Peptides (XXXII)-(XL) were deblocked by treatment with a 2.16 N solution of HCl in AcOH at 0-5°C for 1-2 h, giving, respectively, compounds (I)-(IX). The final purification of the deblocked peptides (I)-(IX) was achieved by gel chromatography on Sephadex LH-20 with elution by the solvent system MeOH–H_2O–0.1 N AcOH (1:0.5:0.2). Results of the amino acid analysis of peptides (I)-(IX): (I) – Phe 2.93 (3), Gly 1.02 (1), His 0.89 (1), Met 0.96 (1); (II) – Glx 1.01 (1), Phe 2.94 (3), Gly 1.01 (1), His 0.91 (1), Met 0.94 (1); (III) – Glx 1.99 (2), Phe 2.97 (3); Gly 0.99 (1), His 0.92 (1), Met 0.92 (1); (IV) – Phe 2.97 (3), Gly 0.99 (1), His 0.92 (1), Met 0.92 (1); (V) – Glx 0.97 (1), Phe 2.98 (3), Gly 1.03 (1), His 0.95 (1), Met 1.01 (1); (VI) – Glx 0.96 (1), Phg 0.99 (1), Phe 1.97 (2), Gly 1.02 (1), His 0.89 (1), Met 0.95 (1); (VII) – Glx 0.97 (1), Phe 2.01 (2), Phg 1.02 (1), Gly 0.97 (1), His 0.92 (1), Met 0.98 (1); (VIII) – Glx 0.94 (1), Phg 1.96 (2), Gly 1.03 (1), His 0.84 (1), Phe 0.92 (1), Met 0.95 (1); (IX) – Phg 1.02 (1), Phe 3.03 (3). Gly 0.97 (1), His 0.86 (1), Met 0.89 (1). The physicochemical characteristics of compounds (I)-(IX) are given in Table 3.

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INSECT PHEROMONES AND THEIR ANALOGUES.

XXXIV. CHIRAL PHEROMONES FROM (S)-(+)-3,7-DIMETHYLOCTA-1,6-DIENE.

2. SYNTHESIS OF OPTICALLY ACTIVE (S)-(–)-DIPRIONYL ACETATE CONFIGURATIONALLY HOMOGENEOUS WITH RESPECT TO THE C^3 ATOM

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UDC 542.91+541.65+547.315.3+632.936.9

A four-stage synthesis has been performed of the optically active (S)-(–)-diprionyl acetate in the form of an equimolar mixture of erythro-(2S,3S,7SR)- and threo-(2R,3S,7SR)-2-acetoxy-3,7-dimethylpentadecanes from a readily available chiral compound – (S)-(+)-3,7-dimethylocta-1,6-diene – with an overall yield of 13%.

The sex pheromone of dangerous pests of coniferous trees – pine sawflies of the genera Diprion and Neodiprion – includes acetates of optically active forms of 3,7-dimethylpentadecan-2-91 [1-3]. Several syntheses of racemic (for the latest, see [4]) and optically active forms configurationally homogeneous with respect to one [5], two [1, 6], or all three [7-9] chiral centers of the pheromone have been described in the literature.

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In the present paper we describe the synthesis of optically active (3S)-3,7-dimethylpentadec-2-yl acetate [(S)-(−)-diprionyl acetate] (VII) in the form of an equimolar mixture of two diastereomeric pairs, with the (2S,3S,7SR)- and (2R,3S,7SR)- configurations, from the readily available [10] (S)-(+)-3,7-dimethylocta-1,6-diene (I). The synthetic transformations from the initial chiral synthon (I) to the desired pheromone (VII) included the following operations. Oxidation of the diene (I) with oxygen on a palladium catalyst according to [11] took place selectively, with the exclusive formation of an equimolar mixture of the erythro-(2S,3S)- and threo-(2R,3S)- alcohols (III), which was converted into the corresponding mixtures of acetates (IV), as was shown by the results of capillary GLC and the equal intensities of the doublet signals of the protons of the methyl groups at the C^2 asymmetric atom, which, according to known laws for PMR spectra [9, 12, 13], are present in a weaker field for erythro isomers [δ 1.13 and 1.19 ppm for the alcohol (III) and the acetate (IV), respectively] than for the threo isomers [δ 1.11 and 1.14 for the alcohol (III) and the acetate (IV), respectively].

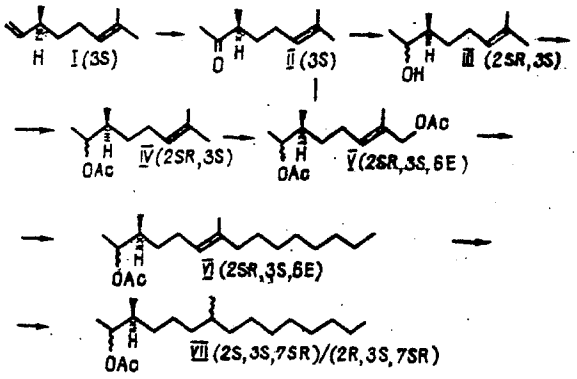
Oxidation of the acetate (IV) with selenium dioxide [14] led, after treatment with sodium tetrahydroborate followed by acetylation, exclusively (GLC results) to (2SR,3S,6E)-2,8-diacetoxy-3,7-dimethyloct-6-ene (V). The position of the signal of the CH_3 group on the double bond in the δ 13.97 region of the ^{13}C NMR spectrum unambiguously indicated its (E)- configuration [15].

It has been found that the allyl oxidation of the isopropylidene group carried out for the acetate (IV) can also be successfully applied to the ketone (II) with the formation of the same diacetate (V), in higher yield. As was to be expected, it was possible to replace the allyl acetoxy group in the diacetate (V) by an alkyl group smoothly under the action of an organocopper reagent generated from n-heptyl bromide. The coupling product, obtained in high yield, consisted of an equimolar (according to capillary GLC and ^1H NMR spectroscopy) mixture of erythro-(2S,3S)- and threo-(2R,3S)-3,7-dimethylpentadec-6E-en-2-yl acetates (VI).

On hydrogenation of the double bond in the alkenyl acetate a third asymmetric center was formed at the C^7 atom, having the (R)- and (S)- configurations with equal probabilities. The saturated acetate (VII) formed therefore consisted of an equimolar mixture of four diastereoisomers, with the (2S,3S,7S)-, (2S,3S,7R)-, (2R,3S,7S)-, and (2R,3S,7R)- configurations.

The erythro-(2S,3S,7SR)- and threo-(2R,3S,7SR)- diastereomers differed from one another chromatographically (two peaks on a capillary GLC chromatogram) and spectrally (in the PMR spectrum there were two doublets of the head methyl group at δ 1.12 and 1.14 ppm and two doublets of the CH_3 group at the C^3 atom in the regions of δ 0.85 and 0.87 ppm; in the ^{13}C NMR spectrum, pairs of quartets were observed for the same groups in the regions of 15.88 and 16.97 ppm and 14.64 and 14.82 ppm; the C^3 atoms also differed, giving two doublets at 37.06 and 37.20 ppm). In the ^{13}C NMR spectrum different chemical shifts were observed for the C^7 atom and for the CH_3 group attached to it in the diastereomers with the 7S- and 7R-configurations (two doublets at 32.95 and 33.04 ppm and two quartets at 19.67 and 19.76 ppm, respectively).

Thus, a four-stage scheme for the synthesis of the optically active pheromone (VII) in the form of an equimolar mixture of erythro-(2S,3S,7SR)- and threo-(2R,3S,7SR)- diastereomers with an overall yield of 13%, calculated on the initial dihydromyrcene (I), has been developed.



EXPERIMENTAL

IR spectra were taken on a UR-20 spectrometer (in a film); ^{13}C NMR spectra (in the regimes of complete and partial decouping from protons) for compounds (II), (V), and (VII) on a AM-300 instrument (working frequency 300 MHz); and PMR spectra for compounds (II), (V), and (VI) on a Tesla BS-567 spectrometer (working frequency 100 MHz); the solvent was $CDCl_3$, and the chemical shifts are given on the δ scale relative to the signal of TMS (internal standard). GLC analysis was conducted on a Shimadzu instrument with the stationary phase PEG-20M in a 0.2 mm × 25 m glass capillary column at working temperatures of 100°C [for (III) and (IV)] and 170°C [for (II) and (V-VII)], with helium as the carrier gas. $[\alpha]_D$ values were determined in $CHCl_3$ on a Perkin-Elmer 241 MC polarimeter. The elementary analyses of the compounds synthesized corresponded to the calculated figures.

(3S)-3,7-Dimethyloct-6-en-2-one (II). A mixture of 0.4 g ($2.2 \cdot 10^{-3}$ mole) of PdCl, 2.3 g ($23.2 \cdot 10^{-3}$ mole) of CuCl, 12 ml of DMFA, and 1.5 ml of water was stirred in an atmosphere of oxygen for 1 h, and then 2.8 g of redistilled technical dihydromyrcene containing, according to GLC, ~70% of (S)-(+)-3,7-dimethylocta-1,6-diene (I) with an optical purity of ~50%, according to [16], was added. The reaction mixture was stirred (O_2) for another 24 h, and, after the addition of 15 ml of 3 N HCl, it was extracted with diethyl ether (3 × 10 ml), and the combined extract was washed with saturated NaCl solution (3 × 10 ml), dried with $MgSO_4$, and evaporated. The residue was distilled, with the collection of a fraction having bp 73-74°C (2 mm), which was chromatographed [SiO_2, hexane–diethyl ether (10:1)], to give 1.36 g (63%) of the ketone (II) with a purity of 96% (GLC results), n_D^{28} 1.4470 [17], $[\alpha]_D^{21}$ −5.8° (c 1.6; $CHCl_3$).

IR spectrum (ν, cm^{-1}): 850 (C=C-H), 1390 (CH_3), 1720 (C=O). PMR spectrum (100 MHz, $CDCl_3$): 1.08 (d, 3H, J = 7.0 Hz, CH_3-3), 1.2-1.5 (m, 2H, H-4), 1.59 and 1.68 (s, 6H, H-8, CH_3-7), 1.8-2.04 (m, 2H, H-5), 2.12 (s, 3H, H-1), 2.38-2.64 (m, 1H, H-3), 5.07 (t, 1H, J = 6.0 Hz, H-6). ^{13}C NMR spectrum (75.47 MHz, $CDCl_3$): 28.00 (q, C-1), 212.87 (s, C-2), 46.61 (d, C-3), 16.19 (q, CH_3-3), 32.90 (t, C-4), 25.65 (t, C-5), 123.70 (d, C-6), 132.18 (s, C-7), 17.62 and 25.65 (q, CH_3-7, C-8).

(2SR,3S)-3,7-Dimethyloct-6-en-2-one (III). At 10-15°C, 0.19 g ($5.0 \cdot 10^{-3}$ mole) of $NaBH_4$ was added to a solution of 0.7 g ($4.5 \cdot 10^{-3}$ mole) of ketone (II) in 7 ml of methanol and the mixture was stirred at room temperature for 12 h and was then treated with 12 ml of 10% AcOH and evaporated. The residue was extracted with diethyl ether (3 × 50 ml), and the combined extract was washed with saturated NaCl solution, dried with Na_2SO_4, and evaporated. This gave 0.6 g (85%) of the alcohol (III) with a purity of 96% (results of capillary GLC), n_D^{28} 1.4535, $[\alpha]_D^{21}$ −3.0° (c 2.5; $CHCl_3$).

IR spectrum (ν, cm^{-1}): 850 (C=C-H), 1390 (CH_3), 1675 (C=C), 3400 (OH). PMR spectrum (300 MHz, $CDCl_3$): 0.87 and 0.89 (d, totaling 3H, J = 6.8 Hz, CH_3-3), 1.11 (d, 1.5H, J = 7.0 Hz, H-1), 1.13 (d, 1.5H, J = 6.5 Hz, H-1), 1.38-1.6 (m, 3H, H-3, H-4), 1.61 and 1.68 (s, 6H, H-8, CH_3-7), 1.8-2.15 (m, 3H, OH, H-5), 3.6-3.75 (m, 1H, H-2), 5.11 (t, 1H, J = 7.0 Hz, H-6). ^{13}C NMR spectrum (75.47 MHz, $CDCl_3$): 19.24/20.23 (q, C-1), 71.30/71.65 (d, C-2), 39.40/39.61 (d, C-3), 14.13/14.38 (q, CH_3-3), 32.76 (t, C-4), 25.61 (t, C-5), 124.72 (d, C-6), 131.35 (s, C-7), 17.63 and 25.77 (q, CH_3-7, C-8).

(2SR,3S)-2-Acetoxy-3,7-dimethyloct-6-ene (IV). A mixture of 0.6 g ($3.87 \cdot 10^{-3}$ mole) of the alcohol (III), 0.46 ml of Ac_2O, and 1.9 ml of Et_3N in 8 ml of dry CH_2Cl_2 was treated wth 10 ml of dimethylaminopyridine (DMAP), and the mixture was kept at 20-25°C for 40 h and was then poured into ice water and extracted with diethyl ether (3 × 50 ml). The organic layer was washed with saturated solutions of $CuSO_4$, $NaHCO_3$, and NaCl, and it was dried over $MgSO_4$. The residue after evaporation was chromatographed [SiO_2, hexane–diethyl ether (10:1)], to give 0.7 g (92%) of the acetate (IV) with a purity of 96% (results of capillary GLC), n_D^{20} 1.4398, $[\alpha]_D^{19}$ −2.6° (c 2.9; $CHCl_3$).

IR spectrum (ν, cm^{-1}): 870 (C=C-H), 1260 (C-O-C), 1390 (CH_3), 1740 (C=O). PMR spectrum (300 MHz, $CDCl_3$): 0.89 and 0.91 (d, totaling 3H, J = 6.8 Hz, CH_3-3), 1.14 (d, 1.5H, J = 6.4 Hz, H-1), 1.19 (d, 1.5H, J = 6.9 Hz, H-1), 1.34-1.6 (m, 3H, H-3, H-4), 1.61 and 1.68 (s, 6H, H-8, CH_3-7), 1.8-2.04 (m, 2H, H-5), 2.03 (s, 3H, CH_3CO), 4.75-4.92 (m, 1H, H-2), 5.09 (t, 1H, J = 7.0 Hz, H-6). ^{13}C NMR spectrum (75.47 MHz, $CDCl_3$): 15.95/16.95 (q, C-1), 73.92/74.29 (d, C-2), 36.95/37.22 (d, C-3), 14.56/14.67 (q, CH_3-3), 32.69/32.75 (t, C-4), 25.61 (t, C-5), 124.50 (d, C-6), 131.54 (s, C-7), 17.65 and 25.70 (q, CH_3-7, C-8), 21.32 (q, H_3CCO), 170.65/170.71 (s, H_3CCO).

(2R,3S,6E)-2,8-Diacetoxy-3,7-dimethyloct-6-ene (V). a) A solution of 2.0 g ($10.1 \cdot 10^{-3}$ mole) of the acetate (IV) in 15 ml of absolute ethanol was treated with 1.15 g ($10.3 \cdot 10^{-3}$ mole) of selenium dioxide, and the mixture was boiled for 3.5 h and was then cooled to 15°C; after the successive addition of 10 ml of MeOH and 0.76 g ($20 \cdot 10^{-3}$ mole) of sodium tetrahydroborate it was stirred at room temperature for 15 h and was then treated with 10 ml of a mixture (10:1) of water and AcOH and was evaporated. The residue was extracted with diethyl ether (3 × 100 ml), and the extract was washed with saturated NaCl solution, dried with Na_2SO_4, and evaporated. To the residue was added 6 ml of Ac_2O–Py (2:3), and the mixture was kept at room temperature for 24 h and was then diluted with 100 ml of CH_2Cl_2, and it was washed successively with 10% HCl and with saturated solutions of $NaHCO_3$ and NaCl, dried with $MgSO_4$ and evaporated. The resulting residue was chromatographed [SiO_2, hexane–diethyl ether (7:3)], to give 0.71 g (27%) of the diacetate (V) with a purity of not less than 96% (results of capillary GLC), n_D^{21} 1.4660, $[\alpha]_D^{24}$ −2.0° (c 3.4; $CHCl_3$).

IR spectrum (ν, cm^{-1}): 855 (C=C–H), 1255 (C–O–C), 1380 (CH_3), 1730, 1740 (C=O). PMR spectrum (100 MHz, $CDCl_3$): 0.92 (d, 3H, J = 6.3 Hz, CH_3-3), 1.14 (d, 1.5H, J = 6.3 Hz, H-1), 1.16 (d, 1.5H, J = 6.6 Hz, H-1), 1.3-1.6 (m, 3H, H-3, H-4), 1.66 (s, 3H, CH_3-7), 1.8-2.16 (m, 2H, H-5), 2.03 (s, 3H, CH_3CO-2), 2.07 (s, 3H, CH_3CO-8), 4.45 (s, 2H, H-8), 4.65-4.95 (m, 1H, H-2), 5.42 (t, 1H, J = 7 Hz, H-6). ^{13}C NMR spectrum (75.47 MHz, $CDCl_3$): 70.17 (t, C-8), 130.29 (s, C-7), 13.97 (q, CH_3-7), 129.44 (d, C-6), 25.33 (t, C-5), 32.12 (t, C-4), 37.01/37.21 (d, C-3), 14.62 (q, CH_3-3), 73.63/74.09 (d, C-2), 16.06/16.91 (q, C-1), 21.02 (q, $H_3\underline{C}CO$-8), 170.64 (s, $H_3C\underline{C}O$-8), 21.35 (q, $H_3\underline{C}CO$-2), 170.9 (s, $H_3C\underline{C}O$-2).

b) A solution of 1.56 g ($10.1 \cdot 10^{-3}$ mole) of the ketone (II) in 15 ml of absolute ethanol was treated with 1.15 g ($10.3 \cdot 10^{-3}$ mole) of selenium dioxide, and the mixture was boiled for 3.5 h and was then cooled to 15°C, and 10 ml of MeOH and 1.14 g ($30 \cdot 10^{-3}$ mole) of sodium tetrahydroborate were added successively; the resulting mixture was stirred at room temperature for 15 h and was then treated with 15 ml of a mixture (10:1) of water and AcOH and was evaporated to dryness. The residue was extracted with diethyl ether (3 × 100 ml) and $CHCl_3$ (50 ml) and the extract was dried with Na_2SO_4 and evaporated; 40 ml of dry CH_2CL_2, 1.2 g of Ac_2O, 5 ml of Et_3N, and 20 ml of DMAP were added successively to the residue so obtained and the resulting mixture was kept at 20-25°C for 40 h and was then poured into ice water and extracted with diethyl ether (3 × 100 ml). The organic layer was washed successively with saturated solutions of $CuSO_4$, $NaHCO_3$, and NaCl, and was dried over $MgSO_4$ and evaporated. The residue was chromatographed [SiO_2, hexane–diethyl ether (7:3)], to give 0.65 g (35%) of the diacetate (V), identical with that obtained in the preceding experiment.

(2SR,3S,6E)-2-Acetoxy-3,7-dimethylpentadec-6-ene (VI). To a stirred solution of the Grignard reagent obtained from 0.08 g ($3.3 \cdot 10^{-3}$ g-atom) of magnesium and 0.54 g ($3 \cdot 10^{-3}$ mole) of n-heptyl bromide in an absolute THF was added (−15°C, Ar) 0.48 g ($2.5 \cdot 10^{-3}$ mole) of CuI and the mixture was stirred at −10°C for 0.5 h, after which a solution of 0.3 g ($1.17 \cdot 10^{-3}$ mole) of the diacetate (V) in 3 ml of absolute THF was added dropwise and the mixture was stirred for 4 h, treated with 10 ml of saturated NH_4Cl solution, and extracted with diethyl ether (3 × 50 ml). The combined organic layer was washed with saturated NaCl solution, dried with $MgSO_4$, and evaporated. The residue was chromatographed [SiO_2, hexane–diethyl ether (9:1)], to give 0.29 g (84%) of the acetate (VI) with a purity of not less than 96% (results of capillary GLC), n_D^{20} 1.4498, $[\alpha]_D^{20}$ −2.7° (c 2.8; $CHCl_3$).

IR spectrum (ν, cm^{-1}): 865 (C=C–H), 1255 (C–O–C), 1390 (CH_3), 1740 (C=O). PMR spectrum (100 MHz, $CDCl_3$): 0.85 (t, 3H, J = 7 Hz, H-15), 0.89 (d, 3H, J = 6.5 Hz, CH_3-3), 1.14 (d, 1.5H, J = 6.5 Hz, H-1), 1.16 (d, 1.5H, J = 6.5 Hz, H-1), 1.2-1.7 (m, 16H, CH_2, CH), 1.6 (s, 3H, CH_3-7), 1.8-2.1 (m, 4H, H-5, H-8), 2.03 (s, 3H, CH_3CO, 4.76-4.90 (m, 1H, H-2), 5.1 (t, 1H, J = 7.0 Hz, H-6).

(2S-3S,7SR/2R,3S,7SR)-2-Acetoxy-3,7-dimethylpentadecane (VII). A mixture of 0.148 g ($0.5 \cdot 10^{-3}$ mole) of compound (VI), 10 ml of absolute ethanol, and 0.06 g of 5% Pd/C was stirred in an atmosphere of hydrogen until the absorption of hydrogen ceased (30 h), and it was then filtered and evaporated. This gave 0.145 g (97%) of the acetate (VII) with a purity of not less than 96% (results of capillary GLC), n_D^{20} 1.4400, $[\alpha]_D^{22}$ −3.1° (c 1.8; $CHCl_3$) [5].

IR spectrum (ν, cm^{-1}): 1260 (C–O–C), 1380 (CH_3), 1740 (C=O). PMR spectrum (300 MHz, $CDCl_3$): 0.84 (d, 3H, J = 6.5 Hz, CH_3-7), 0.85 and 0.87 (d, totaling 3H, J = 6.4 Hz, CH_3-3), 1.12 (d, 1.5H, J = 6.4 Hz, H-1), 1.14 (d, 1.5H, J = 6.4 Hz, H-1), 1.2-1.7 (22H, CH_2, CH),

2.03 (s, 3H, CH_3CO), 4.76-4.90 (m, 1H, H-2). ^{13}C NMR spectrum (75.47 MHz, $CDCl_3$): 15.88/16.97 (q, C-1), 74.12/74.39 (d, C-2), 37.06/37.20 (d, C-3), 14.64/14.82 (q, CH_3-3), 32.76 (t, C-4), 24.56 (t, C-5), 37.30/37.64 (t, C-6), 32.95/33.04 (d, C-7), 19.67/19.76 (q, CH_3-7), 37.30 (t, C-8), 27.11 (t, C-9), 30.07 (t, C-10), 29.72 (t, C-11), 29.38 (t, C-12), 31.97 (t, C-13), 22.71 (t, C-14), 14.12 (q, C-15), 21.34 (q, $\underline{C}H_3CO$), 170.76 (S, $CH_3\underline{C}O$).

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