ISSN 1070-4280, Russian Journal of Organic Chemistry, 2009, Vol. 45, No. 10, pp. 1464–1467. © Pleiades Publishing, Ltd., 2009.
Original Russian Text © V.N. Odinokov, E.R. Shakurova, L.M. Khalilov, U.M. Dzhemilev, 2009, published in Zhurnal Organicheskoi Khimii, 2009, Vol. 45, No. 10, pp. 1479–1482.

First Examples of Hydroxycyclopropanation in the Series of Lupane Triterpenoids

V. N. Odinokov, E. R. Shakurova, L. M. Khalilov, and U. M. Dzhemilev

Institute of Petroleum Chemistry and Catalysis, Russian Academy of Sciences,
pr. Oktyabrya 141, Ufa, 450075 Bashkortostan, Russia
e-mail: ink@anrb.ru

Received October 23, 2008

Abstract—Hydroxycyclopropanation at the ester group in the series of lupane triterpenoids was performed for the first time using 3,3-ethylenedioxybetulonic acid methyl ester and its 20,29-dihydro analog as substrates.

DOI: 10.1134/S1070428009100078

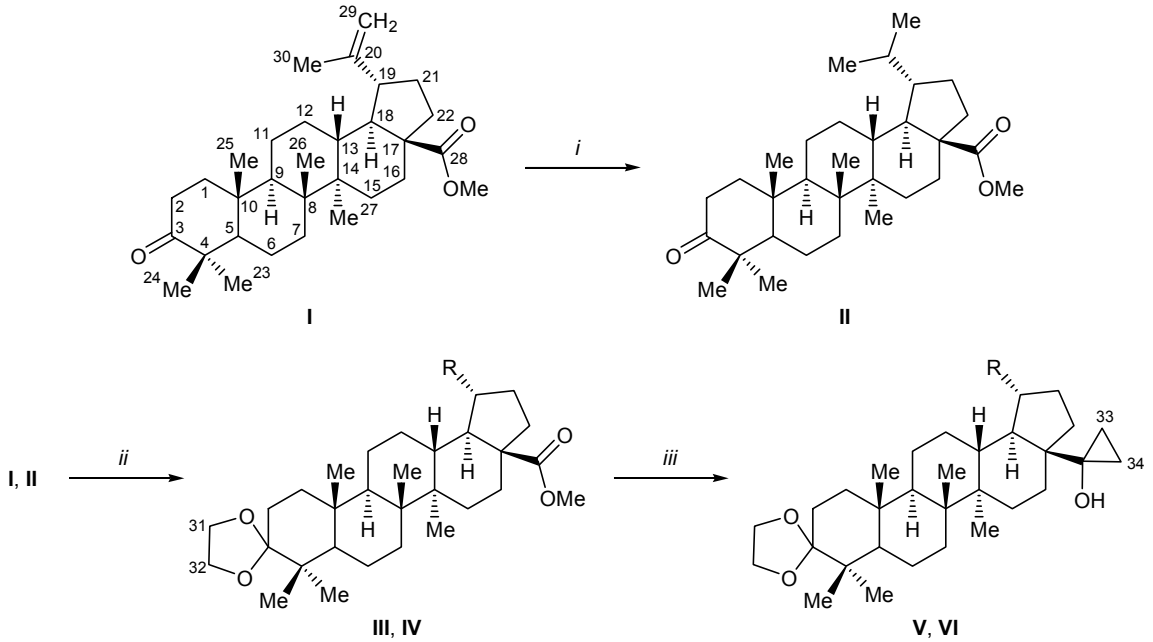
Hydroxycyclopropanation of carboxylic acid esters by the action of ethylmagnesium bromide in the presence of titanium(IV) isopropoxide as catalyst (Kulinkovich reaction) opened a way of introducing a pharmacologically important cyclopropane fragment into biologically active molecules [1] and determined new synthetic approaches to complex natural compounds and their analogs [2]. In the recent years, modification of betulin and its derivatives has attracted increased interest [3], and specific attention has been given to transformations of carboxy group with a view to obtain compounds possessing versatile biological activity [4].

We were the first to effect hydroxycyclopropanation in the series of lupane triterpenoids using 3,3-ethylenedioxybetulonic acid methyl ester (**III**) and its 20,29-dihydro analog **IV** as substrates. Ethylene acetals **III** and **IV** were obtained by reactions of betulonic acid methyl ester **I** and its 20,29-dihydro derivative **II**, respectively, with ethylene glycol in the presence of *p*-toluenesulfonic acid. 20,29-Dihydro analog **II** was synthesized by hydrogenation of ester **I** over platinum oxide. The formation of **II** was confirmed by the ^{1}H and ^{13}C NMR data. The ^{1}H NMR spectrum of **II** contained two doublets at δ 0.73 and 0.84 ppm (J = 6.4, 6.8 Hz) from protons in diastereotopic methyl groups in the isopropyl substituent instead of signals typical of isopropylidene group (δ_C 150.25, 109.51, 19.49 ppm). The structure of ethylene acetals **III** and **IV** follows from the presence in their ^{13}C NMR spectra of signals from carbon atoms in the dioxolane ring [δ_C, ppm: **III**: 113.21 (C^3), 64.75, 64.64 (C^{31}, C^{32}); **IV**: 113.33 (C^3), 64.86, 64.73 ppm (C^{31}, C^{32}) instead of signal typical of the C^3=O carbonyl carbon atom in **I** and **II** (δ_C 217.83 and 217.96 ppm, respectively). Compounds **III** and **IV** displayed in the ^{1}H NMR spectra additional multiplet signals in the region δ 3.81–3.93 ppm due to protons in the ethylenedioxy group.

While studying Kulinkovich hydroxycyclopropanation of esters **III** and **IV** we found that target compounds **V** and **VI** were formed only when a solution of ester **III** or **IV** and Ti(OPr-*i*)$_4$ in diethyl ether was added to a freshly prepared suspension of ethylmagnesium bromide. If the reactants were mixed in a different order, e.g., when a solution of EtMgBr and Ti(OPr-*i*)$_4$ in diethyl ether was added to a solution of the substrate, no hydroxycyclopropanation occurred. Presumably, the presence of the initial reactants at the moment of formation of the true hydroxycyclopropanating agent, diisopropoxytitanacyclopropane, is necessary for successful reaction [1].

The yields of 3,3-ethylenedioxy-28,28-ethanolup-20(29)-en-28-ol (**V**) and its 20,29-dihydro analog **VI** were 84 and 75%, respectively. Their structure was confirmed by the NMR data (^{1}H, ^{13}C, DEPT-135°), including two-dimensional experiments (COSY, HSQC). In the ^{13}C NMR spectrum of **V**, we observed a signal at δ_C 60.17 ppm (C^{28}OH) and two new triplets from the cyclopropane fragment (δ_C 15.80 and 15.63 ppm, DEPT-135°) instead of signals from the C^{28}(O)OMe ester group (δ_C 176.53, 51.14 ppm), while signals from C^{17} (δ_C 41.37 ppm) and C^{18} (δ_C 38.98 ppm) were displaced upfield relative to the corresponding signals of its precursor **III** ($\Delta\delta_C$ 15.09

Scheme 1.



III, **V**, R = CH_2=C(Me); **IV**, **VI**, R = Me_2CH; *i*: H_2/PtO_2, MeOH–$CHCl_3$, 2 : 1; *ii*: $HOCH_2CH_2OH$, TsOH, benzene, 80°C; *iii*: (1) $Ti(OPr\text{-}i)_4$/EtMgBr, Et_2O; (2) 5% NaOH.

and 3.05 ppm, respectively). Signals from the other carbon atoms in the ^{13}C NMR spectra of compounds **V** and **III** coincided within 1 ppm. The ^1H NMR spectrum of **V** contained two new strongly coupled two-proton signals (δ 0.83 and 0.67 ppm; COSY), which gave rise to cross peaks with C^{33} and C^{34} in the HSQC spectrum. Analogous differences were observed in the ^1H and ^{13}C NMR spectra of compound **VI** and its precursor **IV**. The above data indicated that the C^{28}(O)OMe group in **III** and **IV** was converted into hydroxycyclopropane fragment, the configuration of the other chiral centers remaining unchanged.

EXPERIMENTAL

The IR spectra were recorded on a Specord 75IR instrument. The ^1H and ^{13}C NMR spectra were measured on Bruker AM-300 (300.13 MHz for ^1H and 75.48 MHz for ^{13}C) and Bruker Avance-400 spectrometers (400.13 MHz for ^1H and 100.62 MHz for ^{13}C) using $CDCl_3$ as solvent and tetramethylsilane as internal reference. Homo- and heteronuclear correlations experiments (DEPT-135°, COSY, HSQC) were performed on a Bruker Avance-400 instrument (400.13 and 100.62 MHz for ^1H for ^{13}C, respectively). The optical rotations were determined on a Perkin–Elmer-141 polarimeter. Elemental analysis was performed using a Carlo Erba 1106 analyzer. The melting points were determined on a Boetius melting point apparatus. Column chromatography was performed on KSKG silica gel, and Silufol UV-254 plates were used for TLC; spots were detected by treatment with a 20% solution of phosphotungstic acid in ethanol, followed by heating at 100–120°C for 2–3 min.

Betulonic acid methyl ester (**I**) was synthesized according to the procedures described in [5, 6] from betulin isolated from *Betula pendula* bark [7].

Methyl 3-oxolupan-28-oate (II, 20,29-dihydrobetulonic acid methyl ester). Platinum(IV) oxide, 0.005 g, was added to a solution of 0.50 g (1.06 mmol) of compound **I** in 30 ml of a 2 : 1 methanol–chloroform mixture, and the mixture was stirred for 8 h under nitrogen. The mixture was filtered, the filtrate was evaporated, and the residue was subjected to chromatography in a column charged with 5 g of silica gel using chloroform–methanol (50 : 1) as eluent. Yield 0.47 g (94%), colorless crystals, mp 182–184°C (from methanol), $[\alpha]_D^{20}$ = +5.059° (*c* = 1.64, $CHCl_3$). IR spectrum (KBr), ν, cm^{-1}: 2950, 1725, 1470. ^1H NMR spectrum (400.13 MHz), δ, ppm: 0.73 d (3H, $C^{29}H_3$, *J* = 6.4 Hz), 0.81 s (3H, $C^{23}H_3$), 0.84 d (3H, $C^{30}H_3$, *J* = 6.8 Hz), 0.85 s (3H, $C^{24}H_3$), 0.89 s (3H, $C^{26}H_3$), 0.93 s (3H, $C^{25}H_3$), 1.01 m and 1.92 m (2H, 16-H), 1.04 m (1H, 5-H), 1.05 s (3H, $C^{27}H_3$), 1.05 m and 1.32 m (2H, 7-H), 1.13 m and 1.44 m (2H, 1-H), 1.19 m (1H,

18-H), 1.21 m and 1.40 m (2H, 6-H), 1.29 m and 1.48 m (2H, 2-H), 1.32 m and 1.48 m (2H, 21-H), 1.37 m (1H, 9-H), 1.39 m and 1.89 m (2H, 22-H), 1.41 m and 1.48 m (2H, 11-H), 1.44 br.s and 1.76 br.s (2H, 15-H), 1.66 m (1H, 20-H), 1.76 m and 1.05 m (2H, 12-H), 1.90 m (1H, 19-H), 2.23 d.d (1H, 13-H, J = 11.2, 9.6 Hz), 3.64 s (3H, OCH_3). ^{13}C NMR spectrum (100.62 MHz), δ_C, ppm: 14.53 (C^{27}), 14.67 (C^{29}), 15.77 (C^{24}), 15.88 (C^{25}), 19.63 (C^{30}), 21.03 (C^6), 21.42 (C^{26}), 22.75 (C^{11}), 22.96 (C^{23}), 26.59 (C^{12}), 26.90 (C^{15}), 29.64 (C^{20}), 29.72 (C^{16}), 31.99 (C^{22}), 33.68 (C^2), 34.11 (C^{21}), 36.86 (C^7), 37.26 (C^{10}), 38.17 (C^1), 39.58 (C^{13}), 40.61 (C^8), 42.57 (C^{18}), 44.14 (C^{14}), 47.30 (C^4), 48.86 (C^{19}), 49.65 (C^9), 51.15 (OCH_3), 54.94 (C^5), 56.96 (C^{17}), 176.77 (C^{28}), 217.96 (C^3). Found, %: C 79.36; H 9.98. $C_{31}H_{50}O_3$. Calculated, %: C 79.10; H 10.71.

Methyl 3,3-ethylenedioxylup-20(29)-en-28-oate (III). Compound **I**, 0.30 g (0.64 mmol), was dissolved in 50 ml of anhydrous benzene, 0.49 g (8 mmol) of anhydrous ethylene glycol and 0.02 g (0.10 mmol) of *p*-toluenesulfonic acid were added, and the mixture was heated for 3 h under reflux in a flask equipped with a Dean–Stark trap. The mixture was evaporated to 1/4 of the initial volume and extracted with diethyl ether (2 × 15 ml), the extract was washed with a saturated solution of sodium hydrogen carbonate (2× 15 ml), water, and a saturated solution of sodium chloride and dried over magnesium sulfate, the solvent was distilled off, and the residue was subjected to column chromatography on silica gel (5 g) using chloroform–methanol (50:1) as eluent. Yield 0.31 g (99%), colorless crystals, mp 156–158°C (from MeOH), $[\alpha]_D^{20}$ = –6.6° (c = 0.62, EtOAc). IR spectrum (KBr), ν, cm^{-1}: 2547, 1730, 1475. ^1H NMR spectrum (300.13 MHz), δ, ppm: 0.81 s (3H, $C^{23}H_3$), 0.85 s (3H, $C^{24}H_3$), 0.94 s (3H, $C^{26}H_3$), 0.96 s (3H, $C^{25}H_3$), 1.01 m and 1.92 m (2H, 16-H), 1.02 s (3H, $C^{27}H_3$), 1.04 m (1H, 5-H), 1.06 m and 1.32 m (2H, 7-H), 1.12 m and 1.53 m (2H, 1-H), 1.18 m (1H, 18-H), 1.22 m and 1.40 m (2H, 6-H), 1.31 m and 1.57 m (2H, 2-H), 1.32 m and 1.57 m (2H, 21-H), 1.36 m (1H, 9-H), 1.39 m and 1.89 m (2H, 22-H), 1.41 m and 1.57 m (2H, 11-H), 1.45 br.s and 1.76 br.s (2H, 15-H), 1.67 s (3H, $C^{30}H_3$), 1.72 m and 1.06 m (2H, 12-H), 2.19 d.d (1H, 13-H, J = 12.0, 8.0 Hz), 2.98 d.t (1H, 19-H, J = 12.0, 4.0 Hz), 3.65 s (3H, OCH_3), 3.81–3.93 m (4H, CH_2), 4.58 s and 4.72 s (2H, 29-H). ^{13}C NMR spectrum (75.48 MHz), δ_C, ppm: 14.73 (C^{27}), 15.86 (C^{24}), 18.36 (C^{25}), 19.28 (C^{23}), 19.89 (C^{30}), 20.82 (C^{26}), 22.71 (C^{11}), 22.84 (C^6), 25.38 (C^{12}), 26.82 (C^{15}), 29.58 (C^{16}), 30.52 (C^{22}), 32.07 (C^2), 34.11 (C^{21}), 36.87 (C^7), 36.99 (C^{10}), 37.06 (C^1), 38.18 (C^{13}), 40.63 (C^8), 42.03 (C^{18}), 42.34 (C^{14}), 46.90 (C^4), 49.36 (C^{19}), 50.19 (C^9), 51.14 (OCH_3), 53.30 (C^5), 56.46 (C^{17}), 64.64 (C^{32}), 64.75 (C^{31}), 109.49 (C^{29}), 113.21 (C^3), 150.47 (C^{20}), 176.53 (C^{28}). Found, %: C 77.60; H 10.07. $C_{33}H_{52}O_4$. Calculated, %: C 77.30; H 10.22.

Methyl 3,3-ethylenedioxylupan-28-oate (IV). Compound **II**, 0.20 g (0.42 mmol), was dissolved in 50 ml of anhydrous benzene, 0.33 g (5.25 mmol) of anhydrous ethylene glycol and 0.02 g (0.10 mmol) of *p*-toluenesulfonic acid were added, and the mixture was heated for 3 h under reflux in a flask equipped with a Dean–Stark trap and was then treated as described above in the synthesis of **III**. Yield 0.22 g (99%), colorless crystals, mp 206–208°C (from MeOH), $[\alpha]_D^{20}$ = –31.6° (c = 0.75, $CHCl_3$). IR spectrum (KBr), ν, cm^{-1}: 2935, 1720, 1450. ^1H NMR spectrum (400.13 MHz), δ, ppm: 0.74 d (3H, $C^{29}H_3$, J = 6.8 Hz), 0.84 d (3H, $C^{30}H_3$, J = 5.6 Hz), 0.86 s (3H, $C^{23}H_3$), 0.87 s (3H, $C^{24}H_3$), 0.87 s (3H, $C^{26}H_3$), 0.95 s (3H, $C^{25}H_3$), 1.01 m and 1.82 m (2H, 16-H), 1.02 s (3H, $C^{27}H_3$), 1.05 m (1H, 5-H), 1.07 m and 1.32 m (2H, 7-H), 1.12 m and 1.54 m (2H, 1-H), 1.17 m (1H, 18-H), 1.22 m and 1.40 m (2H, 6-H), 1.30 m and 1.57 m (2H, 2-H), 1.32 m and 1.57 m (2H, 21-H), 1.37 m and 1.82 m (2H, 22-H), 1.37 m (1H, 9-H), 1.41 m and 1.57 m (2H, 11-H), 1.46 br.s and 1.76 br.s (2H, 15-H), 1.67 m (1H, 20-H), 1.76 m and 1.07 m (2H, 12-H), 1.79 m (1H, 19-H), 2.19 d.d (1H, 13-H, J = 12.0, 4.0 Hz), 3.65 s (3H, OCH_3), 3.89–3.98 m (4H, CH_2). ^{13}C NMR spectrum (100.62 MHz), δ_C, ppm: 14.69 (C^{27}), 14.72 (C^{29}), 15.93 (C^{24}), 14.55 (C^{25}), 18.45 (C^{30}), 19.99 (C^6), 20.94 (C^{26}), 22.77 (C^{11}), 22.92 (C^{23}), 22.98 (C^{12}), 26.91 (C^{15}), 26.93 (C^{20}), 29.66 (C^{16}), 29.74 (C^{22}), 32.07 (C^2), 34.25 (C^{21}), 37.05 (C^7), 37.15 (C^{10}), 37.31 (C^1), 38.12 (C^{13}), 40.72 (C^8), 42.14 (C^{18}), 42.58 (C^{14}), 44.21 (C^4), 48.93 (C^{19}), 50.03 (C^9), 51.13 (OCH_3), 53.37 (C^5), 57.01 (C^{17}), 64.73 (C^{32}), 64.86 (C^{31}), 113.33 (C^3), 176.89 (C^{28}). Found, %: C 76.60; H 10.32. $C_{33}H_{54}O_4$. Calculated, %: C 76.99; H 10.57.

28,28-Ethano-3,3-ethylenedioxylup-20(29)-en-28-ol (V). A solution of 0.75 g (1.46 mmol) of compound **III** and 0.04 g (0.15 mmol) of titanium(IV) isopropoxide in 40 ml of anhydrous diethyl ether was added with stirring under argon at ~25°C to a solution of ethylmagnesium bromide prepared from 0.17 g (7.3 mmol) of metallic magnesium and 0.79 g (7.3 mmol) of ethyl bromide in 10 ml of anhydrous diethyl ether. The mixture was stirred for 0.5 h, treated with 50 ml of 5% aqueous sodium hydroxide, and ex-

tracted with diethyl ether (3×40 ml). The extract was washed with water and a saturated solution of sodium chloride, dried over sodium sulfate, and evaporated, and the residue was subjected to column chromatography on silica gel (5 g) using chloroform–methanol (50:1) as eluent. Yield 0.63 g (84%), colorless crystals, mp 112–114°C (from MeOH), $[\alpha]_D^{20}$ = –5.7° (*c* = 2.54, EtOAc). IR spectrum (KBr), ν, cm^{-1}: 3445, 2595, 1225. ^1H NMR spectrum (400.13 MHz), δ, ppm: 0.67 m (2H, 34-H), 0.80 s (3H, $C^{23}H_3$), 0.83 s (3H, $C^{24}H_3$), 0.83 m (2H, 33-H), 0.88 s (3H, $C^{26}H_3$), 0.98 s (3H, $C^{27}H_3$), 0.99 s (3H, $C^{25}H_3$), 1.01 m and 1.93 m (2H, 16-H), 1.04 m (1H, 5-H), 1.06 m and 1.72 m (2H, 12-H), 1.11 m and 1.53 m (2H, 1-H), 1.18 m (1H, 18-H), 1.23 m and 1.40 m (2H, 6-H), 1.31 m and 1.06 m (2H, 7-H), 1.32 m and 1.57 m (2H, 2-H), 1.37 m (1H, 9-H), 1.38 m and 1.90 m (2H, 22-H), 1.42 m and 1.55 m (2H, 11-H), 1.57 m and 1.32 m (2H, 21-H), 1.67 s (3H, $C^{30}H_3$), 1.75 br.s and 1.42 br.s (2H, 15-H), 2.31 d.d (1H, 13-H, *J* = 12.0, 3.0 Hz), 2.75 d.t (1H, 19-H, *J* = 11.0, 6.0 Hz), 3.86–3.92 m (4H, CH_2), 4.56 s and 4.71 s (2H, 29-H). ^{13}C NMR spectrum (100.62 MHz), δ_C, ppm: 14.96 q (C^{27}), 15.63 t (C^{34}), 15.80 t (C^{33}), 16.20 q (C^{24}), 16.27 q (C^{25}), 18.65 t (C^6), 18.88 q (C^{30}), 20.18 q (C^{26}), 21.21 t (C^{11}), 23.12 q (C^{23}), 25.57 t (C^{12}), 27.13 t (C^{15}), 29.21 t (C^{16}), 29.89 t (C^{22}), 31.67 t (C^2), 34.41 t (C^{21}), 34.67 t (C^7), 37.24 s (C^{10}), 37.37 t (C^1), 38.13 d (C^{13}), 38.41 s (C^8), 38.98 d (C^{18}), 41.37 s (C^{17}), 42.34 s (C^{14}), 42.94 s (C^4), 48.52 d (C^{19}), 50.43 d (C^9), 53.57 d (C^5), 60.17 s (C^{28}), 64.92 t (C^{32}), 65.03 t (C^{31}), 110.03 t (C^{29}), 113.54 s (C^3), 151.26 s (C^{20}). Found, %: C 79.86; H 10.71. $C_{34}H_{54}O_3$. Calculated, %: C 79.95; H 10.66.

28,28-Ethano-3,3-ethylenedioxylupan-28-ol (VI). A solution of 0.70 g (1.42 mmol) of compound **IV** and 0.04 g (0.15 mmol) of titanium(IV) isopropoxide in 40 ml of anhydrous diethyl ether was added with stirring under argon at ~25°C to a solution of ethylmagnesium bromide prepared from 0.17 g (7.3 mmol) of metallic magnesium and 0.79 g (7.3 mmol) of ethyl bromide in 10 ml of anhydrous diethyl ether. The mixture was stirred for 0.5 h and treated as described above in the synthesis of **V**. Yield 0.52 g (75%), colorless crystals, mp 176–177°C (from methanol), $[\alpha]_D^{20}$ = –32.36° (*c* = 1.26, $CHCl_3$). IR spectrum (KBr), ν, cm^{-1}: 1227, 2590, 3435. ^1H NMR spectrum (400.13 MHz), δ, ppm: 0.67 m (2H, 34-H), 0.77 d (3H, $C^{29}H_3$, *J* = 6.8 Hz), 0.81 s (3H, $C^{23}H_3$), 0.82 d (3H, $C^{30}H_3$, *J* = 8.8 Hz), 0.83 s (3H, $C^{24}H_3$), 0.85 m (2H, 33-H), 0.88 s (3H, $C^{26}H_3$), 0.96 s (3H, $C^{27}H_3$), 0.99 s (3H, $C^{25}H_3$), 1.01 m and 1.91 m (2H, 16-H), 1.05 m (1H, 5-H), 1.05 m and 1.31 m (2H, 7-H), 1.18 m and 1.53 m (2H, 1-H), 1.18 m (1H, 18-H), 1.23 m and 1.41 m (2H, 6-H), 1.34 m and 1.57 m (2H, 2-H), 1.34 m and 1.57 m (2H, 21-H), 1.37 m (1H, 9-H), 1.37 m and 1.87 m (2H, 22-H), 1.41 m and 1.55 m (2H, 11-H), 1.65 m (1H, 20-H), 1.71 m and 1.05 m (2H, 12-H), 1.75 br.s and 1.45 br.s (2H, 15-H), 1.89 m (1H, 19-H), 2.19 d.d (1H, 13-H, *J* = 12.0, 6.0 Hz), 3.88–3.95 m (4H, CH_2), ^{13}C NMR spectrum (100.62 MHz), δ_C, ppm: 14.07 (C^{27}), 14.57 (C^{29}), 14.81 (C^{34}), 15.94 (C^{33}), 16.22 (C^{24}), 17.45 (C^{25}), 18.43 (C^{30}), 20.00 (C^6), 21.05 (C^{26}), 22.93 (C^{11}), 23.09 (C^{23}), 23.89 (C^{12}), 26.92 (C^{15}), 28.72 (C^{20}), 29.69 (C^{16}), 29.91 (C^{22}), 33.62 (C^2), 34.30 (C^{21}), 37.00 (C^7), 37.13 (C^{10}), 37.82 (C^1), 40.28 (C^{13}), 41.24 (C^8), 42.13 (C^{18}), 43.00 (C^{17}), 45.31 (C^{14}), 48.62 (C^4), 49.52 (C^{19}), 49.97 (C^9), 53.31 (C^5), 59.83 (C^{28}), 64.72 (C^{32}), 64.83 (C^{31}), 113.32 (C^3). Found, %: C 79.94; H 11.06. $C_{34}H_{56}O_3$. Calculated, %: C 79.63; H 11.01.

This study was performed under financial support by the Russian Foundation for Basic Research (project no. 07-03-00438) and by the Presidium of the Russian Academy of Sciences (Program P-08 "Development of Methods of Organic Synthesis and Design of Compounds with Practically Important Properties" and "Fundamental Sciences for Medicine").

REFERENCES

1. Kulinkovich, O.G., *Chem. Rev*., 2003, vol. 103, p. 2597.
2. Bekish, A.V., Isakov, V.E., and Kulinkovich, O.G., *Tetrahedron Lett*., 2005, vol. 46, p. 6979.
3. Hiroya, K., Takahashi, T., Miura, N., Naganuma, A., and Sakamoto, T., *Bioorg. Med. Chem. Lett*., 2002, vol. 10, p. 3229.
4. Flekhter, O.B., Medvedeva, N.I., Karachurina, L.T., Baltina, L.A., Zarudii, F.S., Galin, F.Z., and Tolstikov, G.A., *Khim.-Farm. Zh*., 2002, vol. 36, p. 29.
5. Tolstikov, G.A., Goryaev, M.I., Kim, Kh.O., and Khegai, R.A., *Zh. Prikl. Khim*., 1967, vol. 40, p. 920.
6. Flekhter, O.B., Nigmatullina, L.R., Baltina, L.A., Karachurina, L.T., Galin, F.Z., Zarudii, F.S., Tolstikov, G.A., Boreko, E.I., Pavlova, N.I., Nikolaeva, S.N., and Savinova, O.V., *Khim.-Farm. Zh*., 2002, vol. 36, p. 26.
7. *Weygand–Hilgetag Organisch-chemische Experimentierkunst*, Hilgetag, G. and Martini, A., Eds., Leipzig: Johann Ambrosius Barth, 1964, 3rd ed. Translated under the title *Metody eksperimenta v organicheskoi khimii*, Moscow: Khimiya, 1968, p. 548.