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Electronic Supplementary Information

Regioselective lactonization of unsymmetrical 1,4-diols: an efficient access to lactone lignans

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1) General Methods

All manipulations of oxygen and moisture-sensitive materials were conducted under purified argon atmosphere (BASF–Catalyst R3-11) using standard Schlenk techniques. Unless otherwise noted, all reagents and solvents were purchased and used as delivered. All reactions were performed in commercially available anhydrous solvents unless otherwise specified. Column chromatography was performed using Kanto spherical silica gel 60N (63-210 μm). The ^{1}H, and $^{13}C\{^{1}H\}$, and $^{31}P\{^{1}H\}$ NMR spectra were recorded using $CDCl_3$ at 25 ºC on a JEOL LA 300 and ECA 400 spectrometers and chemical shifts were referenced to $(CH_3)_4Si$ (0.0 ppm), $(CH_3)_4Si$ (0.0 ppm), and H_3PO_4 (0.0 ppm), respectively, unless otherwise specified. The coupling constants were described in herz (Hz). Elemental analyses were performed by the Analytical Facility at the Research Laboratory of Resources Utilization, Tokyo Institute of Technology or on a Perkin-Elmer 2400II CHN analyzer.

Chemicals The Cp*RuCl(PN) complexes **1a**,[S1] **1b,**[S2] **1d–e**,[S2] and **1g–i**,[S2] and the PN ligands, **2c**,[S2] **2j**,[S2] **2k**,[S2] and **2l**[S2] were prepared as we reported previously. The PN ligand **2m** (Aldrich 43163) was purchased and used as delivered. The PN ligand **2n** was prepared according to the literature method[S3] with slight modifications. Unsymmetrical 1,4-diols **3a–d** were prepared as we reported previously.[S4] 2,3-bis(hydroxymethyl)-1-phenylnaphthalene (**8**)[S5] was prepared in 40% yield by the reduction of 1-phenyl-2,3-naphthalenedicarboxylic anhydride (Aldrich 456047) with $LiAlH_4$ (3.0 equiv) in refluxing THF for 13 h. In a similar manner, the diol **9** was prepared in 59% yield by $LiAlH_4$ reduction of 6,7-methylenedioxy-1-(3,4-methylenedioxyphenyl)naphthalene-2,3-dicarboxylic anhydride,[S6a] which was prepared from piperonal (Aldrich P49104) in three steps including Corey-Fuchs reaction followed by CO_2 trap[S6a] and dehydrative cyclization with acetic anhydride.[S6b] The reference S6 details preparative methods and spectroscopic data for all synthetic intermediates except those for 3,4-(methylenedioxyphenyl)propiolic acid. Therefore, only its NMR spectra are shown. ^{1}H NMR (300.40 MHz, CD_3OD) δ 6.02 (s, 2H), 6.86 (d, J = 8, 1H), 7.00 (d, J = 2), 7.14 (dd, J = 8 & 2, 1H); $^{13}C\{^{1}H\}$ NMR (75.4 MHz, CD_3OD) δ 80.7, 87.3, 103.4, 109.8, 113.2, 113.8, 129.8, 149.3, 151.6, 156.8.

2) Preparation of Cp*RuCl(PN) complexes

Preparation of $Ph_2PC(CH_2)_3CH_2NH_2$ (2f) The PN ligand **2f** was newly synthesized from $Ph_2POC_2H_5$ in four steps including cyanomethylation,[S7a] double alkylation of the methylene group,[S7b,c,d] deoxygenation of the phosphorus group,[S7e,f] and reduction of the cyano group as follows. $Ph_2P(O)CH_2CN$ was prepared by heating $Ph_2POC_2H_5$ (Aldrich 149489, 3.12 g, 13.6 mmol) in $BrCH_2CN$ (Aldrich 242489, 1.89 mL, 27.1 mmol) at 160 ºC for 4 h followed by collection of the resulting precipitates by filtration through a membrane filter (Advantec, PTFE T050A047A) using *n*-hexane (3.11 g, 95% yield; ^{1}H NMR (399.8 MHz) δ 3.34 (d, J = 15 Hz, 2H), 7.54–7.58 (m, 4H), 7.62–7.67 (m, 2H), 7.82–7.87 (m, 4H); $^{13}C\{^{1}H\}$ NMR (75.4 MHz) δ 21.3 (d, J = 62), 113.4 (d, J = 7), 129.1 (d, J = 13), 129.6 (d, J = 106), 131.1 (d, J = 10), 133.2 (d, J = 3); $^{31}P\{^{1}H\}$ NMR (162 MHz) δ +25.1). Then, $Ph_2P(O)CH_2CN$ (847 mg, 3.51 mmol) was subjected to the reaction with 1,3-dibromopropane (Aldrich 125903, 0.72 mL, 7.0 mmol) in DMSO (Kanto 10378-01, 35 mL) containing K_2CO_3 (1.94 g, 14.1 mmol) at room temperature for 62 h under air. The reaction mixture was quenched by adding water and extracted with diethyl ether. The combined extracts were washed with water, dried over Na_2SO_4, and filtered through a pad of cotton wool. Evaporation of the filtrate followed by recrystallization of the residue from CH_2Cl_2/*n*-hexane to give

$Ph_2P(O)C(CH_2)_3CN$ as colorless solids (650 mg, 66% yield, ^1H NMR (399.8 MHz) δ 2.23–2.28 (m, 1H), 2.40–2.53 (m, 3H), 3.00–3.09 (m, 2H), 7.49–7.62 (m, 6H), 7.83–7.88 (m, 4H); $^{13}C\{^1H\}$ NMR (100.5 MHz) δ 18.3 (d, *J* = 9), 27.7 (d, *J* = 3), 34.2 (d, *J* = 70), 122.0 (d, *J* = 5), 128.7 (d, *J* = 102), 128.8 (d, *J* = 12), 131.7 (d, *J* = 9), 132.8 (d, *J* = 3); $^{31}P\{^1H\}$ NMR (162 MHz) δ +29.2; Anal. calcd. for $C_{17}H_{16}NOP$: C, 72.59; H, 5.73; N, 4.98. Found: C, 72.27; H, 5.75; N 5.08). Next, $Ph_2P(O)C(CH_2)_3CN$ (326 mg, 1.16 mmol) was placed in a 50-mL pressure bottle and dissolved in toluene (20 mL). To this was added trichlorosilane (Wako 307-60742, 2.34 mL, 23.2 mmol) at 0 ºC and the resulting mixture was heated at 150 ºC for 38 h and then cooled. The reaction mixture was concentrated under reduced pressure, and the residue was diluted with CH_2Cl_2. The insoluble materials were removed by filtration through a pad of Celite under argon. The filtrate was concentrated *in vacuo* to give $Ph_2PC(CH_2)_3CN$ as viscous oil (291 mg, ca. 95% yield, ^1H NMR (399.8 MHz) δ 2.27–2.39 (m, 2H), 2.43–2.53 (m, 2H), 2.64–2.74 (m, 2H), 7.30–7.55 (m, 10H); $^{31}P\{^1H\}$ NMR (162 MHz) δ +6.1), which was diluted with THF (5.0 mL) and to this was slowly added BH_3•THF (Kanto 05012-25, 1.06 M in THF, 3.6 mL). The resulting mixture was refluxed for 20 h, cooled, and then quenched by adding degassed methanol (2.5 mL) slowly. The mixture was concentrated under reduced pressure to leave white solids, which was dissolved in degassed 2 M HCl aq.(15 mL) and then heated at 100 ºC for 4 h. The mixture was cooled, basicified to pH 11 by adding finely-crushed KOH slowly, and then extracted with degassed ethyl acetate under argon repeatedly. The combined extracts were dried over $MgSO_4$ and filtered through a pad of Celite under argon. The filtrate was concentrated *in vacuo* to give $Ph_2PC(CH_2)_3CH_2NH_2$ as viscous oil (254 mg, 86% yield), which was used for the complexation without further purification. ^1H NMR (399.8 MHz) δ 1.36–1.47 (m, 3H), 1.81–1.91 (m, 1H), 2.03–2.12 (m, 2H), 2.19–2.28 (m, 2H), 3.03 (d, *J* = 10, 2H), 7.32–7.36 (m, 6H), 7.43–7.47 (m, 4H); $^{31}P\{^1H\}$ NMR (162 MHz) δ –2.1.

Preparation of Cp*RuCl(PN) complexes, 1f, 1n, and 1m. By the similar procedure to that we reported previously for **1a**,[S1,2] these complexes were prepared by adding a solution of the corresponding PN compounds in degassed CH_2Cl_2 (0.1–0.2 M) to a solution of an equimolar amount of Cp*RuCl(isoprene)[S8] in degassed CH_2Cl_2 (0.1–0.2 M) over a period of 1 h and by stirring for additional 1 h at 30 °C followed by the standard work-up including concentration, washing with anhydrous *n*-hexane or diethyl ether, and drying under vacuum.

Cp*RuCl[$Ph_2PC(CH_2)_3CH_2NH_2$] (1f) 81% yield; ^1H NMR (399.8 MHz, CD_2Cl_2) δ 1.37 (s, 15H), 1.56 (brs, 1H), 1.72–1.82 (m, 2H), 1.90–2.00 (m, 1H), 2.13–2.20 (m, 1H), 2.47–2.57 (m, 1H), 2.84–2.90 (m, 1H), 3.03 (brs, 1H), 3.17–3.18 (m, 1H), 3.51–3.62 (m, 1H), 7.07 (brs, 2H), 7.29 (brs, 3H), 7.45–7.49 (m, 3H), 8.06–8.10 (m, 2H); $^{13}C\{^1H\}$ NMR (100.5 MHz, CD_2Cl_2) δ 9.56 (s), 16.0 (d, *J* = 7.7), 27.2 (d, *J* = 5.8), 30.5 (s), 45.0 (d, *J* = 12.5), 60.0 (d, *J* = 18.2), 80.8 (s), 127.6 (d, *J* = 9.6), 127.6 (d, *J* = 7.7), 128.2 (s), 129.9 (s), 132.1 (d, *J* = 8.6), 132.8 (d, *J* = 33.5), 137.0 (d, *J* = 12.5), 139.5 (d, *J* = 34.5); $^{31}P\{^1H\}$ NMR (161.8 MHz) δ +71.9; Anal. calcd. For $C_{27}H_{35}ClNPRu$•$0.5H_2O$: C, 58.95; H, 6.60; N, 2.55. Found: C, 58.70; H, 6.66; N, 2.48.

Cp*RuCl[$Ph_2P(CH_2)_3NH_2$] (1m) 90% yield; ^1H NMR (300.4 MHz, CD_2Cl_2) δ 1.35 (s, 15H), 1.68–1.97 (m, 2H), 2.28 (brs, 1H), 2.39–2.56 (m, 2H), 2.80 (brs, 1H), 3.12 (brs, 1H), 3.27 (t, *J* = 10.8, 1H), 7.32–7.48 (m, 6H), 7.54–7.63 (m, 2H), 7.65–7.77 (m, 2H); $^{13}C\{^1H\}$ NMR (100.5 MHz, CD_2Cl_2) δ 9.7 (s), 25.7 (d, *J* = 3.1), 28.0 (d, *J* = 17.9), 41.0 (d, *J* = 2.7), 80.7 (d, *J* = 2.7), (Since signals in the range from 127 to 134 ppm were too complicated due to the intensive couplings with the phosphorous nuclei,

only those for $C_5(CH_3)_5$, and $(CH_2)_3$ were assigned); $^{31}P\{^1H\}$ NMR (121.6 MHz, CD_2Cl_2) δ +34.9; Anal. calcd. for $C_{25}H_{33}ClNPRu$: C, 58.30; H, 6.46; N, 2.72. Found: C, 58.56; H, 6.33; N, 2.74.

Cp*RuCl[(2-Ph$_2$P)C$_6$H$_4$CH$_2$NH$_2$] (1n). 75% yield; ^1H NMR (300.4 MHz, CD_2Cl_2) δ 1.44 (d, *J* = 1.3 15H), 3.03 (brs, 1H), 3.25 (brs, 1H), 3.36–3.44 (m, 1H), 3.92–4.19 (m, 1H), 7.04–7.05 (m, 1H), 7.18–7.80 (m, 13H); $^{31}P\{^1H\}$ NMR (121.6 MHz, CD_2Cl_2) δ +44.5; Anal. calcd. for $C_{29}H_{33}ClNPRu•1/3CH_2Cl_2$: C, 59.57; H, 5.74; N, 2.37. Found: C, 59.65; H, 5.56; N, 2.39.

3) General procedure for the lactonization of diols

A degassed solution of **3a** (213.0 mg, 1.18 mmol) in acetone (2.4 mL) was transferred to a mixture of **1f** (6.4 mg, 11.8 μmol) and KO*t*-Bu (1.3 mg, 11.8 μmol) placed in a 20-mL Schlenk tube. The reaction mixture was vigorously stirred at 30 °C for 1 h and quenched by filtration through a plug of silica gel. The filtrate was evaporated to give an isomeric mixture of **4a** and **5a** (207 mg, >99% yield), whose molar ratio was determined as 90:10 by integration of well-resolved signals in ^1H NMR spectroscopy.

β-benzyl-γ-butyrolactone (4a)[S9a] ^1H NMR (300.4 MHz, $CDCl_3$) δ 2.19 (dd, *J* = 17.4, 6.7 Hz, 1H), 2.53 (dd, *J* = 17.4, 7.9 Hz, 1H), 2.69–2.81 (m, 3H), 3.97 (dd, *J* = 9.0, 6.0 Hz, 1H), 4.27 (d, *J* = 9.0, 6.8 Hz, 1H), 7.07–7.28 (m, 5H).

α-benzyl-γ-butyrolactone (5a)[S4b] ^1H NMR (399.8 MHz, $CDCl_3$) δ 1.94–2.04 (m, 1H), 2.21–2.28 (m, 1H), 2.75 (dd, *J* = 13.7, 9.5 Hz, 1H), 2.81–2.89 (m, 1H), 3.25 (dd, *J* = 13.7, 4.0 Hz, 1H), 4.11–4.25 (m, 2H), 7.20–7.26 (m, 3H), 7.29–7.33 (m, 2H).

β-(*p*-methoxybenzyl)-γ-butyrolactone (4b)[S9b] ^1H NMR (300.4 MHz, $CDCl_3$) δ 2.27 (dd, *J* = 17.5, 6.6 Hz, 1H), 2.52–2.72 (m, 4H), 3.80 (s, 3H), 4.04 (dd, *J* = 8.6, 5.9 Hz, 1H), 4.31 (d, *J* = 8.6, 6.7 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 2H).

α-(*p*-methoxybenzyl)-γ-butyrolactone (5b)[S4b] ^1H NMR (399.8 MHz, $CDCl_3$) δ 1.95–2.03 (m, 1H), 2.20–2.28 (m, 1H), 2.73 (dd, *J* = 13.4, 9.1 Hz, 1H), 2.77–2.90 (m, 1H), 3.16 (dd, *J* = 13.4, 4.0 Hz, 1H), 3.79 (s, 3H), 4.11–4.23 (m, 2H), 6.84 (d, *J* = 8.6 Hz, 2H), 7.12 (d, *J* = 8.6 Hz, 2H).

β-(*m*-benzyloxybenzyl)-γ-butyrolactone (4c)[S9c] ^1H NMR (300.4 MHz, $CDCl_3$) δ 2.19 (dd, *J* = 17.3, 6.8 Hz, 1H), 2.51 (dd, *J* = 17.6, 7.9 Hz, 1H), 2.60–2.76 (m, 3H), 3.93 (dd, *J* = 9.0, 5.9 Hz, 1H), 4.23 (dd, *J* = 9.0, 6.8 Hz, 1H), 4.98 (s, 2H), 6.65–6.80 (m, 3H), 7.15–7.20 (m, 1H), 7.25–7.40 (m, 5H).

α-(*m*-benzyloxybenzyl)-γ-butyrolactone (5c)[S4b] ^1H NMR (399.8 MHz, $CDCl_3$) δ 1.90–2.00 (m, 1H), 2.17–2.25 (m, 1H), 2.71 (dd, *J* = 13.7, 9.5 Hz, 1H), 2.78–2.86 (m, 1H), 3.22 (dd, *J* = 13.7, 4.0 Hz, 1H), 4.10–4.16 (m, 1H), 4.19–4.24 (m, 1H), 5.06 (s, 2H), 6.81–6.88 (m, 3H), 7.21–7.26 (m, 1H), 7.31–7.44 (m, 5H).

β-(3,4-methylenedioxyphenylmethyl)-γ-butyrolactone (4d)[S9d] ^1H NMR (300.4 MHz, $CDCl_3$) δ 2.19 (dd, *J* = 17.4, 6.7 Hz, 1H), 2.52 (dd, *J* = 17.4, 8.0 Hz, 1H), 2.58–2.74 (m, 3H), 3.94 (dd, *J* = 9.2, 6.0 Hz, 1H), 4.25 (dd, *J* = 9.2, 6.8 Hz, 1H), 5.87 (s, 2H), 6.52–6.70 (m, 3H).

α-(3,4-methylenedioxyphenylmethyl)-γ-butyrolactone (5d)[S4b] ^1H NMR (399.8 MHz, $CDCl_3$) δ 1.93–2.04 (m, 1H), 2.21–2.29 (m, 1H), 2.70 (dd, *J* = 13.8, 9.2 Hz, 1H), 2.75–2.83 (m, 1H), 3.14 (dd, *J* = 13.8, 4.3 Hz, 1H), 4.12–4.18 (m, 1H), 4.21–4.26 (m, 1H), 5.94 (s, 2H), 6.64 (dd, *J* = 7.9, 1.8 Hz, 1H), 6.69 (d, *J* = 1.8 Hz, 1H), 6.74 (d, *J* = 7.9 Hz, 1H).

10[S9e] ^{1}H NMR (300.4 MHz, $CDCl_3$) δ 5.26 (s, 2H), 7.38–7.40 (m, 2H), 7.48–7.62 (m, 5H), 7.79–7.83 (m, 1H), 8.07–8.10 (m, 1H), 8.52 (s, 1H).

Justicidin E[S9f] ^{1}H NMR (300.4 MHz, DMSO-d_6) δ 5.26 (d, J = 15.0 Hz, 1H), 5.33 (*d*, J = 15.0 Hz, 1H), 6.13 (s, 2H), 6.19 (s, 2H), 6.90 (d, J = 7.8 Hz, 1H), 7.00 (s, 1H), 7.04 (s, 1H), 7.09 (dd, J = 7.8, 2.9 Hz, 1H), 7.63 (d, J = 2.9 Hz, 1H), 8.36 (s, 1H).

4) Preparation of (±)-*O,O'*-dibenzyl enterolactone

To a solution of diisopropylamine (0.30 mL, 2.13 mmol) in THF (8.0 mL) was slowly added *n*-BuLi (Kanto 04937, 1.58 M in *n*-hexane, 1.12 mL, 1.77 mmol) at –78 °C and subsequently HMPA (0.37 mL, 2.13 mmol) and the resulting mixture was stirred at the same temperature for 30 min. To this was added a solution of a mixture of **4c** and **5c** (92:8, 0.20 g, 0.71 mmol) in THF (3.0 mL) and the resulting mixture was further stirred at –78 °C for 90 min. Then a solution of 3-(benzyloxy)-1-(bromomethyl)benzene[S10] (0.39 g, 1.42 mmol) in THF (2.0 mL) was added and the mixture was allowed to warm up to room temperature. After stirring at 30 °C for additional 24 h, the mixture was quenched by adding sat. NH_4Cl aq. and extracted with ethyl acetate, washed with brine, dried over Na_2SO_4, and filtered through a pad of Celite. The filtrate was concentrated and the residue was chromatographed on silica gel eluting with gradient *n*-hexane/ethyl acetate (10:1 to 6:1 (v/v)) to give the title compound (135.7 mg, 43% yield) R_f = 0.69, *n*-hexane/ethyl acetate = 2/1; ^{1}H NMR (300.4 MHz, $CDCl_3$) δ 2.36–2.58 (m, 4H), 2.87 (dd, J = 13.9, 7.2 Hz, 1H), 3.03 (dd, J = 13.9, 4.9 Hz, 1H), 3.79 (dd, J = 8.5, 7.8 Hz, 1H), 4.02 (dd, J = 7.8, 6.8 Hz, 1H), 4.99 (s, 2H), 5.00 (s, 2H), 6.56–6.58 (m, 2H), 6.74–6.98 (m, 4H), 7.13–7.41 (m, 12H); ^{13}C{^{1}H} NMR (75.4 MHz, $CDCl_3$) δ 35.0, 38.4, 41.1, 46.2, 69.8, 71.0, 112.8, 113.3, 115.3, 115.6, 121.1, 121.8, 127.4, 127.8, 127.9, 128.4, 128.5, 129.6, 136.7, 136.8, 139.2, 139.5, 158.9, 178.4.[S10]

5) References

S1 M. Ito, M. Hirakawa, A. Osaku and T. Ikariya, *Organometallics,* 2003, **22**, 4190–4192.

S2 M. Ito, A. Osaku, C. Kobayashi, A. Shiibashi and T. Ikariya, *Organometallics,* 2009, **28**, 390–393.

S3 (*a*) S. J. Coote, G. J. Dawson, C. G. Frost and M. J. Williams, *Synlett,* 1993, 509–510; (*b*) A. Brunner, F. M. Kuhnle and D. Seebach, *Helv. Chim. Acta,* 1996, **79**, 319–345; (*c*) M. Hingst, M. Tepper and O. Stelzer, *Eur. J. Inorg. Chem.,* 1998, 73–82.

S4 (*a*) T. Ikariya, M. Ito, A. Shiibashi and T. Ootsuka, *PCT Int. Pat. Appl. WO*, 2010/004883 A1, Jan 14, 2010; (*b*) M. Ito, T. Ootsuka, R. Watari, A. Shiibashi, A. Himizu and T. Ikariya, *manuscript in preparation.*

S5 C. L. Bradford, S. A. Fleming and S. C. Ward, *Tetrahedron Lett.,* 1995, **36**, 4189–4192.

S6 (*a*) H. Sai, T. Ogiku, T. Nishitani, H. Hiramatsu, H. Horikawa and T. Iwasaki, *Synthesis,* 1995, 582–586; (*b*) T. L. Holms and R. Stevenson, *J. Chem. Soc. C*, 1971, 2091–2094.

S7 (*a*) M. Regitz and W. Anshutz, *Chem. Ber.* 1969, **102**, 2216–2229; (*b*) N. M. Vinogradova, I. L. Odinets, K. A. Lyssenko, A. A. Korlyukov, P. V. Petrovskii, G. V. Roeschenthaler and T. A. Mastryukova, *Mendeleev Commun.* 2001, **12**, 133–135; (*c*) P. V. Kazakov, I. L. Odinets, A. P. Laretina, T. M. Shcherbina, P. V. Petrovskii, L. V. Kovalenko and T. A. Mastryukova, *Izv. Akad. Nauk SSSR,*

Ser. Khim. 1990, **8**, 1873–1880; (*d*) J. Nasser, E. About-Jauder and N. Collignon, *Phosphous and Sulfur*, 1990, **54**, 171–179; (*e*) N. M. Vinogradova, I. L. Odinets, K. A. Lyssenko, M. P. Pasechnik, P. V. Petrovskii and T. A. Mastryukova, *Mendeleev Commun.* 2001, **11**, 219–221; (*f*) O. Dahl and F. K. Jensen, *Acta Chim. Scand.* 1975, **B29**, 863–866.

S8 P. J. Fagan, W. S. Mahoney, J. C. Calabrese and I. D. Williams, *Organometallics,* 1990, **9**, 1843–1852.

S9 (*a*) G. Bencivenni, T. Lanza, R. Leardini, M. Minozzi, D. Nanni, P. Spagnolo and G. Zanardi, *Org. Lett.,* 2008, **10**, 1127–1130; (*b*) S. Koul, B. Singh, S. C. Taneja and G. N. Qazi, *Tetrahedron,* 2003, **59**, 3487–3491; (*c*) A. Srikrishna, S. Danieldoss, S. Venkateswarlu and J. A. Sattigeri, *J. Ind. Chem. Soc.,* 1997, **74**, 864–869; (*d*) J. W. Bode, M. P. Doyle, M. Protopopova and Q.-L. Zhou, *J. Org. Chem.,* 1996, **61**, 9146–9155; (*e*) N. Eghbali, J. Eddy and P. T. Anastas, *J. Org. Chem.,* 2008, **73**, 6932–6935; (*f*) S. R. Flanagan, D. C. Harrowven and M. Bradley, *Tetrahedron,* 2002, **58**, 5989–6001.

S10 A. van Oeveren, J. F. G. A. Jansen and B. L. Feringa, *J. Org. Chem.* 1994, **59**, 5999–6007.