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# 10,13,17-Trimethyl-3-oxoperhydro-1*H*-cyclopenta[*a*]phenanthren-17-yl chloroacetate

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**Key indicators**

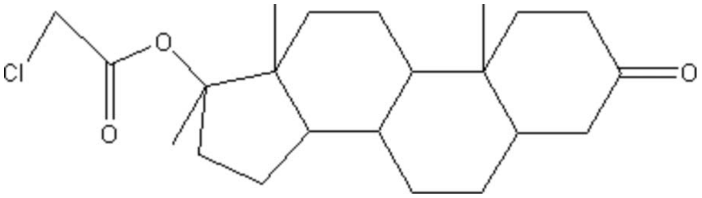
Single-crystal X-ray study
*T* = 298 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
*R* factor = 0.060
*wR* factor = 0.150
Data-to-parameter ratio = 18.3

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

The molecule of the title compound, $C_{22}H_{33}ClO_3$, is built up from four fused rings, three of which are six-membered and one five-membered. The three six-membered rings all have chair conformations. C—H···O hydrogen-bonding interactions give rise to chains in the solid state.

## Comment

Testosterone derivatives exhibit a high level of biological activity and have been widely used as hormone treatments (Alvarez-Ginarte *et al.*, 2005). As part of our continuing interest in the structure–activity relationship of testosterone derivatives, we have isolated the title compound, (I), the product of the reaction between 2-chloroacetyl chloride and 17-hydroxy-10,13,17-trimethyltetradecahydro-2*H*-cyclopenta-[*a*]phenanthren-3(4*H*)-one, as colourless crystals suitable for X-ray analysis.



(I)

The molecular structure of (I) is built up from four fused rings, three of which are six-membered and one five-membered (Fig. 1). The three six-membered rings have chair conformations (Cremer & Pople, 1975). The dihedral angles between the C6/C7/C9/C10 plane and the C1/C2/C4/C5 and C8/C11/C12/C14 planes are 8.90 (15) and 3.40 (14)°, respectively.

C—H···O interactions in the crystal structure of (I) lead to the formation of hydrogen-bonded chains (Table 1).

## Experimental

To a solution of 17-hydroxy-10,13,17-trimethyltetradecahydro-2*H*-cyclopenta[*a*]phenanthren-3(4*H*)-one (1.52 g, 5 mmol) and pyridine (0.79 g, 10 mmol) in dichloromethane (25 ml) was added 2-chloroacetyl chloride (0.678 g, 6 mmol). The reaction mixture was left at room temperature for 3 h and then concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel eluted with petroleum ether (303–333 K)–diethyl ether (10:1) to give the product (1.22 g) in 64.1% yield. A solution of the compound in ethanol was concentrated slowly at room temperature to afford (I) as colourless prisms (m.p. 417–419 K).

*Crystal data*

$C_{22}H_{33}ClO_3$
$M_r = 380.93$
Orthorhombic, $P2_12_12_1$
$a = 11.332$ (7) Å
$b = 13.225$ (8) Å
$c = 14.667$ (8) Å
$V = 2198$ (2) Å$^3$

$Z = 4$
$D_x = 1.151$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.19$ mm$^{-1}$
$T = 298$ (2) K
Prism, colourless
0.25 × 0.20 × 0.20 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.954$, $T_{\max} = 0.963$
9937 measured reflections

4541 independent reflections
3063 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.053$
$\theta_{\max} = 26.5°$
3 standard reflections
frequency: 60 min
intensity decay: 4.2%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.150$
$S = 0.96$
4541 reflections
248 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0839P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.35$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.24$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.021 (2)
Absolute structure: Flack (1983), with 1756 Friedel pairs
Flack parameter: 0.03 (12)

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C22$A$—H22$B\cdots$O1$^{\rm i}$ | 0.97 | 2.51 | 3.464 (4) | 169 |

Symmetry code: (i) $-x+\frac{3}{2}, -y+1, z+\frac{1}{2}$.

H atoms were included in calculated positions and refined using a riding model, with C—H distances of 0.96 Å for methyl H atoms, 0.97 Å for methylene H atoms or 0.98 Å for methine H atoms, and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C), or $1.5U_{\rm eq}$(C) for methyl H atoms. The chloromethyl group was found to display large displacement ellipsoids with unrealistic C—Cl bond lengths. Cl1, C22 and attached H atoms were modelled as disordered, with occupancy factors in the ratio 0.7:0.3; the ratio was initially set at 0.5:0.5, but the ratio of 0.7:0.3 was confirmed by the refinement.

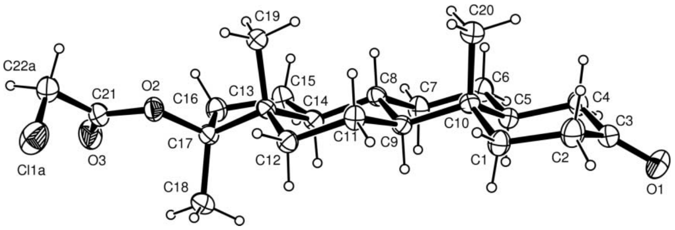


**Figure 1**
The molecular structure of (I), showing the atom-labelling scheme. Ellipsoids are drawn at the 30% probability level.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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