Chemistry of Natural Compounds and Bioorganic Chemistry

Synthesis of optically active analogs of α-tocopherol based on (*S*)-(+)-dihydromyrcene

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New optically active analogs of α-tocopherol (vitamin E) were synthesized starting from enantiomerically enriched (*ee* ≈ 50%) (*S*)-(+)-dihydromyrcene.

Key words: analogs of α-tocopherol (vitamin E), isoprenoid vinyl alcohols, (*S*)-(+)-3,7-dimethylocta-1,6-diene ((*S*)-(+)-dihydromyrcene), (3*S*)-3,7-dimethyloct-6-en-2-one, (3*S*)-3,7-dimethyloctan-2-one.

In recent years, considerable attention has been given to the synthesis of analogs of α-tocopherol (vitamin E).[1] Compounds of this series, prepared based on linalool or dihydrolinalool (these analogs are named C_6-chromans from the number of carbon atoms in the side chain), were found to exhibit high biological activity.[2]

We used bishomoisoprenoids enantiomerically enriched at the C(4) atom, *viz.*, (3*R*/*S*,4*S*)-3,4,8-trimethylnon-1-en-3-ol and (3*R*/*S*,4*S*)-3,4,8-trimethylnona-1,7-dien-3-ol, as the starting compounds for preparing new optically active analogs of α-tocopherol. The starting compounds were synthesized from readily accessible[3] (*S*)-(+)-dihydromyrcene (**1**) (*ee* ≈ 50%), which was converted into (3*S*)-(+)-3,7-dimethyloct-6-en-2-one (**2**).[4] Catalytic hydrogenation of the latter afforded saturated ketone **3** (Scheme 1). The reactions of ketones **2** and **3** with vinylmagnesium bromide gave rise to the required vinyl alcohols **4** and **8**, respectively (mixtures of diastereomers). Their condensation with trimethylhydroquinone (TMHQ) in the presence of $BF_3 \cdot OEt_2$ yielded mixtures of stereoisomeric optically active C_8-chromans **5** and **9** (*erythro* : *threo* ≈ 1 : 1, GLC data), respectively. Since the starting dihydromyrcene **1** is characterized by *ee* equal to ~50%, it is evident that the *erythro* and *threo* diastereomers of chromans **5** and **9** are enriched to the same extent with epimers with the *S* configuration at the C(1′) atom.

The ^{13}C NMR spectra of allyl alcohols **4** and **8** and chromans **5** and **9** have double sets of signals for some carbon atoms, which is characteristic of mixtures of diastereomers.[5] In the spectra of alcohols **4** and **8**, the most pronounced difference in the chemical shifts is

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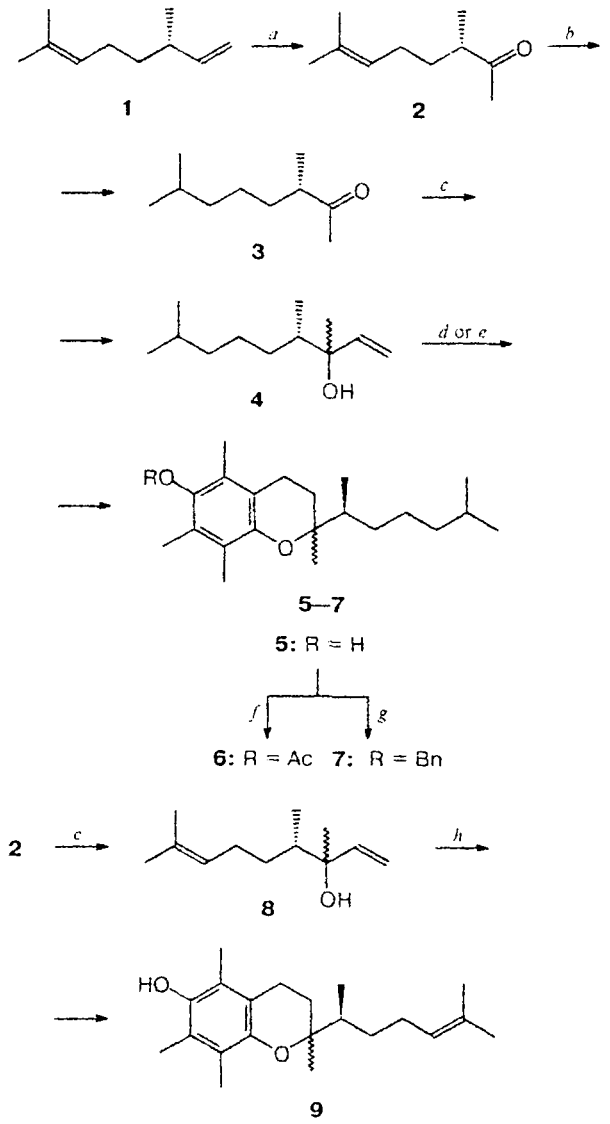
Scheme 1



Reagents and conditions: *a*. O_2/Cu_2Cl_2—$PdCl_2$, DMF—H_2O; *b*. H_2/Pd—C; *c*. CH_2=CHMgBr, THF; *d*. TMHQ, $BF_3 \cdot OEt_2$, *n*-C_7H_{16}; *e*. TMHQ, Tseokar-10, *n*-C_9H_{20}; *f*. Ac_2O; *g*. BnCl, K_2CO_3, DMF; *h*. TMHQ, $BF_3 \cdot OEt_2$, PhH.

observed for the C(2) atoms ($\Delta\delta$ = 0.46 and 0.52, respectively). In the spectra of chromans **5** and **9**, the largest differences are observed for the C(3) ($\Delta\delta$ = 0.43 and 0.51, respectively) and C(2′) atoms ($\Delta\delta$ = 0.97 and 0.92, respectively). The methyl groups at the C(1′), C(7), and C(8) atoms in saturated chroman **5** also give two signals each ($\Delta\delta$ = 0.49, 0.92, and 0.42, respectively), whereas the distinct differences between the diastereomers of its acetyl (**6**) and benzyl (**7**) derivatives are manifested only in the chemical shifts of the signals for the C(2′) atom ($\Delta\delta$ = 0.82 and 0.91, respectively) and the Me group at position 7 ($\Delta\delta$ = 0.60 and 0.84, respectively). The fact that the ^{1}H NMR spectra of compounds **5**—**7** have two doublets with equal intensities, which correspond to the protons of the methyl group at the C(1′) atom (δ 0.9—1.1), indicates that these compounds exist as equimolar mixtures of (2*R*,1′*S*)-*erythro* and (2*S*,1′*S*)-*threo* diastereomers.

It should be noted that the key stage in the synthesis of C_8-chromans, *viz.*, condensation of TMHQ with vinyl alcohols **4** and **8**, catalyzed by $BF_3 \cdot OEt_2$ proceeded in 71 and 33% yields, respectively, whereas the yields obtained in this stage in the presence of other Lewis acids used by us ($ZnCl_2$ or $AlCl_3$) were lower. We found that the yield of chroman **5** was increased to 93% in the presence of zeolite Tseokar-10. Unfortunately, this catalyst did not allow us to obtain chroman **9** in more acceptable yield.

Experimental

The IR spectra were recorded on a Specord 75-IR spectrometer (in thin layers). The UV spectra were measured on a Specord M-40 spectrometer in $CHCl_3$. The ^{1}H and ^{13}C NMR spectra were obtained on Bruker AM-300 (300.13 and 75 MHz for ^{1}H and ^{13}C, respectively) and Jeol FX 90Q (90 and 22.5 MHz for ^{1}H and ^{13}C, respectively; for compounds **4** and **8**) instruments in $CDCl_3$. The chemical shifts are given in the δ scale relative to Me_4Si (internal standard). The GLC analysis was carried out on a Chrom-5 chromatograph (a 2400×4-mm column, SE-30 (5%) as the stationary phase on Chromaton N-AW-DMCS, the temperature range was 50—300 °C (8 deg min^{-1}), helium as the carrier gas). HPLC was performed on an LKB liquid chromatograph (Sweden) equipped with a UV detector (λ = 249 nm) and a column with Separon-C18 as the adsorbent (125×4 mm, 5 μm); the 87 : 13 MeOH—H_2O system was used as the eluent (0.3 mL min^{-1}, 20 °C). The specific angles of rotation were measured on a Perkin—Elmer-141 polarimeter.

(3*S*)-3,7-Dimethyloctan-2-one (3). A mixture of ketone **2** (3.65 g, 23.7 mmol; **2** was prepared according to a procedure reported previously[4]; n_D^{20} 1.4470, $[\alpha]_D^{20}$ −5.8° (*c* 1.6, $CHCl_3$)), the 5% Pd—C catalyst (0.365 g), and MeOH (36.5 mL) was stirred under an atmosphere of hydrogen for 24 h. The catalyst was filtered off and the filtrate was concentrated *in vacuo*. Ketone **3** was obtained in a yield of 3.36 g (92.5%), n_D^{20} 1.4350, $[\alpha]_D^{20}$ −4.7° (*c* 3.5, $CHCl_3$). Found (%): C, 76.97; H, 12.81. $C_{10}H_{20}O$. Calculated (%): C, 76.86; H, 12.90. IR, ν/cm^{-1}: 1710 (C=O). ^{1}H NMR, δ: 0.86 (d, 6 H, H(8), C(7)CH_3, *J* = 6.3 Hz); 1.08 (d, 3 H, C(3)CH_3, *J* = 7.0 Hz); 1.20—1.59 (m, 7 H, H(4), H(5), H(6), H(7)); 2.13 (s, 3 H, H(1)); 2.38 (m, 1 H, H(3)). ^{13}C NMR, δ: 15.78 (C(3)$\underline{C}H_3$); 22.18 (C(8), C(7)$\underline{C}H_3$); 24.59 (C(5)); 27.42 (C(7)); 27.60 (C(1)); 38.54 (C(6)); 32.71 (C(4)); 46.71 (C(3)); 212.41 (C(2)).

(3*R*/*S*,4*S*)-3,4,8-Trimethylnon-1-en-3-ols (4). A solution of ketone **3** (0.45 g, 2.92 mmol) in THF (2 mL) was added to a solution of the Norman reagent, which was prepared from Mg chips (0.27 g, 11.3 mmol) and vinyl bromide (1.22 g, 11.4 mmol), in THF (7 mL) under Ar at −25 °C. The reaction mixture was stirred for 3 h and then treated with a saturated NH_4Cl solution (6 mL). The organic layer was separated and the aqueous layer was extracted with Et_2O. The combined organic layers were washed with a saturated solution of NaCl and dried with $MgSO_4$. The solvent was evaporated *in vacuo*. After chromatog-

raphy (SiO_2, a 10 : 1 hexane—Et_2O mixture as the eluent), a mixture of alcohols **4** was obtained in a yield of 0.45 g (86%), n_D^{20} 1.4486, $[\alpha]_D^{15}$ −6.17° (*c* 0.84, $CHCl_3$). Found (%): C, 78.32; H, 13.20. $C_{12}H_{24}O$. Calculated (%): C, 78.19; H, 13.13. IR, ν/cm^{-1}: 910, 995, 3090 (CH=CH_2); 1640 (C=C); 3400 (OH). ^1H NMR, δ: 0.86 (d, 6 H, H(9), C(8)CH_3, J = 6.1 Hz); 0.90 (d, 3 H, C(4)CH_3, J = 6.1 Hz); 1.04—1.65 (m, 8 H, H(4), H(5), H(6), H(7), H(8)); 1.22 (s, 3 H, C(3)CH_3); 1.94 (s, 1 H, OH); 4.98—5.29 (m, 2 H, H(1)); 5.91 (dd, 1 H, H(2), J = 17.6 and 11.8 Hz). ^{13}C NMR, δ: 14.05, 14.24 (C(4)$\underline{C}H_3$); 22.57 (C(8)$\underline{C}H_3$); 22.89 (C(9)); 24.52 (C(6)); 25.95 (C(3)$\underline{C}H_3$); 28.03 (C(8)); 31.41, 31.54 (C(5)); 39.34 (C(7)); 43.11 (C(4)); 75.57 (C(3)); 112.11, 112.24 (C(1)); 144.17, 144.63 (C(2)).

2-(1′*S*,5′-Dimethylhexyl)-6-hydroxy-2*R*/*S*,5,7,8-tetramethylchromans (5). ***A.*** $BF_3 \cdot OEt_2$ (0.24 mL, 0.17 mmol) was added with stirring to a suspension of TMHQ (0.18 g, 1.19 mmol) in anhydrous heptane (3 mL) under Ar at 98 °C. After 5 min, a mixture of alcohols **4** (0.39 g, 2.11 mmol) was added dropwise. The reaction mixture was refluxed with stirring for 12 h, cooled to −20 °C, and filtered. The filtrate was concentrated. The residue was chromatographed on a column with SiO_2; elution was carried out first with *n*-hexane and then with a 10 : 1 hexane—Et_2O mixture. Chromans **5** were obtained in a yield of 0.27 g (71%). According to the HPLC data, these compounds existed as a mixture (1 : 1) of the *erythro* and *threo* diastereomers (t = 25.2 and 28.8 min, respectively). $[\alpha]_D^{15}$ −1.67° (*c* 2.0, $CHCl_3$). Found (%): C, 79.35; H, 10.88. $C_{21}H_{34}O_2$. Calculated (%): C, 79.19; H, 10.76. UV, λ_{max}/nm (ε): 295 (3900). ^1H NMR, δ: 0.80—0.88 (m, 6 H, H(6′), C(5′)CH_3); 0.90 (d, 1.5 H, C(1′)CH_3, J = 6.7 Hz); 0.95 (d, 1.5 H, C(1′)CH_3, J = 6.8 Hz); 1.11 (s, 3 H, C(2)CH_3); 1.2—1.9 (m, 10 H, H(3), H(1′), H(2′), H(3′), H(4′), H(5′)); 2.10 (s, 6 H, ArCH_3); 2.15 (s, 3 H, ArCH_3); 2.57 (t, 2 H, H(4), J = 6.7 Hz); 4.25 (s, 1 H, OH). ^{13}C NMR, δ: 11.35 (C(5)$\underline{C}H_3$); 11.86, 12.28 (C(8)$\underline{C}H_3$); 13.47, 14.39 (C(7)CH_3); 19.45, 19.94 (C(1′)$\underline{C}H_3$); 20.52, 20.60 (C(4)); 22.62 (C(6′)); 22.80, 22.86 (C(2)$\underline{C}H_3$); 25.89, 25.97 (C(3′)); 27.84, 27.97 (C(5′)); 29.24, 29.67 (C(3)); 30.69, 31.66 (C(2′)); 39.29 (C(4′)); 39.90 (C(1′)); 77.15, 77.21 (C(2)); 117.63 (C(5)); 118.45, 118.55 (C(10)); 121.04, 121.05 (C(8)); 122.65 (C(7)); 144.49 (C(9)); 145.35 (C(6)).

B. A mixture of alcohols **4** (0.7 g, 3.8 mmol) was added dropwise (Ar, 150 °C) to a suspension of TMHQ (0.24 g, 1.59 mmol) and the powdered catalyst Tseokar-10 (0.52 g; a zeolite-containing aluminosilicate catalyst of cracking of petroleum fractions; the Salavatnefteorgsintez Joint-Stock Company) in anhydrous *n*-nonane (7 mL). The reaction mixture was refluxed for 4 h, cooled to −20 °C, and filtered. The filtrate was concentrated and the residue was chromatographed on SiO_2 as described above (experiment *A*). Chromans **5** were isolated in a yield of 0.47 g (93%). The resulting compounds were identical (the IR spectra and the ^1H and ^{13}C NMR spectra) to those obtained in experiment *A*.

6-Acetoxy-2-(1′*S*,5′-dimethylhexyl)-2*R*/*S*,5,7,8-tetramethylchromans (6). A solution of a mixture of chromans **5** (0.15 g, 10.47 mmol) in Ac_2O (0.15 mL) was refluxed for 3 h, cooled, poured into ice water (10 mL), and extracted with Et_2O. The extract was successively washed with saturated solutions of $NaHCO_3$ and NaCl, dried with $MgSO_4$, concentrated, and chromatographed (SiO_2, hexane as the eluent). A 1 : 1 mixture of the *erythro* and *threo* diastereomers of **6** (GLC data) was obtained in a yield of 0.13 g (77%), $[\alpha]_D^{14}$ −4.75° (*c* 1.8, $CHCl_3$). Found (%): C, 76.91; H, 10.20. $C_{23}H_{36}O_3$. Calculated (%): C, 76.62; H, 10.07. IR, ν/cm^{-1}: 1740 (C=O). UV, λ_{max}/nm (ε): 277 (1760), 286 (3100). ^1H NMR, δ: 0.83—0.92 (m, 6 H, H(6′), C(5′)Me); 0.95 (d, 1.5 H, C(1′)Me, J = 6.8 Hz); 1.05 (d, 1.5 H, C(1′)CH_3, J = 6.7 Hz); 1.18 (s, 3 H, C(2)CH_3); 1.3—1.9 (m, 10 H, H(3), H(1′), H(2′), H(3′), H(4′), H(5′)); 2.02 (s, 3 H, ArCH_3); 2.09 (s, 3 H, ArCH_3); 2.18 (s, 3 H, ArCH_3); 2.39 (s, 3 H, O=CCH_3); 2.60 (t, 2 H, H(4), J = 6.6 Hz). ^{13}C NMR, δ: 11.85, 12.09 (C(8)$\underline{C}H_3$); 12.94 (C(5)$\underline{C}H_3$); 13.55, 14.15 (C(7)$\underline{C}H_3$); 20.33, 20.41 (C(4)); 20.56 (C(1′)$\underline{C}H_3$, O=C$\underline{C}H_3$); 22.55 (C(6′)); 22.78 (C(2)$\underline{C}H_3$); 25.85, 25.90 (C(3′)); 27.83, 27.92 (C(5′)); 30.73, 31.55 (C(2′)); 31.91 (C(3)); 39.25 (C(1′), C(4′)); 77.67 (C(2)); 117.60 (C(10)); 124.80, 124.86 (C(8)); 123.06 (C(5)); 126.63 (C(7)); 140.48 (C(9)); 149.20 (C(6)); 169.75 (C=O).

6-Benzyloxy-2-(1′*S*,5′-dimethylhexyl)-2*R*/*S*,5,7,8-tetramethylchromans (7). Potassium carbonate (0.38 g) and then benzyl chloride (0.33 g, 2.9 mmol) were added with stirring to a solution of a mixture of chromans **5** (0.33 g, 1.04 mmol) in anhydrous DMF (21 mL). The reaction mixture was kept at −20 °C for 38 h, poured onto ice, and extracted with Et_2O. After standard treatment and flash chromatography (SiO_2; hexane as the eluent), a mixture of ethers **7** was obtained in a yield of 0.26 g (62%), $[\alpha]_D^{15}$ −3.33° (*c* 3.4, $CHCl_3$). Found (%): C, 82.51; H, 9.72. $C_{28}H_{40}O_2$. Calculated (%): C, 82.30, H, 9.87. UV, λ_{max}/nm (ε): 270 (2300), 294 (4400). ^1H NMR, δ: 0.91—1.02 (m, 6 H, H(6′), C(5′)CH_3); 1.00 (d, 1.5 H, C(1′)CH_3, J = 6.8 Hz); 1.09 (d, 1.5 H, C(1′)CH_3, J = 6.8 Hz); 1.20 (s, 3 H, C(2)CH_3); 1.3—2.0 (m, 10 H, H(3), H(1′), H(2′), H(3′), H(4′), H(5′)); 2.20 (s, 3 H, ArCH_3); 2.25 (s, 3 H, ArCH_3); 2.30 (s, 3 H, ArCH_3); 2.65 (t, 2 H, H(4), J = 6.8 Hz); 4.80 (s, 2 H, OCH_2); 7.36—7.56 (m, 5 H, C_6H_5). ^{13}C NMR, δ: 11.82, 11.95 (C(8)$\underline{C}H_3$); 12.61 (C(5)$\underline{C}H_3$); 13.51, 14.35 (C(7)$\underline{C}H_3$); 20.40, 20.47 (C(4)); 20.65, 20.74 (C(1′)$\underline{C}H_3$); 22.52 (C(2)$\underline{C}H_3$, C(6′)); 25.80, 25.90 (C(3′)); 27.75, 27.90 (C(5′)); 30.67, 31.58 (C(2′)); 39.16, 39.21 (C(1′)); 39.91, 40.03 (C(4′)); 74.66 (Ph—CH_2); 77.43 (C(2)); 117.81 (C(10)); 122.93 (C(5)); 125.79 (C(8)); 125.87 (C(7)); 127.64 (C(2″), C(4″), C(6″)); 128.41 (C(3″), C(5″)); 138.03 (C(9)); 147.75 (C(1″)); 148.09 (C(6)).

(3*R*/*S*,4*S*)-3,4,8-Trimethylnona-1,7-dien-3-ols (8). A mixture of alcohols **8** was prepared as described above by the reaction of ketone **2** (3.0 g, 19.5 mmol) with the Norman reagent, which was synthesized from Mg (1.8 g, 75 mmol) and vinyl bromide (8.05 g, 75.2 mmol) in THF (26 mL), in a yield of 3.0 g (85%), n_D^{20} 1.6350, $[\alpha]_D^{13}$ −7.8° (*c* 6.9, $CHCl_3$). Found (%): C, 79.33, H, 12.29. $C_{12}H_{22}O$. Calculated (%): C, 79.06; H, 12.16. IR, ν/cm^{-1}: 905, 995, 3080 (CH=CH_2); 1630 (C=C); 3400 (OH). ^1H NMR, δ: 0.90 (d, 3 H, C(4)CH_3, J = 6.8 Hz); 1.23 (s, 3 H, C(3)CH_3); 1.02—1.97 (m, 5 H, H(4), H(5), H(6)); 1.59 (s, 3 H, H(9)); 1.67 (s, 3 H, C(8)CH_3); 4.99—5.29 (m, 2 H, H(1)); 5.59 (m, 1 H, H(7)); 5.92 (dd, 1 H, H(2), J = 17.3 and 11.0 Hz). ^{13}C NMR, δ: 13.82, 14.15 (C(4)$\underline{C}H_3$); 17.73 (C(8)$\underline{C}H_3$); 24.88 (C(9)); 25.73 (C(3)$\underline{C}H_3$); 26.51 (C(6)); 31.38, 31.58 (C(5)); 42.57, 42.70 (C(4)); 75.73 (C(3)); 112.15, 112.35 (C(1)); 124.70 (C(7)); 131.27, 131.40 (C(8)); 144.08, 144.60 (C(2)).

2-(1′*S*,5′-Dimethyl-4-hexen-1-yl)-6-hydroxy-2*R*/*S*,5,7,8-tetramethylchromans (9). A mixture of alcohols **8** (3.1 g, 17.05 mmol) was added dropwise to a stirred solution of TMHQ (11.3 g, 8.53 mmol) and $BF_3 \cdot OEt_2$ (0.25 mL) in anhydrous benzene (38 mL) at the boiling temperature under Ar. The reaction mixture was refluxed for 3 h, cooled to −20 °C, successively washed with water and saturated solutions of $NaHCO_3$ and NaCl, dried with $MgSO_4$, and concentrated. The residue was chromatographed on SiO_2 (a 10 : 1 *n*-hexane—Et_2O mixture as the eluent). A mixture of chromans **9** was obtained in a yield of 0.9 g (33%), $[\alpha]_D^{14}$ −2.0° (*c* 3.4, $CHCl_3$). Found (%): C, 79.52, H, 10.02. $C_{21}H_{32}O_2$. Calculated (%): C, 79.70; H, 10.19. UV, λ_{max}/nm (ε): 296 (3800). ^1H NMR, δ: 1.02—1.95 (m, 7 H, H(1′), H(2′), H(3′)); 1.15 (s, C(2)CH_3); 1.63 (s, 3 H,

H(6′)); 1.70 (s, 3 H, C(5′)CH_3); 2.12 (s, 3 H, ArCH_3); 2.13 (s, 3 H, ArCH_3); 2.17 (s, 3 H, ArCH_3); 2.58 (t, 2 H, H(4), J = 6.8 Hz); 4.55 (s, 1 H, OH); 5.10 (m, 1 H, H(4′)). ^{13}C NMR, δ: 11.17 (C(5)CH_3); 11.69, 11.88 (C(8)$\underline{C}H_3$); 12.14, 12.18 (C(7)$\underline{C}H_3$); 15.74 (C(5′)$\underline{C}H_3$); 17.88 (C(1′)$\underline{C}H_3$); 19.33, 19.79 (C(2)$\underline{C}H_3$); 20.94 (C(4)); 25.48, 25.59 (C(6′)); 26.41 (C(3′)); 29.12, 29.63 (C(3)); 30.76, 31.68 (C(2′)); 38.80, 39.12 (C(1′)); 77.00 (C(2)); 117.41 (C(5)); 118.54, 118.66 (C(10)); 121.24 (C(8)); 122.41 (C(7)); 124.66, 124.80 (C(4′)); 131.07, 131.36 (C(5′)); 144.51 (C(9)); 145.75 (C(6)).

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