Supplementary material for JO026897V

Zirconium(IV) chloride Catalyzed New and Efficient Protocol for the Selective Cleavage of *p*-Methoxybenzyl (PMB) Ethers

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Procedure for preparation and spectral data:

1-(4-Methoxybenzyloxy)-2-phenyl ethane (1):

To a stirred solution of phenyl ethanol (0.5 g, 4.09 mmol) in dry THF (10 mL), NaH (0.33 g, 8.19 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, *p*-methoxyphenyl methyl bromide (MPMBr) (0.82 g, 4.09 mmol) was added through a syringe and stirred at room temperature for 3h. Excess of NaH was quenched by slow and careful addition of MeOH (5 mL), diluted with water (10 mL) and extracted into ether (2 20 mL). Combined ether extracts were washed with brine solution (10 mL), dried (Na₂SO₄) and concentrated under reduced pressure and purified the residue by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **1** (0.8 g, 81%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.18 (m, 6H, Ar-H), 7.0 (d, *J* = 8.0 Hz, 1H, Ar-H), 6.76 (t, *J* = 8.0 Hz, 2H, Ar-H), 4.40 (s, 2H), 3.76 (s, 3H), 3.60 (m, 2H), 2.80 (m, 2H); EIMS (*m/z*): 242 (*M*⁺); IR (neat): 2937, 2859, 1623, 1455, 1247, 1077 cm⁻¹. Anal. Calcd. for C₁₆H₁₈O₂ (242): C, 79.31; H, 7.49. Found: C, 79.27; H, 7.44%.

1-Methoxy-4-octyloxymethyl benzene (2):

To a stirred solution of 1-octanol (0.3 g, 2.30 mmol) in dry THF (10 mL), NaH (0.18 g, 4.61 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, MPMBr (0.46 g, 2.30 mmol) was added through a syringe and stirred at room temperature for 4h. The reaction mixture was worked up as described

for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **2** (0.51 g, 88%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.20 (d, *J* = 8.3 Hz, 2H, Ar-H), 6.84 (d, *J* = 8.3 Hz, 2H, Ar-H), 4.40 (s, 2H), 3.75 (s, 3H), 3.45 (t, *J* = 8.2 Hz, 2H), 1.60-1.50 (m, 2H), 1.45-1.25 (m, 10H), 0.90 (t, *J* = 5.1 Hz, 3H); EIMS (*m/z*): 251 (*M*⁺+1); IR (neat): 2934, 2862, 1515, 1260, 1046 cm⁻¹. Anal. Calcd. for C₁₆H₂₆O₂ (250): C, 76.75; H, 10.47. Found: C, 76.72; H, 10.43%.

1-(4-Methoxybenzyloxy)-6-tetrahydro-2H-2-pyranyloxyhexane (3)⁸:

To a stirred solution of 1-(4-methoxybenzyloxy)-6-hexanol (0.5 g, 2.1 mmol) in CH₂Cl₂ (10 mL), were added dihydropyran (0.18 g, 2.10 mmol), catalytic PTSA at 0°C and stirred it at room temperature for 6h. The reaction mixture was neutralized with saturated aqueous NaHCO₃ solution and extracted with CH₂Cl₂ (20 mL), organic layer was washed with brine (10 mL), dried (Na₂SO₄), concentrated under reduced pressure and purified by column chromatography (silicagel, EtOAc: Hexane; 1:4) to afford **3** (0.63 g, 93%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.22 (d, *J* = 8.4 Hz, 2H, Ar-H), 6.83 (d, *J* = 8.4 Hz, 2H, Ar-H), 4.55 (br.s, 1H), 4.40 (s, 2H), 3.88-3.75 (m, 5H), 3.50-3.30 (m, 4H), 1.90-1.30 (m, 14H); EIMS (*m/z*): 237 (*M*⁺-85); IR (neat): 2937, 2859, 1612, 1513, 1247, 1034 cm⁻¹. Anal. Calcd. for C₁₉H₃₀O₄ (322): C, 70.77; H, 9.38. Found: C, 70.72; H, 9.33%.

1-Methoxy-4- 6-(2-ethoxyethoxymethoxy)hexyloxymethyl benzene(4) :

To a stirred solution of 1-(4-methoxybenzyloxy)-6-hexanol (0.3 g, 1.26 mmol) in dry THF (10 mL), NaH (0.1 g, 2.52 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, MEMCl (0.16 g, 1.26 mmol) was added through a syringe and stirred at room temperature for 4h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:4) to afford **4** (0.35 g, 85%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.24 (d, *J* = 8.3 Hz, 2H, Ar-H), 6.84 (d, *J* = 8.3 Hz, 2H, Ar-H), 4.73 (d, *J* = 5.92 Hz, 1H), 4.68 (s, 1H), 4.39 (s, 2H), 3.79 (s, 3H), 3.69-3.62 (m,

2H), 3.58-3.41(m, 6H), 3.39 (s, 3H), 1.6-1.3 (m, 8H); EIMS (m/z): 326 (M^+); IR (neat): 2934, 1614, 1513, 1253, 1105, 1036 cm^{-1} . Anal. Calcd. for $\text{C}_{18}\text{H}_{30}\text{O}_5$ (326): C, 66.23; H, 9.26. Found: C, 66.19; H, 9.21%.

6-(4-Methoxybenzyloxy) hexyl acetate (5): ^{6a}

To a stirred solution of 1-(4-methoxybenzyloxy)-6-hexanol (0.5 g, 2.10 mmol) in CH_2Cl_2 (10 mL) were added triethylamine (0.42 g, 4.20 mmol), acetic anhydride (0.21 g, 2.10 mmol) at 0°C and stirred it at room temperature for 2h. The reaction mixture was diluted with water (20 mL) and extracted with CH_2Cl_2 (20 mL). Organic layer was washed with brine (10 mL), dried (Na_2SO_4), concentrated under reduced pressure and purified the residue by column chromatography (silicagel, EtOAc: Hexane; 1:4) to give **5** (0.52 g, 88%) as a syrup.

^1H NMR (CDCl_3 , 200 MHz): 7.22 (d, $J = 8.2$ Hz, 2H, Ar-H), 6.88 (d, $J = 8.2$ Hz, 2H, Ar-H), 4.38 (s, 2H), 4.04-3.93 (m, 2H), 3.78 (s, 3H), 3.42-3.39 (t, $J = 5.9$ Hz, 2H), 2.1 (s, 3H), 1.69-1.32 (m, 8H); FABMS (m/z): 280 (M^+), 279(8), 137(30), 121(100); IR (neat): 2910, 1750, 1560, 1260, 1039 cm^{-1} . Anal. Calcd. for $\text{C}_{16}\text{H}_{24}\text{O}_4$ (280): C, 68.54; H, 8.63. Found: C, 68.49; H, 8.59%.

4-Methoxy-1- (6-phenylcarbonyloxy hexyloxymethyl) benzene (6)⁸:

To a stirred solution of 1-(4-methoxybenzyloxy)-6-hexanol (0.5 g, 2.10 mmol) in CH_2Cl_2 (10 mL) were added triethylamine (0.42 g, 4.20 mmol), benzoyl chloride (0.29 g, 2.10 mmol) at 0°C and stirred at room temperature for 4h. The reaction mixture was worked up as described for **5** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:4) to afford **6** (0.68 g, 94%) as a syrup.

^1H NMR (CDCl_3 , 200 MHz): 8.02 (d, $J = 7.5$ Hz, 2H), 7.55-7.35 (m, 3H), 7.20 (d, $J = 8.5$ Hz, 2H, Ar-H), 6.80 (d, $J = 8.5$ Hz, 2H, Ar-H), 4.40 (s, 2H), 4.30 (t, $J = 6.2$ Hz, 2H), 3.80 (s, 3H), 3.40 (t, $J = 6.2$ Hz, 2H), 1.80-1.40 (m, 8H); EIMS (m/z): 342 (M^+); IR (neat): 2923, 1755, 1562, 1266, 1032 cm^{-1} . Anal. Calcd. for $\text{C}_{21}\text{H}_{26}\text{O}_4$ (342): C, 73.66; H, 7.65. Found: C, 73.61; H, 7.59%.

1-(6-Allyloxy hexyloxymethyl)-4-methoxybenzene (7):

To a stirred solution of 1-(4-methoxybenzyloxy)-6-hexanol (0.5 g, 2.10 mmol) in dry THF (10 mL), NaH (0.16 g, 4.20 mmol, 60% suspension) was added in portions with constant stirring at 0° C. After 10 min, allyl bromide (0.25 g, 2.10 mmol) was added through a syringe and stirred at room temperature for 5h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **7** (0.51 g, 89%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.23 (d, *J* = 8.4 Hz, 2H, Ar-H), 6.91 (d, *J* = 8.4 Hz, 2H, Ar-H), 5.96-5.81 (m, 1H), 5.24-5.18 (m, 2H), 4.39 (s, 2H), 3.94 (d, *J* = 6.1 Hz, 2H), 3.89 (s, 3H), 3.42-3.37 (m, 4H), 1.64 -1.31 (m, 8H); FABMS (*m/z*): 278 (M⁺), 191(8), 147(16), 137(25), 121(100); IR (neat): 2912, 1590, 1510, 1260, 1110, 1039 cm⁻¹. Anal. Calcd. for C₁₇H₂₆O₃ (278): C, 73.34; H, 9.41. Found: C, 73.29; H, 9.35%.

1-Methoxy-4- 6-(3-methyl-2-butenyloxy) hexyloxymethyl benzene (8):

To a stirred solution of 1-(4-methoxybenzyloxy)-6-hexanol (0.3 g, 1.26 mmol) in dry THF (10 mL), NaH (0.1 g, 2.52 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, prenyl bromide (0.19 g, 1.26 mmol) was added through a syringe and stirred at room temperature for 3h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:4) to afford **8** (0.33 g, 85%) as a syrup.

¹H NMR (CDCl₃, 300 MHz): 7.22 (d, *J* = 8.3 Hz, 2H, Ar-H), 6.92 (d, *J* = 8.3 Hz, 2H, Ar-H), 5.34-5.29 (t, *J* = 6.6 Hz, 1H), 4.39 (s, 2H), 3.9 (d, *J* = 4.8 Hz, 2H), 3.75 (s, 3H), 3.41-3.34 (m, 4H), 1.72 (s, 3H), 1.68 (s, 3H), 1.61-1.32 (m, 8H); EIMS (*m/z*): 237 (M⁺-69), 152, 121, 83; IR (neat): 2937, 2858, 2359, 1512, 1180, 1037 cm⁻¹. Anal. Calcd. for C₁₉H₃₀O₃ (306): C, 74.47; H, 9.87. Found: C, 74.42; H, 9.82%.

5-(4-Methoxybenzyloxymethyl)-2,2,7,7-tetramethyl-(3aR,5R,5aS,8aS,8bR)-perhydrodi 1,3 dioxolo 5,4-b:5,4-d pyran (9)⁸:

To a stirred solution of diacetone galactose (0.3 g, 1.15 mmol) in dry THF (10 mL), NaH (0.1 g, 2.30 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, MPMBBr (0.23 g, 1.15 mmol) was added through a syringe and stirred at room temperature for 5h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:9) to give **9** (0.40 g, 91%) as a syrup.

¹H NMR (CDCl₃, 300 MHz): 7.35 (d, *J* = 8.1 Hz, 2H, Ar-H), 6.85 (d, *J* = 8.1 Hz, 2H, Ar-H), 5.50 (d, *J* = 5.1 Hz, 1H), 4.58-4.48 (m, 3H), 4.30-4.20 (m, 2H), 3.9 (dt, *J* = 6.0, 2.3 Hz, 1H), 3.80 (s, 3H), 3.63-3.50 (m, 2H), 1.53 (s, 3H), 1.45 (s, 3H), 1.31 (s, 6H); EIMS (*m/z*): 379 (*M*⁺-1); IR (neat): 2985, 2935, 1611, 1509, 1377, 1216, 1077 cm⁻¹. Anal. Calcd. for C₂₀H₂₈O₇ (380): C, 63.14; H, 7.42. Found: C, 63.09; H, 7.38%.

6-Benzyloxy-5-(4-methoxybenzyloxymethyl)-2,2-dimethyl-(3aR,5R,6S, 6aR)-perhydrofuro 2,3-d 1,3 dioxole (10):

To a stirred solution of 5-(4-methoxybenzyloxy)-1,2-*O*-isopropylidene- -D-xylofuranose (0.3 g, 0.96 mmol) in dry THF (10 mL), NaH (0.08 g, 1.93 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, benzyl bromide (0.16 g, 0.93 mmol) was added through a syringe and stirred at room temperature for 6h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:4) to afford **10** (0.38 g, 88%) as a syrup.

¹H NMR (CDCl₃, 300 MHz): 7.42-7.30 (m, 7H, Ar-H), 6.80 (d, *J* = 8.6 Hz, 2H, Ar-H), 5.95 (d, *J* = 4.6 Hz, 1H), 4.61 (d, *J* = 4.8 Hz, 1H), 4.53-4.46 (m, 4H), 4.32 (m, 1H), 3.9 (d, *J* = 5.8 Hz, 1H), 3.77 (s, 3H), 3.69-3.61 (m, 2H), 1.45 (s, 3H), 1.39 (s, 3H); FABMS (*m/z*): 400 (*M*⁺), 307(4), 237(14), 136(28), 121(100); IR (neat): 2933, 2859, 1612, 1463, 1247, 1024 cm⁻¹. Anal. Calcd. for C₂₃H₂₈O₆ (400): C, 68.98; H, 7.05. Found: C, 68.92; H, 7.01%.

2- 2-(4-Methoxybenzyloxy)ethyl -6,6-dimethylbicyclo 3.1.1 hept-2-ene (11)⁸:

To a stirred solution of nopol (0.3 g, 1.80 mmol) in dry THF (10 mL), NaH (0.14 g, 3.61 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, MPMBr (0.36 g, 1.80 mmol) was added through a syringe and stirred at room temperature for 4h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **11** (0.42 g, 81%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.28 (d, *J* = 8.8 Hz 2H, Ar-H), 6.86 (d, *J* = 8.8 Hz, 2H, Ar-H), 5.28 (s, 1H), 4.42 (s, 2H), 3.80(s, 3H), 3.50 (t, *J* = 6.8 Hz, 2H), 2.35-1.62 (m, 8H), 1.28 (s, 3H), 1.04 (s, 3H); EIMS (*m/z*): 286 (M⁺); IR (neat): 2914, 1612, 1464, 1248, 1174, 1096 cm⁻¹. Anal. Calcd. for C₁₉H₂₆O₂ (286): C, 79.68; H, 9.15. Found: C, 79.62; H, 9.09%.

1-Isopropyl-2-(4-methoxybenzyloxy)-4-methyl-(1S,2R,4R)-cyclohexane (12)⁸:

To a stirred solution of menthol (0.5 g, 3.20 mmol) in dry THF (10 mL), NaH (0.25 g, 6.40 mmol, 60% suspension) was added in portions with constant stirring at 0°C. After 10 min, MPMBr (0.64 g, 3.20 mmol) was added through a syringe and stirred at room temperature for 6h. The reaction mixture was worked up as described for **1** and purified by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **12** (0.63 g, 71%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.25(d, *J* = 8.5 Hz, 2H, Ar-H), 6.85 (d, *J* = 8.5 Hz, 2H, Ar-H), 4.45 (s, 2H), 3.80 (s, 3H), 3.40 (dt, *J* = 9.7,4.2 Hz, 1H), 2.25-2.10 (m, 1H), 2.0-1.90 (m, 1H), 1.70-1.57 (m, 2H), 1.10-0.85 (m, 11H), 0.80 (d, *J* = 3.4 Hz, 3H); EIMS (*m/z*): 276 (M⁺); IR (neat): 2954, 2869, 1454, 1258, 1043 cm⁻¹. Anal. Calcd. for C₁₈H₂₈O₂ (276): C, 78.21; H, 10.21. Found: C, 78.18; H, 10.15%.

1-(4-Methoxybenzyloxy)-(S)-2-(Boc-amino)-3-phenyl propane (13):

To a stirred solution of N-Boc-L-phenylalaninol (0.3 g, 1.19 mmol) in dry acetone (10 mL), K₂CO₃ (0.33 g, 2.38 mmol) was added in portions with constant stirring at 0°C. After 10 min, MPMBr (0.24 g, 1.19 mmol) was added through a syringe

and stirred at room temperature for 3h. The solvent evaporated and residue diluted with water (10 mL) and extracted into ether (2 20 mL). Combined ether extracts were washed with brine solution (10 mL), dried (Na₂SO₄) and concentrated under reduced pressure and purified the residue by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **13** (0.35 g, 79%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.34-7.29 (m, 5H), 7.24 (d, *J* = 8.4 Hz, 2H, Ar-H), 6.86 (d, *J* = 8.4 Hz, 2H, Ar-H), 4.39 (s, 2H), 3.80 (s, 3H), 3.79-3.76 (m, 1H), 3.58 (d, *J* = 5.9 Hz, 2H), 2.81 (d, *J* = 6.2 Hz, 2H), 1.4 (s, 9H); FABMS (*m/z*): 372 (*M*⁺+1); IR (neat): 2985, 2865, 1745, 1465, 1375, 1260 cm⁻¹.

1-(4-Methoxybenzyloxy)-4- trityloxy butane (14):

To a stirred solution of 1-(4-methoxybenzyloxy)-4-butanol (0.5 g, 2.38 mmol) in CH₂Cl₂ (10 mL) were added triethylamine (0.48 g, 4.76 mmol), trityl chloride (0.66 g, 2.38 mmol) at 0°C and stirred it at room temperature for 2h. The reaction mixture was diluted with water (20 mL) and extracted with CH₂Cl₂ (20 mL). Organic layer was washed with brine (10 mL), dried (Na₂SO₄), concentrated under reduced pressure and purified the residue by column chromatography (silicagel, EtOAc: Hexane; 1:9) to give **14** (0.9 g, 85%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 7.42-7.37 (m, 6H), 7.29-7.24 (m, 9H), 7.22 (d, *J* = 8.3 Hz, 2H, Ar-H), 6.83 (d, *J* = 8.3 Hz, 2H, Ar-H), 4.39 (s, 2H), 3.79 (s, 3H), 3.42-3.39 (t, *J* = 6.1Hz, 2H), 3.1 (t, *J* = 5.9 Hz, 2H), 1.74-1.69 (m, 4H); FABMS (*m/z*): 452 (*M*⁺); IR (neat): 2936, 2859, 1612, 1510, 1246, 1039 cm⁻¹.

4-methoxybenzyl hexanoate (15):

To a stirred solution of MPMOH (0.5 g, 3.62 mmol) in CH₂Cl₂ (10 mL) were added triethylamine (0.73 g, 7.24 mmol), hexanoyl chloride (0.48 g, 3.62 mmol) at 0°C and stirred it at room temperature for 1h. The reaction mixture was diluted with water (20 mL) and extracted with CH₂Cl₂ (20 mL). Organic layer was washed with brine (10 mL), dried (Na₂SO₄), concentrated under reduced pressure and purified the residue by

column chromatography (silicagel, EtOAc: Hexane; 1:4) to give **15** (0.85 g, 92%) as a syrup.

¹H NMR (CDCl₃, 300 MHz): 7.22 (d, *J* = 8.2 Hz, 2H, Ar-H), 6.86 (d, *J* = 8.2 Hz, 2H, Ar-H), 4.98 (s, 2H), 3.78 (s, 3H), 2.24 (t, *J* = 5.9 Hz, 2H), 1.65-1.61 (m, 2H), 1.32-1.27 (m, 4H), 0.9 (t, *J* = 6.2 Hz, 3H); EIMS (*m/z*): 236 (*M*⁺); IR (neat): 2926, 2858, 1750, 1540, 1039 cm⁻¹.

4-methoxybenzyl isonicotinate (16):

To a stirred solution of isonicotinic acid (0.5 g, 4.06 mmol) in dry acetone (10 mL), K₂CO₃ (1.12 g, 8.13 mmol) was added in portions with constant stirring at 0°C. After 10 min, MPMBBr (0.81 g, 4.06 mmol) was added through a syringe and stirred at room temperature for 3h. The solvent evaporated and residue diluted with water (10 mL) and extracted into ether (2 20 mL). Combined ether extracts were washed with brine solution (10 mL), dried (Na₂SO₄) and concentrated under reduced pressure and purified the residue by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **16** (0.80 g, 81%) as a syrup.

¹H NMR (CDCl₃, 200 MHz): 8.73 (d, *J* = 8.2 Hz, 2H), 7.86 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H, Ar-H), 6.86 (d, *J* = 8.4 Hz, 2H, Ar-H), 5.27 (s, 2H), 3.80 (s, 3H). EIMS (*m/z*): 243 (*M*⁺); IR (neat): 2985, 2865, 1745, 1465, 1375, 1260 cm⁻¹.

1-(4-Methoxybenzyl)-(S)-2-(Boc-amino) propanoate (17):

To a stirred solution of N-Boc-alanine (0.5 g, 2.64 mmol) in dry acetone (10 mL), K₂CO₃ (0.73 g, 5.29 mmol) was added in portions with constant stirring at 0°C. After 10 min, MPMBBr (0.53 g, 2.64 mmol) was added through a syringe and stirred at room temperature for 3h. The solvent evaporated and residue diluted with water (10 mL) and extracted into ether (2 20 mL). Combined ether extracts were washed with brine solution (10 mL), dried (Na₂SO₄) and concentrated under reduced pressure and purified the residue by column chromatography (silicagel, EtOAc: Hexane; 1:9) to afford **17** (0.70 g, 86%) as a syrup.

^1H NMR (CDCl_3 , 200 MHz): 7.22 (d, $J = 8.3$ Hz, 2H, Ar-H), 6.85 (d, $J = 8.2$ Hz, 2H, Ar-H), 5.09 (s, 2H), 4.26 (m, 1H), 3.78 (s, 3H), 1.4 (s, 9H), 1.38 (d, $J = 5.6$ Hz, 3H); EIMS (m/z): 310 ($\text{M}^+ + 1$); IR (neat): 2926, 1750, 1540, 1039 cm^{-1} .

General Experimental procedure for deprotection of PMB ethers:

To a stirred solution of PMB ether (1 mmol) in dry acetonitrile, ZrCl_4 (0.2 mmol) was added and stirred at room temperature until the starting material completely disappeared (TLC analysis). The solvent was removed under reduced pressure and residue treated with EtOAc (20 mL). It was washed with water (15 mL), brine (15 mL), dried (Na_2SO_4) and evaporated under reduced pressure. The residue was purified by column chromatography (silicagel, 60-120; hexane: EtOAc) to furnish the alcohols, which were characterized by IR, ^1H NMR and Mass spectra.

2-Phenyl-1-ethanol (1a):

Data is in accordance with the data of a commercially available sample.

1-Octanol (2a):

Data is in accordance with the data of a commercially available sample.

6-Tetrahydro-2H-2-Pyran-2-yl-1-hexanol (3a):

^1H NMR (CDCl_3 , 200 MHz): 4.56 (br.s, 1H), 3.88 (m, 2H), 3.53-3.34 (m, 4H), 1.92-1.34 (m, 14H); EIMS (m/z): 203 ($\text{M}^+ + 1$); IR (neat): 3459, 2936, 1614, 1518 cm^{-1} . Anal. Calcd. for $\text{C}_{11}\text{H}_{22}\text{O}_3$ (202): C, 65.31; H, 10.96. Found: C, 65.25; H, 10.90%.

6-(2-Methoxyethoxy methoxy) -1- hexanol (4a):

^1H NMR (CDCl_3 , 200 MHz): 4.68 (d, $J = 5.61$ Hz, 1H), 4.64 (s, 1H), 3.66-3.61 (t, $J = 5.8$ Hz, 2H), 3.56-3.44 (m, 6H), 3.41 (s, 3H), 1.64-1.32 (m, 8H); EIMS (m/z): 206 (M^+); IR (neat): 3469, 2926, 1521, 1471 cm^{-1} . Anal. Calcd. for $\text{C}_{10}\text{H}_{22}\text{O}_4$ (206): C, 58.23; H, 10.75. Found: C, 58.19; H, 10.70%.

6-Hydroxy hexyl acetate (5a)^{6a}:

¹H NMR (CDCl₃, 200 MHz): 4.14-4.06 (t, *J* = 5.9 Hz, 2H), 3.43-3.40 (t, *J* = 5.9 Hz, 2H), 2.1 (s, 3H), 1.68-1.34 (m, 8H); EIMS (m/z): 160 (M⁺), 142, 121, 91; IR (neat): 3482, 2910, 1750, 1560, 1260 cm⁻¹. Anal. Calcd. for C₈H₁₆O₃ (160): C, 59.97; H, 10.07. Found: C, 59.91; H, 10.02%.

6-Hydroxy hexyl benzoate (6a):

¹H NMR (CDCl₃, 200 MHz): 8.12 (d, *J* = 7.5 Hz, 2H), 7.58-7.38 (m, 3H), 4.34 (t, *J* = 6.2 Hz, 2H), 3.64 (t, *J* = 6.3 Hz, 2H), 1.80-1.44 (m, 8H); EIMS (m/z): 222 (M⁺); IR (neat): 3471, 2914, 1755, 1569, 1268 cm⁻¹. Anal. Calcd. for C₁₃H₁₈O₃ (222): C, 70.24; H, 8.16. Found: C, 70.19; H, 8.13%.

6-Allyloxy-1-hexanol (7a):

¹H NMR (CDCl₃, 200 MHz): 5.96-5.84 (m, 1H) 5.26-5.20 (m, 2H), 3.92 (d, *J* = 6.2 Hz, 2H), 3.58-3.63 (m, 2H) 3.42-3.38 (t, *J* = 5.6 Hz, 2H), 1.63 -1.30 (m, 8H); EIMS (m/z): 158 (M⁺), 141, 121, 83; IR (neat): 3512, 2912, 1590, 1510, 1260 cm⁻¹. Anal. Calcd. for C₉H₁₈O₂ (158): C, 68.31; H, 11.47. Found: C, 68.25; H, 11.42%.

6-(3-Methyl-2-butenyloxy)-1-hexanol (8a):

¹H NMR (CDCl₃, 200 MHz): 5.38-5.32 (t, *J* = 6.6 Hz, 1H), 3.96 (d, *J* = 4.8 Hz, 2H), 3.62 (t, *J* = 6.4 Hz, 2H), 3.36-3.39 (t, *J* = 6.4 Hz, 2H), 1.71 (s, 3H), 1.66 (s, 3H), 1.62-1.34 (m, 8H); EIMS (m/z): 186 (M⁺), 169, 141, 83, 69; IR (neat): 3506, 2942, 2861, 1523 cm⁻¹. Anal. Calcd. for C₁₁H₂₂O₂ (186): C, 70.92; H, 11.90. Found: C, 70.87; H, 11.86%.

2,2,7,7-tetramethyl-(3aR,5R,5aS,8aS,8bR)-perhydrodi 1,3 dioxolo 5,4-b:5,4-d pyran -5-yl methanol (9a):

Data is in accordance with the data of a commercially available sample.

6-Benzyloxy-2,2-dimethyl-(3aR,5R,6S,6aR)-perhydrofuro 2,3-d 1,3 dioxol-5-yl methanol (10a):

¹H NMR (CDCl₃, 200 MHz): 7.23-7.36 (m, 5H), 5.89 (d, *J* = 4.6 Hz, 1H), 4.64 (d, *J* = 4.8 Hz, 1H), 4.51-4.43 (m, 2H), 4.26 (m, 1H), 3.9 (d, *J* = 5.8 Hz, 1H), 3.82-3.78 (m, 2H), 1.47 (s, 3H), 1.38 (s, 3H); FABMS (*m/z*): 281 (*M*⁺+1), 223(62), 181(31), 137(24), 91(100); IR (neat): 3485, 2936, 2849, 1622, 1469 cm⁻¹. Anal. Calcd. for C₁₅H₂₀O₅ (280): C, 64.27; H, 7.19. Found: C, 64.21; H, 7.15%.

2-(6,6-Dimethyl bicyclo 3.1.1 hept-2-en-2-yl)-1-ethanol (11a):

Data is in accordance with the data of a commercially available sample.

2-Isopropyl-5-methyl- (1R,2S,5R)-cyclohexan-1-ol (12a):

Data is in accordance with the data of a commercially available sample.

(S)-2-(Boc-amino)-3-phenyl -1-propanol (13a):

Data is in accordance with the data of a commercially available sample.

1,4-butanediol (14a):

Data is in accordance with the data of a commercially available sample.

Hexanoic acid (15a):

Data is in accordance with the data of a commercially available sample

Isonicotinic acid (16a):

Data is in accordance with the data of a commercially available sample

N-Boc-L-alanine (17a):

Data is in accordance with the data of a commercially available sample