Supporting Information

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Chemoselective Deprotection of Cyclic *N*,*O*-Aminals Using Catalytic Bismuth(III) Bromide in Acetonitrile

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Experimental

General Methods. All reactions were carried out under argon or nitrogen in oven-dried glasswares using standard gastight syringes, cannulas, and septa. Solvents and reagents were purified and dried by standard methods prior to use. Optical rotations were measured at rt. IR spectra were recorded on an FT-IR instrument. ^{1}H NMR spectra were recorded at 300 MHz and are reported in parts per million (δ) downfield relative to TMS as internal standard, and ^{13}C NMR spectra were recorded at 75 MHz and assigned in parts per million (δ). Flash column chromatographies were performed on silica gel (10-40 μm) using mixtures of petroleum ether and ethyl acetate as the eluents.

General procedure for deprotection of cyclic *N,O*-aminals: To a solution of cyclic *N,O*-aminal **1** (1.0 mmol) in MeCN (10 mL) was added bismuth (III) bromide (45 mg, 0.1 mmol) at room temperature. The reaction mixture was stirred at rt, and the progress of the reaction was monitored by TLC. When all starting material had disappeared, the reaction mixture was quenched by adding saturated aqueous $NaHCO_3$ (5 mL). The layers were separated and the aqueous layer was extracted with ethyl acetate. The combined organic layers were washed with brine, dried over Na_2SO_4 and concentrated. The residue was purified by flash column chromatography on silica gel to afford the pure product **2**.

(*S*)-4-Methoxymethoxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid benzyl ester (3). To a solution of (*R*)-4-hydroxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid benzyl ester [1] (408 mg, 1.6 mmol) in anhydrous CH_2Cl_2 (16 mL) was added *N,N*-diisopropylethylamine (1.1 mL, 6.4 mmol). The mixture was cooled to 0^{o}C under an atmosphere of nitrogen. MOMCl (0.24 mL, 3.2 mmol) was added dropwise *via* sypringe at 0^{o}C, and the reaction mixture was stirred at room temperature overnight. After all starting material disappeared by TLC monitoring, sat. aq. $NaHCO_3$ (10 mL) was added to quench the reaction. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3x10 mL). The combined organic layers were washed with brine (3x10 mL), dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=10:1) to give pure MOM ether **3** (430 mg, 88%) as a colorless oil. $[\alpha]_D^{22}$ -24.7 (*c* 1.47, $CHCl_3$). IR (neat): 2985, 2939, 2885, 1708, 1409, 1351, 1261, 1155, 1091, 1075, 1050, 1027cm^{-1}. ^{1}H NMR (300

MHz, 55 ^{o}C, $CDCl_3$): δ 1.51 (s, 3H), 1.58 (s, 3H), 3.31 (s, 3H), 3.44-3,50 (m, 1H), 3.67(brs, 1H), 3.96-4.04 (m, 1H), 4.09 (brs, 1H), 4.58 (s, 2H), 5.12, 5.17 (AB, *J*=12.6Hz, 2H), 7.29-7.36 (m, 5H) ppm. ESI-MS (*m/z*, %): 332.2 ($M+Na^+$, 100%), 332.2 ($M+K^+$, 20%). Anal. calcd for $C_{16}H_{23}NO_5$ (%): C 62.12, H 7.49, N 4.53; Found: C 62.40, H 7.64, N 4.35.

(*S*)-4-(*tert*-Butyldimethylsilanyloxymethyl)-2,2-dimethyl-oxazolidine-3-carboxylic acid benzyl ester (5). To a solution of (*R*)-4-hydroxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid benzyl ester [1] (470 mg, 1.84 mmol), DMAP (22mg, 0.18mmol) and imidazole (275 mg, 4.05 mmol) in anhydrous DMF (1.8 mL) was added TBDMSCl (305mg, 2.02mmol) at 0^{o}C under an atmosphere of nitrogen. The reaction mixture was stirred at room temperature overnight. After all starting material disappeared as indicated by TLC monitoring, sat. aq. $NaHCO_3$ (2 mL) was added to quench the reaction. The mixture was extracted with CH_2Cl_2 (3x10 mL). The combined organic layers were washed with brine (3x10 mL), dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=20:1) to give pure **5** (674 mg, 97%) as a colorless oil. $[\alpha]_D^{22}$ -22.7 (*c* 1.95, $CHCl_3$). IR (neat): 2984, 2957, 2931, 2859, 1712, 1408, 1349, 1259, 1095, 1073, 1029cm^{-1}. ^{1}H NMR (300 MHz, 55 ^{o}C, $CDCl_3$): δ 0.10 (s, 6H), 0.89 (s, 9H), 1.52 (s, 3H), 1.58 (s, 3H), 3.50 (brs, 1H), 3.76 (brs, 1H), 3.94-3.96(m, 2H), 4.05-4.07 (m, 1H), 5.11, 5.18 (AB, *J*=12.3Hz, 2H), 7.31-7.38 (m, 5H) ppm. ESI-MS (*m/z*, %): 380.2 ($M+H^+$, 40%), 402.2 ($M+Na^+$, 20%). Anal. calcd for $C_{20}H_{33}NO_4Si$ (%): C 63.29, H 8.76, N 3.69; Found: C 63.52, H 8.97, N 3.61.

(2-Hydroxy-1-hydroxymethyl-ethyl)-carbamic acid benzyl ester (6).[2] TBDMS ether **5** (84 mg, 0.22 mmol) was converted to diol **6** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=1:2) to give pure diol **6** (42 mg, 85%) as a white solid. Mp 105-108 ^{o}C. ^{1}H NMR (300 MHz, CD_3COCD_3): δ 2.98 (brs, 2H), 3.67 (brs, 4H), 3.93 (brs, 1H), 5.07 (s, 2H), 6.11 (brs, 1H), 7.31-7.39 (m, 5H) ppm.

(*S*)-4-Acetoxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid benzyl ester (7). To a solution of (*R*)-4-hydroxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid benzyl ester [1] (267 mg, 1.0 mmol) in anhydrous CH_2Cl_2 (10 mL) was added Et_3N (0.35 mL, 2.4 mmol). The mixture was cooled to 0^{o}C under

an atmosphere of nitrogen. Acetyl chloride (0.08 mL, 1.2 mmol) was added dropwise *via* sypringe at 0^oC and the reaction mixture was stirred at room temperature for 30 min. After all starting material disappeared, sat. aq. $NaHCO_3$ (5 mL) was added to quench the reaction. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3x10 mL). The combined organic layers were washed with brine (3x10 mL), dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=8:1) to give pure **7** (306 mg, 99%) as a colorless oil. $[\alpha]_D^{22}$ -27.0 (*c* 2.00, $CHCl_3$). IR (neat): 2986, 2939, 2885, 1747, 1708, 1409, 1353, 1237, 1091, 1076, 1043 cm^{-1}. ^1H NMR (300 MHz, 55 ^oC, $CDCl_3$): δ 1.50 (s, 3H), 1.60 (s, 3H), 2.01 (s, 3H), 3.90-4.00 (m, 2H), 4.05 (brs, 1H), 4.15 (brs, 1H), 4.23-4.27 (m, 1H), 5.15 (s, 2H), 7.24-7.34 (m, 5H) ppm. ESI-MS (*m/z*, %): 330.2 ($M+Na^+$, 100%). Anal. calcd for $C_{16}H_{21}NO_5$ (%): C 62.53, H 6.89, N 4.56; Found: C 62.80, H 7.08, N 4.33.

Acetic acid (2*S*)-benzyloxycarbonylamino-3-hydroxy-propyl ester (8). Acetate **7** (146 mg, 0.48 mmol) was converted to alcohol **8** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=1:1) to give pure alcohol **8** (123 mg, 97%) as a colorless oil. $[\alpha]_D^{22}$ -0.4 (*c* 1.57, $CHCl_3$). IR (neat): 3340, 2957, 2891, 1739, 1539, 1456, 1368, 1240, 1159, 1037 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 2.08 (s, 3H), 2.42 (br, 1H), 3.62-3.71 (m, 2H), 3.93-3.71 (m, 1H), 4.21-4.23 (m, 2H), 5.11 (brs, 2H), 5.24 (d, *J*=8.4 Hz, 1H), 7.33-7.39 (m, 5H) ppm. ESI-MS (*m/z*, %): 290.1 ($M+Na^+$, 100%), 306.1 ($M+K^+$, 20%). Anal. calcd for $C_{13}H_{17}NO_5$ (%): C 58.42, H 6.41, N 5.24; Found: C 58.55, H 6.47, N 5.02.

(*S*)-4-Methoxymethoxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid *tert*-butyl ester (9). (*R*)-4-hydroxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid *tert*-butyl ester [3] (337 mg, 1.45 mmol) was converted to MOM ether **9** by the same procedure for compound **3**. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=10:1) to give MOM ether **9** (380 mg, 92%) as a colorless oil. $[\alpha]_D^{23}$ -20.7 (*c* 1.75, $CHCl_3$). IR (neat): 2980, 2937, 2884, 1700, 1391, 1367, 1262, 1176, 1159,1110, 1078, 1051, 1027 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.47 (s, 9H), 1.50-1.56 (m, 6H), 3.36 (s, 3H), 3.42-3,48 (m, 1H), 3.68-3.75(m, 1H), 3.94-4.11 (m, 3H), 4.61-4.67 (m, 2H) ppm. ESI-MS (*m/z*, %): 298.2 ($M+Na^+$, 100%). HR-ESI-MS calcd. for $C_{13}H_{25}NO_5Na$ ($M+Na^+$): 298.1625; Found: 298.1631.

(*S*)-(2-Hydroxy-1-methoxymethoxymethyl-ethyl)-carbamic acid *tert*-butyl ester (10). MOM ether **9** (119 mg, 0.43 mmol) was converted to alcohol **10** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=2:1) to give pure alcohol **10** (101 mg, 99%) as a colorless oil. $[\alpha]_D^{23}$ 3.3 (*c* 1.35, $CHCl_3$). IR (neat): 3357, 2978, 2943, 2887, 1694, 1521, 1367, 1249, 1173, 1114, 1044, 920 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.45 (s, 9H), 2.71 (br, 1H), 3.37 (s, 3H), 3.62-3.75 (m, 3H), 3.77-3.82 (m, 2H), 4.63 (s, 2H), 5.17 (br, 1H) ppm. ESI-MS (*m/z*, %): 258.1 ($M+Na^+$, 100%). Anal. calcd for $C_{10}H_{21}NO_5$ (%): C 51.05, H 9.00, N 5.95; Found: C 51.00, H 9.14, N 5.82.

(2-Hydroxy-1-hydroxymethyl-ethyl)-carbamic acid *tert*-butyl ester (12).[4] TBDMS ether **11**[5] (207 mg, 0.6 mmol) was converted to diol **11** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=1:3) to give pure diol **12** (42 mg, 85%) as a white solid. Mp 86-87°C. ^1H NMR (300 MHz, $CDCl_3$): δ 1.45 (s, 9H), 3.32 (br, 2H), 3.67-3.82 (m, 5H), 5.35 (br, 1H) ppm.

(*S*)-4-Acetoxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid *tert*-butyl ester (13). (*R*)-4-hydroxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid *tert*-butyl ester[3] (341 mg, 1.47 mmol) was converted to acetate **13** by the same procedure for compound **7**. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=8:1) to give pure **13** (381 mg, 95%) as a colorless oil. $[\alpha]_D^{23}$ -34.0 (*c* 1.36, $CHCl_3$). IR (neat): 2982, 2939, 2883, 1749, 1702, 1390, 1367, 1236, 1174, 1105, 1080, 1042 cm^{-1}. ^1H NMR (300 MHz, 55°C, $CDCl_3$): δ 1.49 (s, 9H), 1.51 (s, 3H), 1.58 (s, 3H), 2.05 (s, 3H), 3.87-4.04 (m, 4H), 4.25-4.29 (m, 1H) ppm. ESI-MS (*m/z*, %): 296.2 ($M+Na^+$, 100%). HR-ESI-MS calcd. for $C_{13}H_{23}NO_5Na$ ($M+Na^+$): 296.1468; Found: 296.1469.

Acetic acid (2*S*)-*tert*-butoxycarbonylamino-3-hydroxy-propyl ester (14). Acetate **13** (175 mg, 0.64 mmol) was converted to alcohol **14** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=2:1) to give pure alcohol **14** (140 mg, 94%) as a colorless oil. $[\alpha]_D^{23}$ -3.71 (*c* 1.30, $CHCl_3$). IR (neat): 3367, 2979, 2937, 2893, 1744, 1715, 1527, 1368, 1264, 1171, 1045 cm^{-1}. ^1H NMR (300 MHz,

$CDCl_3$): δ 1.45 (s, 9H), 2.10 (s, 3H), 2.48 (br, 1H), 3.60-3.70 (m, 2H), 3.88-3.91 (m, 1H), 4.19 (s, 1H), 4.21 (s, 1H), 5.24 (d, *J*=5.7 Hz, 1H) ppm. ESI-MS (*m/z*, %): 256.1 ($M+Na^+$, 100%). Anal. calcd for $C_{10}H_{19}NO_5$ (%): C 51.49, H 8.21, N 6.00; Found: C 51.78, H 8.28, N 5.76.

(*S*)-4-(*tert*-Butyl-diphenyl-silanyloxymethyl)-2,2-dimethyl-oxazolidine-3-carboxylic acid *tert*-butyl ester (15). To a solution of (*R*)-4-hydroxymethyl-2,2-dimethyl-oxazolidine-3-carboxylic acid *tert*-butyl ester[3] (342 mg, 1.48 mmol) and imidazole (151 mg, 2.20 mmol) in anhydrous CH_2Cl_2 (15 mL) was added TBDPSCl (0.42 mL, 1.62 mmol) at 0°C under an atmosphere of nitrogen. The reaction mixture was stirred at room temperature for 2 hours. After all starting material disappeared, sat. aq. $NaHCO_3$ (2 mL) was added to quench the reaction. The mixture was extracted with CH_2Cl_2 (3x20 mL). The combined organic layers were washed with brine (3x10 mL), dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=30:1) to give pure **15** (690 mg, 99%) as a white solid. Mp: 105-106°C. $[\alpha]_D^{23}$ -21.7 (*c* 1.41, $CHCl_3$). IR (KBr): 3073, 2976, 2933, ,2888, 2859, 1702, 1691, 1471, 1382, 1263, 1112, 1085, 1077 cm^{-1}. ^1H NMR (300 MHz, 55°C, $CDCl_3$): δ 1.05 (s, 9H), 1.35 (brs, 9H), 1.47 (s, 3H), 1.48 (s, 3H) 3.58 (brs, 1H), 3.80-3.83 (m, 1H), 3.95-3.98 (m, 2H), 4.13-4.15 (m, 1H), 7.32-7.41 (m, 6H), 7.62-7.70 (m, 4H) ppm. ESI-MS (*m/z*, %): 492.4 ($M+Na^+$, 100%), 508.5 ($M+K^+$, 40%). Anal. calcd for $C_{27}H_{39}NO_4Si$ (%): C 69.04, H 8.37, N 2.98; Found: C 69.09, H 8.40, N 2.50.

(2*S*)-[1-(*tert*-Butyl-diphenyl-silanyloxymethyl)-2-hydroxy-ethyl]-carbamic acid *tert*-butyl ester (16). TBDPS ether **15** (350 mg, 0.75 mmol) was converted to alcohol **16** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=6:1) to give pure **16** (140 mg, 92%) as a white solid. Mp: 72-73°C. $[\alpha]_D^{23}$ -4.1 (*c* 1.12, $CHCl_3$). IR (KBr): 3411, 3286, 3075, 3057, 2930, 2856, 1675, 1546, 1474, 1482, 1179, 1112, 1085, 1043 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.07 (s, 9H), 1.45 (s, 9H), 2.40 (br, 1H) 3.66-3.85 (m, 5H), 5.10 (d, *J*=5.7Hz, 1H), 7.40-7.46 (m, 6H), 7.63-7.66 (m, 4H) ppm. ESI-MS (*m/z*, %): 452.4 ($M+Na^+$, 100%), 568.5 ($M+K^+$, 10%). Anal. calcd for $C_{24}H_{35}NO_4Si$ (%): C 67.10, H 8.21, N 3.26; Found: C 67.25, H 8.25, N 3.06.

(4*R*,2*E*)-4-*tert*-Butoxycarbonylamino-5-hydroxy-pent-2-enoic acid ethyl ester (18). Ester **17**[6] (275 mg, 0.92 mmol) was converted to alcohol **18** by the general procedure using of 20 mol% $BiBr_3$. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=4:1) to give pure **18** (215 mg, 90%) as a white solid. Mp: 46-47°C. $[\alpha]_D^{23}$ -4.7 (*c* 1.10, $CHCl_3$). IR (KBr): 3353, 2986, 2881, 1527, 1369, 1297, 1167, 1013 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.29 (t, *J*=7.2Hz, 3H), 1.45 (s, 9H), 2.45 (br, 1H) 3.75-3.78 (m, 2H), 4.20 (q, *J*=7.5Hz, 2H), 4.43 (brs, 1H) 5.12 (d, *J*=7.5Hz, 1H), 6.01 (brd, *J*=15.6Hz, 1H), 6.92 (dd, *J*=5.1, 15.6Hz, 1H) ppm. ESI-MS (*m/z*, %): 282.2 ($M+Na^+$, 100%), 298.2 ($M+K^+$, 50%). Anal. calcd for $C_{12}H_{21}NO_5$ (%): C 55.58, H 8.16, N 5.40; Found: C 55.72, H 8.08, N 5.37.

***N*-((*R*)-2-(Benzyloxy)-1-((4*R*,5*S*)-2,2-dimethyl-5-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-1,3-dioxola n-4-yl)ethyl)acetamide (19).** To a solution of (2*R*)-1-*O*-benzyl-2-azido-2-deoxy-3,4:5,6-di-*O*-isopropylidene-D-mannitol[7] (286 mg, 0.76 mmol), DMAP (10 mg, 0.08 mmol) and Et_3N (0.3 mL, 2.28 mmol) in anhydrous CH_2Cl_2 (8 mL) was added acetic anhydride (0.14 mL, 1.52 mmol) at 0°C under an atmosphere of nitrogen. The reaction mixture was stirred at room temperature overnight. After all starting material disappeared, sat. aq. $NaHCO_3$ (5 mL) was added to quench the reaction. The mixture was extracted with CH_2Cl_2 (3x10 mL). The combined organic layers were washed with brine (3x10 mL), dried (Na_2SO_4) and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=2:1) to give pure acetamide **19** (272 mg, 91%) as a white solid. Mp: 67-69°C. $[\alpha]_D^{24}$ -5.4 (*c* 2.59, $CHCl_3$). IR (KBr): 3258, 2986, 2934, 2881, 1651, 1538, 1456, 1373, 1218, 1066 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.35 (s, 3H), 1.38 (s, 9H), 1.99 (s, 3H), 3.63 (m, 1H), 3.77 (m, 1H), 3.82 (m, 1H), 3.91-4.05 (m, 2H), 4.12-4.19(m, 3H), 4.52, 4.59 (AB, *J*=12Hz, 2H), 5.97 (d, *J*=6.9Hz, 1H), 7.29-7.36 (m, 5H) ppm. ESI-MS (*m/z*, %): 394.2 ($M+H^+$, 40%), 416.2 ($M+Na^+$, 100%). Anal. calcd for $C_{21}H_{31}NO_6$ (%): C 64.10, H 7.94, N 3.56; Found: C 64.26, H 7.91, N 3.26.

***N*-((*R*)-2-(Benzyloxy)-1-((4*R*,5*R*)-5-((*R*)-1,2-dihydroxyethyl)-2,2-dimethyl-1,3-dioxolan-4-yl)ethyl)acetamide (20).** Acetamide **19** (106 mg, 0.27 mmol) was converted to diol **20** by the general procedure using 10 mol% of bismuth(III) bromide. The crude product was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=2:1 to1:5) to give pure diol **20** (16 mg, 84% based on 19% conversion) as a white solid. Mp: 108-110°C. $[\alpha]_D^{24}$ -5.1 (*c* 2.04, $CHCl_3$). IR (KBr): 3308,

2992, 2936, 2870, 1651, 1551, 1455, 1371, 1238, 1082, 1028 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.37 (s, 6H), 1.86 (br, 1H), 1.98 (s, 3H), 2.63 (br, 1H), 3.62-3.71 (m, 4H), 3.75-3.92 (m, 2H), 4.21-4.23 (m, 2H), 4.54 (s, 2H), 6.34 (d, *J*=6.9Hz, 1H), 7.30-7.36 (m, 5H) ppm. ESI-MS (*m/z*, %): 354.2 ($M+H^+$, 35%), 376.2 ($M+NA^+$, 100%). ^{13}C NMR (75 MHz, CD_3OD): δ 23.3, 27.0, 27.2, 51.7, 63.9, 68.2, 73.2, 73.4, 78.9, 78.9, 109.8, 127.8, 127.9, 128.0, 128.5 (2C), 137.8, 170.8 ppm. HR-ESI-MS calcd. for $C_{18}H_{27}NO_6$ (%): 376.1730; Found: 376.1735.

1-((*R*)-2,2-Dimethyl-4-((4*R*,5*S*)-2,2-dimethyl-5-((*R*)-2,2-dimethyl-1,3-dioxolan-4-yl)-1,3-dioxolan-4-yl)oxazolidin-3-yl)ethanone (21). To a solution of (2*R*)-2-acetamido-2-deoxy-3,4:5,6-di-*O*-isopropylidene-D-mannitol **22**[7] (502 mg, 1.66 mmol), *p*-TsOH•H_2O (32 mg, 0.17 mmol) in anhydrous DMF (16 mL) was added 2,2-dimethoxypropane (2.1 mL, 16.6 mmol) at room temperature under an atmosphere of nitrogen. The reaction mixture was stirred at 70°C for 48 hours. After all the starting material disappeared, Et_3N (2 mL) was added and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (petroleum ether/ethyl acetate=4:1) to give pure compound **21** (363 mg, 66%) as a pale-yellow oil. $[\alpha]_D^{24}$ 1.4 (*c* 2.15, $CHCl_3$). IR (neat): 2988, 2939, 2881, 1655, 1401, 1373, 1264, 1214, 1156, 1070, 844 cm^{-1}. ^1H NMR (300 MHz, 55°C, $CDCl_3$): δ 1.32 (s, 3H), 1.35 (s, 3H), 1.38 (s, 3H), 1.42 (s, 3H), 1.57 (s, 3H), 1.64 (s, 3H) 2.14 (s, 3H), 3.58-3.67(m, 1H), 3.90-4.25 (m, 7H) ppm. ^{13}C NMR (75 MHz, CD_3OD): δ 23.6, 25.2, 25.5, 26.7, 27.2, 27.3, 27.4, 58.8, 63.8, 68.0, 77.4, 78.9, 81.8, 96.0, 109.8, 110.4, 167.8 ppm. ESIMS (*m/z*, %): 344.2 ($M+H^+$, 100%). HR-ESI-MS calcd. for $C_{17}H_{29}NO_6Na$ ($M+Na^+$):366.1887; Found: 366.1888.

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