An Alternative Synthesis of (+)-1,7-Dimethyl-4-*endo*-isopropyl-8-*syn*-(2-hydroxyethyl)bicyclo[3.2.1]oct-6-ene, the Key Intermediate for the Synthesis of (+)-Sativene and (+)-Cyclosativene[1]

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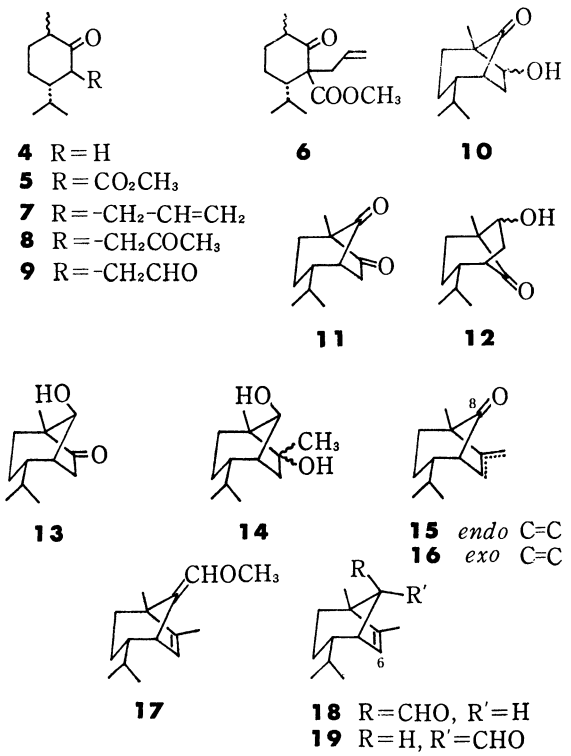
The title compound, the key intermediate for the synthesis of (+)-sativene and (+)-cyclosativene, has been synthesized through the alternative route starting with (−)-carvone.

Recently Piers and co-workers[2] reported the stereoselective total synthesis of (+)-sativene (**1**)[3,4] and (+)-cyclosativene (**2**)[5,6] through the common intermediate, (+)-1,7-dimethyl-4-*endo*-isopropyl-8-*syn*-(2-hydroxyethyl)bicyclo[3.2.1]oct-6-ene (**3**). We have independently investigated an approach to the synthesis of these sesquiterpenes in the optically active forms using the compound **3** as the key intermediate and wish to report our synthetic route of **3** starting from (−)-carvone in this paper.

1 **2** **3**

Introduction of a suitable substituent at the α-methylene of carvomenthone (**4**) for the construction of a five-membered ring was accomplished *via* alkylation of a β-ketoester **5**. Thus, the reaction of **4**, prepared from (−)-carvone[7], with sodium hydride–dimethyl carbonate in dioxane gave **5**, which was allylated to **6** in an excellent overall yield. Demethoxycarbonylation of **6** was achieved by heating in dimethyl sulfoxide in the presence of an equimolar amount of sodium cyanide[8] to produce **7** in 92% yield. It should be noted that decarboxylation of **6** under the usual alkaline conditions was unsatisfactory, resulting in opening of the ring.

Attempts to transform directly the allylic side chain into the acetonyl one, **7**→**8**, by employing oxymercuration-demercuration procedure did not give clear result, even if the various derivatives (alcohol, acetate, or tetrahydropyranyl ether) were used. The modified Lemieux-Johnson oxidation[9] of **7** produced an aldehyde **9** which, without purification, was cyclized to a mixture of bicyclic aldols **10** (50% from **7**) by treating with 20% potassium hydroxide–methanol. The products **10**, which were separable into the crystalline, mp 80—82 °C, and oily isomers by chromatography on silica gel, are epimeric as shown from the close resemblance of the physical properties (IR, NMR, and ORD) and the conversion into a diketone **11**. But the actual configuration of the hydroxyl group in each epimer has not been determined at present and, fortunately, this ambiguity is not essential to our purpose. Then, a choice between the *endo* and *exo* isopropyl structures **10** and **12** can be made by the following consideration and finally on the basis of ORD of the compound **13**. The thermodynamically more stable carbon-framework, having the equatorial isopropyl group on the chair



4 R=H
5 R=CO_2CH_3
7 R=$-CH_2-CH=CH_2$
8 R=$-CH_2COCH_3$
9 R=$-CH_2CHO$

6 **10** **11** **12** **13** **14** **15** *endo* C=C **16** *exo* C=C **17**

18 R=CHO, R′=H
19 R=H, R′=CHO

cyclohexane ring as **10**, would be formed preferentially in such an equilibrium (reversible) reaction.

The mixture **10** was transformed into the isomeric aldol **13** in 50% overall yield by sequential application of the following procedures: (1) acetylation of the hydroxyl group in **10**, (2) reduction of the ketone with sodium borohydride, (3) protection of the newly formed hydroxyl group with dihydropyrane, (4) reduction of the acetate function with lithium aluminum hydride, (5) oxidation of the resulting alcohol with Collins' reagent[10], and finally (6) hydrolytic removal of the pyranyl ether group. Aldol **13** thus obtained was a single crystalline compound, mp 80—81 °C, which shows the negative Cotton effect in ORD. This ORD property establishes the assigned absolute stereochemistry **13** having α (*endo*-equatorial) isopropyl group at C-4 as shown.[11] The configuration of the hydroxyl group was tentatively assigned to be *anti* from the fact that hydride ion would be expected to attack the bridge ketone of **10** from the less hindered side (five-membered ring side) in such a ring system. Treatment of **13** with excess methyl lithium gave a methylated diol **14**. Oxidation of **14** with Collins' reagent followed by dehydration of the resulting ketol with phosphorous oxychloride–pyridine produced a mixture

of the *endo* and *exo* olefinic ketones **15** and **16** in roughly equal amounts, and iodine-catalyzed isomerization of the mixture in refluxing xylene[12] gave exclusively the desired isomer **15**.

Elongation of a two-carbon unit with the appropriate configuration, being *syn* with respect to the five-membered ring, at the C-8 position of **15** was accomplished by applying doubly the Wittig reaction using methoxymethylenetriphenylphosphorane. Reaction of **15** with methoxymethylenetriphenylphosphorane in dimethyl sulfoxide[13] gave **17** which was then hydrolyzed by treating with perchloric acid in ether to give a single aldehyde **18**. This substance must have the thermodynamically unfavorable *anti* (axial on the six-membered ring) formyl group since upon treatment with potassium carbonate in methanol it was changed exclusively to the isomeric *syn* aldehyde **19**. The assignment of the configuration of the formyl group was further supported by NMR spectrum; the signal of the olefinic proton at C-6 in **19** appeared at a higher field (0.1 ppm) than that in **18**, presumably due to the anisotropic effect of the *syn* formyl group. Again, **19** was subjected to the Wittig reaction followed by hydrolysis in the same manner as in the case of **15** to yield a homologous aldehyde, which was reduced to the desired alcohol **3** with lithium aluminum hydride.

The pure sample of **3** collected by preparative glpc, $[\alpha]_D = +133°$, was identical with the sample prepared by Piers, *et al.*[2]

Experimental

All melting points and boiling points are uncorrected. IR spectra were taken on a Hitachi EPI-S2 or a G2 spectrometer. NMR spectra were obtained on a JEOL Model C-60HL instrument using TMS as an internal standard and CCl_4 as the solvent unless otherwise indicated, and chemical shifts are given in δ and coupling constants in Hz. Mass spectra were taken on a Hitachi RMU-6D spectrometer. Specific rotations and ORD curves were recorded on a JASCO Model ORD/UV-5 spectrometer. Glpc analyses were performed on a JEOL Model JGC-1100 instrument. Elemental analyses were performed in the microanalytical laboratory of this institute.

*Methoxycarbonylation of (−)-Carvomenthone (**4**).* To a stirred mixture of sodium hydride (12.5 g, 0.52 mol) and dimethyl carbonate (50 g, 0.55 mol) in dry dioxane (100 ml) was added dropwise over 5 hr a solution of (−)-carvomenthone (**4**) (42 g, 0.27 mol) in dry dioxane (100 ml) at 85—95 °C under nitrogen. Stirring was continued at the same temperature overnight; the mixture turned to wine-red. The cooled mixture was poured into ice-water, acidified with dilute hydrochloric acid to pH 3, and extracted with ether three times. The combined extracts were washed with water twice and then saturated salt solution, and dried over anhydrous sodium sulfate. After removal of the solvent, distillation of the residual liquid gave 50 g (86%) of 3-isopropyl-2-methoxycarbonyl-6-methylcyclohexanone (**5**) as colorless liquid, bp 96—108 °C/3 mmHg, which gave a purple color with alcoholic ferric chloride. IR (neat) 1745, 1710 cm^{-1}; NMR 0.90—1.10 (m, 9H), 1.20—2.50 (m, 8H), 3.70 (s, 3H).

*2-Allyl-3-isopropyl-2-methoxycarbonyl-6-methylcyclohexanone (**6**).* To a suspension of sodium hydride (6.5 g, 0.25 mol) in dry dioxane (100 ml) was added dropwise over 1 hr a solution of **5** (49 g, 0.23 mol) in dry dioxane (100 ml) at 85—90 °C under nitrogen. After the evolution of hydrogen ceased, freshly distilled allyl bromide (30 ml, 0.35 mol) was added over a period of 1 hr, and the mixture was stirred at 80—85 °C overnight. After cooling to room temperature, the reaction mixture was poured into ice-water, neutralized with dilute hydrochloric acid to Congo Red, and extracted with ether three times. The combined extracts were washed with water and saturated salt solution, dried over anhydrous sodium sulfate, and freed from the ether. Distillation of the residual liquid gave 51.3 g (88%) of **6** as colorless oil, bp 110—120 °C/3 mmHg; IR (neat) 3070, 1740, 1715, 1645, 928, 880 cm^{-1}. Found: C, 71.99; H, 9.72%. Calcd for $C_{15}H_{24}O_3$: C, 71.39; H, 9.59%.

*2-Allyl-3-isopropyl-6-methylcyclohexanone (**7**).* Finely powdered sodium cyanide (10 g, 0.2 mol) was added to a solution of **6** (51 g, 0.2 mol) in dimethyl sulfoxide (200 ml), and the mixture was heated at 160 °C for 2 hr with vigorous stirring. The cooled reaction mixture was poured into ice-water and extracted with petroleum ether twice. The extracts were washed thoroughly with water and then saturated salt solution, dried over anhydrous sodium sulfate, and freed from the solvent. Distillation of the residual liquid gave 36 g (92%) of **7** as colorless oil, bp 80—90 °C/0.6 mmHg: IR (neat) 3070, 1705, 1640, 920 cm^{-1}; NMR 0.90—1.00 (m, 9H), 1.50—1.80 (m, 8H), 2.20—2.40 (m, 4H), 4.80—5.10 (m, 3H). Found: C, 80.53; H, 11.38%. Calcd for $C_{13}H_{22}O$: C, 80.35; H, 11.41%.

*7-Hydroxy-4-isopropyl-1-methylbicyclo[3.2.1]octan-8-one (**10**).* To a mechanically stirred solution of **7** (35 g, 0.17 mol) in dioxane (1000 ml) was added osmium tetroxide (1 g, 4 mmol) in the dark with cooling in an ice-bath and then a solution of sodium metaperiodate (50 g, 0.23 mol) in water (1.5 l) over a period of 6 hr. The stirring was continued in the dark overnight. Inorganic salt was removed by filtration and the filtrate was thoroughly extracted with ether. The combined extracts were washed with 10% sodium sulfide solution until the water layer became colorless, and then with water and saturated salt solution. Evaporation of the ether at room temperature under reduced pressure gave 32 g (91%) of the keto aldehyde **9**: IR ($CHCl_3$) 2700, 1715, 1120, 870 cm^{-1}, which was subjected to aldol cyclization without further purification.

To a solution of potassium hydroxide (225 g) in methanol (2.5 l) was added dropwise a solution of **9** (32 g) in methanol (500 ml) with cooling in an ice-bath. The mixture was allowed to stir at room temperature for 36 hr, neutralized with 10% hydrochloric acid, and thoroughly extracted with ether. After removal of the solvent, distillation of the residue gave 15 g of an epimeric mixture of **10**, bp 130—140 °C/0.25 mmHg, which partially crystallized on standing. Crystalline substance was collected and recrystallized from *n*-hexane–ether to afford 5.23 g of an epimer of **10**, mp 80—81 °C. The residual part was chromatographed on silica gel (500 g) using 3 : 1 petroleum ether–ether as eluent to give additional crystals (4.77 g) and a colorless oily epimer (7.23 g). Total yield was 52%. Crystalline epimer: IR ($CHCl_3$) 3600, 3450, 2900, 1735, 1200, 1040 cm^{-1}; NMR ($CDCl_3$) 0.90—1.00 (m, 9H), 1.30—1.90 (m, 7H), 2.20—2.60 (m, 2H), 2.50 (s, 1H, exchangeable with D_2O), 4.08 (m, 1H, changed to q by D_2O addition); ORD (EtOH) $[\phi]_{309}$ $+8.24\times10^3$, $[\phi]_{272}$ -9.02×10^3. Found: C, 73.29; H, 10.33%. Calcd for $C_{12}H_{20}O_2$: C, 73.43; H, 10.27%. Oily epimer: IR ($CHCl_3$) 3600, 3400, 2900, 1735 cm^{-1}; NMR 0.90—1.00 (m, 9H), 1.40—2.50 (m, 9H), 3.65 (s, 1H), 4.00 (m, 1H); ORD (EtOH) $[\phi]_{309}$ $+6.06\times10^3$, $[\phi]_{272}$ -8.20×10^3. Found: C, 72.69 H, 10.22%. MS *m/e* 196

(M^+). Calcd mol wt 196.

Jones oxidation of each epimer in the usual manner gave the same diketone **11** as a viscous oil: IR (CCl_4) 2900, 1770, 1745 (sh), 1730, 1045 cm^{-1}.

*4-Isopropyl-8-hydroxy-1-methylbicyclo[3.2.1]octan-7-one (**13**).* A solution of **10** (7.53 g) in pyridine (30 ml) was treated with acetic anhydride (10 ml) at room temperature for 15 hr. Usual work-up gave 9.24 g (100%) of a mixture of the keto acetate. Analytical sample was purified by evaporative distillation, bp 130 °C (bath temp)/2 mmHg. IR (neat) 1740, 1250, 1030 cm^{-1}; NMR 1.99 (s, 3H), 5.00 (m, 1H). Found: C, 70.49; H, 9.68%. Calcd for $C_{14}H_{22}O_3$: C, 70.55; H, 9.31%.

To a solution of the acetate (9.2 g) in methanol (120 ml) was added sodium borohydride (1.48 g) and one pellet of potassium hydroxide, and the mixture was stirred at room temperature for 2 hr. Usual work-up gave 9.03 g (97%) of a mixture of the oily hydroxy acetate. From the pure oily epimer of **10** the crystalline hydroxy acetate was obtained. Recrystallized sample from *n*-hexane had mp 32 °C. IR ($CHCl_3$) 3550, 3400, 1710, 1250, 1100 cm^{-1}; NMR 2.00 (s, 3H), 3.85 (d, J=6.0, 1H), 4.77 (q, J=3.8 and 8.3, 1H). Found: C, 70.18; H, 9.93%. Calcd for $C_{14}H_{24}O_3$: C, 69.96; H, 10.07%.

A solution of the hydroxy acetate (5.18 g) and dihydropyrane (5 ml) in dry ether (100 ml) containing a catalytic amount of *p*-toluenesulfonic acid was allowed to stir at room temperature for 3 hr. Usual work-up gave 8.2 g of the pyranyl ether. IR ($CHCl_3$) 1720, 1260, 1125, 1020, 970 cm^{-1}; NMR 1.60 (m, 6H), 2.00 (s, 3H), 4.60 (m, 2H).

To a cold suspension of lithium aluminum hydride (1.10 g) in anhydrous ether (150 ml) was added dropwise a solution of the pyranyl ether (8.30 g) in anhydrous ether (20 ml), and the mixture was stirred at room temperature for 4 hr. Usual work-up gave 7.33 g (88%) of the alcohol. IR ($CHCl_3$) 3600, 3400, 1120, 1070, 970 cm^{-1}; NMR 1.70 (m, 6H), 3.30—3.37 (m, 4H), 4.70 (m, 1H).

A solution of the above alcohol (7.33 g) in dichloromethane (500 ml) was treated with the Collins' reagent (31.0 g) at room temperature for 30 min. Usual work-up gave 7.03 g (96%) of the ketone. IR ($CHCl_3$) 1735, 1124, 1075, 1030, 970 cm^{-1}; NMR 1.70 (m, 6H), 3.50—3.90 (m, 3H), 4.75 (m, 1H).

A solution of the above product (212 mg) in 20% aqueous dioxane (5 ml) containing a catalytic amount of *p*-toluenesulfonic acid was heated at 70 °C for 1 hr. The mixture was diluted with water and extracted with ether twice. The combined extracts were washed with water and saturated salt solution, and evaporated to give 105 mg (71%) of crystalline **13**, mp 80—81 °C, recrystallized from *n*-hexane. IR (CCl_4) 3600, 3400, 1740 cm^{-1}; NMR 0.80—1.00 (m, 9H), 1.10—2.20 (m, 8H), 2.30—2.70 (m, 2H, 1H exchangeable with D_2O) 3.85 (d, J=6.0, 1H). ORD (EtOH) $[\phi]_{316}$ -1.99×10^3, $[\phi]_{275}$ $+2.50\times10^3$. Found: C, 73.37; H, 10.35%. Calcd for $C_{12}H_{20}O_2$: C, 73.43; H, 10.27%.

*7,8-Dihydroxy-1,7-dimethyl-4-isopropylbicyclo[3.2.1]octane (**14**).* A solution of **13** (105 mg) in ether (2 ml) was added dropwise to an ethereal solution of methyl lithium, prepared from lithium (400 mg) and methyl iodide (3 ml) in ether (20 ml), with cooling in an ice-bath, and the mixture was stirred overnight at room temperature under nitrogen. The mixture was carefully poured into ice-water and the water layer was extracted with two 30 ml portions of ether. Evaporation of the ether and recrystallization of the residual crystals from *n*-hexane–ether gave 60 mg (53%) of **14**, mp 116—117 °C: IR ($CHCl_3$) 3600, 3400, 1050 cm^{-1}; NMR ($CDCl_3$) 0.75—0.95 (m, 9H), 1.15 (s, 3H), 1.20—1.75 (m, 8H), 2.17 (m, 1H), 3.65 (d, J=6.0, 1H). Found: C, 73.72; H, 11.95%. Calcd for $C_{13}H_{24}O_2$: C, 73.53; H, 11.39%.

*1,7-Dimethyl-4-isopropylbicyclo[3.2.1]oct-6-en-8-one (**15**).* To a stirred slurry of the Collins' reagent (1.0 g) in dichloromethane (10 ml) was added a solution of **14** (123 mg) in dichloromethane (10 ml) at room temperature, and the mixture was stirred for 15 min. After the usual work-up, recrystallization of the residue from *n*-hexane gave 110 mg (90%) of the keto alcohol, mp 124 °C. IR (CCl_4) 3600, 3400, 1730 cm^{-1}; NMR 0.80—1.00 (m, 9H), 1.12 (s, 3H), 1.50—2.50 (m, 10H). Found: C, 74.10; H, 10.30%. Calcd for $C_{13}H_{22}O_2$: C, 74.24; H, 10.54%.

To a solution of the above keto alcohol (64 mg) in pyridine (0.5 ml) was added phosphorous oxychloride (0.04 ml), and the resulting mixture was heated gradually in an oil-bath until the bath temperature reached to 90 °C (30 min) and maintained at that temperature for 15 min. After cooling, the mixture was poured into ice-water in a separatory funnel with the aid of ether and the water layer was extracted with ether. The combined ether layers were successively washed with dilute hydrochloric acid, sodium bicarbonate solution, water, and saturated salt solution. Evaporation of the ether gave 46 mg of a *ca.* 1 : 1 mixture of **15** and the *exo*-isomer **16**, as indicated by glpc on a column of Carbowax 20M (2 m, 155 °C, He 1.4 kg/cm^2, retention times: 5.4 min and 6.3 min). IR (neat) 1755, 1655 cm^{-1}; NMR 1.70 (br. s), 4.90 (m), 5.75 (m).

A solution of the above mixture (550 mg) and a catalytic amount of iodine in anhydrous xylene (20 ml) was heated under reflux for 8 hr under nitrogen. The cooled reaction mixture was successively washed with dilute sodium thiosulfate solution, water, and saturated salt solution. Evaporation of the xylene followed by evaporative distillation of the residual liquid gave 470 mg of pure **15**, bp 100 °C (bath temp)/2 mmHg. IR (neat) 1755 cm^{-1}; NMR 0.80—1.20 (m, 9H), 1.75 (d, J=1.5, 3H), 1.20—2.40 (m, 6H), 2.75 (m, 1H), 5.75 (m, 1H). Found: C, 80.93; H, 10.29%. Calcd for $C_{13}H_{20}O$: C, 81.20; H, 10.48.%

*Wittig Reaction of **15** and Hydrolysis of the Product **17**.* A solution of methoxymethylenetriphenylphosphorane in dimethyl sulfoxide was prepared according to the literature using sodium hydride (0.66 g) and methoxymethyltriphenylphosphonium chloride (9.25 g) in dimethyl sulfoxide (57 ml). To this red solution was added a solution of **15** (1.10 g) in dimethyl sulfoxide (14 ml); and the resulting mixture was stirred at 53 °C overnight under nitrogen. Then, the reaction mixture was poured into ice-water and extracted with petroleum ether three times. The combined extracts were washed with 50% aqueous dimethyl sulfoxide, water (five times), and saturated salt solution, and freed from the solvent.

The crude oily product thus obtained was dissolved in ether and a few drops of 30% hydrogen peroxide was added with cooling in an ice-bath. After stirring for 30 min at room temperature, a large amount of petroleum ether was added and the resulting organic layer was passed through a short column of neutral alumina (Woelm V). Evaporation of the solvent gave 1.03 g (87%) of the oily methoxymethylene compound **17**. IR (CCl_4) 3060, 1705, 1430 cm^{-1}; NMR 1.60 (m, 3H), 2.70 (m, 1H), 3.42 (s, 3H), 5.35 (m, 1H), 5.50 (s, 1H). MS *m/e* 222 (M^+). Calcd for $C_{15}H_{24}O$: mol wt 222.

Compound **17** (213 mg) was dissolved in ether saturated with perchloric acid (5 ml). The reaction mixture was allowed to stand for 2 hr at room temperature and then washed with dilute sodium bicarbonate solution, water, and finally saturated salt solution. Evaporation of the ether gave

200 mg (100%) of the *anti*-aldehyde **18**: IR (CCl_4) 2740, 1725 cm^{-1}; NMR 1.65 (m, 3H), 5.50 (m, 1H), 10.10 (d, J=6.0, 1H).

Isomerization of ***18*** *to the* syn-*Isomer* ***19***. A solution of **18** (150 mg) in ether (3 ml) was added to a saturated methanolic solution of potassium carbonate (2 ml), and the mixture was stirred overnight at room temperature. Then, the mixture was diluted with water and extracted with ether. The extract was washed with water and saturated salt solution, dried, and freed from the ether, giving 128 mg (85%) of an oil, which consisted mainly of the desired compound, 1,7-dimethyl-8-*syn*-formyl-4-isopropylbicyclo[3.2.1]oct-6-ene (**19**): IR (neat) 2700, 1718, 1436, 1120 cm^{-1}; NMR 1.65 (br. s, 3H), 2.75 (m. 1H), 5.40 (m, 1H), 9.50 (d, J=4.0, 1H).

*(+)-1,7-Dimethyl-4-*endo-*isopropyl-8-*syn-*(2-hydroxyethyl)bicyclo[3.2.1]oct-7-ene* (***3***). A solution of **19** (430 mg) in dimethyl sulfoxide (10 ml) was treated with a solution of an excess methoxymethylenetriphenylphosphorane (4 equiv.) in dimethyl sulfoxide (10 ml) at room temperature overnight under nitrogen. The mixture was worked up as before to give 403 mg (87%) of the enol ether as a colorless oil: IR (CCl_4) 3050, 1650, 1430, 1100 cm^{-1}; NMR 1.62 (br. s, 3H), 3.50 (s, 3H), 5.25 (m, 1H), 5.80 (q, J=12.0 and 6.0, 1H), 6.25 (d, J=12.0, 1H).

A solution of the above product (112 mg) in ether (5 ml) was added to cold ether saturated with perchloric acid (5 ml). After stirring for 30 min at room temperature, the mixture was successively washed with dilute sodium bicarbonate solution, water and saturated salt solution, dried over anhydrous sodium sulfate, and freed from the ether, giving 106 mg (89%) of the aldehyde: IR ($CHCl_3$) 2700, 1725, 1450, 1370, 1120 cm^{-1}.

To a stirred suspension of lithium aluminum hydride (21 mg, 4 equiv) in anhydrous ether (5 ml) was added a solution of the above aldehyde (106 mg) in anhydrous ether (3 ml) at 0 °C, and the mixture was stirred for 1 hr at room temperature. Usual work-up gave 101 mg of an oil, which consisted of two components as indicated by glpc analysis (20% Carbowax 20M, 2 m×3 mm, 160 °C, He 0.95 kg/cm^2): the major peak with 77% of area at 7.8 min and the minor one with 23% of area at 9.4 min. The collected sample of the major product was identical with the authentic sample of **3**. IR (CCl_4) 3620, 3040, 1460, 1440, 1380, 1365, 1050 cm^{-1}; NMR 0.80—0.95 (m, 9H), 1.60 (br. s, 3H), 1.00—2.00 (m, 10H, 1H exchangeable with D_2O), 2.55 (m, 1H), 3.65 (d, J=7.5, 2H), 5.45 (m, 1H). $[\alpha]_D$=+133° (c, 1.52×10^{-2}, EtOH). Found: C, 80.46; H, 11.55%. Calcd for $C_{15}H_{26}O$: C, 81.02; H, 11.79%.

Minor product was assigned to be 8-*anti*-isomer from the following spectral data: IR (CCl_4) 3620, 3040, 1460, 1440, 1380, 1120, 1045, 1010 cm^{-1}, NMR 0.75—0.95 (m, 9H), 1.55 (br. s, 3H), 1.00—1.90 (m, 9H), 2.45 (m, 1H), 3.50 (m, 3H), 5.20 (m, 1H).

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