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3,17-Dioxo-11-fluoroandrosta-4,6-diene

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Key indicators

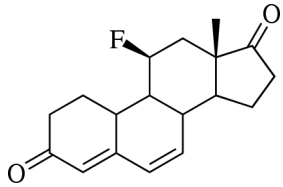
Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.096
Data-to-parameter ratio = 7.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{18}H_{21}FO_2$, (I), is a steroid with a dienone structure and six stereogenic centers. These steroids are substrates for estradiol derivatives with an alkyl chain in the 7α-position [Krause & Thorand (1999). *Inorg. Chim. Acta.* **296**, 1–11; Uerdingen & Krause (2000). *Tetrahedron*, **56**, 2799–2804]. The configuration of the 11-position is *S*. The diene is in conjugation with the carbonyl group and the latter is involved in a weak intermolecular C—H···O hydrogen bond.

Experimental

The title compound, (I), was obtained with 80–90% yield through bromination and dehydrobromination from 3-acetyl-11-fluoro-androsta-4,6-dien-17-one (Vettel, 1998). It was dissolved in a small amount of dichloromethane and cyclohexane, and crystals were obtained by vapour diffusion.



(I)

Crystal data

$C_{18}H_{21}FO_2$
M_r = 288.35
Monoclinic, $P2_1$
a = 9.4058 (5) Å
b = 7.7654 (4) Å
c = 10.2125 (6) Å
β = 94.349 (3)°
V = 743.77 (7) Å^3
Z = 2
D_x = 1.288 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3898 reflections
θ = 3.3–25.3°
μ = 0.09 mm^{-1}
T = 291 (1) K
Plate, colourless
0.35 × 0.30 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
206 frames *via* ω-rotation ($\Delta\omega$ = 1%) at different κ-angles and two times 60 s per frame
3898 measured reflections
1409 independent reflections
876 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 25.3°
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 12$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.096
S = 0.99
1409 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = −0.8 (16)

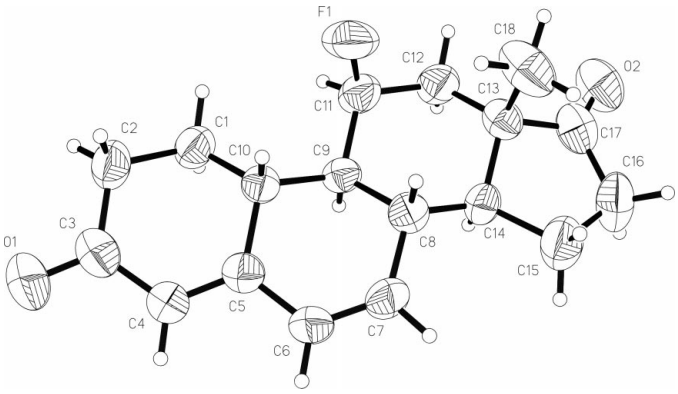


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···O1^{i}	0.93	2.46	3.280 (5)	147

Symmetry code: (i) $-x, \frac{1}{2}+y, 2-z$.

H atoms were placed in calculated positions with U_{iso} constrained to be $1.5U_{eq}$ of the carrier atom for the methyl groups and $1.2U_{eq}$ for the remaining positions. The absolute structure could not be determined reliably and the Friedel reflections were merged before final refinement because of the large s.u. for the Flack parameter [−0.8 (16)].

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

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