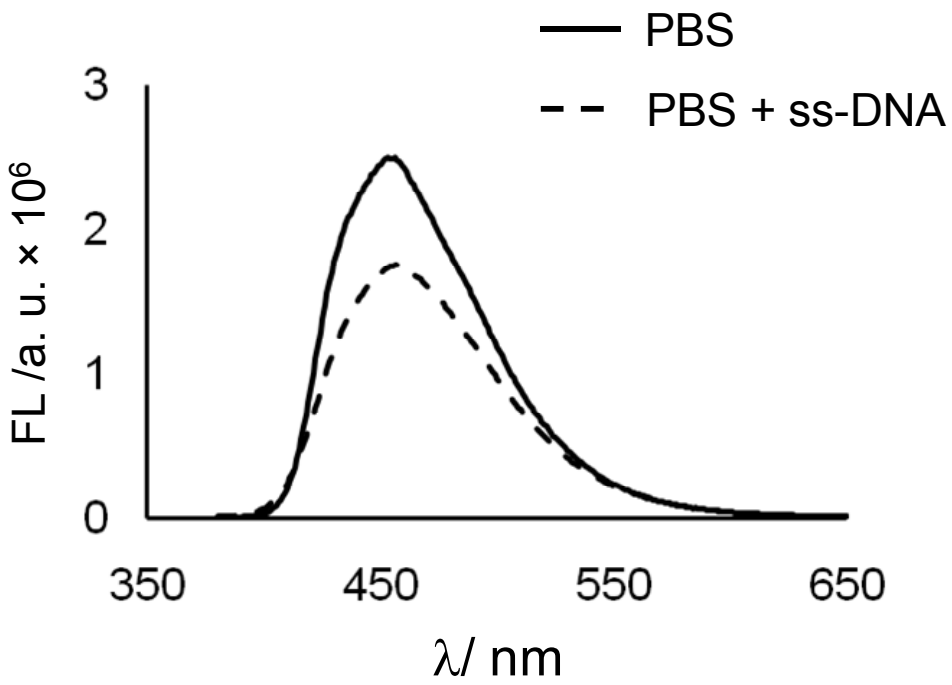

Fig. S4 Fluorescence spectra of **3c** (2.0×10^{-4} M) dissolved in phosphate buffer solution (10 mM pH 7.4) before and after addition of 1.0×10^{-6} M ss-DNA. λ_{ex} = 360 nm. The sequence of the capture probe (ss-DNA) was as follows: 5'-/5ThioMC6-D/AAA AAA AAA TCA CTC GCG GAT GCT GGC CGA-3'.

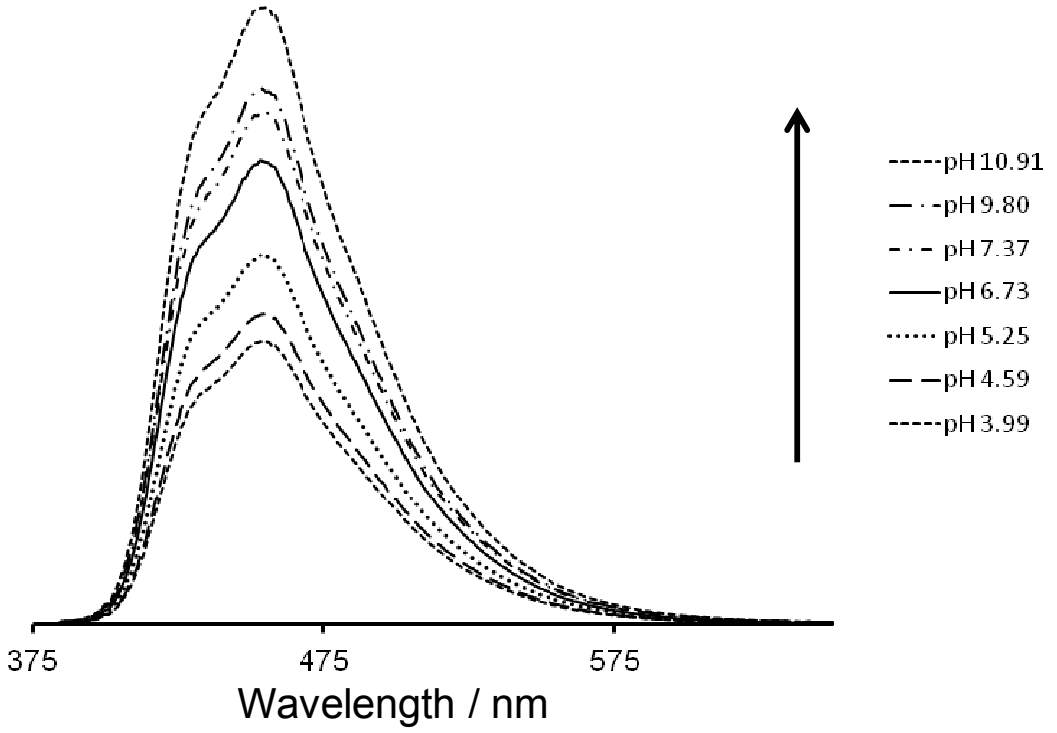

Fig. S5 Fluorescence spectra of **3c** (3.0×10^{-5} M) in Britton-Robinson buffers at different pH values. As the pH values increases, the fluorescence signal also increases. Each line is the average of 5 measurements. Fluorescence spectra were collected on a Tecan Safire2 microplate reader. The λ_{ex} = 360 nm.

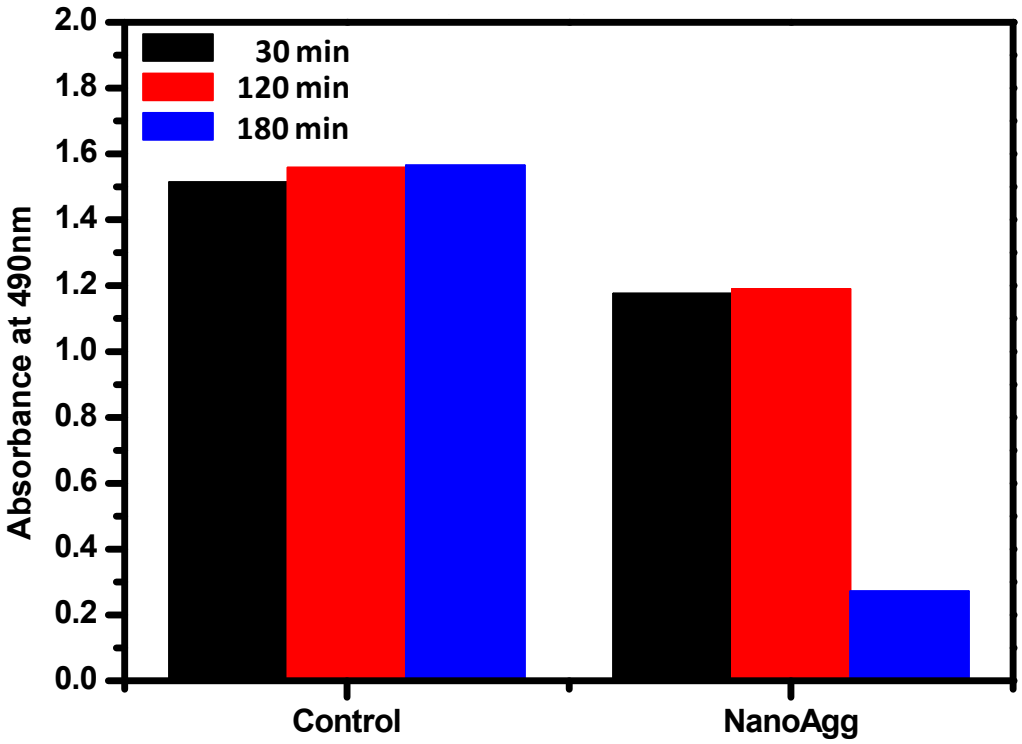

Fig. S6 Fluorescence response of **3c** (~1.0 × 10^{-3} M) dissolved in cell growth medium. Cell cytotoxicity studies were carried out via MTS cell proliferation assay. The λ_{ex} = 360 nm and the λ_{em} was 490 nm.

Acknowledgments

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References

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