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# 17*β*-Ethynyl-17*α*-hydroxyandrosta-1,4-dien-3-one

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In the title molecule, $C_{21}H_{26}O_2$, two C═C bonds [1.327 (3) and 1.338 (3) Å] contribute to the planarity of ring *A* along with the carbonyl group. Rings *B* and *C* have regular chair conformations, and the five-membered ring *D* has an envelope conformation. In the crystal structure, intermolecular O—H⋯O hydrogen bonds link molecules into chains running along the *c* axis.

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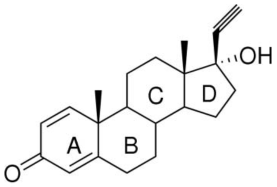
**Key indicators**

Single-crystal X-ray study
$T$ = 296 K
Mean $\sigma$(C–C) = 0.003 Å
$R$ factor = 0.037
$wR$ factor = 0.099
Data-to-parameter ratio = 10.6

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

## Comment

The title compound, (I), is used in the synthesis of prednisolone, because ring *A* is a fully functionalized 1,4-dien-3-one system (Nitta *et al.*, 1985). We present here its crystal structure, which exhibits a crystal packing and unit-cell dimensions close to those observed in 17*α*-hydroxy-4-pregnen-20-yn-3-one, (II) (Reisch *et al.*, 1993), and 17*α*-ethynyl-17*β*-hydroxyandrosta-1,4-diene-3-one, (III) (Sheng *et al.*, 2007).



(I)

In (I) (Fig. 1), all bond lengths and angles are normal and agree well with those observed in (II) and (III). The C1═C2, C4═C5, O1═C3 and O2—C17 distances are 1.327 (3), 1.338 (3), 1.236 (3) and 1.440 (2) Å, respectively. The ethynyl bond length is 1.172 (4) Å. Ring *A* and atoms O1 and C6 are coplanar, with an r.m.s. deviation of 0.018 (2) Å. Rings *B* and *C* show normal chair conformations similar to those reported by Xia *et al.* (2005). Ring *D* has an envelope conformation.

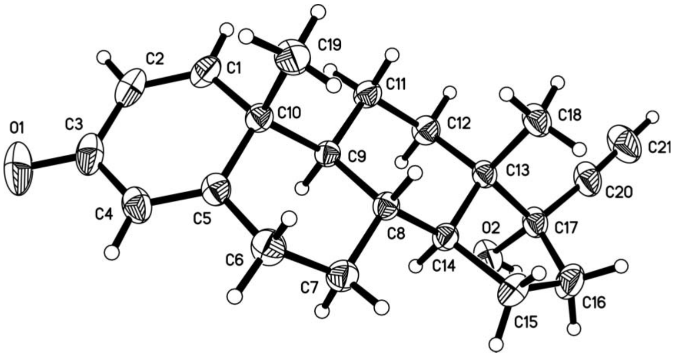


**Figure 1**
The molecular structure of (I), showing the atomic labelling and 30% probability displacement ellipsoids.

Atom C13 deviates by 0.702 (3) Å from the C14–C17 mean plane, which makes a dihedral angle of 71.19 (14)° with the O2/C17/C21/C22 mean plane.

In the crystal structure, intermolecular O—H···O hydrogen bonds (Table 1) link molecules into chains running along the *c* axis.

## Experimental

The title compound was synthesized as a powder according to Nitta *et al.* (1985). Crystals of X-ray quality were obtained as colourless prisms by slow evaporation of a solution in a mixture of tetrahydrofuran, acetone and water (4:4:2 *v*/*v*/*v*).

*Crystal data*

$C_{21}H_{26}O_2$
$M_r$ = 310.42
Orthorhombic, $P2_12_12_1$
$a$ = 7.6330 (12) Å
$b$ = 9.9270 (16) Å
$c$ = 22.766 (4) Å
$V$ = 1725.0 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 296 (2) K
0.30 × 0.20 × 0.20 mm

*Data collection*

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.978, $T_{\max}$ = 0.981
11020 measured reflections
2266 independent reflections
1909 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.030

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
$S$ = 1.03
2266 reflections
214 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{\min}$ = −0.15 e Å$^{-3}$

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O2—H2*X*···O1$^{\rm i}$ | 0.849 (17) | 1.982 (18) | 2.827 (2) | 173 (3) |

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

Hydroxy atom H2*X* was located in a difference Fourier map and refined isotropically, with an O—H bond length restraint of 0.85 (2) Å. The C-bound H atoms were placed at calculated positions (C—H = 0.93–0.98 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2 or 1.5$U_{\rm eq}$(C). In the absence of any significant anomalous scatterers in the molecule, 1793 Friedel pairs were merged before the final refinement. The absolute configuration was assigned to correspond with that of the known chiral centres in a precursor molecule, which remained unchanged during the synthesis of the title compound.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2003); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

## References

Bruker (2003). *SAINT*, *SHELXTL* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Nitta, I., Fujimori, S., Haruyama, T., Inoue, S. & Ueno, H. (1985). *Bull. Chem. Soc. Jpn*, **58**, 981–986.
Reisch, J., Zappel, J., Henkel, G. & Ekiz-Guecer, N. (1993). *Monatsh. Chem.* **124**, 1169–1175.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheng, L.-Q., Liang, W.-G., Chen, G. & Shen, H.-F. (2007). *Acta Cryst.* E**63**, o1182–o1183.
Xia, C.-N., Hu, W.-X. & Zhou, W. (2005). *Acta Cryst.* E**61**, o2896–o2898.

**o2354** Zhang *et al.* • $C_{21}H_{26}O_2$ *Acta Cryst.* (2007). E**63**, o2353–o2354