Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

13-Methyl-3-oxo-2,3,8,9,10,11,12,13,14,-15,16,17-dodecahydro-1*H*-cyclopenta[*a*]-phenanthren-17-yl 2-chloroacetate

Ji-Zhong Yan,* Jian Li and Guo-Wu Rao

College of Pharmaceutical Science, Zhejiang University of Technology, Hangzhou, People's Republic of China

Correspondence e-mail: zyx@zjut.edu.cn

Received 16 May 2006
Accepted 19 May 2006

Key indicators

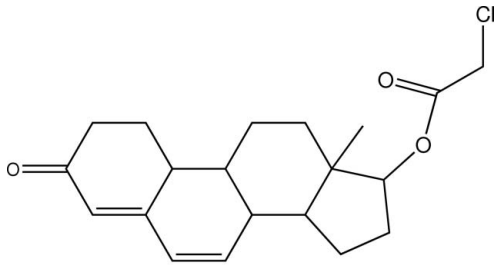
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.066
Data-to-parameter ratio = 18.8

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

The title compound, $C_{20}H_{25}ClO_3$, is built up from four fused rings, three of which are six-membered and one five-membered. The ring adjacent to the five-membered ring adopts a chair conformation.

Comment

Testosterone derivatives have a high biological activity and have been widely used in preparing hormone-based drugs (Alvarez-Ginarte *et al.*, 2005). As part of our continuing interest in the structure–activity relationship of testosterone derivatives, we have isolated the product, (I), from the reaction of 2-chloroacetyl chloride and 17-hydroxy-13-methyl-1,9,10,11,12,13,14,15,16,17-decahydro-2*H*-cyclopenta[*a*]phenanthren-3(8*H*)-one, as colorless crystals suitable for X-ray crystallographic analysis.



(I)

The molecular structure of (I) is illustrated in Fig. 1. It is built up from four fused rings, three of which are six-membered and one five-membered. The C1/C2/C11–C14 ring fused with the five-membered ring adopts a chair conformation (Cremer & Pople, 1975).

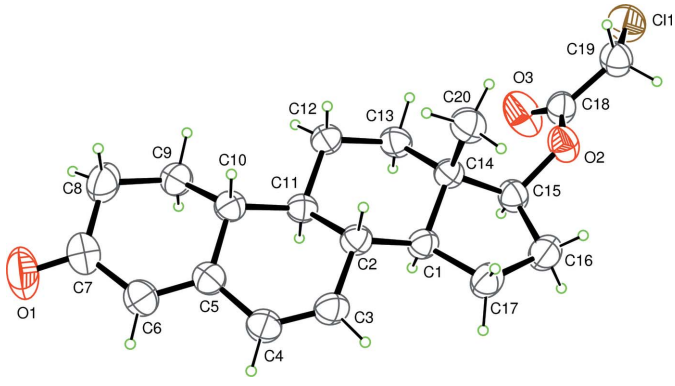


Figure 1
The structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Experimental

The title compound, (I), was prepared according to the procedure of Rao & Hu (2005). An ethanol solution of (I) was concentrated gradually at room temperature to afford colorless chunks.

Crystal data

$C_{20}H_{25}ClO_3$
M_r = 348.85
Orthorhombic, $P2_12_12_1$
a = 7.499 (2) Å
b = 15.365 (5) Å
c = 15.647 (4) Å
V = 1802.9 (9) Å^3
Z = 4
D_x = 1.285 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 298 (1) K
Chunk, colorless
0.26 × 0.21 × 0.14 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.918, T_{max} = 0.969
17271 measured reflections
4116 independent reflections
2498 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.034
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.066
S = 1.01
4116 reflections
219 parameters
H-atom parameters constrained
$w = 1/[1.0800\sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 112 (15)
Absolute structure: Flack (1983), with 1758 Friedel pairs
Flack parameter: 0.054 (5)

H atoms were included in the riding-model approximation, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1993); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

We are grateful to Professor Jian-Ming Gu of the Center of Analysis & Measurement of Zhejiang University for help with the X-ray diffraction experiment.

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Alvarez-Ginarte, Y. M., Crespo, R., Montero-Cabrera, L. A., Ruiz-Garcia, J. A., Ponce, Y. M., Santana, R., Pardillo-Fontdevila, E. & Alonso-Becerra, E. (2005). *QSAR Comb. Sci.* **24**, 218–226.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, C. K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Larson, A. C. (1970). *Crystallographic Computing, edited by F. R. Ahmed, S. R. Hall & C. P. Huber*, pp. 291–294. Copenhagen: Munksgaard.
Rao, G. W. & Hu, W. X. (2005). *Bioorg. Med. Chem. Lett.* **15**, 3174–3176.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.