Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

3,17-Dioxoandrosta-4,6-dien-11-yl acetate

Marc Uerdingen *et al.*

Electronic paper
This paper is published electronically. It meets the data-validation criteria for publication in Acta Crystallographica Section C. The submission has been checked by a Section C Co-editor though the text in the 'Comments' section is the responsibility of the authors.

Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

3,17-Dioxoandrosta-4,6-dien-11-yl acetate

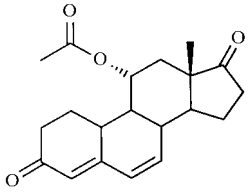
Marc Uerdingen, Markus Schürmann, Hans Preut* and Norbert Krause

Fachbereich Chemie, Universität Dortmund, Otto-Hahn-Straße 6, 44221 Dortmund, Germany
Correspondence e-mail: uch002@uxp1.hrz.uni-dortmund.de

Received 27 June 2000
Accepted 9 August 2000

Data validation number: IUC0000217

The synthesis and crystal structure analysis *via* X-ray diffraction of the title compound, $C_{20}H_{24}O_4$, (I), are described. The title compound is an androstadione bearing a diene in the *A* and *B* rings [Krause & Thorand (1999). *Inorg. Chim. Acta*, **296**, 1–11]. The diene conjugates with the carbonyl group. Intermolecular H···O contacts (2.53 and 2.64 Å; C—H···O angles 161 and 158°) indicate hydrogen bonds.



(I)

Experimental

The title compound was obtained in 80–90% yield through bromination and dehydrobromination of 3,11-diacetylandrosta-4,6-diene-17-one (Vettel, 1998). It was dissolved in a small amount of dichloromethane and cyclohexane, and crystals were obtained by vapor diffusion.

Crystal data

$C_{20}H_{24}O_4$
M_r = 328.39
Orthorhombic, $P2_12_12_1$
a = 9.7567 (3) Å
b = 10.3440 (3) Å
c = 17.0684 (6) Å
V = 1722.60 (9) Å^3
Z = 4
D_x = 1.266 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10 662 reflections
θ = 3.11–27.48°
μ = 0.087 mm^{-1}
T = 291 (1) K
Block, colourless
0.20 × 0.12 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
Method: 208 frames *via* ω-rotation ($\Delta\omega$ = 1°) at different κ values and two times 30 s per frame
10 662 measured reflections
2205 independent reflections
1217 reflections with I > 2sigma(I)
R_{int} = 0.024
θ_{max} = 27.48°
h = −12 → 12
k = −13 → 13
l = −22 → 22

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.078
S = 0.933
2205 reflections
220 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0400P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.10 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0064 (13)
Absolute structure: Flack (1983)
Flack parameter = 0.4 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O2^{i}	0.93	2.64	3.526 (3)	158
C10—H10···O4^{ii}	0.98	2.53	3.472 (2)	161

Symmetry codes: (i) $x-\frac{1}{2}, -\frac{1}{2}-y, -z$; (ii) $x-\frac{1}{2}, \frac{1}{2}-y, -z$.

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 with U_{iso} constrained to be 1.2U_{eq} of the carrier atom for the remaining positions. The absolute configuration was assumed from the synthesis. Friedel pairs were merged in the data set.

Data collection: Nonius KappaCCD software; 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); software used to prepare material for publication: *SHELXL*97.

This work is supported by the Deutsche Forschungsgemeinschaft, the Fonds der Chemischen Industrie and the Schering AG (Berlin)

References

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881
Krause, N. & Thorand, S. (1999). *Inorg. Chim. Acta*, **296**, 1–11.
Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Vettel, S. (1998). Schering AG, Berlin, Germany. Personal communication.