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2,2′-Dihydroxy-[2,2′-bi-1*H*-indene]-1,1′,3,3′-(2*H*,2′*H*)-tetrone dihydrate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.116
Data-to-parameter ratio = 14.4

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

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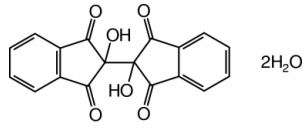
The structure of the title compound, $C_{18}H_{14}O_8$, adopts a symmetric *trans*-configuration of the bisindane rings across the C2—C2′ bond, with this bond lying across an inversion centre. The hydrogen-bonding network consists of associations from the hydroxyl groups to adjacent water O atoms and then from the water H atoms to the keto O atoms.

Comment

Hydrindantin is prepared by the reduction of ninhydrin with ascorbic acid (Moore & Stein, 1954) or alternatively by the action of potassium cyanide (Bruice & Richards, 1958) on ninhydrin. Hydrindantin is, at best, only sparingly soluble in hot water and organic solvents except for ethane-1,2-diol (ethylene glycol), 2-methoxyethanol (methyl cellosolve) and DMSO (Moore, 1968), in which it is very soluble. However, it is soluble, with decomposition, in aqueous Na_2CO_3 and NaOH solutions, in it gives a deep red and a deep blue colour respectively (O'Neil, 2001). The structure of ninhydrin has been twice determined (Medrud, 1969; Fronczek, 1995), but the structure of hydrindantin has not, although The Merck Index (O'Neil, 2001) lists hydrindantin as forming prisms from acetone. Three reported structures (Allen, 2002) that are similar to hydrindantin have been reported (Aliev *et al.*, 1990; Benati *et al.*, 1995; Dopp *et al.*, 2002), and each displays a similar *cis*-configuration of the bisindane rings with respect to the C2—C2′ bond. In contrast, the structure of the title compound, (I), adopts a symmetric *trans*-configuration with the C2—C2′ bond lying across an inversion centre (Fig. 1).

A two molar equivalence of water is produced in the reduction of ninhydrin to hydrindantin. These molecules are an integral part of the packing of hydrindantin and can be removed by heating under vacuum following the preparation of hydrindantin. However, exposure to ambient conditions results in a rehydration of the hydrindantin solid. Therefore, crystallization in organic solvents under ambient conditions will result in the presence of water in the crystal lattice.



(I)

Experimental

Crystals of the title compound were grown from a 50:50 ethyl acetate: chloroform solution.

Crystal data

$C_{18}H_{10}O_6\cdot 2H_2O$
$M_r = 358.29$
Monoclinic, $P2_1/c$
$a = 8.4479\ (4)$ Å
$b = 12.4759\ (6)$ Å
$c = 7.9293\ (4)$ Å
$\beta = 100.634\ (3)^\circ$
$V = 821.36\ (7)$ Å^3
$Z = 2$
$D_x = 1.449$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4659 reflections
$\theta = 2.9$–27.5°
$\mu = 0.12$ mm^{-1}
$T = 120\ (2)$ K
Prism, colourless
$0.14 \times 0.08 \times 0.03$ mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
$T_{min} = 0.984$, $T_{max} = 0.997$
10673 measured reflections
1884 independent reflections
1197 reflections with $I > 2\sigma(I)$
$R_{int} = 0.090$
$\theta_{max} = 27.5^\circ$
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.116$
$S = 1.00$
1884 reflections
131 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.0592P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.038 (5)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1W^{i}	0.96 (3)	1.68 (3)	2.607 (2)	161 (3)
O1W—H1W···O1^{ii}	0.88 (4)	1.95 (4)	2.795 (2)	162 (3)
O1W—H2W···O3^{iii}	0.88 (4)	1.93 (4)	2.798 (2)	174 (3)

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

All H atoms, except for those involved in hydrogen-bonding interactions, were included in the refinement, at calculated positions, as riding models with C—H set to 0.95 Å while the isotropic displacement parameters were set equal to 1.25 times U_{eq} of the carrier atom. The hydroxy and water H atoms were located in difference syntheses and both positional and displacement parameters were refined.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

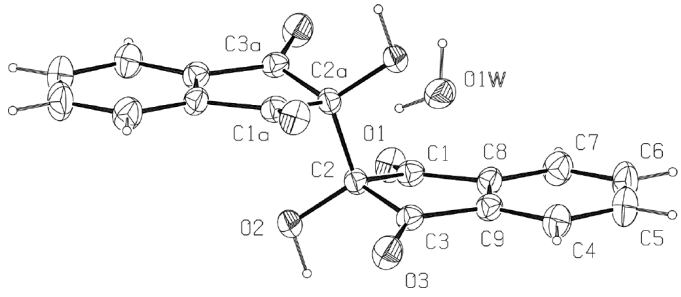


Figure 1
Molecular configuration and atom numbering scheme for the title compound, showing 50% probability ellipsoids [symmetry code: (a) = $1-x, -y, -z$].

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