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4-(4-Methoxybenzoyl)-2,2-dimethyl-5-phenylamino-2,3-dihydro-1,3,4-thiadiazole

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.063
wR factor = 0.124
Data-to-parameter ratio = 13.7

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

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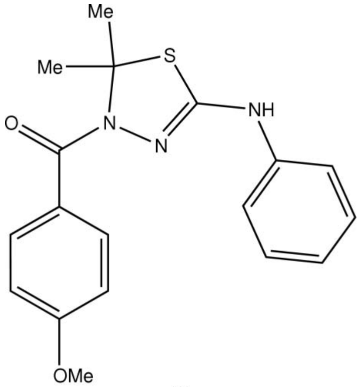
In the title compound, $C_{18}H_{19}N_3O_2S$, the thiadiazole ring adopts an envelope conformation. The crystal structure is stabilized by intermolecular N—H···O hydrogen bonds, forming one-dimensional chains parallel to the *b* axis.

Comment

Thiadiazole derivatives such as 1-(1,3,4-thiadiazol-2-yl)-3-methylthio-6,7-dihydrobenzo[*c*]thiophen-4(5*H*)-one are known to exhibit high antibacterial activity against *Staphylococcus aureus* (Tehranchian *et al.*, 2005).



(I)

In the title compound, (I), the thiadiazole ring adopts an envelope conformation, with atom C8 at the flap position; C8 deviates from the mean plane through the other four atoms by 0.204 (3) Å. The dihedral angle between C1–C6/N1 (Fig. 1) and the fragment C9–C15/O2 is 28.5 (1)°. Atom C16 is displaced by 0.257 (4) Å from the mean plane through the latter fragment. The bond lengths and angles are in normal ranges (Allen *et al.*, 1987) and agree well with those in (5*R*,5′*R*)-2,2′-diacetamido-4,4′-di-*N*-acetyl-5′-(1-deoxy-2,3,4-tri-*O*-acetyl-D-erythritol-1-yl)-bis(1,3,4-thiadiazoline) monohydrate (Alho *et al.*, 2002).

Three intramolecular hydrogen bonds are present (Table 2) and, as a result, three pseudo-six-membered rings are formed (Fig. 1). In the crystal structure, the molecules are linked by intermolecular N—H···O interactions (Table 2), forming one-dimensional chains along the *b* axis (Fig. 2).

Experimental

A solution of *p*-phenylthiosemicarbazide (3.34 g, 20 mmol) in acetone (50 ml) was added dropwise to an acetone solution (50 ml) containing an equimolar amount of *p*-anisoyl chloride (2.52 g, 20 mmol) and ammonium thiocyanate. The solution was refluxed for

1 h and then poured into a beaker containing some ice blocks. The resulting white precipitate was filtered and washed with distilled water and cold ethanol, then dried under a vacuum. Crystals suitable for X-ray investigation were obtained by recrystallization from dimethyl sulfoxide (yield 85%, m.p 447.8–449.3 K).

Crystal data

$C_{18}H_{19}N_3O_2S$
$M_r = 341.42$
Orthorhombic, *Pbca*
$a = 9.4042\ (14)$ Å
$b = 15.603\ (2)$ Å
$c = 23.143\ (4)$ Å
$V = 3395.8\ (9)$ Å^3
$Z = 8$
$D_x = 1.336$ Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 2397 reflections
$\theta = 1.7$–$25.2°$
$\mu = 0.21$ mm^{-1}
$T = 298\ (2)$ K
Block, colourless
0.48 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.907$, $T_{\max} = 0.960$
16783 measured reflections

3073 independent reflections
2446 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 25.2°$
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 18$
$l = -18 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.124$
$S = 1.25$
3073 reflections
224 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0359P)^2 + 2.015P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected bond lengths (Å).

S1—C7	1.745 (3)	N1—C6	1.407 (4)
S1—C8	1.844 (3)	N2—C7	1.286 (3)
O1—C9	1.235 (3)	N2—N3	1.405 (3)
O2—C13	1.371 (3)	N3—C9	1.355 (3)
O2—C16	1.412 (4)	N3—C8	1.481 (3)
N1—C7	1.363 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···N2	0.93	2.42	2.894 (4)	112
C11—H11···N2	0.93	2.51	2.828 (3)	100
C17—H17*A*···O1	0.96	2.40	2.832 (4)	107
N1—H1*B*···O1$^{\rm i}$	0.845 (17)	2.116 (14)	2.923 (3)	160 (2)

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

The H atom of the aniline fragment was located in a difference map and refined isotropically, with a bond length restraint of N—H = 0.844 (10) Å. The remaining H atoms were located in a difference map, repositioned geometrically and refined as riding, with C—H = 0.93–0.96 Å and with $U_{\rm iso}({\rm H}) = xU_{\rm eq}$(carrier atom), where $x = 1.5$ for methyl H and 1.2 for all other H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

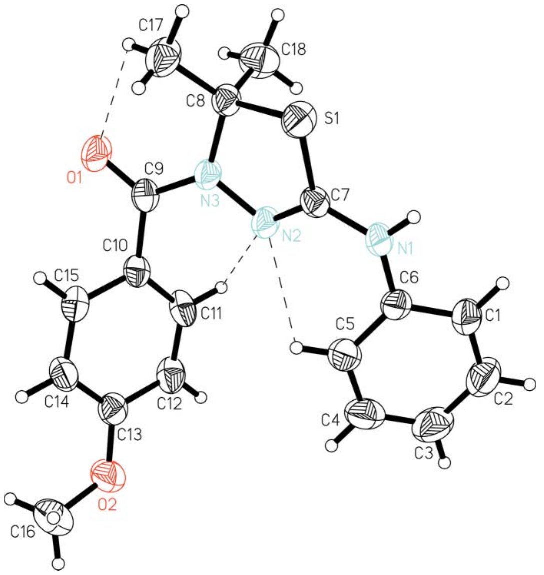


Figure 1
The molecular structure of the title compound, (I). Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate the intramolecular hydrogen bonds.

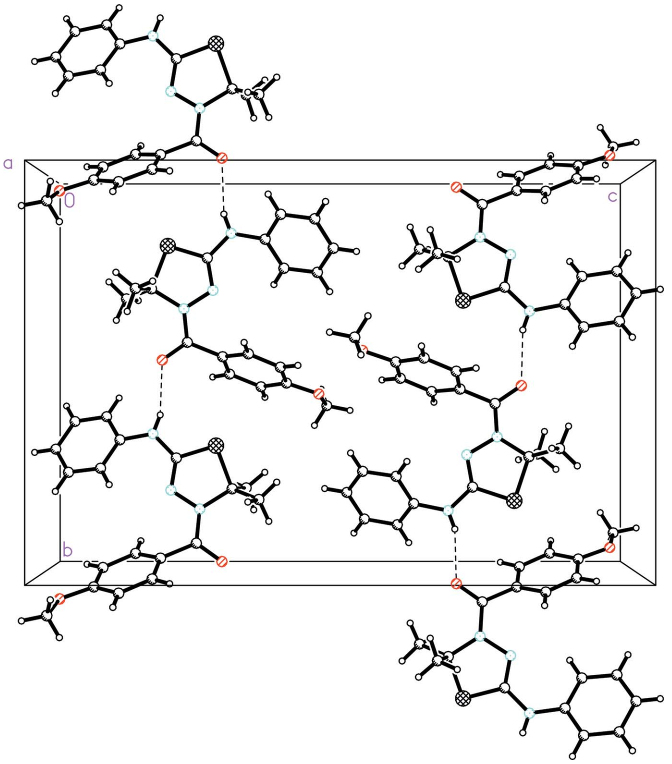


Figure 2
Packing diagram of (I), viewed down the *a* axis. The dashed lines denote the N—H···O intermolecular hydrogen bonds.

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References

Alho, M. A. M., Baggio, R., Garland, M. T., D'Accorso, N. B. & Varela, O. (2002). *Carbohydr. Res.* **337**, 1397–1403.

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2000). *SADABS* (Version 2.01), *SMART* (Version 5.603) and *SAINT* (Version 6.36a). Bruker AXS Inc., Madison, Wisconsin, USA.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Tehranchian, S., Akbarzadeh, T., Fazeli, R. M., Jamalifar, H. & Shafiee, A. (2005). *Bioog. Med. Chem. Lett.* **15**, 1023–1025.

o1074 Yusof *et al.* • $C_{18}H_{19}N_3O_2S$ *Acta Cryst.* (2006). E**62**, o1072–o1074