Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1161). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Fluorinated D-Homoestrone Derivative

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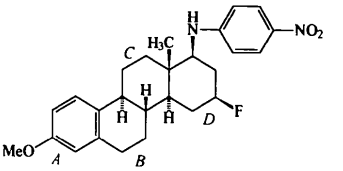
Abstract

The structure of 16β-fluoro-3-methoxy-17aβ-(4-nitroanilino)-17a-homoestra-1,3,5(10)-triene, $C_{26}H_{31}FN_2O_3$, is reported.

Comment

The synthesis of the title compound, (I), *via* a cationic cyclization reaction of the corresponding D-secoestrone imine derivative will be published elsewhere (Wölfling, Schneider, Frank & Tietze, 1996). The product of this reaction has two new stereogenic centers at the C16 and C17*A* positions, and the assignment of the stereochemistry at these positions was the reason for the present study.



(I)

The *B*/*C* and *C*/*D* ring fusions are *trans*. Rings *C* and *D* adopt chair conformations, while ring *B* displays a twisted half-chair conformation. Both substituents of the *D* ring, the F atom at C16 and the 4-nitroanilino group at C17*A*, are equatorial, *i.e.* in the β position. For the crystal structure of a fluorinated estrone derivative see Neeman, Kartha, Go, Santodonato & Dodson-Simmons (1983) and for the structure of a D-homoestrone derivative see Antel, Sheldrick, Tietze & Wölfling (1988).

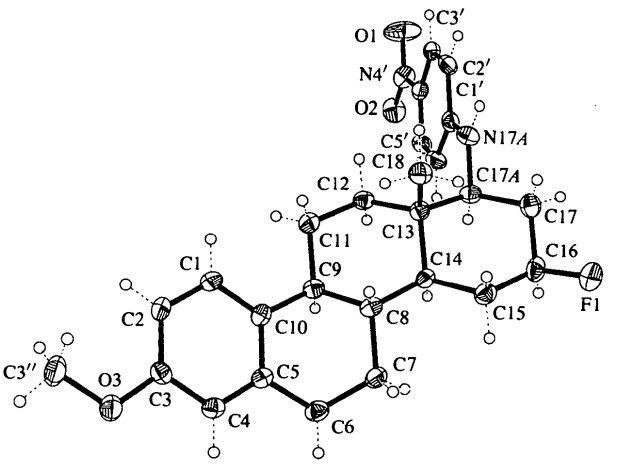


Fig. 1. View of the title compound with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

The title compound was crystallized from acetone. Data were collected by a real-time learnt-profile method (Clegg, 1981).

Crystal data

$C_{26}H_{31}FN_2O_3$
M_r = 438.53
Orthorhombic
$P2_12_12_1$
a = 8.5500 (10) Å
b = 12.1890 (10) Å
c = 20.979 (2) Å

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 48 reflections
θ = 10.0–12.5°
μ = 0.093 mm^{-1}
T = 153 (2) K

V = 2186.3 (4) Å^3
Z = 4
D_x = 1.332 Mg m^{-3}
D_m not measured

Block
0.80 × 0.50 × 0.40 mm
Yellowish

Data collection

Stoe AED-2 four-circle diffractometer
Profile fitted θ-ω scans
Absorption correction: none
2369 measured reflections
2184 independent reflections
1848 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0494
θ_{max} = 24.92°
$h = 0 \rightarrow 10$
$k = -8 \rightarrow 14$
$l = -24 \rightarrow 24$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0459
$wR(F^2)$ = 0.1065
S = 1.073
2184 reflections
291 parameters
H atoms were positioned geometrically and refined as riding
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.7P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.212 e Å^{-3}
$\Delta\rho_{min}$ = −0.246 e Å^{-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)
Absolute configuration: Flack (1983)
Flack parameter = −1.0 (16)

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

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

	x	y	z	U_{eq}
F1	0.0708 (3)	0.6777 (2)	0.80455 (10)	0.0391 (6)
O3	0.2516 (3)	0.0206 (2)	1.15152 (11)	0.0304 (6)
C3''	0.2223 (5)	−0.0956 (3)	1.1464 (2)	0.0366 (10)
C1	0.3362 (4)	0.0863 (3)	0.9830 (2)	0.0257 (8)
C2	0.2994 (4)	0.0242 (3)	1.0368 (2)	0.0263 (8)
C3	0.2860 (4)	0.0747 (3)	1.0955 (2)	0.0229 (8)
C4	0.3082 (4)	0.1887 (3)	1.1003 (2)	0.0221 (7)
C5	0.3430 (4)	0.2500 (3)	1.04668 (15)	0.0201 (7)
C6	0.3681 (4)	0.3728 (3)	1.0479 (2)	0.0260 (8)
C7	0.2849 (5)	0.4327 (3)	0.9924 (2)	0.0263 (9)
C8	0.2759 (4)	0.3658 (3)	0.9293 (2)	0.0210 (7)
C9	0.3976 (4)	0.2719 (3)	0.9307 (2)	0.0217 (8)
C10	0.3598 (4)	0.1988 (3)	0.9871 (2)	0.0212 (8)
C11	0.4104 (5)	0.2101 (3)	0.8677 (2)	0.0254 (8)
C12	0.4354 (4)	0.2894 (3)	0.8117 (2)	0.0229 (8)
C13	0.3040 (4)	0.3752 (3)	0.80619 (15)	0.0200 (7)
C14	0.2965 (4)	0.4402 (3)	0.87016 (15)	0.0198 (7)
C15	0.1726 (4)	0.5309 (3)	0.8664 (2)	0.0255 (8)
C16	0.2012 (4)	0.6069 (3)	0.8111 (2)	0.0264 (8)
C17	0.2271 (4)	0.5513 (3)	0.7479 (2)	0.0265 (8)
C17*A*	0.3491 (4)	0.4599 (3)	0.75396 (15)	0.0235 (8)
C18	0.1485 (4)	0.3190 (3)	0.7894 (2)	0.0265 (8)
N17*A*	0.3798 (4)	0.4118 (2)	0.69154 (13)	0.0268 (7)
C1′	0.5237 (4)	0.3927 (3)	0.6658 (2)	0.0224 (8)
C2′	0.5337 (4)	0.3397 (3)	0.6059 (2)	0.0242 (8)
C3′	0.6759 (4)	0.3192 (3)	0.5782 (2)	0.0236 (8)
C4′	0.8128 (4)	0.3536 (3)	0.6077 (2)	0.0234 (8)
C5′	0.8073 (5)	0.4062 (3)	0.6667 (2)	0.0271 (9)
C6′	0.6647 (4)	0.4240 (3)	0.6954 (2)	0.0263 (8)
N4′	0.9634 (4)	0.3316 (3)	0.5793 (2)	0.0320 (8)
O1	0.9663 (4)	0.2841 (3)	0.52741 (14)	0.0602 (10)
O2	1.0840 (3)	0.3611 (2)	0.60700 (13)	0.0370 (7)

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

F1—C16	1.416 (4)	C13—C17*A*	1.554 (4)
O3—C3	1.379 (4)	C13—C14	1.559 (4)
O3—C3′′	1.442 (4)	C14—C15	1.534 (5)
C5—C6	1.513 (5)	C15—C16	1.504 (5)
C6—C7	1.548 (5)	C16—C17	1.506 (5)
C7—C8	1.556 (4)	C17—C17*A*	1.531 (5)
C8—C14	1.547 (4)	C17*A*—N17*A*	1.459 (4)
C8—C9	1.547 (5)	N17A—C1′	1.363 (4)
C9—C10	1.515 (5)	C4′—N4′	1.444 (5)
C9—C11	1.526 (5)	C5′—C6′	1.376 (5)
C11—C12	1.537 (5)	N4′—O1	1.233 (4)
C12—C13	1.539 (5)	N4′—O2	1.238 (4)
C13—C18	1.537 (5)		
C14—C8—C9	111.8 (3)	C8—C14—C13	113.4 (2)
C14—C8—C7	111.7 (2)	C16—C15—C14	111.8 (3)
C10—C9—C11	113.6 (3)	F1—C16—C15	108.8 (3)
C10—C9—C8	107.8 (3)	F1—C16—C17	107.7 (3)
C11—C9—C8	113.4 (3)	C15—C16—C17	115.2 (3)
C9—C11—C12	111.2 (3)	C16—C17—C17*A*	110.8 (3)
C11—C12—C13	112.5 (3)	N17*A*—C17*A*—C17	110.0 (3)
C18—C13—C12	110.2 (3)	N17*A*—C17*A*—C13	114.2 (3)
C18—C13—C17*A*	110.4 (3)	C17—C17*A*—C13	111.9 (3)
C12—C13—C17*A*	108.9 (3)	C1′—N17*A*—C17*A*	125.9 (3)
C18—C13—C14	112.9 (3)	N17A—C1′—C6′	123.5 (3)
C12—C13—C14	108.1 (3)	N17*A*—C1′—C2′	118.9 (3)
C17*A*—C13—C14	106.2 (2)	O1—N4′—O2	122.3 (3)
C15—C14—C8	112.6 (3)	O1—N4′—C4′	118.0 (3)
C15—C14—C13	110.5 (3)	O2—N4′—C4′	119.7 (3)

Data collection: *DIF*4 (Stoe & Cie, 1991*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXTL/PC*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1183). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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