Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

A new sydnone structure: 4-(phenylamino)-sydno[3,4-*a*]quinoxaline

Gordon B. Riddle, David A. Grossie* and Kenneth Turnbull

Department of Chemistry, Wright State University, Dayton, Ohio 45435, USA

Correspondence e-mail:
david.grossie@wright.edu

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.053
wR factor = 0.134
Data-to-parameter ratio = 15.1

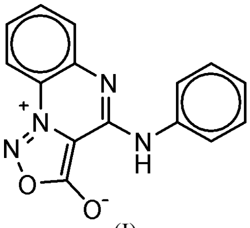
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{15}H_{10}N_4O_2$, a sydnone, is composed of four rings, *viz*. the sydnone ring, two benzene rings, and a central six-membered heterocyclic ring fused to the sydnone ring and one benzene ring.

Received 23 December 2003
Accepted 12 January 2004
Online 23 January 2004

Comment

The title compound, (I), was synthesized as part of a study of a series of sydnones aimed at determining substituent effects on the sydnone ring. Interest lies in finding substituents at the N3 or C4 position that allow for the release of NO *via* ring opening between atoms N2 and N3 and between atoms O1 and C5. Structural details of the compound were compared with previously published sydnone data in the Cambridge Structural Database (CSD; Version of 2003; Allen, 2002). Observed bond distances and angles for the sydnone ring are consistent with the other structures previously published, indicating that the substituents chosen had no significant destabilizing effects.



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. This sydnone has four rings, *viz*. the sydnone ring, two benzene rings, and a central six-membered heterocyclic ring fused to the sydnone ring and one benzene ring. On considering the s.u. values, all the bond distances are within experimental error of other sydnone structures in the CSD. Of particular interest are the N2—N3 and O1—C5 bonds. The average N2—N3 distance is 1.309 (4) Å, while in (I) this distance is 1.315 (2) Å. The average O1—C5 distance is 1.407 (4) Å, and in (I) the same distance is 1.404 (2) Å. A slight variation of the bond angles is observed; however, this is less than one degree on average.

The molecule has a twist of 2.45 (13)° between the planes of the sydnone ring and the nearer benzene ring (C6–C11). The central fused ring is slightly inclined to the sydnone ring by 1.91 (12)°. The phenyl ring (C13–C18) is inclined by only 1.67 (11)° to the central fused ring. Intramolecular N—H···O and C—H···N hydrogen bonds are also present (Table 2), and hence the entire molecule is almost planar.

DOI: 10.1107/S1600536804000777

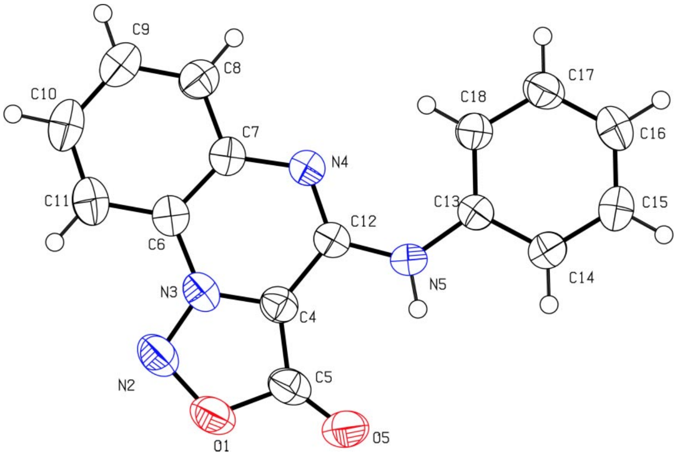


Figure 1
A view of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level.

Experimental

The title compound was prepared by reacting phosphine amide with either isocyanate or isothiocyanate in toluene, and was recrystallized from ethyl acetate (Burson, 1991).

Crystal data

$C_{15}H_{10}N_4O_2$
$M_r = 278.27$
Monoclinic, $C2/c$
$a = 29.427\ (11)$ Å
$b = 5.2611\ (17)$ Å
$c = 17.947\ (10)$ Å
$\beta = 116.20\ (2)°$
$V = 2493.1\ (19)$ Å^3
$Z = 8$
$D_x = 1.483$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4484 reflections
$\theta = 2.3–27.9°$
$\mu = 0.10$ mm^{-1}
$T = 298\ (2)$ K
Rectangular block, yellow
0.45 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 1997–1999)
$T_{min} = 0.800$, $T_{max} = 0.980$
9886 measured reflections
2870 independent reflections
2298 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 28.1°$
$h = -39 \rightarrow 39$
$k = -6 \rightarrow 6$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.134$
$S = 1.06$
2870 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 1.4621P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N5—C12	1.357 (2)	C6—C11	1.393 (2)
N5—C13	1.405 (2)	C6—C7	1.408 (2)
N4—C12	1.306 (2)	C7—C8	1.404 (2)
N4—C7	1.378 (2)	C8—C9	1.367 (3)
N3—N2	1.315 (2)	C9—C10	1.392 (3)
N3—C4	1.347 (2)	C10—C11	1.370 (3)
N3—C6	1.406 (2)	C13—C18	1.387 (2)
O1—N2	1.391 (2)	C13—C14	1.388 (2)
O1—C5	1.405 (2)	C14—C15	1.377 (3)
O5—C5	1.206 (2)	C15—C16	1.375 (3)
C4—C5	1.414 (2)	C16—C17	1.375 (3)
C4—C12	1.432 (2)	C17—C18	1.383 (2)
C12—N5—C13	130.21 (14)	N4—C7—C6	125.11 (15)
C12—N4—C7	117.34 (14)	C8—C7—C6	116.22 (16)
N2—N3—C4	115.01 (15)	C9—C8—C7	120.96 (18)
N2—N3—C6	123.86 (15)	C8—C9—C10	121.12 (19)
C4—N3—C6	121.12 (14)	C11—C10—C9	120.43 (18)
N2—O1—C5	111.67 (13)	C10—C11—C6	118.05 (18)
N3—N2—O1	103.36 (14)	N4—C12—N5	123.44 (14)
N3—C4—C5	106.65 (15)	N4—C12—C4	121.77 (15)
N3—C4—C12	119.89 (15)	N5—C12—C4	114.78 (14)
C5—C4—C12	133.46 (16)	C18—C13—C14	119.15 (15)
O5—C5—O1	122.07 (16)	C18—C13—N5	124.14 (15)
O5—C5—C4	134.67 (18)	C14—C13—N5	116.71 (15)
O1—C5—C4	103.26 (16)	C15—C14—C13	120.81 (17)
C11—C6—N3	122.09 (16)	C16—C15—C14	120.06 (17)
C11—C6—C7	123.21 (17)	C17—C16—C15	119.33 (17)
N3—C6—C7	114.70 (15)	C16—C17—C18	121.42 (17)
N4—C7—C8	118.66 (15)	C17—C18—C13	119.22 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···O5	0.86	2.41	3.101 (3)	138
C18—H18···N4	0.93	2.31	2.924 (3)	123

All H atoms were included in calculated positions as riding atoms, with C—H distances of 0.93 Å and N—H distances of 0.86 Å, and with $U_{iso} = 1.5U_{eq}$ of the carrier C or N atom.

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *OSCAIL* (McArdle, 1995).

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (1997–2000). *SMART* (Version 5.62 for Windows NT/2000) and *SAINT-Plus* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Burson, W. C. (1991). *Synthesis*, **9**, 745–746.
McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.