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Structures of 3*β*-Tetrahydropyranyloxy-5*α*-cholesta-20(21),24-diene and 3*β*-Tetrahydropyranyloxy-21-nor-5*α*-ergost-24-en-20-one

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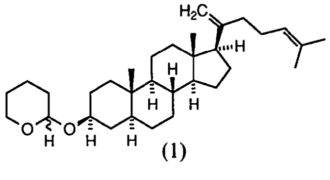
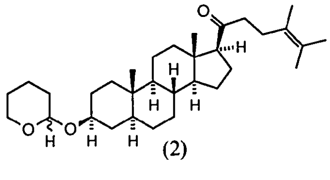
Abstract. 3*β*-Tetrahydropyranyloxy-5*α*-cholesta-20(21),24-diene: $C_{32}H_{52}O_2$, $M_r = 468{\cdot}77$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}710\,(4)$, $b = 11{\cdot}361\,(4)$, $c = 37{\cdot}812\,(11)$ Å, $V = 2882\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}08$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54178$ Å, $\mu = 4{\cdot}60$ cm^{-1}, $F(000) = 1040$, $T = 224$ K, final $R = 0{\cdot}088$ for 1729 unique observed reflections. 3*β*-Tetrahydropyranyloxy-21-nor-5*α*-ergost-24-en-20-one: $C_{32}H_{52}O_3$, $M_r = 484{\cdot}77$, triclinic, $P1$, $a = 6{\cdot}640\,(2)$, $b = 9{\cdot}589\,(2)$, $c = 12{\cdot}202\,(3)$ Å, $\alpha = 111{\cdot}33\,(2)$, $\beta = 101{\cdot}22$, $\gamma = 90{\cdot}27\,(2)°$, $V = 707{\cdot}4\,(3)$ Å^3, $Z = 1$, $D_x = 1{\cdot}14$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}8$ cm^{-1}, $F(000) = 268$, $T = 225$ K, final $R = 0{\cdot}058$ for 2208 unique observed reflections. The configuration at C(17) of these synthetic sterol derivatives, which had been uncertain, is unambiguously established to be 'normal' (possessing a 17*α*-H).

Introduction. The title compounds, 3*β*-tetrahydropyranyloxy-5*α*-cholesta-20(21),24-diene (1) (Rahman & Pascal, 1990) and 3*β*-tetrahydropyranyloxy-21-nor-5*α*-ergost-24-en-20-one (2), are key intermediates in the syntheses of certain inhibitors of ergosterol biosynthesis in parasitic protozoa. During the review of some of the synthetic and biological work for publication, a referee expressed concern that epimerization at C(17) might have occurred during the synthesis of compound (1), which employs strong base in two steps when a carbonyl group is present at C(20). In order to establish unequivocally the configuration at C(17), we performed single-crystal analyses of compound (1) and the related compound (2) which is prepared in a similar way.



(1)



(2)

Experimental. Compound (1): A single crystal (from ethanol) with approximate dimensions 0·05 × 0·15 × 0·52 mm was used for data collection. Intensity measurements were made on a Nicolet *R*3*m* diffractometer, equipped with an LTII low-temperature device, at 224 K by using graphite-monochromated Cu *Kα* radiation and a variable-speed 0·90° *ω* scan. Lattice parameters were determined by a least-squares fit of the 2θ values of 19 reflections having

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$40 \le 2\theta \le 50^\circ$. Three standard reflections used to monitor the data collection (0,4,13, 2,2,12, 312) did not show significant deviations within the period of data collection. 2311 reflections with $3 \le 2\theta \le 114^\circ$ were collected ($0 \le h \le 8$, $0 \le k \le 12$, $0 \le l \le 41$). Systematically absent reflections were eliminated to give 2281 unique data, of which 1729 were considered to be observed [$|F_o| > 3\sigma(F_o)$]. Raw intensities were reduced to structure-factor amplitudes by correction for scan speed, background, Lorentz and polarization effects. The structure was solved by direct methods using the *SHELXTL* package of programs (Sheldrick, 1980). Atomic scattering factors were those incorporated in *SHELXTL*. Of the 34 non-H atoms, 30 positions were derived from an *E* synthesis. A difference Fourier map then revealed the remaining non-H atoms and several additional large peaks which were determined to be due to a second diastereomer [with the opposite configuration at C(29) of the tetrahydropyran (THP) group]. The disordered THP groups were refined with a 1:1 occupancy ratio, isotropic temperature factors and constrained C—C bond distances. All other non-H atoms were refined with anisotropic temperature factors; the H atoms, except those of the disordered THP ring, were included at idealized positions [C—H 0·96 Å, *X*—C—H 109 or 120°, $B(\mathrm{H}) = 1{\cdot}2 \times B(\mathrm{C})$]. 301 parameters were refined by minimizing $\sum w(F_o + F_c)^2$; $R = 0{\cdot}088$, $wR = 0{\cdot}096$, $w = [\sigma^2(F_o) + 0{\cdot}0015F^2]^{-1}$, $S = 1{\cdot}93$, maximum $(\Delta/\sigma) = 0{\cdot}094$, average $(\Delta/\sigma) = 0{\cdot}012$, $\Delta\rho = -0{\cdot}25$ to $+0{\cdot}30$ e Å^{-3}.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (1); *e.s.d.'s are given in parentheses*

	x	*y*	*z*	*U*
C(1)	0·7265 (11)	0·4209 (7)	0·8273 (2)	0·064 (3)*
C(2)	0·7700 (12)	0·4056 (7)	0·8669 (2)	0·075 (3)*
C(3)	0·7063 (11)	0·5114 (7)	0·8882 (2)	0·068 (3)*
C(4)	0·4918 (12)	0·5413 (6)	0·8810 (2)	0·065 (3)*
C(5)	0·4470 (11)	0·5569 (6)	0·8416 (2)	0·056 (2)*
C(6)	0·2338 (11)	0·5983 (6)	0·8347 (2)	0·068 (3)*
C(7)	0·1970 (11)	0·6220 (6)	0·7961 (2)	0·058 (3)*
C(8)	0·2660 (10)	0·5206 (5)	0·7716 (2)	0·049 (2)*
C(9)	0·4789 (10)	0·4831 (5)	0·7800 (2)	0·049 (2)*
C(10)	0·5039 (11)	0·4481 (6)	0·8192 (2)	0·054 (2)*
C(11)	0·5590 (11)	0·3934 (6)	0·7535 (2)	0·061 (3)*
C(12)	0·5239 (10)	0·4273 (6)	0·7144 (2)	0·057 (3)*
C(13)	0·3088 (11)	0·4549 (6)	0·7069 (2)	0·053 (2)*
C(14)	0·2450 (11)	0·5544 (5)	0·7323 (2)	0·052 (2)*
C(15)	0·0496 (11)	0·5974 (6)	0·7176 (2)	0·064 (3)*
C(16)	0·0594 (11)	0·5762 (7)	0·6780 (2)	0·065 (3)*
C(17)	0·2616 (12)	0·5171 (5)	0·6717 (2)	0·057 (2)*
C(18)	0·1830 (12)	0·3445 (6)	0·7116 (2)	0·069 (3)*
C(19)	0·3798 (12)	0·3412 (5)	0·8292 (2)	0·065 (3)*
C(20)	0·2857 (13)	0·4401 (7)	0·6391 (2)	0·066 (3)*
C(21)	0·1398 (15)	0·3749 (7)	0·6261 (2)	0·091 (4)*
C(22)	0·4899 (15)	0·4395 (7)	0·6223 (2)	0·093 (4)*
C(23)	0·5221 (16)	0·5511 (11)	0·5988 (3)	0·121 (5)*
C(24)	0·7207 (19)	0·5659 (15)	0·5836 (3)	0·156 (7)*
C(25)	0·7951 (19)	0·6533 (11)	0·5661 (2)	0·120 (5)*
C(26)	1·0017 (17)	0·6573 (16)	0·5530 (4)	0·190 (8)*
C(27)	0·6690 (22)	0·7558 (10)	0·5561 (4)	0·173 (8)*
O(28)	0·7377 (10)	0·4874 (5)	0·9246 (1)	0·092 (2)*
C(29)	0·7181 (27)	0·5719 (14)	0·9491 (4)	0·087 (5)
C(29′)	0·8938 (30)	0·5321 (16)	0·9413 (4)	0·094 (6)
O(30)	0·8464 (18)	0·6660 (10)	0·9431 (3)	0·060 (4)
O(30′)	0·9177 (40)	0·6575 (20)	0·9418 (7)	0·174 (10)
C(31)	1·0610 (31)	0·6342 (28)	0·9460 (7)	0·152 (10)
C(31′)	1·0912 (46)	0·7032 (27)	0·9571 (6)	0·165 (12)
C(32)	1·1354 (41)	0·5928 (26)	0·9844 (6)	0·135 (9)
C(32′)	1·0820 (40)	0·6534 (20)	0·9955 (6)	0·128 (9)
C(33)	0·9796 (35)	0·4935 (25)	0·9899 (9)	0·136 (9)
C(33′)	1·0937 (44)	0·5175 (22)	0·9943 (9)	0·154 (11)
C(34)	0·7588 (29)	0·5215 (16)	0·9864 (4)	0·083 (5)
C(34′)	0·8931 (36)	0·4721 (19)	0·9791 (5)	0·098 (7)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Preparation of compound (2): A solution of 3β-tetrahydropyranyloxy-5α-pregnan-20-one (2·0 g, 5·2 mmol) (Rahman, Seidel & Pascal, 1988) in dry tetrahydrofuran (50 mL) was cooled to 195 K. Lithium diisopropylamide (3·5 mL of a 1·5 *M* solution in THF, 5·2 mmol) was added, and, after stirring for 30 min, 1-bromo-2,3-dimethyl-2-butene (0·9 g, 5·2 mmol) (McCullough, MacInnis, Lock & Faggiani, 1985) was also added. The solution was allowed to warm to room temperature overnight, methanol was added, and the resulting mixture was poured into ether. This solution was washed with dilute sodium bicarbonate and water, dried over anhydrous magnesium sulfate, and concentrated to dryness. This material was purified by silica-gel column chromatography and crystallized from ether–methanol to give pure compound (2), m.p. 366–367 K. ^{1}H NMR ($CDCl_3$) δ 4·68 (*m*, 1H, THP methine), 3·91 and 3·45 (*m*'s, 2H, THP OCH_2), 3·58 (*m*, 1H, 3α-H), 1·62 (*s*, 9H, side-chain CH_3's), 0·78 (*s*, 3H, 19-H_3), 0·55 (*s*, 3H, 18-H_3); ^{13}C NMR ($CDCl_3$) δ 211·6, 126·4, 124·8, 96·8, 96·6, 75·5, 75·2, 63·1, 62·8, 62·7, 56·8, 54·3, 45·1, 44·8, 44·4, 42·7, 39·3, 37·2, 37·0, 36·2, 35·7, 35·5, 34·3, 32·1, 31·3, 29·5, 28·8, 28·6, 27·8, 25·6, 24·5, 23·0, 21·3, 20·6, 20·1, 19·9, 18·2, 13·7, 12·3; MS, *m*/*z* 484 (7%, M^+), 469 (*M*-CH_3, 18), 385 (*M*-dihydropyran, 8), 257 (41), 85 (100). Exact mass 484·3906, calculated for $C_{32}H_{52}O_3$ 484·3903.

Structure of compound (2): A single crystal (from ethanol) with approximate dimensions of 0·18 × 0·35 × 0·54 mm was used for data collection. Intensity measurements were made on a Nicolet *R*3*m* diffractometer, equipped with an LTII low-temperature device, at 225 K using graphite-monochromated Mo *Kα* radiation and a variable-speed 1·20° ω scan. Lattice parameters were determined by a least-squares fit of the 2θ values of 20 reflections having $25 \le 2\theta \le 30^\circ$. Three standard reflections used to monitor the data collection ($3\bar{4}3$, $\bar{2}26$, 151) did not show significant deviations within the period of data collection. 2619 reflections with $3 \le 2\theta \le 50^\circ$ were collected ($-7 \le h \le 7$, $-11 \le k \le 10$, $0 \le l \le 14$); equivalent reflections were merged ($R_{merge} = 0{\cdot}009$) to give 2600 unique data of which 2208 were considered to be observed [$|F_o| > 3\sigma(F_o)$]. Raw intensities were reduced to structure-factor amplitudes by correction for scan speed, background, Lorentz and polarization effects. The structure was solved by direct

Table 2. *Bond lengths* (Å) *and angles* (°) *for compound* (1); *e.s.d.'s are given in parentheses*

C(1)—C(2)	1·535 (9)	C(1)—C(10)	1·556 (10)
C(2)—C(3)	1·510 (11)	C(3)—C(4)	1·504 (11)
C(3)—O(28)	1·420 (8)	C(4)—C(5)	1·531 (9)
C(5)—C(6)	1·528 (10)	C(5)—C(10)	1·545 (9)
C(6)—C(7)	1·505 (10)	C(7)—C(8)	1·548 (9)
C(8)—C(9)	1·524 (9)	C(8)—C(14)	1·543 (9)
C(9)—C(10)	1·545 (9)	C(9)—C(11)	1·529 (9)
C(10)—C(19)	1·520 (10)	C(11)—C(12)	1·545 (9)
C(12)—C(13)	1·504 (10)	C(13)—C(14)	1·544 (9)
C(13)—C(17)	1·537 (9)	C(13)—C(18)	1·523 (10)
C(14)—C(15)	1·505 (10)	C(15)—C(16)	1·521 (10)
C(16)—C(17)	1·532 (11)	C(17)—C(20)	1·523 (10)
C(20)—C(21)	1·322 (12)	C(20)—C(22)	1·510 (13)
C(22)—C(23)	1·563 (14)	C(23)—C(24)	1·46 (2)
C(24)—C(25)	1·29 (2)	C(25)—C(26)	1·47 (2)
C(25)—C(27)	1·49 (2)	O(28)—C(29)	1·34 (2)
O(28)—C(29′)	1·33 (2)	C(29)—O(30)	1·39 (2)
C(29)—C(34)	1·55 (2)	C(29′)—O(30′)	1·43 (3)
C(29′)—C(34′)	1·58 (3)	O(30)—C(31)	1·49 (3)
O(30′)—C(31′)	1·40 (4)	C(31)—C(32)	1·61 (4)
C(31′)—C(32′)	1·56 (3)	C(32)—C(33)	1·55 (4)
C(32′)—C(33′)	1·55 (3)	C(33)—C(34)	1·52 (3)
C(33′)—C(34′)	1·55 (4)		

C(2)—C(1)—C(10)	113·3 (6)	C(1)—C(2)—C(3)	112·1 (6)
C(2)—C(3)—C(4)	110·7 (6)	C(2)—C(3)—O(28)	108·9 (6)
C(4)—C(3)—O(28)	111·2 (6)	C(3)—C(4)—C(5)	113·0 (6)
C(4)—C(5)—C(6)	112·6 (6)	C(4)—C(5)—C(10)	113·0 (5)
C(6)—C(5)—C(10)	112·6 (6)	C(5)—C(6)—C(7)	111·9 (6)
C(6)—C(7)—C(8)	113·4 (6)	C(7)—C(8)—C(9)	111·3 (5)
C(7)—C(8)—C(14)	111·3 (5)	C(9)—C(8)—C(14)	110·8 (5)
C(8)—C(9)—C(10)	111·9 (5)	C(8)—C(9)—C(11)	112·3 (5)
C(10)—C(9)—C(11)	114·9 (5)	C(1)—C(10)—C(5)	106·8 (5)
C(1)—C(10)—C(9)	110·1 (5)	C(5)—C(10)—C(9)	106·9 (5)
C(1)—C(10)—C(19)	108·6 (6)	C(5)—C(10)—C(19)	111·7 (6)
C(9)—C(10)—C(19)	112·5 (5)	C(9)—C(10)—C(12)	114·1 (6)
C(11)—C(12)—C(13)	112·3 (5)	C(12)—C(13)—C(14)	107·5 (5)
C(12)—C(13)—C(17)	117·2 (6)	C(14)—C(13)—C(17)	98·3 (5)
C(12)—C(13)—C(18)	109·7 (6)	C(14)—C(13)—C(18)	112·1 (6)
C(17)—C(13)—C(18)	111·4 (6)	C(8)—C(14)—C(13)	113·1 (5)
C(8)—C(14)—C(15)	121·0 (6)	C(13)—C(14)—C(15)	104·5 (5)
C(14)—C(15)—C(16)	105·9 (6)	C(15)—C(16)—C(17)	105·1 (6)
C(13)—C(17)—C(16)	104·5 (5)	C(13)—C(17)—C(20)	114·5 (5)
C(16)—C(17)—C(20)	118·1 (6)	C(17)—C(20)—C(21)	123·0 (8)
C(17)—C(20)—C(22)	116·1 (7)	C(21)—C(20)—C(22)	120·9 (7)
C(20)—C(22)—C(23)	111·2 (8)	C(22)—C(23)—C(24)	116·3 (10)
C(23)—C(24)—C(25)	129·9 (14)	C(24)—C(25)—C(26)	124·0 (13)
C(24)—C(25)—C(27)	120·8 (12)	C(26)—C(25)—C(27)	115·1 (11)
C(3)—O(28)—C(29)	121·1 (9)	C(3)—O(28)—C(29′)	120·4 (9)
O(28)—C(29)—O(30)	112·2 (13)	O(28)—C(29)—C(34)	110·3 (13)
O(30)—C(29)—C(34)	108·8 (14)	O(28)—C(29′)—O(30′)	118· (2)
O(28)—C(29′)—C(34′)	105· (2)	O(30′)—C(29′)—C(34′)	115· (2)
C(29)—O(30)—C(31)	114· (2)	C(29′)—O(30′)—C(31′)	118 (2)
O(30)—C(31)—C(32)	116· (2)	O(30′)—C(31′)—C(32′)	103· (2)
C(31)—C(32)—C(33)	97· (2)	C(31′)—C(32′)—C(33′)	110· (2)
C(32)—C(33)—C(34)	119· (2)	C(32′)—C(33′)—C(34′)	107· (2)
C(29)—C(34)—C(33)	109· (2)	C(29′)—C(34′)—C(33′)	101· (2)

methods (*SHELXS*86; Sheldrick, 1986), and all further calculations were carried out using the *SHELXTL* package of programs (Sheldrick, 1980). All H atoms were located in difference Fourier syntheses and subsequently entered at idealized positions [C—H 0·96 Å, X—C—H 109 or 120°, B(H) = 1·2B(C)]. In the final stages of refinement, all non-H atoms were refined with anisotropic temperature factors, and H atoms rode the carbons to which they are attached. The largest peaks in the final difference Fourier map were in the vicinity of the THP ring, presumably due to a very small amount of the diastereomer with the opposite configuration at C(30). 316 parameters were refined by minimizing $\sum w(F_o - F_c)^2$; $R = 0·058$, $wR = 0·063$, $w = [\sigma^2(F_o) + 0·00154F^2]^{-1}$, $S = 1·39$, maximum $(\Delta/\sigma) = 0·007$, average $(\Delta/\sigma) = 0·001$, $\Delta\rho = -0·20$ to $+0·55$ e Å^{-3}.

Discussion. The configuration of C(17) of the sterol THP ethers (1) and (2), which had been under scrutiny, is established to be α by the crystallographic studies. Both compounds have all-*trans* ring junctions in the steroid nuclei.

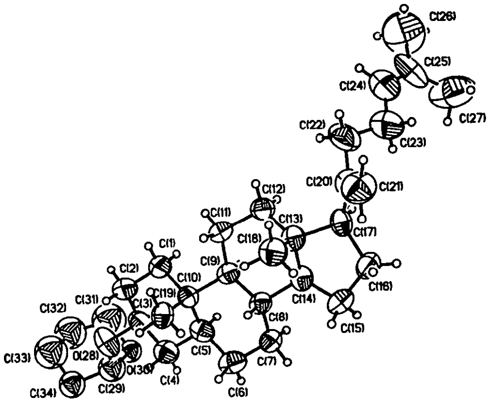

Fig. 1. Perspective drawing and atom labelling of compound (1).

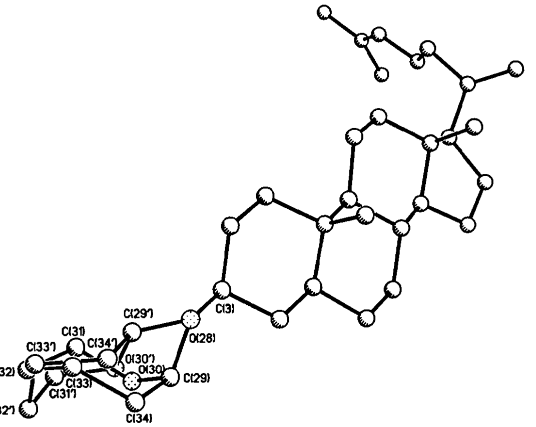

Fig. 2. Perspective drawing of compound (1) showing the positions of the two epimeric tetrahydropyranyl groups in the crystallographic model.

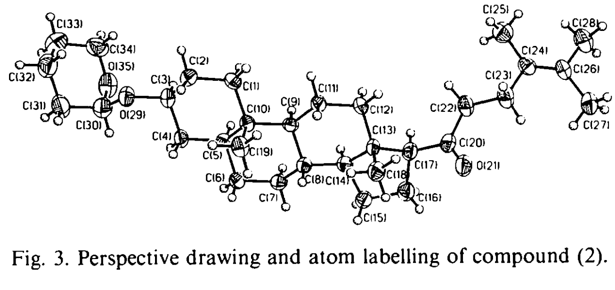

Fig. 3. Perspective drawing and atom labelling of compound (2).

Table 3. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *for compound* (2); *e.s.d.'s are given in parentheses*

	x	*y*	*z*	*U*
C(1)	0·3173 (6)	0·7245 (5)	0·7381 (4)	0·038 (2)*
C(2)	0·3189 (7)	0·6906 (5)	0·6064 (4)	0·041 (2)*
C(3)	0·2342 (6)	0·5299 (4)	0·5271 (3)	0·036 (2)*
C(4)	0·0251 (6)	0·4954 (5)	0·5469 (4)	0·037 (2)*
C(5)	0·0241 (6)	0·5333 (4)	0·6793 (4)	0·032 (1)*
C(6)	−0·1836 (6)	0·4876 (5)	0·6972 (4)	0·039 (2)*
C(7)	−0·1790 (7)	0·5113 (5)	0·8283 (4)	0·040 (2)*
C(8)	−0·0977 (6)	0·6716 (4)	0·9136 (3)	0·033 (1)*
C(9)	0·1129 (6)	0·7155 (4)	0·8928 (4)	0·030 (1)*
C(10)	0·1020 (6)	0·6978 (4)	0·7595 (3)	0·030 (1)*
C(11)	0·2090 (7)	0·8684 (5)	0·9846 (4)	0·041 (2)*
C(12)	0·2163 (7)	0·8845 (5)	1·1155 (4)	0·040 (2)*
C(13)	0·0050 (6)	0·8508 (4)	1·1337 (3)	0·036 (2)*
C(14)	−0·0765 (6)	0·6921 (4)	1·0446 (4)	0·036 (2)*
C(15)	−0·2625 (7)	0·6546 (5)	1·0873 (4)	0·044 (2)*
C(16)	−0·2096 (7)	0·7371 (6)	1·2252 (4)	0·051 (2)*
C(17)	0·0013 (7)	0·8262 (5)	1·2530 (4)	0·042 (2)*
C(18)	−0·1414 (7)	0·9700 (5)	1·1191 (4)	0·046 (2)*
C(19)	−0·0419 (7)	0·8082 (5)	0·7266 (4)	0·041 (2)*
C(20)	0·0414 (7)	0·9682 (5)	1·3664 (4)	0·041 (2)*
O(21)	−0·0879 (5)	1·0577 (4)	1·3895 (3)	0·064 (1)*
C(22)	0·2487 (7)	0·9887 (5)	1·4468 (4)	0·044 (2)*
C(23)	0·2895 (8)	1·1274 (5)	1·5609 (4)	0·050 (2)*
C(24)	0·4695 (7)	1·1212 (5)	1·6569 (4)	0·040 (2)*
C(25)	0·6579 (8)	1·0651 (6)	1·6087 (5)	0·058 (2)*
C(26)	0·4613 (7)	1·1600 (5)	1·7716 (4)	0·042 (2)*
C(27)	0·2762 (8)	1·2194 (6)	1·8230 (4)	0·059 (2)*
C(28)	0·6407 (8)	1·1552 (6)	1·8670 (5)	0·058 (2)*
O(29)	0·2244 (4)	0·5164 (3)	0·4040 (3)	0·043 (1)*
C(30)	0·1790 (8)	0·3705 (6)	0·3159 (4)	0·054 (2)*
C(31)	0·1641 (9)	0·3849 (7)	0·1947 (5)	0·061 (2)*
C(32)	0·3669 (9)	0·4293 (7)	0·1796 (5)	0·069 (3)*
C(33)	0·5230 (9)	0·3197 (7)	0·2030 (5)	0·076 (3)*
C(34)	0·5263 (7)	0·3132 (7)	0·3237 (5)	0·065 (2)*
O(35)	0·3258 (6)	0·2722 (4)	0·3343 (3)	0·062 (2)*

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 4. *Bond lengths* (Å) *and angles* (°) *for compound* (2); *e.s.d.'s are given in parentheses*

C(1)—C(2)	1·522 (7)	C(1)—C(10)	1·539 (6)
C(2)—C(3)	1·524 (5)	C(3)—C(4)	1·510 (6)
C(3)—O(29)	1·448 (5)	C(4)—C(5)	1·523 (6)
C(5)—C(6)	1·524 (6)	C(5)—C(10)	1·544 (5)
C(6)—C(7)	1·526 (7)	C(7)—C(8)	1·532 (5)
C(8)—C(9)	1·552 (6)	C(8)—C(14)	1·516 (6)
C(9)—C(10)	1·560 (6)	C(9)—C(11)	1·526 (6)
C(10)—C(19)	1·538 (6)	C(11)—C(12)	1·538 (7)
C(12)—C(13)	1·514 (6)	C(13)—C(14)	1·533 (5)
C(13)—C(17)	1·562 (7)	C(13)—C(18)	1·541 (7)
C(14)—C(15)	1·520 (7)	C(15)—C(16)	1·542 (6)
C(16)—C(17)	1·549 (7)	C(17)—C(20)	1·524 (5)
C(20)—O(21)	1·216 (6)	C(20)—C(22)	1·493 (6)
C(22)—C(23)	1·511 (5)	C(23)—C(24)	1·521 (6)
C(24)—C(25)	1·505 (7)	C(24)—C(26)	1·323 (7)
C(26)—C(27)	1·508 (7)	C(26)—C(28)	1·509 (7)
O(29)—C(30)	1·407 (5)	C(30)—C(31)	1·519 (9)
C(30)—O(35)	1·402 (7)	C(31)—C(32)	1·475 (9)
C(32)—C(33)	1·541 (9)	C(33)—C(34)	1·492 (10)
C(34)—O(35)	1·429 (7)		

C(2)—C(1)—C(10)	113·1 (3)	C(1)—C(2)—C(3)	111·8 (4)
C(2)—C(3)—C(4)	111·2 (3)	C(2)—C(3)—O(29)	106·4 (4)
C(4)—C(3)—O(29)	111·5 (3)	C(3)—C(4)—C(5)	112·5 (3)
C(4)—C(5)—C(6)	112·0 (3)	C(4)—C(5)—C(10)	113·4 (4)
C(6)—C(5)—C(10)	112·7 (3)	C(5)—C(6)—C(7)	112·1 (3)
C(6)—C(7)—C(8)	112·0 (4)	C(7)—C(8)—C(9)	111·0 (3)
C(7)—C(8)—C(14)	112·2 (4)	C(9)—C(8)—C(14)	109·4 (3)
C(8)—C(9)—C(10)	112·2 (3)	C(8)—C(9)—C(11)	112·5 (3)
C(10)—C(9)—C(11)	113·8 (4)	C(1)—C(10)—C(5)	106·9 (3)
C(1)—C(10)—C(9)	111·1 (3)	C(5)—C(10)—C(9)	107·5 (4)
C(1)—C(10)—C(19)	109·6 (4)	C(5)—C(10)—C(19)	111·3 (3)
C(9)—C(10)—C(19)	110·4 (3)	C(9)—C(11)—C(12)	113·1 (4)
C(11)—C(12)—C(13)	111·3 (3)	C(12)—C(13)—C(14)	108·1 (3)
C(12)—C(13)—C(17)	115·6 (3)	C(14)—C(13)—C(17)	98·4 (4)
C(12)—C(13)—C(18)	111·1 (4)	C(14)—C(13)—C(18)	112·3 (3)
C(17)—C(13)—C(18)	110·7 (4)	C(8)—C(14)—C(13)	114·2 (4)
C(8)—C(14)—C(15)	119·4 (3)	C(13)—C(14)—C(15)	104·4 (3)
C(14)—C(15)—C(16)	104·9 (3)	C(15)—C(16)—C(17)	105·3 (4)
C(13)—C(17)—C(16)	103·4 (3)	C(13)—C(17)—C(20)	115·3 (4)
C(16)—C(17)—C(20)	115·2 (4)	C(17)—C(20)—O(21)	121·9 (4)
C(17)—C(20)—C(22)	115·4 (4)	O(21)—C(20)—C(22)	122·7 (3)
C(20)—C(22)—C(23)	116·1 (4)	C(22)—C(23)—C(24)	114·3 (4)
C(23)—C(24)—C(25)	113·4 (4)	C(23)—C(24)—C(26)	123·4 (4)
C(25)—C(24)—C(26)	123·1 (4)	C(24)—C(26)—C(27)	124·4 (4)
C(24)—C(26)—C(28)	123·4 (4)	C(27)—C(26)—C(28)	112·2 (4)
C(3)—O(29)—C(30)	116·2 (4)	O(29)—C(30)—C(31)	106·8 (5)
O(29)—C(30)—O(35)	112·4 (4)	C(31)—C(30)—O(35)	111·9 (4)
C(30)—C(31)—C(32)	111·4 (5)	C(31)—C(32)—C(33)	109·2 (6)
C(32)—C(33)—C(34)	109·9 (5)	C(33)—C(34)—O(35)	111·5 (4)
C(30)—O(35)—C(34)	113·3 (5)		

The final atomic coordinates for all non-H atoms of compound (1) are presented in Table 1,* and the derived bond lengths and angles are given in Table 2. The numbering scheme employed for (1) is contained in the thermal-ellipsoid (50%) drawing shown in Fig. 1. Although the constitution of compound (1) is not in doubt, the structure is not particularly well determined due to the presence of two diastereomers in the crystal, having opposite configurations at C(29) (the acetal carbon of the THP group). The crystallographic model for this static disorder is shown in Fig. 2, and the disorder is reflected in the rather large thermal parameters for the THP group and the side-chain carbons.

The final atomic coordinates for all non-H atoms of compound (2) are presented in Table 3,* and the derived bond lengths and angles are given in Table 4. The numbering scheme employed for (2) is contained in the thermal-ellipsoid (50%) drawing shown in Fig. 3. In contrast to compound (1), the crystal of (2) is well ordered, and it appears to consist almost entirely of a single diastereomer (30*S*), despite the fact that both diastereomers were present during crystallization. There are no unusual bond lengths or angles.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54294 (60 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.