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4-Phenyl-1-(1*H*-1,2,4-triazol-3-ylcarbonyl)-thiosemicarbazide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.077
Data-to-parameter ratio = 13.2

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

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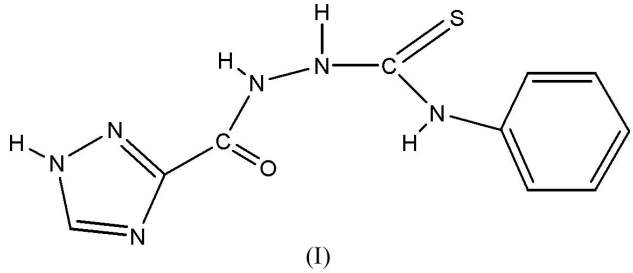
The title compound, $C_{10}H_{10}N_6OS$, was synthesized by the reaction of 1*H*-1,2,4-triazol-3-ylhydrazine with phenyl isocyanate in benzene. The molecule is non-planar, the dihedral angle between the two aromatic rings being 62.07 (7)°. N—H···O, N—H···N and N—H···S hydrogen bonds are observed.

Comment

Azole compounds, such as derivatives of pyrazole, imidazole, triazole (including benzotriazole), tetrazole and indole, exhibit extensive biological activity and have become a central focus in the study of agricultural chemicals and medicines (Ernest & William, 1982). Schiff bases often represent good examples of biologically active substructures and a study of a triazole Schiff base has been reported (Sauter *et al.*, 1991). The hydrazonecarbonyl grouping has also been shown to be bioactive (Zhi *et al.*, 2003) and a number of structures of triazole compounds containing the hydrazonecarbonyl group have been reported (Pan & Yang, 2005; Yang & Pan, 2004). In a search for more effective antibacterial medicines, we have synthesized the title compound, (I).



(I)

The title molecule (Fig. 1) is non-planar; the dihedral angle between the two aromatic rings is 62.07 (7)°. As a result of conjugation, the C═O distance [1.216 (2) Å] is longer than the normal value of 1.20 Å (John, 1998), and the C2—N2 bond distance [1.346 (2) Å] is longer than the C═N double-bond distance (1.32 Å; John, 1998) and shorter than the C—N single-bond distance (1.475 Å; John, 1998).

Intermolecular hydrogen bonds are observed (Table 1 and Fig. 2). The N—H···N and N—H···O hydrogen bonds form a ten-membered ring (Fig. 2).

Experimental

1*H*-1,2,4–Triazol-3-ylhydrazine (0.02 mol, 2.54 g) was dissolved in benzene (50 ml) and phenyl isocyanate (0.02 mol, 2.70 g) was added to the solution. The mixture was refluxed for 8 h and the precipitate formed was collected by filtration and washed with benzene. The

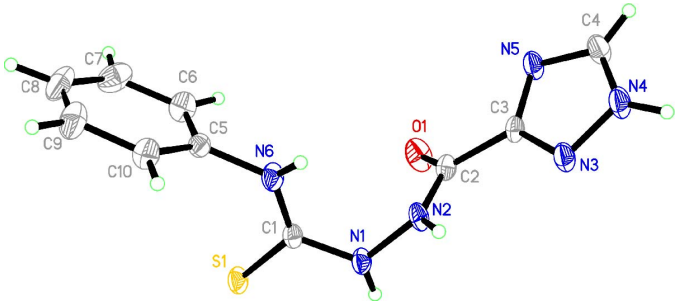


Figure 1
The structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

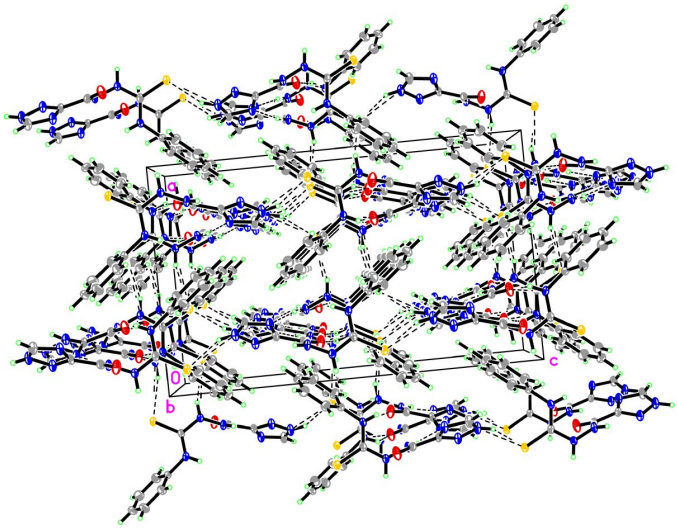


Figure 2
The packing of (I), showing the intermolecular and intramolecular hydrogen bonds as dashed lines.

product was recrystallized from benzene and dried under reduced pressure to give the title compound. The compound (2.0 mmol, 0.53 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 40 d to obtain colourless single crystals, which were collected and washed with distilled water (m.p: 490–491 K). IR ν_{max}(KBr, cm^{-1}): 3327, 3150, 3124, 1704, 1541, 1450, 1252, 1194, 1163, 697. ^{1}H NMR (200 MHz, DMSO): δ 14.71 (1H), 10.58 (1H), 9.80 (2H), 8.78 (1H), 7.05–7.60 (5H).

Crystal data

$C_{10}H_{10}N_6OS$
M_r = 262.30
Orthorhombic, *Pbcn*
a = 12.1981 (10) Å
b = 10.1097 (8) Å
c = 19.9112 (16) Å
V = 2455.4 (3) Å^3
Z = 8
D_x = 1.419 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1478 reflections
θ = 5.2–42.9°
μ = 0.26 mm^{-1}
T = 293 (2) K
Block, colourless
0.38 × 0.25 × 0.09 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.908, T_{max} = 0.977
13557 measured reflections
2684 independent reflections
1653 reflections with $I > 2\sigma(I)$
R_{int} = 0.085
θ_{max} = 27.0°
$h = -15 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -25 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.077
S = 0.85
2684 reflections
203 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0246P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6A···O1^{i}	0.812 (18)	2.44 (2)	2.904 (2)	116.9 (17)
N4—H4A···S1^{ii}	0.88 (2)	2.45 (2)	3.259 (2)	152 (2)
N2—H2···N5^{i}	0.85 (2)	2.08 (2)	2.926 (2)	171 (2)
N1—H1···S1^{iii}	0.890 (15)	2.460 (16)	3.322 (2)	163.0 (17)

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

All H atoms were located in a difference map and their parameters were refined. The N—H distances are in the range 0.812 (18)–0.890 (15) Å and the C—H distances are in the range 0.903 (19)–0.94 (2) Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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