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The sydnone compound 4-hydroxy-4-benzyl-sydno[3,4-*a*]indole

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The title compound (systematic name: 9-benzyl-1,9-dihydroxy-9*H*-indolo[1,2-*c*][1,2,3]oxadiazole), $C_{16}H_{12}N_2O_3$, was synthesized as part of a series of sydnones to determine substituent effects on the sydnone ring. The are two independent molecules in the asymmetric unit. There is interest in finding substituents for the sydnone ring that would allow for the release of NO *via* ring opening. Observed bond distances and angles for the sydnone ring are consistent with previously published structures, suggesting that the substituents used have little effect towards destabilization of the ring.

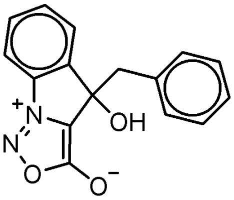
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.146
Data-to-parameter ratio = 15.8

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

Comment

The title compound, (I), has two molecules in the asymmetric unit and is composed of four rings; the sydnone ring, two phenyl rings and another five-membered ring formed by a carbon bridge between the sydnone and attached phenyl ring (C6–C11). No significant difference in bond lengths is observed between molecule *A*, which, for the purpose of this determination, has an *R* configuration at C12*a*, and molecule *B*, which has an *S* configuration at C12*b*. The bond distances (Table 1) are statistically within the average found for the 24 structures determined in-house or cited in the Cambridge Structural Database (Allen, 2002). The bond angles within molecules *A* and *B* are similar to each other, however, when compared to other data, both molecules have slightly smaller N3—N2—O1 angles and slightly larger N2—N3—C4 angles. The average N3—N2—O1 angle of published data is 103.8 (2)°, while molecules *A* and *B* have angles of 102.05 (10) and 101.68 (12)°, respectively. The average N2—N3—C4 angle of published data is 115.7 (2)°, while molecules *A* and *B* have angles of 117.14 (11) and 117.92 (12)°, respectively.



(I)

In (I), molecule *A* has a twist of 4.64 (7)° between the plane of the sydnone ring and the plane of the fused phenyl ring (C6a–C11*a*). The central ring (N3*a*/C4*a*/C6*a*/C7*a*/C12*a*) of the fused ring system is slightly offset from the sydnone ring by 1.49 (8)°. The isolated phenyl ring (C14*a*–C19*a*) is noticeably bent at 51.34 (6)° from the fused ring system. Molecule *B* has a twist of 2.37 (12)° between the plane of the sydnone ring and

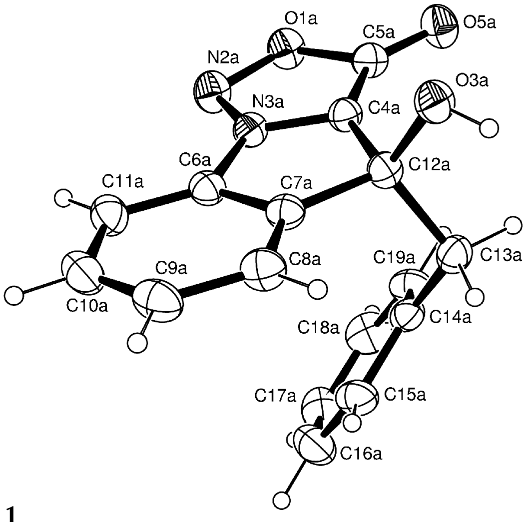


Figure 1
Molecule *A* of (I), shown with 30% probability displacement ellipsoids. H atoms are represented by small spheres.

the fused phenyl ring (C6*b*–C11*b*). The fused ring (N3*b*/C4*b*/C6*b*/C7*b*/C12*b*) is offset from the sydnone ring by 2.07 (8)°. The isolated phenyl ring (C14*b*–C19*b*) is again bent at 57.79 (5)° from the plane of the fused ring system. Each independent molecule forms a centrosymmetric O—H···O hydrogen-bonded pair. Details of the hydrogen-bonding geometries are given in Table 2.

Experimental

The title compound was made by treating 3-(2-bromophenyl)sydnone with *n*-butyllithium and reacting with ethyl phenylacetate, readily forming stable crystals (Krein, 1996).

Crystal data

$C_{16}H_{12}N_2O_3$
$M_r = 280.28$
Triclinic, $P\bar{1}$
$a = 9.502 (3)$ Å
$b = 11.906 (3)$ Å
$c = 13.676 (3)$ Å
$\alpha = 105.712 (17)°$
$\beta = 108.29 (2)°$
$\gamma = 101.13 (2)°$
$V = 1347.1 (7)$ Å^3

$Z = 4$
$D_x = 1.382$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6477 reflections
$\theta = 2.3$–$28.1°$
$\mu = 0.10$ mm^{-1}
$T = 298 (2)$ K
Rectangular block, colorless
0.40 × 0.30 × 0.25 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 1997–1999)
$T_{min} = 0.901$, $T_{max} = 0.976$
11 422 measured reflections

6005 independent reflections
4886 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 28.2°$
$h = -12 \rightarrow 12$
$k = -15 \rightarrow 15$
$l = -17 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.146$
$S = 1.06$
6005 reflections
381 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

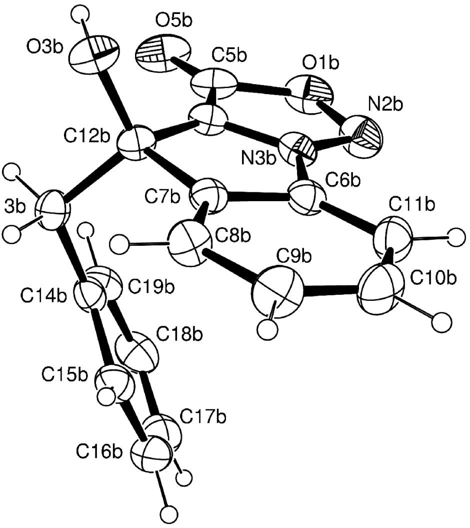


Figure 2
Molecule *B* of (I), shown with 30% probability displacement ellipsoids. H atoms are represented by small spheres.

Table 1
Selected geometric parameters (Å, °).

O1*a*—N2*a*	1.3835 (16)	O1*b*—N2*b*	1.3826 (18)
O1*a*—C5*a*	1.4098 (16)	O1*b*—C5*b*	1.425 (2)
O3*a*—C12*a*	1.4124 (16)	O3*b*—C12*b*	1.4224 (16)
N3*a*—N2*a*	1.3000 (16)	N3*b*—N2*b*	1.2994 (16)
N3*a*—C4*a*	1.3435 (17)	N3*b*—C4*b*	1.3313 (18)
N3*a*—C6*a*	1.4305 (17)	N3*b*—C6*b*	1.4282 (17)
O5*a*—C5*a*	1.2214 (16)	O5*b*—C5*b*	1.2179 (18)
C4*a*—C5*a*	1.3962 (18)	C4*b*—C5*b*	1.3995 (18)
C4*a*—C12*a*	1.5147 (18)	C4*b*—C12*b*	1.5092 (19)
C7*a*—C8*a*	1.3813 (18)	C7*b*—C8*b*	1.3767 (19)
C7*a*—C6*a*	1.3873 (19)	C7*b*—C6*b*	1.3821 (19)
C7*a*—C12*a*	1.5284 (17)	C7*b*—C12*b*	1.5290 (17)
C13*a*—C12*a*	1.5500 (18)	C13*b*—C12*b*	1.5402 (19)
C6*a*—C11*a*	1.3712 (19)	C6*b*—C11*b*	1.372 (2)
C8*a*—C9*a*	1.389 (2)	C8*b*—C9*b*	1.385 (2)
C11*a*—C10*a*	1.385 (2)	C11*b*—C10*b*	1.379 (2)
C9*a*—C10*a*	1.380 (2)	C9*b*—C10*b*	1.385 (3)
N2*a*—O1*a*—C5*a*	111.76 (9)	N2*b*—O1*b*—C5*b*	111.80 (10)
N2*a*—N3*a*—C4*a*	117.14 (11)	N2*b*—N3*b*—C4*b*	117.92 (12)
N2*a*—N3*a*—C6*a*	130.36 (11)	N2*b*—N3*b*—C6*b*	129.62 (12)
C4*a*—N3*a*—C6*a*	112.47 (11)	C4*b*—N3*b*—C6*b*	112.45 (11)
N3*a*—C4*a*—C5*a*	105.29 (11)	N3*b*—C4*b*—C5*b*	105.60 (13)
N3*a*—C4*a*—C12*a*	110.17 (11)	N3*b*—C4*b*—C12*b*	110.63 (11)
C5*a*—C4*a*—C12*a*	144.44 (12)	C5*b*—C4*b*—C12*b*	143.64 (13)
O5*a*—C5*a*—C4*a*	137.25 (12)	O5*b*—C5*b*—C4*b*	137.41 (16)
O5*a*—C5*a*—O1*a*	119.01 (11)	O5*b*—C5*b*—O1*b*	119.61 (13)
C4*a*—C5*a*—O1*a*	103.75 (11)	C4*b*—C5*b*—O1*b*	102.95 (12)
C8*a*—C7*a*—C6*a*	118.16 (13)	C8*b*—C7*b*—C6*b*	118.48 (12)
C8*a*—C7*a*—C12*a*	129.99 (12)	C8*b*—C7*b*—C12*b*	129.84 (12)
C6*a*—C7*a*—C12*a*	111.84 (11)	C6*b*—C7*b*—C12*b*	111.67 (11)
N3*a*—N2*a*—O1*a*	102.05 (10)	N3*b*—N2*b*—O1*b*	101.68 (12)
O3*a*—C12*a*—C4*a*	110.28 (10)	O3*b*—C12*b*—C4*b*	114.01 (11)
O3*a*—C12*a*—C7*a*	111.74 (10)	O3*b*—C12*b*—C7*b*	107.73 (10)
C4*a*—C12*a*—C7*a*	99.50 (10)	C4*b*—C12*b*—C7*b*	99.37 (11)
O3*a*—C12*a*—C13*a*	111.97 (10)	O3*b*—C12*b*—C13*b*	109.53 (11)
C4*a*—C12*a*—C13*a*	111.31 (10)	C4*b*—C12*b*—C13*b*	112.89 (10)
C7*a*—C12*a*—C13*a*	111.42 (10)	C7*b*—C12*b*—C13*b*	112.93 (11)
C11*a*—C6*a*—C7*a*	125.12 (13)	C11*b*—C6*b*—C7*b*	124.48 (13)
C11*a*—C6*a*—N3*a*	129.06 (13)	C11*b*—C6*b*—N3*b*	129.66 (13)
C7*a*—C6*a*—N3*a*	105.75 (11)	C7*b*—C6*b*—N3*b*	105.86 (11)
C7*a*—C8*a*—C9*a*	118.24 (15)	C7*b*—C8*b*—C9*b*	118.45 (14)
C6*a*—C11*a*—C10*a*	115.44 (15)	C6*b*—C11*b*—C10*b*	116.18 (15)
C10*a*—C9*a*—C8*a*	121.63 (14)	C10*b*—C9*b*—C8*b*	121.51 (14)
C9*a*—C10*a*—C11*a*	121.40 (14)	C11*b*—C10*b*—C9*b*	120.89 (14)

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

D—H$\cdots a$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3a—H3$a\cdots$O5$a^{\rm i}$	0.82	1.91	2.7240 (18)	169
O3b—H3$b\cdots$O5$b^{\rm ii}$	0.82	1.90	2.7091 (19)	169

Symmetry codes: (i) $1-x, 2-y, 1-z$; (ii) $2-x, 2-y, -z$.

All H atoms were placed in calculated positions, with O—H = 0.82 Å, C—$H_{\rm methylene}$ = 0.97 Å and C—H = 0.93 Å for all others. They were included in the refinement in the riding-model approximation, with $U_{\rm iso} = 1.2U_{\rm eq}$ [$1.5U_{\rm eq}$(O)] of the carrier atom. The hydroxyl H-atom position was calculated by generating a difference electron density map in a circle at the appropriate O—H distance and C—O—H angle. The point of maximum electron density was taken as the starting point for the H-atom position. The position was re-idealized at the beginning of each interation of least-squares refinement.

Data collection: *SMART* (Bruker, 1997–2000); cell refinement: *SAINT-Plus* (Bruker, 1997–1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *OSCAIL* (McArdle, 1995).

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