Enantioselective Total Synthesis of Colomitides and their absolute configuration determination and Structural Revision

Hongguang Yang, Xiaoyu Liu, Xiaoyu Li, Xiang Shi, Feilong Yang, Xiaozhen Jiao, Ping Xie

State Key Laboratory of Bioactive Substances and Functions of Natural Medicines, Institute of Materia Medica, Beijing Key Laboratory of Active Substances Discovery and Druggability Evaluation, Peking Union Medical College and Chinese Academy of Medical Sciences, Beijing 100050, PR China.

E-mail: xp@imm.ac.cn

Table of contents

Experimental..........................................................2

1H and 13C NMR Spectra of Reported Compounds ........................................................................5

1H NMR analysis of the ratio of 2/1c epimers ........................................................................24

X-Ray Crystallographic Data.................................................................25

NMR Comparison Table of natural colomitide B and synthetic 2........................................27
Experimental

(R)-4-((R)-but-3-yn-2-yl)dihydrofuran-2(3H)-one (9a). 9a (490 mg) was prepared from 15 (700 mg, 5.0 mmol) following the procedure described for 9b in 70% yield. [α]D^18.5 = -56.8 (c 0.38, CHCl₃). ¹H NMR (400 MHz, CDCl₃): δ 4.46 (dd, J = 9.2, 7.1 Hz, 1H), 4.23 (dd, J = 9.2, 6.7 Hz, 1H), 2.66–2.57 (m, 3H), 2.44 (dd, J = 13.4, 11.7 Hz, 1H), 2.17 (d, J = 2.0 Hz, 1H), 1.23 (d, J = 6.6 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 176.4, 84.1, 71.2, 70.6, 40.1, 32.6, 28.5, 18.7. IR (film)νmax/cm⁻¹ 3283, 2978, 2937, 2937, 1778, 1177, 1037, 1004, 656. HRMS (ESI): Calcd for C₆H₁₂O₂ [M+H]^+ 223.1304, found 223.1304.

(3R,4S)-4-((R)-but-3-yn-2-yl)-3-((R)-1-hydroxy-2-methylbutyl)dihydrofuran-2(3H)-one (7a). To a solution of 9a (150 mg, 1.1 mmol) in CH₂Cl₂ (5 mL) was added Bu₂BOTf (1 M in CH₂Cl₂, 1.63 mL, 1.6 mmol) at -78 °C under argon. After stirred for 15 min, EtNPF₅ (0.29 mL, 1.7 mmol) was added and the solution was stirred for 1 hour at -78 °C. A solution of 8a (187 mg, 2.2 mmol) in CH₂Cl₂ was added within 1 hour by syringe pump. After the system stirred for 4 hours at -78 °C, the reaction was quenched by addition of aqueous phosphate buffer (pH 7.4, 4 mL), MeOH (2 mL), 30% H₂O₂ (1 mL). The mixture was warmed to rt and stirred for 1 hour, CH₂Cl₂ (6 mL) was added to the mixture, the organic layer was separated, and the aqueous layer was extracted with CH₂Cl₂ (5 mL x 3). The combined organic layers were washed with saturated aqueous Na₂SO₄ solution and dried over Na₂SO₄ and concentrated. The residue was purified by column chromatography on silica gel (CH₂Cl₂ / petroleum ether = 3:2) to recycle 9a (100 mg) and then eluent with CH₂Cl₂ / MeOH = 50:1 to give 7a (60 mg, 25% yield) as a colorless oil. ¹H NMR (400 MHz, CDCl₃): δ 5.79 (t, J = 8.7 Hz, 1H), 4.23 (m, 1H), 3.59 (dd, J = 6.4, 5.1 Hz, 0.4H), 3.44 (dd, J = 7.5, 4.0 Hz, 0.5H), 2.93 – 2.83 (m, 1H), 2.79 (m, 1H), 2.63 (m, 0.7H), 2.48 (m, 0.5H), 2.22 (dd, J = 5.7, 2.4 Hz, 1H), 1.94 – 1.87 (m, 0.5H), 1.72 – 1.63 (m, 1H), 1.60 – 1.50 (m, 0.5H), 1.38 – 1.30 (m, 0.5H), 1.30 – 1.24 (m, 0.6H), 1.22 (dd, J = 7.1, 2.6 Hz, 3H), 0.99 (dd, J = 6.7, 3.5 Hz, 3H), 0.94 (td, J = 7.4, 1.3 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 172.9, 136.0, 116.9, 80.6, 65.4, 37.5, 37.5, 35.6, 28.0. IR (film)νmax/cm⁻¹ 3431, 2978, 2930, 1728, 1368, 1155, 1037, 915. HRMS (ESI): Calcd for C₁₁H₁₄O₃ [M+Na]^+ 225.1485, found 225.1485.

(S)-tert-butyl 3-(hydroxymethyl)hex-5-enoate (ent-13) ent-13 (10 g) was prepared from Evans’ auxiliary (22 g 0.059 mol) following the procedure described for 13 in 85% yield. [α]D^10.6 = -6.3 (c 1.20, CHCl₃). ¹H NMR (400 MHz, CDCl₃): δ 5.84–5.73 (m, 1H), 5.11–5.02 (m, 2H), 3.64 (dd, J = 11.0, 4.7 Hz, 1H), 3.52 (dd, J = 11.0, 6.0 Hz, 1H), 2.29 (dd, J = 6.4, 1.2 Hz, 2H), 2.19–2.03 (m, 3H), 1.45 (s, 9H). ¹³C NMR (101 MHz, CDCl₃): δ 172.9, 136.0, 116.9, 80.6, 65.4, 37.5, 37.5, 35.6, 28.0. IR (film)νmax/cm⁻¹ 3431, 2978, 2930, 1728, 1368, 1155, 1037, 915. HRMS (ESI): Calcd for C₁₂H₁₄O₂Na [M+Na]^+ 223.1304, found 223.1289.

(S)-4-allyldihydrofuran-2(3H)-one (ent-14) ent-14 (5.86 g) was prepared from ent-13 (10 g 0.050 mol) following the procedure described for 14 in 93% yield. [α]D^20.6 = -26.5 (c 1.10, CHCl₃). ¹H NMR (400 MHz, CDCl₃): δ 5.79–5.67 (m, 1H), 5.16–5.07 (m, 2H), 4.40 (dd, J = 9.1, 7.4 Hz, 1H), 4.00 (dd, J = 9.1, 5.9 Hz, 1H), 2.68–2.58 (m, 2H), 2.29–2.20 (m, 3H). ¹³C NMR (101 MHz, CDCl₃): δ 176.9, 134.2, 117.8, 72.6, 37.1, 34.7, 33.8. IR (film)νmax/cm⁻¹ 2978, 2910, 1779, 1172, 1014, 920. HRMS (ESI): Calcd for C₇H₁₂O₂Na [M+Na]^+ 149.0573, found 149.0223.
(3S,4S)-4-allyl-3-methyldihydrofuran-2(3H)-one (ent-10) ent-10 (4.7 g) was prepared from ent-14 (5.85 g 0.046 mol) following the procedure described for 10 in 72% yield. $[\alpha]_D^{20.7} = +58.6$ (c 1.05, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 5.81–5.69 (m, 1H), 5.16–5.08 (m, 2H), 4.37 (dd, $J = 9.1$, 7.0 Hz, 1H), 3.82 (t, $J = 9.1$ Hz, 1H), 2.45–2.36 (m, 1H), 2.29–2.12 (m, 3H), 1.27 (d, $J = 6.8$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 179.59, 134.1, 117.7, 70.8, 42.9, 39.8, 35.9, 13.9. IR (film) $\nu_{max}$/cm$^{-1}$ 2976, 2905, 2340, 1776, 1642, 1385, 1177, 1014, 921. HRMS (ESI): Calcd for C$_8$H$_{13}$O [M+H]$^+$ 141.0910, found 141.0899.

(S)-2-((S)-3-yn-2-yl)pent-4-en-1-ol (ent-15) ent-15 (1.7 g) was prepared from ent-10 (2.5 g 17.83 mmol) following the procedure described for 15 in 70% yield. $[\alpha]_D^{20.9} = +43.6$ (c 0.99, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 5.81 (m, 1H), 5.15–5.04 (m, 2H), 3.80 (dd, $J = 11.3$, 5.3 Hz, 1H), 3.72 (dd, $J = 11.3$, 5.3 Hz, 1H), 2.68–2.60 (m, 1H), 2.33–2.18 (m, 2H), 2.11 (d, $J = 2.5$ Hz, 1H), 1.70 (s, 1H), 1.66–1.57 (m, 1H), 1.24 (d, $J = 7.1$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 136.5, 116.8, 87.3, 69.9, 63.3, 44.8, 33.9, 26.6, 18.6. IR (film) $\nu_{max}$/cm$^{-1}$ 3303, 3077, 2976, 2935, 2110, 1640, 1442, 1041, 916. 636. HRMS (ESI): Calcd for C$_8$H$_{13}$O [M+H]$^+$ 139.1117, found 139.1115.

(S)-2-((S)-4-(trimethylsilyl)but-3-yn-2-yl)pent-4-en-1-ol (ent-16) ent-16 (2.2 g) was prepared from ent-15 (1.5 g 10.85 mmol) following the procedure described for 16 in quant yield. $[\alpha]_D^{20.9} = +40.0$ (c 0.98, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 5.81 (m, 1H), 5.15–5.01 (m, 2H), 3.81 (dd, $J = 11.4$, 4.7 Hz, 1H), 3.70 (dd, $J = 11.4$, 5.3 Hz, 1H), 2.61 (dd, $J = 7.1$, 5.5 Hz, 1H), 2.24 (q, $J = 7.1$ Hz, 2H), 1.85 (brs, 1H), 1.58 (q, $J = 6.7$ Hz, 1H), 1.22 (d, $J = 7.1$ Hz, 3H), 0.15 (s, 9H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 136.5, 116.7, 110.2, 86.4, 63.5, 45.1, 34.2, 27.9, 18.9. IR (film) $\nu_{max}$/cm$^{-1}$ 3332, 3078, 2960, 2898, 2165, 1640, 1443, 1250, 1041, 916, 844, 760. HRMS (ESI): Calcd for C$_{19}$H$_{33}$OSi [M+H]$^+$ 211.1512, found 211.1499.

(S)-4-((S)-4-(trimethylsilyl)but-3-yn-2-yl)dihydrofuran-2(3H)-one (ent-9b) ent-9b (1.55 g) was prepared from ent-16 (2.2 g 10.46 mmol) following the procedure described for 9b in 71% yield. $[\alpha]_D^{21} = +65.2$ (c 0.46, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 4.43 (dd, $J = 9.2$, 7.0 Hz, 1H), 4.22 (dd, $J = 9.1$, 6.8 Hz, 1H), 2.66–2.55 (m, 3H), 2.48–2.39 (m, 1H), 1.20 (d, $J = 6.4$ Hz, 3H), 0.15 (s, 9H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 176.6, 106.3, 87.7, 70.7, 70.0, 40.2, 32.7, 29.7, 18.8. 0.03. IR (film) $\nu_{max}$/cm$^{-1}$ 2962, 2170, 1796, 1247, 1174, 916, 842, 759. HRMS (ESI): Calcd for C$_{23}$H$_{39}$O$_2$Si [M+H]$^+$ 211.1148, found 211.1144.

(3S,4R)-3-((15,2R)-1-hydroxy-2-methylbutyl)-4-((S)-4-(trimethylsilyl)but-3-yn-2-yl)dihydrofuran-2(3H)-one (ent-17) ent-17 (635 mg) was prepared from ent-9b (550 mg 2.6 mmol) following the procedure described for 7b in 82% yield. $[\alpha]_D^{21.5} = +118.8$ (c 0.7, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 4.37 (t, $J = 8.9$ Hz 1H), 4.21 (t, $J = 8.9$ Hz, 1H), 3.56 (t, $J = 6.0$ Hz, 1H), 2.86 (dd, $J = 9.5$, 6.2 Hz, 1H), 2.82 (dd, $J = 7.1$, 4.3 Hz, 1H), 2.48 (m, 1H), 1.72–1.66 (m, 1H), 1.58–1.51 (m, 1H), 1.37–1.30 (m, 1H), 1.18 (d, $J = 7.2$ Hz, 3H), 1.99 (d, $J = 6.7$ Hz, 3H), 0.93 (t, $J = 7.5$ Hz, 3H), 0.15 (s, 9H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 179.0, 105.6, 89.3, 68.2, 45.5, 42.7, 37.5, 28.2, 26.4, 18.5, 13.4, 11.3. 0.02. IR (film) $\nu_{max}$/cm$^{-1}$ 3470, 2965, 2170, 1740, 1250, 1196, 843, 759. HRMS (ESI): Calcd for C$_{25}$H$_{36}$O$_2$Si [M+H]$^+$ 297.1880, found 297.1872.
(2R,3R,4S,5R)-2-((S)-but-3-yn-2-yl)-3-(hydroxymethyl)-5-methylheptane-1,4-diol \textit{(ent-18)}

\textit{ent-18} (250 mg) was prepared from \textit{ent-17} (600 mg 2.0 mmol) following the procedure described for \textit{6} in 60\% yield over two steps. $[\alpha]_D^{21.1} = +16.6$ (c 0.65, CHCl$_3$). $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 4.04–3.93 (m, 2H), 3.84 (td, $J = 12.6, 11.8, 2.7$ Hz, 2H), 3.63 (t, $J = 5.6$ Hz, 1H), 2.77 (m, 1H), 2.09 (d, $J = 2.5$ Hz, 1H), 1.88–1.84 (m, 1H), 1.81–1.76 (m, 1H), 1.67–1.57 (m, 1H), 1.49–1.39 (m, 1H), 1.29 (d, $J = 7.0$ Hz, 3H), 1.24–1.15 (m, 1H), 0.97 (d, $J = 6.7$ Hz, 3H), 0.92 (t, $J = 7.4$ Hz, 3H). $^{13}$C NMR (101 MHz, CDCl$_3$): $\delta$ 86.8, 75.8, 70.0, 60.7, 59.8, 44.5, 41.3, 37.3, 26.0, 25.7, 19.4, 13.9, 11.1. IR (film) $\nu_{\text{max}}$/cm$^{-1}$ 3305, 2964, 2933, 2109, 1461, 1034, 635. HRMS (ESI): Calcd for C$_{13}$H$_{23}$O$_3$Na [M+Na]$^+$ 251.1617, found 251.1609.

(15,2R)-1-((1R,4R,5S,8S)-1,8-dimethyl-2,7-dioxabicyclo[3.2.1]octan-4-yl)-2-methylbutan-1-ol \textit{(ent-19)}

\textit{ent-19} (150 mg) was prepared from \textit{ent-18} (220 mg 0.96 mmol) following the procedure described for \textit{5} in 70\% yield. $[\alpha]_D^{21.1} = +37.5$ (c 0.16, acetone). $^1$H NMR (400 MHz, Acetone-$d_6$): $\delta$ 3.97 (d, $J = 8.5$ Hz, 1H), 3.91 (dd, $J = 11.5, 5.9, 1.1$ Hz, 1H), 3.84 (ddt, $J = 8.5, 4.4, 0.8$ Hz, 1H), 3.60 (t, $J = 11.3$ Hz, 1H), 3.45–3.40 (m, 1H), 2.13 (d, $J = 4.1$ Hz, 1H), 2.04–1.95 (m, 1H), 1.83 (q, $J = 7.0$ Hz, 1H), 1.52–1.38 (m, 2H), 1.33–1.28 (m, 1H), 1.25 (s, 3H), 0.93–0.88 (m, 6H), 0.85 (d, $J = 6.7$ Hz, 3H). $^{13}$C NMR (101 MHz, Acetone-$d_6$): $\delta$ 108.0, 73.7, 67.9, 64.9, 48.1, 44.0, 43.4, 37.2, 37.2, 27.5, 20.3, 14.6, 12.5, 12.2. IR (film) $\nu_{\text{max}}$/cm$^{-1}$ 3463, 2963, 2936, 2880, 1460, 1384, 1121, 1043, 842. HRMS (ESI): Calcd for C$_{13}$H$_{25}$O$_3$ [M+H]$^+$ 229.1798, found 229.1792.
$^1$H and $^{13}$C NMR Spectra of Reported Compounds

$^1$H NMR spectra of compound 13

$^{13}$C NMR spectra of compound 13
$^1$H NMR spectra of compound 14

$^{13}$C NMR spectra of compound 14
$^1$H NMR spectra of compound 10

$^{13}$C NMR spectra of compound 10
$^{1}$H NMR spectra of compound 15

$^{13}$C NMR spectra of compound 15
$^1$H NMR spectra of compound 16

$^{13}$C NMR spectra of compound 16
$^{1}$H NMR spectra of compound 9b

$^{13}$C NMR spectra of compound 9b
$^1$H NMR spectra of compound 7b

$^{13}$C NMR spectra of compound 7b
$^1$H NMR spectra of compound 6

$^{13}$C NMR spectra of compound 6
$^1$H NMR spectra of compound 5

$^{13}$C NMR spectra of compound 5
$^1$H NMR spectra of compound 1a

$^{13}$C NMR spectra of compound 1a
$^1$H NMR spectra of compound 17

$^{13}$C NMR spectra of compound 17
$^1$H NMR spectra of compound 18

$^{13}$C NMR spectra of compound 18
**1H NMR spectra of compound 19**

![1H NMR spectra of compound 19](image)

<table>
<thead>
<tr>
<th>f1 (ppm)</th>
<th>12.241</th>
<th>12.542</th>
<th>14.654</th>
<th>20.307</th>
<th>27.553</th>
<th>37.154</th>
<th>37.169</th>
<th>43.421</th>
<th>44.022</th>
<th>48.159</th>
<th>64.892</th>
<th>67.948</th>
<th>73.661</th>
<th>73.795</th>
</tr>
</thead>
</table>

**13C NMR spectra of compound 19**

![13C NMR spectra of compound 19](image)
$^{1}H$ NMR spectra of compound 1b

$^{13}C$ NMR spectra of compound 1b
$^1$H NMR spectra of compound 3

$^{13}$C NMR spectra of compound 3
$^{1}H$ NMR spectra of compound 20

$^{13}C$ NMR spectra of compound 20
S21

$^{13}$C NMR spectra of compound 1c

$^{1}H$ NMR spectra of compound 1c

C NMR spectra of compound 1c
$^1$H NMR spectra of compound 2 in (CD$_3$)$_2$CO

$^{13}$C NMR spectra of compound 2 in (CD$_3$)$_2$CO
$^{1}H$ NMR spectra of compound 2 in CD$_3$OD

$^{13}C$ NMR spectra of compound 2 in CD$_3$OD
$^1$H NMR analysis of the ratio of 2/1c epimers

$^1$H NMR spectra of compounds 2 and 1c epimers (entry 2 in Table 2)

$^1$H NMR spectra of compounds 2 and 1c epimers (entry 6 in Table 2)
X-Ray Crystallographic Data

Crystal data and structure refinement for Data CCDC 1533934

Empirical formula  
Formula weight  
Temperature / K  
Crystal system  
Space group  
a / Å, b / Å, c / Å  
α/°, β/°, γ/°  
Volume / Å³  
Z  
ρ calc / mg mm⁻³  
μ / mm⁻¹  
F(000)  
Crystal size / mm³  
2θ range for data collection  
Index ranges  
Reflections collected  
Independent reflections  
Data/restraints/parameters  
Goodness-of-fit on F²  
Final R indexes [I>2σ(I)] i.e. Fo>4σ(Fo)  
Final R indexes [all data]  
Largest diff. peak/hole / e Å⁻³  
Flack Parameters  
Completeness  

C₁₆H₂₈O₃Si  
296.47  
104.6  
monoclinic  
P2₁  
90, 91.679(3), 90  
1780.22(10)  
4  
1.106  
1.200  
648  
0.200 × 0.130 × 0.100  
9.67 to 142.256°  
-12 ≤ h ≤ 11, -11 ≤ k ≤ 11, -22 ≤ l ≤ 21  
13060  
6632[R(int) = 0.0299 (inf-0.9Å)]  
6632/1/375  
1.099  
R₁ = 0.0579, wR₂ = 0.1552  
R₁ = 0.0597, wR₂ = 0.1572  
0.941/-0.237  
0.03(2)  
0.999
### Crystal data and structure refinement for Data CCDC 1533935

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Empirical formula</strong></td>
<td>C_{16}H_{28}O_{3}Si</td>
</tr>
<tr>
<td><strong>Formula weight</strong></td>
<td>296.47</td>
</tr>
<tr>
<td><strong>Temperature / K</strong></td>
<td>105.3</td>
</tr>
<tr>
<td><strong>Crystal system</strong></td>
<td>monoclinic</td>
</tr>
<tr>
<td><strong>Space group</strong></td>
<td>P2₁</td>
</tr>
<tr>
<td><strong>a / Å, b / Å, c / Å</strong></td>
<td>12.0052(10), 9.6664(6), 15.8157(12)</td>
</tr>
<tr>
<td><strong>α°, β°, γ°</strong></td>
<td>90.00, 96.318(8), 90.00</td>
</tr>
<tr>
<td><strong>Volume / Å³</strong></td>
<td>1824.2(2)</td>
</tr>
<tr>
<td><strong>Z</strong></td>
<td>4</td>
</tr>
<tr>
<td><strong>ρ_{calc} / mg mm³</strong></td>
<td>1.079</td>
</tr>
<tr>
<td><strong>μ / mm⁻¹</strong></td>
<td>1.171</td>
</tr>
<tr>
<td><strong>F(000)</strong></td>
<td>648</td>
</tr>
<tr>
<td><strong>Crystal size / mm²</strong></td>
<td>0.42 × 0.17 × 0.05</td>
</tr>
<tr>
<td><strong>2Θ range for data collection</strong></td>
<td>8.8 to 142.72°</td>
</tr>
<tr>
<td><strong>Index ranges</strong></td>
<td>-14 ≤ h ≤ 14, -11 ≤ k ≤ 11, -19 ≤ l ≤ 19</td>
</tr>
<tr>
<td><strong>Reflections collected</strong></td>
<td>12860</td>
</tr>
<tr>
<td><strong>Independent reflections</strong></td>
<td>6840[R(int) = 0.0300 (inf-0.9Å)]</td>
</tr>
<tr>
<td><strong>Data/restraints/parameters</strong></td>
<td>6840/1/375</td>
</tr>
<tr>
<td><strong>Goodness-of-fit on F²</strong></td>
<td>1.036</td>
</tr>
<tr>
<td><strong>Final R indexes [I&gt;2σ (I) i.e. F_o&gt;4σ (F_o)]</strong></td>
<td>R¹ = 0.0404, wR² = 0.1070</td>
</tr>
<tr>
<td><strong>Final R indexes [all data]</strong></td>
<td>R¹ = 0.0456, wR² = 0.1142</td>
</tr>
<tr>
<td><strong>Largest diff. peak/hole / e Å⁻³</strong></td>
<td>0.454/-0.205</td>
</tr>
<tr>
<td><strong>Flack Parameters</strong></td>
<td>0.03(2)</td>
</tr>
<tr>
<td><strong>Completeness</strong></td>
<td>0.986</td>
</tr>
</tbody>
</table>
**NMR Comparison Table of natural colomitide B and synthetic 2**

Table S1 The comparison of $^1$H and $^{13}$C NMR data of reported natural colomitide B and synthetic 2 in (CD$_3$)CO

<table>
<thead>
<tr>
<th>Position</th>
<th>$\delta$ H (600 MHz $J$ in Hz)</th>
<th>$\delta$ C (150MHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Natural colomitide B</td>
<td>Synthetic 2</td>
</tr>
<tr>
<td>1</td>
<td>109.1</td>
<td>109</td>
</tr>
<tr>
<td>3</td>
<td>A 4.04 (d, 11.7)</td>
<td>A 4.04 (d, 11.7)</td>
</tr>
<tr>
<td></td>
<td>B 3.79 (dd, 11.8, 5.1)</td>
<td>3.79 (dd, 11.7, 5.1)</td>
</tr>
<tr>
<td>4</td>
<td>2.76 (t, 8.1, 3.5)</td>
<td>2.77 m</td>
</tr>
<tr>
<td>5</td>
<td>2.71 (d, 3.5)</td>
<td>2.72 m</td>
</tr>
<tr>
<td>6</td>
<td>A 4.11 (dd, 8.3, 4.5)</td>
<td>A 4.12 (dd, 8.4, 4.5)</td>
</tr>
<tr>
<td></td>
<td>B 3.95 (d, 8.4)</td>
<td>3.96 (d, 8.4)</td>
</tr>
<tr>
<td>8</td>
<td>1.79 (dd, 14.0, 7.0)</td>
<td>1.80 (dd, 14.0, 7.0)</td>
</tr>
<tr>
<td>9</td>
<td>1.21 (s)</td>
<td>1.22 (s)</td>
</tr>
<tr>
<td>10</td>
<td>0.87 (d, 7.0)</td>
<td>0.88 (d, 7.1)</td>
</tr>
<tr>
<td>1'</td>
<td>214</td>
<td>214</td>
</tr>
<tr>
<td>2'</td>
<td>2.86 (dd, 13.4, 6.7)</td>
<td>2.86 (dd, 13.4, 6.7)</td>
</tr>
<tr>
<td>3'</td>
<td>A 1.62-1.57 (m)</td>
<td>A 1.68–1.61 (m)</td>
</tr>
<tr>
<td></td>
<td>B 1.34-1.29 (m)</td>
<td>B 1.38–1.31 (m)</td>
</tr>
<tr>
<td>4'</td>
<td>0.84 (t, 7.4)</td>
<td>0.84 (t, 7.4)</td>
</tr>
<tr>
<td>5'</td>
<td>1.01 (d, 6.7)</td>
<td>1.02 (d, 6.8)</td>
</tr>
</tbody>
</table>