Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)—C(2)	1.360 (4)	C(1)—C(10a)	1.400 (3)
C(2)—C(3)	1.383 (3)	C(3)—C(4)	1.387 (3)
C(4)—C(4a)	1.418 (3)	C(4)—C(41)	1.523 (3)
C(4a)—C(4b)	1.467 (3)	C(4a)—C(10a)	1.430 (3)
C(4b)—C(5)	1.417 (3)	C(4b)—C(8a)	1.425 (3)
C(5)—C(6)	1.383 (3)	C(5)—C(51)	1.502 (3)
C(6)—C(7)	1.381 (4)	C(7)—C(8)	1.361 (4)
C(8)—C(8a)	1.399 (4)	C(8a)—C(9)	1.435 (4)
C(9)—C(10)	1.335 (4)	C(10)—C(10a)	1.430 (3)
C(41)—C(42)	1.514 (3)	C(41)—O(4)	1.422 (3)
C(51)—O(52)	1.457 (2)	O(52)—C(52)	1.304 (3)
C(52)—O(53)	1.199 (3)	C(52)—C(53)	1.481 (3)
C(10a)—C(1)—C(2)	120.8 (2)	C(3)—C(2)—C(1)	119.5 (2)
C(4)—C(3)—C(2)	122.1 (2)	C(4a)—C(4)—C(3)	119.0 (2)
C(41)—C(4)—C(3)	116.7 (2)	C(41)—C(4)—C(4a)	123.6 (2)
C(4b)—C(4a)—C(4)	125.4 (2)	C(10a)—C(4a)—C(4)	117.4 (2)
C(10a)—C(4a)—C(4b)	117.2 (2)	C(5)—C(4b)—C(4a)	124.8 (2)
C(8a)—C(4b)—C(4a)	117.7 (2)	C(8a)—C(4b)—C(5)	117.4 (2)
C(6)—C(5)—C(4b)	119.2 (2)	C(51)—C(5)—C(4b)	123.1 (2)
C(51)—C(5)—C(6)	117.0 (2)	C(7)—C(6)—C(5)	121.6 (3)
C(8)—C(7)—C(6)	119.9 (3)	C(8a)—C(8)—C(7)	120.5 (3)
C(8)—C(8a)—C(4b)	119.9 (3)	C(9)—C(8a)—C(4b)	119.6 (2)
C(9)—C(8a)—C(8)	120.1 (3)	C(10)—C(9)—C(8a)	120.7 (2)
C(10a)—C(10)—C(9)	121.6 (3)	C(4a)—C(10a)—C(1)	119.9 (2)
C(10)—C(10a)—C(1)	120.5 (2)	C(10)—C(10a)—C(4a)	119.5 (2)
C(42)—C(41)—C(4)	110.6 (2)	O(4)—C(41)—C(4)	113.3 (2)
O(4)—C(41)—C(42)	111.3 (2)	O(52)—C(51)—C(5)	105.1 (2)
C(52)—O(52)—C(51)	117.5 (2)	O(53)—C(52)—O(52)	121.8 (2)
C(53)—C(52)—O(52)	113.6 (2)	C(53)—C(52)—O(53)	124.6 (3)

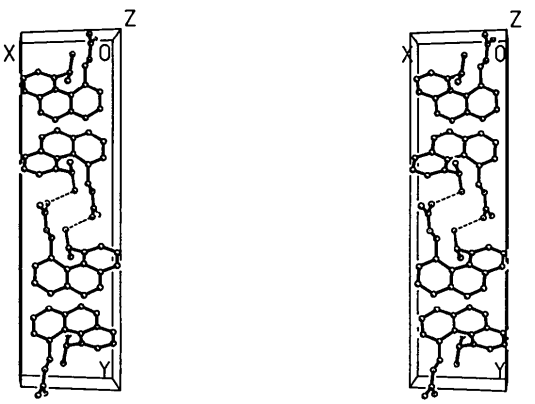

Fig. 2. Stereographic packing dragram (without H atoms) along the *z* axis. Hydrogen bonds are indicated by dashed lines.

The structure was solved by direct methods and subjected to anisotropic full-matrix least-squares refinement on *F*. H atoms were included using a riding model. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.0002F^2$; final $R = 0.057$, $wR = 0.059$, for 209 parameters; $S = 2.4$; maximum $\Delta/\sigma = 0.001$, maximum $\Delta\rho = 0.28$, minimum $\Delta\rho = -0.26$ e Å^{-3}. Atomic scattering factors and f', f'' values were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The program system used was *SHELXTL-Plus* (Sheldrick, 1989). Final atom coordinates are given in Table 1,* with derived bond lengths and angles in Table 2. Fig. 1 shows the atomic labelling scheme and Fig. 2 shows the crystal packing.

Related literature. Similar helical ring systems are described by Schrumpf & Jones (1988) and Jones & Schrumpf (1988).

We thank the Fonds der Chemischen Industrie for financial support. The crystals were provided by Professor G. Schrumpf of the University of Göttingen, where the intensity measurements were performed.

* 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 55328 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0105]

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9,10-Dihydrophenanthrene-4,5-dimethanol

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Abstract. $C_{16}H_{16}O_2$, $M_r = 240.29$, orthorhombic, $P2_12_12$, $a = 15.6464$ (12), $b = 23.027$ (3), $c = 6.9320$ (8) Å, $V = 2497.5$ (7) Å^3, $Z = 8$, $D_x = 1.278$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.08$ mm^{-1}, $F(000) = 1024$, $T = 293$ K, $R = 0.076$ for 3362 reflections. The two independent molecules are essentially identical except for the orientation of one hydroxyl group O(4); however, they display opposite

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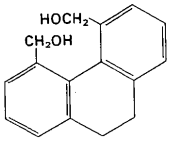
helicities. The helicity of the ring system is greater than that of the 9,10-dehydro analogue [Jones (1992). *Acta Cryst.* C**48**, 2244–2245], with torsion angles C(4)—C(4a)—C(4b)—C(5) ±45° (standard numbering for phenanthrene systems). The largest torsion angle of the central ring is 60° about C(9)—C(10). Several O···O contacts <3.1 Å probably correspond to hydrogen bonds.

Experimental. A colourless prism, 0.7 × 0.2 × 0.2 mm, of the title compound (I), was mounted in a glass capillary. Using a Stoe–Siemens four-circle diffractometer, 5685 intensities were registered by θ/ω scans to $2\theta_{max} = 50°$ with monochromated Mo $K\alpha$ radiation. Of 4385 unique reflections ($R_{int} = 0.021$, index ranges h −18 to 18, k 0 to 27, l 0 to 8), 3362 with $F > 3\sigma(F)$ were considered observed. The cell constants were refined from ±ω angles of 58 reflections in the 2θ range 20–22°. Three check reflections showed no significant intensity variation. No absorption correction was applied.



(I)

The structure was solved by direct methods and subjected to anisotropic full-matrix least-squares refinement on F. H atoms were included using a riding model, except for the hydroxyl H at O(4′), which was not found; the O atom displays a high U_{eq} value of 0.127 Å^2, and may be slightly disordered. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.00025F^2$; final $R = 0.076$, with $wR = 0.065$. 325 parameters were refined; $S = 2.0$; maximum $\Delta/\sigma = 0.02$; maximum/minimum $\Delta\rho = 0.7/-0.5$ e Å^{-3}. *SHELXTL-Plus* (Sheldrick, 1989) was used for computations, and was the source of atomic scattering factors. Final atom coordinates are given in Table 1, with derived bond lengths and angles in Table 2.* The two independent molecules are shown in Fig. 1. The high R factor is attributable to the weak diffraction and ultimately to the thermal motion of some terminal atoms (see above).

Related literature. Similar helical ring systems are decribed by Schrumph & Jones (1988), Jones & Schrumpf (1988) and Jones (1992).

* 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 55367 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0104]

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

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

	x	y	z	U_{eq}
C(1)	1991 (3)	2538 (2)	5624 (6)	61 (2)
C(2)	1124 (3)	2425 (2)	5752 (6)	68 (2)
C(3)	823 (3)	1905 (2)	5050 (6)	57 (2)
C(4)	1354 (2)	1512 (2)	4112 (5)	45 (1)
C(4a)	2214 (2)	1666 (2)	3800 (5)	41 (1)
C(4b)	2807 (2)	1300 (2)	2613 (6)	49 (1)
C(5)	2610 (3)	1054 (2)	823 (6)	50 (1)
C(6)	3194 (4)	686 (2)	−40 (8)	80 (2)
C(7)	3988 (4)	590 (3)	769 (11)	98 (3)
C(8)	4227 (3)	893 (3)	2421 (10)	88 (2)
C(8a)	3644 (3)	1255 (2)	3365 (7)	60 (2)
C(9)	3898 (3)	1642 (2)	5047 (7)	80 (2)
C(10)	3493 (3)	2231 (2)	4762 (7)	67 (2)
C(10a)	2541 (3)	2160 (2)	4692 (6)	47 (1)
C(41)	1001 (3)	923 (2)	3707 (6)	54 (1)
O(4)	1073 (2)	588 (1)	5347 (4)	96 (2)
C(51)	1847 (3)	1218 (2)	−356 (6)	59 (2)
O(5)	1323 (2)	739 (1)	−907 (5)	96 (1)
C(1′)	8152 (3)	2364 (2)	10160 (7)	61 (2)
C(2′)	9018 (3)	2248 (2)	10316 (6)	64 (2)
C(3′)	9321 (3)	1723 (2)	9689 (6)	56 (2)
C(4′)	8800 (2)	1321 (2)	8776 (5)	43 (1)
C(4a′)	7948 (2)	1471 (2)	8409 (5)	40 (1)
C(4b′)	7341 (2)	1108 (2)	7269 (5)	42 (1)
C(5′)	7537 (2)	842 (2)	5515 (6)	45 (1)
C(6′)	6957 (3)	461 (2)	4672 (7)	62 (2)
C(7′)	6165 (3)	382 (2)	5489 (8)	73 (2)
C(8′)	5931 (3)	692 (2)	7082 (8)	67 (2)
C(8a′)	6504 (3)	1062 (2)	7988 (6)	50 (1)
C(9′)	6240 (3)	1458 (2)	9632 (6)	63 (2)
C(10′)	6646 (3)	2048 (2)	9285 (6)	57 (2)
C(10″)	7611 (3)	1981 (2)	9258 (6)	47 (1)
C(41′)	9132 (3)	727 (2)	8420 (6)	56 (2)
O(4′)	9451 (3)	497 (2)	10142 (5)	127 (2)
C(51′)	8333 (2)	992 (2)	4332 (6)	51 (1)
O(5′)	8777 (2)	486 (1)	3748 (4)	67 (1)

Table 2. *Interatomic distances including possible hydrogen-bond lengths* (Å) *and bond angles* (°)

For bond distances and angles the second value is for the second independent molecule with atoms labelled with primes in Table 1. E.s.d.'s for the possible O···O hydrogen-bond lengths are 0.01 Å. The H atom at O(4′) was not located.

C(1)—C(2)	1.384 (7)	1.386 (7)	C(1)—C(10a)	1.385 (6)	1.374 (6)
C(2)—C(3)	1.377 (7)	1.369 (6)	C(3)—C(4)	1.389 (6)	1.386 (6)
C(4)—C(4a)	1.409 (5)	1.400 (5)	C(4)—C(41)	1.492 (6)	1.484 (6)
C(4a)—C(4b)	1.499 (5)	1.494 (5)	C(4a)—C(10a)	1.392 (5)	1.414 (5)
C(4b)—C(5)	1.398 (6)	1.396 (5)	C(4b)—C(8a)	1.414 (6)	1.405 (5)
C(5)—C(6)	1.383 (7)	1.391 (6)	C(5)—C(51)	1.495 (6)	1.530 (5)
C(6)—C(7)	1.380 (9)	1.375 (6)	C(7)—C(8)	1.393 (9)	1.364 (7)
C(8)—C(8a)	1.398 (7)	1.387 (6)	C(8a)—C(9)	1.520 (7)	1.518 (6)
C(9)—C(10)	1.509 (7)	1.518 (6)	C(10)—C(10a)	1.499 (6)	1.519 (6)
C(41)—O(4)	1.379 (5)	1.398 (5)	C(51)—O(5)	1.427 (5)	1.415 (5)

O(4)(x, y, $-1+z$)···O(5)	2.65	O(4)···H(O5)	1.81
O(4)($1-x$, $-y$, z)···O(5′)	2.72	O(5′)···H(O4)	1.88
O(4′)(x, y, $-1+z$)···O(5′)	2.71	O(4′)···H(O5′)	1.85
O(4′)($2-x$, $-y$, z)···O(4′)	2.86		
O(5)($1+x$, y, $1+z$)···O(4′)	3.07		

C(2)—C(1)—C(10a)	121.4 (4)	120.8 (4)	C(1)—C(2)—C(3)	118.5 (4)	119.0 (4)
C(2)—C(3)—C(4)	121.8 (4)	122.1 (4)	C(3)—C(4)—C(4a)	118.6 (4)	118.5 (3)
C(3)—C(4)—C(41)	117.3 (3)	119.0 (3)	C(4a)—C(4)—C(41)	123.6 (3)	122.1 (3)
C(4)—C(4a)—C(4b)	122.3 (3)	124.3 (3)	C(4)—C(4a)—C(10a)	119.3 (3)	119.0 (3)
C(4b)—C(4a)—C(10a)	118.4 (3)	116.6 (3)	C(4a)—C(4b)—C(5)	125.3 (3)	124.5 (3)
C(4a)—C(4b)—C(8a)	114.4 (4)	116.6 (3)	C(5)—C(4b)—C(8a)	120.1 (4)	118.8 (3)
C(4b)—C(5)—C(6)	119.1 (4)	119.9 (4)	C(4b)—C(5)—C(51)	124.0 (4)	123.2 (3)
C(6)—C(5)—C(51)	116.4 (4)	116.6 (4)	C(5)—C(6)—C(7)	121.2 (5)	119.8 (4)
C(6)—C(7)—C(8)	119.7 (5)	120.5 (4)	C(7)—C(8)—C(8a)	120.5 (5)	120.9 (4)
C(4b)—C(8a)—C(8)	118.4 (4)	119.2 (4)	C(4b)—C(8a)—C(9)	118.8 (4)	118.3 (3)
C(8)—C(8a)—C(9)	122.6 (4)	122.2 (4)	C(8a)—C(9)—C(10)	108.5 (4)	107.8 (3)
C(9)—C(10)—C(10a)	108.8 (4)	109.0 (3)	C(1)—C(10a)—C(4a)	119.5 (4)	119.6 (4)
C(1)—C(10a)—C(10)	122.3 (4)	122.7 (4)	C(4a)—C(10a)—C(10)	118.0 (4)	117.5 (3)
C(4)—C(41)—O(4)	108.8 (3)	109.4 (3)	C(5)—C(51)—O(5)	114.3 (3)	111.6 (3)

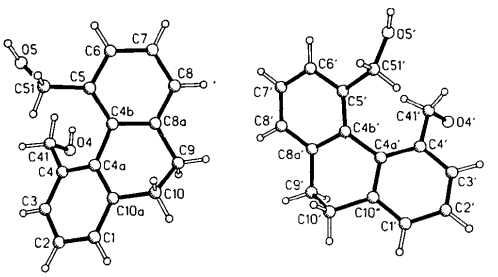


Fig. 1. The two independent molecules of the title compound in the crystal. Atomic radii are arbitrary.

I thank the Fonds der Chemischen Industrie for financial support. The crystals were provided by Professor G. Schrumpf of the University of Göttingen, where the intensity measurements were performed.

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Structure of 5β-Pregnane-3α,6α,17α-triol Triacetate

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Abstract. $C_{27}H_{40}O_7$, $M_r = 476.61$, monoclinic, $P2_1$, $a = 17.440$ (5), $b = 13.267$ (1), $c = 12.168$ (2) Å, $\beta = 110.49$ (8)°, $V = 2637.3$ (9) Å^3, $Z = 4$, $D_x = 1.20$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 7.04$ cm^{-1}, $F(000) = 1032$, $T = 293$ K, $R = 0.048$, $wR = 0.068$ for 5590 observed reflections with $(F_o)^2 > 2\sigma[(F_o)^2]$. The structure contains two crystallographically independent molecules in the asymmetric unit that have almost identical geometry. Rings *A*, *B* and *C* have chair conformations and the *D* ring assumes a half-chair conformation in both molecules. The progesterone side chain has a conformation typical for other 17α-ester steroids; the C(16)—C(17)—C(20)—O(20) torsion angles are −18.2 (5) and −15.0 (4)° for the first and the second molecule respectively.

Experimental. Material for crystallization was provided by Dr F. S. LaBella (Templeton, Sashi Kumar, Bose & LaBella, 1989). A crystal with dimensions 0.44 × 0.64 × 0.80 mm was used for data collection on a CAD-4 diffractometer. Cell dimensions and Laue symmetry were determined from 25 centered reflections ($59.8 < 2\theta < 65.4°$) checked with oscillation photographs. Initial orientation indicated possible *B*-centered orthorhombic space group; however, a check of equivalent reflections indicated the space group to be *B*-centered monoclinic and was subsequently transformed to the current monoclinic $P2_1$ cell. Data were collected using Cu $K\alpha$ radiation, with scan width $(0.80 + 0.20\tan\theta)°$, for $\theta_{max} = 75°$, $-22 < h < 0$, $0 < k < 16$, $-16 < l < 16$. 6509 reflections were measured using θ–2θ scans. 5665 reflections were unique, $R_{int} = 0.19$. Four standard reflections (217, 7$\bar{1}$3, 0$\bar{9}$1, 10,1,1) were measured every 196 reflections and varied in intensity by less than 5% during the data collection. Intensity corrections were made with the *DREAM* program (Blessing, 1987).

Direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by full-matrix least-squares method on *F* values using 5590 reflections for which $(F_o)^2 > 2\sigma[(F_o)^2]$. The H-atom positions were located in a difference Fourier map and refined with isotropic displacement parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.048$ ($R_{all} = 0.048$), $wR = 0.068$ ($w = 1/\sigma^2$), $S = 2.675$ for 321 variables and 5590 reflections; $(\Delta/\sigma)_{max}$

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