SYNTHESIS AND ABSOLUTE CONFIGURATION AT $C_{(20)}$ OF 21,26,27-TRINOR-5α-CHOLESTAN-25→20-OLIDE DERIVATIVES*

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Reaction of the aldehyde *I* with 4-(methoxymethoxy)butylmagnesium chloride yielded compounds *II* and *VII* which were converted into diols *III* and *VIII*, respectively. By oxidation of the diols *III* and *VIII* were obtained saturated lactones *XVI* and *XX*, respectively, which were converted into unsaturated lactones *XIX* and *XXIII*, respectively. On the basis of chemical correlation with methyl ethers *XIV* and *XV* of a known configuration at $C_{(20)}$, the compounds *II*—*VI*, *XII*, *XVI*—*XIX* were assigned the 20*R*-configuration whereas 20*S* configuration was established for the compounds *VII*—*XI*, *XIII* and *XX*—*XXIII*.

In our preceding communications[1,2] we described the synthesis of 21-nor-5α-cholane derivatives with a 24→20-olide γ-lactone ring. In the present paper there is reported a synthesis of homologous δ-lactones derived from 21,26,27-trinor-5α-cholestan-25→20-olide.

For the construction of the side chain in position 17β, we utilized a reaction of the known[1] aldehyde *I* with 4-(methoxymethoxy)butylmagnesium chloride. The reaction yielded two main products: *II* (yield 28%), and *VII* (yield 38%). Their structures follow from spectral measurements: The IR spectra show bands of a hydroxyl group ($\nu_{(OH)}$ = 3 635 and 3 500 cm^{-1}), in the ^{1}H-NMR spectrum signals (3·33 s 3 H and 4·60 s 2 H) characteristic of a methoxymethoxy grouping are present; the mass spectrum corroborates that the grouping —$CH_2CH_2CH_2CH_2OCH_2OCH_3$ is attached to the aldehyde *I* molecule. Configuration at $C_{(20)}$ of the compounds *II* and *VII* has been established by the following correlation with the methyl ethers *XIV* and *XV* bearing the known[3] configuration at $C_{(20)}$. Reaction of the compound *II* with sodium hydride and methyl iodide in tetrahydrofuran yielded the methyl ether *IV* in which the methoxymethyl protecting group was split off by the action of hydrochloric acid in methanol–benzene[4] to give the hydroxy derivative *V*. Shortening of the carbon side chain was achieved by oxidative decarboxylation[5,6] of the acid *VI* which was obtained from the hydroxy derivative *V* by Jones' oxidation. The

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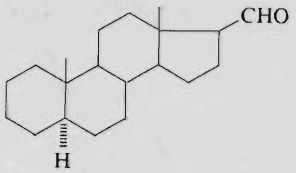
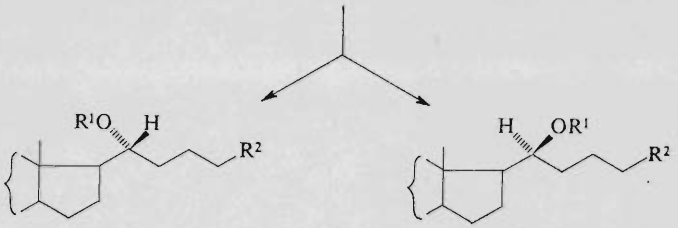
least polar compound isolated in 22% yield was the olefin *XII*. The terminal position of the double bond was confirmed by the IR spectrum (bands at 992 and 920 cm^{-1} characteristic of a vinyl group) and by the ^{1}H-NMR spectrum (for signals *cf.* Table I). Hydrogenation of the olefin *XII* provided the saturated methyl ether *XIV*

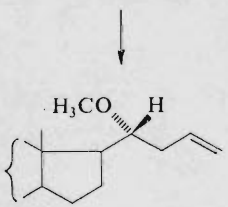


I

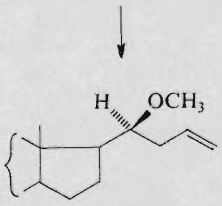


II, $R^1 = H$, $R^2 = CH_2OCH_2OCH_3$
III, $R^1 = H$, $R^2 = CH_2OH$
IV, $R^1 = CH_3$, $R^2 = CH_2OCH_2OCH_3$
V, $R^1 = CH_3$, $R^2 = CH_2OH$
VI, $R^1 = CH_3$, $R^2 = COOH$

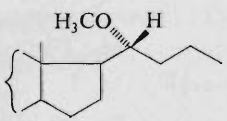
VII, $R^1 = H$, $R^2 = CH_2OCH_2OCH_3$
VIII, $R^1 = H$, $R^2 = CH_2OH$
IX, $R^1 = CH_3$, $R^2 = CH_2OCH_2OCH_3$
X, $R^1 = CH_3$, $R^2 = CH_2OH$
XI, $R^1 = CH_3$, $R^2 = COOH$



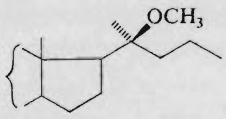
XII



XIII



XIV

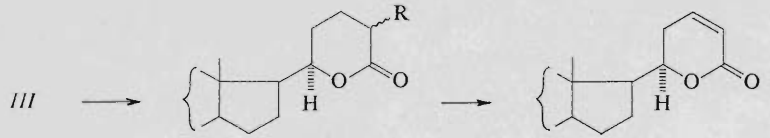


XV

of 20*R*-configuration as a sole isolated product. The same procedure was applied to *VII* and, through the intermediates *IX*, *X*, *XI* and *XIII*, yielded the saturated methyl ether *XV* of 20*S*-configuration.

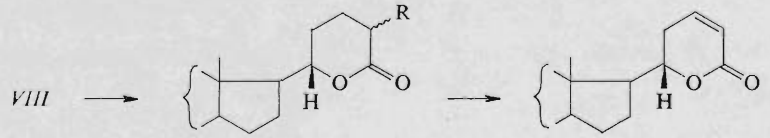
Removal of the methoxymethyl protecting group from *II* was achieved by treatment with hydrochloric acid in methanol–benzene and provided the diol *III*. Under the same conditions, the compound *VII* gives rise to the diol *VIII*. Oxidation of the diol *III* with silver carbonate on Celite[7] furnished a complex mixture. Chromatography on silica gel column led to isolation in 40% yield of the lactone *XVI* with the 20*R* configuration. On the other hand, oxidation of the epimeric diol *VIII* yielded practically pure lactone *XX* of the 20*S*-configuration and was isolated in 73% yield after crystallization from light petroleum.

For preparing the unsaturated lactones *XIX* and *XXIII*, a procedure[8] was applied using sulfenylation and dehydrosulfenylation of a saturated lactones *XVI* and *XX*. The anion prepared from the lactone *XVI* reacted with dimethyl disulfide to give



XVI, R = H
XVII, R = SCH_3
XVIII, R = $SOCH_3$

XIX



XX, R = H
XXI, R = SCH_3
XXII, R = $SOCH_3$

XXIII

the α-methylthiolactone *XVII*. As shown by thin layer chromatography, the reaction gave two isomers differing in configuration at $C_{(24)}$. Oxidation of these compounds with 3-chloroperoxybenzoic acid at −78°C gave the lactone *XVIII*; the latter was heated with calcium carbonate in toluene to furnish the unsaturated lactone *XIX* in 34% overall yield (based on the starting lactone *XVI*). Analogous treatment of the saturated lactone *XX* proceeded through the intermediates *XXI* and *XXII* and provided the unsaturated lactone *XXIII* in 30% overall yield. The structure of both unsaturated lactones is corroborated by IR and ^{1}H-NMR spectra (Table I) showing the presence of signals characteristic of α,β-unsaturated six-membered lactones[9].

EXPERIMENTAL

Melting points were determined on a Kofler block. Optical rotations were measured in chloroform at 25°C with an error of ±3°. The infrared spectra were recorded on a Perkin–Elmer model 580 spectrometer in tetrachloromethane unless stated otherwise. The CD spectra were measured on a Dichrographe II (Jouan-Roussel) in dioxane. The mass spectra were recorded on a AEI MS 901 mass spectrometer. Silica gel according to Pitra (60—120 μm) and neutral aluminum oxide (Reanal, grade II) were used for column chromatography whereas silica gel G according to Stahl (Woelm) was used for thin layer chromatography. Plates with 200 × 200 × × 0·7 mm silica gel layer were used for preparative thin layer chromatography. Analytical samples were dried at 50°C and 26 Pa for 12 h. The identity of samples prepared on different routes was checked by comparison of their IR and ^{1}H-NMR spectra, by thin layer chromatography and mixture melting point determination.

(20*R*)-25-Methoxymethoxy-21,26,27-trinor-5α-cholestan-20-ol (*II*)

A solution of the aldehyde[1] *I* (5 g) in benzene (90 ml) was added in the course of 30 min into a stirred solution of 4-(methoxymethoxy)butylmagnesium chloride (c = 1·6 mol l^{-1}) in tetra-

TABLE I

Characteristic parameters of ^{1}H-NMR spectra

Compound[a]	18-H_3	19-H_3	20-H	23-H	24-H	25-H_2
II[b]	0·73 s	0·77 s	3·46 mt[c]	[d]	[d]	3·46 mt[c]
IV[b,e]	0·67 s	0·78 s	3·17 mt	[d]	[d]	3·51 mt
V[e]	0·67 s	0·78 s	3·17 mt[f]	[d]	[d]	3·62 bt[g]
VII[b]	0·65 s	0·77 s	3·50 mt[c]	[d]	[d]	3·50 mt[c]
IX[b,e]	0·62 s	0·76 s	3·06 mt	[d]	[d]	3·50 mt
X[e]	0·65 s	0·79 s	3·12 mt[h]	[d]	[d]	3·65 bt[i]
XII[e]	0·72 s	0·82 s	3·23 mt	4·8—6·2 brmt		—
XIII[e,j]	0·63 s	0·76 s	3·18 mt	4·8—6·2 brmt		—
XVI[k]	0·77 s	0·80 s	4·23 mt[l]	[d]	[d]	—
XIX[m]	0·70 s	0·79 s	4·34 mt[l]	6·85 mt[n]	6·01 ddd[o]	—
XX[p]	0·69 s	0·78 s	4·22 mt[l]	[d]	[d]	—
XXIII[q]	0·73 s	0·77 s	4·37 mt[l]	6·83 mt[n]	5·97 dt[r]	—

[a] The spectra were measured in deuteriochloroform with tetramethylsilane as internal reference on Tesla B 476 (60 MHz) instrument. Compounds *X*, *XIX* and *XX* were measured on Varian HA-100 (100 MHz) instrument. Chemical shifts are given in ppm (δ-scale). All values were obtained by first order analysis. [b] Other signals: 3·32—3·35 s and 4·59—4·61 s (CH_3OCH_2). [c] Overlapped signals. [d] Undeterminable value. [e] Other signal 3·26—3·32 (CH_3O). [f] $W \approx 20$ Hz. [g] $J \approx 6$ Hz. [h] $W \approx 22$ Hz. [i] $J \approx 6{\cdot}5$ Hz. [j] Other signal: 2·30 bt, $J \approx 6$ Hz (22-H_2). [k] Other signal: 2·47 mt (24-H_2). [l] $W = 25$ Hz. [m] Other signal: 2·41 mt (22-H_2). [n] $W = 18$ Hz. [o] $J_{23,24} = 9{\cdot}7$ Hz; $J_{24,22} = 2{\cdot}5 + 1{\cdot}7$ Hz. [p] Other signal: 2·49 mt (24-H_2). [q] Other signal: 2·27 mt (22-H_2). [r] $J_{23,24} = 10$ Hz, $J_{24,22} = 1{\cdot}7$ Hz.

hydrofuran[4] (30 ml) kept at 0°C. The reaction was carried out under argon. Stirring was continued at 0°C for an additional 30 min, the mixture poured in a saturated aqueous ammonium chloride solution (500 ml), the product taken up in ether, the extract washed with saturated aqueous ammonium chloride solution, water, dried with anhydrous sodium sulfate and the solvent removed under reduced pressure. The residue (10·5 g) was chromatographed on a silica gel (300 g) column. Light petroleum–ether (80 : 2) eluted the alcohol *II* (2 g), m.p. 107—109°C (ether), $[\alpha]_D$ +12° (*c* 1·6). IR spectrum: 3 635, 3 500 (OH) cm^{-1}. Mass spectrum (*m/z*): 406 (M^+), 388 ($M^+ - H_2O$), 374 ($M^+ - CH_3OH$), 343 ($M^+ - H_2O - CH_2OCH_2$), 288 ($M^+ - CH_2CH_2CH_2CH_2OCH_2OCH_3$). For $C_{26}H_{46}O_3$ (406·7) calculated: 76·79% C, 11·40% H; found: 76·52% C, 11·37% H.

(20*R*)-21,26,27-Trinor-5α-cholestan-20,25-diol (*III*)

Concentrated hydrochloric acid (2·5 ml) was added to a solution of the protected diol *II* (1 g) in a mixture of benzene (50 ml) with methanol (100 ml) and the solution was refluxed for 2 h. Evaporation gave a residue which was dissolved in ether, the solution washed with aqueous potassium hydrogen carbonate, water, and dried with anhydrous sodium sulfate. Evaporation of the solvent *in vacuo* yielded chromatographically pure diol *III* (820 mg), m.p. 138—140°C (ether), $[\alpha]_D$ +17° (*c* 1·7). IR spectrum (chloroform): 3 622, 3 400 (OH) cm^{-1}. For $C_{24}H_{42}O_2$ (362·6) calculated: 79·50% C, 11·68% H; found: 79·71% C, 12·01% H.

(20*R*)-20-Methoxy-21,26,27-trinor-5α-cholestan-25-ol (*V*)

A solution of the alcohol *II* (540 mg) in tetrahydrofuran (10 ml) and methyl iodide (6 ml) were added to a stirred suspension of sodium hydride (540 mg) in tetrahydrofuran (20 ml). The mixture was stirred under argon at 41°C for 9 h, decomposed with water, the product taken up in ether, the extract washed with dilute hydrochloric acid, water, potassium hydrogen carbonate solution, water, dried with anhydrous sodium sulfate and the solvent evaporated. The residue (550 mg) was shown to be pure methyl ether *IV* by thin layer chromatography and was dissolved in a mixture of methanol (50 ml) and benzene (20 ml). After addition of concentrated hydrochloric acid (1 ml), the mixture was refluxed for 60 min and evaporated *in vacuo*. The residue was dissolved in ether and the solution was washed with potassium hydrogen carbonate solution, water, dried with anhydrous sodium sulfate and the solvent evaporated to afford chromatographically pure alcohol *V* (500 mg), $[\alpha]_D$ +4° (*c* 1·9). IR spectrum (chloroform): 3 620, 3 420 (OH) cm^{-1}. For $C_{25}H_{44}O_2$ (376·6) calculated: 79·73% C, 11·78% H; found: 79·48% C, 11·67% H.

(20*S*)-25-Methoxymethoxy-21,26,27-trinor-5α-cholestan-20-ol (*VII*)

Further elution with light petroleum–ether (75 : 25) (preparation of the alcohol *II*) provided the alcohol *VII* (2·7 g) m.p. 76—78°C (light petroleum); $[\alpha]_D$ +9° (*c* 2·1). IR spectrum: 3 635, 3 500 (OH) cm^{-1}. Mass spectrum (*m/z*): 406 (M^+), 388 ($M^+ - H_2O$), 374 ($M^+ - CH_3OH$), 343 ($M^+ - H_2O - CH_2OCH_3$), 288 ($M^+ - CH_2CH_2CH_2CH_2OCH_2OCH_3$). For $C_{26}H_{46}O_3$ (406·7) calculated: 76·79% C, 11·40% H; found: 76·50% C, 11·61% H.

(20*S*)-21,26,27-Trinor-5α-cholestan-20,25-diol (*VIII*)

The preparation of the diol *VIII* from the protected diol *VII* was carried out in the same manner as preparation of *III* from *II*. Evaporation of the solvent under reduced pressure yielded chromatographically pure diol *VIII* (800 mg), m.p. 146—147°C (benzene), $[\alpha]_D$ +5° (*c* 1·9). IR spectrum

(chloroform): 3 625, 3 375, 3 270 shoulder (OH) cm^{-1}. For $C_{24}H_{42}O_2$ (362·6) calculated: 79·50% C, 11·68% H; found: 79·71% C, 12·01% H.

(20*S*)-20-Methoxy-21,26,27-trinor-5α-cholestan-25-ol (*X*)

Preparation of the alcohol *X* from the alcohol *VII* through the intermediate *IX* was carried out in the same manner as preparation of the alcohol *V* from *II*. Evaporation of the solvent under reduced pressure gave the alcohol *X* (500 mg), m.p. 112—114°C (ether), $[\alpha]_D$ +19·5° (*c* 2·4). IR spectrum: 3 635, 3 410 (OH) cm^{-1}. For $C_{25}H_{44}O_2$ (376·6) calculated: 79·73% C, 11·78% H; found: 79·91% C, 12·05%H.

(20*R*)-Methoxy-21-nor-5α-chol-23-en (*XII*)

Jones' reagent (6 ml) was added to a solution of the alcohol *V* (400 mg) in acetone (50 ml) the mixture was stirred at room temperature for 15 min, diluted with ether (200 ml), washed with saturated aqueous solution of ammonium sulfate and dried with anhydrous sodium sulfate. Evaporation of the solvent under reduced pressure gave the oily acid *VI* (390 mg). IR spectrum (chloroform): 3 400—2 400, 1 710 (COOH) cm^{-1}. The acid (380 mg) was dissolved in benzene (26 ml) and pyridine (0·06 ml), cupric acetate monohydrate (50 mg) and lead tetraacetate (880 mg) were added in succession. After 5 h stirring and refluxing under argon the mixture was filtered through a column of aluminum oxide (20 g). The column was eluted with benzene, the solvent removed and the residue (190 mg) chromatographed on two preparative silica gel plates in light petroleum–ether (98 : 2). The zones containing the least polar compounds were combined and eluted with dichloromethane to yield the oily olefin *XII* (80 mg), $[\alpha]_D$ −21° (*c* 1·8). IR spectrum: 2 820 shoulder, 1 093 (OCH_3), 3 082, 1 639, 1 413, 992, 920 ($C{=}CH_2$) cm^{-1}. For $C_{24}H_{40}O$ (344·6) calculated: 83·66% C, 11·70% H; found: 83·98% C, 11·63% H.

(20*S*)-Methoxy-21-nor-5α-chol-23-ene (*XIII*)

Preparation of the olefin *XIII* from the alcohol *X via* the acid *XI* was carried out in the same manner as preparation of *XII* from *V*. The yield of the oily olefin *XIII* amounted to 77 mg, $[\alpha]_D$ +2° (*c* 1·0). IR spectrum: 2 820 shoulder, 1 093 (OCH_3), 3 080, 1 640, 1 415, 993, 914, ($C{=}CH_2$) cm^{-1}. For $C_{24}H_{40}O$ (344·6) calculated: 83·66% C, 11·70% H; found: 83·41% C, 11·75% H.

(20*R*)-20-Methoxy-21-nor-5α-cholane (*XIV*)

10% Palladium on carbon (40 mg) was added to a solution of the olefin *XII* (80 mg) in ethyl acetate (20 ml) and shaken under hydrogen for 2 h. The mixture was then filtered and the solvent evaporated under reduced pressure. The residue was chromatographed on a preparative silica gel plate using light petroleum–ether (96 : 4) for development. In this manner, 70 mg of the methyl ether *XIV* was obtained, m.p. 65—68° (ether) $[\alpha]_D$ +2° (*c* 2·1), identical with an authentic sample[3].

(20*S*)-20-Methoxy-21-nor-5α-cholane (*XV*)

A solution of the olefin *XIII* (40 mg) in methanol–ethyl acetate (1 : 1, 20 ml) and 10% palladium on carbon (40 mg) were shaken under hydrogen for 2 h, filtered and the solvent evaporated. Chromatography of the residue on a preparative silica gel plate in light petroleum–ether (96 : 4)

system yielded the saturated methyl ether *XV* (35 mg), m.p. 67—70°C (ether), $[\alpha]_D$ +21° (*c* 1·5), identical with an authentic sample[3].

(20*R*)-21,26,27-Trinor-5α-cholestan-25→20-olide (*XVI*)

Silver carbonate on celite (6 g, ref.[7]) was suspended in benzene (60 ml), 10 ml of benzene was distilled off while stirring and a solution of the diol *III* (500 mg) in benzene (40 ml) was added. Stirring was continued and the mixture was refluxed for 5 h, the mixture filtered through celite, the solvent evaporated and the residue chromatographed on a column of silica gel (50 g). A mixture of light petroleum–ether–ethyl acetate (96 : 2 : 2) eluted the lactone *XVI* (200 mg), m.p. 158—161°C (light petroleum), $[\alpha]_D$ −5·5° (*c* 1·8). IR spectrum: 1 738 (C=O) cm^{-1}. CD spectrum: $\Delta\varepsilon$ +0·26 (235 nm). Mass spectrum (*m/z*): 358 (M^+). For $C_{24}H_{38}O_2$ (358·6) calculated: 80·39% C, 10·68% H; found: 80·26% C, 10·90% H.

(20*R*)-21,26,27-Trinor-5α-cholest-23-en-25→20-olide (*XIX*)

n-Butyllithium in n-hexane (0·92 ml $c = 1{\cdot}6$ mol l^{-1}) was added to a stirred solution of diisopropylamine (152 mg) in tetrahydrofuran (2·5 ml) cooled to −78°C and kept under argon. After standing at −78°C for 15 min, a solution of the lactone *XVI* (150 mg) in tetrahydrofuran (4 ml) was added and the mixture allowed to reach the room temperature which took 30 min. Dimethyl disulfide (122 mg) was added and stirring continued for 1 h, the mixture poured in dilute hydrochloric acid, the product taken up in ether, the extract washed with a potassium hydrogen carbonate solution, water, dried with anhydrous sodium sulfate and the solvent removed under reduced pressure. The residue was chromatographed on two preparative silica gel plates using light petroleum–acetone (9 : 1). The zones containing two least polar compounds were collected and eluted with acetone to give a mixture (100 mg) of two isomeric compounds possessing the structure *XVII* and differing in configuration at $C_{(24)}$. A solution of 3-chloroperoxybenzoic acid (85 mg, 85% content) in dichloromethane (1·5 mol) was added to a solution of the compound *XVII* (100 mg) in dichloromethane (20 ml) chilled at −78°C and the mixture was stirred at −78°C for 10 min and poured into a 10% sodium sulfite solution. The product was taken up in ether, the extract washed with potassium hydrogen carbonate solution, water, and dried with anhydrous sodium sulfate. Evaporation of the solvents under reduced pressure gave a residue (100 mg) which was dissolved in toluene (25 ml) and after addition of calcium carbonate (500 mg) refluxed under argon with stirring for 4 h. Inorganic salts were removed by filtration through a kieselguhr layer. Elution with ether yielded a crude product which was purified by chromatography on two preparative silica gel plates using light petroleum–acetone (9 : 1). The yield of the unsaturated lactone *XIX* amounted to 50 mg, m.p. 179—181°C (light petroleum), $[\alpha]_D$ +65° (*c* 1·8). IR spectrum: 1 736 (C=O), 1 620 (C=C) cm^{-1}. CD spectrum: $\Delta\varepsilon$ +1·46 (265 nm). Mass spectrum (*m/z*): 356 (M^+). For $C_{24}H_{36}O_2$ (356·6) calculated: 80·85% C, 10·80% H; found: 80.61% C, 10·23% H.

(20*S*)-21,26,27-Trinor-5α-cholestan-25→20-olide (*XX*)

Preparation of the lactone *XX* from the diol *VIII* was carried out in the same manner as preparation of *XVI* from *III*. Crystallization of the crude product from light petroleum yielded the lactone *XX* (360 mg), m.p. 172—174°C, $[\alpha]_D$ − 1° (*c* 2·6). IR spectrum: 1 742 (C=O) cm^{-1}. CD spectrum: $\Delta\varepsilon$ −0·29 (235 nm). Mass spectrum (*m/z*): 358 (M^+). For $C_{24}H_{38}O_2$ (358·6) calculated: 80·39% C, 10·68% H; found: 80·27% C, 10·79% H.

(20*S*)-21,26,27-Trinor-5α-cholest-23-en-25→20-olide (*XXIII*)

Preparation of *XXIII* from *XX* was carried out through the intermediates *XXI* and *XXII* in the same manner as preparation of *XIX* from *XVI*. The procedure yielded 45 mg of *XXIII*, m.p. 187–190°C (ether), $[\alpha]_D$ −15° (*c* 0·9). IR spectrum: 1 737 (C=O), 1 630 (C=C) cm^{-1}. CD spectrum: $\Delta\varepsilon$ −2·30 (265 nm). Mass spectrum (*m/z*): 356 (M^+). For $C_{24}H_{36}O_2$ (356·6) calculated: 80·85% C, 10·18% H; found: 80·57% C, 10·14% H.

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