An alternative synthesis of a sex pheromone of the dry bean beetle *Callosobruchus analis*

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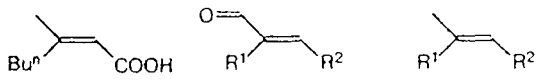
A formal total synthesis of 3-methylhept-2(*Z*)-enoic acid, a sex pheromone of the dry bean beetle *Callosobruchus analis*, was performed on the basis of a highly stereoselective version of the Peterson olefination of ketones developed previously by the authors.

Key words: stereoselective version of the Peterson olefination of ketones, insect pheromones, 3-methylhept-2(*Z*)-enoic acid, 3-phenylthiohexan-2-one, methyl and *tert*-butyl 3-methyl-4-phenylthiohept-2(*Z*)- and -2(*E*)-enoate, 3-methyl-4-phenylthiohept-2(*Z*)- and -2(*E*)-enols.

In the previous communication,[1] we described a highly stereoselective synthesis of the sex pheromone of the *Callosobruchus analis* beetle (**1**), based on the higher thermodynamic stability of the (*E*)-isomers of α,β-disubstituted acroleins (**2**) that we established previously and on the possibility of stereospecific transformation of the latter compounds into the corresponding (*Z*)-methylolefins (**3**).[2]

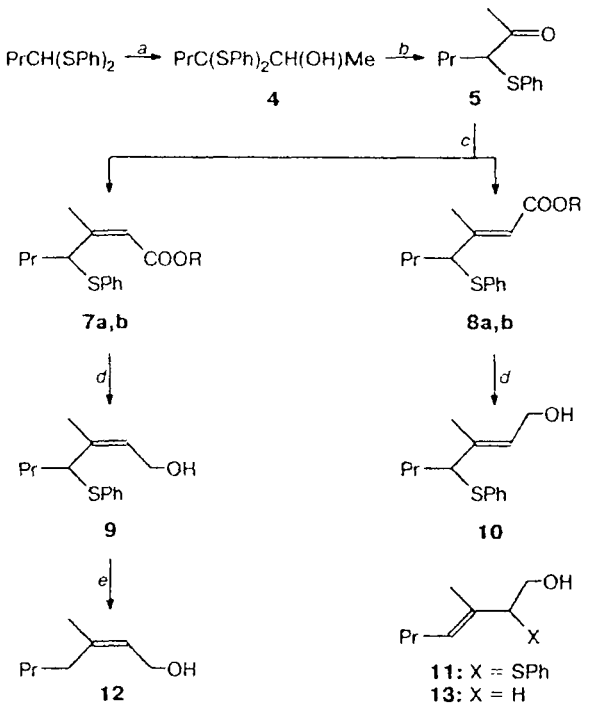


1 **2** **3**

The present communication is devoted to an alternative synthesis of **1**, which makes use of the highly stereoselective version of the Peterson olefination of ketones that we developed earlier.[3,4] In this case, 3-phenylthio ketone **5** and alkyl trimethylsilylacetates **6** serve as the starting compounds for the construction of **1**.

The previously unknown ketone **5** was prepared from butanal bis(phenylthio)acetal by the method developed by Warren *et al.*[5] Condensation of the latter with acetaldehyde yields alcohol **4**, which is smoothly cleaved on refluxing in benzene with ~0.4 equiv. of TsOH for 15 min to give the target ketone **5** (Scheme 1). Condensation of **5** with the methyl trimethylsilylacetate anion (generated from **6a** on treatment with LDA) under the conditions selected previously[4] gives ester **7a**, containing ~8% 2(*E*)-isomer **8a**, in 82% yield. The **7a** : **8a** ratio, which is in full agreement with previous results, can be easily determined from the data of ^{1}H NMR spectra based on the ratio of the integral intensities of the signals for C(3)Me (δ 1.93 and 2.22) and H-2 (δ 5.62 and 5.42) for (*Z*)- and (*E*)-isomers, respectively.[3,4]

Scheme 1



R = Me (**6a**–**8a**); Bu^t (**6b**–**8b**)

Reagents: *a*. Bu^{n}Li, MeCHO; *b*. TsOH/C_6H_6; *c*. Me_3SiCH_2COOR (**6a,b**)/LDA; *d*. $[AlH_3]$; *e*. Na/NH_3—hexane/dibenzo-18-crown-6.

As was to be expected,[3,4] the condensation of **5** with *tert*-butyl trimethylsilylacetate (**6b**) is less stereoselective and gives a mixture of esters **7b** and **8b** in a ratio of

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 7, pp. 1389–1392, July, 1999.

1066-5285/99/4807-1376 $22.00

82 : 18 (^{1}H NMR data). The reduction of the individual esters **7** and **8**, isolated by chromatography, to the corresponding phenylthio alcohols **9** and **10**, unlike the previously studied reduction of (*Z*,*Z*)- and (*E*,*Z*)-methyl (4-phenylthio)farnesoates,[3,4] was found to be a fairly complex process. This reaction occurs regio- and stereoselectively only when the reaction mixture is thoroughly protected from light (Al foil) at all the steps of preparation and isolation of the reaction products and when peroxide-free solvents are used. In this case, ester **7b** was converted into individual alcohol **9** in 83% yield, and ester **8b** was converted into isomeric alcohol **10**. The reduction of **7a** even under the above-specified conditions is accompanied by [1,3]-migration of the PhS group and gives a mixture of **9** and its isomer (**11**) in a ratio of 81 : 19 (^{1}H NMR data). Under typical reduction conditions, esters **7a,b** were converted only into alcohol **11**. The interconversions of alcohols **9**—**11** are considered in greater detail in another publication.[6] The desulfurization of **9** on treatment with a suspension of Na in a liquid NH_3—hexane mixture in the presence of dibenzo-18-crown-6 [7] gives alcohol **12**, which was purified from regioisomer **13** using HPLC. The preparation of **12** formally completes the synthesis of acid **1**, because oxidation of **12** to **1** was described in the previous publication.[1]

Experimental

UV spectra were recorded for alcoholic solutions on a Specord UV VIS spectrometer. IR spectra for KBr pellets or (for alcohols **9**—**13**) for solutions in CCl_4 were recorded on a Perkin—Elmer 577 instrument. The ^{1}H and ^{13}C NMR spectra were measured for solutions in $CDCl_3$ or C_6D_6 (for **9**—**12**) on Bruker AC-200 and Bruker DRX-500 spectrometers. Mass spectra (EI, 70 eV) were run on a Varian MAT CH-6 and Varian MAT 311A instruments (peaks with the intensity $I_{rel} > 10\%$ are given). Preparative flash chromatography was carried out on silica gel L (40—100 μm, Chemapol (Czechoslovakia)); TLC was performed on Silufol plates (Kavalier, Czechoslovakia) in benzene (A) or in CH_2Cl_2 (B). Analytical and preparative-scale HPLC were carried out on a column with Armofer Sil 10 (10 μm, 150×24 mm) using a RIDK-102 detector and an eluent flow rate of 6 mL min^{-1}.

The main solvents were purified as follows: THF and ether were kept over KOH, distilled successively from Na and $LiAlH_4$, refluxed under Ar with benzophenone Na ketyl until a persistent blue color appeared, and distilled directly into the reaction vessel. Hexane and benzene were distilled from Na.

The solution of LDA was prepared directly in the reactor from equivalent amounts of Pr^i_2NH and a 1.3—1.6 *M* solution of Bu^{n}Li in hexane, prepared by a standard procedure. The organic extracts were washed by a saturated solution of $MgSO_4$, dried with anhydrous $MgSO_4$, and concentrated *in vacuo* ("The usual workup").

3,3-Di(phenylthio)hexan-2-ol (4). A 1.6 *M* solution of Bu^{n}Li (25 mL, 40 mmol) in hexane was added dropwise to a solution of butanal diphenylthioacetal (7.4 g, 27 mmol), prepared by a previously described procedure,[5] and TMEDA (4.76 mL, 34 mmol) in 160 mL of THF vigorously stirred at 0 °C (under Ar). The mixture was stirred for 20 min, and a solution of acetaldehyde (2 mL) in 5 mL of THF was added dropwise at the same temperature. The reaction mixture was stirred for 10 min at 0 °C, slowly heated to ~20 °C, stirred for an additional 1 h, and poured into a mixture of 350 mL of H_2O and 100 mL of ether. The resulting mixture was stirred for 10 min and the layers were separated. The aqueous layer was extracted with ether. The subsequent usual workup of the combined extracts gave 7.64 g of an oily product, whose chromatography on 200 g of SiO_2 (gradient elution: benzene→CH_2Cl_2) gave 4.11 g (50%) of alcohol **4**, R_f 0.18 (A), 0.35 (B). IR, ν/cm^{-1}: 3560, 3470, 3080, 3060, 3000, 2960, 2935, 2880, 1470, 1440, 1385, 1270, 1120, 1090, 1070, 1040, 1030, 900, 700. ^{1}H NMR, δ: 0.78 (t, 3 H, Me, J = 7 Hz); 1.43 (d, 3 H, McCHOH, J = 6.2 Hz); 1.68 (m, 4 H, CH_2); 3.93 (q, 1 H, CHOH, J = 6.2 Hz); 7.50 (m, 10 H, Ph). ^{13}C NMR, δ*: 14.1 (C(6)); 17.9 (C(5)); 18.3 (C(1)); 38.1 (C(4)); 71.9 (C(2)); 75.3 (C(3)). MS, m/z (I_{rel} (%)): 273 (1.8), 209 (33), 99 (10), 44 (37), 32 (30), 28 (100).

3-Phenylthiohexan-2-one (5). A vigorously stirred solution of **4** (3.18 g, 10 mmol) in 250 mL of C_6H_6 was heated to reflux, $TsOH \cdot H_2O$ (0.7 g) was added, and the mixture was stirred for 15 min at 80 °C, quickly cooled to ~20 °C, and added to a saturated solution of Na_2CO_3. The mixture was stirred for 10 min and the layers were separated. The usual workup of the benzene layer afforded 2.22 g of a yellow nonviscous oil, whose chromatography on 150 g of SiO_2 (gradient elution: hexane→benzene) gave 1.46 g (70%) of ketone **5**, b.p. 103—105 °C (1 Torr). Found (%): C, 69.21; H, 7.77; S, 15.35. $C_{12}H_{16}OS$. Calculated (%): C, 69.18; H, 7.74; S, 15.39. IR, ν/cm^{-1}: 3080, 3060, 3020, 2960, 2930, 2875, 1710, 1585, 1570, 1480, 1465, 1440, 1380, 1360, 1240, 1195, 1185, 1160, 1105, 1090, 1070, 1030, 750, 700. ^{1}H NMR, δ: 0.95 (t, 3 H, Me, J = 7.2 Hz); 1.45 (m, 2 H, $H_2C(5)$); 1.75 (m, 2 H, $H_2C(4)$); 2.24 (s, 3 H, MeCO); 3.65 (t, 1 H, CHSPh, J = 7.4 Hz); 7.35 (m, 5 H, Ph). ^{13}C NMR, δ: 13.6 (C(6)); 20.4 and 26.2 (C(4), C(5)); 32.3 (C(1)); 57.4 (C(3)); 205.3 (C(2)). MS, m/z (I_{rel} (%)): 208 $[M]^+$ (16.5), 165 (25.7), 137 (11), 123 (89.5).

Methyl 3-methyl-4-phenylthiohept-2(*Z*)-enoate (7a) and its 2(*E*)-isomer (8a). A solution of **6a** (1.75 g, 12 mmol), prepared by a known procedure,[8] in 5 mL of THF was added dropwise to a solution of LDA (14 mmol) in 70 mL of a THF—hexane mixture (10 : 1) stirred at −78 °C (under Ar). The reaction mixture was stirred for 1.5 h at −78 °C, then a solution of **5** (1.34 g, 6.45 mmol) in 5 mL of THF was added dropwise at the same temperature. The reaction mixture was stirred for an additional 2 h, heated to ~4 °C over a period of 2.5 h, and allowed to stand at this temperature for ~16 h. The resulting orange solution was added to a mixture of ether and a saturated solution of NH_4Cl (150 mL, 1 : 1). The mixture was stirred for 15 min and the layers were separated. The aqueous layer was extracted with ether. The subsequent usual workup of the combined extracts gave 2.24 g of a yellow oily product, whose chromatography on 100 g of SiO_2 (gradient elution: petroleum ether→C_6H_6) gave 1.14 g (70%) of **7a** and 0.12 g of a mixture of **7a** and **8a** in a ratio of ~3 : 7. HPLC of this mixture (using 5% (v/v) of AcOEt in heptane) afforded 0.06 g (3.6%) of **8a**. Ester **7a**, b.p. 106—109 °C (0.09 Torr), retention time (R_t) 22 min. Found (%): C, 68.32; H, 7.75; S, 12.29. $C_{15}H_{20}O_2S$. Calculated (%): C, 68.14; H, 7.62; S, 12.13. UV, λ_{max}/nm (ε): 213 (19234), 245 (8674). IR,

* The ^{13}C NMR spectra of compounds **4**, **5**, and **7**—**11** also contain signals for Ph groups at 128—134 ppm.

ν/cm^{-1}: 3080, 3060, 3020, 2960, 2930, 2875, 1710, 1640, 1580, 1480, 1465, 1440, 1380, 1260, 1215, 1200, 1155, 1105, 1095, 1040, 1030, 925, 865, 750, 710, 698. ^{1}H NMR, δ: 0.95 (t, 3 H, Me, J = 7.2 Hz); 1.45 (m, 2 H, $H_2C(6)$); 1.70 (m, 2 H, $H_2C(5)$); 1.93 (s, 3 H, C(3)Me); 3.55 (s, 3 H, MeO); 5.62 (m, 2 H, HC(2), HC(4)); 7.35 (m, 5 H, Ph). ^{13}C NMR, δ: 13.8 (C(7)); 19.1 (C(3)Me); 20.6, 34.4 (C(5), C(6)); 47.5 (C(4)); 50.8 (MeO); 118.3 (C(2)); 157.5 (C(3)); 166.2 (C(1)). MS, m/z (I_{rel} (%)): 264 [M]$^+$ (27.2), 155 (68.1), 125 (17.8), 123 (27.8), 109 (16.0), 108 (11.6), 96 (14.7), 95 (65.4), 80 (22.2). Ester **8a**, R_t 28 min, R_f 0.43 (A). ^{1}H NMR, δ: 0.93 (t, 3 H, Me, J = 7.2 Hz); 1.40 (m, 2 H, $H_2C(6)$); 1.70 (m, 2 H, $H_2C(5)$); 2.22 (s, 3 H, C(3)Me); 3.60 (t, 1 H, HC(4), J = 7.5 Hz); 3.62 (s, 3 H, MeO); 5.42 (s, 1 H, HC(2)); 7.35 (m, 5 H, Ph). ^{13}C NMR, δ: 13.6 (C(7)); 14.4 (C(3)Me); 20.6, 34.2 (C(5), C(6)); 50.7 (MeO); 58.4 (C(4)); 117.0 (C(2)); 157.3 (C(3)); 166.4 (C(1)).

***tert*-Butyl 3-methyl-4-phenylthiohept-2(*Z*)-enoate (7b) and its 2(*E*)-isomer (8b)** were prepared in 82 : 18 ratio and in a total yield of 62% by condensation of **5** (1.66 g, 8 mmol) with **6b** (2.2 g, 11.7 mmol) under the conditions described for **7a** and **8a**. The mixture was separated by flash chromatography on SiO_2 (gradient elution: petroleum ether→C_6H_6). Ester **7b**, b.p. 130—132 °C (0.07 Torr) (bath). Found (%): C, 70.54; H, 8.64; S, 10.08. $C_{18}H_{26}O_2S$. Calculated (%): C, 70.54; H, 8.55; S, 10.46. UV, λ_{max}/nm (ε): 217 (22950), 243 (9792). IR, ν/cm^{-1}: 3080, 3055, 3000, 2970, 2930, 2870, 1705, 1635, 1560, 1480, 1440, 1390, 1370, 1260, 1220, 1180, 1145, 1030, 870, 750, 695. ^{1}H NMR, δ: 1.01 (t, 3 H, Me, J = 7.1 Hz); 1.40 (s, 9 H, Me_3C); 1.45 (m, 2 H, $H_2C(6)$); 1.70 (m, 2 H, $H_2C(5)$); 1.90 (s, 3 H, C(3)Me); 5.54 (s, 1 H, HC(2)); 5.67 (dd, 1 H, HC(4), J_1 = 7.9 Hz, J_2 = 7.0 Hz); 7.35 (m, 5 H, Ph). ^{13}C NMR, δ: 14.0 (C(7)); 19.0 (C(3)Me); 20.7, 34.7 (C(5), C(6)); 28.3 (Me_3C); 46.8 (C(4)); 79.3 (CMe_3); 120.8 (C(2)); 157.7 (C(3)); 164.9 (C(1)). MS, m/z (I_{rel} (%)): 306 [M]$^+$ (2.4), 250 (50.5), 233 (13.1), 142 (11.7), 141 (82.9), 123 (36.9), 110 (27.85), 109 (79.55), 97 (17.3), 96 (11.5), 95 (94.3), 93 (10.1), 78 (15.7), 77 (15.1), 67 (34.3), 65 (23.7), 59 (10.6), 56 (79.3). Ester **8b**, b.p. 140 °C (0.08 Torr) (bath). Found (%): C, 70.73; H, 8.61; S, 10.30. $C_{18}H_{26}O_2S$. Calculated (%): C, 70.54; H, 8.55; S, 10.46. UV, λ_{max}/nm (ε): 213 (17772), 265 (3231). IR, ν/cm^{-1}: 3080, 3060, 3000, 2980, 2960, 2935, 2880, 1710, 1690, 1480, 1470, 1455, 1440, 1390, 1370, 1300, 1240, 1190, 1150, 1110, 1090, 1070, 1050, 1030, 870, 700. ^{1}H NMR, δ: 0.93 (t, 3 H, Me, J = 7.2 Hz); 1.40 (m, 11 H, $H_2C(6)$ + Me_3C); 1.70 (m, 2 H, $H_2C(5)$); 2.18 (s, 3 H, C(3)Me); 3.55 (t, 1 H, HC(4), J = 7.5 Hz); 5.33 (s, 1 H, HC(2)); 7.35 (m, 5 H, Ph). ^{13}C NMR, δ: 13.9 (C(7)); 14.4 (C(3)Me); 20.9, 34.6 (C(5), C(6)); 28.3 (Me_3C); 58.8 (C(4)); 79.1 (CMe_3); 119.4 (C(2)); 154.9 (C(3)); 165.1 (C(1)). MS, m/z (I_{rel} (%)): 306 [M]$^+$ (6.6), 251 (18.5), 250 (53.7), 233 (28.5), 205 (28.15), 142 (10.8), 141 (58. 5), 137 (13.85), 123 (38.9), 110 (31.0), 109 (43.95), 108 (31.4), 99 (11.35), 97 (12.7), 96 (11.8), 95 (61.7), 82 (13.95), 80 (15.0), 69 (14.1), 67 (21.95), 65 (15.1), 57 (45.1), 56 (63.2).

3-Methyl-4-phenylthiohept-2(*Z*)-en-1-ol (9), its 2(*E*)-isomer (10), and 3-methyl-2-phenylthiohept-3(*E*)-en-1-ol (11). A solution of AlH_3 prepared by a known procedure[9] from $AlCl_3$ (0.144 g, 1.08 mmol), 7 mL of ether, and a 0.55 *M* ethereal solution of $LiAlH_4$ (6.6 mL, 3.6 mmol) was added dropwise to a solution of **7b** (0.75 g, 2.43 mmol) in 5 mL of ether stirred at −5 °C (under Ar) and thoroughly protected from light with Al foil. The mixture was stirred for 30 min at 0 °C, heated to ~20 °C, stirred for 1 h, and cooled again to 0 °C. A saturated solution of NH_4Cl (5 mL) was added dropwise, the mixture was stirred for 15 min, and the layers were separated. The aqueous layer was extracted with ether. The subsequent usual workup of the combined extracts gave 0.48 g (83%) of alcohol **9** as a colorless oil, R_f 0.10 (A), 0.31 (B). IR, ν/cm^{-1}: 3600, 3560, 3070, 3000, 2960, 2930, 2880, 1580, 1480, 1465, 1455, 1440, 1380, 1110, 1090, 1080, 1070, 1030, 995, 695. ^{1}H NMR, δ: 1.02 (t, 3 H, Me, J = 7.2 Hz); 1.15 (m, 2 H, $H_2C(6)$); 1.65 (m, 2 H, $H_2C(5)$); 1.98 (s, 3 H, C(3)Me); 3.86 (dd, 2 H, $H_2C(1)$, J_1 = 7.0 Hz, J_2 = 9.0 Hz); 4.30 (dd, 1 H, HC(4), J_1 = 6.2 Hz, J_2 = 9.0 Hz); 5.56 (t, 1 H, HC(2), J = 7.1 Hz); 7.40 (m, 5 H, Ph). ^{13}C NMR, δ: 14.8 (C(7)); 18.8 (C(3)Me); 21.9, 35.5 (C(5), C(6)); 51.3 (C(4)); 59.0 (C(1)); 135.2 (C(2)); 137.5 (C(3)). MS, m/z (I_{rel} (%)): 236 [M]$^+$ (15.9), 205 (37.2), 148 (10.7), 135 (13.2), 127 (36.4), 126 (84.8), 125 (12.25), 123 (23.3), 111 (17.6), 110 (66.5), 109 (86.65), 108 (101.4), 107 (15.0), 99 (10.9), 98 (17.6), 97 (63.8), 95 (23.0), 93 (10.6), 91 (18.0), 84 (18.3), 83 (35.9), 82 (86.2), 81 (20.7), 80 (35.4), 71 (64.1), 69 (62.7), 68 (15.3), 67 (41.8), 66 (29.3), 65 (34.2), 56 (20.9), 55 (106.0), 54 (18.8), 53 (67.6).

Reduction of ester **7a** (0.76 g) by AlH_3 carried out in a similar way gave a mixture of alcohols **9** and previously described[6] **11** in a ratio of 81 : 19 (^{1}H NMR data) and in a total yield of 80%. An attempt to separate this mixture by chromatography on SiO_2 gave 0.44 g (65%) of **11**, 0.06 g (9%) of **9**, and 0.1 g (15%) of alcohol **10**.

The reduction of **7b** carried out as described above but without protection from light gave alcohol **11** (yield 92%), identical (^{1}H and ^{13}C NMR, IR) to that described previously,[6] as the only reaction product.

The reduction of ester **8b** by AlH_3 in the dark afforded alcohol **10** (yield 92%) R_f 0.10 (A), 0.29 (B). IR, ν/cm^{-1}: 3600, 3080, 3060, 3000, 2960, 2935, 2880, 1580, 1480, 1465, 1455, 1440, 1380, 1105, 1090, 1070, 1030, 990, 950, 700. ^{1}H NMR, δ: 0.83 (t, 3 H, Me, J = 7.3 Hz); 0.95 (m, 2 H, $H_2C(6)$); 1.35 (m, 2 H, $H_2C(5)$); 1.59 (s, 3 H, C(3)Me); 3.59 (dd, 2 H, $H_2C(4)$, J_1 = 6.8 Hz, J_2 = 8.3 Hz); 3.85 (d, 2 H, $H_2C(1)$, J = 6.6 Hz); 5.20 (t, 1 H, HC(2), J = 6.6 Hz); 7.40 (m, 5 H, Ph). ^{13}C NMR, δ: 12.7 (C(7)); 14.8 (C(3)Me); 21.9, 35.7 (C(5), C(6)); 58.9 (C(4)); 59.7 (C(1)); 134.0 (C(2)); 137.2 (C(3)). MS, m/z (I_{rel} (%)): 236 [M]$^+$ (8.5), 218 (1.1), 205 (19.2), 127 (15.1), 126 (40.2), 110 (28.15), 109 (48.1), 108 (62.8), 97 (35.9), 95 (10.9), 83 (18.2), 82 (55.4), 80 (19.8), 76 (19.2), 71 (38.4), 69 (26.7), 67 (27.6), 66 (13.8), 65 (23.6), 58 (15.5), 57 (47.1), 55 (54.7) 54 (14.7), 53 (51.15), 52 (23.6), 51 (16.5).

3-Methylhept-2(*Z*)-en-1-ol (12). A solution of **9** (0.66 g) in 3 mL of ether was added dropwise to a suspension of Na (0.5 g) in a mixture of 100 mL of liquid NH_3 and 50 mL of hexane containing dibenzo-18-crown-6 (140 mg)[7] and stirred at −78 °C. The reaction mixture was stirred for 40 min at −78 °C, solid NH_4Cl was added until the mixture became colorless, and the NH_3 was allowed to evaporate. The usual workup of the residue gave 0.43 g of a reaction product, which according to ^{13}C NMR, was a mixture of alcohol **12** and its regioisomer **13** [6] (^{1}H NMR, δ: 0.83 (t, 3 H, $H_3C(7)$, J = 7 Hz); 1.30 (m, 2 H, $H_2C(6)$); 1.46 (s, 3 H, C(3)Me); 1.90 (dt, 2 H, $H_2C(5)$, J_1 = J_2 = 7.2 Hz); 2.10 (t, 2 H, $H_2C(2)$, J = 7 Hz); 3.5 (t, 2 H, $H_2C(1)$, J = 7 Hz); 5.20 (t, 1 H, HC(4), J = 7.2 Hz)) in a ratio of 92 : 8. Individual **12** was isolated by HPLC, yield 70%; the physicochemical characteristics of the sample obtained were fully identical to those of the sample described previously.[1]

This work was financially supported by the Russian Foundation for Basic Research (Project No. 94-03-08904), the State Foundation for the Support of Leading Scientific Schools (grant 96-15-97461), and the International Science Foundation (ISF) (grant NGO 000).

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Received January 11, 1999