SECOND-GENERATION SYNTHESIS OF A CHIRAL BUILDING BLOCK FOR OXYGENATED TERPENOIDS VIA A RING-CONTRACTIVE COUPLING WITH A SECONDARY ALCOHOL

Sayuri Saito, Hiroyuki Yamakoshi, and Seiichi Nakamura*

Graduate School of Pharmaceutical Sciences, Nagoya City University, 3-1 Tanabe-dori, Mizuho-ku, Nagoya 467-8603, Japan

nakamura@phar.nagoya-cu.ac.jp

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1. General Information

Optical rotations were recorded on a digital polarimeter with a sodium lamp (589 nm). Infrared (IR) spectra were recorded on an FT-IR spectrophotometer and absorbance bands are reported in wavenumber (cm$^{-1}$). Proton nuclear magnetic resonance ($^1$H NMR) spectra were recorded with CHCl$_3$ ($\delta_H$ 7.26) as an internal standard. Coupling constants ($J$) are reported in hertz (Hz). Abbreviations of multiplicity are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Data are presented as follows: chemical shift, multiplicity, coupling constants, integration and assignment. Carbon nuclear magnetic resonance ($^{13}$C NMR) spectra were recorded with CDCl$_3$ ($\delta_C$ 77.16) as an internal standard. High-resolution mass spectra (HRMS) were recorded by electrospray ionization (ESI) using a time-of-flight (TOF) analyzer.

Column chromatography was carried out on silica gel 60 N (63–210 $\mu$m or 40–50 $\mu$m). Analytical thin layer chromatography (TLC) was carried out with 0.25 mm silica gel plates. Visualization was accomplished with ultraviolet light and anisaldehyde stain, followed by heating.

Reagents and solvents were purified by standard means or used as received unless otherwise noted. Dehydrated dichloromethane (CH$_2$Cl$_2$) and tetrahydrofuran (THF, stabilizer free) were purchased. 1,1,1,3,3,3-Hexamethyldisilazane (HMDS), chlorotrimethylsilane (TMSCl) and diisopropylamine were distilled from calcium hydride. All reactions were conducted under an argon atmosphere unless otherwise noted.
2. Copies of $^1$H and $^{13}$C NMR Spectra
Supporting Information