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(3*R*,5*S*,6*S*,8*R*,10*S*)-8-Acetyl-7-hydroxy-3-isopropenyl-10-methylperhydrophenanthren-1-one

Huub Kooijman,[a]* Florence C. E. Sarabèr,[b] Aede de Groot[b] and Anthony L. Spek[a]

[a]Bijvoet Center for Biomolecular Research, Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands, and [b]Laboratory of Organic Chemistry, Wageningen University, Dreijenplein 8, 6703 HB Wageningen, The Netherlands

Correspondence e-mail: h.kooijman@chem.uu.nl

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.099
Data-to-parameter ratio = 9.0

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

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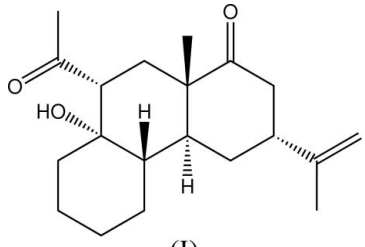
In the crystal structure of the title compound, $C_{20}H_{30}O_3$, there is an intramolecular hydrogen bond between the hydroxyl and acetyl groups [O···O = 2.722 (3) Å].

Comment

The structure of the title compound, (I), was determined in the course of our investigations toward the synthesis of polycyclic systems using Mukaiyama reactions (Sarabèr *et al.*, 2005). The molecular structure of (I) is displayed in Fig. 1. Selected geometric parameters are given in Table 1. All six-membered rings are in chair conformations, and the relevant asymmetry parameters (Duax & Norton, 1975) are all less than 10°. The absolute configuration of atom C5 was assigned as *S*. The other chiral centres, C3, C6, C7, C8 and C10, have configurations *R*, *S*, *S*, *R* and *S*, respectively.



(I)

The title compound displays an intramolecular hydrogen bond between the hydroxyl (O19) and acetyl (O21) groups (Fig. 1). The packing displays no intermolecular hydrogen bonds or short C—H···*A* contacts.

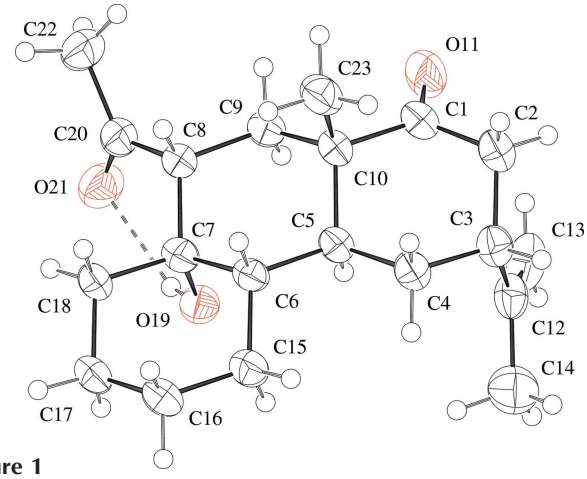


Figure 1
A plot of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

Experimental

Synthesis of the title compound is described elsewhere (Sarabèr *et al.*, 2005). Crystals of (I) suitable for diffraction experiments were obtained by evaporation of a solution of the title compound in hexane.

Crystal data

$C_{20}H_{30}O_3$
M_r = 318.44
Orthorhombic, $P2_12_12_1$
a = 6.1236 (9) Å
b = 13.8160 (18) Å
c = 21.026 (3) Å
V = 1778.9 (4) Å^3
Z = 4
D_x = 1.189 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 290 reflections
θ = 2.0–25.0°
μ = 0.08 mm^{-1}
T = 150 K
Block, colourless
0.3 × 0.2 × 0.1 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offset
Absorption correction: none
19009 measured reflections
1916 independent reflections
1214 reflections with $I > 2\sigma(I)$
R_{int} = 0.104
θ_{max} = 25.4°
$h = -7 \rightarrow 7$
$k = -16 \rightarrow 16$
$l = -24 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.099
S = 1.04
1916 reflections
214 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O11—C1	1.223 (4)	O21—C20	1.221 (4)
O19—C7	1.447 (4)		
O11—C1—C2	121.5 (3)	O21—C20—C22	120.1 (3)
O11—C1—C10	122.3 (3)	O21—C20—C8	122.3 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O19—H19···O21	0.88 (3)	2.01 (3)	2.722 (3)	137 (3)

In the absence of significant anomalous scatterers, Friedel pairs were averaged. The absolute configuration at atom C5 was chosen in accordance with the starting material. The hydroxyl H atom was refined freely. All other H atoms were introduced in calculated positions, with C—H = 0.95–1.00 Å, and refined as riding on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(methyl C) or U_{iso}(H) = 1.2U_{eq}(other C).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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References

Duax, W. L. & Norton, D. A. (1975). In *Atlas of Steroid Structures*. New York: Plenum.
Nonius (1998). *COLLECT Software*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sarabèr, F. C. E., Dratch, S., Bosselaar, G., Jansen, B. J. M. & Groot, A. de (2005). *Tetrahedron*. Submitted.
Sheldrick, G. M. (1985). *SHELXS86*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.