Supporting Information

Directed Studies Towards The Total Synthesis of (+)-13-Deoxytedanolide: Simple and Convenient Synthesis of C8-C16 fragment

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**Usual procedures**

All reagents were obtained from commercial sources and used as supplied unless otherwise stated. Anhydrous THF, Et$_2$O, Toluene and CH$_2$Cl$_2$ were obtained from a MBraun® SPS-800 solvent purification system. Light petroleum refers to the fraction of petrol ether that was distilled between 40 °C and 65 °C.

The reactions were magnetically stirred and monitored by TLC, which were performed on Merck® 60F254 plates and achieved under a 254 nm UV light, visualized with an aqueous solution of potassium permanganate or an ethanolic solution of molybdophosphoric acid, followed by treatment with a heat gun.

Flash chromatography was performed with Merck® Kieselgel 60 (230-400) mesh silica gel.

**Physical data and spectroscopic measurements**

NMR data were recorded on a Bruker Avance 300 and 400 spectrometer in C$_6$D$_6$ or CDCl$_3$ and chemical shifts (δ) were given in ppm relative to the residual non-deuterated solvent signal for $^1$H NMR (C$_6$D$_6$: 7.16 ppm), (CDCl$_3$: 7.26 ppm) and relative to the deuterated solvent signal for $^{13}$C NMR (C$_6$D$_6$: 128.06 ppm), (CDCl$_3$: 77.16 ppm); coupling constants (J) are in Hertz, and the classical abbreviations are used to describe the signal multiplicity (s = singlet, d = doublet, t = triplet, sept = septet, m = multiplet, dd = doublet of doublets, dt = doublets of triplets, br = broad, etc.). NMR Spectra were assigned using information ascertained from DEPT, HMQC and NOE experiments.

High resolution mass spectra (HRMS) have been performed using a mass spectrometer equipped with pneumatically assisted atmospheric pressure ionization. The sample was ionized in positive mode electrospray in the following conditions: electrospray voltage (ISV): 5500 V; orifice voltage (OR): 70 V; nebulising gas flow pressure (air): 0.6 psi. The mass spectrum was obtained using a time of flight analyzer (TOF). The measure was realized in triplicate. The sample was dissolved in methanol (500 µL) then diluted (dilution factor 4/10000) in a methanolic solution of ammonium acetate (3 mM). The sample solution was infused in the ionization source at a 5 µL/min flow rate.
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\[ \text{\textbf{S 3}} \]

\[ \text{\textbf{O}} \]

\[ \text{\textbf{N}} \]

\[ \text{\textbf{O}} \]

\[ \text{\textbf{Bn}} \]

\[ (\text{\textbf{S}})-14 \]

\[ \text{\textbf{O}} \]

\[ \text{\textbf{N}} \]

\[ \text{\textbf{O}} \]

\[ \text{\textbf{Bn}} \]

\[ (\text{\textbf{R}})-14 \]

\[ \text{\textbf{H NMR}} \] (300 MHz, CDCl\textsubscript{3}) \( \delta \)

1.18 (3H, d, \( J = 6.8 \) Hz, CH\textsubscript{3}), 2.19-2.28 (1H, m, CH\textsubscript{2}), 2.48-2.57 (1H, m, CH\textsubscript{2}), 2.69 (1H, dd, \( J = 13.4 \) and 9.8 Hz, CH2), 3.27 (1H, dd, \( J = 13.4 \) and 3.2 Hz, CH\textsubscript{2}), 3.36 (1H, m, \( J = 6.8 \) Hz, CH), 4.11-4.21 (2H, m, CH\textsubscript{2}), 4.64-4.71 (1H, m, CH\textsubscript{3}), 5.04-5.13 (2H, m, CH\textsubscript{2}), 5.76-5.89 (m, 1H, CH), 7.20-7.35 (5H, m, CH\textsubscript{Ar}); \[ \text{\textbf{13C NMR}} \] (CDCl\textsubscript{3}, 75 MHz) \( \delta \)

16.5 (CH\textsubscript{3}), 37.2 (CH), 38.0 (CH\textsubscript{2}), 38.1 (CH\textsubscript{2}), 55.4 (CH), 66.1 (CH\textsubscript{2}), 117.3 (CH\textsubscript{2}), 127.4 (CH\textsubscript{Ar}), 129.0 (2 x CH\textsubscript{Ar}), 129.5 (2 x CH\textsubscript{Ar}), 135.4 (CH), 135.5 (C\textsubscript{Ar}), 153.2 (C), 176.6 (C); \[ (\text{\textbf{S}})-14 \] [\( \alpha \)\textsubscript{\textbf{D}}] = +38.0 (c 1, CHCl\textsubscript{3}); \[ (\text{\textbf{R}})-14 \] [\( \alpha \)\textsubscript{\textbf{D}}] = -39.0 (c 1, CHCl\textsubscript{3})
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\[ \text{OH} \quad \text{(S)-15} \quad \text{OH} \quad \text{(R)-15} \]

\(^1\text{H NMR}\) (200 MHz, CDCl\(_3\)) \(\delta\) 0.92 (3H, d, \(J = 6.7\) Hz, CH\(_3\)), 1.37 (1H, br s, OH), 1.65-1.82 (1H, m, CH), 1.87-2.01 (1H, m, CH\(_2\)), 2.11-2.25 (1H, m, CH\(_2\)), 3.40-3.55 (2H, m, CH\(_2\)), 4.99-5.09 (2H, m, CH\(_2\)), 5.71-5.92 (m, 1H, CH), \(^{13}\text{C NMR}\) (50 MHz, CDCl\(_3\)) \(\delta\) 16.4 (CH\(_3\)), 35.6 (CH), 37.9 (CH\(_2\)), 67.7 (CH\(_2\)), 116.1 (CH\(_2\)), 137.1 (CH); (R)-15 \([\alpha]^{19}_D = +4.3\) (c 1, CHCl\(_3\)); (S)-15 \([\alpha]^{24}_D = -2.6\) (c 1, CHCl\(_3\))
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(H)-12  (R)-12

\[ \begin{align*}
^{1}H \text{ NMR} \ (400 \text{ MHz, CDCl}_{3}) & \ \delta \ 1.07 \ (3H, d, J = 6.8 \text{ Hz, CH}_{3}), \ 2.98-3.07 \ (1H, m, CH), \ 3.85 \\
& \ (1H, dd, J = 8.8 \text{ and } 6.8 \text{ Hz, CH}_{2}), \ 4.37 \ (1H, dd, J = 9.8 \text{ and } 8.8 \text{ Hz, CH}_{2}), \ 4.93 \ (1H, t, J = 2.5 \\
& \text{ Hz, CH}), \ 6.29 \ (1H, t, J = 2.5 \text{ Hz, CH}); \ ^{13}C \text{ NMR} \ (50 \text{ MHz, CDCl}_{3}) \ \delta \ 20.6 \ (\text{CH}_{3}), \ 36.5 \ (\text{CH}), \\
& \ 76.7 \ (\text{CH}_{2}), \ 106.3 \ (\text{CH}), \ 145.2 \ (\text{CH}).
\end{align*} \]
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\[ \alpha^2_d + 21.4, \text{ (c 1.0, CHCl}_3) \]; \textbf{1H NMR} (400 MHz, CDCl\textsubscript{3}) \( \delta \) 0.84-0.90 (15H, m, 3 x CH\textsubscript{3} and 3 x CH\textsubscript{2}), 1.02 (3H, d, \( J = 6.8 \text{ Hz, CH}_3 \)), 1.24-1.53 (12H, m, 6 x CH\textsubscript{2}), 2.34-2.44 (1H, m, CH\textsubscript{2}), 3.38-3.44 (1H, m, CH\textsubscript{2}), 3.47-3.53 (1H, m, CH\textsubscript{2}), 5.79 (1H, dd, \( J = 19.0 \text{ and 7.0 Hz, }^3J_{\text{Sn-H}} = 70 \text{ Hz, CH} \)), 5.99 (1H, br d, \( J = 19.0 \text{ Hz, }^2J_{\text{Sn-H}} = 18 \text{ Hz, CH} \)); \textbf{\textsuperscript{13}C NMR} (75 MHz, CDCl\textsubscript{3}) \( \delta \) 9.5 (3 x CH\textsubscript{2}, \( ^1J_{\text{Sn-C}} = 334 \text{ Hz} \)), 13.4 (3 x CH\textsubscript{3}), 16.1 (CH\textsubscript{3}), 27.3 (3 x CH\textsubscript{2}, \( ^3J_{\text{Sn-C}} = 54 \text{ Hz} \)), 29.2 (3 x CH\textsubscript{2}, \( ^2J_{\text{Sn-C}} = 21 \text{ Hz} \)), 44.5 (CH, \( ^3J_{\text{Sn-C}} = 57 \text{ Hz} \)), 66.9 (CH\textsubscript{2}), 129.5 (C\textsubscript{3}, CH, \( ^2J_{\text{Sn-C}} = 23 \text{ Hz} \)), 151.2 (CH); \textbf{IR} (thin film) \( \nu_{\text{max}} = 3325, 2956, 2923, 2871, 2852, 1597, 1455, 1376, 1072, 1031, 990 \text{ cm}^{-1} \); \textbf{LRMS} \( m/z \) (ESI) 399.(M+Na)\textsuperscript{+}; \textbf{HRMS} \( m/z \) (ESI) calcd for C\textsubscript{17}H\textsubscript{37}OSn [M+H]\textsuperscript{+}: 377.1860, found 377.1861.
[α]_{D}^{21} = -31.8 (c 1.0, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 0.84-0.98 (18H, 4 x CH₃ and 3 x CH₂), 1.16-1.56 (12H, m, 6 x CH₂), 1.88 (3H, d, J = 1.9 Hz, ³J_{Sn-H} = 44Hz, CH₃), 2.77-2.99 (1H, m, CH), 3.30-3.36 (1H, m, CH₂), 3.43-3.50 (1H, m, CH₂), 5.23 (1H, dq, J = 9.0 and 1.9 Hz, ³J_{Sn-H} = 70 Hz, CH); ¹³C NMR (75 MHz, CDCl₃) δ 9.3 (3 x CH₂, ¹J_{Sn-C} = 322 Hz), 13.8 (3 x CH₃), 16.9 (CH₃), 19.7 (CH₃), 27.5 (C9, 3 x CH₂, ³J_{Sn-C} = 54 Hz), 29.3 (3 x CH₂, ²J_{Sn-C} = 20 Hz), 35.3 (CH₃, ³J_{Sn-C} = 53 Hz), 67.7 (CH₂), 141.2 (C), 143.1 (CH, ²J_{Sn-C} = 24 Hz); IR (thin film) ν max = 3330, 2955, 2924, 2871, 2850, 1456, 1377, 1071, 1030, 970 cm⁻¹; HRMS (ESI) m/z calcd for C₁₈H₃₉OSn [M+H]⁺: 391.2017, found 391.2017.
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$[\alpha]^{25}_D$ +23.1, (c 1.0, CHCl$_3$); $^1$H NMR (300 MHz, CDCl$_3$) $\delta$ 1.00 (3H, d, $J = 6.8$ Hz, CH$_3$), 1.88-1.92 (1H, m, OH), 2.33-2.46 (1H, m, CH), 3.40-3.53 (2H, m, CH$_2$), 6.12 (1H, d, $J = 14.5$ Hz, CH), 6.45 (1H, dd, $J = 14.5$ and 7.9 Hz, CH); $^{13}$C NMR (75 MHz, CDCl$_3$) $\delta$ 15.6 (CH$_3$), 43.4 (CH), 66.5 (CH$_2$), 76.2 (CH), 148.6 (CH).
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\[
\begin{align*}
\alpha & = 25^\circ + 20.8, (c \ 1.0, \ CHCl_3); \\
^1H \text{ NMR} & (400 MHz, CDCl}_3) \ \delta 0.05 (6H, s, 2 \times CH_3), 0.90 (9H, s, 3 \times CH_3), 1.01 (3H, d, J = 6.8 Hz, CH_3), 2.33-2.40 (1H, m, CH), 3.45 (1H, dd, J = 9.8 and 6.4 Hz, CH_2), 3.48 (1H, dd, J = 9.8 and 6.4 Hz, CH_2), 6.06 (1H, br dd, J = 14.6 Hz, CH), 6.49 (1H, dd, J = 14.6 and 6.5 Hz, CH); \\
^{13}C \text{ NMR} & (100 MHz, CDCl}_3) \ \delta -5.2 (2 \times CH_3), 15.7 (CH_3), 18.4 (C), 26.0 (3 \times CH_3), 43.3 (CH), 67.0 (CH_2), 75.2 (CH), 149.2 (CH); \\
\text{IR} & \text{ (thin film)} \ \nu_{\text{max}} = 2955, 2928, 2856, 1605, 1471, 1386, 1361, 1252, 1187, 1088, 1024, 1006, 947 \text{ cm}^{-1}; \\
\text{LRMS} \ m/z & \text{ (ESI) 349 (M+Na)}^+; \ \text{HRMS} \ m/z \text{ (ESI) calcd for C}_{11}H_{24}OSi [M+H]^+: 327.0636, found 327.0640
\end{align*}
\]
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Chloroform

77.16
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\[ \alpha \]_D^{25} = -31.3, (c 1.0, CHCl₃); \[^1^H\text{NMR}\] (400 MHz, CDCl₃) δ 0.95 (3H, d, J = 6.8 Hz, CH₃), 1.82-1.85 (1H, m, OH), 2.41 (3H, d, J = 1.5 Hz, CH₃), 2.57-2.68 (1H, m, CH), 3.36-3.51 (2H, m, CH₂), 5.96 (1H, br dq, J = 9.8, 1.5 Hz, CH); \[^{13}\text{C NMR}\] (100 MHz, CDCl₃) δ 16.4 (CH₃), 28.2 (CH₃), 38.6 (CH), 67.0 (CH₂), 95.6 (CH), 143.6 (CH); \[^{1}\text{RMS}\] (thin film) ν max = 3332, 2958, 2926, 2870, 1635, 1429, 1377, 1217, 1119, 1076, 1030, 996, cm⁻¹; \[^{1}\text{RMS}\] m/z (ESI) 249 (M+Na); \[^{1}\text{HRMS}\] m/z (ESI) calcd for C₆H₁₅NOI [M+NH₄]+: 244.0193, found 244.0185.
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1H NMR (400 MHz, CDCl₃) δ 1.21 (3H, d, J = 7.0 Hz, CH₃), 2.46 (3H, d, J = 1.5 Hz, CH₃), 3.23-3.31 (1H, m, CH), 6.06 (1H, br dq, J = 9.3, 1.5 Hz, CHF), 9.51 (1H, d, J = 1.7 Hz, CHF); 13C NMR (100 MHz, CDCl₃) δ 13.8 (CH₃), 28.4 (CH₃), 48.8 (CH), 67.0 (CH₂), 98.1 (CH), 136.6 (CH), 199.3 (CHO).
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Major diastereoisomer only: $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 0.05 (6H, s, 2 x CH$_3$), 0.89 (1H, s, 3 x CH$_3$), 1.00 (6H, d, $J = 6.8$ Hz, 2 x CH$_3$), 1.66 (1H, br s, OH), 2.29-2.36 (1H, m, CH), 2.39 (3H, d, $J = 1.5$ Hz, CH$_3$), 2.50-2.59 (1H, m, CH), 3.40 (1H, dd, $J = 9.8$ and 6.8 Hz, CH$_2$), 3.49 (1H, dd, $J = 9.8$ and 6.3 Hz, CH$_2$), 3.88 (1H, app br t, $J = 6.5$ Hz, CH), 5.45 (1H, br dd, $J = 15.6$ and 6.8 Hz, CH), 5.60 (1H, br dd, $J = 15.6$ and 6.3 Hz, CH), 5.99 (1H, br dq, $J = 9.8$ and 1.5 Hz, CH); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ -5.3 (2 x CH$_3$), 15.9 (CH$_3$), 16.5 (CH$_3$), 18.3 (C), 25.9 (3 x CH$_3$), 28.1 (CH$_3$), 39.0 (CH), 41.7 (CH), 67.9 (CH$_2$), 76.3 (CH), 94.6 (Cl), 129.8 (CH), 135.8 (CH), 142.9 (CH); IR (thin film) $\nu_{\text{max}}$ = 3419, 2958, 2930, 2858, 1638, 1473, 1388, 1257, 1089, 1009, 974 cm$^{-1}$; LRMS $m/z$ (ESI) 447 (M+Na)$^+$; HRMS $m/z$ (ESI) calcd for C$_{17}$H$_{37}$NO$_2$Si [M+NH$_4$]$^+$: 442.1633, found 442.1633.
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\[ \alpha^{21}_D + 52.5 \ (c \ 1.0, \ CHCl_3); \]

\(^1H\) NMR (400 MHz, C\(_6\)D\(_6\)) \( \delta \)

- 0.00 (6H, s, 2 x CH\(_3\)),
- 0.82 (3H, d, \( J = 6.8 \) Hz, CH\(_3\)),
- 0.94 (9H, s, 3 x CH\(_3\)),
- 0.99 (3H, d, \( J = 7.0 \) Hz, CH\(_3\)),
- 2.12 (3H, br d, \( J = 1.5 \) Hz, CH\(_3\)),
- 2.22 (1H, app sept, \( J = 6.3 \) Hz, CH),
- 3.19 (1H, dq, \( J = 9.8 \) and 7.0 Hz, CH),
- 3.28 (2H, d, \( J = 6.0 \) Hz, CH\(_2\)),
- 6.06 (1H, br dd, \( J = 15.8 \) and 1.3 Hz, CH),
- 6.25 (1H, br dq, \( J = 9.8 \) and 1.5 Hz, CH),
- 6.90 (1H, dd, \( J = 15.8 \) and 7.3 Hz, CH);

\(^{13}C\) NMR (100 MHz, C\(_6\)D\(_6\)) \( \delta \)

- -5.3 (2 x CH\(_3\)),
- 15.7 (CH\(_3\)),
- 16.3 (CH\(_3\)),
- 18.5 (C),
- 26.1 (3 x CH\(_3\)),
- 27.9 (CH\(_3\)),
- 39.6 (CH),
- 47.2 (CH),
- 67.1 (CH\(_2\)),
- 96.1 (C),
- 127.5 (CH),
- 140.3 (CH),
- 149.8 (CH),
- 196.9 (C);

IR (thin film) \( \nu_{\text{max}} = 2955, 2927, 2854, 1697, 1673, 1626, 1471, 1459, 1253, 1189, 1129, 1097, 1084, 1029, 980 \text{ cm}^{-1} \);

LRMS m/z (ESI) 445 (M+Na)\(^+\);

HRMS m/z (ESI) calcd for C\(_{17}\)H\(_{32}\)O\(_2\)SiI \[M+H\]\(^+\):

423.1211, found 423.1211.
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\[ \text{[}\alpha\text{]}^{22}_{\text{D}} + 65.6, \ (c \ 1.0, \ \text{CHCl}_3); \]  
\[^1\text{H} \text{NMR} \ (400 \text{ MHz, CDCl}_3) \ \delta \ 0.05 \ (6\text{H}, \text{s, } 2 \times \text{CH}_3), \ 0.87 \ (3\text{H}, \text{d, } J = 6.6 \text{ Hz, CH}_3), \ 0.90 \ (9\text{H}, \text{s, } 3 \times \text{CH}_3), \ 1.16 \ (3\text{H}, \text{d, } J = 6.8 \text{ Hz, CH}_3), \ 1.30-1.43 \ (1\text{H}, \text{m, CH}_2), \ 1.51-1.62 \ (1\text{H}, \text{m, CH}), \ 1.62-1.73 \ (1\text{H}, \text{m, CH}_2), \ 2.36-2.55 \ (2\text{H}, \text{m, CH}_2), \ 2.46 \ (3\text{H}, \text{d, } J = 1.5 \text{ Hz, CH}_3), \ 3.33-3.40 \ (1\text{H}, \text{m, CH}), \ 3.42 \ (2\text{H}, \text{d, } J = 5.9 \text{ Hz, CH}_2), \ 6.12 \ (1\text{H}, \text{dq, } J = 10.0 \text{ and } 1.5 \text{ Hz, CH}_3); \]  
\[^{13}\text{C} \text{NMR} \ (100 \text{ MHz, CDCl}_3) \ \delta \ -5.2 \ (2 \times \text{CH}_3), \ 16.3 \ (\text{CH}_3), \ 16.8 \ (\text{CH}_3), \ 18.5 \ (\text{C}), \ 26.1 \ (3 \times \text{CH}_3), \ 27.4 \ (\text{CH}_2), \ 28.1 \ (\text{CH}_3), \ 35.4 \ (\text{CH}), \ 38.8 \ (\text{CH}_2), \ 48.7 \ (\text{CH}), \ 68.2 \ (\text{CH}_2), \ 96.1 \ (\text{C}), \ 139.9 \ (\text{CH}), \ 209.9 \ (\text{C}); \]  
\(\text{IR (thin film)} \ \nu_{\text{max}} = 2955, \ 2929, \ 2883, \ 2856, \ 1716, \ 1472, \ 1462, \ 1434, \ 1252, \ 1117, \ 1091, \ 1037, \ 1028, \ 1005 \ \text{cm}^{-1}; \]  
\(\text{LRMS} \ m/z \ (\text{ESI}) \ 447 \ (\text{M+Na})^+; \)  
\(\text{HRMS} \ m/z \ (\text{ESI}) \ \text{calcd for C}_{17}\text{H}_{34}\text{O}_2\text{SiI} \ [\text{M+H}]^+: \) 425.1367, found 425.1367.
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\text{[} \alpha \text{]}^{20}_{20} = -23.0, (c 1.0, \text{CHCl}_3); \quad \text{H NMR} \ (300 \text{ MHz}, \text{C}_6\text{D}_6) \quad \delta 0.07 (6\text{H}, \text{s}, 2 \times \text{CH}_3), 0.79 (3\text{H}, \text{d}, \text{J} = 6.9 \text{ Hz}, \text{CH}_3), 0.87 (3\text{H}, \text{d}, \text{J} = 6.7 \text{ Hz}, \text{CH}_3), 0.98 (9\text{H}, \text{s}, 3 \times \text{CH}_3), 1.19-1.45 (5\text{H}, \text{m}, 2 \times \text{CH}_2 \text{ and } \text{OH}), 1.51-1.61 (1\text{H}, \text{m}, \text{CH}), 2.12-2.24 (1\text{H}, \text{m}, \text{CH}), 2.17 (3\text{H}, \text{d}, \text{J} = 1.3 \text{ Hz}, \text{CH}_3), 3.05-3.11 (1\text{H}, \text{m}, \text{CH}), 3.33-3.42 (1\text{H}, \text{m}, \text{CH}_2), 6.16 (1\text{H}, \text{br dq}, \text{J} = 10.0 \text{ and } 1.3 \text{ Hz}, \text{CH}); \quad \text{C NMR} \ (75 \text{ MHz}, \text{C}_6\text{D}_6) \quad \delta -5.1 (2 \times \text{CH}_3), 16.8 (\text{CH}_3), 17.1 (\text{CH}_3), 18.6 (\text{C}), 26.3 (3 \times \text{CH}_3), 28.1 (\text{CH}_3), 29.7 (\text{CH}_2), 32.4 (\text{CH}_2), 36.0 (\text{CH}), 41.9 (\text{CH}), 68.6 (\text{CH}_2), 74.9 (\text{CH}), 94.6 (\text{C}), 143.5 (\text{CH}); \quad \text{IR (thin film)} \quad \nu_{\text{max}} = 3397, 2954, 2928, 2856, 1462, 1377, 1361, 1251, 1090 \text{ cm}^{-1}; \quad \text{LRMS} \quad m/z (\text{ESI}) 449 (\text{M+Na})^+; \quad \text{HRMS} \quad m/z (\text{ESI}) \text{ calcd for } \text{C}_{17}\text{H}_{36}\text{O}_2\text{SiI} [\text{M+H}]^+: 427.1524, \text{found } 427.1523.
\]
[α]$_D^{36}$ -31.8, (c 1.0, CHCl$_3$); $^1$H NMR (300 MHz, C$_6$D$_6$) δ 0.07 (6H, s, 2 x CH$_3$), 0.84 (3H, d, J = 6.8 Hz, CH$_3$), 0.89 (3H, d, J = 6.4 Hz, CH$_3$), 0.98 (9H, s, 3 x CH$_3$), 0.99-1.06 (1H, m, CH$_2$), 1.11-1.21 (1H, m, CH$_2$), 1.25 (1H, br s, OH), 1.35-1.46 (1H, m, CH$_2$), 1.48-1.60 (2H, m, CH and CH$_2$), 2.17 (3H, d, J = 1.5 Hz, CH$_3$), 2.19-2.27 (1H, m, CH), 3.03-3.11 (1H, m, CH), 3.34 (1H, dd, J = 9.8 and 5.6 Hz, CH$_2$), 3.41 (1H, dd, J = 9.8 and 5.6 Hz, CH$_2$), 6.08 (1H, dq, J = 10.0 and 1.5 Hz, CH); $^{13}$C NMR (75 MHz, C$_6$D$_6$) δ -5.1 (2 x CH$_3$), 15.5 (CH$_3$), 17.3 (CH$_3$), 18.6 (C), 26.3 (3 x CH$_3$), 28.0 (CH$_3$), 29.8 (CH$_2$), 32.4 (CH$_2$), 36.1 (CH$_3$), 42.0 (CH), 68.3 (CH$_2$), 75.2 (CH), 94.1 (C), 144.5 (CH); IR (thin film) $\nu_{\text{max}}$ = 3358, 2954, 2928, 2856, 1633, 1462, 1378, 1361, 1252, 1092 cm$^{-1}$; LRMS m/z (ESI) 449 (M+Na)$^+$; HRMS m/z (ESI) calcd for C$_{17}$H$_{36}$O$_2$SiI [M+H]$^+$: 427.1524, found 427.1521.
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![Chemical Structure](image)

\([\alpha]^{25}_D\) -33.3, (c 1.0, CHCl₃); \(^1^H\) NMR (300 MHz, C₆D₆) δ 0.07 (6H, s, 2 x CH₃), 0.08 (6H, s, 2 x CH₃), 0.85 (3H, d, J = 6.8 Hz, CH₃), 0.90 (3H, d, J = 6.5 Hz, CH₃), 0.98 (9H, s, 3 x CH₃), 1.00 (9H, s, 3 x CH₃), 1.10-1.20 (1H, m, CH₂), 1.33-1.61 (4H, m, CH₂ and CH₂ and CH), 2.24 (3H, d, J = 1.5 Hz, CH₃), 2.37-2.49 (1H, m, CH), 3.35-3.45 (3H, m, CH and CH₂), 6.24 (1H, dq, J = 10.0 and 1.4 Hz, CH); \(^1^C\) NMR (100 MHz, C₆D₆) δ -5.2 (2 x CH₃), -4.1 (CH₃), -4.0 (CH₃), 16.4 (CH₃), 16.9 (CH₃), 18.3 (C), 18.6 (CH₃), 26.2 (3 x CH₃), 26.2 (3 x CH₃), 28.0 (CH₃), 28.7 (CH₂), 32.1 (CH₂), 36.4 (CH), 40.8 (CH), 68.5 (CH₂), 75.9 (CH), 94.1 (C), 144.3 (CH); LRMS m/z (ESI) 563 (M+Na)⁺; HRMS m/z (ESI) calcd for C\(_{23}\)H\(_{49}\)NO\(_2\)Si\(_2\)I [M+NH₄]⁺: 558.2654, found 558.2651.
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[α]$_D^{25}$ -32.8, (c 1.0, CHCl$_3$); $^1$H NMR (400 MHz, C$_6$D$_6$) δ 0.05 (3H, s, CH$_3$), 0.06 (3H, s, CH$_3$), 0.83 (2 x 3H, 2 x d overlapped, J = 6.7 Hz, 2 x CH$_3$), 0.97 (9H, s, 3 x CH$_3$), 1.01-1.06 (1H, m, CH$_2$), 1.29-1.49 (4H, m, CH$_2$ and CH$_2$ and CH), 2.22 (3H, d, J = 1.5 Hz, CH$_3$), 2.35-2.44 (1H, m, CH), 3.17 (1H, dd, J = 10.0 and 6.0 Hz, CH$_2$), 3.23 (1H, dd, J = 10.3 and 5.5 Hz, CH$_2$), 3.33 (1H, q, J = 5.0 Hz, CH), 6.23 (1H, br dq, J = 10.0 and 1.5 Hz, CH); $^{13}$C NMR (75 MHz, C$_6$D$_6$) δ -4.2 (CH$_3$), -4.1 (CH$_3$), 16.5 (CH$_3$), 16.8 (CH$_3$), 18.3 (C), 26.2 (3 x CH$_3$), 28.0 (CH$_3$), 28.6 (CH$_2$), 32.2 (CH$_2$), 36.2 (CH), 40.7 (CH), 67.9 (CH$_2$), 75.8 (CH), 94.1 (C), 144.2 (CH); LRMS m/z (ESI) 449 (M+Na)$^+$; HRMS m/z (ESI) calcd for C$_{17}$H$_{36}$O$_2$SiI [M+H]$^+$: 427.1524, found 427.1517.
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\[ \alpha \] D -46.0, (c 1.0, CHCl₃); \(^1\)H NMR (300 MHz, C₆D₆) \( \delta \) 0.00 (3H, s, CH₃), 0.03 (3H, s, CH₃), 0.77 (3H, d, \( J = 6.9 \) Hz, CH₃), 0.78 (3H, d, \( J = 7.1 \) Hz, CH₃), 0.94 (9H, s, 3 x CH₃), 1.07-1.16 (1H, m, CH₂), 1.18-1.32 (2H, m, CH₂), 1.41-1.56 (1H, m, CH₂), 1.73-1.84 (1H, m, CH), 2.20 (3H, d, \( J = 1.5 \) Hz, CH₃), 2.24-2.41 (1H, m, CH), 3.26 (1H, q, \( J = 5.5 \) Hz, CH), 6.14 (1H, br dq, \( J = 10.0 \) and 1.5 Hz, CH), 9.30 (1H, d, \( J = 1.3 \) Hz, CH); \(^{13}\)C NMR (75 MHz, C₆D₆) \( \delta \) -4.2 (CH₃), -4.1(CH₃), 13.3 (CH₃), 16.2 (CH₃), 18.3 (CH₃), 25.8 (CH₂), 26.1 (3 x CH₃), 27.9 (CH₃), 31.6 (CH₂), 40.8 (CH), 46.2 (CH), 75.2 (CH), 94.3 (C), 144.0 (CH), 202.9 (CH); IR (thin film) \( \nu \) max = 2256, 2931, 2858, 1709, 1472, 1464, 1379, 1361, 1254, 1067, 1045, 1027, 1006 cm⁻¹; LRMS m/z (ESI) 447 (M+Na)⁺
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The alcohol is not described because the mixture of diastereomers.

**IR** (thin film) \( \nu_{\text{max}} = 3364, 2957, 2928, 2883, 2857, 1471, 1461, 1406, 1378, 1361, 1253, 1065, 1027, 1004, 942 \text{ cm}^{-1} \); **LRMS** \( m/z \) (ESI) 463 (M+Na); **HRMS** \( m/z \) (ESI) calcd for C\(_{18}\)H\(_{38}\)O\(_2\)SiI [M+H]\(^{+}\): 441.1680, found 441.1667.
[α]_{D}^{28} -28.6, (c 1.0, CHCl₃); \(^1\)H NMR (300 MHz, C₆D₆) δ 0.03 (3H, s, CH₃), 0.04 (3H, s, CH₃), 0.79 (3H, d, J = 6.8 Hz, CH₃), 0.87 (3H, d, J = 7.0 Hz, CH₃), 0.95 (9H, s, 3 x CH₃), 1.15-1.38 (3H, m, CH₂ and CH₂), 1.49-1.59 (1H, m, CH₂), 1.75 (3H, s, CH₃), 2.01-2.10 (1H, m, CH), 2.21 (3H, d, J = 1.5 Hz, CH₃), 2.28-2.40 (1H, m, CH), 3.26-3.31 (1H, m, CH), 6.17 (1H, br dq, J = 10.0 and 1.5 Hz, CH); \(^{13}\)C NMR (75 MHz, C₆D₆) δ -4.2 (CH₃), -4.1 (CH₃), 16.3 (CH₃), 16.5 (CH₃), 18.3 (C), 26.2 (3 x CH₃), 27.8 (CH₃), 28.0 (CH₃), 28.3 (CH₂), 32.3 (CH₂), 40.7 (CH), 47.0 (CH), 75.4 (CH), 94.3 (CH), 144.0 (CH), 209.6 (CH); IR (thin film) ν_max = 2955, 2929, 2856, 1713, 1471, 1461, 1378, 1359, 1253, 1170, 1067, 1043, 1026, 1006, 940 cm\(^{-1}\); LRMS m/z (ESI) 461 (M+Na); HRMS m/z (ESI) calcd for C\(_{18}\)H\(_{36}\)O\(_2\)SiI [M+H]\(^{+}\): 439.1524, found 439.1517.