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4-(2-Benzoylhydrazinothiazol-4-yl)-3-phenylsydnone

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.129
Data-to-parameter ratio = 12.7

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

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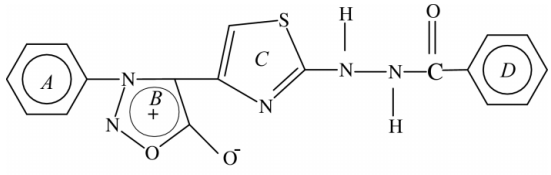
In the title compound, $C_{18}H_{13}N_5O_3S$, both the five- and six-membered rings are planar. The phenyl ring attached to the sydnone ring is nearly perpendicular to both the sydnone ring [78.2 (1)°] and the thiazole ring [80.1 (1)°]. The dihedral angle between the sydnone and thiazole rings is 20.5 (1)°. The molecular packing in the crystal is stabilized by intermolecular N—H···O hydrogen bonds and C—H···O interactions.

Comment

Sydnones are the product of dehydration of *N*-nitroso-α-amino acids, named after the site of their discovery at the University of Sydney (Earl & Mackney, 1935). They have attained importance due, not only their structural features and chemical properties, but also to their biological properties. Sydnones are less toxic (Pillai *et al.*, 1993), potent porphyrinogenic (Sutherland *et al.*, 1986; Marks, 1987), anti-inflammatory (Satyanarayana & Rao, 1995) and have the effect of scavenging free-radicals (Narla & Rao, 1995). Sydnone halogen derivatives change their colour irreversibly under the influence of UV light (λ < 400 nm) (Hašek *et al.*, 1979). The title compound, (I), has been tested for its anti-inflammatory and gastrointestinal effect. It also showed significant inhibition of granuloma formation in anti-inflammatory activity and less ulcerogenicity in gastric mucosa, as compared to aspirin (Yelamaggad *et al.*, 1993). In view of its biological importance, the crystal structure analysis of (I) was carried out.



(I)

X-ray crystallographic data are available in the literature for eight 3,4-disubstituted sydnones (Ueng *et al.*, 1989, 1987*a*,*b*; Hašek *et al.*, 1979, 1978). The bond lengths in the sydnone moiety of (I) agree with the corresponding average values given in Table 1 for these other compounds. The dihedral angles between the sydnone ring and the attached phenyl ring in the 3,4-disubstituted sydnone derivatives (55–79 °) are larger than those in the 3-substituted sydnone derivatives (2–39 °) (Ueng *et al.*, 1987*a*). The phenyl ring attached to the sydnone ring is nearly perpendicular both to the sydnone ring [78.2 (1)°] and to the thiazole ring [80.1 (1)°]. The dihedral angles between the mean planes of rings *B*, *C* and

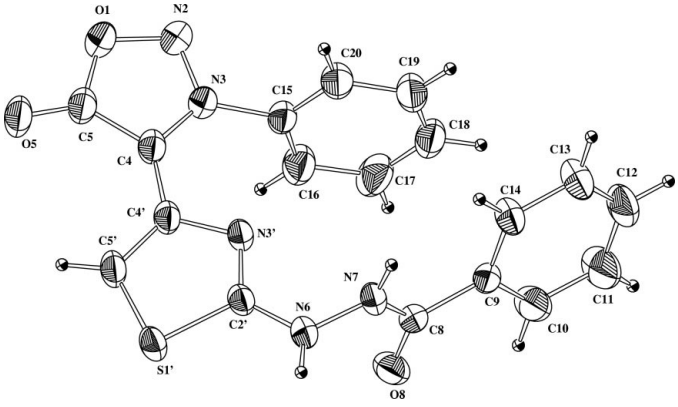


Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

D are *B*/*C* 20.5 (1)°, *B*/*D* 67.2 (1)° and *C*/*D* 77.8 (1)°. In the solid state, the inversion-related molecules are linked by N—H···O hydrogen bonds and C—H···O interactions (Table 2), to form column-like structures along the *c* direction (Fig. 2).

Experimental

To a stirred suspension of 4-bromoacetyl-3-phenylsydnone (2.83 g, 0.01 mol) in absolute ethanol (20 ml) was added a solution of 1-benzoyl thiosemicarbazide (1.9 g, 0.01 mol) in absolute ethanol (20 ml) and the mixture was stirred at room temperature for 20 min. The light-yellow precipitate that separated was filtered off and washed with 10% $NaHCO_3$ solution and water. The solid was dried and crystallized from ethanol (yield 63%, m.p. 468–469 K).

Crystal data

$C_{18}H_{13}N_5O_3S$
M_r = 379.39
Triclinic, $P\overline{1}$
a = 8.290 (2) Å
b = 9.646 (3) Å
c = 11.715 (3) Å
α = 81.54 (2)°
β = 81.74 (2)°
γ = 71.59 (2)°
V = 874.4 (4) Å^3
Z = 2
D_x = 1.441 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14–30°
μ = 1.92 mm^{-1}
T = 293 (2) K
Prism, yellow
0.3 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.637, T_{max} = 0.682
3306 measured reflections
3105 independent reflections
2709 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 69.9°
$h = -9 \rightarrow 10$
$k = -3 \rightarrow 11$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.129
S = 1.04
3105 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0778P)^2 + 0.1813P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0056 (9)

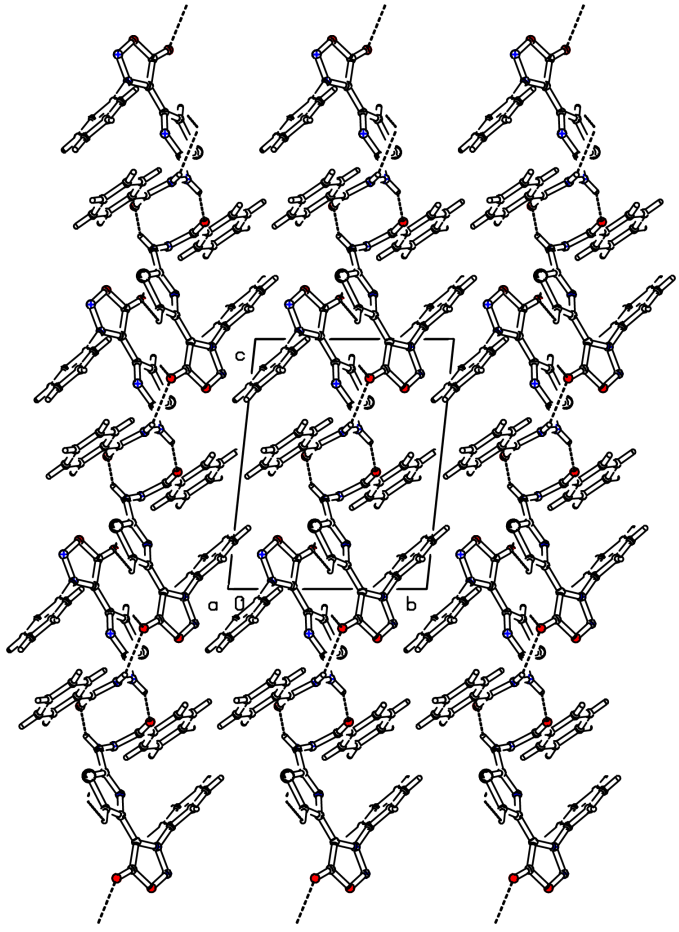


Figure 2
Packing diagram showing N—H···O and C—H···O hydrogen bonds, viewed down the *a* axis.

Table 1
Comparison of the bond lengths (Å) in the sydnone ring of the title compound with the corresponding average values found in 3,4-disubstituted sydnone compounds.

Bond	Title compound	Average value
O1—C5	1.391 (3)	1.407 (4)
O1—N2	1.380 (3)	1.379 (3)
N2—N3	1.319 (3)	1.310 (3)
N3—C4	1.344 (3)	1.351 (3)
C4—C5	1.423 (3)	1.413 (3)
C5—O5	1.210 (3)	1.206 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6···O8^{i}	0.86	2.12	2.827 (2)	139
N7—H7···O5^{ii}	0.86	2.48	3.185 (3)	140
C5′—H5′···O5^{iii}	0.93	2.58	3.144 (3)	120

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

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *CAD*-4 *Software*;

program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *PARST*97 (Nardelli, 1995).

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