Stereoselective Synthesis of a Mevinic Acid Analogue[1]

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Abstract: An efficient and versatile synthetic method for the stereoselective synthesis of a mevinic acid analogue is described. This approach uses a combination of a Cosford protocol with a catecholborane-mediated stereoselective reduction of acyclic β-hydroxy ketones to *syn*-1,3-diols, as key steps.

Key words: mevinic acid analogue, Cosford protocol, β-hydroxy ketones, catecholborane, *syn*-1,3-diols

Mevinolin, a fungal metabolite isolated from the fungus *Aspergillus sp.*, is a potent anti-hypercholesterolemic agent and a competitive inhibitor of 3-hydroxy-3-methylglutaryl coenzyme A (HMGCoA).[2] Mevinolin and its relatives compactin, pravastatin and simvastatin, are useful in helping to reduce cholesterol and other fat levels in the blood. They also show promise in helping to slow the progression of coronary arthrosclerosis. Such drugs act by blocking enzymes that synthesize cholesterol, while simultaneously increasing the level of high-density lipoproteins. Despite the relatively simple structure of these compounds, the lactone moiety has proved to be essential for the biological activity of such compounds.[3] Thus, the synthesis of many analogues of the lactone moiety have been reported in literature.[4]

An efficient protocol for the cross-coupling of alkynes with aryl and heteroaryl substrates using a Pd/C-CuI-PPh_3 catalytic system in aqueous media has been developed by Cosford and coworkers,[5] that has wide applicability to the formation of C–C bonds in organic synthesis. Similarly the stereoselective reduction of acyclic β-hydroxy ketones to *syn*-1,3-diols can be achieved using catecholborane as an effective reagent.[6] We envisaged that by combining these two methods, an effective synthetic strategy for the preparation of mevinic acid analogues could be developed.

The synthesis of mevinic acid analogue **1** could be achieved by combining the Cosford protocol with the stereoselective reduction of acyclic β-hydroxy ketones to *syn*-1,3-diols using catecholborane. Thus, the enantioselective synthesis of mevinic acid analogue started from the key intermediate **4**. Coupling of iodobenzene **2** and alkyne **3**[7] was achieved by heating in 1,2-dimethoxyethane/water solution in the presence of potassium carbonate and catalytic amounts of Pd/C, CuI, and PPh_3, to afford the propargylic alcohol **4** in 90% yield (Scheme 1).

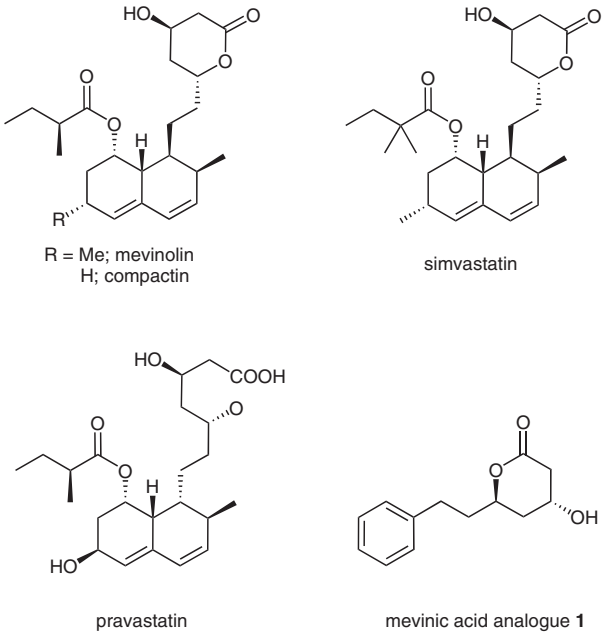
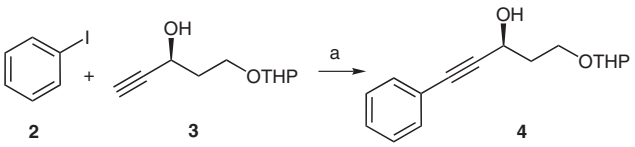


Figure 1



Scheme 1 *Reagents and conditions*: (a) 10% Pd/C (cat.), CuI (4 equiv), Ph_3P (0.1 equiv), K_2CO_3, H_2O–DME, 80 °C, 2 h, 90%.

The triple bond of compound **4** was reduced using Pd/C in ethanol to afford the saturated alcohol **5** in 90% yield. The secondary hydroxy group of **5** was protected as the corresponding silyl ether **6** and the deprotection of the tetrahydropyran (THP) group was achieved using pyridinium *p*-toluenesulfonate (PPTS) in methanol affording **7**. The primary alcohol **7**, upon oxidation, resulted in the corresponding aldehyde **8** in 90% yield, which was converted into β-keto ester **9** (80% yield) by the reaction with ethyl diazoacetate in the presence of a catalytic amount of tin(II)chloride. Desilylation of **9** was achieved using tetrabutylammonium fluoride (TBAF) to afford β-hydroxy-β-keto ester **10** in 85% yield. The stereoselective reduction of **10** to the corresponding *syn*-1,3-diol **11**, in 95% yield with high diastereoselectivity, was achieved using

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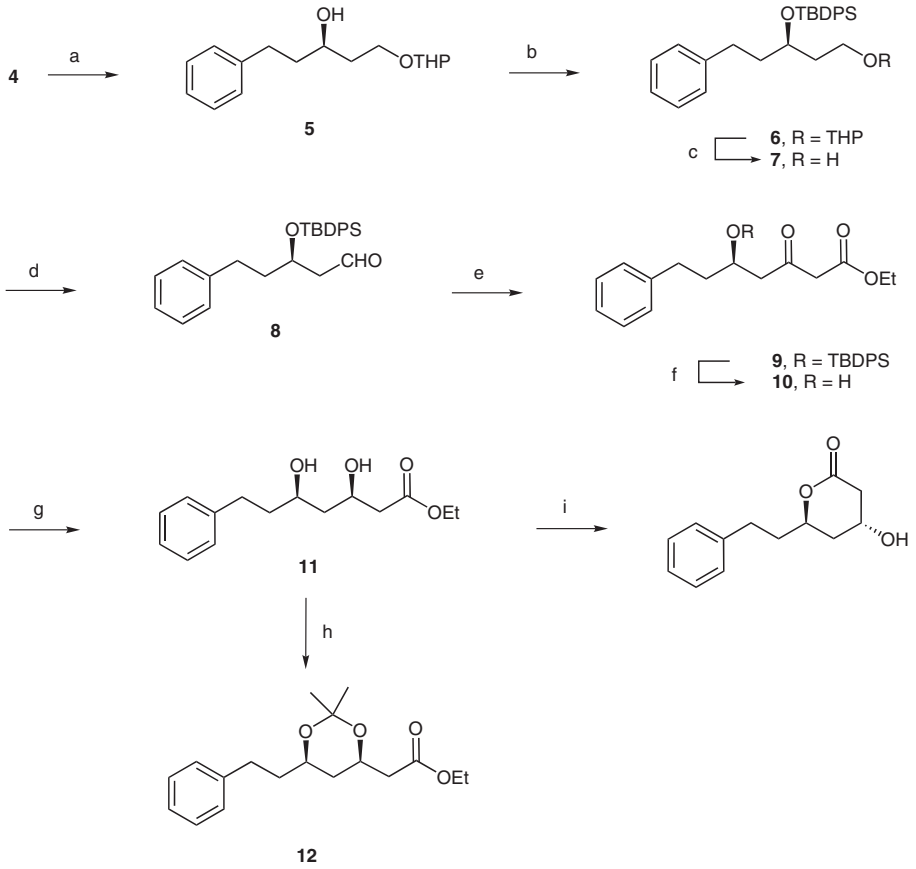

Scheme 2 *Reagents and conditions*: (a) Pd/C, EtOH, 2 h, 90%; (b) TBDPSCl, imidazole, CH_2Cl_2, DMAP, 2 h, 95%; (c) PPTS, MeOH, 12 h, 90%; (d) 2-iodoxybenzoic acid, DMSO, CH_2Cl_2, 0 °C to r.t., 2 h, 90%; (e) anhyd $SnCl_2$ (cat.), $N_2CHCOOEt$, CH_2Cl_2, 0 °C to r.t., 40 min, 80%; (f) TBAF, THF, 2 h, 85%; (g) catecholborane, THF, –10 °C, 4 h, 95%; (h) 2,2-dimethoxypropane, PPTS, 87%; (i) PTSA, CH_2Cl_2, 3 h, 84%.

catecholborane (2.2 equiv). The stereochemistry of *syn*-diol **11** was confirmed by analyzing the ^{13}C NMR spectral data of the corresponding acetonide **12**. Characteristic peaks at δ = 19.8, 30.1 and 99.0 ppm in the ^{13}C NMR spectra of 1,3-diol[8] **12**, confirmed the *syn*-geometry of the diol. Compound **11**, on treatment with *p*-toluenesulfonic acid (PTSA) in dichloromethane, furnished the target lactone **1** in 84% yield (Scheme 2). The lactone **1** showed ^1H and ^{13}C NMR spectral data and optical rotation {$[\alpha]_D^{25}$ +47.2 (*c* 0.3, $CHCl_3$)} in good agreement with the previously reported compound.[9]

In summary, we have applied a Cosford cross-coupling protocol for the reaction of iodobenzene with an acetylenic alcohol, along with a stereoselective reduction of an acyclic β-hydroxy ketone to the *syn*-1,3-diol using catecholborane, as key steps in the synthesis of a mevinic acid analogue. This strategy provides rapid access to a range of analogues of the lactone moiety of mevinolin.

Reactions were conducted under nitrogen atmosphere using anhydrous solvents. All reactions were monitored by thin layer chromatography (TLC) using Merck 60 F-254 silica gel plates with UV visualization. Light petroleum had a distillation range 60–80 °C. Yields refer to chromatographically and spectroscopically (^1H, ^{13}C) homogeneous material. Air sensitive reagents were transferred by syringe or with a double-ended needle. Evaporation of solvents was performed at reduced pressure, using a Büchi rotary evaporator. ^1H NMR spectra were recorded on Varian FT-200MHz (Gemini) or a Bruker UXNMR FT-300MHz (Avance) in $CDCl_3$. Chemical shift values are reported in parts per million (δ) relative to TMS (δ = 0.0) as an internal standard. Mass spectra were recorded under EI at 70eV on an LC-MSD (Agilent technologies). Column chromatography was performed on silica gel (60–120 mesh) supplied by Acme Chemical Co., India. Optical rotations were measured with a JASCO DIP-370 Polarimeter at 20 °C.

(3*S*)-1-Phenyl-5-(tetrahydro-2*H*-2-pyranyloxy)pent-1-yn-3-ol (4)

To a solution of **2** (3 g, 14.7 mmol) in DME (60 mL) was added H_2O (25 mL), K_2CO_3 (5 g, 36.75 mmol), CuI (0.11 g, 0.58 mmol), Ph_3P (0.29 g, 1.17 mmol) and a catalytic amount of10% Pd/C. The resulting mixture was stirred at r.t. for 30 min, then compound **3** (4.3 g, 22.0 mmol) was added and the reaction was warmed to 80 °C for 2 h. The mixture was cooled to r.t., filtered through a celite pad and washed with EtOAc (100 mL). The solution was diluted with H_2O (100 mL) and extracted with EtOAc (2 × 300 mL). The organic phase was washed with brine (100 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by column chromatography (*n*-hexane–EtOAc, 70:30) to afford **4**.

Yield: 5 g (90%); colorless liquid; $[\alpha]_D^{25}$ +12.5 (*c* 1.00, $CHCl_3$).

IR (neat): 2943, 1618, 1490, 1353, 1200, 1073, 1034, 757, 692 cm^{-1}.

^1H NMR (400 MHz, $CDCl_3$): δ = 1.46–1.88 (m, 6 H), 1.93–2.22 (m, 2 H), 2.85 (br s, 1 H), 3.42–4.24 (m, 4 H), 4.59–4.82 (m, 1 H), 4.79 (dd, *J* = 4.6, 7.0 Hz, 1 H), 7.23–7.31 (m, 3 H), 7.35–7.43 (m, 2 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 131.5, 128.1, 122.5, 98.8, 89.4, 84.7, 64.6, 62.1, 61.4, 36.8, 30.3, 25.1, 19.2.

LC-MS: m/z = 283 $[M + Na]^+$.

(3*R*)-1-Phenyl-5-(tetrahydro-2*H*-2-pyranyloxy)pentan-3-ol (5)
To a solution of **4** (3 g, 11.5 mmol) in anhyd EtOAc (10 mL) was added a catalytic amount of 10% Pd/C and the mixture was stirred at r.t. under a H_2 atmosphere for 6 h. The catalyst was filtered off and washed with EtOAc (100 mL) and the filtrate was concentrated under reduced pressure. Purification by column chromatography (*n*-hexane–EtOAc, 75:25) afforded pure **5**.

Yield: 2.7 g (90%); colorless liquid; $[\alpha]_D^{25}$ +40.3 (*c* 0.5, $CHCl_3$).

IR (neat): 3451, 2925, 2364, 1639, 1454, 1354, 1209, 1027, 981 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.45–1.92 (m, 10 H), 2.56–2.87 (m, 2 H), 2.86–2.99 (br s, 1 H, OH), 3.43–3.66 (m, 2 H), 3.74–4.03 (m, 3 H), 4.52–4.63 (m, 1 H) 7.08-7.37 (m, 5H).

LC-MS: m/z = 287 $[M + Na]^+$.

***tert*-Butyl[(1*R*)-1-phenethyl-3-(tetrahydro-2*H*-2-pyranyloxy)propyl]oxydiphenylsilane (6)**
To a stirred solution of alcohol **5** (2.5 g, 9.4 mmol) and imidazole (1.2 g, 18.8 mmol) in anhyd CH_2Cl_2 (15 mL) was added TBDPS-Cl (3.1 g, 11.3 mmol), portion-wise, at 0 °C. The reaction mixture was stirred at the same temperature for 2 h and then quenched with H_2O. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (2 × 20 mL). The combined organic layers were washed with H_2O (40 mL), brine (20 mL) and dried (Na_2SO_4). The solvent was removed in vacuo and the residue was purified by column chromatography (*n*-hexane–EtOAc, 95:5) to afford **6**.

Yield: 4.5 g (95%); colorless liquid; $[\alpha]_D^{25}$ –15.8 (*c* 0.5, $CHCl_3$).

IR (neat): 2929, 2856, 2356, 1558, 1427, 1260, 1067, 857 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.06 (s, 9 H), 1.36–1.61 (m, 6 H), 1.63–1.90 (m, 4 H), 2.55 (t, *J* = 8.30 Hz, 2 H), 3.27–3.45 (m, 2 H), 3.61–3.77 (m, 2 H) 3.87–4.02 (m, 1 H) 4.43 (t, *J* = 3.02 Hz, 1 H), 6.89–6.97 (m, 2 H), 7.02–7.20 (m, 3 H), 7.28–7.43 (m, 5 H), 7.61–7.71 (m, 5 H).

LC-MS: m/z = 525 $[M + Na]^+$.

(3*R*)-3-[1-(*tert*-Butyl)-1,1-diphenylsilyl]oxy-5-phenylpentan-1-ol (7)
To a stirred solution of compound **6** (4.5 g, 8.9 mmol) in MeOH (20 mL) was added a catalytic amount of PPTS (0.2 g, 0.89 mmol). The reaction mixture was stirred at r.t. for 2 h then the MeOH was removed under reduced pressure. The crude residue was purified by column chromatography (*n*-hexane–EtOAc, 80:20) to afford **7**.

Yield: 3.3 g (90%); viscous colorless liquid; $[\alpha]_D^{25}$ –32.6 (*c* 0.5, $CHCl_3$).

IR (neat): 3067, 2931, 2857, 1592, 1426, 1364, 1106, 1058, 820 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.07 (s, 9 H), 1.58 (br s, OH), 1.62–1.92 (m, 4 H), 2.37–2.53 (m, 2 H), 3.56–3.79 (m, 2 H), 3.92–4.02 (m, 1 H), 6.84–6.91 (m, 2 H), 7.03–7.19 (m, 3 H), 7.30–7.46 (m, 3 H), 7.62–7.72 (m, 5 H).

LC-MS: m/z = 441 $[M + Na]^+$.

(3*R*)-3-[1-(*tert*-Butyl)-1,1-diphenylsilyl]oxy-5-phenylpentanal (8)
To an ice-cold solution of 2-iodoxybenzoic acid (2.4 g, 8.6 mmol) in DMSO (3 mL, 42.6 mmol) was added a solution of alcohol **7** (3 g, 7.1 mmol) in anhyd CH_2Cl_2 (10 mL). The mixture was stirred at r.t. for 2 h and then filtered through a celite pad and washed with Et_2O (100 mL). The combined organic filtrates were washed with H_2O (100 mL) and brine (50 mL), dried (Na_2SO_4) and concentrated in vacuo. The crude product was purified by column chromatography (*n*-hexane–EtOAc, 80:20) to afford aldehyde **8**.

Yield: 2.6 g (90%); viscous colorless liquid; $[\alpha]_D^{25}$ –14.4 (*c* 0.7, $CHCl_3$).

IR (neat): 3068, 2929, 2855, 1721, 1590, 1426, 1363, 1188, 1003, 820 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.06 (s, 9 H), 1.76–1.88 (m, 2 H), 2.46–2.58 (m, 4 H), 4.19–4.28 (m, 1 H), 6.88–6.96 (m, 2 H), 7.03–7.21 (m, 3 H), 7.27–7.49 (m, 5 H), 7.58–7.73 (m, 5 H), 9.65 (t, *J* = 2.45 Hz, 1 H).

LC-MS: m/z = 439 $[M + Na]^+$.

Ethyl (5*R*)-5-[1-(*tert*-Butyl)-1,1-diphenylsilyl]oxy-3-oxo-7-phenylheptanoate (9)
To anhyd tin(II) chloride (0.1 g, 0.48 mmol) was added CH_2Cl_2 (15 mL) followed by ethyl diazoacetate (0.6 g, 5.2 mmol) at r.t. while stirring. A few drops of **8** (2 g, 4.8 mmol) in dry CH_2Cl_2 (5 mL) were slowly added and, after nitrogen evolution had begun, the remainder of the solution was added dropwise over 10 min. After nitrogen evolution had stopped (~30 min) the reaction was transferred to a separating funnel with saturated brine (20 mL) and extracted into Et_2O (50 mL). The organic layers were combined, dried (Na_2SO_4) and the solvent was removed in vacuo to give a residue that was purified by silica gel column chromatography (*n*-hexane–EtOAc, 90:10) to afford **9**.

Yield: 1.9 g (80%); viscous colorless liquid; $[\alpha]_D^{25}$ –16.2 (*c* 0.5, $CHCl_3$).

IR (neat): 3448, 2926, 2855, 2361, 1717, 1636, 1461, 1231, 1108, 820 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 1.05 (s, 9 H), 1.28 (t, *J* = 7.03 Hz, 3 H), 2.67 (d, *J* = 5.46 Hz, 2 H), 3.18 (s, 2 H), 4.12 (q, *J* = 7.03 Hz, 2 H), 4.18–4.31 (m, 1 H), 6.87–7.24 (m, 5 H), 7.27–7.48 (m, 5 H), 7.58–7.76 (m, 5 H).

LC-MS: m/z = 525 $[M + Na]^+$.

Ethyl (5*R*)-5-Hydroxy-3-oxo-7-phenylheptanoate (10)
To compound **9** (2 g, 3.9 mmol) in anhyd THF (10 mL) was added TBAF (1 M in THF, 3.9 mL, 3.9 mmol), dropwise at 0 °C, and the mixture was stirred for 30 min. H_2O (2 mL) was added and the mixture was extracted with EtOAc (50 mL). The organic extracts were washed with brine (20 mL), dried (Na_2SO_4), and the solvent was evaporated to give a residue that was purified by column chromatography (*n*-hexane–EtOAc, 60:40) to afford the product **10**.

Yield: 0.89 g (85%); colorless liquid; $[\alpha]_D^{25}$ –12.5 (*c* 1.0, CH_2Cl_2).

IR (film): 3442, 3027, 2931, 1741, 1712, 1496, 1455, 1409, 1368, 1318, 1155, 1029, 750, 701 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 1.29 (t, *J* = 7.70 Hz, 3 H), 1.56–1.88 (m, 2 H), 2.60–2.88 (m, 4 H), 3.39 (s, 2 H), 3.96–4.11 (m, 1 H), 4.18 (q, *J* = 14.4 Hz, 2 H), 7.05–7.43 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 203.5, 166.8, 141.6, 128.3 (2 × C), 125.8, 66.7, 61.4, 49.8, 49.6, 38.0, 31.6, 14.0.

LC-MS: m/z = 287 $[M + Na]^+$.

Ethyl (3*R*,5*R*)-3,5-Dihydroxy-7-phenylheptanoate (11)
A solution of **10** (0.5 g, 1.89 mmol) in anhyd THF (10 mL) was chilled in a MeOH–ice bath (–10 °C) and freshly distilled catecholborane (0.56 g, 4.72 mmol) was added. After 5 h, the reaction mixture was quenched by the addition of anhyd MeOH (1 mL) and sat. aq sodium potassium tartrate (2 mL). The mixture was allowed to stir at r.t. for 1 h, and the desired product was purified by column chromatography (*n*-hexane–EtOAc, 40:60) to afford the diol **11**.

Yield: 0.47 g (95%); colorless liquid; $[\alpha]_D^{25}$ –4.8 (*c* 1.0, $CHCl_3$).

IR (KBr): 3444, 2923, 2853, 2361, 1729, 1456, 1376, 1159, 1028 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.28 (t, *J* = 7.1 Hz, 3 H), 1.51–1.89 (m, 4 H), 2.43 (t, *J* = 7.5 Hz, 2 H), 2.62–2.84 (m, 2 H), 3.79–3.94 (m, 1 H), 4.20–4.26 (m, 1 H), 4.16 (q, *J* = 14.1 Hz, 2 H), 7.09–7.27 (m, 5 H).

LC-MS: *m*/*z* = 289 $[M + Na]^+$.

Ethyl 2-[(4*R*,6*R*)-2,2-Dimethyl-6-phenethyl-1,3-dioxan-4-yl]acetate (12)

To a solution of diol **11** (0.1 g, 3.7 mmol) in anhyd acetone (5 mL), 2,2-dimethoxy propane (0.07 mL, 0.55 mmol) and PPTS (0.009 g, 0.37 mmol) were added. The mixture was stirred at r.t. for 12 h, then $NaHCO_3$ was added to neutralize the PPTS, and filtered. Removal of the solvent under reduced pressure gave a residue that was purified by silica gel column chromatography (*n*-hexane–EtOAc, 90:10) to afford the acetonide **12**.

Yield: 0.1 g (87%); colorless liquid; $[\alpha]_D^{25}$ –8.6 (*c* 1.0, $CHCl_3$).

IR (KBr): 3451, 2989, 2936, 1737, 1636, 1454, 1379, 1263, 1165, 1026, 950 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.26 (t, *J* = 7.1 Hz, 3 H,), 1.36 (s, 3 H), 1.41 (s, 3 H), 1.48–1.86 (m, 4 H), 2.25–2.53 (m, 2 H), 2.55–2.79 (m, 2 H) 3.69–3.82 (m, 1 H), 4.12 (q, *J* = 7.1 Hz, 2 H), 4.14–4.26 (m, 1 H), 7.1–7.24 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 14.2, 19.8, 30.1, 31.0, 37.0, 38.0, 41.5, 60.5, 66.0, 67.7, 99.0, 126.0, 128.4, 128.5, 142.0, 171.0.

LC-MS: *m*/*z* = 329 $[M + Na]^+$.

(4*R*,6*R*)-4-Hydroxy-6-phenethyltetrahydro-2*H*-2-pyranone (1)

To a stirred solution of **11** (0.3 g, 1.12 mmol) in anhyd CH_2Cl_2 (5 mL) was added a catalytic amount of PTSA under an N_2 atmosphere. The mixture was stirred at r.t. for 6 h, then the reaction was quenched by the addition of solid $NaHCO_3$ (0.004 g, 0.05 mmol). The mixture was filtered and the solvent was removed from the filtrate under reduced pressure. The residue was purified by column chromatography (*n*-hexane–EtOAc, 40:60) to afford **1**.

Yield: 0.2 g (84%); colorless solid; $[\alpha]_D^{25}$ +47.2 (*c* 0.3, $CHCl_3$).

IR (KBr): 3442, 2980, 1730, 1650, 1435, 1370, 1135, 1036 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 1.0–1.5 (br s, 1 H), 2.09–1.71 (m, 4 H), 2.59–2.96 (m, 4 H), 4.32–4.39 (m, 1 H), 4.60–4.71 (m, 1 H), 7.11–7.30 (m, 5 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 32.0, 36.0, 37.5, 38.6, 75.0, 77.0, 126.0, 128.3, 128.5, 141.1, 170.5.

LC-MS: *m*/*z* = 243 $[M + Na]^+$.

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