

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## Pregna-1,4,16-triene-3,20-dione

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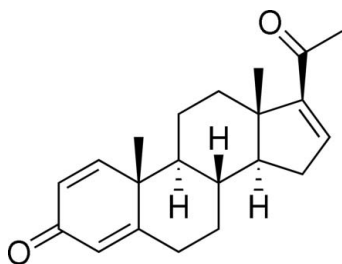
Received 21 July 2007; accepted 26 July 2007

Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.040;  $wR$  factor = 0.102; data-to-parameter ratio = 10.7.

In the title steroid compound,  $\text{C}_{21}\text{H}_{26}\text{O}_2$ , ring *A* is essentially planar due to the presence of two  $\text{C}=\text{C}$  bonds [1.326 (3) and 1.332 (3) Å]. Rings *B* and *C* have regular chair conformations, while ring *D* has an envelope conformation. Weak intermolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds stabilize the crystal packing.

## Related literature

For literature describing similar structures with similar properties to the title compound, see: Nitta *et al.* (1985); Reisch *et al.* (1993); Sheng *et al.* (2007); Xia *et al.* (2005). For related literature, see: Patil *et al.* (2002).



## Experimental

## Crystal data

$\text{C}_{21}\text{H}_{26}\text{O}_2$   
 $M_r = 310.42$

Orthorhombic,  $P2_12_12_1$   
 $a = 6.3625$  (12) Å

$b = 11.745$  (2) Å  
 $c = 23.007$  (4) Å  
 $V = 1719.2$  (5) Å<sup>3</sup>  
 $Z = 4$

Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 296$  (2) K  
 $0.25 \times 0.20 \times 0.15$  mm

## Data collection

Bruker APEXII CCD area-detector diffractometer  
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.981$ ,  $T_{\max} = 0.999$

10912 measured reflections  
2274 independent reflections  
1871 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.028$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$   
 $wR(F^2) = 0.102$   
 $S = 1.05$   
2274 reflections

212 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.15$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.13$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}19-\text{H}19\text{B}\cdots\text{O}1^{\text{i}}$	0.96	2.57	3.356 (4)	139
$\text{C}21-\text{H}21\text{A}\cdots\text{O}1^{\text{ii}}$	0.96	2.59	3.537 (3)	169

Symmetry codes: (i)  $-x + \frac{5}{2}, -y + 1, z + \frac{1}{2}$ ; (ii)  $x - \frac{1}{2}, -y + \frac{3}{2}, -z + 1$ .

Data collection: *APEX2* (Bruker, 2005); 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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

The authors thank Professor Chen for the X-ray data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2284).

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**supplementary materials**

*Acta Cryst.* (2007). E63, o3659 [ doi:10.1107/S1600536807036732 ]

## Pregna-1,4,16-triene-3,20-dione

N.-Q. Wang and T.-J. Wang

### Comment

The title compound, (I), is used in the synthesis of prednisolone, because the ring A is fully functionalized a 1,4-dien-3-one system (Nitta *et al.*, 1985). We present here its crystal structure, which exhibits the crystal packing close to those observed in 17 $\alpha$ -hydroxy-4-pregnen-20-yn-3-one (II) (Reisch *et al.*, 1993) and 17 $\alpha$ -hydroxypregna-1,4-diene-3,20-dione (III) (Sheng *et al.*, 2007). In (I) (Fig. 1), all bond lengths and angles agree with those in (II) and (III). The C1=C2, C4=C5, C16=C17, O1—C3 and O2—C18 distances are 1.326 (3) Å, 1.332 (3) Å, 1.332 (3) Å, 1.226 (3) Å and 1.221 (2) Å, respectively.

Ring A and atoms O1 and C6 are almost coplanar with the r.m.s. deviation of 0.061 (1) Å. Rings B and C show normal chair conformations, which are very similar to those reported by Xia *et al.* (2005). Ring D has an envelope conformation with atom C14 deviating at 0.589 (3) Å from the mean plane C13/C15/C16/C17 [in spite of structures (II) and (III) with the most deviating atom C13].

In the crystal, the weak intermolecular C—H $\cdots$ O hydrogen bonds (Table 1) stabilize the packing (Fig. 2).

### Experimental

Pregna-1,4,16-triene-3,20-dione was synthesized according to Patil *et al.* (2002) in a form of a powder. Crystals of (I) suitable for structure analysis were obtained by slow evaporation from a mixture of tetrahydrofuran, acetone and water (4:4:2, *v/v*).

### Refinement

Due to absence of any significant anomalous scatterers in the molecule, the 1591 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. 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  $U_{\text{iso}}(\text{H}_{\text{methyl}}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$  or  $U_{\text{iso}}(\text{H}_{\text{non-methyl}}) = 1.2U_{\text{eq}}(\text{C}_{\text{non-methyl}})$ .

### Figures



Fig. 1. The structure of (I) with 30% probability displacement ellipsoids.

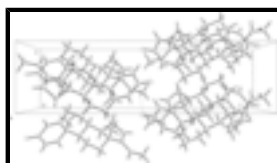


Fig. 2. Packing diagram of (I), viewed along the *b* axis. Hydrogen-bonds are shown as dashed lines.

## Pregna-1,4,16-triene-3,20-dione

### Crystal data

$C_{21}H_{26}O_2$	$F_{000} = 672$
$M_r = 310.42$	$D_x = 1.199 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
Hall symbol: P 2ac 2ab	$\lambda = 0.71073 \text{ \AA}$
$a = 6.3625 (12) \text{ \AA}$	Cell parameters from 3759 reflections
$b = 11.745 (2) \text{ \AA}$	$\theta = 3.2\text{--}26.9^\circ$
$c = 23.007 (4) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$V = 1719.2 (5) \text{ \AA}^3$	$T = 296 (2) \text{ K}$
$Z = 4$	Block, pale yellow
	$0.25 \times 0.20 \times 0.15 \text{ mm}$

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer	2274 independent reflections
Radiation source: fine-focus sealed tube	1871 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.028$
$T = 296(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
$\varphi$ and $\omega$ scans	$\theta_{\text{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 6$
$T_{\text{min}} = 0.981, T_{\text{max}} = 0.999$	$k = -15 \rightarrow 12$
10912 measured reflections	$l = -26 \rightarrow 29$

### Refinement

Refinement on $F^2$	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2 + 0.2215P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.102$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.15 \text{ e \AA}^{-3}$
2274 reflections	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
212 parameters	Extinction correction: SHELXL97,
Primary atom site location: structure-invariant direct methods	$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0073 (16)
Hydrogen site location: inferred from neighbouring sites	Absolute structure: see text
	Flack parameter:
	Rogers parameter: ?

*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.

**Refinement.** Refinement of  $F^2$  against ALL reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.9371 (4)	0.57024 (18)	0.48495 (8)	0.1004 (7)
O2	1.2908 (3)	0.79394 (18)	0.91843 (8)	0.0839 (6)
C1	1.0192 (4)	0.7610 (2)	0.60194 (9)	0.0619 (6)
H1	1.1142	0.8176	0.6123	0.074*
C2	1.0551 (4)	0.7048 (3)	0.55291 (10)	0.0723 (7)
H2	1.1738	0.7224	0.5311	0.087*
C3	0.9126 (5)	0.6160 (2)	0.53236 (10)	0.0683 (7)
C4	0.7407 (4)	0.5879 (2)	0.57074 (9)	0.0625 (6)
H4	0.6524	0.5281	0.5603	0.075*
C5	0.7016 (3)	0.64276 (17)	0.62024 (9)	0.0501 (5)
C6	0.5246 (4)	0.6074 (2)	0.65934 (9)	0.0617 (6)
H6A	0.4533	0.5420	0.6427	0.074*
H6B	0.4237	0.6690	0.6625	0.074*
C7	0.6068 (4)	0.57713 (18)	0.71942 (9)	0.0551 (6)
H7A	0.6922	0.5089	0.7168	0.066*
H7B	0.4891	0.5609	0.7449	0.066*
C8	0.7380 (3)	0.67328 (16)	0.74551 (8)	0.0428 (4)
H8	0.6477	0.7398	0.7515	0.051*
C9	0.9196 (3)	0.70629 (16)	0.70402 (8)	0.0406 (4)
H9	1.0036	0.6371	0.6987	0.049*
C10	0.8374 (3)	0.74005 (16)	0.64175 (8)	0.0461 (5)
C11	1.0676 (3)	0.79469 (18)	0.73120 (8)	0.0506 (5)
H11A	0.9940	0.8668	0.7341	0.061*
H11B	1.1869	0.8057	0.7055	0.061*
C12	1.1488 (3)	0.76146 (18)	0.79171 (9)	0.0500 (5)
H12A	1.2393	0.6953	0.7886	0.060*
H12B	1.2309	0.8235	0.8078	0.060*
C13	0.9649 (3)	0.73475 (15)	0.83207 (8)	0.0431 (5)
C14	0.8354 (3)	0.63945 (16)	0.80305 (8)	0.0437 (4)
H14	0.9376	0.5799	0.7934	0.052*
C15	0.7042 (4)	0.5901 (2)	0.85305 (9)	0.0614 (6)
H15A	0.5775	0.6341	0.8598	0.074*
H15B	0.6674	0.5111	0.8462	0.074*

## supplementary materials

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C16	0.8584 (4)	0.60245 (19)	0.90194 (9)	0.0605 (6)
H16	0.8498	0.5619	0.9366	0.073*
C17	1.0091 (4)	0.67786 (17)	0.89034 (8)	0.0491 (5)
C18	1.1800 (4)	0.7120 (2)	0.92989 (9)	0.0596 (6)
C19	1.2148 (5)	0.6444 (3)	0.98430 (10)	0.0841 (9)
H19A	1.3263	0.6782	1.0065	0.126*
H19B	1.2521	0.5677	0.9743	0.126*
H19C	1.0882	0.6439	1.0070	0.126*
C20	0.8361 (4)	0.84353 (17)	0.84474 (9)	0.0583 (6)
H20A	0.9170	0.8938	0.8689	0.087*
H20B	0.7084	0.8233	0.8644	0.087*
H20C	0.8031	0.8811	0.8088	0.087*
C21	0.7052 (4)	0.85114 (18)	0.64361 (10)	0.0650 (7)
H21A	0.6380	0.8624	0.6067	0.098*
H21B	0.7956	0.9146	0.6518	0.098*
H21C	0.6004	0.8451	0.6735	0.098*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.1292 (18)	0.1085 (15)	0.0636 (11)	0.0078 (15)	0.0199 (12)	-0.0263 (10)
O2	0.0872 (14)	0.0929 (13)	0.0717 (11)	-0.0241 (13)	-0.0179 (10)	0.0001 (10)
C1	0.0630 (14)	0.0734 (15)	0.0493 (11)	-0.0159 (13)	-0.0024 (11)	0.0101 (11)
C2	0.0675 (16)	0.0992 (19)	0.0502 (12)	-0.0030 (16)	0.0145 (12)	0.0093 (13)
C3	0.0827 (18)	0.0691 (15)	0.0532 (13)	0.0111 (15)	0.0042 (13)	-0.0050 (11)
C4	0.0750 (16)	0.0569 (13)	0.0555 (13)	-0.0038 (13)	-0.0047 (12)	-0.0082 (11)
C5	0.0515 (12)	0.0496 (11)	0.0492 (11)	0.0004 (10)	-0.0050 (10)	-0.0017 (9)
C6	0.0530 (13)	0.0698 (14)	0.0623 (13)	-0.0163 (12)	-0.0024 (11)	-0.0081 (11)
C7	0.0536 (13)	0.0549 (12)	0.0568 (12)	-0.0154 (11)	0.0062 (11)	-0.0041 (10)
C8	0.0406 (10)	0.0383 (9)	0.0495 (10)	0.0017 (8)	0.0049 (9)	-0.0015 (8)
C9	0.0394 (10)	0.0378 (9)	0.0445 (9)	0.0014 (8)	0.0026 (8)	-0.0013 (8)
C10	0.0500 (11)	0.0430 (10)	0.0454 (10)	-0.0014 (9)	0.0001 (9)	-0.0007 (8)
C11	0.0497 (12)	0.0530 (11)	0.0491 (10)	-0.0129 (10)	0.0044 (9)	0.0033 (9)
C12	0.0463 (11)	0.0536 (11)	0.0500 (10)	-0.0088 (10)	-0.0002 (10)	-0.0008 (9)
C13	0.0456 (11)	0.0382 (10)	0.0455 (10)	0.0003 (9)	0.0034 (9)	-0.0024 (8)
C14	0.0454 (11)	0.0385 (9)	0.0473 (10)	-0.0009 (9)	0.0058 (9)	0.0005 (8)
C15	0.0670 (14)	0.0599 (13)	0.0573 (13)	-0.0170 (12)	0.0088 (12)	0.0069 (10)
C16	0.0737 (15)	0.0613 (13)	0.0464 (11)	-0.0033 (13)	0.0053 (12)	0.0078 (10)
C17	0.0565 (12)	0.0463 (11)	0.0445 (10)	0.0069 (10)	0.0050 (10)	-0.0010 (9)
C18	0.0605 (14)	0.0669 (14)	0.0514 (12)	0.0079 (13)	-0.0024 (11)	-0.0064 (11)
C19	0.091 (2)	0.103 (2)	0.0587 (14)	0.0107 (19)	-0.0158 (15)	0.0070 (14)
C20	0.0707 (14)	0.0453 (11)	0.0588 (12)	0.0101 (11)	0.0014 (12)	-0.0061 (9)
C21	0.0818 (17)	0.0492 (12)	0.0641 (13)	0.0098 (13)	-0.0145 (13)	0.0035 (10)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

O1—C3	1.226 (3)	C11—H11A	0.9700
O2—C18	1.221 (3)	C11—H11B	0.9700
C1—C2	1.326 (3)	C12—C13	1.526 (3)

C1—C10	1.496 (3)	C12—H12A	0.9700
C1—H1	0.9300	C12—H12B	0.9700
C2—C3	1.461 (4)	C13—C17	1.524 (3)
C2—H2	0.9300	C13—C14	1.542 (3)
C3—C4	1.444 (4)	C13—C20	1.545 (3)
C4—C5	1.332 (3)	C14—C15	1.535 (3)
C4—H4	0.9300	C14—H14	0.9800
C5—C6	1.500 (3)	C15—C16	1.499 (3)
C5—C10	1.516 (3)	C15—H15A	0.9700
C6—C7	1.520 (3)	C15—H15B	0.9700
C6—H6A	0.9700	C16—C17	1.332 (3)
C6—H6B	0.9700	C16—H16	0.9300
C7—C8	1.527 (3)	C17—C18	1.473 (3)
C7—H7A	0.9700	C18—C19	1.499 (3)
C7—H7B	0.9700	C19—H19A	0.9599
C8—C14	1.515 (3)	C19—H19B	0.9599
C8—C9	1.548 (3)	C19—H19C	0.9599
C8—H8	0.9800	C20—H20A	0.9599
C9—C11	1.535 (3)	C20—H20B	0.9599
C9—C10	1.576 (3)	C20—H20C	0.9599
C9—H9	0.9800	C21—H21A	0.9599
C10—C21	1.553 (3)	C21—H21B	0.9599
C11—C12	1.535 (3)	C21—H21C	0.9599
C2—C1—C10	124.9 (2)	C13—C12—C11	110.24 (17)
C2—C1—H1	117.6	C13—C12—H12A	109.6
C10—C1—H1	117.6	C11—C12—H12A	109.6
C1—C2—C3	121.6 (2)	C13—C12—H12B	109.6
C1—C2—H2	119.2	C11—C12—H12B	109.6
C3—C2—H2	119.2	H12A—C12—H12B	108.1
O1—C3—C4	122.7 (3)	C17—C13—C12	118.94 (18)
O1—C3—C2	121.5 (3)	C17—C13—C14	99.28 (15)
C4—C3—C2	115.8 (2)	C12—C13—C14	107.19 (15)
C5—C4—C3	123.6 (2)	C17—C13—C20	107.12 (15)
C5—C4—H4	118.2	C12—C13—C20	110.57 (16)
C3—C4—H4	118.2	C14—C13—C20	113.46 (17)
C4—C5—C6	121.2 (2)	C8—C14—C15	122.14 (18)
C4—C5—C10	122.5 (2)	C8—C14—C13	113.99 (15)
C6—C5—C10	116.17 (17)	C15—C14—C13	103.89 (15)
C5—C6—C7	110.57 (19)	C8—C14—H14	105.1
C5—C6—H6A	109.5	C15—C14—H14	105.1
C7—C6—H6A	109.5	C13—C14—H14	105.1
C5—C6—H6B	109.5	C16—C15—C14	99.80 (18)
C7—C6—H6B	109.5	C16—C15—H15A	111.8
H6A—C6—H6B	108.1	C14—C15—H15A	111.8
C6—C7—C8	111.90 (17)	C16—C15—H15B	111.8
C6—C7—H7A	109.2	C14—C15—H15B	111.8
C8—C7—H7A	109.2	H15A—C15—H15B	109.5
C6—C7—H7B	109.2	C17—C16—C15	112.64 (18)
C8—C7—H7B	109.2	C17—C16—H16	123.7

## supplementary materials

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H7A—C7—H7B	107.9	C15—C16—H16	123.7
C14—C8—C7	111.90 (16)	C16—C17—C18	126.1 (2)
C14—C8—C9	107.43 (16)	C16—C17—C13	109.56 (19)
C7—C8—C9	110.53 (15)	C18—C17—C13	124.0 (2)
C14—C8—H8	109.0	O2—C18—C17	120.5 (2)
C7—C8—H8	109.0	O2—C18—C19	120.8 (2)
C9—C8—H8	109.0	C17—C18—C19	118.7 (2)
C11—C9—C8	112.10 (15)	C18—C19—H19A	109.5
C11—C9—C10	113.81 (16)	C18—C19—H19B	109.5
C8—C9—C10	112.09 (16)	H19A—C19—H19B	109.5
C11—C9—H9	106.0	C18—C19—H19C	109.5
C8—C9—H9	106.0	H19A—C19—H19C	109.5
C10—C9—H9	106.0	H19B—C19—H19C	109.5
C1—C10—C5	111.39 (17)	C13—C20—H20A	109.5
C1—C10—C21	107.31 (18)	C13—C20—H20B	109.5
C5—C10—C21	109.50 (18)	H20A—C20—H20B	109.5
C1—C10—C9	109.97 (17)	C13—C20—H20C	109.5
C5—C10—C9	107.24 (15)	H20A—C20—H20C	109.5
C21—C10—C9	111.48 (16)	H20B—C20—H20C	109.5
C9—C11—C12	113.82 (16)	C10—C21—H21A	109.5
C9—C11—H11A	108.8	C10—C21—H21B	109.5
C12—C11—H11A	108.8	H21A—C21—H21B	109.5
C9—C11—H11B	108.8	C10—C21—H21C	109.5
C12—C11—H11B	108.8	H21A—C21—H21C	109.5
H11A—C11—H11B	107.7	H21B—C21—H21C	109.5
C10—C1—C2—C3	1.2 (4)	C8—C9—C11—C12	51.8 (2)
C1—C2—C3—O1	174.3 (3)	C10—C9—C11—C12	-179.63 (17)
C1—C2—C3—C4	-4.4 (4)	C9—C11—C12—C13	-54.3 (2)
O1—C3—C4—C5	-174.4 (3)	C11—C12—C13—C17	168.19 (16)
C2—C3—C4—C5	4.3 (4)	C11—C12—C13—C14	56.8 (2)
C3—C4—C5—C6	-178.0 (2)	C11—C12—C13—C20	-67.3 (2)
C3—C4—C5—C10	-1.0 (4)	C7—C8—C14—C15	-52.3 (2)
C4—C5—C6—C7	121.5 (2)	C9—C8—C14—C15	-173.83 (17)
C10—C5—C6—C7	-55.8 (3)	C7—C8—C14—C13	-178.48 (16)
C5—C6—C7—C8	54.0 (3)	C9—C8—C14—C13	60.0 (2)
C6—C7—C8—C14	-175.12 (18)	C17—C13—C14—C8	172.40 (16)
C6—C7—C8—C9	-55.4 (2)	C12—C13—C14—C8	-63.3 (2)
C14—C8—C9—C11	-52.2 (2)	C20—C13—C14—C8	59.1 (2)
C7—C8—C9—C11	-174.52 (17)	C17—C13—C14—C15	37.2 (2)
C14—C8—C9—C10	178.41 (15)	C12—C13—C14—C15	161.49 (17)
C7—C8—C9—C10	56.1 (2)	C20—C13—C14—C15	-76.2 (2)
C2—C1—C10—C5	2.1 (3)	C8—C14—C15—C16	-165.34 (18)
C2—C1—C10—C21	-117.7 (3)	C13—C14—C15—C16	-34.8 (2)
C2—C1—C10—C9	120.9 (2)	C14—C15—C16—C17	19.4 (3)
C4—C5—C10—C1	-2.3 (3)	C15—C16—C17—C18	178.0 (2)
C6—C5—C10—C1	174.97 (18)	C15—C16—C17—C13	4.5 (3)
C4—C5—C10—C21	116.3 (2)	C12—C13—C17—C16	-141.8 (2)
C6—C5—C10—C21	-66.5 (2)	C14—C13—C17—C16	-26.2 (2)
C4—C5—C10—C9	-122.6 (2)	C20—C13—C17—C16	92.0 (2)



C6—C5—C10—C9	54.6 (2)	C12—C13—C17—C18	44.5 (3)
C11—C9—C10—C1	56.5 (2)	C14—C13—C17—C18	160.17 (19)
C8—C9—C10—C1	-174.94 (16)	C20—C13—C17—C18	-81.6 (2)
C11—C9—C10—C5	177.80 (16)	C16—C17—C18—O2	-168.9 (2)
C8—C9—C10—C5	-53.7 (2)	C13—C17—C18—O2	3.7 (3)
C11—C9—C10—C21	-62.4 (2)	C16—C17—C18—C19	11.1 (3)
C8—C9—C10—C21	66.2 (2)	C13—C17—C18—C19	-176.3 (2)

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C12—H12B $\cdots$ O2	0.97	2.60	3.076 (3)	111
C19—H19B $\cdots$ O1 <sup>i</sup>	0.96	2.57	3.356 (4)	139
C21—H21A $\cdots$ O1 <sup>ii</sup>	0.96	2.59	3.537 (3)	169

Symmetry codes: (i)  $-x+5/2, -y+1, z+1/2$ ; (ii)  $x-1/2, -y+3/2, -z+1$ .

Fig. 1

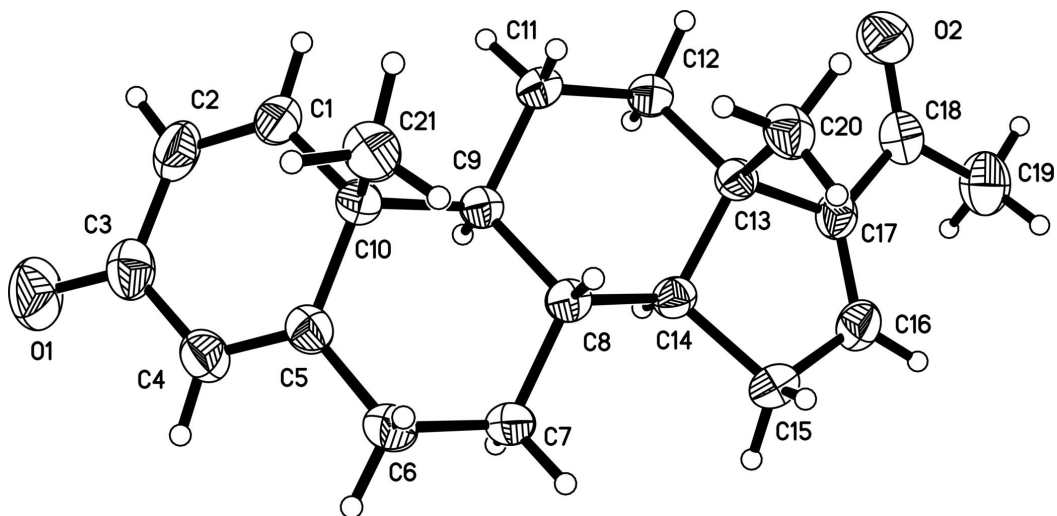


Fig. 2

