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

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.

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 (Sarabér 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

\[ \text{C}_{20}\text{H}_{30}\text{O}_3 \]

\[ M_r = 318.44 \]

Orthorhombic, \( P_{2_1}2_12_1 \)

\( a = 6.1236 \) (9) \( \text{Å} \)

\( b = 13.8160 \) (18) \( \text{Å} \)

\( c = 21.026 \) (3) \( \text{Å} \)

\( V = 1778.9 \) (4) \( \text{Å}^3 \)

\( Z = 4 \)

\( \rho = 1.189 \) Mg \( \text{m}^{-3} \)

Mo Ka radiation

Cell parameters from 290

reflections

\( \theta = 2.0-25.0^\circ \)

\( \mu = 0.08 \) mm\(^{-1} \)

\( T = 150 \) K

Block, colourless

Data collection

Bruker Nonius KappaCCD area-detector diffractometer

\( \psi \) scans, and \( \omega \) scans with \( \kappa \) offset

Absorption correction: none

19699 measured reflections

1916 independent reflections

1214 reflections with \( I > 2\sigma(I) \)

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

\( \Delta F_{	ext{max}} = 0.15 \) e \( \text{Å}^{-3} \)

\( \Delta F_{	ext{min}} = -0.19 \) e \( \text{Å}^{-3} \)

Table 1

<table>
<thead>
<tr>
<th></th>
<th>( \text{O}11-\text{C}1 )</th>
<th>( \text{O}19-\text{C}7 )</th>
<th>( \text{O}11-\text{C}1-\text{C}2 )</th>
<th>( \text{O}11-\text{C}1-\text{C}10 )</th>
<th>( \text{O}21-\text{C}20 )</th>
<th>( \text{O}21-\text{C}20-\text{C}22 )</th>
<th>( \text{O}21-\text{C}20-\text{C}8 )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.223 (4)</td>
<td>1.447 (4)</td>
<td>121.5 (3)</td>
<td>122.3 (3)</td>
<td>1.221 (4)</td>
<td>120.1 (3)</td>
<td>122.3 (3)</td>
</tr>
</tbody>
</table>

Table 2

Hydrogen-bond geometry (\( \text{Å} \), \( ^\circ \)).

<table>
<thead>
<tr>
<th>( \text{D}--\text{H}--\text{A} )</th>
<th>( \text{D}--\text{H} )</th>
<th>( \text{H}--\text{A} )</th>
<th>( \text{D}--\text{A} )</th>
<th>( \text{D}--\text{H}--\text{A} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>O19--H19---O21</td>
<td>0.88 (3)</td>
<td>2.01 (3)</td>
<td>2.722 (3)</td>
<td>137 (3)</td>
</tr>
</tbody>
</table>

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 \( U_{	ext{iso}}(\text{H}) = 1.5U_{	ext{eq}}(\text{methyl C}) \) or \( U_{	ext{iso}}(\text{H}) = 1.2U_{	ext{eq}}(\text{other C}) \).

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO (Otwinski & 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


