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SYNTHESIS OF TRIDECAPRENOL WT_3C_7SCOH AND TETRAKISNOROCTAHYDRO ANALOGS OF PENTA- AND HEXAPRENOL

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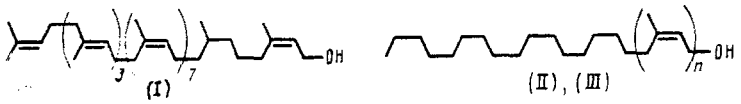
Tridecaprenol WT_3C_7SCOH and two modified prenols, having a saturated hydrocarbon residue bound to one or two isoprenoid units, were synthesized by a stepwise scheme using a C_5-saturated and cis-C_5-blocks.

The development of a stepwise scheme of a stereospecific synthesis of polyprenols enabled preparation of also their modified representatives [1]. The two series of compounds were found to be materials for studying the mechanism of the biosynthesis of carbon-containing biopolymers of cellular wall membranes of certain organisms [2, 3]. In continuation of this investigation, we discuss in the present article a stepwise synthesis of the modified prenols (I)-(III) indicated in the title. The first of these is distinguished from natural prenols by the presence of a (±)-S-unit inside the 1,5-polyene chain, while the cetyl residue of the other two was used to simulate a completely demethylated and reduced block of four WT/C units

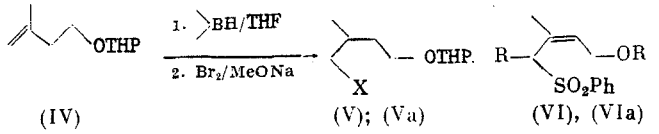


(I) (II), (III)

= 1 (II); 2 (III).

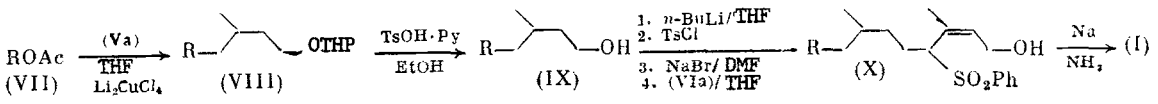
The tetrahydropyranyl (THP) ether (IV) of isobutenylcarbinol served as a precursor of a saturated C_5-syntone required for constructing the molecule of (I). Hydrobromination—bromination [4] of olefin (IV) led in 53% yield to bromide (V), recently obtained in a four-stage reaction from the same ether [5]. The known Z-hydroxy-sulfone (VI) [6] was used as another building block in the above-designated sequence of transformations, leading to compounds (I)-(III).

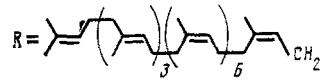
The Li_2CuCl_4-catalyzed condensation of the available [7] undecaprenol WT_3C_7OH in the form of its acetate (VII) with the Grignard reagent (Va), prepared from bromide (V), followed by TsOH·Py-catalyzed removal of the THP protective function in the intermediate (VIII), readily results in (cf. [8]) the dolichol-like prenol, WT_3C_7SOH (IX).



(IV) (V); (Va) (VI), (VIa)

X = Br (V), MgBr (Va); R = H (VI), Li (VIa).





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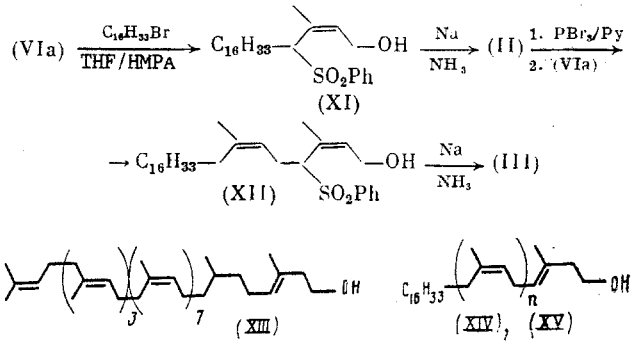
The structure of these two compounds was confirmed spectrally. In particular, there are characteristic doublet signals in their PMR spectra of the CH_3 group of a saturated α-isoprene unit, $\delta \approx 0.9$ ppm; the multiplet signals of CH_2O protons of the same unit are located in the region of $\delta \approx 3.7$ ppm (see [8, 9]). The mass spectrum of (IX) is characterized by the peak of M^+ and by a fragmentation usual for linear isoprenoids, due to successive cleavages of allylic C–C bonds (Δm/z 68) [10].

A further, Z-C_5-homologization of the saturated alcohol (IX) was carried out by its initial two-stage conversion (cf. [11]) into the corresponding tosylate and bromide. Alkylation by the latter of the dilithium derivative (VIa) of hydroxy-sulfone (VI) led to compound (X) in a good yield (a mixture of diastereomers), the reductive splitting of the C–S bond thereof leading to the desired prenol (I). Comparison of its PMR spectrum with the specimen of precursor (IX), taking into account the criteria previously established for these compounds [1, 7, 9], indicates the presence of an additional α-Z-isoprene unit in the molecule (I).

The application of the stepwise approach to the synthesis of the cisoid allylic alcohols (II) and (III) is based on the use of hydroxy-sulfone (VI). Thus, the alkylation of (VIa) with cetyl bromide smoothly leads to sulfone (XI), the reductive desulfonation of which gives alcohol (II). A standard transformation of (II) into the corresponding allyl bromide and repetition of the above operations via the hydroxy-sulfone (XII) stage gave the desired allyl alcohol (III).



n=0 (XIV), 1 (XV).

The structure of the compounds thus synthesized was confirmed spectrally. In particular, the proton signals in the PMR spectra of hydroxy-sulfones (XI) and (XII) of the CH_3 and CH_2 groups of the α-isoprene units are located in the expected regions with $\delta \approx 1.85$ and 3.85 ppm, respectively (cf. [9]); in a similar way, the position and form of proton signals of these groups in the spectra of the desulfonation products (II) and (III) correspond to these characteristics previously found for the similar Z-isoprenoid structures.

It should be noted that the reductive splitting of the C–S bond in sulfones (X)-(XII) is accompanied, as usual in these cases [1-9], by the allylic shift of the C=C bond with the formation of the homoallylic regioisomers (XIII)-(XV), which are difficult to separate from their corresponding alcohols (I)-(III). The content of (XIII)-(XV) was established by measuring the integral intensity in the PMR spectra of the mixtures of the CH_2O proton signals at $\delta \approx 3.7$ ppm belonging to these regioisomers (cf. [1, 9]), which varies within 20-35%. The homoallylic admixtures of (XIII)-(XV), however, do not hinder carrying out biochemical investigations of allyl alcohols (I)-(III), since they are eliminated under the conditions [12] of the conversion of the latter into phosphates. The admixture in alcohol (II) of its regioisomer (XIV) is also eliminated under the conditions [13] of the conversion of (II) into the corresponding allyl bromide, and does not influence (cf. [1]) the purity of sulfone (XII) and the modified prenol (III).

EXPERIMENTAL

The IR spectra (in CCl_4) were run on a UR-20 spectrophotometer. The PMR spectra of the solutions in $CDCl_3$ were measured on a Bruker WM-250 spectrometer. The mass spectra were obtained on Varian MAT CH-6 and Varian MAT 311A spectrometers at 70 eV. The R_f values are given (unless otherwise noted) for a stationary Silufol SiO_2 layer in an ether–hexane (1:1) system.

Bromo Ether (V). A 16 ml portion of a 1.5 M solution of BH_3 in THF (24 mmoles) was added over a period of 5 min to a stirred (0°C, Ar) solution of 10 g (58.7 mmoles) of (IV) [5] in 50 ml of THF. The reaction mixture was allowed to stand at 0°C for 45 min, and then was

successively treated at −5°C with a solution of 5.86 g (108.5 mmoles) of MeONa in 30 ml of MeOH added in the course of 5 min and then with 9.32 g (58.3 mmoles) of added Br_2 in the course of 15 min. The reaction mixture was heated for 1 h to 25°C, washed with ether, and neutralized with 20% H_2SO_4. The aqueous layer was separated, extracted with ether, the combined organic solution was washed with water, dried over $MgSO_4$, evaporated under vacuum, and the residue (13 g) was chromatographed on 250 g of SiO_2. Elution with a hexane–ether (9:1) mixture gave 7.8 g (53%) of (V), bp 74-76°C (0.09 mm), n_D^{20} 1.4786. Literature data [5], bp 82-84°C (0.5 mm). The PMR spectrum of (V) practically coincided with that given in [5].

2-Tetrahydropyranyl Ether of (±)-3,7,11,15,19,23,27,31,35,39,43,47-Dodecamethyloctatetraconta-6Z,10Z,14Z,18Z,22Z,26Z,30Z,34E,38E,42E,46-undecaen-1-ol (VIII). A 0.7 ml portion of a 0.1 M solution of Li_2CuCl_4 in THF (0.07 mmole) was added to a stirred (−20°C, Ar) Grignard reagent, prepared from 0.44 g (1.75 mmoles) of (V) and 0.05 g (2.06 mg·atom) of Mg in 5 ml of THF, and then a solution of 0.2 g (0.25 mmole) of (VII) in 10 ml of THF was added over a period of 5 min. The reaction mixture was stirred at −20°C for 30 min, allowed to stand for 10 h at 5°C, and was then decomposed by a saturated aqueous solution of NH_4Cl. The aqueous layer was separated, extracted with ether, the combined organic solution was washed with water, dried over $MgSO_4$, evaporated under vacuum, and the residue (0.6 g) was chromatographed on 20 g of SiO_2. Gradient elution from hexane to ether (up to 5% of the latter) gave 0.18 g (78%) of (VIII) in the form of a colorless oil, R_f 0.68 (hexane–ether, 9:1). IR spectrum (ν, cm^{-1}): 870, 910, 985, 1035, 1080, 1120, 1140, 1200, 1380, 1450, 1560, 1670, 2680, 2930, 2970. PMR spectrum (δ, ppm, J, Hz): 0.92 d (3H, CH_3–C^3, J = 7), 1.1-1.8 m (11H, CH_2, HC^3), 1.61 br. s (12H, cis-CH_3C=C), 1.70 br. s (24H, trans-CH_3C=C), 2.0 m (42H, CH_2C=C), 3.4-3.9 m (4H, CH_2O), 4.58 m (1H, OCHO), 5.1 m (11H, HC=C).

(±)-3,7,11,15,19,23,27,31,35,39,43,47-Dodecamethyloctatetraconta-6Z,10Z,14Z,18Z,22Z,26Z,30Z,34E,38E,42E,46-undecaen-1-ol (IX). A solution of 0.48 g of ether (VIII) and 10 mg of TsOH·Py in 10 ml of abs. EtOH was stirred at 65°C for 1 h, then evaporated under vacuum, and the residue (0.5 g) was chromatographed on 10 g of SiO_2. Gradient elution from hexane to ether (up to 10% of the latter) gave 0.41 g (94%) of (IX) in the form of colorless oil, R_f 0.45. IR spectrum (ν, cm^{-1}): 840, 980, 1035, 1095, 1220, 1375, 1450, 1660, 2860, 2920, 2960, 3630. PMR spectrum (δ, ppm, J, Hz): 0.92 d (3H, CH_3–C^3, J = 7), 1.1-1.6 m (5H, HC^2, HC^3, HC^4), 1.61 br. s (12H, cis-CH_3C=C), 1.70 br. s (24H, trans-CH_3C=C), 2.0 m (42H, CH_2C=C), 3.70 m (2H, CH_2O), 5.1 m (11H, HC=C). Mass spectrum (m/z): 836 (M^+), 769, 768, 701, 700, 683, 682, 633, 632, 614, 565, 546, 496, 476, 427, 409, 359, 291, 271, 203, 137, 135, 69, 68. Calculated for $C_{60}H_{100}O$: Mol. wt. 837.5.

(±)-4-Phenylsulfonyl-3,7,11,15,19,23,27,31,35,39,43,47,51-tridecamethyldopentaconta-2Z,10Z,14Z,18Z,22Z,26Z,30Z,34Z,38E,42E,46E,50-dodecaen-1-ol (X). A 0.88 ml portion of a 2 M solution of n-BuLi in hexane (1.76 mmoles) was added over the period of 3 min to a stirred (−70°C, Ar) solution of 0.2 g (0.88 mmole) of (VI) [6] in 5 ml of THF and 1.5 ml of HMPA, and then after 10 min, a solution of a bromide, freshly prepared according to [11] from 0.4 g (0.48 mmole) of (IX) in 5 ml of THF was added at −40°C in the course of 5 min. The mixture was allowed to stand at −40°C for 2 h, and then was heated in the course of 1 h to 0°C, and treated with ether and water. The aqueous layer was separated, neutralized with 20% H_2SO_4, and extracted with ether. The usual treatment of the extract gave 0.8 g of a compound, which was chromatographed on 30 g of SiO_2. Gradient elution from hexane to ether (up to 40% of the latter) gave 0.25 g (50%) of (X) in the form of a colorless oil, R_f 0.27. IR spectrum (ν, cm^{-1}): 835, 885, 1025, 1085, 1150, 1215, 1310, 1320, 1375, 1445, 1660, 2850, 2920, 2960, 3520. PMR spectrum (δ, ppm, J, Hz): 0.83 m (3H, CH_3–C^7), 1.0-1.6 m (7H, HC^5, HC^6, HC^7, HC^8), 1.62 br. s (12H, cis-CH_3C=C), 1.68 br. s (24H, trans-CH_3C=C), 1.83 br. s (3H, CH_3–C^3), 2.0 m (42H, CH_2C=C), 3.84 m (2H, CH_2O), 4.08 m (1H, HCS), 5.1 m (11H, HC=C), 5.90 br. t (1H, HC^2, J = 6.5), 7.6-7.9 m (C_6H_5). Found: S 3.14%; 903 $(M-C_6H_5SO_2)^+$. $C_{71}H_{112}O_3S$. Calculated: S 3.07%, Mol. wt. 1045.7.

3,7,11,15,19,23,27,31,35,39,43,47,51-Tridecamethyldopentaconta-2Z,10Z,14Z,18Z,22Z,26Z,30Z,34Z,38E,42E,46E,50-dodecaen-1-ol (I). A 10 ml portion of hexane, and then, in the course of 5 min, a solution of 0.22 g (0.21 mmole) of (X) in 3 ml of THF were successively added to a stirred (−65°C, Ar) solution of 40 mg (1.74 mg·atom) of Na in 10 ml of NH_3. The reaction mixture was allowed to stand for 30 min at −65°C, and was then decomposed with an excess of NH_4Cl, NH_3 was evaporated, and the residue treated with ether and water. The aqueous layer was separated and extracted with ether. The usual treatment of the combined organic layer gave 0.2 g of a compound, which was chromatographed on 10 g of SiO_2. Gradient elution from hexane to ether (up to 15% of the latter) gave 0.11 g (58%) of (I) in the form of a colorless

oil, R_f 0.48, containing (PMR) ∿20% of homoallyl alcohol (XIII). IR spectrum (ν, cm^{-1}): 830, 990, 1035, 1085, 1125, 1230, 1310, 1380, 1450, 1665, 2850, 2940, 3015, 3530, 3620. PMR spectrum of (I) (δ, ppm, J, Hz): 0.87 d (3H, CH_3–C^7, J = 6.5), 1.1-1.6 m (7H, HC^5, HC^6, HC^7, HC^8), 1.61 br. s (12H, cis-CH_3C=C), 1.69 br. s (24H, trans-CH_3C=C), 1.75 br. s (3H, CH_3–C^3), 2.0 m (44H, CH_2C=C), 4.13 d (2H, CH_2O, J = 7), 5.1 m (11H, HC=C), 5.42 br. t (1H, HC^2, J = 7). Mass spectrum (m/z): 904 (M^+), 886 $(M-H_2O)^+$, 838, 819, 751, 684, 683, 614, 546, 478, 477, 409, 341, 273, 272, 271, 203, 137, 135, 69, 68. Calculated for $C_{65}H_{108}O$; Mol. wt. 905.6.

4-Phenylsulfonyl-3-methyleicos-2Z-en-1-ol (XI). A 14.5 ml portion of a 2M solution of n-BuLi in hexane (29 mmoles) was added over a period of 10 min to a stirred (–70°C, Ar) solution of 3.27 g (14.5 mmoles) of (VI) in 70 ml of THF and 4 ml of HMPA, and then after 20 min, a solution of 2.6 g (8.5 mmoles) of cetyl bromide [14] in 20 ml of THF was added at –25°C in the course of 10 min. The reaction mixture was allowed to stand at –25°C for 2.5 h and then was treated as described for (X). Yield ∿6 g of a compound, which was chromatographed on 300 g of SiO_2. Gradient elution from hexane to ether (up to 60% of the latter) gave 3.32 g (87%) of (XI) in the form of colorless prisms, mp 50-51°C (pentane). IR spectrum (ν, cm^{-1}): 740, 1010, 1090, 1150, 1295, 1310, 1320, 1450, 1460, 1540, 1650, 2860, 2930, 3520, 3610. PMR spectrum (δ, ppm, J, Hz): 0.87 t (3H, CH_3, J = 7), 1.2 m (28H, CH_2), 1.80 br. s (3H, CH_3–C^3), 1.90 m (2H, HC^5), 3.84 m (2H, CH_2O), 4.09 d. d (1H, HCS, J = 5.5 and 11), 5.87 br. t (1H, HC^2, J = 7.5), 7.5-7.9 m (5H, C_6H_5). Found: S 6.74%; 309 $(M-C_6H_5SO_2)^+$. $C_{27}H_{46}O_3S$. Calculated: S 7.11%, Mol. wt. 450.7.

3-Methyleicos-2Z-en-1-ol (II). In a similar way as described for (I), from 0.71 g (1.58 mmoles) of (XI), 0.29 g (12.6 mg·atom) of Na in 30 ml of NH_3, 20 ml of hexane and 10 ml of THF, 0.6 g of a compound was obtained, which was chromatographed on 30 g of SiO_2. Gradient elution from hexane to ether (up to 30% of the latter) gave 0.29 g (59%) of (II) in the form of a colorless oil, R_f 0.63 containing (PMR) ∿35% of the regioisomer (XIV). IR spectrum (ν, cm^{-1}): 735, 995, 1100, 1210, 1380, 1455, 1465, 1510, 1520, 1540, 1560, 1640, 2850, 2930, 3610. PMR spectrum of (II) (δ, ppm, J, Hz): 0.89 t (3H, CH_3, J = 6.5), 1.3 m (28H, CH_2), 1.59 br. s (3H, CH_3–C^3), 1.60 m (2H, HC^5), 2.05 m (2H, HC^4), 4.12 d (2H, CH_2O, J = 7.5), 5.41 br. t (1H, HC^2, J = 7.5). High resolution mass spectrum for m/z 310 (M^+) — Found: 310.32116; Calculated for $C_{21}H_{42}O$: 310.32354.

4-Phenylsulfonyl-3,7-dimethyltetracosa-2Z,6Z-dien-1-ol (XII). In the same way as described for (XI), from 0.53 g (2.34 mmoles) of (VI), 2.35 ml of 2 M solution of n-BuLi in hexane (4.7 mmoles) and a bromide freshly prepared according to [13] from 0.43 g (1.38 mmoles) of (II) in 10 ml of THF and 1 ml of HMPA, 1.1 g of a compound was obtained, which was chromatographed on 60 g of SiO_2. Gradient elution from hexane to ether (up to 60% of the latter) gave 0.33 g (46%) of (XII) in the form of a colorless oil, R_f 0.40 (ether–hexane, 2:1). IR spectrum (ν, cm^{-1}): 735, 1010, 1085, 1150, 1310, 1325, 1380, 1450, 1460, 1540, 1555, 1665, 2860, 2930, 3520, 3610. PMR spectrum (δ, ppm, J, Hz): 0.88 t (3H, CH_3, J = 6.5), 1.3 m (28H, CH_2), 1.6 m (2H, HC^9), 1.62 br. s (3H, CH_3–C^7), 1.85 m (2H, HC^8), 1.87 br. s (3H, CH_3–C^3), 2.59 m (2H, HC^5), 3.90 m (2H, CH_2O), 4.10 m (1H, HCS), 4.79 br. t (1H, HC^6, J = 6), 5.93 m (1H, HC^2), 7.6-7.9 m (5H, C_6H_5). Found: S 5.79%; 377 $(M-C_6H_5SO_2)^+$. $C_{32}H_{54}O_3S$. Calculated: S 6.18%, Mol. wt. 518.9.

3,7-Dimethyltetracosa-2Z,6Z-dien-1-ol (III). In a similar way as described for (I), from 0.3 g (0.58 mmole) of (XII), 0.11 g (4.78 mg·atom) of Na in 10 ml of NH_3, 10 ml of hexane and 3 ml of THF, 0.25 g of a compound was obtained, which was chromatographed on 20 g of SiO_2. Gradient elution from hexane to ether (up to 20% of the latter) gave 60 mg (27%) of (III) in the form of a colorless oil, R_f 0.64, containing (PMR) ∿30% of homoallyl alcohol (XV). IR spectrum (ν, cm^{-1}): 960, 1000, 1060, 1185, 1310, 1380, 1470, 1670, 2855, 2930, 3570, 3630. PMR spectrum of (III) (δ, ppm, J, Hz): 0.87 t (3H, CH_3, J = 7), 1.3 m (28H, CH_2), 1.67 br. s (3H, CH_3–C^7), 1.70 m (2H, HC^9), 1.75 br. s (3H, CH_3–C^3), 2.0 m (6H, CH_2C=C), 4.09 d (2H, CH_2O, J = 7.5), 5.09 br. t (1H, HC^6, J = 7), 5.45 br. t (1H, HC^2, J = 7.5). High resolution mass spectrum for m/z 378 (M^+) — Found: 378.38821; Calculated for $C_{26}H_{50}O$: 378.38616.

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ADDITION OF BENZHYDROXAMIC ACID TO ALKOXYETHYLENES

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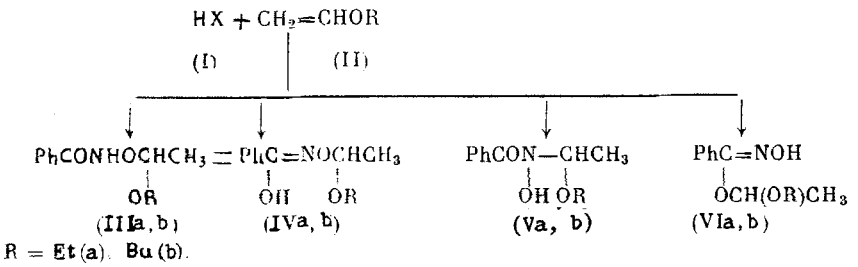
A method for the isolation of 1-alkoxyethyl esters of benzhydroxamic acid, based on the addition of this acid to alkoxyethylenes in the presence of the catalyst HBr, was developed. The possible structure of the adducts is discussed.

The interaction of hydroxamic acids with alkyl vinyl ethers has not been studied up to now. Taking the example of benzhydroxamic acid (I), we showed that the indicated acids undergo addition to the activated double bond of the alkoxyethylenes (II) according to the Markownikoff rule. The reaction proceeds at 20°C in the absence of traces of moisture. The catalyst for this was the solution of hydrogen bromide in acetone or benzene, prepared directly before application by means of the addition of the calculated amount of bromine to the acetone or the solution of the vinyl alkyl ether in benzene. The amount of the HBr introduced into the reaction comprised 5-10 mole % in relation to (I). The molar ratio (I):(II) is 1:10-20.

Since the anion (X^-) of hydroxamic acid may theoretically exist in four tautomeric forms (X^- = $PhCONHO^-$, $PhC(OH)=NO^-$, $PhC(O)N^-OH$, $PhC(O^-)=NOH$), it is difficult to assign, a priori, one of the structures (III)-(VI) to the adducts which are formed.



However, it can be concluded from the data of mass spectrometry and IR spectroscopy, taking into consideration the ^1H, ^{13}C, and ^{14}N NMR spectra, that the isolated (1-alkoxy)ethyl esters of hydroxamic acid occur mainly in the form of the hydroxamates (III).

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