A concise synthesis of (5*R*,6*S*)-*tert*-butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate

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Dedicated to Professor Richard R. Schmidt on the occasion of his 78th birthday

Abstract

An efficient synthesis of (5*R*,6*S*)-*tert*-butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate **1**, a common intermediate for various polyhydroxylated piperidines is reported in six steps with 32% overall yield starting from Garner's aldehyde. The key steps include the diastereoselective nucleophilic addition and intramolecular cyclization. (5*R*,6*S*)-*tert*-butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate is a common precursor for the synthesis of 1-deoxy-L-mannojirimycin, 1-deoxy-L-idonojirimycin, L-fagomycin and related analogues.

Keywords: Garner's aldehyde, nucleophilic addition, cyclization, polyhydroxy piperidines

Introduction

Recent years have witnessed an increasing interest in the synthesis and naturally occurring polyhydroxylated piperidine alkaloids as biological tools and potential therapeutics.[1] This has resulted in onslaught of activity towards construction of various versatile chiral building blocks that would provide us with powerful tools for the synthesis of various biologically active natural products.[2]

Reported non-chiral approach towards synthesizing chiral building blocks for synthesis of 1-deoxy-L-mannojirimycin **(2)**, 1-deoxy-L-idonojirimycin **(3)** and 1-deoxy-L-allonojirimycin **(4)** involves multi-steps and highly expensive reagents.[3] Compound **1** serves as versatile synthetic intermediate for a variety of hydroxylated piperidine derivatives, such as **2-7** (Figure 1).

Though, there are few synthesis of this chiral building block employing α-amino aldehyde[2a,4] as the synthetic precursor, they are mainly derived with use of some specific reagents or suffer from harsh reaction conditions. Carbohydrates based approach[5] towards these compounds, in general requires a large number of steps to reach a specific target. Because of the role of

polyhydroxylated piperidines as potential drugs to treat a variety of carbohydrate- mediated diseases[6] and the exceptional usage of α-amino aldehyde as a building block, we considered it worthwhile using Garner's aldehyde as starting material in synthesizing different hydroxylated piperidine alkaloids and its congeners of biological interest. As a part of our research interest on the enantioselective synthesis of polyhydroxylated alkaloids,[7] herein we report the synthesis of (5*R*,6*S*)-*tert*-butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate starting from Garner's aldehyde, using diasteroselective nucleophilic addition and intramolecular cyclization as key steps.

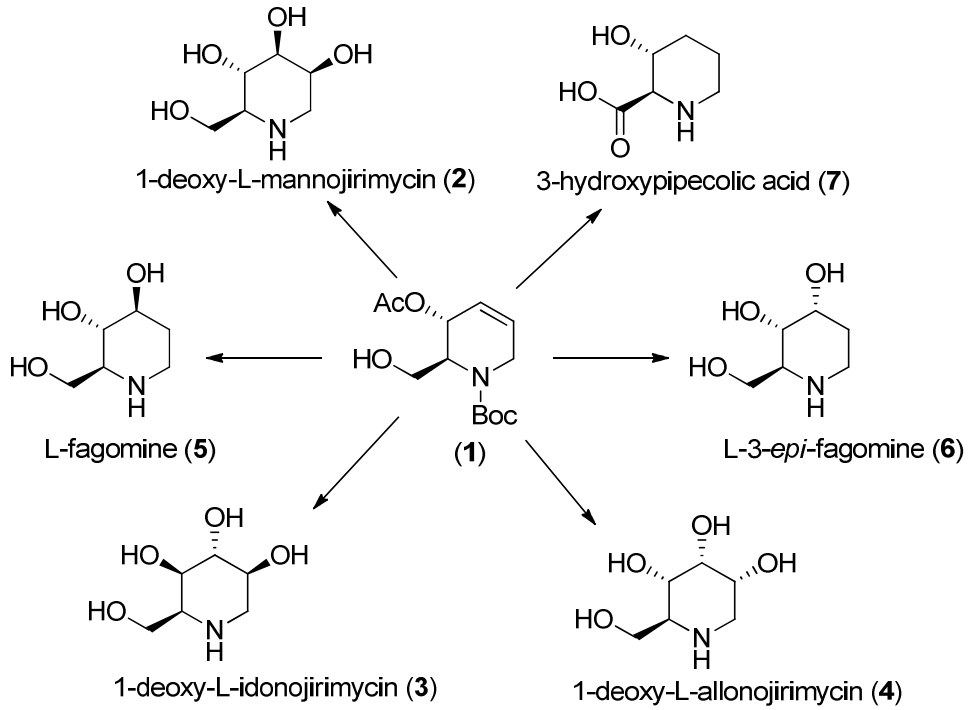
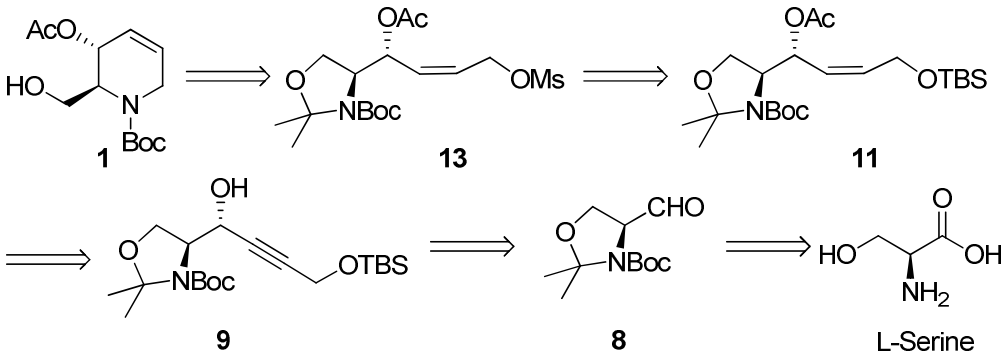


Figure 1. Structures of polyhydroxylated piperidine alkaloids.

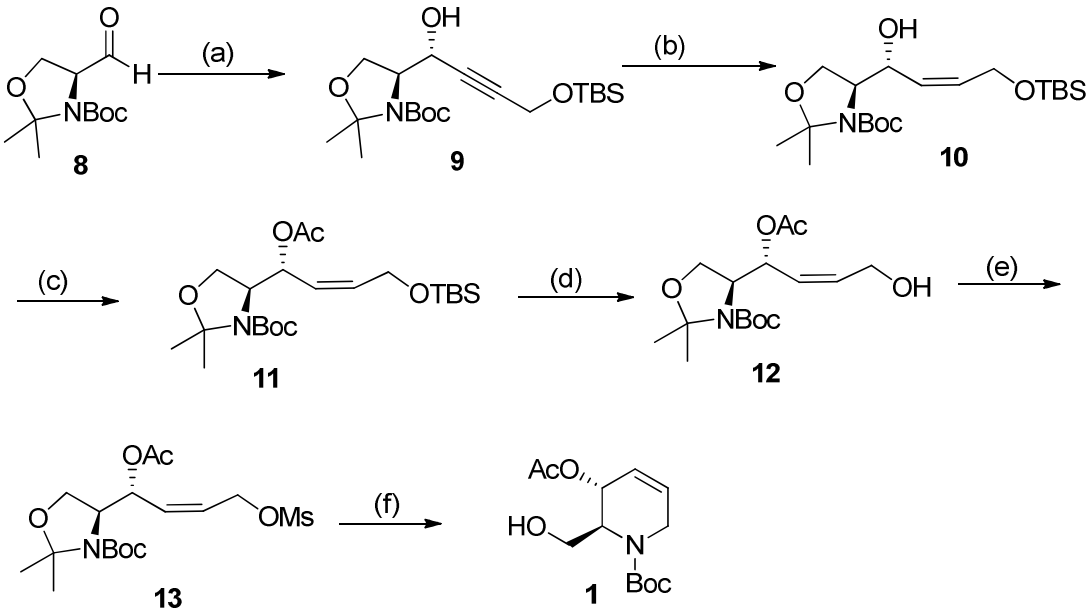


Scheme 1. Retrosynthetic route to the target molecule (**1**).

As seen from the retrosynthetic analysis, compound**9** is a key intermediate for the synthesis of desired chiral building block**1** which could then be easily transformed into various polyhydroxypiperidine alkaloids and its congeners such as 1-deoxy-L-mannonojirimycin (**2)**, 1-deoxy-L-allonojirimycin**(4)** by direct asymmetric dihydroxylation reaction using different chiral ligands. Inversion at C-4 position using Mitsunobu conditions gives the intermediate which can be easily transformed into other polyhydroxylated piperidine alkaloids such as, 1-deoxy-L-idonojirimycin **(3)** whereas L-fagomine (**5)**, L-3-*epi*-fagomine (**6)** and 3-hydroxypipecolic acid **(7)** could be synthesized by routine organic transformations which renders our synthetic intermediate **1** as a powerful tool for the synthesis of various polyhydroxy piperidine alkaloids.

Results and Discussion

We envisioned that the synthesis of chiral building block **1** could be achieved by the diasteroselective addition of lithium derivative of *tert*-butyldimethylsilyl propargyl ether to Garner aldehyde, followed by reduction of the double bond to give the *cis* olefin which could be easily transformed into the desired chiral building block **1** by standard synthetic manipulation.
As illustrated in Scheme 2, our synthesis towards the target molecule **1** began with commercially available chiral pool starting material Garner's aldehyde.[8] Addition of *tert*-butyldimethylsilyl propargyl ether to Garner's aldehyde using *n*-butyl lithium/HMPA in toluene at -78 ^{o}C gave the addition product **9** in the ratio of 5:95 (*syn:anti*) in 71% yield.[9] Partial reduction of **9** with Lindlar's catalyst gave the *cis*-olefin **10** in essentially quantitative yield. The secondary hydroxyl group of **10** was converted into its acetate **11** in 92% yield using acetic anhydride/dry pyridine in anhydrous CH_2Cl_2. The *t*-butyldimethylsilyl group of **11** was deprotected using TBAF in dry THF to give **12** in 85% yield. The primary alcohol functionality of **12** was mesylated using methanesulfonyl chloride in presence of pyridine at 0 °C to give **13** in 86% yield. Global deprotection using PTSA followed by intramolecular cyclization of **13** using DIPEA as a base in anhydrous CH_2Cl_2 and *in situ* Boc protection furnished the required chiral building block **1** in 68% yield.



Reagents and Conditions: (a) *t*-Butyldimethylsilylpropargyl ether, *n*-BuLi, HMPA, -78 °C, 6 h, 71% (b) Lindlar's catalyst, ethyl acetate, H_2, 6 h, 99% (c) Ac_2O, pyridine, DMAP, CH_2Cl_2, 0 °C, 4 h, 92% (d) TBAF/THF, 0 °C, 8 h, 85% (e) MsCl, pyridine, CH_2Cl_2 (dry), 0 °C, 4 h, 86% (f) *p*-TSA, CH_2Cl_2, 10 h; DIPEA, 2 h; $(Boc)_2O$, rt, 6 h, 68%.

Scheme 2. Synthesis of (5*R*,6*S*)-*tert*-butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate (**1**).

Conclusions

In conclusion, we have developed a simple, short and efficient route to (5*R*,6*S*)-*tert*-butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate which can easily be transformed into various polyhydroxypiperidine alkaloids.

Experimental Section

General. All reactions were carried out under argon or nitrogen in oven-dried glassware using standard gas-light syringes, cannulas, and septa. Solvents and reagents were purified and dried by standard methods prior to use. ^{1}H NMR spectra were recorded on 200 MHz, 300 MHz, 400 MHz NMR machine and are reported in parts per million (δ) downfield relative to $CDCl_3$ as internal standard and ^{13}C NMR spectra were recorded at 50 MHz, 75 MHz and 125 MHz and assigned in parts per million (δ) relative to $CDCl_3$. Optical rotations were measured using sodium

D line on JASCO-181 digital polarimeter. Infrared spectra were recorded on Perkin-Elmer FT-IR spectrophotometer. Mass spectra were obtained with a Finnigan MAT-1020-B-70 e*V* mass spectrometer. Elemental analysis was carried out on Carlo Erba CHNS-O analyzer. Column chromatography was performed on silica gel (60-120 mesh) using a mixture of petroleum ether and ethyl acetate as eluent.

(*S*)-*tert*-Butyl 4-((*R*)-4-((*tert*-butyldimethylsilyl)oxy)-1-hydroxybut-2-yn-1-yl)-2,2-dimethyl-oxazolidine-3-carboxylate (**9**). A solution of *tert*-butyldimethylsilyl propargyl ether (10.41 g, 61.13 mmol) and toluene (100 mL) was cooled under argon at -78 °C, and then n-butyllithium (1.6 M in toluene 37.1 mL, 59.30 mmol) was added followed by HMPA (10.37 mL, 59.57 mmol). The stirring was continued for another 2 h, then the solution of the protected serinal **8** (7.9 g, 34 mmol) in toluene (30 mL) was added dropwise. After 3 h, the reaction mixture was allowed to warm to room temperature and stirred for additional 1 h. Then the reaction mixture was poured into 1 M aqueous NaH_2PO_4 (20 mL). It was then extracted with ethyl acetate (3 × 100 mL), and the organic layer was worked up in a usual manner to give crude adduct which after column chromatography using petroleum ether-ethyl acetate (95:5 to 85:15) afforded 9.6 g of *anti* **9** (71% yield) and 0.70 g of *syn* product. $[\alpha]_D^{20}$**:-** 41.1 [*c*, 1, $CHCl_3$] {lit.[9] $[\alpha]_D^{20}$: - 40.7 (*c*, 1.2, $CHCl_3$)} IR (Neat, cm^{-1}): ν_{max} 3447, 2310, 2207, 1734, 1695, 1472, 1368,1252. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.10 (s, 6H), 0.89, (s, 9H), 1.50 (s, 12H); 1.60 (s, 3H), 1.72 (brs, 1H), 3.87-3.97 (m, 1H), 4.00-4.10 (m, 1H), 4.10-4.19 (m, 1H), 4.34 (s, 2H), 4.58 (m, 0.5H), 4.81 (m, 0.5 H), 4.81 (m, 0.5 H). ^{13}C NMR (125 MHz, $CDCl_3$): δ -5.8, 17.9, 25.5, 26.7, 62.3, 63.4, 77.6, 79.4, 95.8, 108.7, 180.5. Mass: 439 (M^+ +K), 423 (M^+ +Na), 400 (M+), 382, 345, 289.

(*S*)-*tert*-Butyl 4-[(*R,Z*)-4-[(*tert*-butyldimethylsilyl)oxy]-1-hydroxybut-2-en-1-yl)-2,2-dimeth-yloxazolidine-3-carboxylate (**10**). A solution of **9** (2 g, 5 mmol) and Lindlar's catalyst (25 mg) in anhydrous ethyl acetate (50 mL) was stirred under H_2 atmosphere for 6 h. After completion of the reaction, the reaction mixture was filtered through a pad of celite and concentrated to give **10** (2 g, 99%) as pale yellow oil. $[\alpha]_D^{20}$**:**-38.19 (*c*, 0.98, $CHCl_3$) IR (Neat): ν_{max} 3385, 3019, 2400, 1690, 1503, 1472, 1392, 1368, 1255, 1215 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.05 (s, 6H), 0.85 (s, 9H), 1.47 (s, 12H), 1.54 (s, 3H), 3.25-4.17 (m, 4H), 4.20-4.38 (m, 2H), 4.60 (s, 1H), 5.45- 5.56 (m, 1H), 5.65-5.75 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 5.4, 18.1, 20.9, 25.8, 28.3, 59.6, 63.8, 68.8, 69.4, 80.3, 94.5, 124.9, 135.8, 152.4, 169.5. Mass: 440 (M^+ +K), 424 (M^+ +Na), 402 (M+) 358, 301. Analysis calcd for $C_{20}H_{39}NO_5Si$ (401.758): Found C, 59.80; H, 9.20; N, 4.21; required C, 59.56; H, 9.31; N, 4.40.

(*S*)-*tert*-Butyl 4-((*R,Z*)-1-acetoxy-4-((*tert*-butyldimethylsilyl)oxy)but-2-en-1-yl)-2,2-dimethyl-oxazolidine-3-carboxylate (**11**). To an ice cold stirred solution of compound **10** (1.8 g, 4.47 mmol) in anhydrous CH_2Cl_2 (50 mL) was added pyridine (0.32 mL, 3.7 mmol) and acetic anhydride (332 mg, 0.21 mL, 2.3 mmol) and stirred for 4 h. After the reaction completion, the reaction mixture was poured into 50 g of crushed ice. The organic layer after separation was washed with dilute solution of $CuSO_4$. After drying over anhydrous Na_2SO_4 and concentration, the crude product was purified through silica gel column chromatography using petroleum ether-

EtOAc (90:10) as eluent to give compound **11** (1.82 g, 92%) as pale yellow oil. **$[\alpha]_D^{20}$:** –15.26 (*c*, 1.04, $CHCl_3$). IR (Neat): ν_{max} 3016, 1754, 1722, 1471, 1406, 1389, 1362, 1216, 1256, 1152, 1103 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 0.06 (s, 6H), 0.88 (s, 9H), 1.49 (s, 15H), 2.05 (s, 3H), 3.87-4.05 (m, 3H), 4.22-4.36 (m, 1H), 4.41 -4.51 (m, 1H), 5.22 – 5.40 (m, 1H), 5.65-5.84 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$): δ -5.6, -5.5, 17.8, 20.7, 22.9, 24.1, 25.5, 25.7, 26.7, 28.0, 58.8, 59.3, 63.3, 63.5, 68.3, 69.1, 79.8, 93.3, 94.2, 124.5, 124.7, 134.8, 135.4, 151.3, 152.1, 168.8, 169.2. LCMS: 466 (M^+ +Na). Anal: calcd for $C_{22}H_{41}NO_6Si$ (443.653): C, 59.56; H; 9.31; N, 3.16.; found C, 59.32; H, 9.27; N, 3.36%.

(*S*)-*tert*-Butyl 4-((*R*,*Z*)-1-acetoxy-4-hydroxybut-2-en-1-yl)-2,2-dimethyloxazolidine-3-carboxylate (**12**). A mixture of compound **11** (1.40 g, 3.16 mmol) and TBAF (140 mg) in 25 mL of dry THF is stirred together at 0 °C for a time period of 8 h under N_2 atmosphere. After completion of the reaction, it was quenched with water and concentrated to remove THF. The remaining aqueous phase was extracted with EtOAc (3 × 50 mL) to give crude compound **12**. Purification by silica gel column chromatography using petroleum ether-EtOAc (85:15) as eluent gave **12** (0.88 g, 85%) as colorless oil. $[\alpha]_D^{20}$: – 35.36 (*c*, 1.02, $CHCl_3$). IR (neat): ν_{max} 3401, 2989, 1742, 1716, 1475, 1375 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ 1.46 (s, 15H), 1.48 (brs, 1H), 2.02 (s, 3H), 3.90-4.38 (m, 4H), 4.66-4.82 (m, 2H), 5.62-5.66 (m, 1H), 5.76-5.87 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 20.9, 24.4, 26.4, 28.3, 60.4, 61.9, 64.5, 69.0, 81.2, 94.4, 126.7, 132.5, 154.2, 170.9. Mass: 368 (M^+ +K), 352(M^+ +Na), 330(M^+ +1), 274, 263, 246. Analysis calcd for $C_{16}H_{27}NO_6$ (329.39): C, 58.34; H, 8.26; N, 4.25; found C, 58.23; H, 8.03; N, 4.05 %.

(*S*)-*tert*-Butyl 4-[(*R*,*Z*)-1-acetoxy-4-[(methylsulfonyl)oxy]but-2-en-1-yl]-2,2-dimethyloxazolidine-3-carboxylate (**13**). To a stirred ice cold solution of **12** (0.6 g, 1.82 mmol) and pyridine (1.27, 9.1 mmol) in anhydrous CH_2Cl_2, methanesulfonyl chloride (0.472 ml, 5.47 mmol) was added. The stirring was continued for 4 h. After completion of the reaction it was quenched with water and extracted with CH_2Cl_2 (3 × 25 mL), washed with $CuSO_4$ and brine solution, and dried over anhydrous Na_2SO_4. After concentration, the crude product was passed through silica gel column chromatography to give **13** (0.64g, 86%) as pale yellow oil. **$[\alpha]_D^{20}$**: + 18.21 **(***c*, 1.04; $CHCl_3$). IR (neat): ν_{max} 3012, 2973, 1737, 1713, 1608, 1556, 1375, 1215 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ 1.49 (s, 12H), 1.51 (s, 3H), 2.07 (s, 3H), 2.99 (s, 3H), 3.74 (d, 2H, *J* = 6 Hz), 4.00 (m, 1H), 4.13 (d, 2H, *J* = 6Hz), 4.22 (m, 1H), 5.51 (m, 1H), 5.81 (m, 1H). Mass: 408 (M^+ +1), 330 (M^+ +1- SO_2CH_3), 316 (M^+- OSO_2CH_3), 310 (M^+ -COO^tBu), 301, 296, 272, 250, 217. Analysis: calcd for $C_{17}H_{29}NO_8S$ (407.543): C, 50.10; H, 7.19; N, 3.44; found C, 50.34; H, 6. 95; N, 3.19 %.

(5*R*,6*S*)-*tert*-Butyl 5-acetoxy-6-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate (**1**). A solution of **13** (0.4 g, 0.98 mmol) in dry CH_2Cl_2 and *p*-TSA (180 mg, 1.048 mmol) were stirred together at room temperature for 10 h. The progress of the reaction was monitored by TLC. After complete deprotection, the reaction mixture was quenched with excess of *N*,*N*-diisopropylethylamine and stirred for another 2 h at room temperature, Boc anhydride (320 mg, 1.47 mmol) was added and stirring was continued further 6 h at room temperature. The reaction mixture was then concentrated under reduced pressure and residue was purified by column

chromatography using petroleum ether-EtOAc (75:25) as eluent to give **1 (**0.113 mg, 68%) as a pale yellow oil. $[\alpha]_D^{20}$: + 17.44 (c, 1.05; $CHCl_3$). IR (Neat): νmax 3465 (br), 3014, 2976, 2815, 1738, 1719, 1597, 1452, 1372, 1125 cm^{-1}. ^1H NMR (200MHz, $CDCl_3$): δ 1.48 (s, 9H), 2.06 (s, 3H), 3.94 (m, 2H), 4.10-4.30 (m, 4H), 5.47 (m, 1H,), 5.79 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$): δ 21.0, 28.2, 38.9, 59.3, 63.7, 68.7, 80.5, 128.7, 130.2, 152.5, 169.7. Mass: 294 (M^+ +Na), 272 (M^+ +1), 234 (294 – CH_3COO), 229 (M^+ +1– $COCH_3$), 228 (271- $COCH_3$), 212 (M^+ -$OCOCH_3$), 203, 202, 197, 196, 195, 190, 173, 172. Analysis: calcd for $C_{13}H_{21}NO_5$ (271.31): 57.55; H, 7.80; N, 5.16; found C, 57.43; H, 7.67; N, 4.95 %.

Acknowledgements

We are grateful to CSIR, New Delhi for financial support (Grant No. SR/S1/OC-44/2009). ADB, KS thank CSIR New Delhi for the award of Senior Research Fellowship.

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