Supplementary Data

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1. Experimental

1.1 General Methods:

All reagents were obtained from commercial suppliers and were used without further purification. CH_3CN was distilled over CaH_2. Analytical thin-layer chromatography was performed using silica gel 60 F254 plates (Merck). The ^1H and ^{13}C NMR spectra were recorded with Bruker AM 300 (300 MHz) spectrometers. Chemical shifts are expressed in ppm with residual $CHCl_3$ as reference. Low- and high-resolution mass spectra were recorded under electron ionization (EI) conditions. UV-vis spectra were recorded by using HP-8453 spectrophotometer with a diode array detector, and the resolution was set at 1 nm. Fluorescence spectra were recorded on a Cary Eclipse Fluorescene spectrophotometer.

1-bromo-2-(bromomethyl)naphthalene (1)

To a solution of 1-bromo-2-methylnaphthalene (2.01 g, 9.1 mmol) in dry acetonitrile was added *N*-bromosuccinimide (1.90 g, 10.7 mmol), AIBN (0.40 g, 2.4 mmol). The reaction mixture was stirred 2 hours under reflux, the mixture was concentrated, and then purified by silica column chromatography (Hexane) to give **1** (2.30 g, 85%) as a white solid; ^1H NMR ($CDCl_3$, 300 MHz) δ 8.31 (d, J = 8.7 Hz, 1H), 7.81−7.75 (m, 2H), 7.62−7.47 (m, 3H), 4.84 (s, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 134.9, 134.1, 132.5, 128.3, 128.1, 127.8, 127.7, 127.6, 127.2, 124.9, 34.7; HRMS (EI): Calcd for $C_{11}H_8Br_2$ (M^+), m/z 297.8993, found m/z 297.9001.

1,2-bis(2-((1-bromonaphthalen-2-yl)methoxy)ethoxy)ethane (2)

To a solution of triethylene glycol (0.2 mL, 1.5 mmol) in dry THF was added sodium

hydride (60%) (0.15 g, 3.8 mmol) at 0℃. The mixture was stirred for 10 mines, **1** (1.01 g, 3.4 mmol) was added and stirred for 4 hours at room temperature. Quench with 5 mL H_2O and then concentrated. The mixture was dissolved in CH_2Cl_2 and extracted, filtered, concentrated. The mixture was purified by silica column chromatography (hexane/EtOAc = 8∶1) to give **2** (0.57 g, 65%) as a yellow oil;

^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.29 (d, *J* = 8.4 Hz, 2H), 7.77−7.74 (m, 4H), 7.65−7.62 (m, 2H), 7.57−7.46 (m, 4H), 4.86 (s, 4H), 3.75−3.72 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 135.7, 133.7, 131.9, 127.9, 127.5, 127.1, 126.7, 126.2, 125.8, 122.1, 73.0, 70.6, 70.5, 69.9; HRMS (EI): Calcd for $C_{28}H_{28}Br_2O_4$ (M^+), m/z 586.0354, found m/z 586.0363.

fluorescent sensor (3)

To a solution of **2** (1.01 g, 1.7 mmol) in dry THF were added *n*-BuLi (1.6M) (2.3 mL, 3.7 mmol) at -78℃. The reaction mixture was stirred for 1 hour at -78℃, then $B(OMe)_3$ (1.0 mL, 8.8 mmol) was added. The resulting mixture was stirred overnight at room temperature. The reaction mixture was acidified with aq 10% HCl solution and extracted with Et_2O. Remove the organic solvent, the mixture was purified by silica column chromatography (hexane/EtOAc = 6∶1) to give **3** (0.59 g, 66%) as a yellow oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.85−7.81 (m, 6H), 7.52−7.46 (m, 6H), 4.73 (s, 4H), 3.72−3.69 (m, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 135.7, 133.2, 132.9, 128.0, 127.8, 127.6, 126.4, 125.9, 125.7, 73.2, 70.6 (2C), 69.3

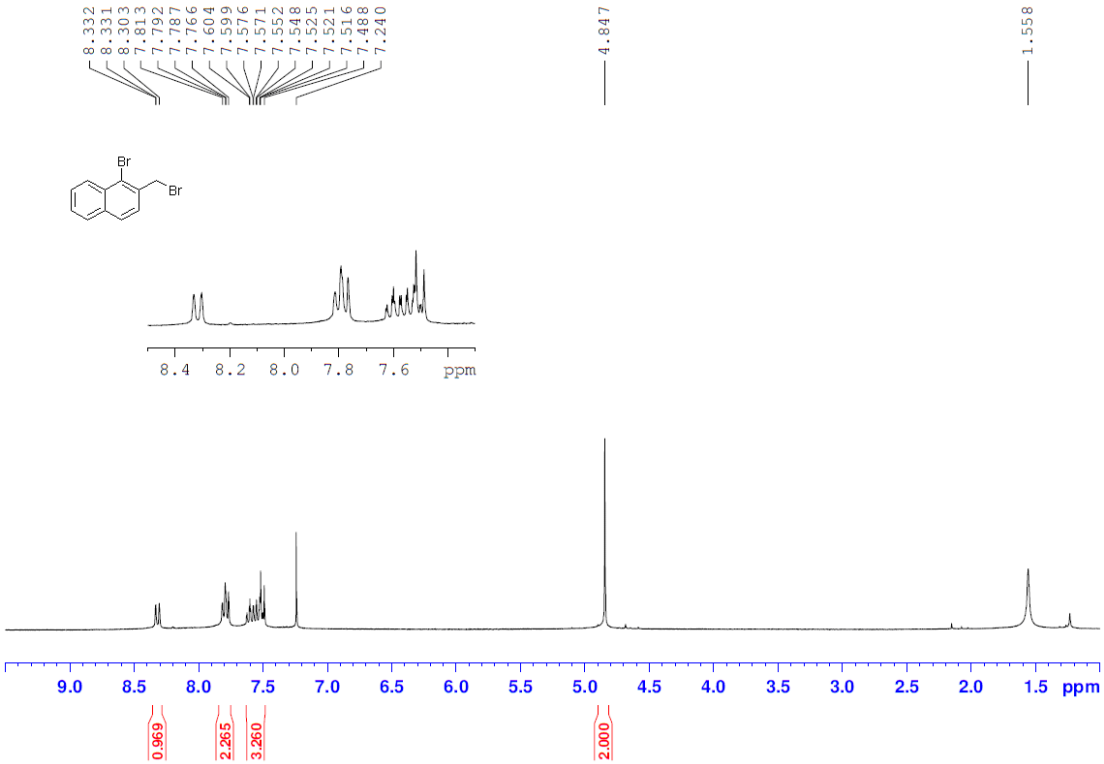

Figure S1 ^{1}H NMR (300 MHz) Spectrum of **1** in $CDCl_3$

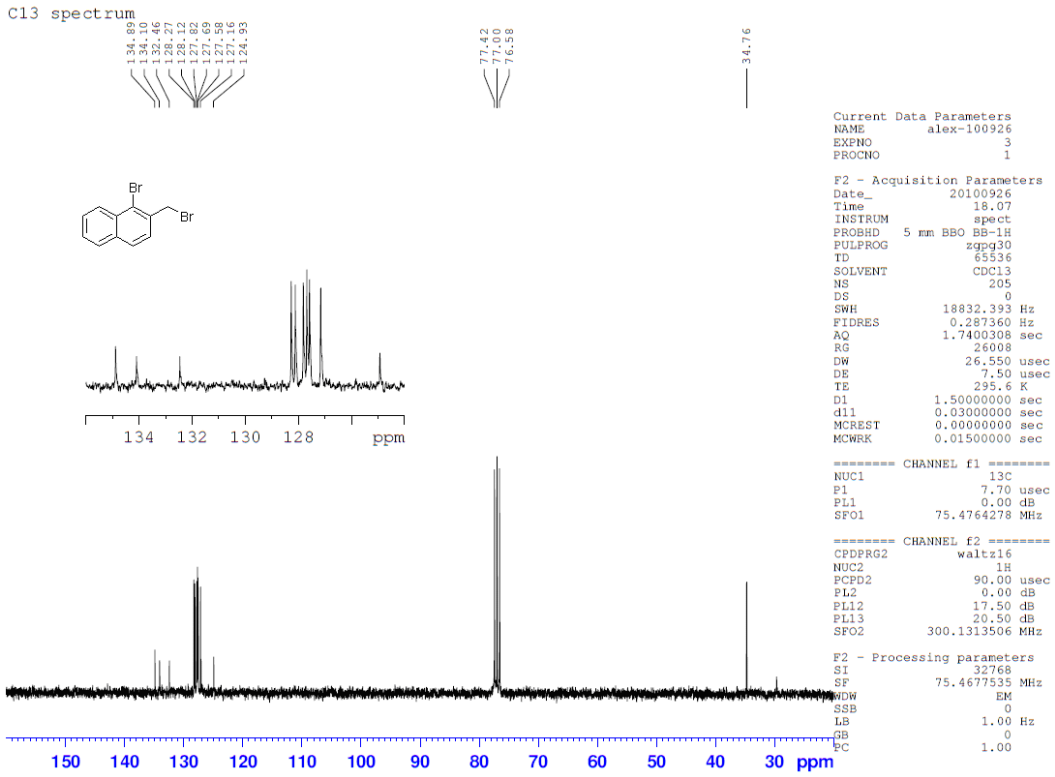

Figure S2 ^{13}C NMR (75.4 MHz) spectrum of **1** in $CDCl_3$

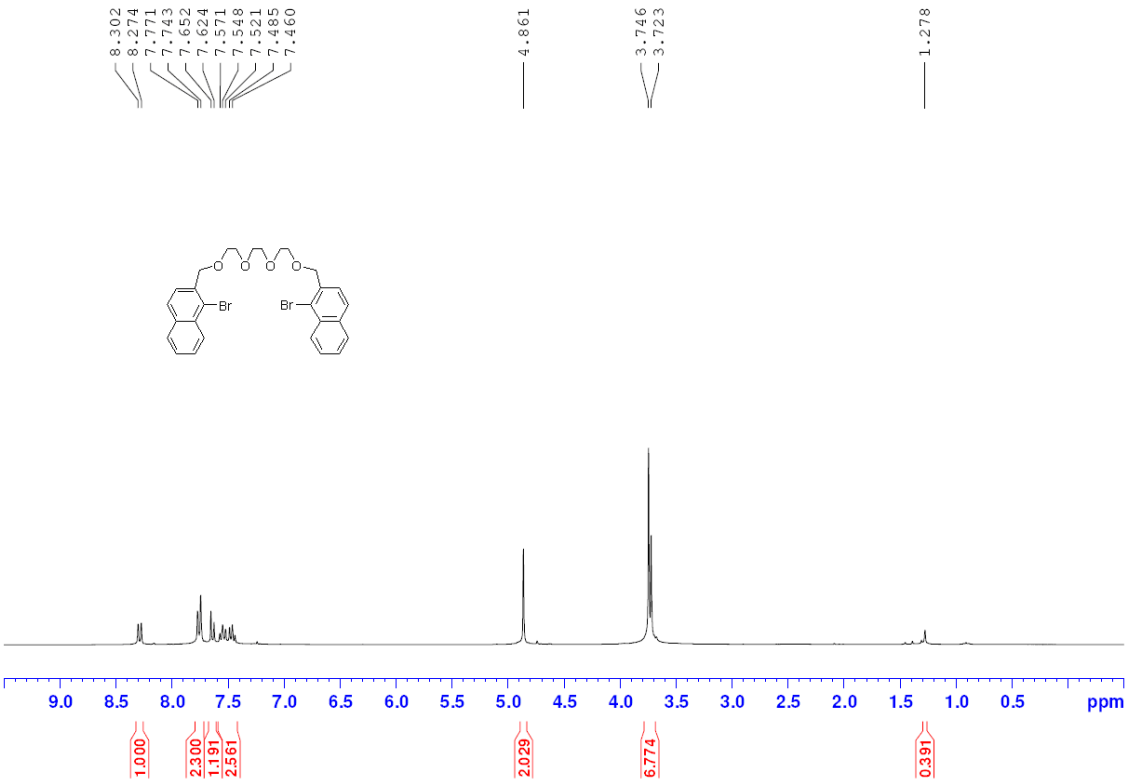

Figure S3 ^{1}H NMR (300 MHz) Spectrum of **2** in $CDCl_3$

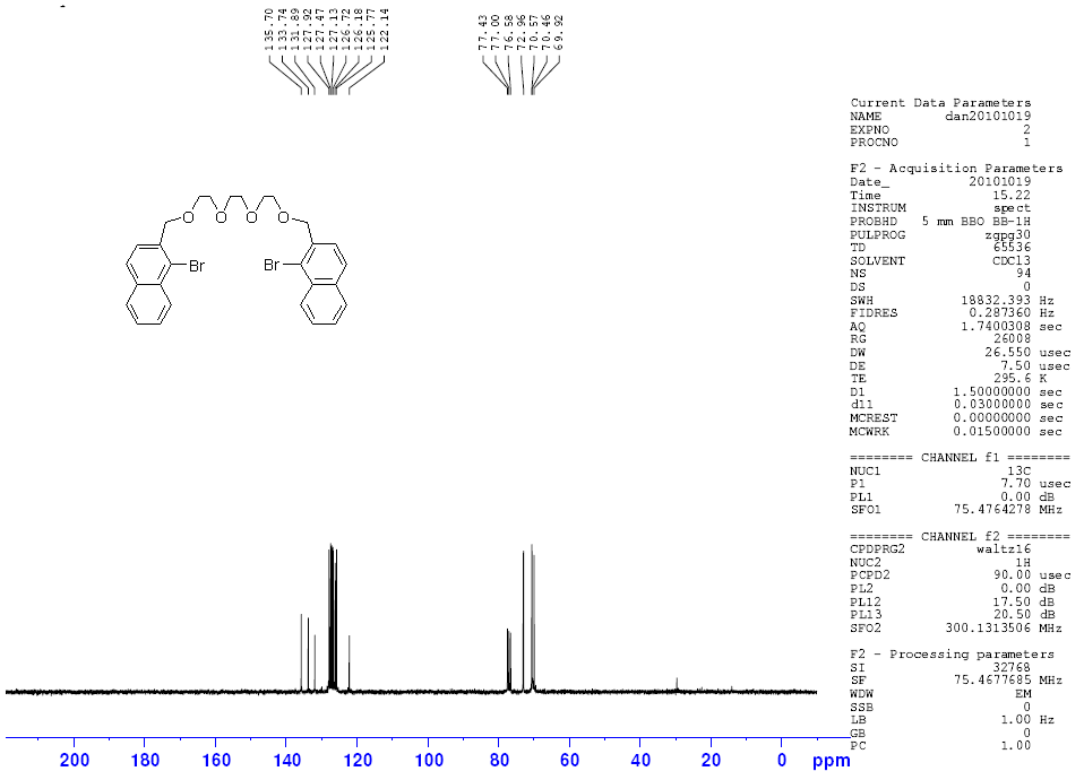

Figure S4 ^{13}C NMR (75.4 MHz) spectrum of **2** in $CDCl_3$

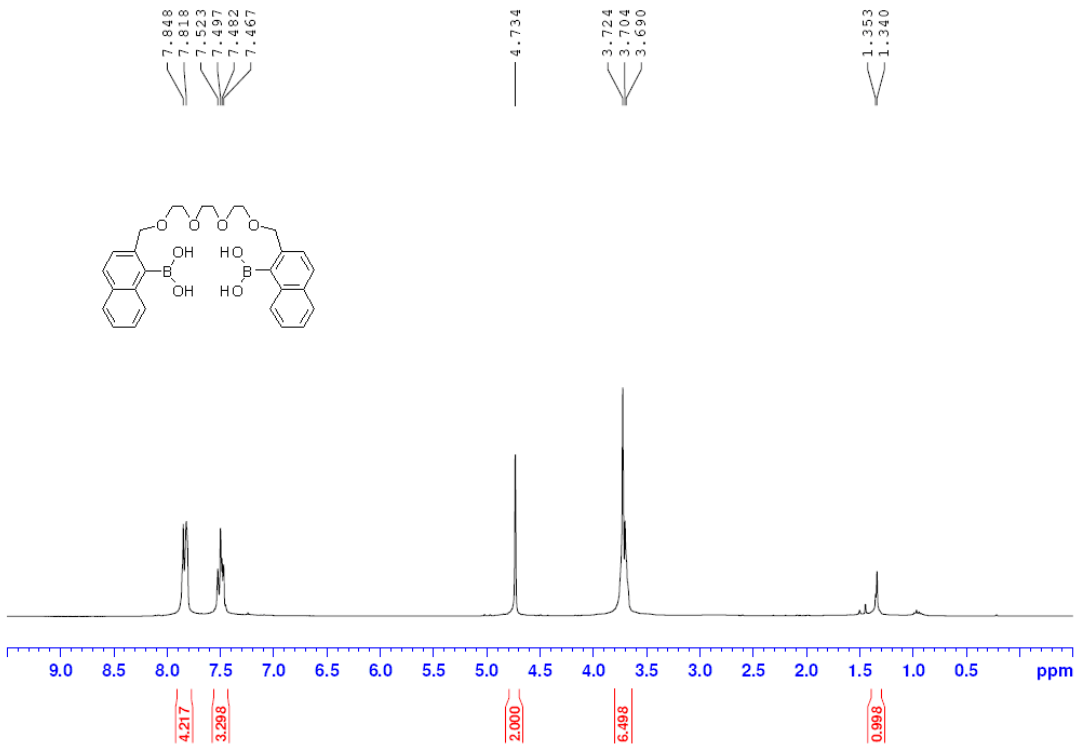

Figure S5 ^{1}H NMR (300 MHz) Spectrum of **3** in $CDCl_3$

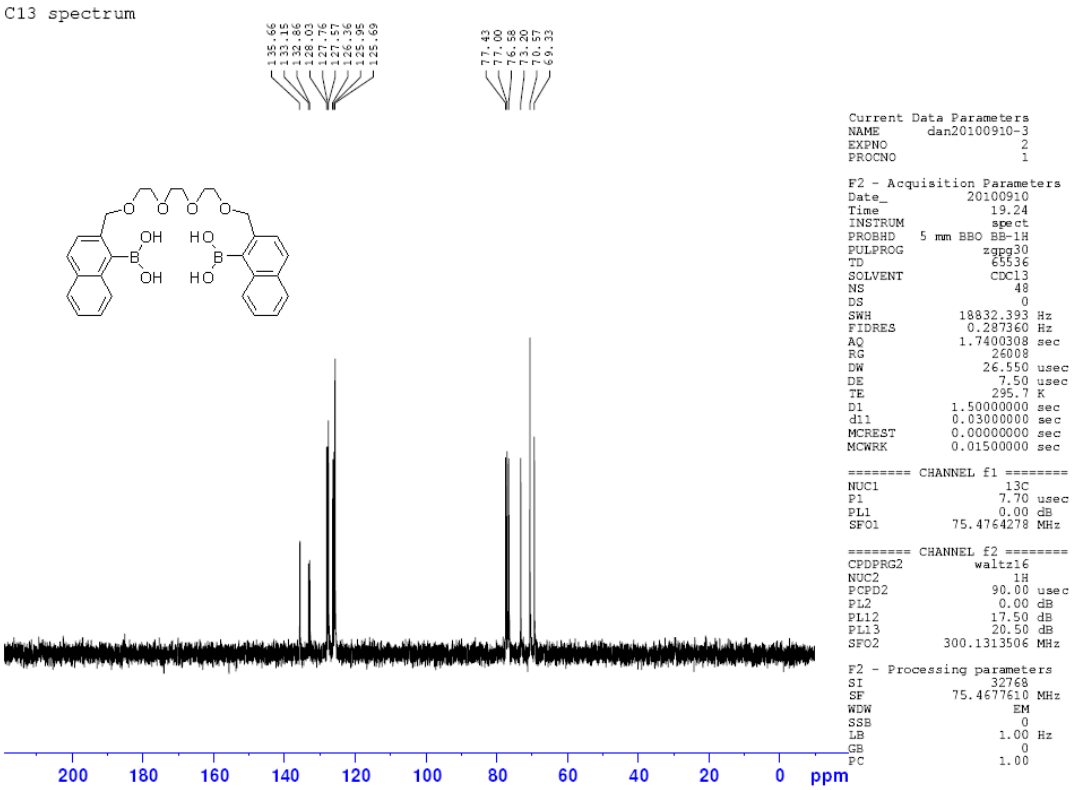

Figure S6 ^{13}C NMR (75.4 MHz) spectrum of **3** in $CDCl_3$

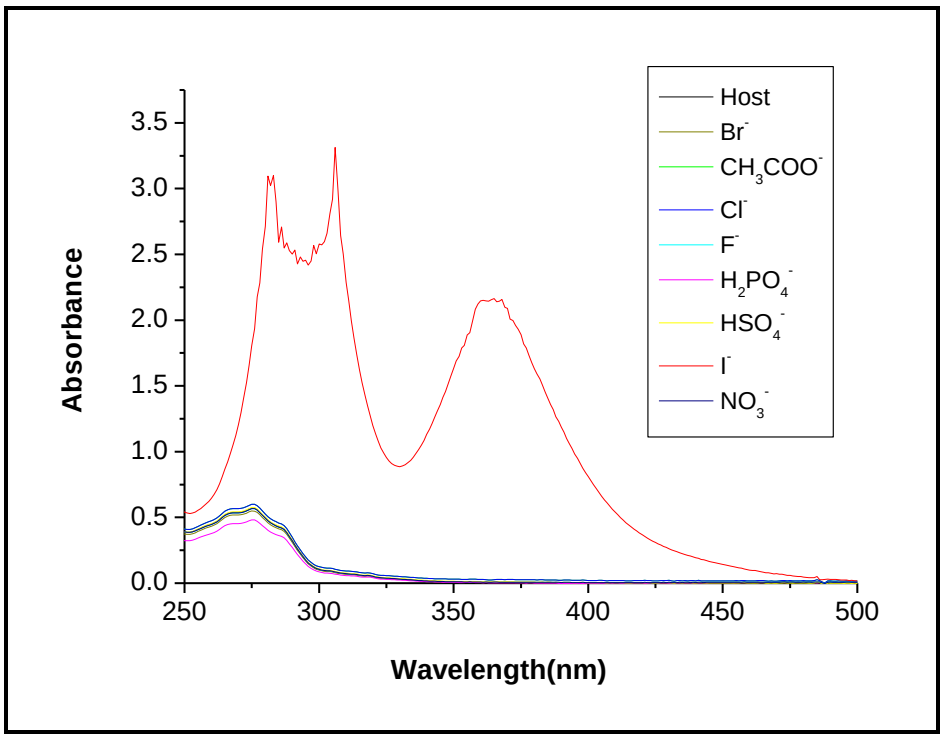

Figure S7 (a) UV-*vis* spectra of **3** (52 μM) in the presence of 10 equivalents of various anion in H_2O/THF (1/1, v/v)

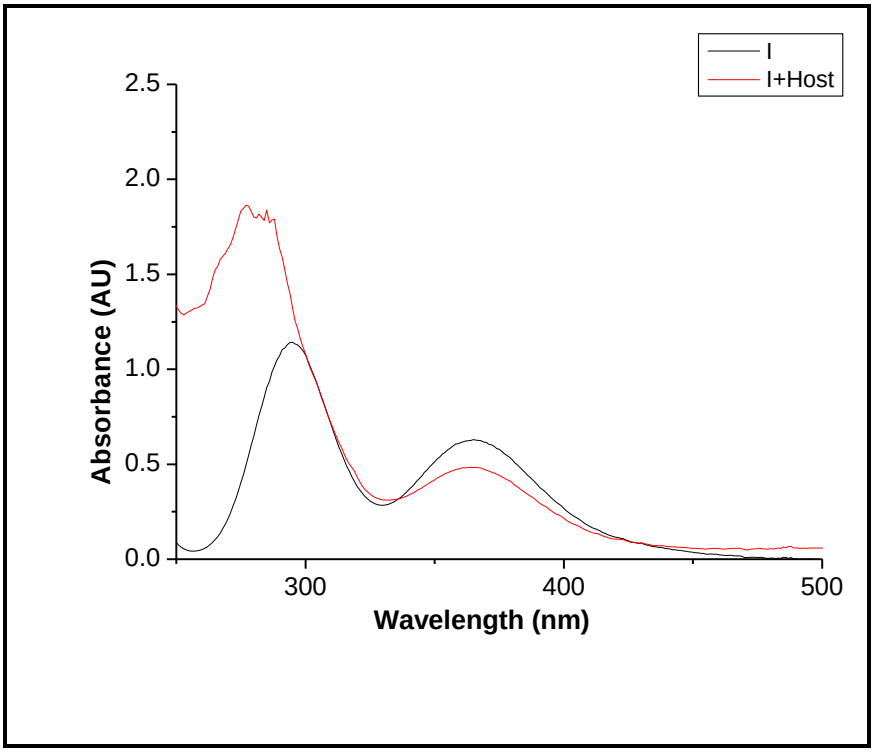

Figure S7 (b) UV-*vis* spectra of **3** (170 μM) in the presence of the same equivalents of iodide (170 μM) in H_2O/THF (1/1, v/v)

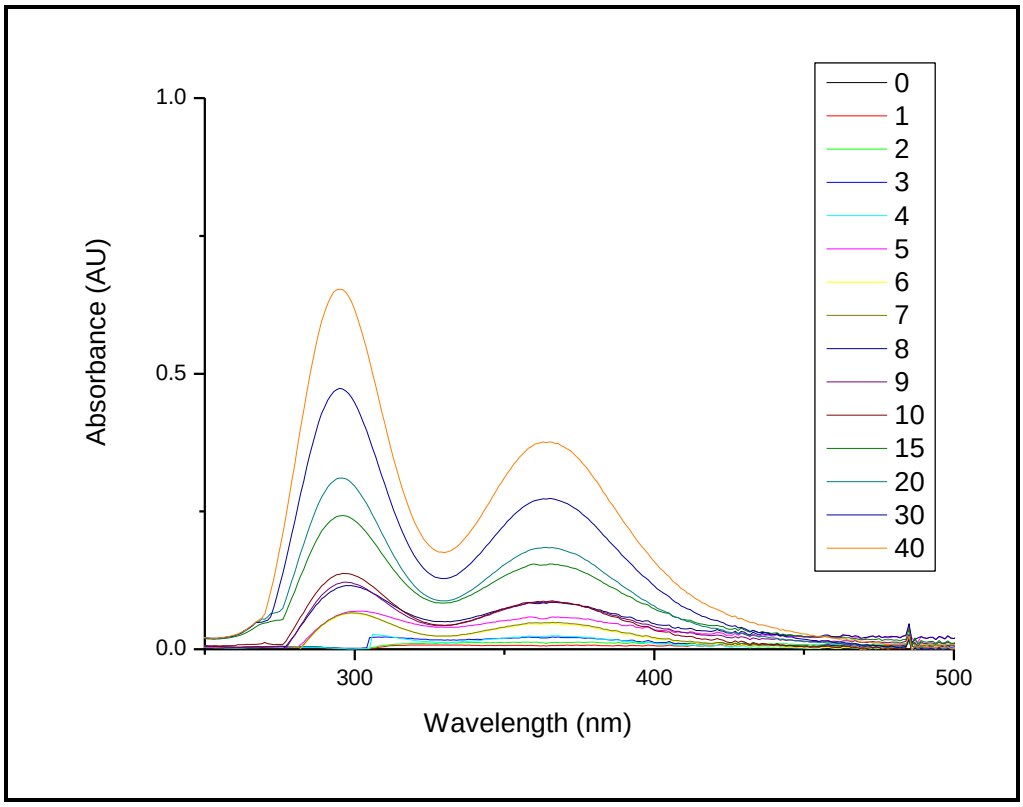

Figure S8 UV-*vis* spectra of **3** (13 μM) in H_2O/THF (1:1, v/v) upon addition of increasing concentrations of I(TBA).

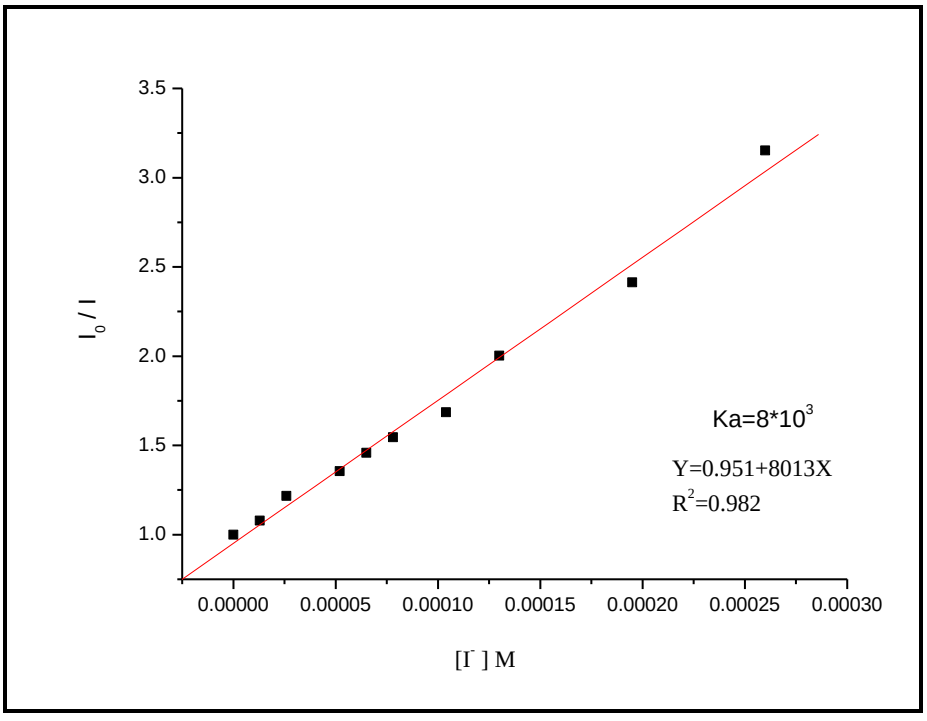

Figure S9 Stern-Volmer plot of **3** with $(n\text{-Bu})_4NI$ in H_2O/THF (1:1, v/v)

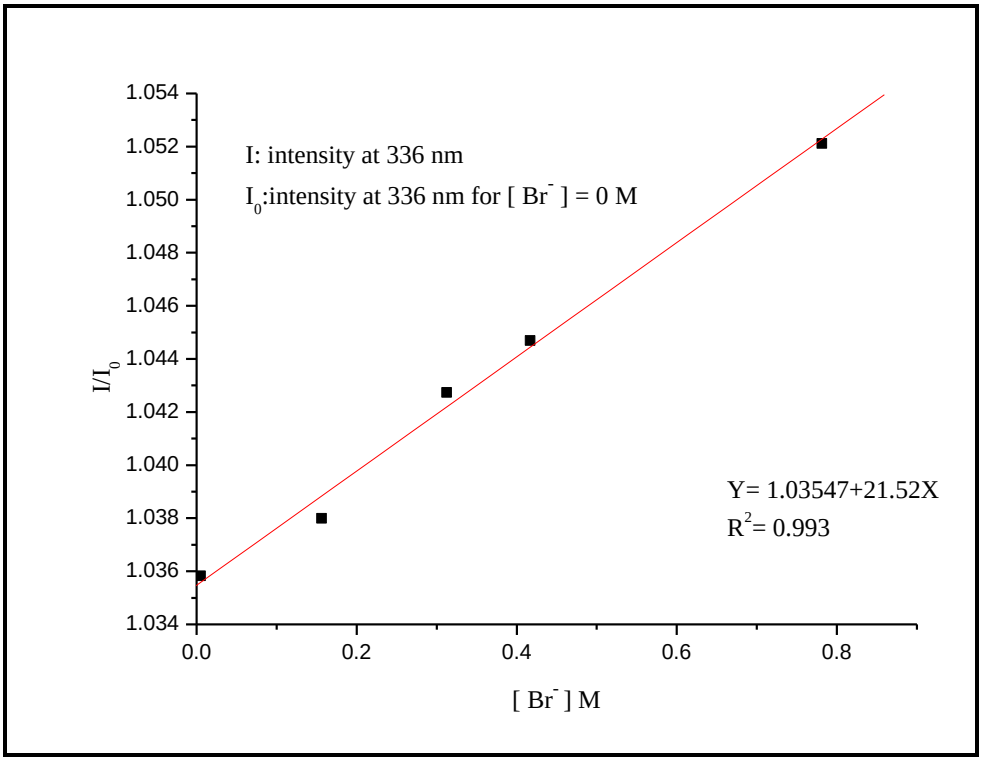

Figure S10 Stern-Volmer plot of **3** with (*n*-Bu)$_4$NBr in H_2O/THF (1:1, v/v)

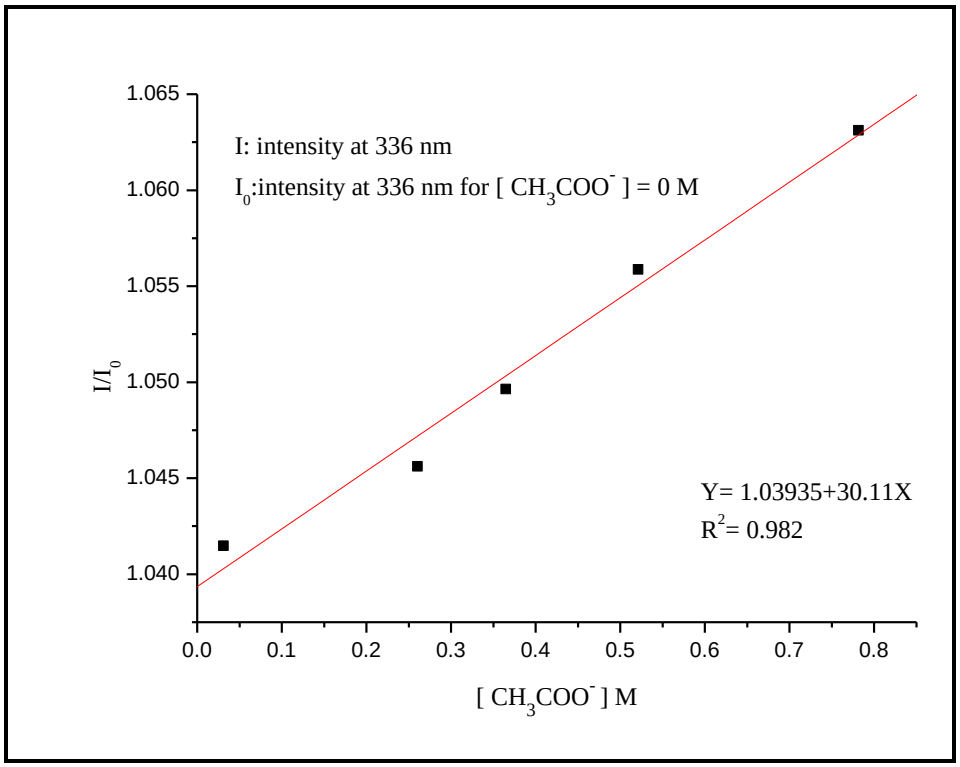

Figure S11 Stern-Volmer plot of **3** with (*n*-Bu)$_4$NOAc in H_2O/THF (1:1, v/v)

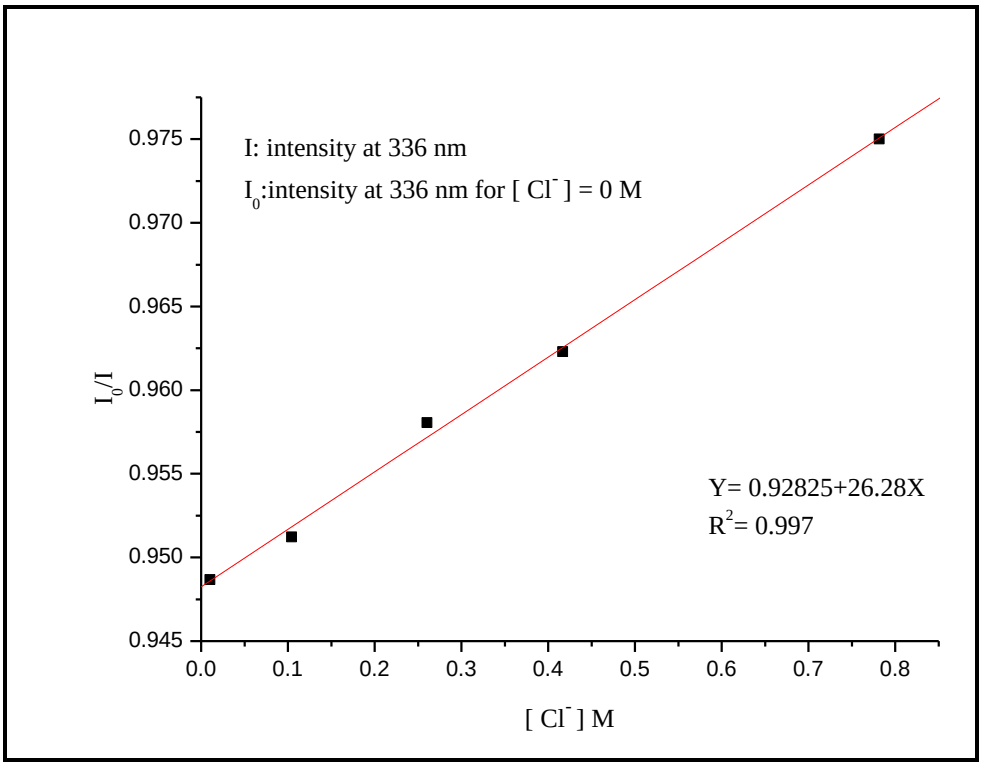

Figure S12 Stern-Volmer plot of **3** with (*n*-Bu)$_4$NCl in H_2O/THF (1:1, v/v)

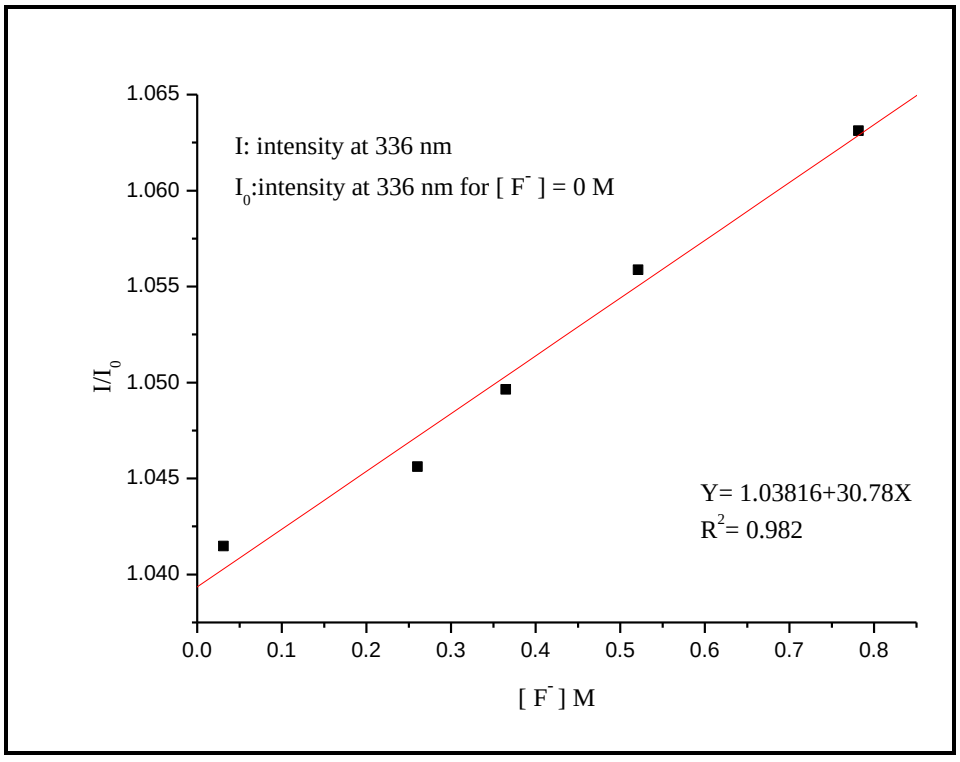

Figure S13 Stern-Volmer plot of **3** with (*n*-Bu)$_4$NF in H_2O/THF (1:1, v/v)

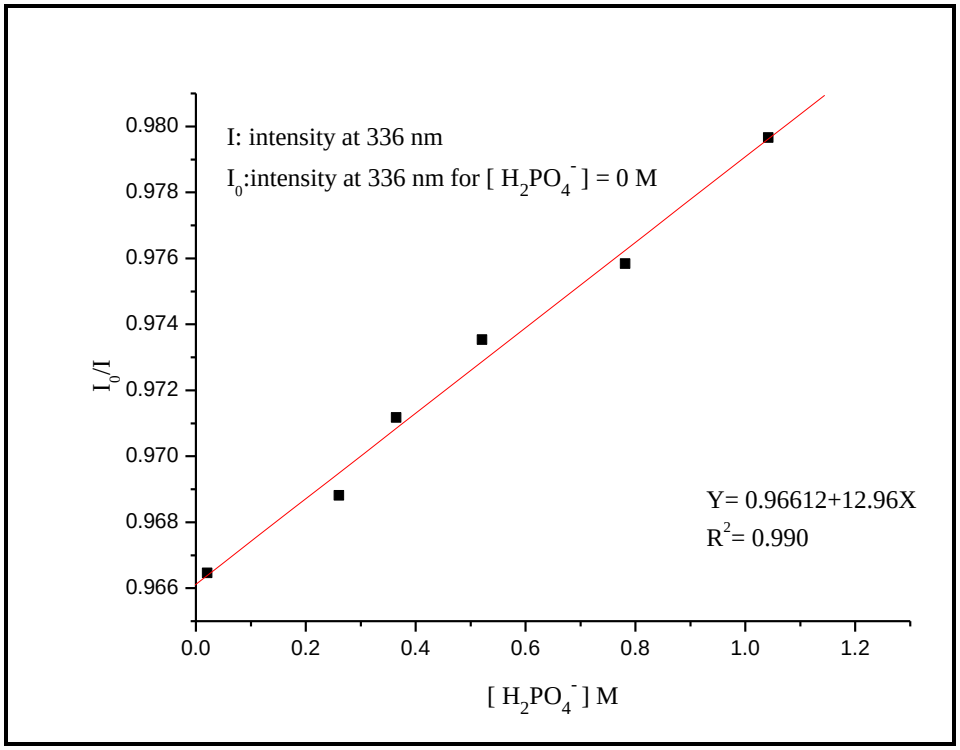

Figure S14 Stern-Volmer plot of **3** with $(n\text{-Bu})_4NH_2PO_4$ in H_2O/THF (1:1, v/v)

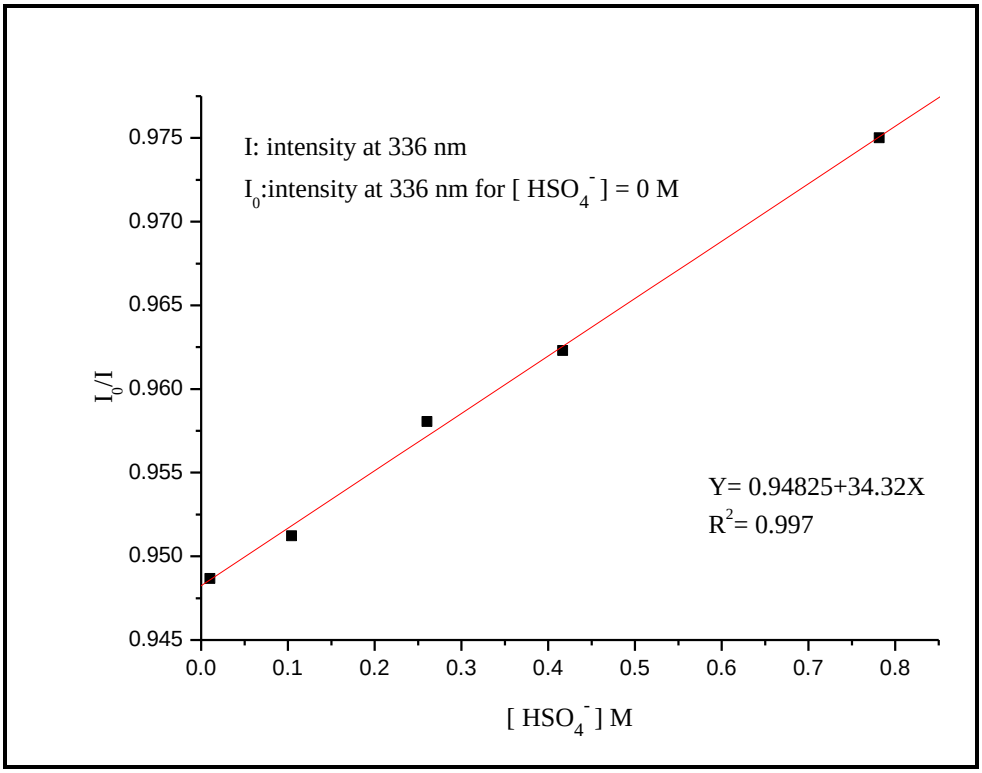

Figure S15 Stern-Volmer plot of **3** with $(n\text{-Bu})_4NHSO_4$ in H_2O/THF (1:1, v/v)

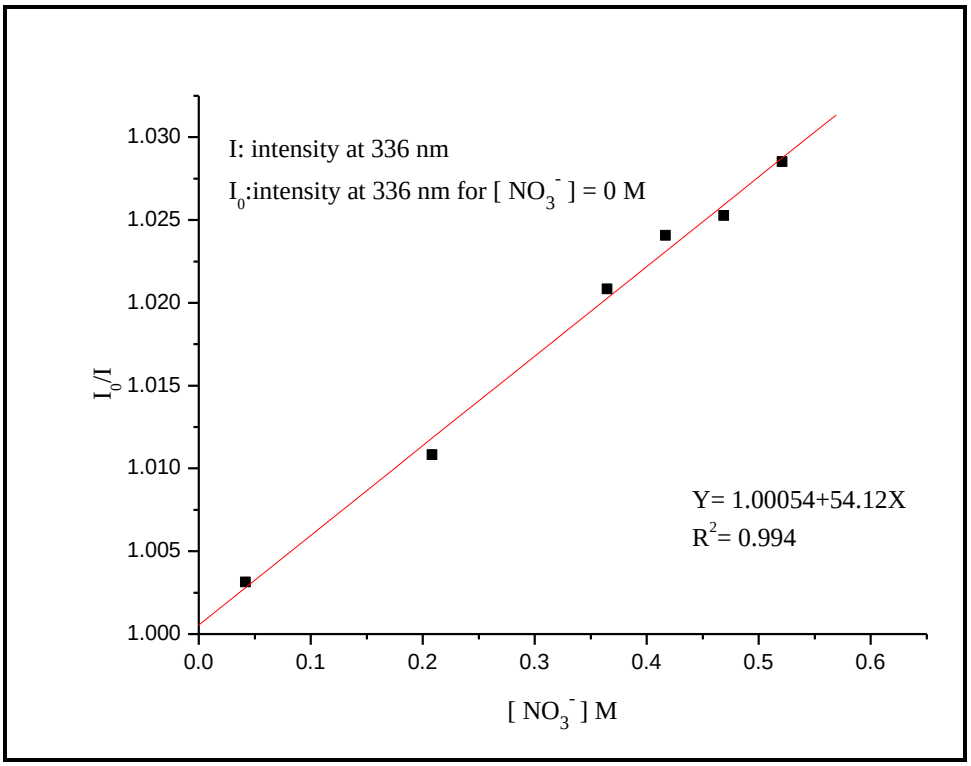

Figure S16 Stern-Volmer plot of **3** with $(n\text{-Bu})_4NNO_3$ in H_2O/THF (1:1, v/v)

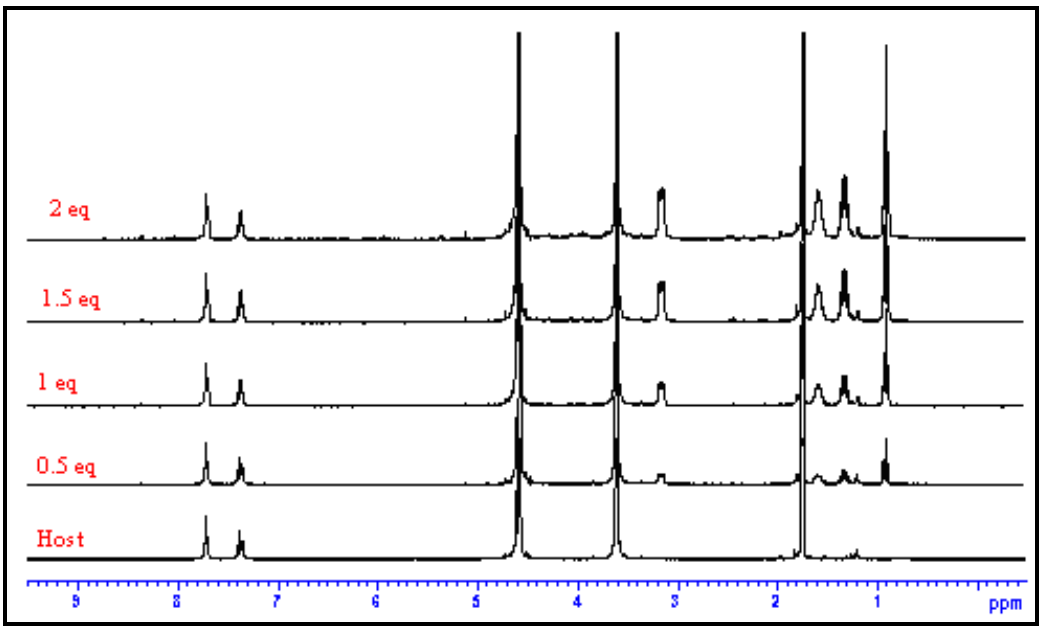

Figure S17 ^{1}H NMR spectra of **3** (10 mM) in the presence of 0.5-2.0 equiv of $(n\text{-Bu})_4NI$ in $_{d\text{-}8}$THF/D_2O (1:1, v/v).

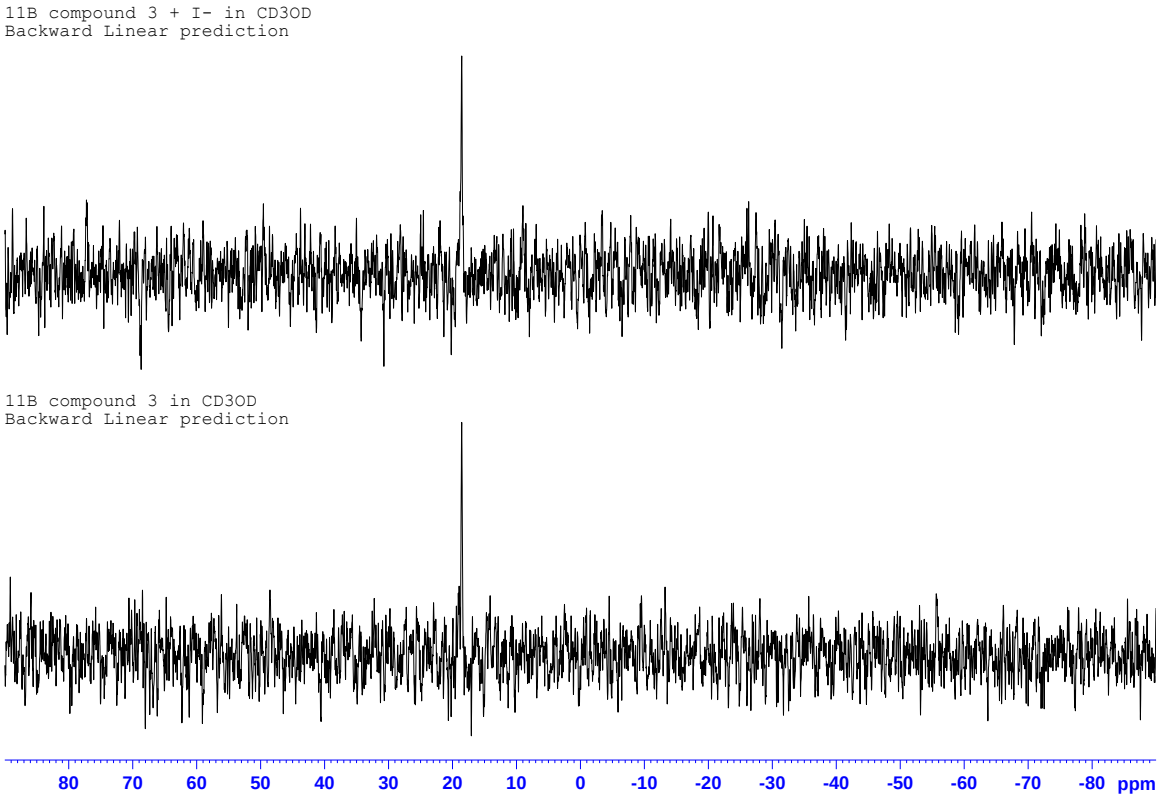

Figure S18 ^{11}B NMR spectra (CD_3OD) of (a) **3** and (b) **3** upon adding 1 equiv of (n-Bu)$_4$NI

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