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(2*SR*,3*RS*,1′*SR*,2′*SR*,3′*RS*)-2-(2′,3′-Dihydro-1′-hydroxy-3′-phenyl-1*H*-inden-2′-yl)-2,3-dihydro-3-phenylinden-1-one: isomer (II)

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Isomer (II) of the title compound, $C_{30}H_{24}O_2$, has been obtained as pale-orange needle-shaped crystals. The dihydrobiindene skeleton is approximately planar, and there are two phenyl substituents on different sides of the skeleton plane. There is an intramolecular O—H···O hydrogen bond.

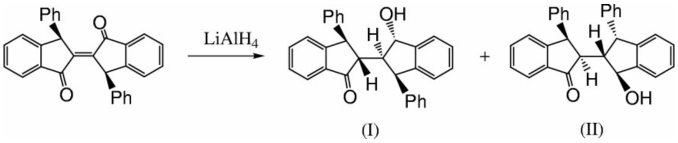
Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.006 Å
R factor = 0.057
wR factor = 0.170
Data-to-parameter ratio = 17.5

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

Comment

In a series of studies of the crystal photochromism of biindenylidene compounds (Tanaka *et al.*, 2003; Ohba *et al.*, 2003), two geometrical isomers, (I) and (II), were obtained as a mixture by the reaction shown in the scheme. They were separated as yellow plate-like crystals of (I) and pale-orange needles of (II). X-ray structure analyses have been carried out to determine the geometrical structures of these compounds. The structure of (II) is reported in this paper, and that of (I) is reported in the preceding paper (Ohba *et al.*, 2004).



The major difference between isomers (I) and (II) is the relative configuration at atom C13. The arrangement of the Ph group bonded to atom C14 and the H atom bonded to atom C13 is *trans* for (II) (Fig. 1). The dihydrobiindene skeleton is approximately planar, the dihedral angle between the C6–C11 and C15–C20 rings being 14.0 (1)°. The five-membered rings are each in an envelope form, with atom C4 displaced from the C3/C7/C6/C5 plane by 0.352 (6) Å in the C4—H4 direction and atom C13 shifted from the C12/C16/C15/C14 plane by 0.326 (6) Å in the C13—H13 direction, where atoms H4 and

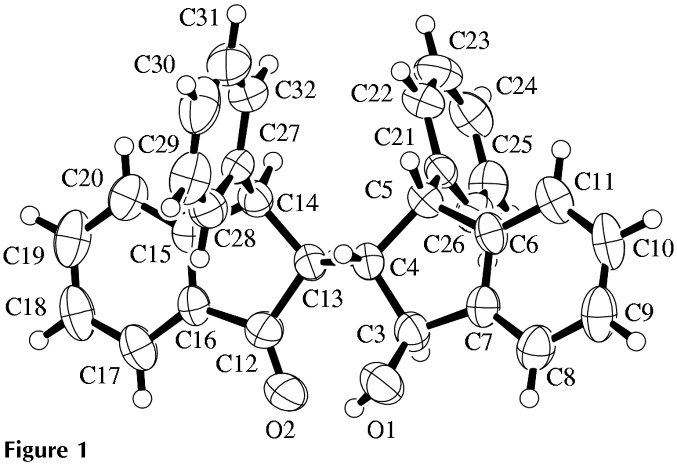


Figure 1
The molecular structure of (II), showing 50% probability displacement ellipsoids.

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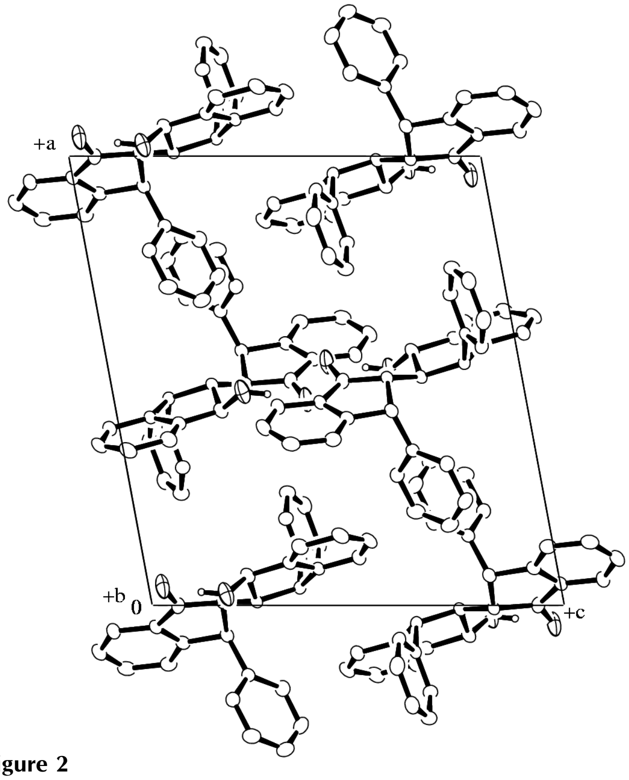

Figure 2
The crystal structure of (II) projected along the *b* axis. H atoms have been omitted, except for that of the hydroxy group.

H13 are in *trans* positions. As a result, hydroxy atom O1 and keto atom O2 are on the same side of the central C4—C13 bond in (II), forming an intramolecular O—H···O hydrogen bond (Table 2).

The space group is $P2_1/n$, with $Z = 4$ for both (I) and (II). The cell volume of (II), 2216.7 (13) Å^3, is slightly smaller than that of (I), 2222.6 (10) Å^3, indicating that the packing efficiency is higher for molecules similar to (II), having the two phenyl substituents on different sides of the biindenyl skeleton plane. A similar difference in the cell volume was observed between the *meso* and racemic isomers of the bis(3,5-dimethylphenyl)biindenylidene derivative (Ohba *et al.*, 2003).

Experimental

Reduction of rac-(*E*)-2,3-dihydro-2-[2′,3′-dihydro-1′-oxo-3′-phenyl-1*H*-inden-2′-ylidene]-3-phenylinden-1-one (1.5 g, 3.6 mmol) with $LiAlH_4$ (0.13 g, 3.6 mmol) in dry tetrahydrofuran (60 ml) at room temperature for 10 min gave a mixture of (I) (0.24 g) and (II) (0.67 g) in 16 and 44% yields, respectively. Pale-orange crystals of (II) were grown from a methanol solution (m.p. 484–486 K).

Crystal data

$C_{30}H_{24}O_2$
$M_r = 416.52$
Monoclinic, $P2_1/n$
$a = 14.883 (4)$ Å
$b = 11.290 (5)$ Å
$c = 13.418 (4)$ Å
$\beta = 100.52 (2)^\circ$
$V = 2216.7 (13)$ Å^3
$Z = 4$
$D_x = 1.248$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.3$–13.6°
$\mu = 0.08$ mm^{-1}
$T = 300$ K
Block cut from a needle, pale orange
0.45 × 0.25 × 0.10 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.982$, $T_{\max} = 0.993$
5794 measured reflections
5088 independent reflections
1507 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.027$
$\theta_{\max} = 27.5^\circ$
$h = -7 \rightarrow 19$
$k = 0 \rightarrow 14$
$l = -17 \rightarrow 17$
3 standard reflections
every 150 reflections
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.170$
$S = 0.92$
5088 reflections
290 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.361 (5)	O2—C12	1.219 (6)
C3—C4—C5—C21	103.0 (3)	C5—C4—C13—C12	165.9 (3)
C3—C4—C13—C12	40.5 (4)	C5—C4—C13—C14	−67.3 (4)
C3—C4—C13—C14	167.3 (3)	C12—C13—C14—C27	102.2 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	2.19	2.841 (4)	136

The hydroxy H atom was located in a difference synthesis and allowed to ride on the O atom, with O—H = 0.82 Å and $U_{\rm iso}({\rm H}) = U_{\rm eq}({\rm O})$. The other H atoms were positioned geometrically, with C—H set equal to 0.95 Å, and fixed with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent atom). The positions of the H atoms were recalculated after each set of cycles of refinement, except for the last.

Data collection: *WinAFC Diffractometer Control Software* (Rigaku, 1999); cell refinement: *WinAFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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