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N-(4-Acetylphenyl)-*N*′-[(4-sydnon-3-yl)-phenyl]urea dimethylformamide solvate

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In the title compound, $C_{17}H_{14}N_4O_4 \cdot C_3H_7NO$, the dihedral angle between the planes of the sydnone ring and the attached benzene ring is 13.56 (8)°. The benzene rings are inclined at an angle of 21.02 (8)° with respect to one another. In the solid state, intermolecular N—H···O hydrogen bonds and weak intermolecular C—H···O interactions are observed.

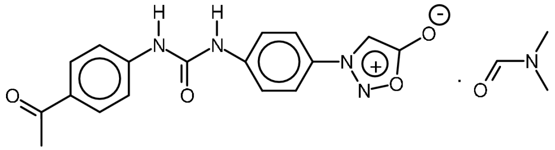
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.116
Data-to-parameter ratio = 11.2

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

Comment

Sydnones are less toxic, but potent, porphyrinogenic and anti-inflammatory compounds (Thamotharan, Parthasarathi, Sanyal *et al.*, 2003; Thamotharan, Parthasarathi, Hunnur *et al.*, 2003, and references therein). The present study was undertaken to determine the crystal and molecular structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles in the sydnone moiety in (I) are comparable to those of related compounds (Ueng *et al.*, 1985; Thamotharan, Parthasarathi, Sanyal *et al.*, 2003; Thamotharan *et al.*, 2004). The dihedral angle between the sydnone moiety and the attached benzene ring in 3-substituted sydnone derivatives ranges from 2 to 39° (Ueng *et al.*, 1987). In (I), the dihedral angle between the planes of the sydnone ring and the attached benzene ring is 13.56 (8)°. The two benzene rings are inclined at an angle of 21.02 (8)° with respect to one another.

In the crystalline state, carbonyl atom O1*A* of dimethylformamide accepts two different intermolecular N—H···O hydrogen bonds (Table 1) from the same neighbouring molecule, one from the N12—H12 group and the other from the N14—H14 group. These hydrogen bonds lead to an $R_2^1(6)$

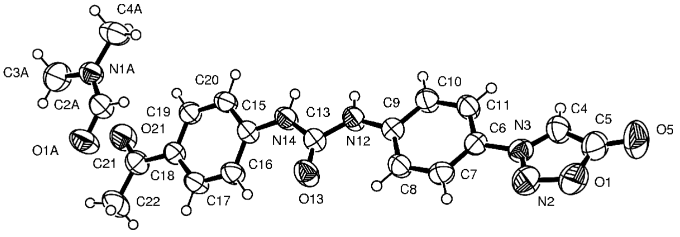


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

motif (Bernstein *et al.*, 1995). Atom C4 acts as a donor for a weak intermolecular C—H···O interaction with atom O13 of an adjacent molecule. This interaction links the molecules into a chain, which runs parallel to the *b* axis and has a graph-set motif of *C*(10). Atom C2*A* of the DMF molecule has a weak intermolecular C—H···O interaction with atom O5 of the sydnone moiety of an adjacent molecule. An intramolecular short contact is observed between atoms H12 and H14 (1.99 Å).

Experimental

The title compound was prepared by refluxing 3-(*p*-azidocarbonyl)-phenylsydnone (0.001 *M*) with *p*-aminoacetophenone (0.001 *M*) in anhydrous toluene (15 ml) for 4 h at 393 K. The crystalline product that separated out was collected and washed with toluene and petroleum ether. The title compound, (I), was recrystallized from an ethanol–DMF mixture (m.p. 489–490 K).

Crystal data

$C_{17}H_{14}N_4O_4 \cdot C_3H_7NO$
M_r = 411.42
Triclinic, $P\overline{1}$
a = 7.7954 (8) Å
b = 8.3922 (8) Å
c = 16.9225 (17) Å
α = 79.057 (2)°
β = 84.267 (2)°
γ = 65.049 (2)°
V = 985.26 (17) Å^3
Z = 2
D_x = 1.387 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1983 reflections
θ = 2.0–25.0°
μ = 0.10 mm^{-1}
T = 273 (2) K
Prism, colourless
0.30 × 0.22 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
5038 measured reflections
3153 independent reflections
2493 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.016
$\theta_{\rm max}$ = 25.0°
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.116
S = 1.03
3153 reflections
282 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 0.2508P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.16 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.13 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H12···O1*A*$^{\rm i}$	0.85 (2)	2.07 (2)	2.880 (2)	159.3 (19)
N14—H14···O1*A*$^{\rm i}$	0.88 (2)	1.99 (2)	2.831 (2)	159 (2)
C4—H4···O13$^{\rm ii}$	0.93	2.44	3.332 (3)	161
C2*A*—H2*A*···O5$^{\rm iii}$	0.93	2.36	3.267 (3)	166

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

The positions of the amine H atoms were determined from a difference Fourier map and refined freely, along with their isotropic displacement parameters. The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). All remaining H atoms were placed in idealized positions (C—H = 0.93 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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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