Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2-Hydroxy-2′-oxo-1,2,3,3a,8,8a,1′,2′,3′,4′-decahydrospiro[cyclopenta[*a*]indene-3,1′-naphthalen]-8-yl acetate

Hong-Tao Mu,* He-Yang Li, Hai-Bin Song and Wen-Bin Chen

State Key Laboratory and Institute of Elemento-Organic Chemistry, Nankai University, Tianjin, Weijin Road No. 94, Tianjin, People's Republic of China

Correspondence e-mail:
muhongtao@mail.china.com

Received 17 June 2005
Accepted 23 June 2005
Online 30 June 2005

The title compound, $C_{23}H_{22}O_4$, contains two crystallographically independent molecules in the asymmetric unit. In both molecules, the indan ring system is almost planar, and the cyclopentane and cyclohexanone rings adopt distorted envelope conformations. In the crystal structure, the molecules exist as O—H···O hydrogen-bonded dimers.

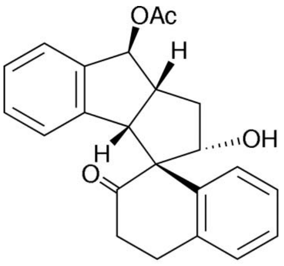
Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.005 Å
R factor = 0.034
wR factor = 0.087
Data-to-parameter ratio = 6.6

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

Comment

The natural enediyne antitumor antibiotic neocarzinostatin (NCS-chrom) can induce highly efficient site-specific strand cleavage at either single- or two-base bulge-containing nucleic acids depending on the particular mode of activation of the drug (Williams & Goldberg, 1988; Kappen & Goldberg, 1993; Xi, Mao & Goldberg, 1999). General-base post-activated neocarzinostatin chromophore (NCSi-gb) (Hensens *et al.*, 1994) can bind to bulged DNA (Yang *et al.*, 1995). During the preparation of biomimics of NCSi-gb, the title compound, (I), an *exo* spiroalcohol, was obtained as a by-product. We report here the crystal structure of (I).



(I)

The molecules of (I) crystallize as an enantiomeric pair in the asymmetric unit of a non-centrosymmetric space group (Fig. 1). The corresponding bond distances and angles in this enantiomeric pair agree with each other (Table 1). In both molecules, the cyclopentane and cyclohexanone rings adopt distorted envelope conformations. The indan ring system is almost planar and the acetate group is twisted away from it by 56.6 (1)° in one of the independent molecules and 56.2 (1)° in the other. In the crystal structure, the independent molecules are linked by O4—H4···O6^{i} and O8—H8···O3^{ii} hydrogen bonds [symmetry codes are given in Table 2] to form a dimer (Fig. 2).

Experimental

Compound (I) was synthesized as a by-product of base-induced spiro-aldolization of the corresponding ketoaldehyde according to the reported procedure of Xi, Jones *et al.* (1999) and Xi *et al.* (2002).

doi:10.1107/S1600536805019835

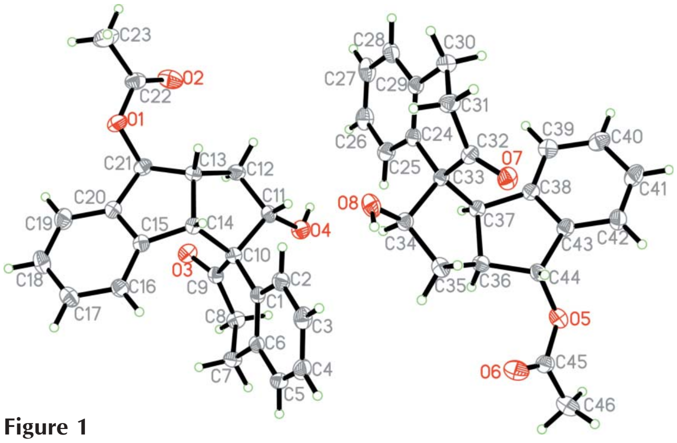


Figure 1

The asymmetric unit of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

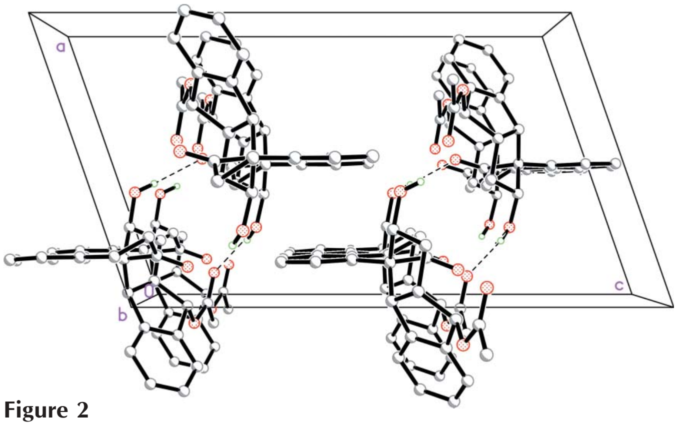


Figure 2

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

Single crystals of (I) suitable for X-ray diffraction were obtained by recrystallization from methanol.

Crystal data

$C_{23}H_{22}O_4$
$M_r = 362.41$
Monoclinic, *Pc*
$a = 10.949$ (4) Å
$b = 9.344$ (3) Å
$c = 18.961$ (5) Å
$\beta = 109.154$ (16)°
$V = 1832.5$ (10) Å^3
$Z = 4$
$D_x = 1.314$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 845 reflections
$\theta = 2.9$–25.9°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.30 × 0.28 × 0.24 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.970$, $T_{\max} = 0.979$
9262 measured reflections
3227 independent reflections
2702 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 13$
$k = -11 \rightarrow 9$
$l = -22 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.087$
$S = 1.06$
3227 reflections
491 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.27P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

O1—C22	1.342 (4)	O5—C45	1.329 (4)
O1—C21	1.462 (4)	O5—C44	1.465 (4)
O2—C22	1.200 (5)	O6—C45	1.210 (5)
O3—C9	1.225 (4)	O7—C32	1.211 (4)
O4—C11	1.419 (4)	O8—C34	1.421 (4)
C9—C10—C1	112.9 (3)	C24—C33—C32	112.8 (3)
C9—C10—C11	105.7 (2)	C24—C33—C37	111.9 (2)
C1—C10—C11	113.2 (2)	C32—C33—C37	112.3 (2)
C9—C10—C14	112.9 (2)	C24—C33—C34	113.1 (2)
C1—C10—C14	111.3 (2)	C32—C33—C34	106.1 (2)
C11—C10—C14	100.1 (2)	C37—C33—C34	99.8 (2)
O4—C11—C12	115.7 (3)	O8—C34—C35	115.7 (3)
O4—C11—C10	111.3 (3)	O8—C34—C33	112.4 (3)
C12—C11—C10	105.3 (3)	C35—C34—C33	105.2 (3)
C12—C13—C21	117.9 (3)	C35—C36—C44	117.8 (3)
C12—C13—C14	106.4 (2)	C35—C36—C37	105.8 (2)
C21—C13—C14	106.1 (2)	C44—C36—C37	106.3 (3)
C15—C14—C13	105.0 (2)	C38—C37—C36	104.8 (2)
C15—C14—C10	117.4 (2)	C38—C37—C33	117.9 (2)
C13—C14—C10	107.2 (2)	C36—C37—C33	107.3 (2)

Table 2

Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O6$^{\rm i}$	0.82	2.09	2.891 (4)	165
O8—H8···O3$^{\rm ii}$	0.82	2.18	2.994 (3)	177

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

The H atoms were placed in idealized positions and allowed to ride on their parent atoms, with O—H = 0.82 Å and C—H = 0.93–0.98 Å, and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O,C$_{\rm methyl}$) and 1.2$U_{\rm eq}$(C). Rotating group refinement was used for the hydroxy and methyl groups. Friedel pairs were merged before the final refinement because of the absence of significant anomalous scattering effects.

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*.

The authors thank the National Natural Science Foundation of China (grant Nos. 20272029 and 20432010), the Natural Science Foundation of Tianjin (grant No. 013616311), the Ministry of Science and Technology (grant No. 2003CB114403) and the Ministry of Education of China for financial support.

References

Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hensens, O. D., Chin, D.-H., Stassinopoulos, A., Zink, D. L., Kappen, L. S. & Goldberg, I. H. (1994). *Proc. Natl Acad. Sci. USA*, **91**, 4534–4538.
Kappen, L. S. & Goldberg, I. H. (1993). *Biochemistry*, **32**, 13138–13145.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Williams, L. D. & Goldberg, I. H. (1988). *Biochemistry*, **27**, 3004–3011.

Xi, Z., Hwang, G.-S., Goldberg, I. H., Harris, J. L., Pennington, W. T., Fouad, F. S., Qabaja, G., Wright, J. M. & Jones, G. B. (2002). *Chem. Biol.* **9**, 925–931.

Xi, Z., Jones, G. B., Qabaja, G., Wright, J., Johnson, F. S. & Goldberg, I. H. (1999). *Org. Lett.* **1**, 1375–1377.

Xi, Z., Mao, Q. K. & Goldberg, I. H. (1999). *Biochemistry*, **38**, 4342–4354.

Yang, C. F., Stassinopoulos, A. & Goldberg, I. H. (1995). *Biochemistry*, **34**, 2267–2275.