SUPPLEMENTARY INFORMATION FOR:

Distance-dependent Fluorescence Quenching and Binding of CdSe Quantum Dots by Functionalized Nitroxide Radicals

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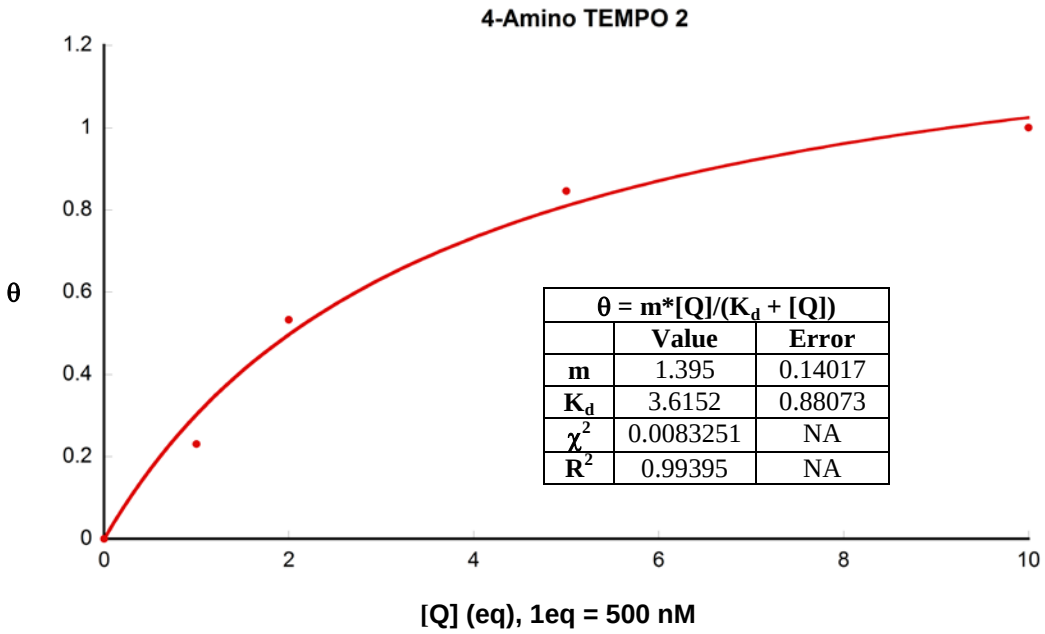


Figure S1. Nonlinear curve fit of the saturation curve for 4-amino TEMPO **2**.

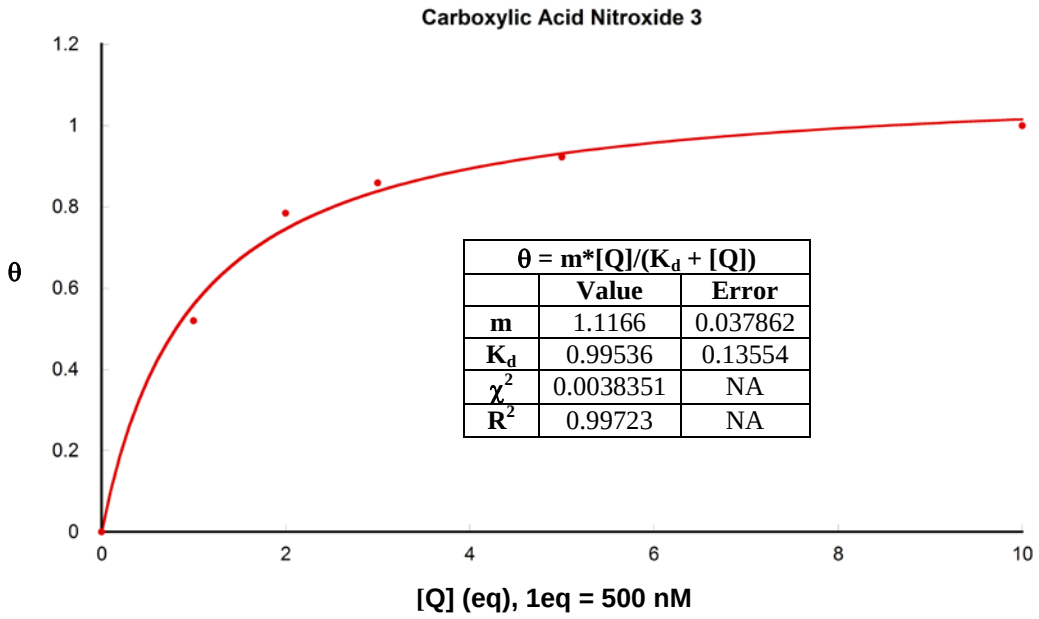


Figure S2. Nonlinear curve fit of the saturation curve for carboxylic acid nitroxide **3**.

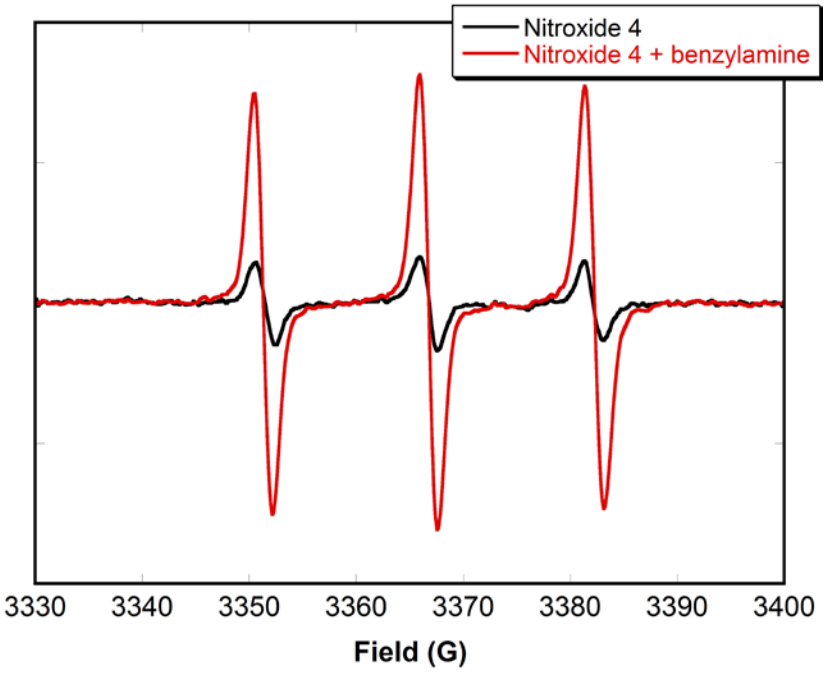


Figure S3. EPR spectra of bisamino nitroxide **4** (0.5 μM) in toluene before and after the addition of a large excess benzylamine (6×10^5:1 moles of benzylamine:nitroxide **4**).

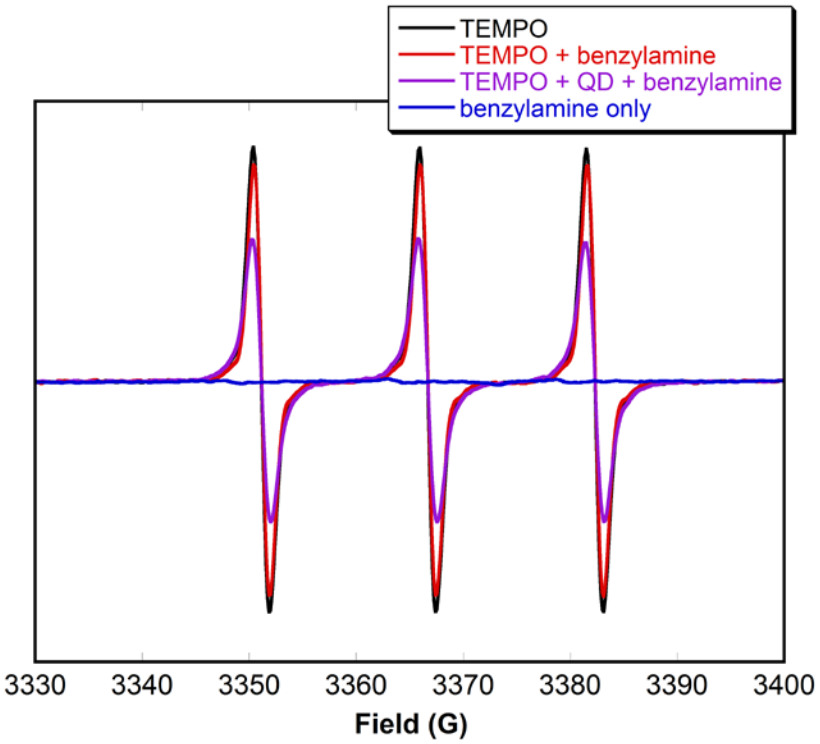


Figure S4. EPR spectra of TEMPO (0.5 μM) in toluene before and after the addition of a large excess of benzylamine (6×10^5:1 moles of benzylamine:TEMPO) and CdSe QDs (4 μM).

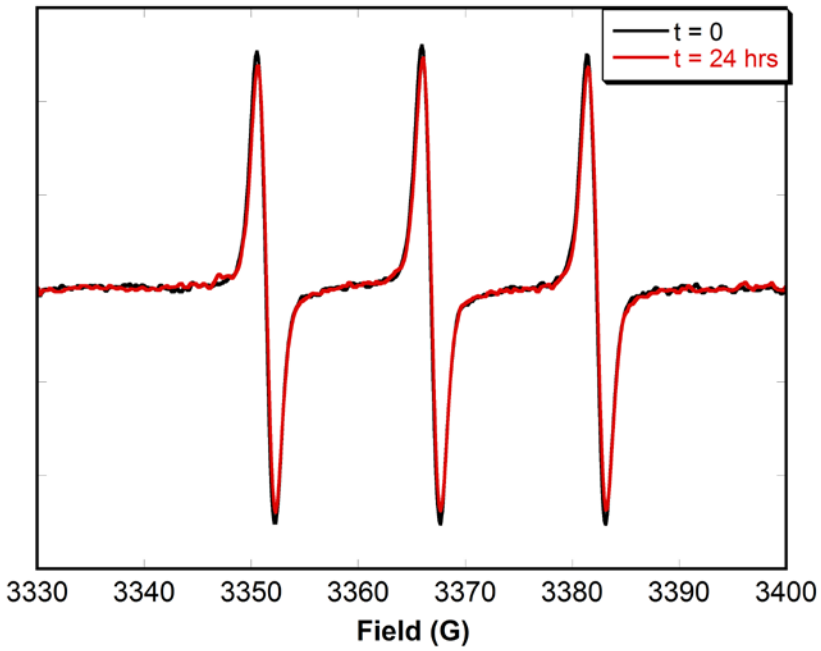
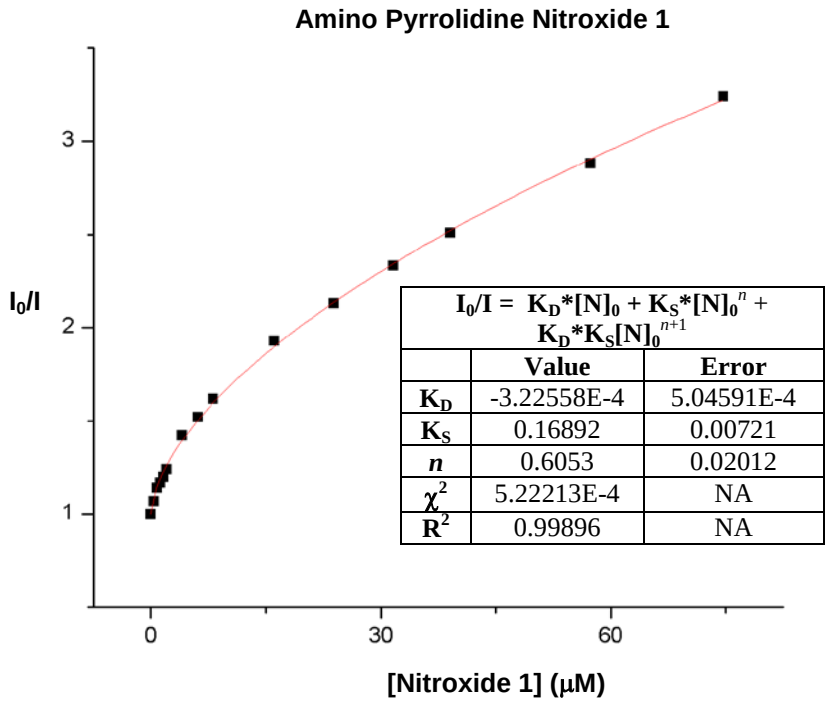


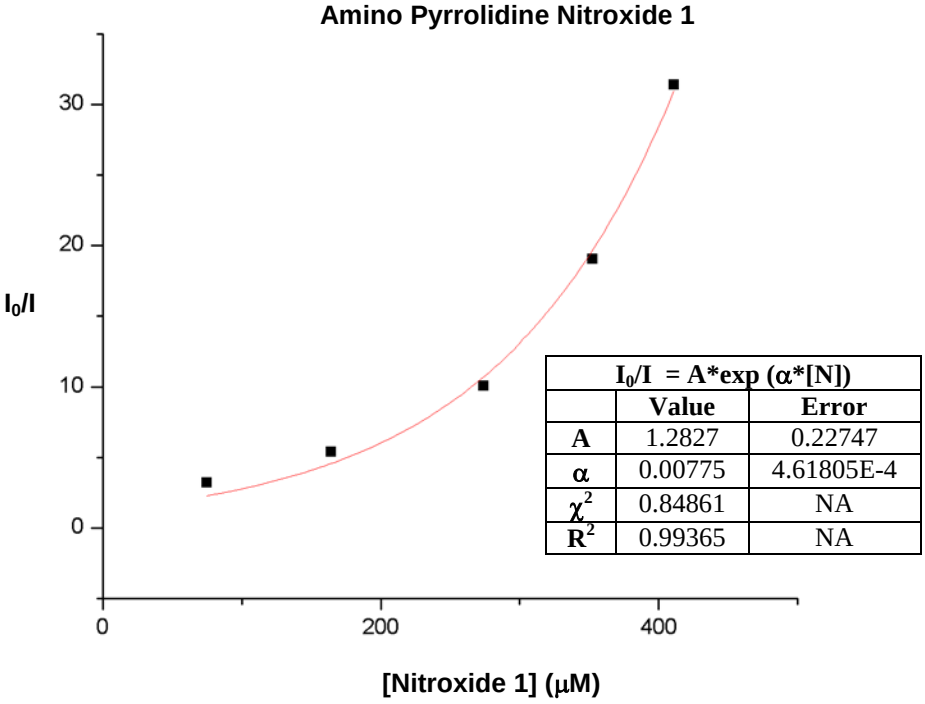
Figure S5. EPR spectra of carboxylic acid nitroxide **3** (0.5 μM) in toluene taken at t = 0 and t = 24 hrs.



$I_0/I = K_D*[N]_0 + K_S*[N]_0^n + K_D*K_S[N]_0^{n+1}$

	Value	Error
K_D	-3.22558E-4	5.04591E-4
K_S	0.16892	0.00721
n	0.6053	0.02012
χ^2	5.22213E-4	NA
R^2	0.99896	NA

Figure S6. Nonlinear curve fit of the downward curvature in the Stern-Volmer plot for amino pyrrolidine nitroxide **1** using the "multi-binding-site" model (see supporting information in reference 42 for the derivation of equation **4**). (Note: $[N]_0$ = [nitroxide]$_0$)



$I_0/I = A*\exp(\alpha*[N])$

	Value	Error
A	1.2827	0.22747
α	0.00775	4.61805E-4
χ^2	0.84861	NA
R^2	0.99365	NA

Figure S7. Nonlinear curve fit of the upward curvature in the Stern-Volmer plot for amino pyrrolidine nitroxide **1** using equation **5**.

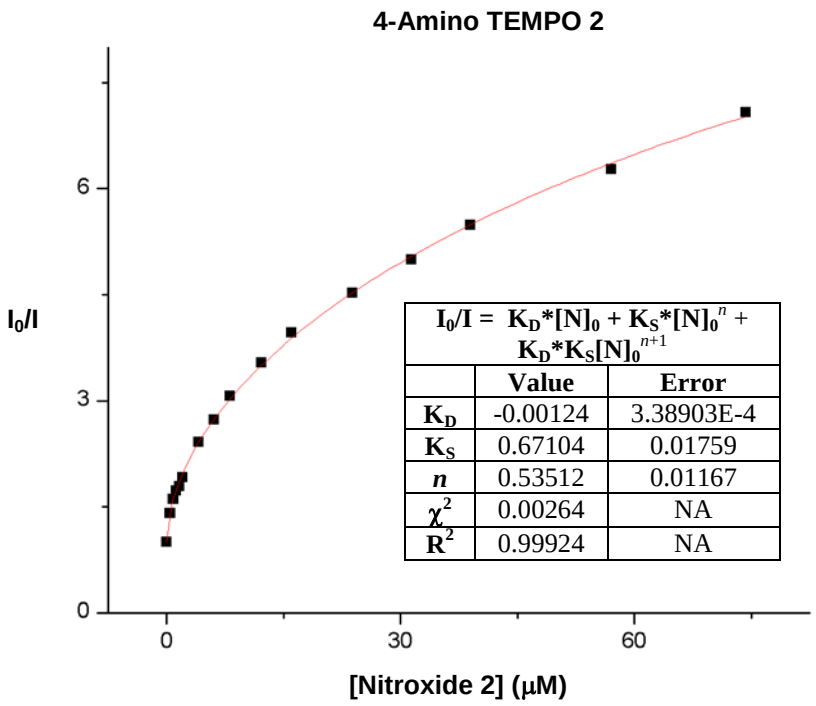


Figure S8. Nonlinear curve fit of the downward curvature in the Stern-Volmer plot for 4-amino TEMPO **2** using the "multi-binding-site" model.

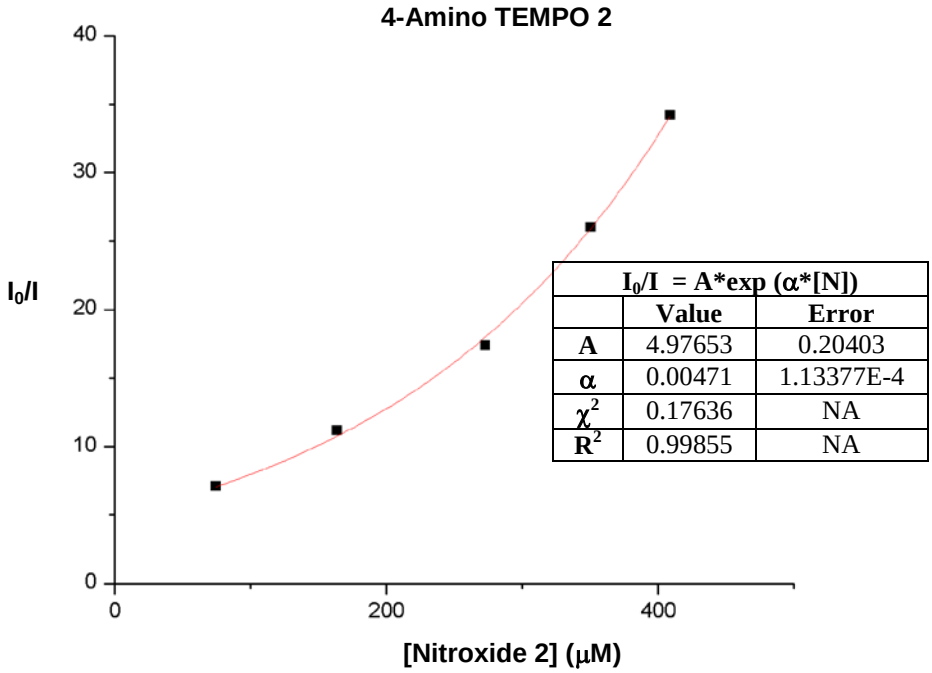


Figure S9. Nonlinear curve fit of the upward curvature in the Stern-Volmer plot for 4-amino TEMPO **2** using equation **5**.

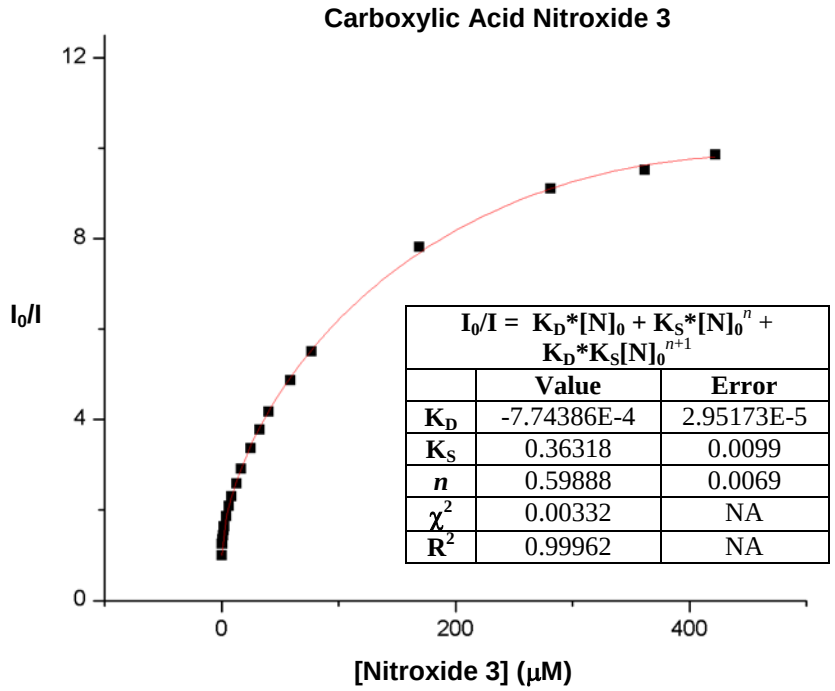


Figure S10. Nonlinear curve fit of the Stern-Volmer plot for carboxylic acid nitroxide **3** using the "multi-binding-site" model.

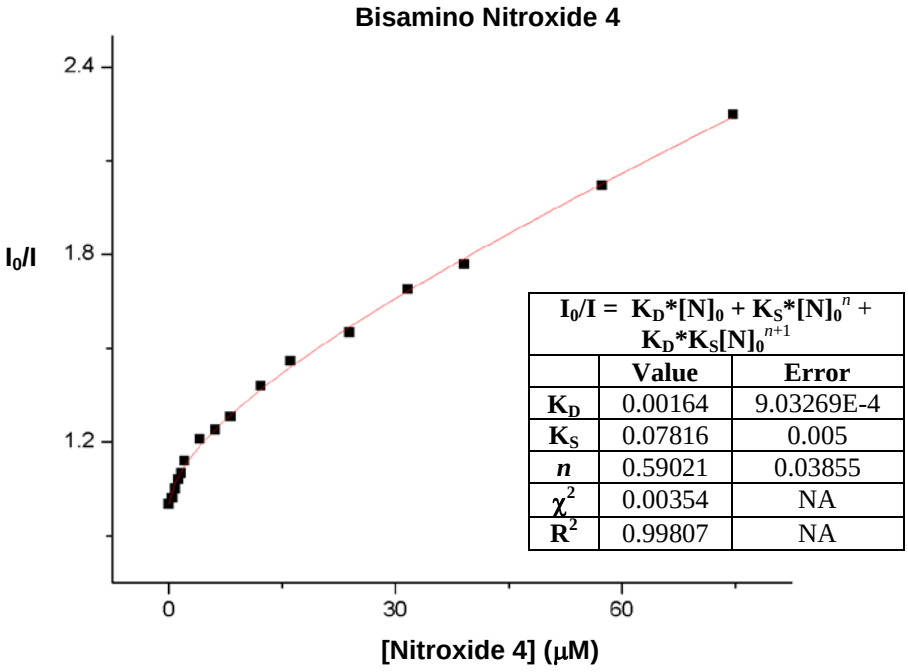


Figure S11. Nonlinear curve fit of the downward curvature in the Stern-Volmer plot for bisamino nitroxide **4** using the "multi-binding-site" model.

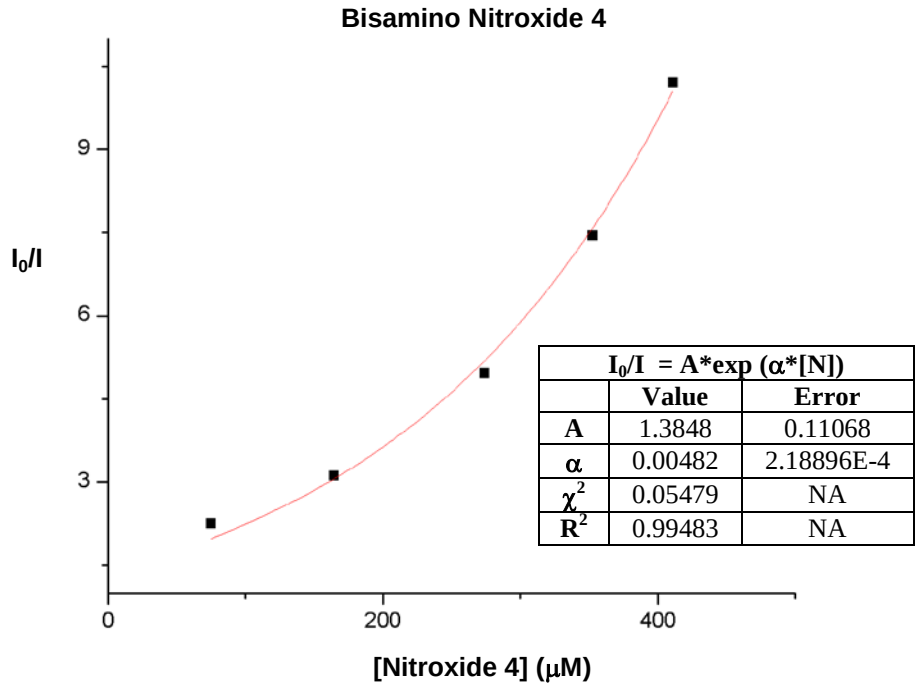


Figure S12. Nonlinear curve fit of the upward curvature in the Stern-Volmer plot for bisamino nitroxide **4** using equation **5**.

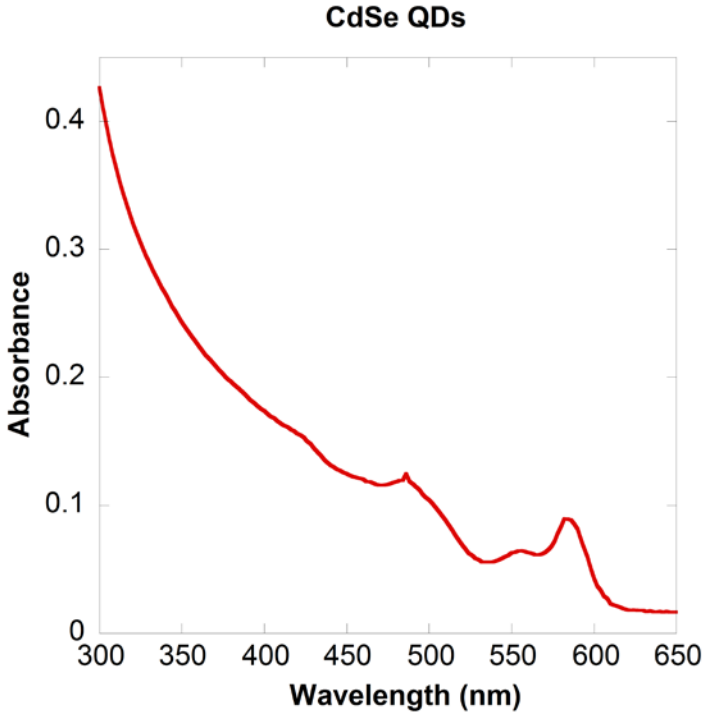


Figure S13. Absorption spectrum of of 3.7 nm CdSe QDs (0.4 μM) in toluene.

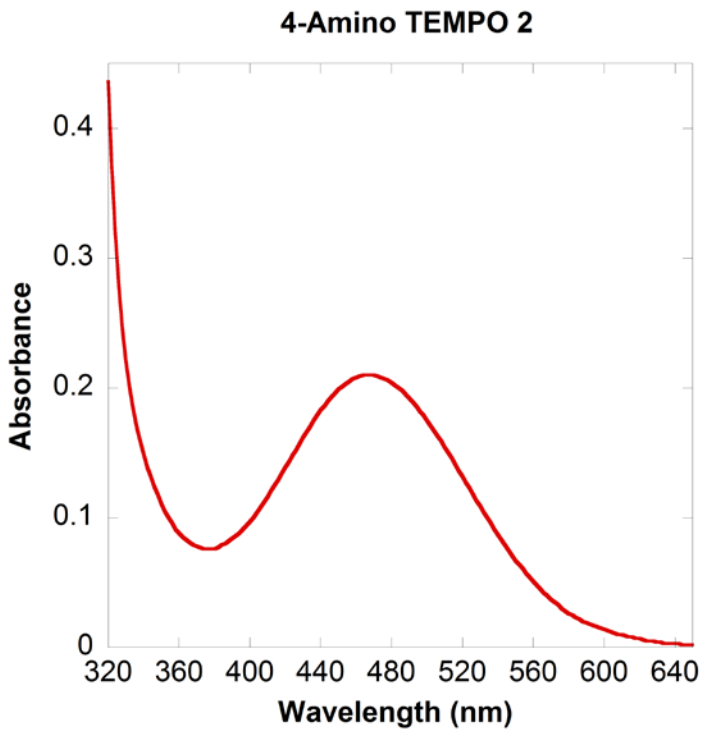


Figure S14. Absorption spectrum of 4-amino TEMPO **2 (**20 mM) in toluene.

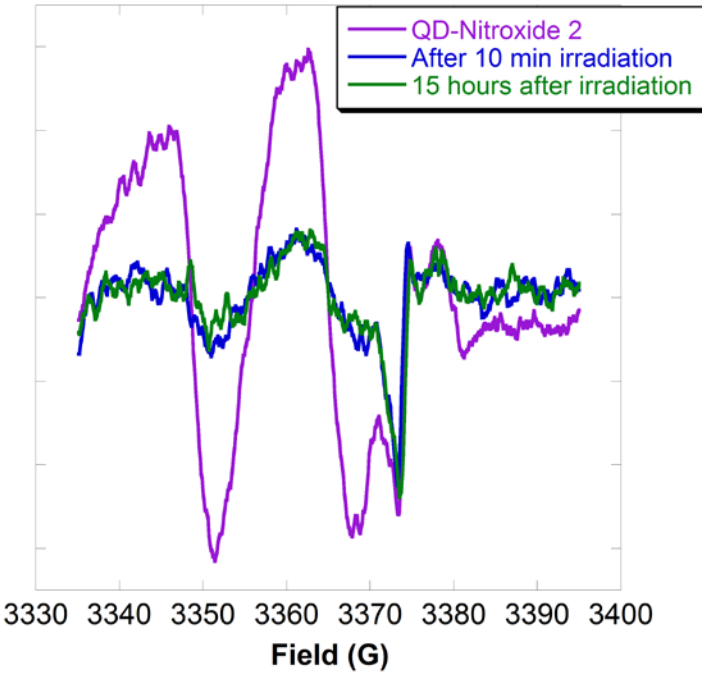


Figure S15. EPR spectra of a toluene solution containing CdSe QDs (2 μM) and 4-amino TEMPO **2** (1 μM) from photoexcitation studies: before irradiation, 10 minutes, and 15 hours after irradiation.

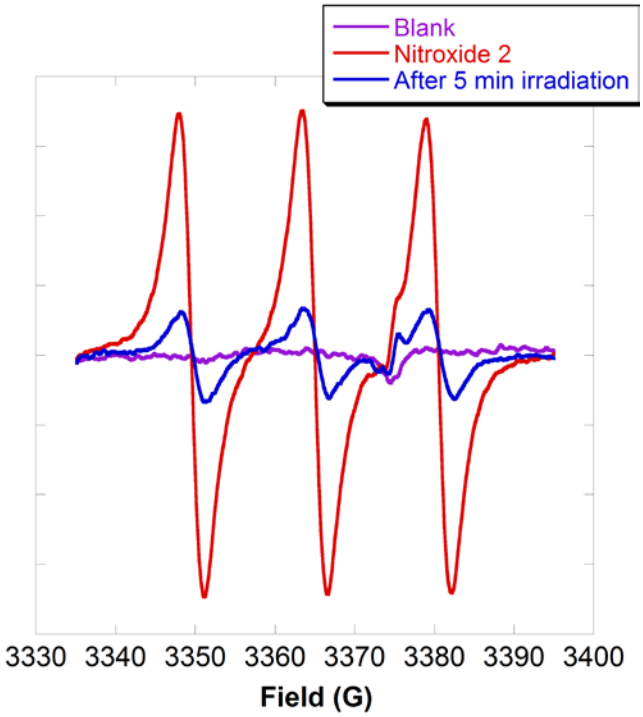


Figure S16. EPR spectra show the photoinduced reduction of 4-amino TEMPO **2** (1 μM) in toluene after photoexcitation at 365 nm.

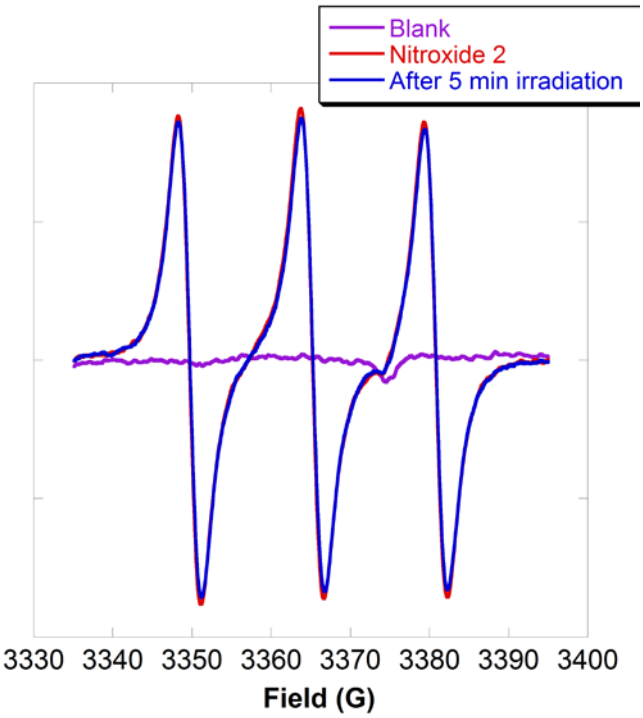
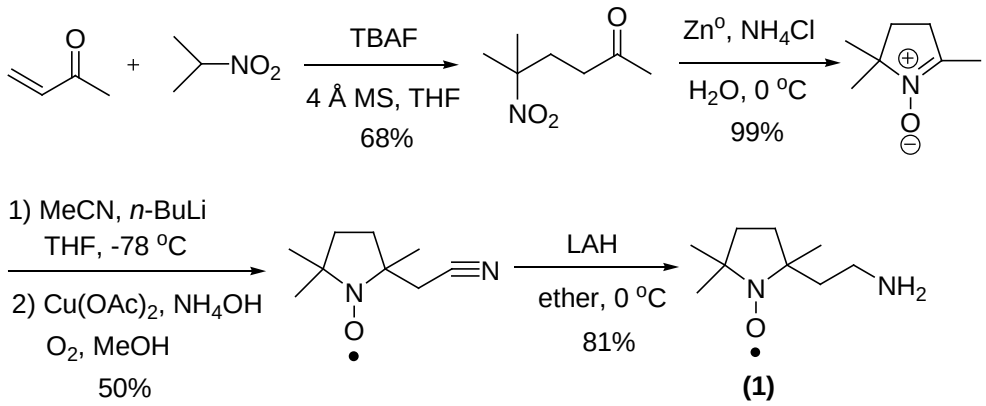


Figure S17. EPR spectra of 4-amino TEMPO **2** (1 μM) in benzene before and after photoexcitation at 365 nm.

Experimental Section

Synthesis



2-(2-Aminoethyl)-2,5,5-trimethylpyrrolidin-1-oxyl (1). Following a procedure of Miller *et al*,[1] to a solution of 2-nitropropane (4.20 mL, 46.7 mmol) and methyl vinyl ketone (4.19 mL, 51.4 mmol) with activated 4 Å molecular sieves in tetrahydrofuran (20 mL) was slowly added tetrabutylammonium fluoride (TBAF, 1.0 M in THF, 20 mL, 20 mmol). The solution was allowed to stir overnight under nitrogen. The reaction mixture was filtered through a celite pad, and rinsed with dichloromethane. The filtrate was dried over magnesium sulfate, filtered, and concentrated *in vacuo* to give 5.24 g of a brownish oil. The oil was purified by flash column chromatography with 4:1 hexane/ethyl acetate to give 5.06 g (31.8 mmol, 68% yield) of 5-methyl-5-nitrohexan-2-one as a non-viscous gold oil. TLC: 4:1 hexanes/ethyl acetate, UV, I_2, R_f = 0.40. ^{1}H-NMR (500 MHz, $CDCl_3$): δ 2.46 (t, *J* = 8.0 Hz, 2H), 2.19 (t, *J* = 8.0 Hz, 2H), 2.18 (s, 3H), 1.59 (s, 6H) ppm.

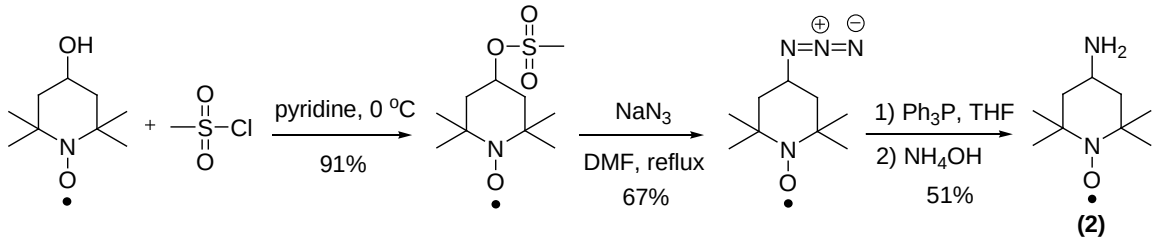
To a solution of 5-methyl-5-nitrohexan-2-one (4.68 g, 29.4 mmol) and ammonium chloride (1.70 g, 31.8 mmol) in water (39 mL) chilled in an ice bath was added zinc metal (7.69 g, 118 mmol) over an hour. The solution was allowed to warm slowly to room temperature overnight. The reaction mixture was filtered through a celite pad, and rinsed

with methanol. Volatiles were reduced *in vacuo*. The resulting aqueous solution was extracted with chloroform (5 × 20 mL). The combined organic layer was dried over magnesium sulfate, filtered, and concentrated *in vacuo* to give 3.68 g (28.9 mmol, 99% yield) of 3,4-dihydro-2,2,5-trimethyl-2*H*-pyrrole-1-oxide[2] as a gold oil. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.59 (td, 2H, *J* = 7.2, 1.2 Hz), 2.03 (t, 3H, *J* = 1.2 Hz), 2.00 (t, 2H, *J* = 7.2 Hz), 1.40 (s, 6H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 141.2, 73.2, 32.3, 29.1, 25.5, 13.2 ppm.

Following a modified procedure of Hideg *et al*,[3] acetonitrile (3.57 g, 86.9 mmol) in tetrahydrofuran (5 mL) was added dropwise to a stirred solution of *n*-butyllithium (46.3 mL, 2.5 M in hexanes, 116 mmol) in anhydrous tetrahydofuran (75 mL) at -78 ^{o}C under nitrogen. After 15 minutes, to this solution was added 3,4-dihydro-2,2,5-trimethyl-2*H*-pyrrole-1-oxide (3.68 g, 29.0 mmol) in anhydrous tetrahydrofuran (10 mL). After 10 minutes the reaction was quenched with saturated ammonium chloride (50 mL), and allowed to warm to room temperature. The organic layer was separated, and the aqueous layer was extracted with chloroform (3 × 30 mL). The combined organic layers were dried over magnesium sulfate, filtered and concentrated *in vacuo*. The resulting oil was taken up in methanol (145 mL), and ammonium hydroxide (11 mL) and then cupric acetate (263 mg, 1.45 mmol) were added. This solution was stirred while bubbling air through the system until the yellow solution turned green in color (15 minutes). The reaction mixture was concentrated *in vacuo*, taken up in chloroform (100 mL), washed with a saturated solution of sodium bisulfate (50 mL), and then with water (50 mL). The combined aqueous layer was extracted with chloroform (3 × 50 mL). The combined organic layers were dried over magnesium sulfate, filtered, and concentrated *in vacuo*.

The resulting oil was purified by flash column chromatography with 2:1 hexanes:ethyl acetate to give 2.43 g (14.5 mmol, 50% yield) of 2-cyanomethyl-1-oxyl-2,5,5-trimethylpyrrolidine as an orange oil. TLC: 2:1 hexanes/ethyl acetate, UV, I_2, R_f = 0.36. ^{1}H-NMR (500 MHz, $CDCl_3$, $PhNHNH_2$): δ 2.46 (s, 2H), 1.84 - 1.78 (m, 2H), 1.67 (m, 2H), 1.30 (s, 3H), 1.16 (s, 3H), 1.13 (s, 3H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$, $PhNHNH_2$): δ 119.2, 64.0, 63.8, 34.3, 33.3, 30.4, 27.6, 23.3, 23.1 ppm.

Following a modified procedure of Hideg *et al*,[4] a solution of 2-cyanomethyl-1-oxyl-2,5,5-trimethylpyrrolidine (2.43 g, 14.5 mmol) in anhydrous diethyl ether (48.5 mL) was cooled to 0 ^{o}C. To this solution was added dropwise lithium aluminum hydride (LAH, 1.0 M in diethyl ether, 72.7 mL, 72.7 mmol) under nitrogen. The solution was allowed to warm to room temperature for 3 hours. The reaction was quenched by cooling to 0 ^{o}C before adding sequentially: 2.8 mL of water, 2.8 mL of 15% sodium hydroxide solution, 9.0 mL of water, and then 2.00 g of magnesium sulfate. This mixture was stirred for an additional 15 minutes, and filtered through a celite pad. The filtrate was concentrated *in vacuo* and taken up in chloroform (30 mL). To this solution was added lead dioxide (174 mg, 0.727 mmol), and the solution was stirred while air was bubbled through the system for 30 minutes. The solution was filtered through a celite pad, and then concentrated *in vacuo* to give 2.03 g (11.9 mmol, 81% yield) of the title product as an orange-yellowish oil. ^{13}C NMR (125 MHz, $CDCl_3$, $PhNHNH_2$): δ 64.5, 62.3, 42.8, 37.0, 34.7, 33.2, 29.2, 22.6, 20.5 ppm.

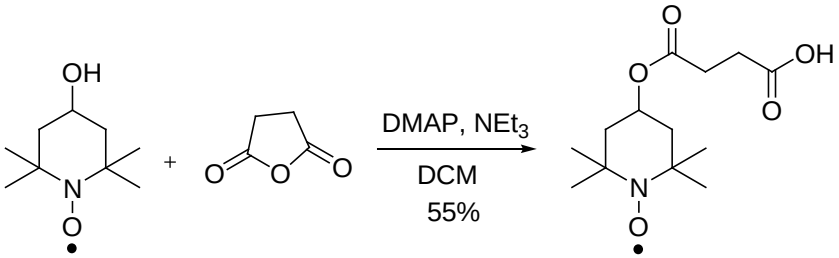


4-Amino-2,2,6,6-tetramethylpiperidine-1-oxyl (2). Following a modified procedure of Bushmakina *et al*,[4] to an ice-cooled solution of 4-hydroxy-2,2,6,6-tetramethylpiperidine-*N*-oxyl (TEMPOL, 2.008 g, 11.66 mmol) in anhydrous pyridine (8.20 mL) was added methanesulfonyl chloride (1.80 mL, 23.1 mmol). The solution was allowed to warm to room temperature under nitrogen for 6 h, and then cold saturated sodium bicarbonate was added to the reaction mixture cooled over an ice bath. The solution was extracted with chloroform (4 × 40 mL). The combined organic layer was washed with saturated sodium bicarbonate (2 × 40 mL), and then with water (2 × 40 mL), dried over sodium sulfate, filtered, and concentrated *in vacuo* to give 2.651 g (10.59 mmol, 91% yield) of 4-methanesulfonyl-oxy-2,2,6,6-tetramethylpiperidine-1-oxyl as an orange solid. m.p. = 85-87 °C. ^{1}H-NMR (500 MHz, $CDCl_3$, $PhNHNH_2$): δ 4.94 (m, 1H), 3.01 (s, 3H), 2.08 (dt, 2H, *J* = 13, 2.0 Hz), 1.80 (dd, 2H, *J* = 13, 13 Hz), 1.26 (s, 6H), 1.22 (s, 6H) ppm.

To a solution of 4-methanesulfonyl-oxy-2,2,6,6-tetramethylpiperidine-1-oxyl (1.003 g, 4.006 mmol) in dimethylformamide (13 mL) was added sodium azide (521 mg, 8.01 mmol). A condenser was attached, and the solution was heated to 110 °C under nitrogen overnight. Upon cooling, diethyl ether (100 mL) was added to the reaction mixture. The organic layer was washed with saturated sodium bicarbonate (50 mL), and water (4 × 50 mL), dried over sodium sulfate, filtered, and concentrated *in vacuo* to give 490 mg (2.48 mmol, 62% yield) of 4-azido-2,2,6,6-tetramethylpiperidine-1-oxyl as an orange solid.

m.p. = 60-61 °C. ^{1}H-NMR (500 MHz, $CDCl_3$, $PhNHNH_2$): δ 4.60 (m, 1H), 1.87 (dt, 2H, *J* = 12.6, 1.8 Hz), 1.57 (dd, 2H, *J* = 12.6, 12.6 Hz), 1.23 (s, 6H), 1.16 (s, 6H) ppm.

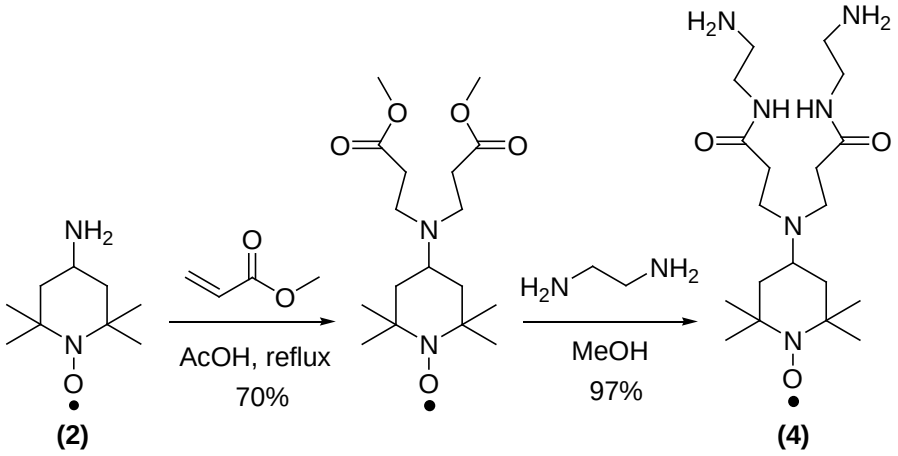
To a solution of 4-azido-2,2,6,6-tetramethylpiperidine-1-oxyl (490 mg, 2.48 mmol) in anhydrous tetrahydrofuran (5.30 mL) was added triphenylphosphine (2.376 g, 8.968 mmol) in portions over 3 h. Concentrated ammonia solution (5.30 mL) was then added. The solution was allowed to stir overnight under nitrogen. Solvent was removed *in vacuo* using only gentle heating (~40 °C). The reaction mixture was taken up in chloroform (50 mL), extracted with 10% acetic acid (2 × 25 mL). The combined aqueous layer was neutralized with saturated sodium bicarbonate using a few drops of 2 M sodium hydroxide to make it slightly basic, extracted with chloroform (3 × 50 mL), dried over sodium sulfate, filtered, and concentrated *in vacuo* to give 357 mg (2.08 mmol, 84% yield) of the title product as an orange oil. ^{1}H-NMR (500 MHz, $CDCl_3$, $PhNHNH_2$): δ 3.05 (m, 1H), 1.75 (dt, 2H, *J* = 12, 1.5 Hz), 1.26 (dd, 2H, *J* = 12, 12 Hz), 1.17 (s, 6H), 1.14 (s, 6H) ppm.



(3)

4-(1-Oxyl-2,2,6,6-tetramethylpiperidin-4-yloxy)-4-oxobutanoic acid (3). To a solution of TEMPOL (200 mg, 1.16 mmol) and 4-dimethylaminopyridine (DMAP, 10 mg, 0.083 mmol) in anhydrous dichloromethane (5 mL) cooled over ice bath was added anhydrous triethylamine (0.90 mL, 6.5 mmol). A solution of succinic anhydride (209 mg, 2.09 mmol) in anhydrous dichloromethane (5 mL) was added slowly. The solution was

allowed to warm to room temperature overnight under nitrogen. Water (10 mL) and 10% hydrochloric acid (3 mL) were added into the reaction mixture. The aqueous layer was extracted with dichloromethane (3 × 10 mL). The organic layers were combined, dried over sodium sulfate, filtered, and concentrated *in vacuo* to give an orange oil. The oil was purified by silica gel column chromatography with 1:2 hexanes/ethyl acetate to give 175 mg (0.643 mmol, 55% yield) of the title product as an orange solid. TLC: 1:2 hexanes/ethyl acetate, UV, *p*-anisaldehyde stain, R_f = 0.40. m.p. = 86-87 °C. IR: ($CDCl_3$) 2977, 1731, 1711, 1219, 1167 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$, $PhNHNH_2$): δ 4.99 (m, 1H), 2.66 (t, 2H, *J* = 6.0 Hz), 2.48 (t, 2H, 6.0 Hz), 2.28 (dd, 2H, *J* = 12, 2.5 Hz), 1.94 (dd, 2H, *J* = 12, 12 Hz), 1.40 (s, 6H), 1.31 (s, 6H) ppm. ^{13}C-NMR/DEPT (125 MHz, $CDCl_3$, $PhNHNH_2$): δ 179.1 (C), 173.3 (C), 65.0 (CH), 60.8 (C), 41.9 (CH_2), 31.0 (CH_2), 30.5 (C), 28.1 (CH_3), 21.1 (CH_3) ppm. HRMS: M+1 (addition of $PhNHNH_2$, $C_{13}H_{24}NO_5^+$) 274.1649 calcd; 274.1646 obsd.

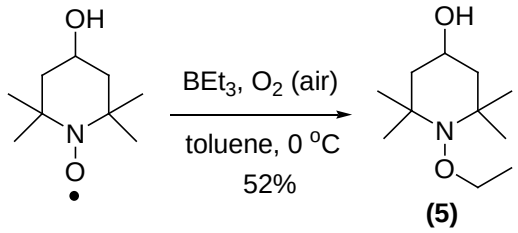


3,3'-(1-Oxyl-2,2,6,6-tetramethylpiperidin-4-yl-azanediyl)bis(*N*-(2-aminoethyl) propanamide) (4). Following the modified procedure of Yao *et al*,[5] to a 10 mL round bottom flask equipped with a stir bar, distilled methyl acrylate (1.90 mL, 21.1 mmol) and

glacial acetic acid (0.15 mL, 2.6 mmol) was added to 4-amino-2,2,6,6-tetramethylpiperidine-*N*-oxyl (compound **1**, 357 mg, 2.08 mmol). The solution was allowed to stir overnight under nitrogen. Excess methyl acrylate was removed under reduced pressure. The residue was taken up in ethyl acetate (15 mL) and washed with saturated sodium bicarbonate (15 mL), water (15 mL), and brine (30 mL). The organic layer was dried over magnesium sulfate, filtered, and concentrated *in vacuo* to give 504 mg (1.47 mmol, 70% yield) of 3,3′-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl-amino) bispropionic acid dimethyl ester as an orange oil. IR: ($CDCl_3$) 2974, 2950, 1798, 1437, 1362, 1201, 1173 cm^{-1}. ^{1}H-NMR (600 MHz, $CDCl_3$, $PhNHNH_2$): δ 3.98 (dd, 1H, *J* = 6.6, 6.6 Hz), 3.66 (s, 6H), 2.79 (t, 4H, *J* = 6.6 Hz), 2.43 (t, 4H, *J* = 6.6 Hz), 1.64 (d, 2H, *J* = 12 Hz), 1.45 (d, 2H, *J* = 8.4 Hz), 1.24 (s, 6H), 1.19 (s, 6H) ppm. ^{13}C-NMR/DEPT (150 MHz, $CDCl_3$, $PhNHNH_2$): δ 173.1 (C), 59.4 (C), 51.6 (CH_3), 50.7 (CH), 46.3 (CH_2), 41.3 (CH_2), 34.8 (CH_2), 32.8 (CH_3), 20.0 (CH_3) ppm. HRMS: M+1 (addition of $PhNHNH_2$, $C_{17}H_{33}N_2O_5^+$) 345.2384 calcd; 345.2367 obsd.

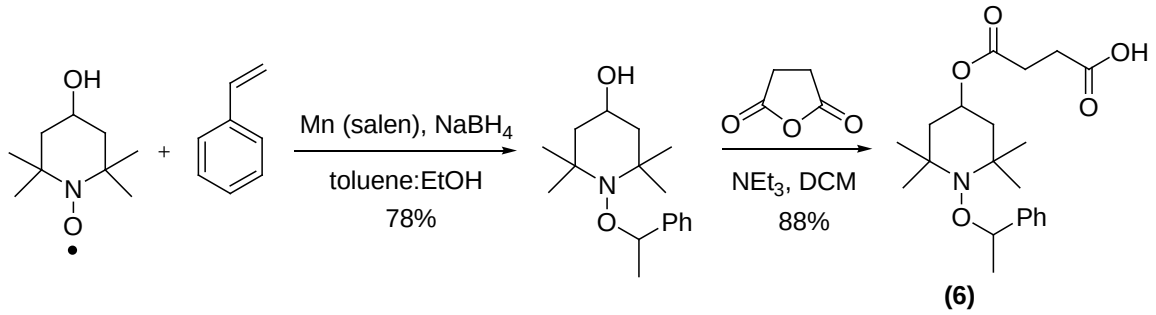
To a solution of 3,3′-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl-amino) bispropionic acid dimethyl ester (482 mg, 1.40 mmol) in methanol (5.30 mL) was added ethylenediamine (9.50 mL, 140 mmol). The solution was allowed to stir at room temperature overnight under nitrogen. Excess ethylenediamine was removed *in vacuo* using toluene/methanol as an azeotrope[6] to give 540 mg (1.35 mmol, 97% yield) of the title product as an orange oil. IR: ($CDCl_3$) 3365, 2975, 2938, 1644, 1555, 1463, 1380, 1363, 1242, 1121, 1034 cm^{-1}. ^{1}H-NMR (600 MHz, $CDCl_3$, $PhNHNH_2$): δ 3.98 (dd, 1H, *J* = 6.0, 6.0 Hz), 3.29 (t, 4H, *J* = 6.0 Hz), 2.82 (t, 4H, 6.0 Hz), 2.76 (t, 4H, *J* = 6.0 Hz), 2.35 (t, 4H, *J* = 6.0 Hz), 1.48 (m, 4H), 1.16 (s, 6H), 1.11 (s, 6H) ppm. ^{13}C-NMR/DEPT (150 MHz, $CDCl_3$, $PhNHNH_2$): δ

172.9 (C), 59.2 (C), 50.2 (CH), 46.6 (CH_2), 42.0 (CH_2), 41.6 (CH_2), 40.7 (CH_2), 35.5 (CH_2), 33.0 (CH_3), 20.0 (CH_3) ppm. HRMS: M+1 (addition of $PhNHNH_2$, $C_{19}H_{41}N_6O_3^+$) 401.3235 calcd; 401.3225 obsd.



(5)

1-Ethoxy-2,2,6,6-tetramethylpiperidin-4-ol (5). To a solution of TEMPOL (206 mg, 1.20 mmol) in anhydrous toluene (4 mL) was added triethylborane (1.0 M in THF, 1.40 mL, 1.40 mmol). The solution was allowed to stir open to the atmosphere overnight. After five days, TEMPOL was not completely consumed, so more triethylborane (1.0 M in THF, 1.40 mL, 1.40 mmol) was added. The solution turned from orange to yellow within 15 minutes after the second addition of triethylborane. After ten days, the reaction mixture was concentrated *in vacuo* to give a yellowish oil. The oil was purified by silica gel column chromatography first with 10:1 hexanes/ethyl acetate, and then 4:1 hexanes/ethyl acetate to afford 126 mg (0.626 mmol, 52% yield) of the title product as a colorless oil. TLC: 10:1 hexanes/ethyl acetate, UV, *p*-anisaldehyde stain, R_f = 0.64. IR: ($CDCl_3$) 3267, 2972, 1372, 1360, 1040, 730 cm^{-1}. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 3.94 (m, 1H), 3.77 (q, 2H, *J* = 7.2 Hz), 1.80 (dt, 2H, 12, 1.8 Hz), 1.44 (dd, 2H, *J* = 12, 12 Hz), 1.18 (s, 6H), 1.14 (s, 6H), 1.11 (t, 3H, *J* = 7.2 Hz) ppm. ^{13}C-NMR/DEPT (150 MHz, $CDCl_3$): δ 72.2 (CH_2), 63.8 (CH), 59.9 (C), 48.3 (CH_2), 33.4 (CH_3), 21.1 (CH_3), 13.8 (CH_3) ppm. HRMS: M+1 ($C_{11}H_{24}NO_2^+$) 202.1802 calcd; 202.1766 obsd.



(6)

4-(1-Phenylethoxy-2,2,6,6-tetramethylpiperidin-4-yloxy)-4-oxobutanoic acid (6). Following a modified procedure of Hawker *et al*,[7] TEMPOL (302 mg, 1.75 mmol) was dissolved in 2:3 v/v of toluene (6.40 mL) and ethanol (8.90 mL) followed by the addition of distilled styrene (0.40 mL, 3.5 mmol). Manganese salen catalyst[8] (196 mg, 0.549 mmol) was added followed by sodium borohydride (198 mg, 5.23 mmol); the reaction was allowed to stir open to atmosphere overnight. The reaction mixture was concentrated *in vacuo* and combined with dichloromethane (15 mL) and water (15 mL). The aqueous layer was extracted with dichloromethane (2 × 15 mL). The combined organic layer was washed with saturated sodium bicarbonate, dried over magnesium sulfate, filtered, and concentrated *in vacuo* to give a brownish oil. The oil was purified by silica gel column chromatography first with 4:1 hexanes/ethyl acetate, and then 2:1 hexanes/ethyl acetate to give 379 mg (1.37 mmol, 78% yield) of 1-(1-phenylethoxy)-2,2,6,6-tetrametylpiperidin-4-ol as a white solid. TLC: 4:1 hexanes/ethyl acetate, UV, *p*-anisaldehyde stain, R_f = 0.23. m.p. = 95-97 $^{\circ}$C. IR: ($CDCl_3$) 3286, 2969, 1449, 1373, 1361, 1043, 1031, 697 cm^{-1}. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.31-7.21 (m, 5H), 4.77 (q, 1H, *J* = 6.6 Hz), 3.93 (m, 1H), 1.84 (dt, 2H, *J* = 12.6, 3.6 Hz), 1.70 (dt, 2H, *J* = 12.6, 3.6 Hz), 1.48 (d, 3H, 6.6 Hz), 1.33 (s, 3H), 1.22 (s, 3H), 1.07 (s, 3H), 0.67 (s, 3H) ppm. ^{13}C-NMR/DEPT (150 MHz, $CDCl_3$): δ 145.4 (C), 128.0 (CH), 126.9 (CH), 126.6 (CH), 83.3 (CH), 63.3 (CH), 60.2

(C), 60.0 (C), 48.9 (CH_2), 48.8 (CH_2), 34.5 (CH_3), 34.2 (CH_3), 23.4 (CH_3), 21.3 (CH_3) ppm. HRMS: M+1 ($C_{17}H_{28}NO_2^+$) 278.2115 calcd; 278.2114 obsd.

To a solution of 1-(1-phenylethoxy)-2,2,6,6-tetrametylpiperidin-4-ol (355 mg, 1.28 mmol) and DMAP (9.9 mg, 0.081 mmol) in anhydrous dichloromethane (11 mL) was added distilled triethylamine (0.90 mL, 6.5 mmol). Succinic anhydride (200 mg, 2.00 mmol) was added slowly while cooling the reaction mixture over an ice bath. The solution was allowed to warm to room temperature overnight under nitrogen. Distilled water (15 mL) and 10% hydrochloric acid (5 mL) were added and the aqueous layer was extracted with dichloromethane (3 × 10 mL). The organic layers were combined, dried over sodium sulfate, filtered, and concentrated *in vacuo* to give a golden-brown oil. The oil was purified by silica gel column chromatography with 1:2 hexanes/ethyl acetate to give 425 mg (1.12 mmol, 88% yield) of the title product as a slightly yellowish solid. TLC: 1:2 hexanes/ethyl acetate, UV, *p*-anisaldehyde stain, R_f = 0.78. m.p. = 76-78 $^{\circ}$C. IR: ($CDCl_3$) 2975, 2934, 1734, 1714, 1363, 1174, 700 cm^{-1}. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.30-7.21 (m, 5H), 5.02 (m, 1H), 4.77 (q, 1H, *J* = 6.6 Hz), 2.64 (t, 2H, *J* = 6.6 Hz), 2.57 (t, 2H, *J* = 6.6 Hz), 1.86 (d, 1H, *J* = 12 Hz), 1.74 (dt, 2H, *J* = 12, 3.6 Hz), 1.58 (t, 1H, *J* = 12 Hz), 1.48 (d, 3H, 6.6 Hz), 1.33 (s, 3H), 1.26 (s, 3H), 1.11 (s, 3H), 0.66 (s, 3H) ppm. ^{13}C-NMR/DEPT (150 MHz, $CDCl_3$): δ 177.9 (C), 171.7 (C), 145.3 (C), 128.1 (CH), 127.0 (CH), 126.6 (CH), 83.4 (CH), 67.5 (CH), 61.3 (C), 60.2 (C), 44.5 (CH_2), 34.3 (CH_3), 34.0 (CH_3), 29.2 (CH_2), 29.0 (CH_2), 23.4 (CH_3), 21.1 (CH_3) ppm. HRMS: M+1 ($C_{21}H_{32}NO_5^+$) 378.2275 calcd; 378.2276 obsd.

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