Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(2*RS*,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 (I)

Shigeru Ohba,[a]* Yohei Yamamoto[b] and Koichi Tanaka[b]‡

[a]Department of Chemistry, Keio University, Hiyoshi 4-1-1, Kohoku-ku, Yokohama 223-8521, Japan, and [b]Department of Applied Chemistry, Faculty of Engineering, Ehime University, Matsuyama, Ehime 790-8577, Japan

‡ Present address: Department of Applied Chemistry, Faculty of Engineering, Kansai University, Suita, Osaka 564-8680, Japan.

Correspondence e-mail: ohba@flet.keio.ac.jp

Received 6 September 2004
Accepted 9 September 2004
Online 18 September 2004

Isomer (I) of the title compound, $C_{30}H_{24}O_2$, has been obtained as yellow plate-like crystals. The dihydrobiindene skeleton is approximately planar, and there are two phenyl substituents on the same side of the skeleton plane. The molecules are connected *via* an O—H···O hydrogen bond, forming spiral chains along the *b* axis.

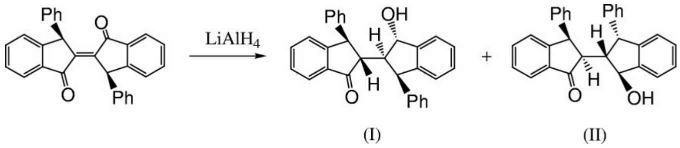
Key indicators

Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.004 Å
R factor = 0.054
wR factor = 0.167
Data-to-parameter ratio = 17.6

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 isomers. The structure of (I) is reported in this paper, and that of (II) is reported in the following 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 *cis* for (I) (Fig. 1). The dihydrobiindene skeleton is approximately planar, the dihedral angle between the C6–C11

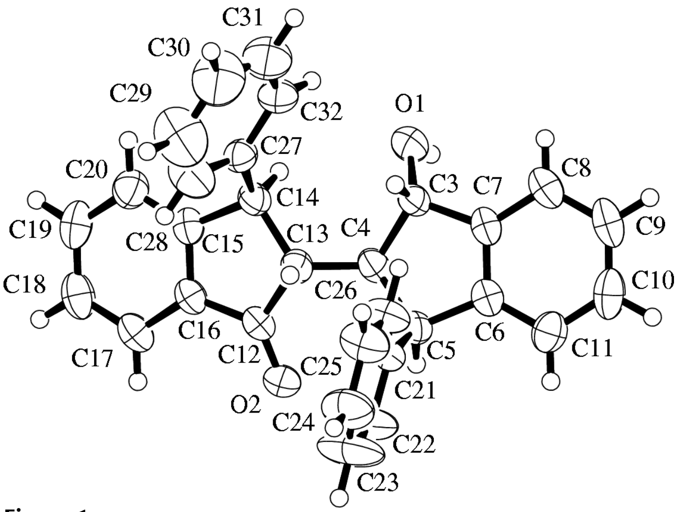


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

DOI: 10.1107/S1600536804022457

and C15–C20 rings being 17.5 (1)°. The five-membered rings are each in an envelope form, with atom C4 displaced from the C3/C7/C6/C5 plane by 0.377 (4) Å in the C4—H4 direction and atom C13 atom shifted from the C12/C16/C15/C14 plane by 0.187 (4) Å in the C13—H13 direction, where atoms H4 and H13 are in *trans* positions. As a result, hydroxy atom O1 and keto atom O2 are on different sides of the central C4—C13 bond in (I). In the crystal structure, molecules are connected *via* an O—H···O hydrogen bond (Table 2), forming spiral chains around 2_1 axes parallel to *b* (Fig. 2).

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 for10 min gave a mixture of (I) (0.24 g) and (II) (0.67 g) in 16 and 44% yields, respectively. Yellow plate-like crystals of (I) were grown from a methanol solution (m.p. 493–495 K).

Crystal data

$C_{30}H_{24}O_2$
$M_r = 416.52$
Monoclinic, $P2_1/n$
$a = 17.985$ (3) Å
$b = 9.529$ (3) Å
$c = 13.154$ (4) Å
$\beta = 99.63$ (2)°
$V = 2222.6$ (10) Å^3
$Z = 4$

$D_x = 1.245$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10.1$–12.8°
$\mu = 0.08$ mm^{-1}
$T = 297$ K
Plate, yellow
0.5 × 0.3 × 0.1 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: by integration (Coppens *et al.*, 1965)
$T_{\min} = 0.974$, $T_{\max} = 0.993$
6053 measured reflections
5110 independent reflections
2464 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.035$
$\theta_{\max} = 27.5°$
$h = -8 \rightarrow 23$
$k = -12 \rightarrow 4$
$l = -17 \rightarrow 17$
3 standard reflections
every 150 reflections
intensity decay: 0.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.167$
$S = 1.01$
5110 reflections
290 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0731P)^2 + 0.206P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.401 (3)	O2—C12	1.222 (3)
C3—C4—C5—C21	96.8 (2)	C5—C4—C13—C12	−65.6 (3)
C3—C4—C13—C12	165.8 (2)	C5—C4—C13—C14	168.4 (2)
C3—C4—C13—C14	39.7 (3)	C12—C13—C14—C27	132.7 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2$^{\rm i}$	0.82	2.16	2.969 (3)	168

Symmetry code: (i) $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

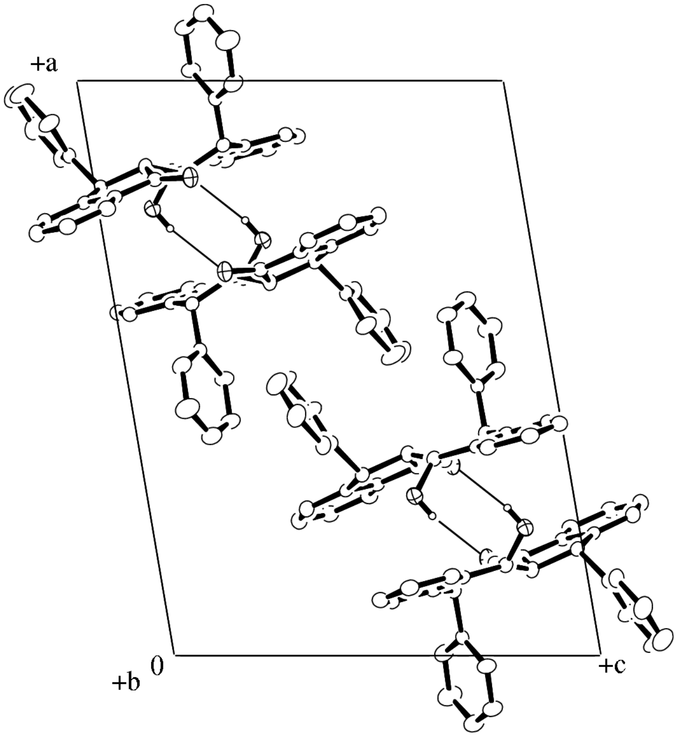


Figure 2
The crystal structure of (I), projected along the *b* axis. H atoms have been omitted, except for that of the hydroxy group. The thin lines indicate hydrogen bonds.

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 (C—H = 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. The $U_{\rm eq}$ values of atoms C22, C23, C24, C28, C29, C30 and C31 are relatively large, 0.07–0.11 Å^2, suggesting some librational disorder of the phenyl groups.

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*.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (2001). *TEXSAN*. Version 1.11. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Ohba, S., Yamamoto, Y. & Tanaka, K. (2003). *Acta Cryst.* C**59**, o370–o372.

Ohba, S., Yamamoto, Y. & Tanaka, K. (2004). *Acta Cryst.* E**60**, o1795–o1796.

Rigaku (1999). *WinAFC Diffractometer Control Software*. Rigaku Corporation, Tokyo, Japan.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Tanaka, K., Yamamoto, Y. & Ohba, S. (2003). *Chem. Commun.* pp. 1866–1867.