Supporting Information

Development of A Catalytic Electron Transfer System Mediated by Transition Metal Ate Complexes: Applicability and Tunability of Electron-releasing Potential for Organic Transformations

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General Comments. Melting points were determined with a Yazawa micro melting point apparatus and uncorrected. ^{1}H-NMR spectra were recorded on a Varian Gemini 2000 using tetramethylsilane as an internal standard. Chemical shifts are expressed in δ (ppm) values, and coupling constants are expressed in herts (Hz). The following abbreviations are used : s = singlet, d = doublet, t = triplet, quint = quintet, m = multiplet, and brs = broad singlet.[i] Mass spectra were recorded on a JOEL JMS-O1SG-2 spectrometer.

Materials. Unless otherwise noted, materials were purchased from Tokyo Kasei Co., Aldrich Inc., and other commercial suppliers and were used after appropriate purification (distillation or recrystalization) before use. $CoCl_2$, $FeCl_2$, and $MnCl_2$ were obtained from Wako Pure Chemical Industries, Ltd. MeLi in Et_2O, ^{n}BuLi in n-hexane and ^{t}BuLi in n-pentane were obtained from Kanto Chemical Co. Ltd. The concentrations of MeLi, ^{n}BuLi and ^{t}BuLi were determined by titration prior to use.[1]

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Preparation of ET-ate complexes[2]

Me_3CoLi

Under Ar atmosphere, MeLi (0.83 M Et_2O solution, 5.4 mL, 4.5 mmol) was added to the mixture of dry THF (4 mL) and $CoCl_2$ (200.8 mg, 1.5 mmol) at -78°C and the mixture was stirred for 2 h at -78°C.

Me_3FeLi

Under Ar atmosphere, MeLi (0.83 M Et_2O solution, 5.4 mL, 4.5 mmol) was added to the mixture of dry THF (4 mL) and $FeCl_2$ (196.0 mg, 1.5 mmol) at -78°C and the mixture was stirred for 2 h at -40°C.

Me_3MnLi

Under Ar atmosphere, a THF suspension of $MnCl_2$ (188.8 mg, 1.5 mmol) was sonicated for 20 min. The mixture was cooled to 0°C, and MeLi (0.83 M Et_2O solution, 5.4 mL, 4.5 mmol) was added and was stirred for 30 min at 0°C.

IR Spectroscopic Analyses. Samples were recorded using a ReactIR™ 1000 from ASI Applied Systems fitted with a one in an immerscible DiComp™ ATR (Attenuated Total Reflectance) probe optimized for maximum sensitivity. The spectra were acquired in 128 scans per spectrum at a gain of one and a resolution of two using system ReactIR™ 2.1 software. A representative reaction was carried out as follows: The IR probe was inserted through a nylon adapter and O-ring seal into an oven-dried, cylindrical two necked flask fitted with magnetic stir bar. Following evacuation under full vacuum and flushing with Ar, the flask was charged with a suspension of Na (161 mg, 7.0 mmol) in THF (3 ml) at 23°C. Following the recording of a background spectrum, a 3M THF solution of benzophenone (2 ml, 6.0 mmol) was added neat with stirring. IR spectra were recorded every 2 minutes over the course of the reaction. To account for mixing and temperature equilibration, spectra recorded in the first 2 min were discarded. Data manipulation and statistical analyses were carried out using the system 2.1 ReactIR™

software.

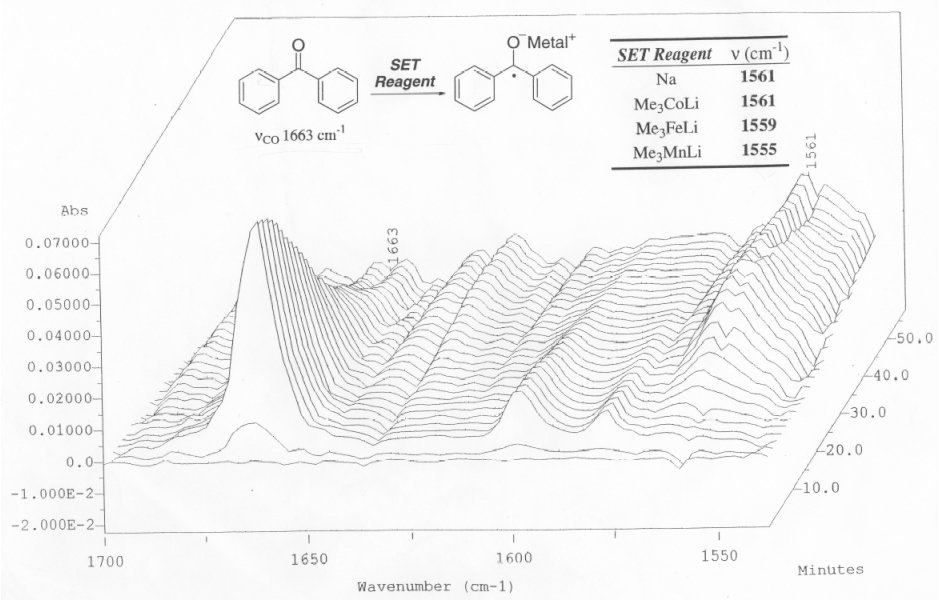

Figure *S*-1 FT-IR Spectroscopic Analysis of SET Reaction of Benzophenone

Electrochemical Analyses.. Cyclic voltammetry data were collected with a Hokuto Denko HA-501 potentiostat connected to a Graphtec WX 1200 XY recorder. Differential pulse voltammetry (DPV) data were recorded with a Yanaco P-1100 polarographic analyzer connected to a Watanabe WX 4401 XY recorder. All of the electrochemical experiments were carried out at 0°C under Ar atmosphere in dry THF solution containing 0.1 M tetrabutylammonium perchlorate (TBAP) as supporting electrolyte. Conventional three-electrode cells were used, in which the working electrode was a glassy carbon (area = 0.07 cm^2), and the counter electrode was a Pt wire and the reference electrode was Ag/AgCl.

Preparation of *N* -Phenylsulfonylindole (1)

A mixture of indole (5.9 g, 50 mmol), 50 % aq. NaOH (50 mL), H_2O (75 mL), toluene (75 mL) and tetra-n-butylammonium bromide (1.6 g, 5.0 mmol) was stirred at room temperature. Benzenesulfonyl chloride (1.6 g, 5.0 mmol) was added dropwode to

the mixture at 0°C. The mixture was stirred at roomtemperature for 2 h. After evaporation of the solvent, the residue was diluted with H_2O (50 mL) and extracted with Et_2O (50 x 3 mL). the Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using AcOEt-hexane (1 : 5) as an eluet to give *N* -phenylsulfonylindole (**1**) (16.1 g, 84 %) as colorless prisms: mp 76.5-77°C (recrystallized from EtOH, colorless prisms)(lit.[5] mp 76-76.5 °C): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 6.66 (1H, m), 7.10-7.60 (7H, m), 7.80-7.90 (2H, m), 8.0 (1H, d, *J* = 8.8 Hz).

Preparation of *N* -Phenylsulfonyl -1, 2, 3, 4-tetrahydroquinoline (2)

Benzenesulfonyl chloride (3.8 mL, 30 mmol) was added to a pyridine solution of 1,2,3,4-tetrahydroquinoline (2.10 g, 15 mmol) and stirred for 1 h at room temperature. After evaporation of the solvent, the residue was diluted with H_2O (50 mL) and extracted with Et_2O (50 x 3 mL). the Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using AcOEt-hexane (1 : 5) as an eluet to give *N* -phenylsulfonyl-1,2,3,4-tetrahydroquinoline (**2**) (4.10 g, 100 %): mp 61-63°C (recrystallized from n-hexane/ether, colorless prisms): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 1.63 (2H, quint, *J* = 6.0, 6.3 Hz), 2.42 (2H, t, *J* = 6.3 Hz), 3.82 (2H, t, *J* = 6.0 Hz), 7.01-7.08 (2H, m), 7.18-7.22 (1H, m), 7.38-7.43 (2H, m), 7.51-7.61 (3H, m), 7.78-7.81 (1H, m). MS *m/z*: 273 (M^+). HRMS Calcd for $C_{15}H_{15}NO_2S$: 273.08226, Found: 273.0828. *Anal*. Calcd for $C_{15}H_{15}NO_2S$: C, 65.91; H, 5.53; N, 5.12. Found: C, 65.86; H, 5.57; N, 5.09.

Preparation of *N* -Phenylsulfonyl -1, 2, 3, 4-tetrahydroisoquinoline (3)

Benzenesulfonyl chloride (3.83 mL, 30 mmol) was added to a pyridine solution of 1,2,3,4-tetrahydroisoquinoline (2.15 g, 15 mmol) and stirred for 1 h at room temperature. After evaporation of the solvent, the residue was diluted with H_2O (50 mL) and

extracted with Et_2O (50 x 3 mL). the Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using AcOEt-hexane (1 : 5) as an eluet to give *N* -phenylsulfonyl-1,2,3,4-tetrahydroisoquinoline (**3**) (3.77 g, 92 %): mp 156-157°C (recrystallized from ethyl acetate, colorless needles): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 2.93 (2H, t, *J* = 6.0 Hz), 3.39 (2H, t, *J* = 6.0 Hz), 4.28 (2H, s), 7.05-7.09 (2H, m), 7.13-7.16 (2H, m), 7.51-7.60 (3H, m), 7.84-7.87 (2H, m). MS *m/z*: 273 (M^+). HRMS Calcd for $C_{15}H_{15}NO_2S$: 273.08226, Found: 273.0810. *Anal.* Calcd for $C_{15}H_{15}NO_2S$: C, 65.91; H, 5.53; N, 5.12. Found: C, 65.85; H, 5.60; N, 5.11.

General Procedure for Catalytic Deprotection of *N* -Phenylsulfonyl Moiety Using the ET-Ate Complex.

Under Ar atmosphere, *N* -phenylsulfonyl moiety (1.0 mmol) in THF (3 mL) was added dropwise to a THF suspension of ET-ate complex (0.2 mmol; 0.1eq) and magnesium turnings (390 mg, 16 mmol). After allowing to warm to room temperature, the mixture was sitirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with NH_4Cl (30 mL) followed by extraction with $CHCl_3$ (30 mL x 3). The $CHCl_3$ layer was dried over $MgSO_4$ and $CHCl_3$ was removed under reduced pressure. The residue was purified by SiO_2 column chromatography to give a corresponding deprotected product.

General Procedure for the Catalytic Deprotection of Phenols Using the ET-Ate Complex. Under Ar atmosphere, an *O*-allyl compound (1.0 mmol) in THF (3 mL) was added dropwise to a THF suspension of the ET-ate complex (0.2 mmol; 0.1 eq) and magnesium turnings (390 mg, 16 mmol). After allowing to warm to room temperature, the mixture was stirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with aq. NH_4Cl (30 mL) followed by extraction with $CHCl_3$ (30

mL x 3). The $CHCl_3$ layer was dried over $MgSO_4$ and $CHCl_3$ was removed under reduced pressure. The residue was purified by SiO_2 column chromatography to give a corresponding deprotected product.

General Procedure for the Catalytic Half Reduction of the 1,2-Diketone Using the ET-Ate Complex. Under Ar atmosphere, benzil (210.2 mg; 1.0 mmol) in THF (3 mL) was added dropwise to a THF suspension of the ET-ate complex (0.4 mmol; 0.2 eq) and magnesium turnings (390 mg, 16 mmol). After allowing to warm to room temperature, the mixture was stirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with aq. NH_4Cl (30 mL) followed by extraction with $CHCl_3$ (30 mL x 3). The $CHCl_3$ layer was dried over $MgSO_4$ and $CHCl_3$ was removed under reduced pressure. The residue was purified by SiO_2 column chromatography using AcOEt-n-hexane (1 : 10) as an eluent to give benzoin (**17**) (154 mg, 74 %): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 4.55 (1H, d, J = 6.3 Hz), 5.96 (1H, d, J = 6.3 Hz), 7.39 (8H, m), 7.92 (2H, dt, J = 0.6, 7.1 Hz). MS m/z: 212 (M^+). HRMS Calcd for $C_{14}H_{12}O_2$ 212.0837, found 212.0833.

General Procedure for the Catalytic Reduction of the Nitro Compounds Using the ET-Ate Complex. Under Ar atmosphere, 4-nitroanisol (149.0 mg; 1.0 mmol) in THF (3 mL) was added dropwise to a THF suspension of the ET-ate complex (0.4 mmol; 0.067 eq) and magnesium turnings (390 mg, 16 mmol). After allowing to warm to room temperature, the mixture was stirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with aq. NH_4Cl (30 mL) followed by extraction with $CHCl_3$ (30 mL x 3). The $CHCl_3$ layer was dried over $MgSO_4$ and $CHCl_3$ was removed under reduced pressure. The residue was purified by SiO_2 column chromatography using AcOEt-n-hexane (1 : 5) as an eluent to give 4-methoxyaniline (**22**) (107.9 mg, 86 %): mp 53-55°C (recrystallized from Et_2O-n-hexane, colorless prisms,

lit.[3] mp 57-60°C): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 3.42 (2H, br), 3.72 (3H, s), 6.62 (2H, dt, *J* = 2.7, 4.7 Hz), 6.72 (2H, dt, *J* = 2.7, 4.7 Hz). MS *m*/*z*: 123 (M^+). HRMS Calcd for C_7H_9ON 123.0684, found 123.0637.

General Procedure for Reductive Coupling Using the ET-Ate Complex. Under Ar atmosphere, diphenyliodonium chloride (316.3 mg; 1.0 mmol) was added to a THF suspension of the ET-ate complex (0.4 mmol) and magnesium turnings (18 mmol). After allowing to warm to room temperature, the mixture was stirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with aq. NH_4Cl (30 mL) followed by extraction with $CHCl_3$ (30 mL x 3). The $CHCl_3$ layer was dried over $MgSO_4$ and $CHCl_3$ was removed under reduced pressure. The residue was purified by SiO_2 column chromatography to give biphenyl (134.9 mg, 88 %): 300 MHz ^{1}H-NMR ($CDCl_3$ / TMS) δ (ppm): 7.34 (2H, t, *J* = 7.0 Hz), 7.43 (4H, t, *J* = 7.3 Hz), 7.59 (4H, d, *J* = 7.3 Hz).

General Procedure for the Catalytic Intramolecular Coupling Reaction of Aryl Halides Using the ET-Ate Complex. Under Ar atmosphere, Bis(2-bromophenyl)methylamine (0.5 mmol) in THF (3 mL) was added dropwise to a THF suspension of the ET-ate complex (0.2 mmol; 0.1 eq) and magnesium turnings (195 mg, 8 mmol). After allowing to warm to room temperature, the mixture was stirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with aq. NH_4Cl (30 mL) followed by extraction with Et_2O (30 mL x 3). The Et_2O layer was dried over $MgSO_4$ and Et_2O was removed under reduced pressure. The residue was purified by SiO_2 column chromatography using n-hexane as an eluent to give *N*-methylcarbazole: mp 88-90°C (recrystallized from n-hexane, white powder, lit.[4] mp 87 °C). 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 3.87 (3H, s), 7.24 (2H, dt, *J* = 1.1, 7.0 Hz), 7.41 (2H, d, *J* =8.0 Hz), 7.49 (2H, dt, *J* = 1.1, 6.8 Hz), 8.10 (2H, d, *J* = 7.7 Hz).

MS *m/z*: 181 (M^+). HRMS calcd. for $C_{13}H_{11}N$ 181.0891, found 181.0890.

Preparation of *B*is (2-bromophenyl)amine. A mixture of 2-bromoiodobenzene (1.50 mL, 12.0 mmol), 2-bromoaniline (1.72 g, 10.0 mmol), NaO^tBu (1.34 g, 14 mmol), $Pd_2(dba)_3$ (457.9 mg, 0.5 mmol), DPPF (554.4 mg, 1 mmol), and toluene (20 mL) was refluxed for 18 h. After evaporation of the solvent, the residue was diluted with H_2O (50 mL) and extracted with Et_2O (50 x 3 mL). The Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using n-hexane as an eluet to give bis (2-bromophenyl)amine (2.91 g, 87%) as a colorless oil: 300 MHz ^{1}H NMR ($CDCl_3$/TMS) δ (ppm): 7.59 (2H, dd, *J*=1.4, 7.9 Hz), 7.30 (2H, dd, *J*=1.6, 8.2 Hz), 7.22 (2H, dt, *J*=1.1, 7.1 Hz), 6.85 (2H, dt, *J*=1.1, 7.9 Hz), 6.44 (1H, brs). MS: *m/z*: 325 (M^+). HRMS calcd for $C_{12}H_9Br_2N$ 324.9102, found 324.9085.

Preparation of Bis(2-bromophenyl)methylamine. Under Ar atmosphere, to a suspension of sodium hydride (546.0 mg, 13.7 mmol) in dry THF (5 mL), a THF solution of bis(2-bromophenyl)amine (2.80 g, 8.5 mmol) was added and stirred for 30 min at 0°C. The mixture was cooled to 0°C and methyl iodide (0.85 mL, 13.7 mmol) was added at the same temperature. The mixture was then allowed to warm to ambient temperature and then stirred for 3 h. After reaction, the mixture was diluted with H_2O (30 mL) and extracted with Et_2O (50 x 3 mL). The Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using n-hexane as an eluet to give Bis(2-bromophenyl)methylamine (1.75 g, 78 %): mp 105-106°C (recrystallized from n-hexane, colorless prisms): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 7.56 (2H, dd, *J*=1.4, 7.9 Hz), 7.25 (2H, dd, *J*=1.7, 8.0 Hz), 7.01 (dd, *J*=1.7, 8.0 Hz), 6.95 (2H, dt, *J*=1.7, 8.0 Hz), 3.24 (3H, s). MS *m/z*: 339 (M^+). HRMS Calcd for $C_{13}H_{11}Br_2N$ 338.9858 found 338.9273.

Anal Calcd for $C_{13}H_{11}Br_2N$: C, 45.78; H, 3.25; N, 4.11; Br, 46.86. Found: C, 45.87; H, 3.22; N, 4.08; Br, 46.90.

Preparation of Bis(2-chlorophenyl)amine. A mixture of 2-chloroiodobenzene (1.47 g, 12 mmol), 2-chloroaniline (1.27 g, 10 mmol), NaO^{t}Bu (1.34 g, 14 mmol), $Pd_2(dba)_3$ (457.9 mg, 0.5 mmol), DPPF (554.4 mg, 1 mmol) and toluene (20 mL) was refluxed for 18 h. After evaporation of the solvent, the residue was diluted with H_2O (50 mL) and extracted with Et_2O (50 x 3 mL). The Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using hexane as an eluent to give bis(2-chlorophenyl)amine (2.26 g, 94 %) as a colorless oil: 300 MHz ^{1}H NMR ($CDCl_3$/TMS) δ (ppm):7.41 (2H, dd, J = 8.0, 1.4 Hz), 7.32 (2H, dd, J = 8.2, 1.4 Hz), 7.18 (2H, dt, J = 8.2, 1.1 Hz), 6.91 (2H, dt,J = 7.7, 1.6 Hz), 6.43 (1H, s). MS: m/z: 237 (M^+). HRMS calcd for $C_{12}H_9Cl_2N$ 237.0113. found 237.0101.

Preparation of Bis(2-chlorophenyl)methylamine. Under Ar atmosphere, to a suspension of sodium hydride (546.0 mg, 13.7 mmol) in dry THF (5 mL), a THF solution of di (2-chlorophenyl)amine (2.19 g, 9.1 mmol) was added and stirred for 30 min at 0°C. The mixture was cooled to 0°C and methyliodide (0.85 mL, 13.7 mmol) was added at the temperature. The mixture was then allowed to warm to ambient temperature and then srirred for 3 h. After reaction, the mixture was diluted with H_2O (30 mL) and extracted with Et_2O (50 x 3 mL). The Et_2O layer was dried over $MgSO_4$ and the Et_2O was removed under reduced pressure. The residue was purefied by SiO_2 column chromatography using hexane as an eluet to give bis(2-chlorophenyl)methylamine (1.75 g, 78 %): mp. 79-81°C (recrystallized from n-hexane, colorless prisms): 300 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 3.24 (3H, s), 6.99-7.04 (4H, m), 7.20 (2H, m), 7.36 (2H, dd, J = 8.2, 1.6 Hz). MS m/z: 251 (M^+). HRMS Calcd for $C_{13}H_{11}NCl_2$: 251.0269 found: 251.0242. *Anal* Calcd for $C_{13}H_{11}Cl_2N$: C, 61.93; H, 4.40; N, 5.56, Cl, 28.12. Found: C, 61.67; H, 4.38; N, 5.52, Cl, 28.23.

General Procedure for the Catalytic Reductive Coupling of the Carbonyl Compounds.

Under Ar atmosphere, acetophenone (0.5 mmol) in THF (3 mL) was added dropwise to a THF suspension of the ET-ate complex (0.2 mmol; 0.1 eq) and magnesium turnings (195 mg, 8 mmol) at -78°C. After allowing to warm to 0°C, the mixture was sitirred for 12 h. The solvent was removed under reduced pressure and the residue was treated with NH_4Cl (30 ml) followed by extraction with Et_2O (30 ml x 3). The Et_2O layer was dried over $MgSO_4$ and Et_2O was removed under reduced pressure. The residue was purified by SiO_2 column chromatography using AcOEt-hexane (1 : 5) as an eluent to give a mixture of *dl* and *meso* isomers of 2,3-diphenyl-2,3-butandiol.

1,2-Diphenyl-*1,2*-ethanediol (*dl* and [*meso*]) 400 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 2.28 [2.29] (2H, brs), 4.59 [4.72] (2H, s), 7.01-7.23 (10H, m).

1,2-Bis(4-methoxyphenyl)-*1,2*-ethanediol (*dl* and [*meso*]) 400 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 2.1 [2.8] (2H, brs), 3.75 [3.79] (6H, s), 4.63 [4.72] (2H, s), 6.73-7.26 (8H, m).

1,2-Bis(4-fluorophenyl)-*1,2*-ethanediol (*dl* and [*meso*]) 400 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 3.56 [2.4] (2H, brs), 4.49 [4.8] (2H, s), 6.99-6.81 [7.0-7.4] (8H, m).

2,3-Diphenyl-*2,3*-buthanediol (*dl* and [*meso*]) 400 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 1.39 [1.46] (6H, s), 2.23 [2.52] (2H, brs), 7.06-7.11 (10H, m).

2,3-Bis(*4*-methylphenyl)-*2,3*-buthanediol (*dl* and [*meso*]) 400 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 1.44 [1.51] (6H, s), 2.26 [2.52] (2H, brs), 2.32 [2.31] (6H, s), 7.06-7.11 (8H, m).

2,3-Bis(*4*-methoxyphenyl)-*2,3*-buthanediol (*dl* and [*meso*]) 400 MHz ^{1}H-NMR ($CDCl_3$/TMS) δ (ppm): 1.42 [1.50] (6H, s), 3.0 [2.7] (2H, brs), 3.76 [3.8] (6H, s), 6.7 (4H, m), 7.2 (4H, m).

References for *Supporting Information*

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