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CRYSTALLOGRAPHIC COMMUNICATIONS

Structure of Equilenin at 100 K: an estrone related steroid

Christopher S. Frampton* and David D. MacNicol

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SCIENTIFIC MANUSCRIPT

For review only.

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Category: *research communications*

Co-editor:
Professor H. Stoeckli-Evans
Telephone: +41 32 7182400
Fax: +41 32 7182511
Email: helen.stoeckli-evans@unine.ch

Contact author:
Christopher S. Frampton
United Kingdom
Telephone: 01895 265337
Fax: ?
Email: chris.frampton@brunel.ac.uk

checkCIF/PLATON results for paper su5383

checkCIF/PLATON results
Ellipsoid plot

checkCIF/PLATON results

No syntax errors found. CIF dictionary Interpreting this report

Datablock: I

Bond precision: C-C = 0.0020 A Wavelength=1.54184
Cell: a=7.27709(7) b=7.32686(6) c=25.5179(2)
alpha=90 beta=90 gamma=90
Temperature: 100 K

	Calculated	Reported
Volume	1360.57(2)	1360.57(2)
Space group	P 21 21 21	P 21 21 21
Hall group	P 2ac 2ab	P 2ac 2ab
Moiety formula	C18 H18 O2	C18 H18 O2
Sum formula	C18 H18 O2	C18 H18 O2
Mr	266.32	266.32
Dx,g cm-3	1.300	1.300
Z	4	4
Mu (mm-1)	0.658	0.658
F000	568.0	568.0
F000'	569.63	
h,k,lmax	9,9,31	9,9,31
Nref	2767[1632]	2769
Tmin,Tmax	0.911,0.955	0.853,0.960
Tmin'	0.765	

Correction method= # Reported T Limits: Tmin=0.853 Tmax=0.960 AbsCorr = MULTI-SCAN
Data completeness= 1.70/1.00
Theta(max)= 74.465
R(reflections)= 0.0292(2754) wR2(reflections)= 0.0804(2769)
S = 1.008 Npar= 186

Alert level G

PLAT142_ALERT_4_G s.u. on b - Axis Small or Missing ..... 0.00006 Ang.
PLAT143_ALERT_4_G s.u. on c - Axis Small or Missing ..... 0.00020 Ang.
PLAT791_ALERT_4_G The Model has Chirality at C13 (Chiral SPGR) S Verify
PLAT791_ALERT_4_G The Model has Chirality at C14 (Chiral SPGR) S Verify
PLAT802_ALERT_4_G CIF Input Record(s) with more than 80 Characters 1 Info
PLAT978_ALERT_2_G Number C-C Bonds with Positive Residual Density. 19 Note

0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
6 **ALERT level G** = General information/check it is not something unexpected

0 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
1 ALERT type 2 Indicator that the structure model may be wrong or deficient
0 ALERT type 3 Indicator that the structure quality may be low
5 ALERT type 4 Improvement, methodology, query or suggestion
0 ALERT type 5 Informative message, check

database duplication summary

Datablock: ciftbxwarning

- **Chemical name =**
- *R* factor =
- Space group =
- Formula =
- a= b= c=
- alpha= beta= gamma=

Datablock: Inputlinelengthexceedsline_

- **Chemical name =**
- *R* factor =
- Space group =
- Formula =
- a= b= c=
- alpha= beta= gamma=

Datablock: I

- **Chemical name = Equilenin**
- *R* factor = 0.029
- Space group =
- Formula = C18 H18 O2
- a=7.27709 b=7.32686 c=25.5179
- alpha=90 beta=90 gamma=90

- Ohrt, J. M., Haner, B. A. & Norton, D. A. (1967). *Acta Cryst.***23**, 1100-1100 details
  [Cell: 7.48,25.528,7.279(90,90,90) *R*= *T*= Room Temp.(283-303)]

  Author Response: This paper was a unit cell determination only. No 3-dimensional structural data exists for this important steroid.

reference checking results

The following references were not checked in detail as they were not recognized as journal references

Fieser, L. F. & Fieser, M. (1959). *Steroids*. Reinhold Publishing Corporation, New York, 460--461.

Marshall, P. G. (1970). *Rodd's Chemistry of Carbon Compounds*, 2nd Edition., ed. Coffey S. Vol. IID, Elsevier, B. V., 216--222.

Rigaku Oxford Diffraction (2015). *CrysAlis PRO*. Rigaku Corporation, Oxford, UK.

The following references may be incorrectly formatted

Bossche, G. van den (1971). *Bull. Soc. Roy. Sci. Liege*, **40**, 614--?.
[*Unrecognized journal title.*]

Cruickshank, D. W. J. & Sparks, R. A. (1960). Proc. Roy. Soc. A. 258, 270--285.
[*Unrecognized journal title.*]

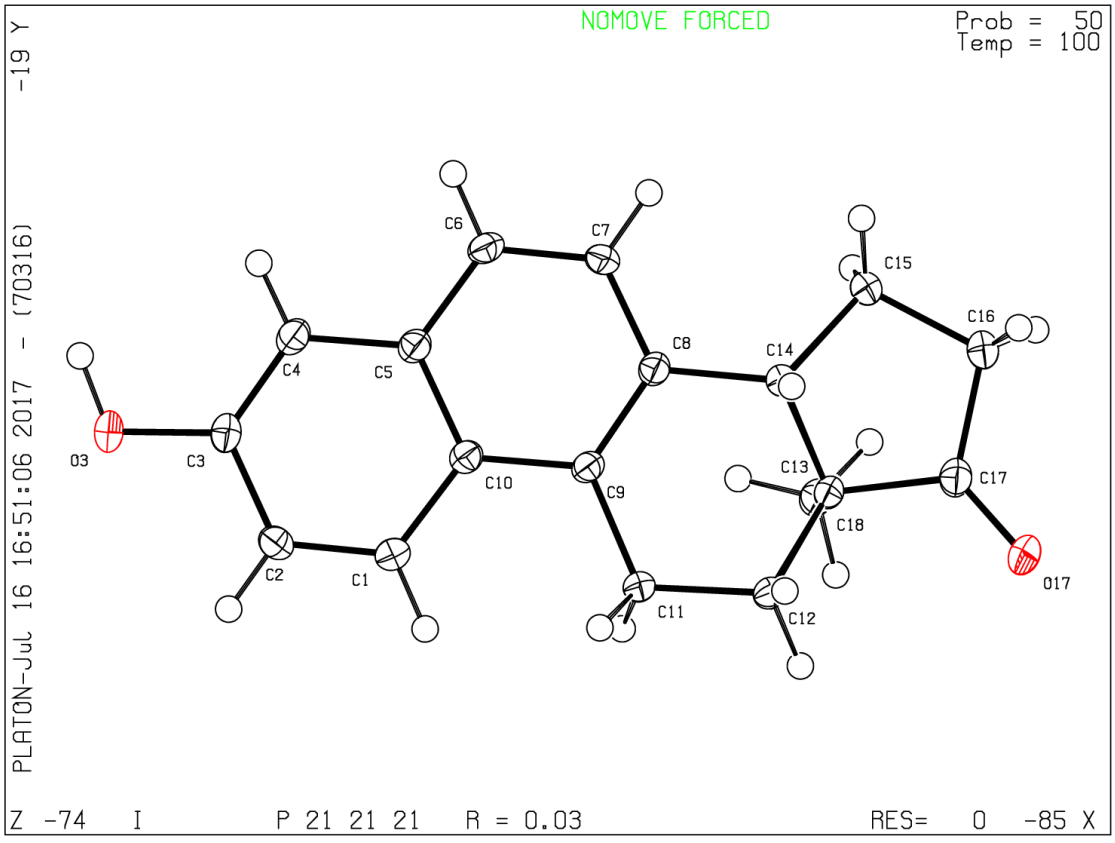
Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). Crystal Structures of Steroids, in Topics in Stereochemistry, eds. Allinger, N. L., Eliel, E. L., 9, John Wiley & Sons, Inc., Hoboken, NJ, USA, pp. 271--383.
[*Missing final page numbers?*]

All references appear to be cited unambiguously

Citation comments

1 date found in data_I _publ_body_contents that could be part of a citation but not found in reference list: 2017

Datablock I - ellipsoid plot



Structure of Equilenin at 100 K: an estrone related steroid

Christopher S. Frampton[a]* and David D. MacNicol[b]

[a]Wolfson Centre for Materials Processing, Brunel University London, Kingston Lane, Uxbridge, UB8 3PH, U.K., and [b]Department of Chemistry, University of Glasgow, Glasgow, G12 8QQ, Scotland, U.K.

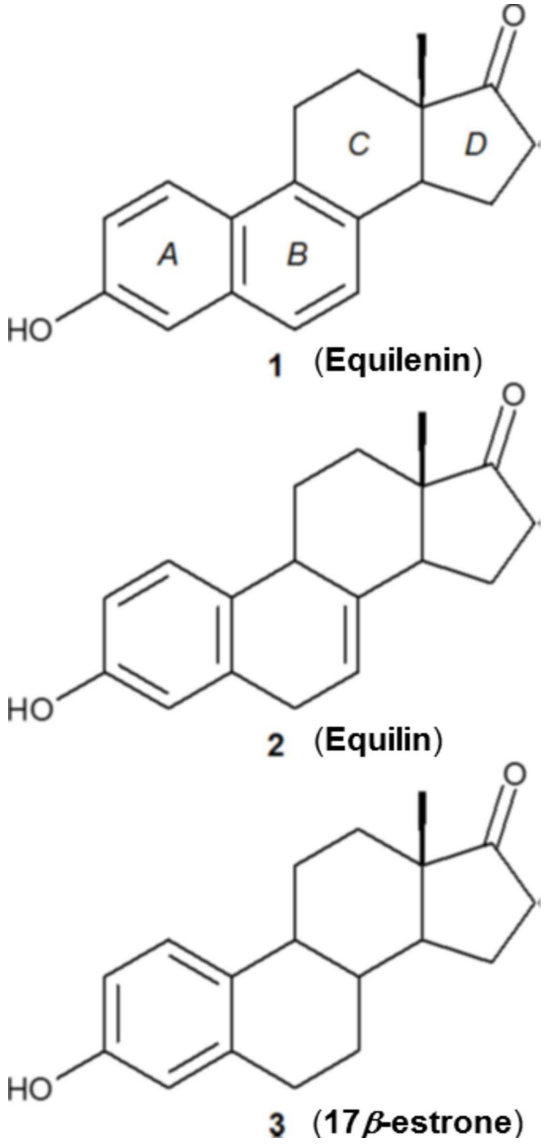
Correspondence email: Chris.Frampton@Brunel.ac.uk

Abstract

The structure of the estrone related steroid, Equilenin, $C_{18}H_{18}O_2$ (systematic name 3-hydroxy-13-methyl-11,12,13,14,15,16-hexahydro-cyclopenta[*a*]phenanthren-17-one), has been determined at 100 K. The crystals are orthorhombic, $P2_12_12_1$, and the absolute structure of the molecule in the crystal has been determined by resonant scattering [Flack parameter = 0.05 (4)]. The carbon atoms of the *A* and *B* rings, are coplanar with an r.m.s. deviation from planarity of 0.0104 Å. The *C* ring has a sofa conformation while the D ring has an envelope conformation with the methine C atom as the flap. The keto oxygen and the methyl group are translated 0.78 Å and 0.79 Å, respectively, from the equivalent positions on 17β-estrone **3**. In the crystal, molecules are linked by O—H···O hydrogen bonds forming chains parallel to the *c*-axis direction.

Keywords: crystal structure; Equilenin; Equilin; estrone; steroid; conformation; hydrogen bonding.

33scheme1.tif



1. Chemical context

The title compound, Equilenin **1**, is one member of a series of three estrogenic steroids, the other members being Equilin **2** and 17β-estrone **3**, that are components of the hormone replacement therapy medication, *'Premarin'*, a mixture of natural estrogens isolated from the urine of pregnant equine mares. It can be seen from the scheme that on going from 17β-estrone **3** through to the title compound Equilenin (**1**), there is a progressive aromatization of the *B* ring of the steroid framework where in **1** rings *A* and *B* comprise a fully aromatic naphthalene core. The structure of Equilin **2**, was determined by Sawicki *et al.* (1999*b*), who demonstrated that the presence of the unsaturated C7—C8 bond in the *B* ring rotates the *C* and *D* rings of the steroid such that the 17-keto oxygen atom, O17, is translated by 0.73 Å with respect to the analogous oxygen atom of **3** when an overlay of the two structures was performed based on the superposition of the *A* rings. The translation of the oxygen atom was implicated in the increased anti-human estrogenic 17β-hydroxysteroid dehydrogenase 1 (17β-HSD1) inhibitory behaviour of **2** with respect to 17β-estone **3** (Sawicki *et al.*, 1999*a*). The impact of the inhibitory behaviour of **2** is that it causes a reduction of the active estrogen, 17β-estradiol, which is present in elevated concentrations in human breast tumour tissues and responsible for the accelerated growth of the tumour tissue. It is

therefore of great interest to investigate what the structural and conformational consequences are on the *C* and *D* rings of the steroid framework of **1** by having fully unsaturated *A* and *B* rings. Although the unit-cell parameters of **1** at room temperature have been previously reported by Ohrt *et al.* (1967), no three-dimensional structure analysis of this important estrone steroid has been determined. Herein, we report on the crystal structure of this final member of the estrone series of steroids, Equilenin **1**, at 100 K.

2. Structural commentary

The crystal structure of Equilenin **1**, is orthorhombic, space group $P2_12_12_1$ (Z' = 1) and its molecular structure is illustrated in Fig. 1. The unit cell data agree with the previously reported values (Ohrt *et al.*, 1967) with the caveat that they are slightly smaller owing to some modest isotropic contraction due to the lower temperature. The atoms C1 through C10, which define the *AB* (napthalene) plane, are little affected by the chiral centres at C13 and C14, and are coplanar with an r.m.s. deviation of the fitted atoms of 0.0104 Å and a total puckering amplitude, Q, of 0.033 (2) Å. The greatest displacement from the ten atom mean plane is atom C10 at −0.019 (1) Å. The C—C bond lengths of the *AB* rings follow the pattern in which C1–C2, C3–C4, C6–C7 and C8–C9 are significantly shorter, (mean value 1.372 Å), than the remaining 7 bonds (mean value 1.421 Å) [Ahmed & Cruickshank, 1952; Cruickshank & Sparks, 1960], thus demonstrating that the *AB* ring is a true aromatic naphthalene core. The aromatization of ring *B* does however, have a significant effect on the conformations of both the *C* and *D* rings of **1**, compared to **2** and **3.** In contrast to the regular chair conformation of the *C* rings of **2** and **3**, the *C* ring of **1**, has a highly symmetric 13*β*-envelope conformation characterized by a $\Delta C_s(9)$ asymmetry parameter of 0.50° (Duax *et al.*, 1976); and related pairs of torsion angles [C14–C8–C9–C11, C8–C9–C11–C12, −4.1 (2), 4.1 (2)°; C9–C11–C12–C13, C9–C8–C14–C13, −32.6 (2), 32.7 (2)°; C11–C12–C13–C14, C12–C13–C14–C8, 60.4 (2), −61.3 (1)°]. The downside impact of this conformational change in the *C* ring of **1** is such that in place of the asymmetric twist or half-chair *D* ring conformation demonstrated by **2** and **3**, the *D* ring of Equilenin **1** displays a 14*α*-envelope conformation with a $\Delta C_s(14)$ of 4.20°; the torsion angles for **1**, (with related torsion angles for **2**/**3** are given in [/]) are C13–C14–C15–C16, C17–C13–C14–C15) −41.3 (2)[−40.2/-39.0]°, 43.3 (2) [44.5/42.9]°; C14–C13–C17–C16, C14–C15–C16–C17, −28.6 (2)[−31.0/-30.9]°, 22.3 (2)[19.6/19.4]°; and C15–C16–C17–C13, 3.6 (2)[8.1/7.5]°. Torsional angle data for **2** and **3** was extracted from structures GODTIC (Sawicki *et al.*, 1999*b*) and ESTRON13 (Shikii *et al.*, 2004), respectively [see Section 4, Database survey]. Compounds **1** and **2**, possibly owing to increased conformational constraint in the *B* ring, have lower oestrongenic activity than 17*β*-estrone itself, which has the *B* ring as the principal point of molecular flexibility (Duax *et al.*, 1976; Busetta *et al.*, 1973). Interestingly this reduction in activity (Marshall, 1970) does not directly relate to the crystallographically determined degree to which the *A* and *B* rings of the steroid are constrained to coplanarity, since **1**, possessing an essentially planar naphthalene core, is about five times more estrogenic than **2** which features only approximate coplanarity of its *A* and *B* rings with an r.m.s. deviation of the fitted atoms of 0.102 Å, and a total puckering amplitude of 0.270 (2) Å (Sawicki *et al.*, 1999*b*). An overlay of structures **1** (red), **2** (blue) and **3** (green) is shown in Fig. 2. The overlay was performed by a superposition of the atoms in the *A* ring only. From this overlay it can be calculated that the keto oxygen atom is translated by 0.78 Å and 0.69 Å, respectively, for compounds **1** and **2** from its position on **3**. Perhaps more significant is the degree of translation of the methyl group C18 which is translated by 0.79 Å and 1.40 Å, respectively, for compounds **1** and **2** from its position on **3** which may account for the increased estrogenic activity of **1** over **2**. The stereochemistry assignments at C13 and C14 are *S*, *S*; confirmed by resonant scattering through the Flack *x* parameter value of −0.05 (4).

3. Supramolecular features

In the crystal, the Equilenin **1** molecules are linked head-to-tail by a single O—H···O^{i} hydrogen bond (Table 1), to form chains propagating along the *c*-axis direction. A view along the *b*-axis of the crystal packing of the title compound is shown in Fig. 3.

4. Database survey

A search of the Cambridge Structural Database (CSD, Version 5.38, last update February 2017; Groom *et al.*, 2016) for the basic steroid *ABCD* ring framework yielded 401 hits although the hits for 1 and 2 could only be accessed by introducing the aromaticity into the *B* ring. Of the 401 hits there were 8 hits for the structure of 17*β*-estrone, **3** (ESTRON03–05 and ESTRON10–15) which exists in three polymorphic forms. They include, form I, orthorhombic $P2_12_12_1$ (ESTRON11: Busetta *et al.*, 1973), form II, orthorhombic $P2_12_12_1$ (ESTRON03: Debaerdemaeker, 1972; ESTRON04: van den Bossche, 1971); ESTRON10: Busetta *et al.*, 1973; ESTRON13: Shikii *et al.*, 2004; ESTRON14: Zhurova *et al.*, 2006) and form III, monoclinic $P2_1$ [Z' = 2] (ESTRON05, unit cell determination only: Ohrt *et al.*, 1964; ESTRON12: Busetta *et al.*, 1973). The polymorphic behaviour appears to be attributable to the crystal packing and has no significant influence on the conformation of the steroid framework. There was a single entry for **2** (GODTIC: Sawicki *et al.*, 1999*b*) and a single entry for **1** (QQQAMM, unit cell determination only: Ohrt *et al.*, 1967).

5. Synthesis and crystallization

In common with Equilin **2** the title compound, **1**, was isolated from the urine of a pregnant mare (Girard *et al.*, 1932; Fieser & Fieser, 1959). The sample used for the X-ray data collection was gifted to us from the J. W. Cook collection of the University of Glasgow. Suitable crystals were obtained as needles from ethanol, m.p. 531–532 K (evacuated sealed capillary).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The O-bound H atom was located from a difference-Fourier map and freely refined. All remaining H atoms were placed geometrically in idealized positions and refined using a riding model (including free rotation about the methyl C–C bond): C–H = 0.95–0.99 Å with U_{iso}(H) = 1.5U_{eq}(*C*-methyl) and 1.2U_{eq}(C) for other H atoms. The absolute stereochemistry of **1**, was confirmed through the Flack *x* parameter value of −0.05 (4). This was determined using 1130 quotients [(I+)-(I–)]/[(I+)+(I–)] (Parsons *et al.*, 2013).

fig1.tif

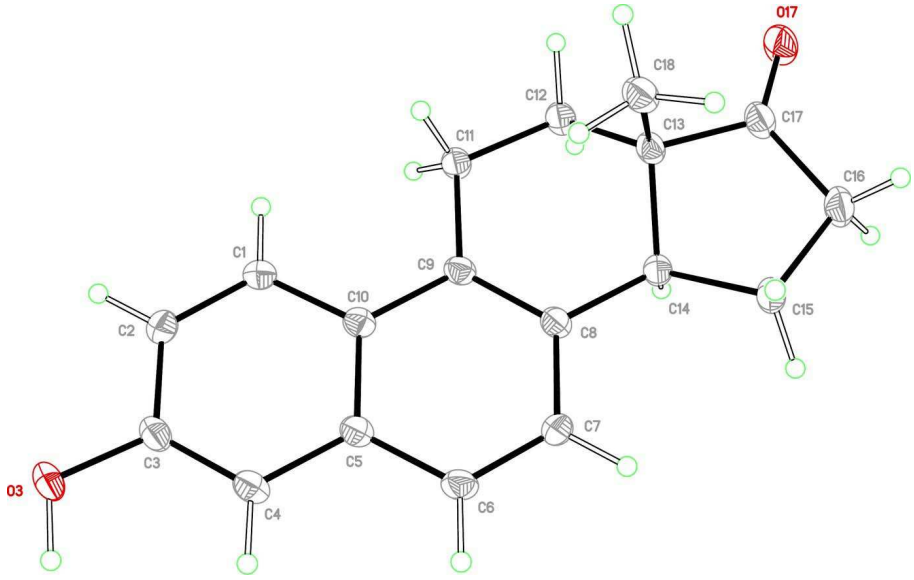


Figure 1

View of the molecular structure of compound **1**, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

fig2.tif

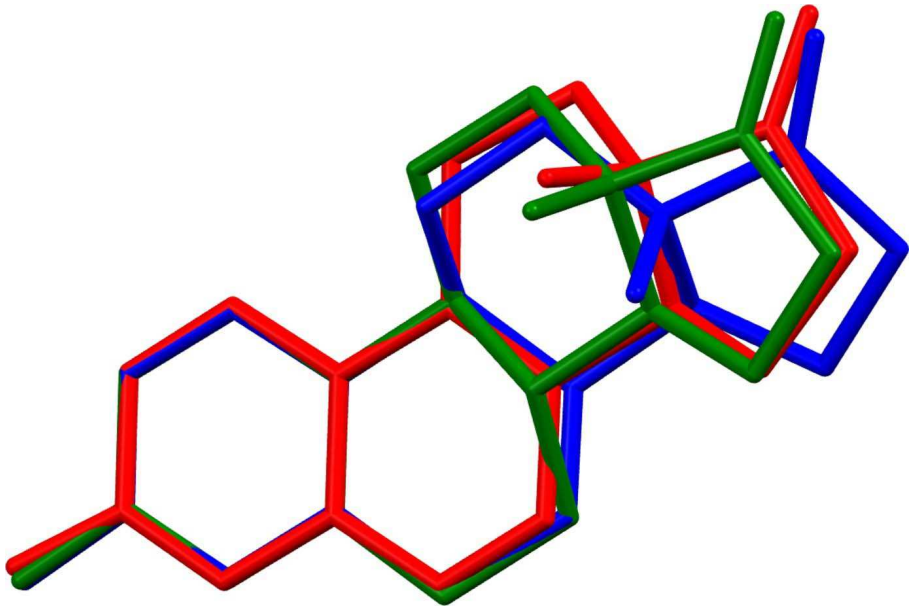


Figure 2

View of the structure overlay of compounds **1** (red), **2** (blue) and **3** (green). The overlay was performed by a superposition of the atoms in the *A* ring only.

fig3.tif

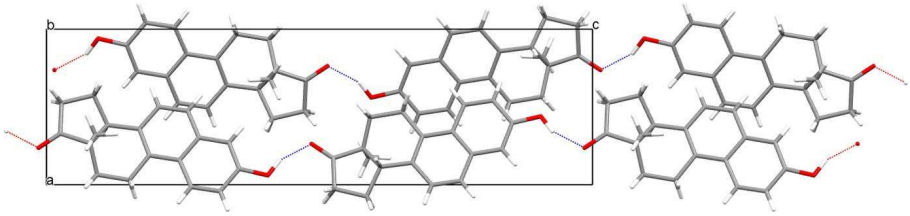


Figure 3

View along the *b* axis of the crystal packing of compound **1**. The intermolecular O—H···O hydrogen bonds are shown as dashed lines (see Table 1).

Table 1

Experimental details

Crystal data	
Chemical formula	$C_{18}H_{18}O_2$
M_r	266.32
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, *b*, *c* (Å)	7.27709 (7), 7.32686 (6), 25.5179 (2)
V (Å^3)	1360.57 (2)
Z	4
Radiation type	Cu *Kα*
μ (mm^{-1})	0.66
Crystal size (mm)	0.41 × 0.12 × 0.07
Data collection	
Diffractometer	SuperNova, Dualflex, AtlasS2
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.853, 0.960
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16660, 2769, 2754
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.029, 0.080, 1.01
No. of reflections	2769
No. of parameters	186
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.17
Absolute structure	Flack x determined using 1130 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons et al., 2013)
Absolute structure parameter	−0.05 (4)

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXD2014*/6 (Sheldrick, 2010), *SHELXL2014*/6 (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008) and *Mercury CSD 2.0* (Macrae *et al.*, 2008), *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

Table 2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···O17^{i}	0.95 (3)	1.82 (3)	2.7153 (17)	157 (3)

Symmetry code: (i) −*x*+1/2, −*y*+1, *z*−1/2.

Acknowledgements

We thank the University of Glasgow for the gift of the sample from the J. W. Cook collection.

References

Ahmed, F. R. & Cruickshank, D. W. J. (1952). *Acta Cryst.* **5**, 852–853.

Bossche, G. van den (1971). *Bull. Soc. Roy. Sci. Liege*, **40**, 614–?.

Busetta, B., Courseille, C. & Hospital, M. (1973). *Acta Cryst.* B**29**, 298–313.

Cruickshank, D. W. J. & Sparks, R. A. (1960). Proc. Roy. Soc. A. 258, 270–285.

Debaerdemaeker, T. D. J. (1972). *Cryst. Struct. Commun.*, **1**, 39–42.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Crystal Structures of Steroids, in Topics in Stereochemistry*, eds. Allinger, N. L., Eliel, E. L., 9, John Wiley & Sons, Inc., Hoboken, NJ, USA, pp. 271–383.

Fieser, L. F. & Fieser, M. (1959). *Steroids*. Reinhold Publishing Corporation, New York, 460–461.

Girard, A., Sandulesco, G., Fridenson, A., Gudefroy, C. & Rutgers, J. J. (1932). Compt. Rend. Acad. Sci. 194, 1020-1022.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Marshall, P. G. (1970). *Rodd's Chemistry of Carbon Compounds*, 2nd Edition., ed. Coffey S. Vol. IID, Elsevier, B. V., 216–222.

Ohrt, J. M., Haner, B. A. & Norton, D. A. (1964). *Acta Cryst.* **17**, 1611.

Ohrt, J. M., Haner, B. A. & Norton, D. A. (1967). *Acta Cryst.* **23**, 1100.

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.

Rigaku Oxford Diffraction (2015). *CrysAlis PRO*. Rigaku Corporation, Oxford, UK.

Sawicki, M. W., Erman, M., Puranen, T., Vihko, P. & Ghosh, D. (1999*a*). Proc. Natl. Acad. Sci. 96, 840–845.

Sawicki, M. W., Li, N. & Ghosh, D. (1999*b*). *Acta Cryst.* C**55**, 425–427.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Sheldrick, G. M. (2010). *Acta Cryst.* D**66**, 479–485.

Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

Shikii, K., Sakamoto, S., Seki, H., Utsumi, H. & Yamaguchi, K. (2004). *Tetrahedron*, **60**, 3487–3492.

168 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

169 Zhurova, E. A., Matta, C. F., Wu, N., Zhurov, V. V. & Pinkerton, A. A. (2006). *J. Am. Chem. Soc.* **128**, 8849–8861.

supporting information

Structure of Equilenin at 100 K: an estrone related steroid

Christopher S. Frampton* and David D. MacNicol

Computing details

Data collection: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); cell refinement: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); data reduction: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015); program(s) used to solve structure: *SHELXD2014*/6 (Sheldrick, 2010); program(s) used to refine structure: *SHELXL2014*/6 (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury CSD 2.0* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

3-Hydroxy-13-methyl-11,12,13,14,15,16-hexahydro- cyclopenta[a]phenanthren-17-one

Crystal data

$C_{18}H_{18}O_2$
$M_r = 266.32$
Orthorhombic, $P2_12_12_1$
$a = 7.27709\ (7)$ Å
$b = 7.32686\ (6)$ Å
$c = 25.5179\ (2)$ Å
$V = 1360.57\ (2)$ Å^3
$Z = 4$
$F(000) = 568$
$D_x = 1.300$ Mg m^{-3}
Melting point: 531 K
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 13177 reflections
$\theta = 3.5–76.4°$
$\mu = 0.66$ mm^{-1}
$T = 100$ K
Rod, colourless
0.41 × 0.12 × 0.07 mm

Data collection

SuperNova, Dualflex, AtlasS2 diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Detector resolution: 5.2921 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
$T_{min} = 0.853$, $T_{max} = 0.960$
16660 measured reflections
2769 independent reflections
2754 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 74.5°$, $\theta_{min} = 3.5°$
$h = -9\rightarrow7$
$k = -9\rightarrow9$
$l = -31\rightarrow31$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.080$
$S = 1.01$
2769 reflections
186 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.310P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Absolute structure: Flack x determined using 1130 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons et al., 2013)
Absolute structure parameter: −0.05 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.06030 (17)	0.42177 (17)	0.09478 (4)	0.0234 (3)
H3A	0.155 (4)	0.472 (4)	0.0738 (10)	0.058 (8)*
O17	0.23720 (17)	0.44067 (19)	0.51461 (4)	0.0277 (3)
C1	−0.0140 (2)	0.4004 (2)	0.23492 (6)	0.0184 (3)
H1A	−0.1120	0.3698	0.2579	0.022*
C2	−0.0417 (2)	0.3930 (2)	0.18189 (6)	0.0191 (3)
H2A	−0.1585	0.3588	0.1685	0.023*
C3	0.1025 (2)	0.4357 (2)	0.14700 (6)	0.0184 (3)
C4	0.2710 (2)	0.4870 (2)	0.16578 (6)	0.0185 (3)
H4A	0.3673	0.5152	0.1420	0.022*
C5	0.3030 (2)	0.4983 (2)	0.22075 (6)	0.0164 (3)
C6	0.4744 (2)	0.5542 (2)	0.24123 (6)	0.0191 (3)
H6A	0.5719	0.5844	0.2181	0.023*
C7	0.5009 (2)	0.5651 (2)	0.29424 (6)	0.0182 (3)
H7A	0.6172	0.6022	0.3073	0.022*
C8	0.3584 (2)	0.5223 (2)	0.32991 (6)	0.0162 (3)
C9	0.1889 (2)	0.4662 (2)	0.31167 (6)	0.0156 (3)
C10	0.1585 (2)	0.4530 (2)	0.25640 (6)	0.0156 (3)
C11	0.0307 (2)	0.4177 (2)	0.34819 (6)	0.0181 (3)
H11A	−0.0010	0.2877	0.3427	0.022*
H11B	−0.0777	0.4910	0.3379	0.022*
C12	0.0654 (2)	0.4477 (2)	0.40723 (6)	0.0188 (3)
H12A	0.0321	0.5743	0.4169	0.023*
H12B	−0.0125	0.3634	0.4279	0.023*
C13	0.2666 (2)	0.4136 (2)	0.41985 (5)	0.0174 (3)
C14	0.3840 (2)	0.5486 (2)	0.38816 (6)	0.0175 (3)
H14A	0.3364	0.6733	0.3965	0.021*
C15	0.5749 (2)	0.5349 (3)	0.41347 (6)	0.0233 (3)
H15A	0.6510	0.6432	0.4053	0.028*
H15B	0.6404	0.4234	0.4020	0.028*
C16	0.5263 (2)	0.5265 (3)	0.47245 (6)	0.0278 (4)
H16A	0.6101	0.4423	0.4911	0.033*
H16B	0.5360	0.6491	0.4886	0.033*
C17	0.3293 (2)	0.4572 (2)	0.47493 (6)	0.0210 (3)
C18	0.3214 (2)	0.2130 (2)	0.41011 (6)	0.0209 (3)

H18A	0.2300	0.1321	0.4261	0.031*
H18B	0.4422	0.1896	0.4257	0.031*
H18C	0.3269	0.1899	0.3723	0.031*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0254 (6)	0.0327 (6)	0.0121 (5)	−0.0031 (5)	−0.0025 (4)	0.0006 (4)
O17	0.0239 (6)	0.0460 (7)	0.0131 (5)	−0.0037 (6)	0.0025 (4)	−0.0009 (5)
C1	0.0173 (7)	0.0209 (7)	0.0170 (7)	−0.0030 (6)	0.0015 (6)	0.0020 (5)
C2	0.0163 (7)	0.0216 (7)	0.0193 (7)	−0.0032 (6)	−0.0028 (6)	0.0011 (6)
C3	0.0219 (7)	0.0202 (7)	0.0132 (7)	0.0004 (6)	−0.0015 (6)	0.0014 (6)
C4	0.0191 (7)	0.0218 (7)	0.0146 (7)	0.0019 (6)	0.0025 (6)	0.0023 (5)
C5	0.0160 (7)	0.0183 (7)	0.0149 (7)	0.0011 (6)	0.0011 (5)	0.0020 (5)
C6	0.0146 (7)	0.0254 (8)	0.0171 (7)	0.0000 (6)	0.0037 (6)	0.0035 (6)
C7	0.0122 (7)	0.0243 (8)	0.0182 (7)	−0.0020 (6)	−0.0012 (6)	0.0030 (6)
C8	0.0157 (7)	0.0192 (7)	0.0136 (7)	0.0012 (6)	0.0005 (5)	0.0017 (6)
C9	0.0145 (7)	0.0183 (7)	0.0141 (6)	0.0004 (6)	0.0020 (5)	0.0019 (5)
C10	0.0153 (7)	0.0163 (7)	0.0152 (7)	0.0014 (6)	0.0012 (5)	0.0014 (5)
C11	0.0132 (7)	0.0269 (8)	0.0142 (7)	−0.0015 (6)	0.0011 (5)	0.0009 (6)
C12	0.0132 (7)	0.0291 (8)	0.0143 (6)	0.0007 (6)	0.0021 (5)	0.0000 (6)
C13	0.0146 (7)	0.0256 (7)	0.0121 (6)	0.0002 (6)	0.0006 (5)	0.0010 (6)
C14	0.0145 (7)	0.0241 (7)	0.0140 (7)	−0.0019 (6)	−0.0004 (5)	0.0004 (6)
C15	0.0154 (7)	0.0393 (9)	0.0153 (7)	−0.0042 (7)	−0.0021 (5)	0.0029 (7)
C16	0.0200 (8)	0.0483 (10)	0.0151 (7)	−0.0047 (8)	−0.0027 (6)	0.0019 (7)
C17	0.0193 (7)	0.0287 (8)	0.0148 (7)	0.0018 (7)	−0.0010 (5)	0.0011 (6)
C18	0.0195 (7)	0.0261 (8)	0.0170 (7)	0.0013 (6)	0.0010 (6)	0.0030 (6)

Geometric parameters (Å, º)

O3—C3	1.3714 (17)	C11—C12	1.5433 (19)
O3—H3A	0.95 (3)	C11—H11A	0.9900
O17—C17	1.220 (2)	C11—H11B	0.9900
C1—C2	1.369 (2)	C12—C13	1.520 (2)
C1—C10	1.423 (2)	C12—H12A	0.9900
C1—H1A	0.9500	C12—H12B	0.9900
C2—C3	1.411 (2)	C13—C17	1.5118 (19)
C2—H2A	0.9500	C13—C14	1.537 (2)
C3—C4	1.369 (2)	C13—C18	1.544 (2)
C4—C5	1.4243 (19)	C14—C15	1.535 (2)
C4—H4A	0.9500	C14—H14A	1.0000
C5—C6	1.413 (2)	C15—C16	1.547 (2)
C5—C10	1.429 (2)	C15—H15A	0.9900
C6—C7	1.369 (2)	C15—H15B	0.9900
C6—H6A	0.9500	C16—C17	1.523 (2)
C7—C8	1.414 (2)	C16—H16A	0.9900
C7—H7A	0.9500	C16—H16B	0.9900
C8—C9	1.381 (2)	C18—H18A	0.9800

C8—C14	1.5103 (19)	C18—H18B	0.9800
C9—C10	1.4309 (19)	C18—H18C	0.9800
C9—C11	1.5234 (19)		
C3—O3—H3A	111.0 (17)	C13—C12—H12A	109.7
C2—C1—C10	121.43 (14)	C11—C12—H12A	109.7
C2—C1—H1A	119.3	C13—C12—H12B	109.7
C10—C1—H1A	119.3	C11—C12—H12B	109.7
C1—C2—C3	120.34 (14)	H12A—C12—H12B	108.2
C1—C2—H2A	119.8	C17—C13—C12	116.95 (12)
C3—C2—H2A	119.8	C17—C13—C14	100.69 (12)
C4—C3—O3	124.14 (14)	C12—C13—C14	108.58 (12)
C4—C3—C2	120.39 (13)	C17—C13—C18	105.82 (13)
O3—C3—C2	115.46 (14)	C12—C13—C18	111.80 (13)
C3—C4—C5	120.47 (14)	C14—C13—C18	112.60 (12)
C3—C4—H4A	119.8	C8—C14—C15	121.14 (13)
C5—C4—H4A	119.8	C8—C14—C13	111.54 (13)
C6—C5—C4	121.68 (14)	C15—C14—C13	103.87 (12)
C6—C5—C10	118.77 (13)	C8—C14—H14A	106.5
C4—C5—C10	119.55 (14)	C15—C14—H14A	106.5
C7—C6—C5	120.43 (14)	C13—C14—H14A	106.5
C7—C6—H6A	119.8	C14—C15—C16	101.83 (12)
C5—C6—H6A	119.8	C14—C15—H15A	111.4
C6—C7—C8	121.33 (14)	C16—C15—H15A	111.4
C6—C7—H7A	119.3	C14—C15—H15B	111.4
C8—C7—H7A	119.3	C16—C15—H15B	111.4
C9—C8—C7	120.24 (13)	H15A—C15—H15B	109.3
C9—C8—C14	118.66 (13)	C17—C16—C15	105.59 (13)
C7—C8—C14	120.98 (14)	C17—C16—H16A	110.6
C8—C9—C10	119.37 (13)	C15—C16—H16A	110.6
C8—C9—C11	122.58 (13)	C17—C16—H16B	110.6
C10—C9—C11	118.06 (13)	C15—C16—H16B	110.6
C1—C10—C5	117.81 (13)	H16A—C16—H16B	108.8
C1—C10—C9	122.30 (13)	O17—C17—C13	125.78 (15)
C5—C10—C9	119.86 (14)	O17—C17—C16	125.78 (15)
C9—C11—C12	116.13 (13)	C13—C17—C16	108.43 (13)
C9—C11—H11A	108.3	C13—C18—H18A	109.5
C12—C11—H11A	108.3	C13—C18—H18B	109.5
C9—C11—H11B	108.3	H18A—C18—H18B	109.5
C12—C11—H11B	108.3	C13—C18—H18C	109.5
H11A—C11—H11B	107.4	H18A—C18—H18C	109.5
C13—C12—C11	109.95 (12)	H18B—C18—H18C	109.5
C10—C1—C2—C3	0.7 (2)	C10—C9—C11—C12	−175.62 (14)
C1—C2—C3—C4	−0.7 (2)	C9—C11—C12—C13	−32.63 (19)
C1—C2—C3—O3	179.27 (14)	C11—C12—C13—C17	173.40 (14)
O3—C3—C4—C5	179.87 (14)	C11—C12—C13—C14	60.42 (17)
C2—C3—C4—C5	−0.2 (2)	C11—C12—C13—C18	−64.41 (16)

C3—C4—C5—C6	−178.75 (15)	C9—C8—C14—C15	155.31 (15)
C3—C4—C5—C10	1.0 (2)	C7—C8—C14—C15	−28.7 (2)
C4—C5—C6—C7	179.52 (14)	C9—C8—C14—C13	32.65 (19)
C10—C5—C6—C7	−0.2 (2)	C7—C8—C14—C13	−151.31 (14)
C5—C6—C7—C8	−0.4 (2)	C17—C13—C14—C8	175.33 (13)
C6—C7—C8—C9	0.7 (2)	C12—C13—C14—C8	−61.31 (16)
C6—C7—C8—C14	−175.24 (15)	C18—C13—C14—C8	63.05 (16)
C7—C8—C9—C10	−0.4 (2)	C17—C13—C14—C15	43.25 (15)
C14—C8—C9—C10	175.63 (14)	C12—C13—C14—C15	166.61 (13)
C7—C8—C9—C11	179.82 (14)	C18—C13—C14—C15	−69.03 (16)
C14—C8—C9—C11	−4.1 (2)	C8—C14—C15—C16	−167.57 (14)
C2—C1—C10—C5	0.1 (2)	C13—C14—C15—C16	−41.33 (17)
C2—C1—C10—C9	178.36 (15)	C14—C15—C16—C17	22.97 (18)
C6—C5—C10—C1	178.82 (14)	C12—C13—C17—O17	33.7 (3)
C4—C5—C10—C1	−0.9 (2)	C14—C13—C17—O17	151.02 (18)
C6—C5—C10—C9	0.5 (2)	C18—C13—C17—O17	−91.6 (2)
C4—C5—C10—C9	−179.25 (14)	C12—C13—C17—C16	−145.92 (15)
C8—C9—C10—C1	−178.42 (14)	C14—C13—C17—C16	−28.56 (16)
C11—C9—C10—C1	1.3 (2)	C18—C13—C17—C16	88.83 (16)
C8—C9—C10—C5	−0.2 (2)	C15—C16—C17—O17	−175.96 (18)
C11—C9—C10—C5	179.60 (13)	C15—C16—C17—C13	3.62 (19)
C8—C9—C11—C12	4.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O17^{i}	0.95 (3)	1.82 (3)	2.7153 (17)	157 (3)

Symmetry code: (i) −*x*+1/2, −*y*+1, *z*−1/2.

other supporting information

Crystallographic Information File. su5383.cif

Structure factors. su5383Isup2.hkl