organic papers

3-[4-(1-Oxo-3-phenylprop-2-en-1-yl)phenyl]sydnone

S. Thamotharan,[a]
V. Parthasarathi,[a]*
Raveendra K. Hunnur,[b]
Prashant S. Shinge,[b]
Bharati Badami[b] and
Kurt J. Schenk[c]

[a]Department of Physics, Bharathidasan University, Tiruchirappalli 620 024, India, [b]Post-Graduate Department of Studies in Chemistry, Karnatak University, Dharwad 580 003, India, and [c]Institut de Cristallographie, Université de Lausanne, BPB Dorigny, CH-1015 Lausanne, Switzerland

Correspondence e-mail: vpsarati@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.065
wR factor = 0.207
Data-to-parameter ratio = 19.5

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

In the title sydnone compound, $C_{17}H_{12}N_2O_3$, the two benzene rings are oriented at angles of 7.37 (7) and 15.27 (7)° with respect to the plane of the sydnone moiety. In the crystalline state, the molecules are linked *via* weak intermolecular C—H···O interactions, forming $C(4)$ and $C(7)$ chains, and $R_2^2(14)$ and $R_2^1(7)$ rings.

Comment

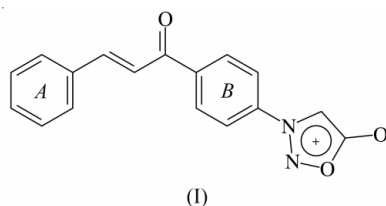The title sydnone derivative, (I), serves as a synthetic precursor for a variety of heterocyclic systems. Sydnones are less toxic, but potent, porphyrinogenic and anti-inflammatory compounds (Thamotharan *et al.*, 2003, and references therein). In view of their importance, the crystal structure analysis of (I) was undertaken.

(I)

A view of the molecule of (I), with the atomic numbering scheme, is shown in Fig. 1. The Cambridge Structural Database (November 2002 Release; Allen, 2002) currently contains nine entries that include the 3-substituted sydnone moiety, excluding 3,4-disubstituted sydnone derivatives. The bond lengths and angles in the sydnone moiety in (I) are comparable to those of related compounds (Bärnighausen *et al.*, 1963; Hope & Thiessen, 1969; Wang *et al.*, 1984; Ueng *et al.*, 1985). The dihedral angles between the planes of the sydnone ring and the attached benzene ring in the 3-substitued sydnone derivatives (2–39°) are smaller than those in the 3,4-disubstituted sydnone derivatives (55–79°; Ueng *et al.*, 1987). Rings A and B are oriented at angles of 7.37 (7) and 15.27 (7)° with respect to the plane of the sydnone moiety.
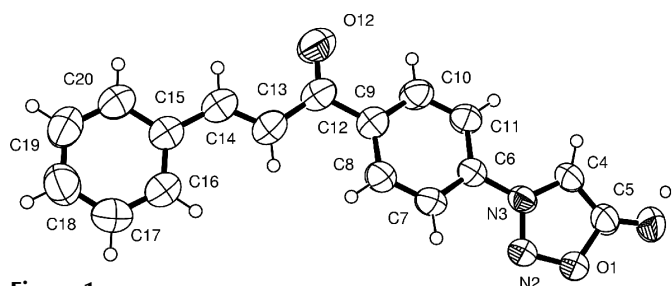

Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

The carbonyl group (C12==O12) is in an *s-cis* conformation, as can be seen from the C14—C13—C12—O12 torsion angle [-1.5 (4)°]. The dihedral angle between the planes of rings *A* and *B* is 8.56 (7)°. Atoms H13 and H14 are *trans* to each other. The C15—C14—C13 bond angle [129.3 (2)°] is significantly larger than the normal value (120°), and this difference may be due to steric repulsion between atoms H13 and H16 (2.24 Å). A close intramolecular contact is observed between atoms O12 and H14 (2.51 Å).

In the crystal structure, atom C20 acts as a donor in a weak intermolecular C—H···O interaction with carbonyl atom O12 of an adjacent molecule at $(1 - x, 2 - y, -z)$, leading to an $R_2^2(14)$ motif. Atom C4 is involved in a weak intermolecular C—H···O interaction with atom O5 of the sydnone moiety of an adjacent molecule at $(2 - x, \frac{1}{2} + y, \frac{1}{2} - z)$ (Table 1). This interaction produces a continuous chain that runs parallel to the *b* axis and has a graph-set motif of $C(4)$. Atom C11 participates in a weak intermolecular C—H···O interaction with atom O5 of the sydnone moiety of an adjacent molecule at $(2 - x, \frac{1}{2} + y, \frac{1}{2} - z)$. This interaction links the molecules into another chain, which runs parallel to the *b* axis and has a graph-set motif of $C(7)$. These two chains combine to form an $R_2^1(7)$ ring (Bernstein *et al.*, 1995).

Experimental

3-(4′-Acetylphenyl)sydnone (2 g, 0.01 mol) was suspended in sodium hydroxide solution (0.5 g sodium hydroxide in 5 ml water and 5 ml ethanol) and benzaldehyde (1 g, 0.01 mol) was added. The reaction mixture was stirred for 30 min at room temperature and the resulting precipitate was collected immediately and washed with water. Recrystallization from methanol–dioxane (2:3) afforded crystals of (I) suitable for X-ray analysis (m.p. 471 K).

Crystal data

$C_{17}H_{12}N_2O_3$	$D_x = 1.372$ Mg m^{-3}
$M_r = 292.29$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 6020
$a = 7.5839$ (8) Å	reflections
$b = 7.9343$ (7) Å	$\theta = 1.9$–28.0°
$c = 23.543$ (2) Å	$\mu = 0.10$ mm^{-1}
$\beta = 92.954$ (8)°	$T = 293$ (2) K
$V = 1414.8$ (2) Å^3	Parallelepiped, translucent orange
$Z = 4$	$0.65 \times 0.26 \times 0.20$ mm

Data collection

Stoe IPDS diffractometer	$R_{int} = 0.026$
φ scans	$\theta_{max} = 29.5°$
13 010 measured reflections	$h = -10 \rightarrow 10$
3882 independent reflections	$k = -10 \rightarrow 10$
2947 reflections with $I > 2\sigma(I)$	$l = -32 \rightarrow 32$

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O5^i	0.93	2.28	3.187 (2)	164
C11—H11···O5^i	0.93	2.51	3.429 (3)	169
C20—H20···O12^{ii}	0.93	2.54	3.285 (3)	137

Symmetry codes: (i) $2 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 - x, 2 - y, -z$.

All H atoms were placed in idealized positions (C—H = 0.93 Å) and constrained to ride on their parent atoms [$U_{iso}(H) = 1.2U_{eq}(C)$]. The ratio of maximum-to-minimum residual density is 2.3. The highest peak in the final difference map was found near atom H20, at a distance of 0.83 Å.

Data collection: *IPDS Software* (Stoe & Cie, 1997); cell refinement: *IPDS Software*; data reduction: *IPDS Software*; 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*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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