INSECT PHEROMONES AND THEIR ANALOGUES. XLV. SYNTHESIS OF MONO-AND DIENIC COMPONENTS OF INSECT PHEROMONES FROM ISOPROPYL NONA-3E,8-DIENOATE

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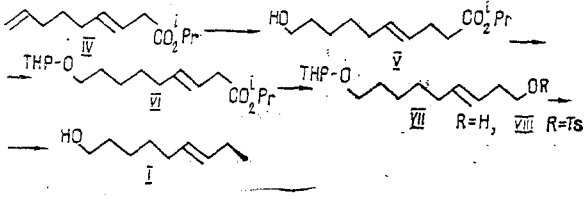
Non-6E-en-1-ol, dodec-3E-en-1-yl acetate, and dodeca-7E, 9Z-dien-1-yl acetate, which are components of sex pheromomes of insects of the order Lepidoptera, have been synthesized from the readily accessible isopropyl nona-3E,8-dienoate.

In the present communication, using as examples syntheses of non-6E-en-1-ol (I) and the acetates of dodec-3E-en- and dodeca-7E,9Z-dien-1-ols (II and III), we demonstrate the rich possibilities of derivatives of nona-3E,8-dienoic acid as applied to the synthesis of mono- and dienic components of pheromomes of insects of the order Lepidoptera.

The product of the copolymerization of butadiene and carbon monoxide catalyzed by complex compounds of palladium is an accessible [1] and convenient starting compound for the synthesis of pheromomes but it has been used for this purpose only once – in the synthesis of brevicomin [2].

A whole series of syntheses starting from various compounds have been described for compounds (I-III), which are the main components of sex pheromomes of the Mediterranean fruit fly (Ceratitis capitata), the beet-mining moth (Scrobipalpa ocellatella), and the European grape moth (Lobesia botrana), respectively [3-17]. In the scheme of synthesis that we have developed, isopropyl nona-3E,8-dienoate (IV) is used as the initial compound.

The 9-atom carbon skeleton of (IV) corresponds to the structure of the first of the above-mentioned pheromomes. The conversion of the dienic ester (IV) into the 6E-monoenic alcohol (I) was achieved after five successive transformations with the participation of the terminal double bond and the ester group. The hydroboration of the ester (IV) with the aid of 9-borabicyclo[3.3.1]nonane (BBN) took place regiospecifically and, after oxidation of the organoboron intermediate, led to isopropyl 9-hydroxynon-3E-enoate (V), which was converted into the tetrahydropyranol ether (VI), and the latter was reduced with diisobutylaluminum hydride (DIBAH) to the selectively protected diol 9-(tetrahydropyran-2-yloxy)non-3E-en-1-ol (VII). The synthesis of pheromome (I) was completed by the conversion of compound (VII) into its tosylate (VIII) and the reduction of the latter with lithium tetrahydroaluminate, the overall yield of the pheromome (I) calculated on the initial (IV) being 41%. According to the results of GLC analysis on a capillary column the purity of the product was not less than 98%.



In the molecule of pheromome (II), as in the initial compound, a double bond with the E-configuration is present in position 3, and the transformation of the structure of (IV) con-

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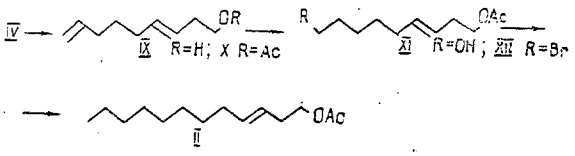
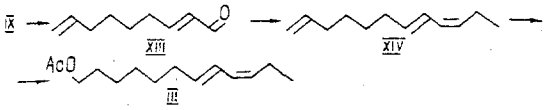
sisted in the growth of the carbon skeleton by three atoms. This transformation was performed in five stages. In the first of them the ester was (IV) was converted into nona-3E, 8-dien-1-ol (IX), and this into the corresponding acetate (X). Hydroboration gave 9-hydroxynon-3E-en-1-yl acetate (XI), from which 9-bromonon-3E-en-1-yl acetate (XII) was obtained. The interaction of n-propylmagnesium bromide with the bromoacetate (XII) took place selectively at the bromine atom and led to the desired pheromome (II), the overall yield of which, calculated on the initial (IV), was 28.5%.



For the alcohol (IX), we proposed a transformation leading unambiguously to the pheromome with (E, Z)-conjugated double bonds – the acetate (III). The oxidation of alcohol (IX) was accompanied by the shifting of a double bond into conjugation with the aldehyde group, which was finally completed on subsequent heating in the presence of an antioxidant. The olefination of the resulting nona-2E,8-dienal (XIII) with n-propylidenetriphenylphosphorane gave doceca-1,7E,9Z-triene (XIV). This C_{12} triene was converted into the desired pheromome (III) by hydroboration, which took place exclusively at the terminal double bond. The completing operation in this synthesis was the acetylation of the dienic alcohol produced by hydroboration. The overall yield of pheromome (III) calculated on the (IV) was 16.5%, and the content of the main (7E, 9Z)-stereoisomer exceeded 93% (results of capillary chromatography).



EXPERIMENTAL

IR spectra were taken on a UR-20 spectrometer (in a film), and PMR spectra for compounds (XI), (XII), and (XIV) were recorded on a Tesla BS-467 instrument (working frequency 60 MHz) with CCl_4 as solvent. Other PMR spectra were taken on a AM-300 spectrometer (300 MHz) for (XIII)) or a Tesla BS-567 (100 MHz). ^{13}C NMR spectra were recorded on an AM-300 instrument (75.47 MHz) (for (XIII)) or a JEOL FX-90 Q (22.5 MHz) with broad-band and off-resonance suppression of proton effects; the solvent was $CDCl_3$ and the chemical shifts are given in the δ scale relative to the signals of TMS (internal standard). GLC analysis was conducted on a Chrom-5 instrument [with the stationary phase SE-30 silicone liquid (5%) on Chromaton N-AW-DMCS (0.16-0.20 mm) at a working temperature of 50-300°C] or a Shimadzu instrument [for (I), (II), and (III)); stationary phase PEG-20M, glass capillary column, 2 mm × 25 m; working temperature 50-180°C], with helium as the carrier gas. The elementary analyses of the compounds synthesized corresponded to the calculated figures.

Isopropyl 9-Hydroxynon-3E-enoate (V). A solution of 5.06 g ($25.8\cdot10^{-3}$ mole) of the ester (IV) [1] in 15 ml of abs. THF was added at 10°C over 0.5 h to a suspension of 4.5 g ($36.9\cdot10^{-3}$ mole) of BBN in 45 ml of abs. THF; after 2 h, the mixture was treated at 0°C with a solution of 7.5 g of AcONa in 18 ml of water, and then 27 ml of 30% H_2O_2 was added dropwise over 1 h. The reaction mixture was stirred at 25°C for 2 h and was then diluted with 300 ml of diethyl ether and was washed successively with saturated NaCl solution, 0.1 N $Na_2S_2O_3$ solution, and NaCl again, and was dried with Na_2SO_4 and evaporated. The residue was chromatographed (SiO_2, gradient elution from hexane to hexane containing 45% of diethyl ether). This gave 3.76 g (68%) of compound (V), bp·125-126°C (3 mm)), n_D^{23} 1.4586. IR spectrum (ν, cm^{-1}): 985 and 1660 (E–CH=CH), 1070 (C–O), 1730 (C=O), 3400 (OH). PMR spectrum (100 MHz, $CDCl_3$): 1.23 (d, 6H, J = 6.4 Hz, CH_3), 1.15-1.7 (m, 6H, H-6, H-7, H-8), 1.96-2.14 (m, 2H, H-5), 2.96-3.14 (m, 2H, H-2), 3.61 (t, 2H, J = 6.3 Hz, H-9), 5.0-5.24 (m, 1H, CH), 5.54-5.76 (m, 2H, H-3, H-4).

Isopropyl 9-(Tetrahydropyran-2-yloxy)non-3E-enoate (VI). At 10-15°C, 1.5 ml (1.34 g; $16.0\cdot10^{-3}$ mole) of 2,3-dihydropyran was added to a solution of 1.0 g ($4.7\cdot10^{-3}$ mole) of the alcohol (V) and 0.03 g of TsOH in 15 ml of abs. diethyl ether, and then the mixture was heated to room temperature and was stirred for 20 h, after which 100 ml of diethyl ether was added and the whole was washed successively with saturated solutions of $NaHCO_3$ and NaCL, dried with $MgSO_4$, and evaporated.

The residue was chromatographed (SiO_2, hexane–diethyl ether (4:1)) and yielded 1.24 g (89%) of compound (VI), n_D^{23} 1.4602. IR spectrum (ν, cm^{-1}): 975 and 1665 (E–CH=CH), 1040, 1080, 1110, 1140, and 1180 (C–O), 1740 (C=O). PMR spectrum (100 MHz, $CDCl_3$): 1.23 (d, 6H, J = 6.1 Hz, CH_3), 1.1-1.8 (m, 12H, CH_2), 1.97-2.25 (m, 2H, H-5), 2.94-3.15 (m, 2H, H-2), 3.3-4.1 (m, 4H, CH_2O), 4.56 (br.s, 1H, OCHO), 4.98-5.28 (m, 1H, HCO), 5.55-5.78 (m, 2H, H-3, H-4).

9-(Tetrahydropyran-2-yloxy)non-3E-en-1-ol (VII). A solution of 0.7 g ($2.3 \cdot 10^{-3}$ mole) of the ester (VI) in 10 ml of abs. diethyl ether was treated (Ar, −20°C) with 2 ml of a 73% solution ($8.8 \cdot 10^{-3}$ mole) of DIBAH in toluene, and the mixture was stirred for 2 h and was heated to 0°C; then 20 ml of water was added and, after two hours' stirring at room temperature, the product was extracted with diethyl ether (2 × 50 ml). The combined extracts were washed with saturated NaCl solution, dried with Na_2SO_4, and evaporated, to give 0.55 g (96%) of compound (VII), n_D^{23} 1.4664. IR spectrum (ν, cm^{-1}): 985 and 1660 (E–CH=CH), 1040, 1050, 1090, 1135, and 1150 (C–O), 3400 (OH). PMR spectrum (100 MHz, $CDCl_3$): 1.2-1.8 (m, 12H, CH_2), 1.87 (br.s, 1H, OH), 1.98-2.48 (m, 4H, H-2, H-5), 3.32-4.1 (m, 6H, CH_2O), 4.56 (br.s, 1H, OCHO), 5.4-5.73 (m, 2H, H-3, H-4).

9-(Tetrahydropyran-2-yloxy)-1-tosyloxynon-3E-ene (VIII). In small portions, 0.79 g ($4.1 \cdot 10^{-3}$ mole) of TsCl was added to a solution of 0.5 g ($2.06 \cdot 10^{-3}$ mole) of compound (VII) in 1.2 ml of dry pyridine cooled to 0°C, and the mixture was stirred for 1 h and was kept at the same temperature for 15 h, and it was then diluted with 100 ml of diethyl ether and was washed successively with cooled 10% HCl and saturated solutions of $NaHCO_3$ and NaCl, and was dried with $MgSO_4$ and evaporated. This gave 0.75 g (92%) of the tosylate (VIII). IR spectrum (ν, cm^{-1}): 970 and 1660 (E–CH=CH), 1040, 1080, 1100 and 1130 (C–O), 1185 and 1370 (SO_2), 1600 and 3030 (Ar).

Non-6E-1-ol (I). Under Ar at 0°C, 0.038 g ($1.0 \cdot 10^{-3}$ mole) of lithium tetrahydroaluminate was added to a solution of 0.4 g ($1.0 \cdot 10^{-3}$ mole) of compound (VIII) in 10 ml of absolute ether, and the mixture was stirred at 0°C for 1 h and at 20°C for 2 h; then it was again cooled to 0°C and, after the addition of 2 ml of water, it was stirred at room temperature for 2 h and was extracted with diethyl ether (3 × 30 ml), and the extract was dried with $MgSO_4$ and evaporated. The residue was dissolved in 2 ml of methanol containing 0.02 g of TsOH and 0.4 ml of water, and the solution was stirred for 15 h and was then evaporated. The new residue was treated with 50 ml of diethyl ether, and the resulting solution was washed with saturated $NaHCO_3$, dried with Na_2SO_4, and evaporated. The residue was chromatographed (SiO_2, hexane–diethyl ether (1:1)), to give 0.11 g (76%) of the alcohol (I) (with a purity of no less than 98% according to GLC), n_D^{25} 1.4470; IR and PMR spectra identical with those given in the literature [5].

Nona-3E,8-dien-1-ol (IX). Under Ar at −20°C, 41 ml (0.181 mole) of a 73% solution of DIBAH in toluene was added to a solution of 11.2 g ($57.1 \cdot 10^{-3}$ mole) of the ester (IV) in 160 ml of absolute diethyl ether, and the mixture was worked up as described above for compound (VII). This gave 7.6 g (95%) of the alcohol (IX), n_D^{20} 1.4678, having IR and PMR spectra identical with those given in the literature [18].

Nona-3E,8-dien-1-yl Acetate (X). The alcohol (IX) (2.8 g; 0.02 mole) was treated with 22 ml of a 2:3 mixture of acetic anhydride and pyridine, and the reaction mixture was kept at room temperature for 24 h and was then diluted with 250 ml of CH_2Cl_2 and was washed successively with 10% HCl and with saturated solutions of $NaHCO_3$ and NaCl, dried with $MgSO_4$ and evaporated. The residue was chromatographed (SiO_2, hexane–diethyl ether (10:1)), to give 3.34 g (92%) of the acetate (X), n_D^{24} 1.4460. IR spectrum (ν, cm^{-1}): 925, 980, 1010, 1645 and 3090 (E-CH=CH, $CH=CH_2$), 1250 and 1740 (OAc). PMR spectrum (100 MHz, $CDCl_3$): 1.1-1.62 (m, 2H, H-6), 1.8-2.42 (m, 9H, H-2, H-5, H-7, CH_3CO), 4.07 (t, 2H, J = 6.8 Hz, H-1), 4.8-6.0 (m, 5H, H-3, H-4, H-8, H-9).

9-Hydroxynon-3E-en-1-yl Acetate (XI). A solution of 1.57 g ($8.6 \cdot 10^{-3}$ mole) of the acetate (X) in 5 ml of abs. THF was added over 15 min at 10°C to a suspension of 1.5 g ($12.3 \cdot 10^{-3}$ mole) of BBN in 15 ml of abs. THF, and the reaction mixture was kept for 2 h, and then, at 0°C, s solution of 2.5 g of AcONa in 6 ml of water was added to it and, after this, dropwise over 0.5 h, 9 ml of 30% H_2O_2. The subsequent working up procedure was the same as for compound (V), and it led to 1.22 g (71%) of compound (XI), n_D^{23} 1.4570. Ir spectrum (ν, cm^{-1}): 985 and 1660 (E-CH=CH), 1060 (C-O), 1260 and 1740 (OAc), 3400 (OH). PMR spectrum (60 MHz, CCl_4): 1.15-1.60 (m, 6H, CH_2), 1.93 (s, 3H, CH_3CO), 2.0-2.4 (m, 4H, H-2, H-5), 2.9 (br.s, 1H, OH), 3.47 (t, 2H, J = 6 Hz, H-9), 3.93 (t, 2H, J = 7 Hz, H-1), 5.18-5.47 (m, 2H, H-3, H-4).

9-Bromonon-3E-en-1-yl Acetate (XII). In portions, 0.5 g ($2{,}6 \cdot 10^{-3}$ mole) of TsCl was added to a solution of 0.44 g ($2.2 \cdot 10^{-3}$ mole) of compound (XI) in 0.7 ml of dry pyridine cooled to 0°C, and the mixture was stirred for 1 h and was kept at the same temperature for 15 h and was then diluted with 50 ml of diethyl ether and was washed successively with 10% HCl and saturated solutions of $NaHCO_3$ and NaCl, and was dried with $MgSO_4$ and evaporated. The residue (0.72 g) was dissolved in 9 ml of bs. acetone, and 0.26 g ($3.0 \cdot 10^{-3}$ mole) of LiBr was added; the mixture was boiled for 3 h and was then evaporated, and the residue extracted with abs. hexane (3 × 20 ml), and the extract was evaporated. The new residue was chromatographed (SiO_2, pentane–diethyl ether (10:1)), giving 0.47 g (82%) of the bromide (XII), n_D^{23} 1.4764. IR spectrum (ν, cm^{-1}): 560 and 650 (C-Br), 970 and 1660 (E-CH=CH), 1240 and 1740 (OAc). PMR spectrum (60 MHz, CCl_4): 1.18-1.6 (m, 6H, CH_2), 1.93 (s, 3H, $CH_3C{=}O$), 2.0-2.45 (m, 4H, H-2, H-5), 3.30 (t, 2H, J = 6.5 Hz, H-9), 3.93 (t, 2H, J = 7 Hz, H-1), 5.23-5.48 (m, 2H, H-3, H-4).

Dodec-3E-en-1-yl Acetate (II). Under argon at −15°C, 0.05 ml of a 0.1 M solution of Li_2CuCl_4 in THF was added to a solution of 0.26 g ($1.0 \cdot 10^{-3}$ mole)) of the bromide (XII) in 0.5 ml of abs. THF, and the mixture was stirred for 10 min, and then the Grignard reagent obtained from 0.065 g ($2.73 \cdot 10^{-3}$ g-atom) of magnesium and 0.33 g ($2.73 \cdot 10^{-3}$ mole) of n-propyl bromide in 5 ml of abs. THF was added, and stirring was continued for 4 h, after which 3 ml of saturated NH_4Cl solution was added, the mixture was stirred for 1 h and was extracted with diethyl ether, and the extract was dried with $MgSO_4$ and evaporated. The residue was chromatographed (SiO_2, hexane–diethyl ether (15:1)), to give 0.13 g (56%) of the acetate (II) (with a purity of not less than 98% according to capillary GLC), n_D^{20} 1.4490; IR and PMR spectra identical with those given in the literature [9].

Non-2E,8-dienal (XIII). To a stirred suspension of 7.26 g of pyridine chlorochromate in 110 ml of abs. CH_2Cl_2 was added a solution of 2.8 g ($20.0 \cdot 10^{-3}$ mole) of the alcohol (IX) in 5 ml of CH_2Cl_2, and the mixture was stirred at room temperature for 1.5 h and was then diluted with 100 ml of diethyl ether and filtered through a layer (20 g) of SiO_2 on a porous glass filter, and the residue on the filter was washed with 50 ml of diethyl ether. The filtrate was evaporated, and 0.1 g of antioxidant (fenozan-43) was added to the residue (2.5 g), and the mixture was heated (140°C, 0.3 h; Ar) and was then chromatographed (SiO_2; pentane–diethyl ether (10:1)) to give 1.4 g (51%) of the aldehyde (XIII), n_D^{23} 1.4580 (compare [19]). IR spectrum (ν, cm^{-1}): 920, 980, 1000, 1650 and 3085 (E-CH=CH, $CH{=}CH_2$), 1700 and 2740 (CHO). PMR spectrum (300 MHz, $CDCl_3$): 1.4-1.6 (m, 4H, H-5, H-6), 2.1-2.2 (m, 2H, H-7), 2.2-2.4 (m, 2H, H-4), 4.9-5.2 (m, 2H, H-9), 5.6-5.9 (m, 1H, H-8), 6.12 (dd, 1H, J_1 = 15.9 Hz, J_2 = 7.86 Hz, H-2), 6.85 (dt, 1H, J_1 = 15.91 Hz, J_2 = 6.6 Hz, H-3), 9.50 (d, 1H, J = 7.86 Hz, H-1). ^{13}C NMR spectrum (75.47 MHz, $CDCl_3$); 193.48 (d, C-1), 133.23 (d, C-2), 160.12 (d, C-3), 27.22 (t, C-4), 32.51 (t, C-5), 28.33 (t, C-6), 33.42 (t, C-7), 138.06 (d, C-8), 114.95 (t, C-9).

Dodeca-1,7E,9Z-triene (XIV). To a solution of 0.92 g ($5.03 \cdot 10^{-3}$ mole) of sodium bis-(trimethylsilyl)amide in 15 ml of abs. THF (Ar, 20°C) was added 1.94 g ($5.0 \cdot 10^{-3}$ mole) of n-propyltriphenylphosphonium bromide, and the mixture was stirred at 25°C for 0.5 h and at the boil for 1 h, and it was then cooled to −78°C and a cooled solution of 0.69 g ($5.0 \cdot 10^{-3}$ mole) of the aldehyde (XIII) in 5 ml of abs. THF was added dropwise; the reaction mixture was stirred for 1 h, and then its temperature was raised to that of the room, and it was allowed to stand for 12 h and was then evaporated. The residue was dissolved in 50 ml of a 1:3 mixture of diethyl ether and hexane, the solution was filtered, and the filtrate was washed successively with 40% $NaHCO_3$ solution and with saturated solutions of $NaHCO_3$ and NaCl and was dried with $MgSO_4$ and evaporated.

The residue was chromatographed (SiO_2, pentane) to give 0.52 g (64%) of the triene (XIV) (with a purity of not less than 96% according to the results of capillary chromatography, n_D^{23} 1.4774. IR spectrum (ν, cm^{-1}): 925, 960, 1000, 1645, 3030 and 3090 ($CH{=}CH_2$), CH=CHCH=CH). PMR spectrum (60 MHz, CCl_4): 0.95 (t, 3H, J = 7 Hz, H-12), 1.15-1.6 (m, 4H, H-4, H-5), 1.8-2.3 (m, 6H, H-3, H-6, H-11), 4.7-6.4 (m, 7H, H-1, H-2, H-7, H-8, H-9, H-10). ^{13}C NMR spectrum (22.5 MHz, $CDCl_3$): 114.34 (t, C-1), 139.00 (d, C-2), 33.68 (t, C-3), 28.92 (t, C-4), 28.54 (t, C-5), 32.73 (t, C-6), 131.77 (d, C-7), 125.71 (d, C-8), 128.07 (d, C-9), 134.45 (d, C-10), 21.06 (t, C-11), 14.33 (q, C-12).

Dodeca-7E,9Z-dien-1-yl Acetate (III). A solution of 0.48 g ($2.9 \cdot 10^{-3}$ mole) of the triene (XIV) in 2 ml of abs. THF was added over 10 min to a suspension of 0.5 g ($4.1 \cdot 10^{-3}$ mole) of BBN in 5 ml of abs. THF at 10°C, and the mixture was stirred for 2 h; after this, at 0°C,

a solution of 0.83 g of AcONa in 2 ml of water and then, dropwise over 15 min, 3 ml of 30% H_2O_2 were added, and the reaction mixture was stirred at 25°C for 2 h and was then diluted with 100 ml of diethyl ether and was washed successively with saturated NaCl solution, 0.1 N $Na_2S_2O_3$, and NaCl again, and was dried with Na_2SO_4 and evaporated.

The residue (0.8 g) was treated with 9 ml of a 2:3 mixture of acetic anhydride and pyridine and the reaction mixture was left at room temperature for 24 h and was then diluted with 100 ml of CH_2Cl_2 and was washed successively with 10% HCl and saturated solutions of $NaHCO_3$ and NaCl, and was dried with $MgSO_4$ and evaporated. The residue was distilled, to give 0.35 g (54%) of the acetate (III) (with a purity of not less than 83%, according to capillary chromatography), n_D^{23} 1.4720, bp 118-121 (1 mm); its IR, ^{13}C NMR, and PMR spectra were identical with those given in the literature [12].

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