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Supporting Information

4-(3,4,5-Trimethoxybenzyl)- γ -butyrolactone (5b). 59% yield from 3,4,5-trimethoxybenzaldehyde; mp 98-9 °C (AcOEt-hexane); IR (KBr) 1771 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.30 (dd, 1H, $J=6.5, 17.3$ Hz), 2.64 (dd, 1H, $J=7.9, 17.3$ Hz), 2.68-2.98 (m, 3H), 3.83 (s, 3H), 3.85 (s, 6H), 4.06 (dd, 1H, $J=5.6, 9.1$ Hz), 4.36 (dd, 1H, $J=6.6, 9.1$ Hz), 6.36 (s, 2H); MS m/z (relative intensity, %): 266 (M^+ , 50), 151 (73), 135 (100). Anal. Calcd for $\text{C}_{14}\text{H}_{18}\text{O}_5$: C, 63.15; H, 6.81. Found: C, 63.08; H, 6.53.

(3*R,4*R**)-3-(3,4-Methylenedioxybenzyl)-4-(3,4,5-trimethoxybenzyl)- γ -butyrolactone (1m).** 88% yield from **5b**; mp 110-1 °C (AcOEt) (Lit.^{11d} amorphous solid); IR (KBr) 1763 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.41-2.68 (m, 4H), 2.85 (dd, 1H, $J=6.3, 13.8$ Hz), 2.99 (dd, 1H, $J=4.6, 13.8$ Hz), 3.82 (s, 9H), 3.90 (dd, 1H, $J=6.8, 9.2$ Hz), 4.19 (dd, 1H, $J=6.6, 9.4$ Hz), 5.90-5.98 (m, 2H), 6.20 (s, 2H), 6.58 (dd, 1H, $J=1.6, 9.2$ Hz), 6.61 (s, 1H), 6.72 (d, 1H, $J=8.1$ Hz); MS m/z (relative intensity, %): 400 (M^+ , 8), 266 (20), 182 (100), 135 (70). Anal. Calcd for $\text{C}_{22}\text{H}_{24}\text{O}_7$: C, 65.99; H, 6.04. Found: C, 65.75; H, 5.84.

(3*R,4*R**)-3-(3-Methoxy-4-benzyloxybenzyl)-4-(3,4-dimethoxybenzyl)- γ -butyrolactone (1n).** 86% yield from **5a**; mp 94-5 °C (AcOEt-hexane) (Lit.^{2c} amorphous solid); IR (KBr) 1767 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.35-2.72 (m, 4H), 2.81-3.05 (m, 2H), 3.80 (s, 3H), 3.84 (s, 3H), 3.85 (s, 3H), 3.78-3.97 (m, 1H), 4.11 (dd, 1H, $J=6.8, 9.4$ Hz), 5.13 (s, 2H), 6.47 (d, 1H, $J=1.9$ Hz); 6.52 (dd, 1H, $J=1.9, 8.1$ Hz), 6.59 (dd, 1H, $J=1.9, 8.1$ Hz), 6.71 (d, 1H, $J=2.4$ Hz), 6.74 (d, 1H, $J=9.4$ Hz), 6.79 (d, 1H, $J=8.2$ Hz), 7.22-7.48 (m, 5H); MS m/z (relative intensity, %): 462 (M^+ , 35), 151 (47), 91 (100). Anal. Calcd for $\text{C}_{28}\text{H}_{30}\text{O}_6$: C, 72.71; H, 6.54. Found: C, 72.45; H, 6.48.

(3*R,4*R**)-3-benzyl-4-(3,4,5-trimethoxybenzyl)- γ -butyrolactone (1o).** 89% yield from **5b**; mp 98-9 °C (AcOEt-hexane); IR (KBr) 1761 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.40-2.71 (m, 4H),

2.94 (dd, 1H, $J=7.1, 13.9$ Hz), 3.11 (dd, 1H, $J=5.2, 13.9$ Hz), 3.80 (s, 6H), 3.82 (s, 3H), 3.81-3.97 (m, 1H), 4.10-4.22 (m, 1H), 6.16 (s, 2H), 7.11-7.37 (m, 5H); MS m/z (relative intensity, %): 356 (M^+ , 93), 182 (100), 167 (19), 151 (23), 91 (45). Anal. Calcd for $C_{21}H_{24}O_5$: C, 70.77; H, 6.79. Found: C, 70.83; H, 6.69.

(3*S,4*S**)-3-Hydroxy-3-(3,4-methylenedioxybenzyl)-4-(3,4,5-trimethoxybenzyl)- γ -butyrolactone (1e).** mp 165-6 °C (AcOEt); IR (KBr) 3445, 1751 cm^{-1} ; 1H NMR (δ in $CDCl_3$) 2.37-2.64 (m, 3H), 2.84-3.02 (m, 1H), 2.92 (d, 1H, $J=13.8$ Hz), 3.09 (d, 1H, $J=13.8$ Hz), 3.83 (s, 3H), 3.84 (s, 3H), 4.07 (br. d, 2H, $J=6.4$ Hz), 5.94 (s, 2H), 6.20 (s, 2H), 6.60 (dd, 1H, $J=1.7, 7.9$ Hz), 6.67 (d, 1H, $w=1.5$ Hz), 6.74 (d, 1H, $J=7.9$ Hz); MS m/z (relative intensity, %): 416 (M^+ , 23), 181 (16), 135 (100). Anal. Calcd for $C_{22}H_{24}O_8$: C, 63.45; H, 5.81. Found: C, 63.43; H, 5.65.

(3*R,4*S**)-3-Hydroxy-3-(3,4-methylenedioxybenzyl)-4-(3,4,5-trimethoxybenzyl)- γ -butyrolactone (2e).** mp 141-2 °C (AcOEt); IR (KBr) 3445, 1751 cm^{-1} ; 1H NMR (δ in $CDCl_3$) 2.62 (dd, 1H, $J=11.3, 13.2$ Hz), 2.77 (s, 1H), 2.85-3.07 (m, 3H), 3.14 (dd, 1H, $J=3.9, 13.3$ Hz), 3.84 (s, 3H), 3.86 (s, 6H), 3.95 (dd, 1H, $J=9.3, 10.2$ Hz), 4.24 (dd, 1H, $J=7.7, 9.2$ Hz), 5.96 (s, 2H), 6.39 (s, 2H), 6.67 (dd, 1H, $J=1.7, 7.9$ Hz), 6.72 (d, 1H, $J=1.4$ Hz), 6.78 (d, 1H, $J=7.8$ Hz); MS m/z (relative intensity, %): 416 (M^+ , 17), 181 (15), 135 (100). Anal. Calcd for $C_{22}H_{24}O_8$: C, 63.45; H, 5.81. Found: C, 63.39; H, 5.52.

(3*S,4*S**)-3-Hydroxy-3-(3-methoxy-4-benzyloxybenzyl)-4-(3,4-dimethoxybenzyl)- γ -butyrolactone (1f).** mp 78-9 °C (AcOEt-hexane) [Lit.^{2c} (-)-1f: mp 76-8 °C (ether)]; IR (KBr) 3429, 1771 cm^{-1} ; 1H NMR (δ in $CDCl_3$) 2.41-2.63 (m, 3H), 2.85-3.01 (m, 1H), 2.93 (d, 1H, $J=13.7$ Hz), 3.10 (d, 1H, $J=13.7$ Hz), 3.83 (s, 3H), 3.85 (s, 3H), 3.86 (s, 3H), 3.92-4.12 (m, 2H), 5.13 (s, 2H), 6.57-6.89 (m, 6H), 7.21-7.48 (m, 5H); MS m/z (relative intensity, %): 478 (M^+ , 23), 277 (49), 151 (48), 91 (100). Anal. Calcd for $C_{28}H_{30}O_7$: C, 70.28; H, 6.32. Found: C, 70.09; H, 6.04.

(3*R,4*S**)-3-Hydroxy-3-(3-methoxy-4-benzyloxybenzyl)-4-(3,4-dimethoxybenzyl)- γ -butyrolactone (2f).** mp 110-1 °C (AcOEt-hexane) [Lit.^{2c} (-)-2f: mp 145-6 °C (CH_2Cl_2 -ether)]; IR

3

(KBr) 3418, 1761 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.66 (dd, 1H, $J=11.3, 13.2$ Hz), 2.70 (s, 1H), 2.83-3.06 (m, 1H), 2.97 (s, 2H), 3.14 (dd, 1H, $J=3.9, 13.2$ Hz), 3.79-3.95 (m, 1H), 3.87 (s, 3H), 3.89 (s, 3H), 4.18 (dd, 1H, $J=7.7, 9.1$ Hz), 5.15 (s, 2H), 6.64-6.91 (m, 6H), 7.28-7.50 (m, 5H); MS m/z (relative intensity, %): 478 (M^+ , 13), 277 (40), 151 (42), 91 (100). Anal. Calcd for $\text{C}_{28}\text{H}_{30}\text{O}_7$: C, 70.28; H, 6.32. Found: C, 70.24; H, 6.08.

(3*S, 4*S**)-3-Hydroxy-3-benzyl-4-(3,4,5-trimethoxybenzyl)- γ -butyrolactone (1g).** mp 142-3 $^{\circ}\text{C}$ (AcOEt); IR (KBr) 3469, 1764 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.48-2.63 (m, 3H), 2.82-3.03 (m, 1H), 3.02 (d, 1H, $J=13.6$ Hz), 3.18 (d, 1H, $J=13.6$ Hz), 3.82 (s, 3H), 3.83 (s, 6H), 3.96-4.14 (m, 2H), 6.31 (s, 2H), 7.11-7.38 (m, 5H); MS m/z (relative intensity, %): 372 (M^+ , 79), 181 (100), 91 (33), 59 (36). Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{O}_6$: C, 67.73; H, 6.50. Found: C, 67.62; H, 6.38.

(3*R, 4*S**)-3-Hydroxy-3-benzyl-4-(3,4,5-trimethoxybenzyl)- γ -butyrolactone (2g).** mp 116-7 $^{\circ}\text{C}$ (AcOEt-hexane); IR (KBr) 3449, 1769 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.65 (dd, 1H, $J=11.3, 13.2$ Hz), 2.72 (s, 1H), 2.87-3.09 (m, 3H), 3.17 (dd, 1H, $J=3.8, 13.2$ Hz), 3.84 (s, 3H), 3.86 (s, 6H), 3.93 (dd, 1H, $J=9.4, 10.1$ Hz), 4.24 (dd, 1H, $J=7.7, 9.1$ Hz), 6.39 (s, 2H), 7.19-7.45 (m, 5H); MS m/z (relative intensity, %): 372 (M^+ , 66), 181 (100), 91 (32), 59 (12). Anal. Calcd for $\text{C}_{21}\text{H}_{24}\text{O}_6$: C, 67.73; H, 6.50. Found: C, 67.61; H, 6.29.

(3*R, 4*R**)-3-(3,4-Dimethoxybenzyl)-4-(3,4-methylenedioxybenzoyl)- γ -butyrolactone (10b):** 71% yield from **8b**; mp 98-9 $^{\circ}\text{C}$ (AcOEt); IR (KBr) 1774, 1665 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.92-3.14 (m, 2H), 3.45-3.60 (m, 1H), 3.75 (s, 3H), 3.82 (s, 3H), 4.00 (dd, 1H, $J=8.3, 16.8$ Hz), 4.12 (t, 1H, $J=8.4$ Hz), 4.38 (t, 1H, $J=8.4$ Hz), 6.06 (s, 2H), 6.58-6.85 (m, 4H), 7.16-7.25 (m, 2H); MS m/z (relative intensity, %): 384 (M^+ , 63), 208 (100), 151 (61). Anal. Calcd for $\text{C}_{21}\text{H}_{20}\text{O}_7$: C, 65.62; H, 5.24. Found: C, 65.71; H, 5.11.

(3*R, 4*R**)-3-(3-Methoxy-4-benzyloxybenzyl)-4-(3,4-dimethoxybenzoyl)- γ -butyrolactone (10c).** 72% yield from **8a**; mp 146-7 $^{\circ}\text{C}$ (AcOEt); IR (KBr) 1777, 1665 cm^{-1} ; ^1H NMR

4

(δ in CDCl_3) 3.02 (br. d, 2H, $J=6.1$ Hz), 3.45-3.63 (m, 1H), 3.75 (s, 3H), 3.90 (s, 3H), 3.95 (s, 3H), 4.00-4.20 (m, 2H), 4.30-4.48 (m, 1H), 5.06 (s, 2H), 6.50-6.85 (m, 4H), 7.16-7.48 (m, 7H); MS m/z (relative intensity, %): 476 (M^+ , 28), 165 (65), 91 (100). Anal. Calcd for $\text{C}_{28}\text{H}_{28}\text{O}_7$: C, 70.58; H, 5.92. Found: C, 70.27; H, 5.69.

(3*R,4*R**)-3-(3,4-Dimethoxybenzyl)-4-[(α S*)- α -hydroxy-3,4-methylenedioxybenzyl]- γ -butyrolactone (11b).** 94% yield; syrup; IR (KBr) 3508, 1740 cm^{-1} ; ^1H NMR (δ in CDCl_3) 1.97 (d, 1H, $J=2.8$ Hz), 2.60 (quintet, 1H, $J=7.3$ Hz), 2.88-3.16 (m, 3H), 3.84 (s, 3H), 3.86 (s, 3H), 3.91 (dd, 2H, $J=1.7, 7.1$ Hz), 4.61 (dd, 1H, $J=2.8, 6.8$ Hz), 5.95-6.01 (m, 2H), 6.63-6.83 (m, 6H); MS m/z (relative intensity, %): 386 (M^+ , 40), 151 (100). Anal. Calcd for $\text{C}_{21}\text{H}_{22}\text{O}_7$: C, 65.28; H, 5.74. Found: C, 65.28; H, 5.71.

(3*R,4*R**)-3-(3-Methoxy-4-benzyloxybenzyl)-4-[(α S*)- α -hydroxy-3,4-dimethoxybenzyl]- γ -butyrolactone (11c).** 89% yield; syrup; IR (KBr) 3500, 1765 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.06 (d, 1H, $J=2.8$ Hz), 2.48-3.17 (m, 4H), 3.62-4.03 (m, 2H), 3.83 (s, 3H), 3.87 (s, 3H), 4.61 (dd, 1H, $J=2.7, 6.8$ Hz), 5.12 (s, 2H), 6.54-6.86 (m, 6H), 7.22-7.49 (5H, m); MS m/z (relative intensity, %): 478 (M^+ , 5), 460 (6), 151 (9), 91 (100). Anal. Calcd for $\text{C}_{28}\text{H}_{30}\text{O}_7$: C, 70.28; H, 6.32. Found: C, 70.08; H, 6.13.

(3*R,4*S**,5*R**)-3-(3,4-Dimethoxybenzyl)-4-methoxymethyloxymethyl-5-(3,4-methylenedioxyphenyl)- γ -butyrolactone (3b).** 74% yield; syrup; IR (KBr) 1771 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.19-2.37 (m, 1H), 2.94-3.20 (m, 3H), 3.27 (dd, 1H, $J=3.9, 10.3$ Hz), 3.32 (s, 3H), 3.42 (dd, 1H, $J=2.8, 10.6$ Hz), 3.85 (s, 3H), 3.86 (s, 3H), 4.47 (d, 1H, $J=6.5$ Hz), 4.56 (d, 1H, $J=6.6$ Hz), 5.12 (d, 1H, $J=9.1$ Hz), 5.94 (s, 2H), 6.46-6.84 (m, 6H); MS m/z (relative intensity, %): 430 (M^+ , 43), 177 (37), 151 (100). Anal. Calcd for $\text{C}_{23}\text{H}_{26}\text{O}_8$: C, 64.18; H, 6.09. Found: C, 64.22; H, 5.99.

(3*R,4*S**,5*R**)-3-(3-Methoxy-4-benzyloxybenzyl)-4-methoxymethyloxymethyl-5-(3,4-**

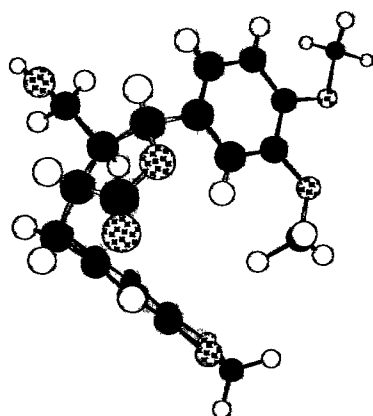
dimethoxyphenyl)- γ -butyrolactone (3c). 80% yield; syrup; IR (KBr) 2937, 1771 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.20-2.38 (m, 1H), 3.00-3.22 (m, 3H), 3.28 (dd, 1H, $J=3.8, 10.3$ Hz), 3.32 (s, 3H), 3.44 (dd, 1H, $J=3.6, 10.3$ Hz), 3.76 (s, 3H), 3.86 (s, 6H), 4.46 (d, 1H, $J=6.6$ Hz), 4.61 (d, 1H, $J=6.5$ Hz), 5.10 (s, 2H), 5.13 (d, 1H, $J=10.7$ Hz), 6.50-6.87 (m, 6H), 7.23-7.47 (m, 5H); MS m/z (relative intensity, %): 522 (M^+ , 17), 151 (29), 91 (100). Anal. Calcd for $\text{C}_{30}\text{H}_{34}\text{O}_8$: C, 68.95; H, 6.56. Found: C, 68.82; H, 6.50.

(3*S,4*S**,5*R**)-3-Hydroxy-3-(3,4-dimethoxybenzyl)-4-methoxymethyloxymethyl-5-(3,4-methylenedioxyphenyl)- γ -butyrolactone (4b).** mp 161-2 $^{\circ}\text{C}$ (AcOEt - i - Pr_2O); IR (KBr) 3457, 1775 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.42-2.55 (m, 1H), 3.06 (d, 1H, $J=13.5$ Hz), 3.16 (d, 1H, $J=13.5$ Hz), 3.36 (s, 3H), 3.48 (s, 1H), 3.57 (dd, 1H, $J=4.3, 10.1$ Hz), 3.78 (dd, 1H, $J=5.8, 10.1$ Hz), 3.84 (s, 3H), 3.87 (s, 3H), 4.57 (d, 1H, $J=6.6$ Hz), 4.61 (d, 1H, $J=6.6$ Hz), 5.25 (d, 1H, $J=8.1$ Hz), 5.95 (s, 2H), 6.40-6.90 (m, 6H); MS m/z (relative intensity, %): 446 (M^+ , 50), 177 (27), 151 (100). Anal. Calcd for $\text{C}_{23}\text{H}_{26}\text{O}_9$: C, 61.88; H, 5.87. Found: C, 61.93; H, 5.72.

(3*S,4*S**,5*R**)-3-Hydroxy-3-(3-methoxy-4-benzyloxybenzyl)-4-methoxymethyloxymethyl-5-(3,4-dimethoxyphenyl)- γ -butyrolactone (4c).** mp 118-9 $^{\circ}\text{C}$ (AcOEt -hexane); IR (KBr) 3481, 1770 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.43-2.59 (m, 1H), 3.06 (d, 1H, $J=13.4$ Hz), 3.19 (d, 1H, $J=13.4$ Hz), 3.35 (s, 3H), 3.48 (s, 1H), 3.55 (dd, 1H, $J=4.1, 10.1$ Hz), 3.71 (s, 3H), 3.73-3.91 (m, 1H), 3.84 (s, 3H), 3.86 (s, 3H), 4.56 (d, 1H, $J=6.5$ Hz), 4.61 (d, 1H, $J=6.5$ Hz), 5.10 (s, 2H), 5.24 (d, 1H, $J=8.4$ Hz), 6.42 (d, 1H, $J=1.9$ Hz), 6.61-6.90 (m, 5H), 7.21-7.49 (m, 5H); MS m/z (relative intensity, %): 538 (M^+ , 12), 238 (28), 193 (37), 177 (37), 165 (43), 91 (100). Anal. Calcd for $\text{C}_{30}\text{H}_{34}\text{O}_9$: C, 66.90; H, 6.36. Found: C, 66.72; H, 6.08.

(3*R,4*S**)-3-Hydroxy-3-(3-methoxy-4-hydroxybenzyl)-4-(3,4-dimethoxybenzyl)- γ -butyrolactone (2a).** 82% yield from 4c; mp 128-9 $^{\circ}\text{C}$ (AcOEt -hexane); IR (KBr) 3451, 1752 cm^{-1} ; ^1H NMR (δ in CDCl_3) 2.67 (dd, 1H, $J=11.3, 13.1$ Hz), 2.74 (s, 1H), 2.84-3.06 (m, 3H), 3.15 (dd, 1H, $J=3.8, 13.2$ Hz), 3.78-3.98 (m, 1H), 3.87 (s, 3H), 3.88 (s, 3H), 4.18 (dd, 1H, $J=7.8, 9.1$ Hz), 5.61 (s,

6
1H), 6.62-6.94 (m, 6H); MS m/z (relative intensity, %): 388 (M^+ , 13), 151 (13), 137 (100). Anal. Calcd for $C_{21}H_{24}O_7$: C, 64.94; H, 6.23. Found: C, 64.56; H, 5.85.



X-ray crystal structure of 12a