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2-(1-Phenylsulfonyl-1*H*-indol-3-yl)-1,2-dihydronaphthalen-1-ol

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.058
wR factor = 0.155
Data-to-parameter ratio = 16.3

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

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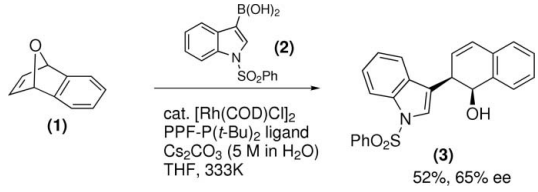
In the title molecule, $C_{24}H_{19}NO_3S$, the dihedral angles formed by the essentially planar indole group with the benzene ring of the naphthalen-1-ol group and the phenyl ring of the benzenesulfonyl group are 77.64 (8) and 70.56 (7)°, respectively, giving rise to a U-shaped molecule. In the crystal structure, molecules are linked to form centrosymmetric dimers *via* weak C—H···O hydrogen bonds (H···O = 2.43 Å). These dimers are, in turn, linked into extended tapes *via* intermolecular O—H···O hydrogen bonds [H···O = 2.00 (3) Å].

Comment

The addition of carbon-based nucleophiles to activated alkenes represents one of the fundamental methods for the controlled construction of C—C bonds in organic synthesis. The use of Rh^I catalysts for the highly enantioselective addition of organoborons and other organometals to a variety of activated alkenes has become an important synthetic method in recent years (for a review, see Hayashi & Yamasaki, 2003). We have reported that heterobicyclic alkenes are effective substrates for metal-catalysed ring-opening reactions with a variety of nucleophiles (for a review, see Lautens *et al.*, 2003). More specifically, we have discovered that boronic acids can add to oxabicyclic alkenes in an enantioselective fashion to give a variety of substituted carbocycles, using Rh^I catalysts (Lautens *et al.*, 2002). Here, we report the crystal structure of alcohol (3) derived from the Rh^I-catalysed ring-opening of oxabicycle (1) with boronic acid (2). The crystals obtained from the reaction contain both enantiomers of (3), although the reaction was enantioselective with 65% ee prior to the crystallization.



A view of the molecular structure of (3) is shown in Fig. 1 and selected bond lengths and angles are given in Table 1. The bonding geometry around atom N1 is slightly pyramidal, as reflected in the sum of the angles (348.2°). The conformational analysis of the cyclohexene ring (C1/C2/C7–C10) (Duax *et al.*, 1976) shows that the conformation is a half-chair, with a local pseudo-twofold axis running through the midpoint of the C1—C10 bond. In the crystal structure, molecules are linked *via* weak C—H···O hydrogen bonds (Table 2), forming $R_2^2(22)$ rings (Bernstein *et al.*, 1995). These rings are, in turn,

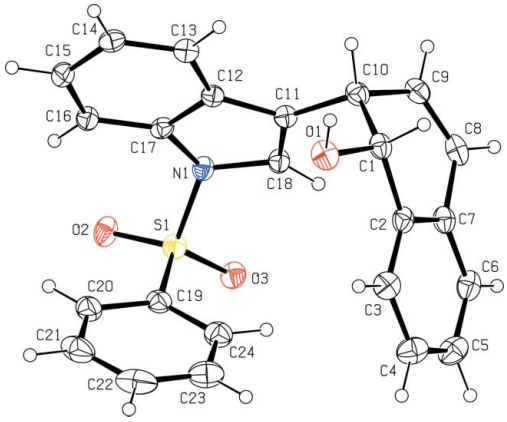


Figure 1
A view of the molecular structure of (3), showing 30% probability displacement ellipsoids (arbitrary spheres for the H atoms).

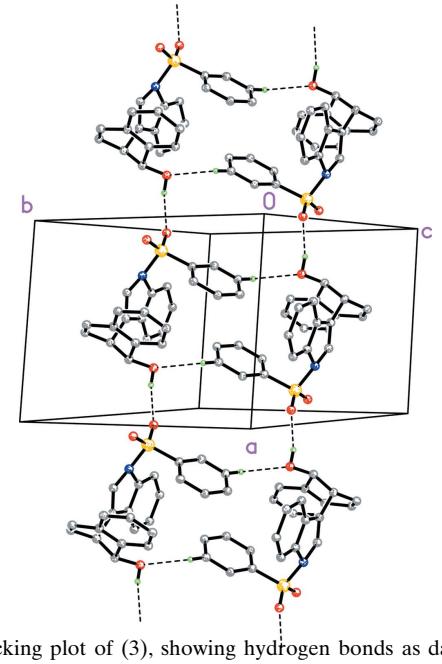


Figure 2
A partial packing plot of (3), showing hydrogen bonds as dashed lines. Colour codes: yellow S, red O, blue N and green H. H atoms not involved in hydrogen bonds have been omitted.

connected by intermolecular O—H···O hydrogen bonds, with graph-set $C(9)$, forming molecular tapes propagating in the *a*-axis direction (Fig. 2). A search of the Cambridge Structural Database (CSD; Version 5.27, with updates to January 2006; Allen, 2002) revealed only five structures containing the 2-substituted-1,2-dihydronaphthalen-1-ol group [CSD refcodes DAHHEA, HUMSIR, IVOXUM, MIYGOQ and PUYXAI (Fagnou *et al.*, 2002*a*; Fagnou *et al.*, 2002*b*; Leong & Lautens, 2004; Li *et al.*, 2004; Lough *et al.*, 2002)].

Experimental

A 5 ml flask with stirrer bar was charged with $[Rh(COD)Cl]_2$ (COD = 1,5-cyclooctadiene) (4.3 mg, 0.0087 mmol) and (*S*)-(*R*)-PPF-P(*t*-Bu)$_2$ {(*S*)-1-[(*R*)-2-(diphenylphosphino)ferrocenyl]ethyldi-*tert*-butylphosphine} ligand (10.3 mg, 0.0190 mmol). The flask was sealed and flushed with N_2 before distilled tetrahydrofuran (THF, 0.5 ml) was added, followed by Cs_2CO_3 (5 *M*) in H_2O (35 µl, 0.175 mmol Cs_2CO_3), and a solution of oxabenzonorbornadiene (1) (50 mg, 0.347 mmol) and 1-(phenylsulfonyl)-1*H*-indol-3-yl boronic acid (2) (purchased from Maybridge Chemical Co., 125 mg, 0.415 mmol) in THF (total 1.8 ml used to dissolve and transfer) *via* a syringe from a vial under N_2. The reaction was stirred for 22 h at room temperature, but thin-layer chromatography analysis (25% EtOAc/hexanes) indicated that the reaction was incomplete. The reaction was then heated at 333 K for 3 h until it was judged to be complete. The crude reaction was evaporated, redissolved in 5% EtOAc/hexanes (with several drops of dichloromethane) and transferred on to a glass column packed with silica gel (1/2" × 3") and eluted with 5–50% EtOAc/hexanes [yield: 73 mg, 52% (unoptimized), ee = 65%]. Product (3) was isolated as a pale-yellow solid after a second purification by flash column to obtain analytically pure material. X-ray quality crystals were obtained from a 15% EtOAc/hexanes solution of the purified compound left standing in a test tube. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (1H, *d*, *J* = 8.1 Hz), 7.66 (2H, *dm*, *J* = 8.8 Hz), 7.53 (1H, *dm*, *J* = 7.7 Hz), 7.47–7.13 (12H, *m*), 6.67 (1H, *dd*, *J* = 9.5, 1.8 Hz), 6.02 (1H, *dd*, *J* = 9.5, 4.0 Hz), 4.92 (1H, *d*, 5.1 Hz), 4.05 (1H, *m*), 1.42 (1H, *br s*)

Crystal data

$C_{24}H_{19}NO_3S$
M_r = 401.46
Monoclinic, $P2_1/n$
a = 9.4904 (4) Å
b = 12.2689 (5) Å
c = 16.5644 (10) Å
β = 97.4327 (15)°
V = 1912.39 (16) Å^3
Z = 4
D_x = 1.394 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 12837 reflections
θ = 2.6–27.5°
μ = 0.20 mm^{-1}
T = 150 (1) K
Block, colourless
0.08 × 0.08 × 0.06 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.920, T_{max} = 0.988
12837 measured reflections
4360 independent reflections
2563 reflections with $I > 2\sigma(I)$
R_{int} = 0.083
θ_{max} = 27.5°
h = −12 → 12
k = −15 → 15
l = −18 → 21

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.155
S = 1.01
4360 reflections
267 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0080 (16)

Table 1
Selected geometric parameters (Å, °).

S1—N1	1.648 (2)	C1—C10	1.542 (4)
O1—C1	1.413 (3)	C2—C7	1.414 (4)
N1—C17	1.429 (3)	C7—C8	1.474 (4)
N1—C18	1.438 (4)	C8—C9	1.328 (4)
C1—C2	1.518 (4)	C9—C10	1.511 (4)
C17—N1—C18	107.1 (2)	O1—C1—C2	110.3 (2)
C17—N1—S1	122.17 (19)	O1—C1—C10	112.2 (2)
C18—N1—S1	118.92 (19)	C2—C1—C10	111.0 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O···O2^{i}	0.93 (3)	2.00 (3)	2.901 (3)	162 (3)
C21—H21A···O1^{ii}	0.95	2.43	3.274 (4)	147

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+1$.

H atoms bonded to C atoms were placed in calculated positions with C—H distances of 0.95 (for Csp^2 atoms) and 1.00 Å (for Csp^3 atoms). They were included in the refinement in the riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C). The hydroxyl H atom was refined independently with an isotropic displacement parameter.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2001); molecular graphics: *SHELXTL* and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL*.

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