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5,6,6a,7,8,8a-Hexahydro-6a,7-dihydroxy-13b*H*-indeno[2,1-*c*]phenanthren-9(13c*H*)-one hemihydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.102
Data-to-parameter ratio = 14.9

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

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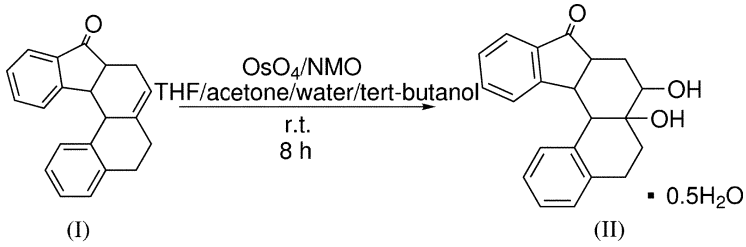
In the title compound, $C_{21}H_{20}O_3\cdot0.5H_2O$, the water molecule lies on a crystallographic twofold rotation axis. The cyclohexane ring adopts a twist–boat conformation and the cyclohexene ring is in an envelope conformation. In the crystal structure, the molecules are linked *via* strong O—H···O intermolecular hydrogen bonds to form chains along the *c* axis.

Comment

Hairpins, knots, pseudoknots, triple helices, loops, helical junctions and bulges constitute the richly diverse structures of nucleic acids (Sanger, 1994). Among these motifs, bulged structures are of general biological significance, and compounds capable of binding to bulges could have significant therapeutic potential (Xi *et al.*, 2002). General-base post-activated neocarzinostatin chromophore (NCSi-gb) is the metabolite of an intramolecular cyclization of neocarzinostatin chromophore in the absence of bulged DNA (Hensens *et al.*, 1994), and it can bind to bulged DNA (Yang *et al.*, 1995). During the preparation of mimics of NCSi-gb, the Diels–Alder ene, (I), can be oxidized to the title diol, (II), with osmium tetroxide/4-methylmorpholine *N*-oxide (NMO) (Xi *et al.*, 1999, 2002). We report here the crystal structure of diol (II).



(I) (II)

X-ray analysis reveals that, in the molecule of (II) (Fig. 1), the two hydroxyl O atoms are almost *cis* [O1—C1—C2—O2 — 38.6 (2)°]. Selected bond lengths and angles are listed in Table 1. The indan-1-one ring system is essentially planar. The cyclohexane ring (C1–C4/C12/C13) adopts a twist–boat conformation and the cyclohexene ring (C1/C13/C14/C19–C21) is in an envelope conformation.

The crystal packing reveals that the molecules of (II) are linked by strong O—H···O intermolecular hydrogen bonds (Table 2) involving the hydroxyl groups and water molecules, to form chains along the *c* axis (Fig. 2).

Experimental

The Diels–Alder ene, (I), was oxidized with osmium tetroxide/4-methylmorpholine *N*-oxide to give the title diol, (II) (Minuti *et al.*,

1995; Xi *et al.*, 1999). Colourless prismatic single crystals of (II) were grown from a saturated solution in tetrahydrofuran.

Crystal data

$C_{21}H_{20}O_3\cdot0.5H_2O$
M_r = 329.38
Monoclinic, $C2/c$
a = 27.942 (9) Å
b = 14.203 (6) Å
c = 8.353 (3) Å
β = 90.291 (6)°
V = 3315 (2) Å^3
Z = 8
D_x = 1.320 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 768 reflections
θ = 2.9–26.0°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colourless
0.30 × 0.15 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
8142 measured reflections
3388 independent reflections
2194 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.4°
$h = -27 \rightarrow 34$
$k = -15 \rightarrow 17$
$l = -10 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.102
S = 1.02
3388 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.6586P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.4345 (17)	O3—C5	1.220 (2)
O2—C2	1.4209 (19)		
O1—C1—C21	109.66 (12)	O3—C5—C6	126.23 (18)
O1—C1—C13	103.87 (12)	C6—C5—C4	108.77 (15)
C21—C1—C13	111.52 (13)	C11—C6—C5	109.78 (15)
O1—C1—C2	109.52 (13)	C7—C6—C5	128.26 (17)
C21—C1—C2	110.39 (13)	C6—C11—C12	111.48 (14)
C13—C1—C2	111.67 (12)	C10—C11—C12	129.73 (15)
O2—C2—C3	112.56 (13)	C11—C12—C4	104.69 (13)
O2—C2—C1	110.99 (13)	C11—C12—C13	117.51 (13)
C3—C2—C1	112.49 (14)	C4—C12—C13	112.72 (13)
C5—C4—C3	109.64 (15)	C14—C13—C1	114.00 (13)
C5—C4—C12	105.22 (14)	C1—C13—C12	113.79 (13)
C3—C4—C12	113.27 (13)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O1^{i}	0.86 (1)	1.87 (1)	2.716 (2)	168 (2)
O2—H2···O4^{ii}	0.82	1.95	2.735 (2)	160
O1—H1···O2^{iii}	0.82	2.03	2.777 (2)	151

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

The unique H atom of the water molecule was located in a difference Fourier map and refined isotropically, with the O—H distance restrained to 0.86 (1) Å and U_{iso}(H) fixed at 0.08 Å^2. The other H atoms were placed in idealized positions and allowed to ride on their parent atoms, with O—H distances of 0.82 Å and C—H distances in the range 0.93–0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(O) and 1.5U_{eq}(C).

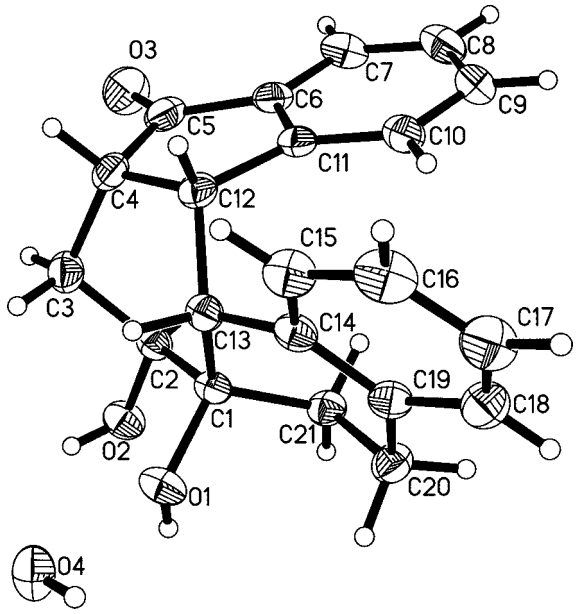


Figure 1
The structure of (II), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

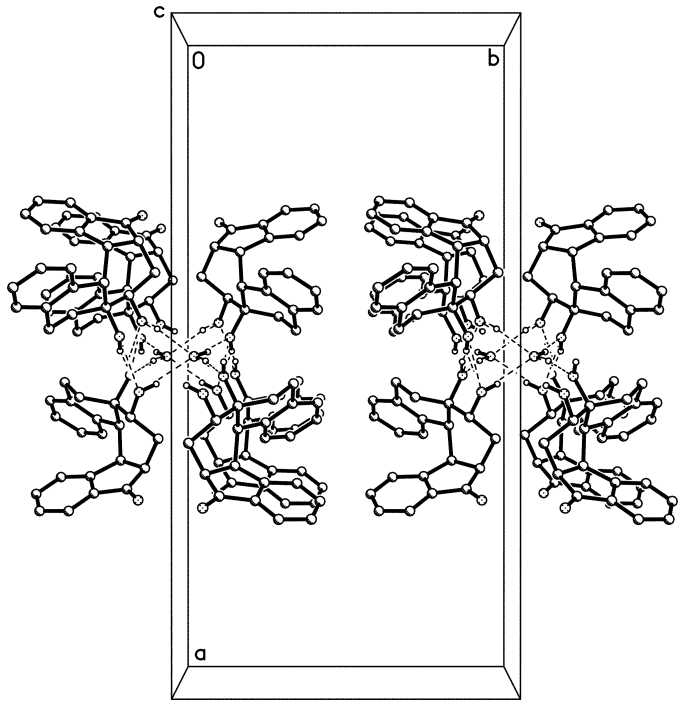


Figure 2
Part of the crystal packing of (II), viewed down the *c* axis. Only H atoms involved in hydrogen bonding (dashed lines) are shown.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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