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A D-Homotestosterone Derivative

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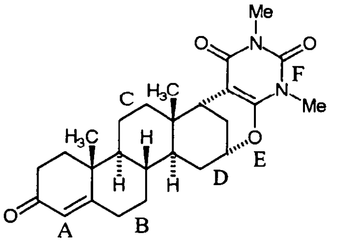
Abstract

The structure of [6*R*-(6α,7aβ,7bα,13aα,13bβ,15aα,16α)]-4,6,7,7a,7b,8,9,11,12,13,13a,13b,14,15,15a,16-hexadecahydro-2,4,13a,15a-tetramethyl-6,16-methano-1*H*-phenanthro[2',1':5,6]oxocino[2,3-*d*]pyrimidine-1,3(2*H*),11-trione, $C_{26}H_{34}N_2O_4$, is reported.

Comment

Rings *B* and *C* and rings *C* and *D* are *trans* fused, whereas rings *D* and *E* are *cis* fused (6α,16α). Rings *B* and *C* adopt chair conformations, whereas rings *A*, *D* and *E* show a 1α,2β-half-chair, a distorted chair and a 17α-sofa conformation, respectively.



(I)

For some related reactions see Tietze (1984), Tietze, Wölfling & Schneider (1991), and Tietze & Beifuss (1993). For the structure of testosterone see Roberts, Pettersen, Sheldrick, Isaacs & Kennard (1973), and for several testosterone derivatives see Duax & Norton (1975), and Griffin, Duax & Weeks (1984).

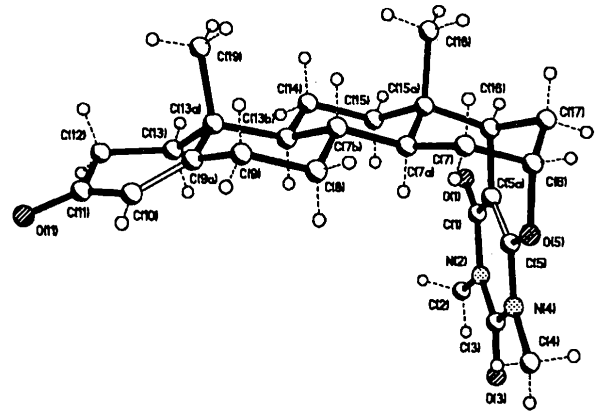


Fig. 1. View of the title compound with atomic numbering scheme.

Experimental

The synthesis of the title compound *via* a tandem-Knoevenagel hetero-Diels–Alder reaction will be published elsewhere (Tietze, Wölfling & Schneider, 1994).

Crystal data

$C_{26}H_{34}N_2O_4$
M_r = 438.57
Orthorhombic
$P2_12_12_1$
a = 10.532 (2) Å
b = 13.812 (3) Å
c = 16.048 (3) Å
V = 2334.5 (8) Å^3
Z = 4
D_x = 1.248 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 35 reflections
θ = 10–12.5°
μ = 0.084 mm^{-1}
T = 293 (2) K
Plates
1.00 × 1.00 × 0.70 mm
Light yellow

Data collection

Stoe AED2 four-circle diffractometer
Profile fitting collection method
Absorption correction: none
2275 measured reflections
2028 independent reflections
1830 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int}$ = 0.0166
$\theta_{\rm max}$ = 22.49°
$h = -2 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0419
$wR(F^2)$ = 0.1140

$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.153 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.159 e Å^{-3}

$S = 0.995$
2025 reflections
289 parameters
H-atom parameters not refined
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0790P)^2 + 0.8100P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C1	0.4770 (4)	0.3329 (3)	0.8693 (2)	0.0483 (9)
O1	0.4460 (3)	0.4156 (2)	0.8853 (2)	0.0707 (8)
N2	0.6001 (3)	0.3136 (2)	0.8389 (2)	0.0497 (8)
C2	0.6793 (4)	0.3965 (3)	0.8177 (3)	0.0711 (13)
C3	0.6460 (4)	0.2233 (3)	0.8204 (2)	0.0529 (10)
O3	0.7530 (3)	0.2120 (2)	0.7922 (2)	0.0798 (10)
N4	0.5686 (3)	0.1460 (2)	0.8374 (2)	0.0491 (8)
C4	0.6147 (5)	0.0499 (3)	0.8147 (3)	0.0809 (15)
C5	0.4462 (3)	0.1611 (3)	0.8657 (2)	0.0431 (9)
C5a	0.3977 (3)	0.2496 (3)	0.8807 (2)	0.0427 (9)
O5	0.3841 (2)	0.0769 (2)	0.8772 (2)	0.0549 (7)
C6	0.2555 (3)	0.0820 (3)	0.9121 (2)	0.0552 (10)
C7	0.1597 (3)	0.0828 (3)	0.8410 (2)	0.0496 (9)
C7a	0.1658 (3)	0.1754 (2)	0.7888 (2)	0.0396 (8)
C7b	0.0692 (3)	0.1787 (2)	0.7165 (2)	0.0400 (8)
C8	0.0758 (4)	0.0876 (2)	0.6634 (2)	0.0548 (10)
C9	−0.0165 (5)	0.0913 (3)	0.5907 (2)	0.0665 (13)
C9a	−0.0002 (4)	0.1795 (3)	0.5384 (2)	0.0462 (9)
C10	0.0037 (4)	0.1733 (3)	0.4551 (2)	0.0642 (11)
C11	−0.0025 (4)	0.2552 (4)	0.3995 (2)	0.0698 (12)
O11	−0.0131 (4)	0.2450 (3)	0.3240 (2)	0.1096 (14)
C12	−0.0065 (6)	0.3522 (3)	0.4386 (2)	0.084 (2)
C13	0.0551 (5)	0.3538 (3)	0.5245 (2)	0.0656 (12)
C13a	0.0035 (4)	0.2758 (2)	0.5836 (2)	0.0440 (9)
C13b	0.0928 (3)	0.2677 (2)	0.6613 (2)	0.0400 (8)
C14	0.0963 (4)	0.3607 (2)	0.7131 (2)	0.0504 (9)
C15	0.1892 (4)	0.3541 (3)	0.7865 (2)	0.0499 (9)
C15a	0.1613 (3)	0.2678 (3)	0.8436 (2)	0.0442 (9)
C16	0.2645 (3)	0.2591 (3)	0.9134 (2)	0.0485 (9)
C17	0.2430 (4)	0.1686 (3)	0.9672 (2)	0.0618 (12)
C18	0.0309 (4)	0.2815 (3)	0.8866 (2)	0.0646 (12)
C19	−0.1350 (4)	0.3000 (3)	0.6092 (3)	0.0641 (11)

Table 2. *Selected geometric parameters* (Å, °)

C1—O1	1.216 (5)	C7b—C13b	1.535 (4)
C1—N2	1.410 (5)	C8—C9	1.520 (5)
C1—C5a	1.433 (5)	C9—C9a	1.489 (5)
N2—C3	1.371 (5)	C9a—C10	1.339 (5)
N2—C2	1.457 (5)	C9a—C13a	1.516 (5)
C3—O3	1.225 (5)	C10—C11	1.442 (6)
C3—N4	1.371 (5)	C11—O11	1.224 (5)
N4—C5	1.383 (5)	C11—C12	1.480 (6)
N4—C4	1.459 (5)	C12—C13	1.524 (6)
C5—C5a	1.346 (5)	C13—C13a	1.534 (5)
C5—O5	1.347 (4)	C13a—C19	1.552 (5)
C5a—C16	1.504 (5)	C13a—C13b	1.566 (4)
O5—C6	1.468 (4)	C13b—C14	1.531 (4)
C6—C17	1.493 (6)	C14—C15	1.533 (5)
C6—C7	1.523 (5)	C15—C15a	1.532 (5)
C7—C7a	1.530 (5)	C15a—C18	1.548 (5)
C7a—C7b	1.544 (4)	C15a—C16	1.566 (5)
C7a—C15a	1.551 (4)	C16—C17	1.535 (5)
C7b—C8	1.521 (5)		
O1—C1—N2	119.8 (3)	C10—C9a—C9	120.9 (3)
O1—C1—C5a	124.8 (4)	C10—C9a—C13a	122.2 (3)
N2—C1—C5a	115.4 (3)	C9—C9a—C13a	116.8 (3)
C3—N2—C1	124.8 (3)	C9a—C10—C11	124.5 (4)
C3—N2—C2	117.6 (3)	O11—C11—C10	121.8 (4)
C1—N2—C2	117.3 (3)	O11—C11—C12	121.4 (4)
O3—C3—N2	121.4 (4)	C10—C11—C12	116.6 (3)
O3—C3—N4	121.4 (4)	C11—C12—C13	112.6 (4)
N2—C3—N4	117.1 (3)	C12—C13—C13a	113.5 (4)
C3—N4—C5	120.1 (3)	C9a—C13a—C13	109.2 (3)
C3—N4—C4	117.4 (3)	C9a—C13a—C19	106.9 (3)
C5—N4—C4	122.0 (3)	C13—C13a—C19	110.2 (3)
C5a—C5—O5	125.1 (3)	C9a—C13a—C13b	109.5 (3)
C5a—C5—N4	123.3 (3)	C13—C13a—C13b	109.3 (3)
O5—C5—N4	111.5 (3)	C19—C13a—C13b	111.6 (3)
C5—C5a—C1	119.0 (3)	C14—C13b—C7b	111.3 (3)
C5—C5a—C16	119.7 (3)	C14—C13b—C13a	112.8 (3)
C1—C5a—C16	121.2 (3)	C7b—C13b—C13a	114.8 (3)
C5—O5—C6	117.2 (3)	C13b—C14—C15	112.5 (3)
O5—C6—C17	110.3 (3)	C14—C15—C15a	112.6 (3)
O5—C6—C7	109.0 (3)	C15—C15a—C18	110.0 (3)
C17—C6—C7	112.3 (3)	C15—C15a—C7a	107.1 (3)
C6—C7—C7a	112.9 (3)	C18—C15a—C7a	112.4 (3)
C7—C7a—C7b	114.1 (3)	C15—C15a—C16	110.8 (3)
C7—C7a—C15a	112.1 (3)	C18—C15a—C16	107.8 (3)
C7b—C7a—C15a	112.5 (3)	C7a—C15a—C16	108.7 (3)
C8—C7b—C13b	109.4 (2)	C5a—C16—C17	105.2 (3)
C8—C7b—C7a	111.5 (3)	C5a—C16—C15a	113.9 (3)
C13b—C7b—C7a	110.5 (3)	C17—C16—C15a	111.3 (3)
C9—C8—C7b	111.9 (3)	C6—C17—C16	107.8 (3)
C9a—C9—C8	112.8 (3)		

Data collection: *DIF*4 (Stoe & Cie, 1988*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1988*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71821 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1057]

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