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3-(1,5-Dimethylpyrazol-4-yl)-4-phenyl-1*H*-1,2,4-triazole-5(4*H*)-thione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.037
wR factor = 0.100
Data-to-parameter ratio = 11.4

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

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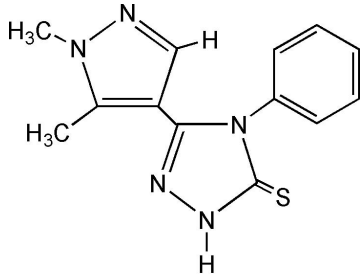
The title compound, $C_{13}H_{13}N_5S$, possesses crystallographically imposed mirror symmetry. In the crystal structure, the molecules interact through strong intermolecular N—H···N hydrogen bonds to form ribbons running parallel to the *a* axis.

Comment

In recent years, 1,2,4-triazole derivatives have been extensively studied due to their broad spectrum of biological activities, the most interesting of which are microbicidal (Ismalel *et al.*, 1984), antifungal (Mishra *et al.*, 1991), antibacterial (Mohan *et al.*, 1990), anti-inflammatory (Rani *et al.*, 1990) and plant-growth regulation activities (Wang *et al.*, 1998). Pyrazoles have been widely investigated due to their close association with various biological effects (Hu *et al.*, 2002). In the course of our systematic studies aimed at the synthesis of new bioactive compounds incorporating these groups, we have synthesized the title compound, (I), the structure of which is reported here.



(I)

Bond distances and angles (Table 1) are as expected for this type of compound. The molecule has crystallographically imposed mirror symmetry, with the triazole and pyrazole rings lying on the mirror plane and the phenyl ring strictly orthogonal to it.

In the crystal structure, the molecules are linked through strong N—H···N hydrogen-bond interactions (Table 2) to form ribbons running parallel to the *a* axis (Fig. 2).

Experimental

A mixture of 1,5-dimethyl-4-pyrazolylcarbohydrazide (0.925 g, 6 mmol) and phenyl isothiocynate (0.811 g, 6 mmol) in ethanol (10 ml) was refluxed for 3 h. The solid was separated by filtering after cooling, washed with cold ethanol, dried and recrystallized from ethanol to give *N*-(1′,5′-dimethyl-4′-pyrazolylcarbonyl)-*N*′-phenyl-3-thiosemicarbazide (0.581 g, 2 mmol), which was refluxed in aqueous sodium carbonate solution (30 ml) for 7 h. The reaction mixture was cooled and acidified to pH 5 with 2 *M* hydrochloric acid. The resulting

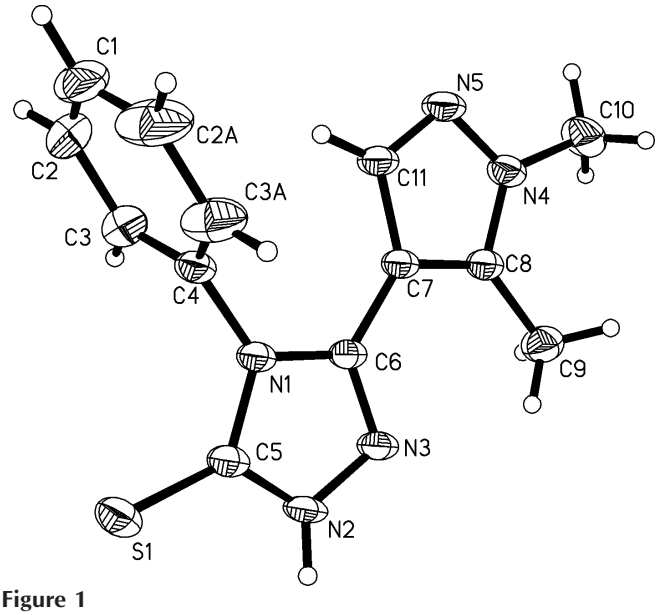


Figure 1
View of the title compound, with 35% probability displacement ellipsoids. Only one disorder component is shown. [Symmetry code (A): $x, \frac{1}{2} - y, z$.]

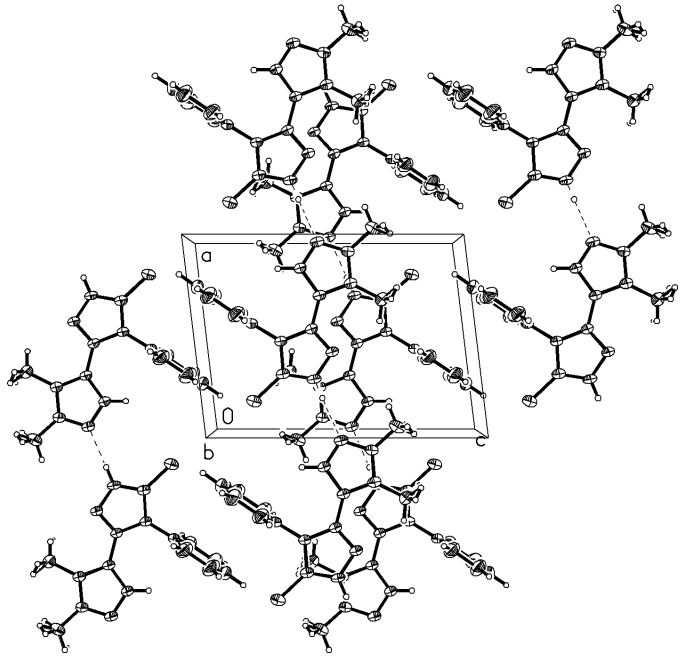


Figure 2
The molecular packing of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

white crude precipitate was filtered, washed with cold water, dried, and recrystallized from DMF to give the title compound (m.p. 573 K).

Crystal data

$C_{13}H_{13}N_5S$
$M_r = 271.35$
Monoclinic, $P2_1/m$
$a = 8.363$ (7) Å
$b = 6.898$ (6) Å
$c = 11.524$ (10) Å
$\beta = 96.879$ (12)°
$V = 660.0$ (10) Å^3
$Z = 2$
$D_x = 1.365$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 912 reflections
$\theta = 2.5$–23.0°
$\mu = 0.24$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.32 × 0.24 × 0.16 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.933$, $T_{max} = 0.962$
3602 measured reflections
1266 independent reflections
972 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 25.0°$
$h = -8 \rightarrow 9$
$k = -8 \rightarrow 8$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.100$
$S = 1.07$
1266 reflections
111 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2 + 0.199P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.662 (3)	N4—C8	1.340 (4)
N1—C5	1.373 (3)	N5—C11	1.308 (3)
N1—C6	1.374 (3)	C6—C7	1.450 (4)
N2—C5	1.319 (3)	C7—C8	1.381 (4)
N3—C6	1.298 (3)	C7—C11	1.399 (3)
C5—N1—C6	108.0 (2)	N2—C5—S1	127.38 (19)
C5—N2—N3	114.5 (2)	N1—C5—S1	129.5 (2)
C6—N3—N2	103.9 (2)	N3—C6—N1	110.5 (2)
C8—N4—N5	112.3 (2)	C8—C7—C11	104.6 (2)
C11—N5—N4	105.5 (2)	N4—C8—C7	106.3 (2)
N2—C5—N1	103.1 (2)	N5—C11—C7	111.4 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N5^{i}	0.86	1.94	2.803 (2)	179.5

Symmetry code: (i) $1 + x, y, z$.

All H atoms were placed in calculated positions, with C—H = 0.93–0.96 Å, N—H = 0.91 Å, and refined using a riding model, with U_{iso}(H) = 1.2 or 1.5 (for methyl H atoms) times U_{eq}(C,N). The H atoms on C9 and C10 were found to be disordered. They were refined over two positions (occupancies of 0.5 for each atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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