

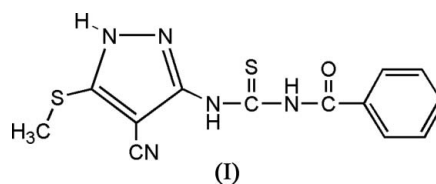
Li-Rong Wen, Ming Li,* Jian-Xia
Zhou and Peng LiuCollege of Chemistry and Molecular
Engineering, Qingdao University of Science and
Technology, Qingdao 266042, People's
Republic of China

Correspondence e-mail: liming928@263.net

Key indicators

Single-crystal X-ray study
 $T = 294\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
Disorder in main residue
 R factor = 0.037
 wR factor = 0.095
Data-to-parameter ratio = 13.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.1-Benzoyl-3-(4-cyano-5-methylsulfanyl-
1H-pyrazol-3-yl)thioureaIn the title compound, $\text{C}_{13}\text{H}_{11}\text{N}_5\text{OS}_2$, the dihedral angle between the planes of the phenyl and pyrazole rings is $25.62(2)^\circ$. The crystal packing is stabilized by intra- and intermolecular hydrogen bonds which link the molecules into a three-dimensional network.Received 4 January 2006
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Comment

Pyrazole and its derivatives represent one of the most active classes of compounds; they possess a wide spectrum of biological activities, such as antibacterial, fungicidal, herbicidal and insecticidal activities (Li *et al.*, 1997; Zhao *et al.*, 2001). In the course of our systematic studies aimed at the synthesis of new bioactive compounds, we synthesized the title compound, (I), the structure of which is reported here.

In (I), the central S2/O1/N3/N4/C6/C7 system is roughly planar (plane $p1$), with a maximum deviation from the plane of $0.114(2)\text{ \AA}$ for N4, and forms dihedral angles of $24.82(5)^\circ$ and $50.54(6)^\circ$ with the planes of the pyrazole ($p2$) and phenyl ($p3$) rings, respectively. The dihedral angle between $p2$ and $p3$ is $25.62(2)^\circ$. Bond distances and angles (Table 1) are as expected for this type of compound. The most interesting feature of (I) is the combination of intra- and intermolecular hydrogen-bond interactions (Table 2), forming an extended three-dimensional network in the crystal structure (Fig. 2).

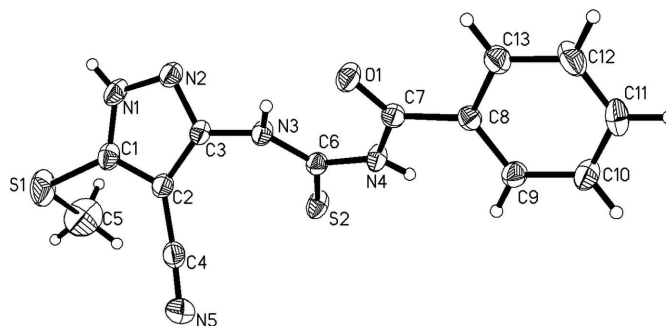


Figure 1

View of the title compound, shown with 35% probability displacement ellipsoids. Only the major component of the disordered methyl group is shown.

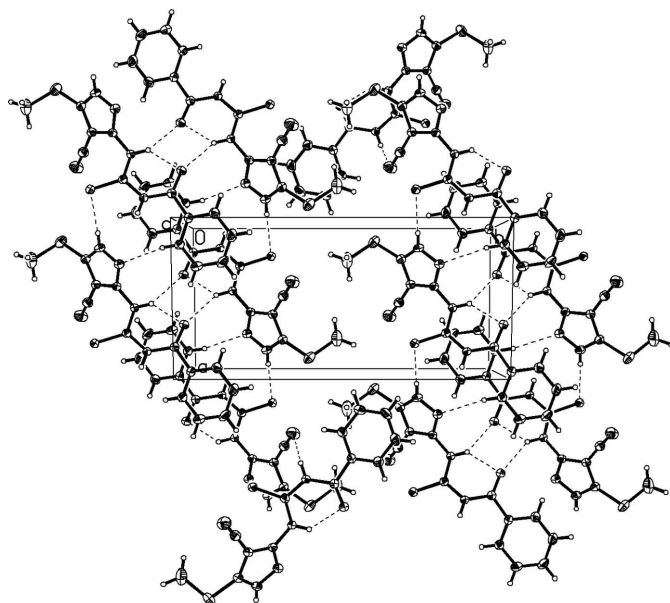


Figure 2
The molecular packing of the title compound, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines. Only the major component of the disordered methyl group is shown.

Experimental

A solution of 5-amino-4-cyano-3-methylsulfanyl-1*H*-pyrazole (0.4 g, 2.6 mmol), obtained according to a previously reported procedure (Wen *et al.*, 2005), and an equimolar amount of benzoyl isothiocyanate in acetone (40 ml) were stirred under microwave irradiation for 20 min (700 W, 313 K). Single crystals of the title compound suitable for X-ray analysis were obtained by recrystallization from ethyl acetate as a light-yellow solid (m.p. 466 K).

Crystal data

$C_{13}H_{11}N_5OS_2$	$D_x = 1.454 \text{ Mg m}^{-3}$
$M_r = 317.39$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 3337 reflections
$a = 7.6970 (11) \text{ \AA}$	$\theta = 2.6\text{--}26.2^\circ$
$b = 15.892 (2) \text{ \AA}$	$\mu = 0.37 \text{ mm}^{-1}$
$c = 12.0775 (17) \text{ \AA}$	$T = 294 (2) \text{ K}$
$\beta = 100.999 (2)^\circ$	Block, light yellow
$V = 1450.2 (3) \text{ \AA}^3$	$0.34 \times 0.28 \times 0.24 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	2938 independent reflections
φ and ω scans	2287 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.026$
$T_{\text{min}} = 0.882$, $T_{\text{max}} = 0.914$	$\theta_{\text{max}} = 26.3^\circ$
8039 measured reflections	$h = -6 \rightarrow 9$
	$k = -19 \rightarrow 19$
	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.7311P]$
$R[F^2 > 2\sigma(F^2)] = 0.037$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
2938 reflections	$\Delta\rho_{\text{min}} = -0.34 \text{ e \AA}^{-3}$
212 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (\AA , $^\circ$).

S1—C5	1.803 (5)	N3—C6	1.338 (2)
S2—C6	1.6677 (19)	N4—C6	1.383 (2)
O1—C7	1.219 (2)	N5—C4	1.143 (3)
N1—C1	1.333 (3)	C1—C2	1.390 (3)
N1—N2	1.353 (2)	C2—C3	1.405 (3)
N2—C3	1.325 (2)		
C1—N1—N2	114.01 (17)	N2—C3—C2	111.98 (16)
C3—N2—N1	103.70 (16)	N5—C4—C2	176.9 (2)
N1—C1—C2	105.74 (17)	N3—C6—N4	116.55 (16)
C1—C2—C3	104.57 (17)	O1—C7—N4	121.81 (17)
C4—C2—C3—N3	0.4 (4)	C6—N4—C7—O1	2.5 (3)
C3—N3—C6—S2	−1.5 (3)		

Table 2
Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N3—H3 \cdots O1	0.86 (2)	2.00 (2)	2.662 (2)	133.9 (18)
N3—H3 \cdots O1 ⁱ	0.86 (2)	2.46 (2)	3.149 (2)	137.5 (18)
N4—H4 \cdots N5 ⁱⁱ	0.83 (2)	2.40 (2)	3.111 (3)	143.1 (19)
N1—H1 \cdots S2 ⁱⁱⁱ	0.80 (2)	2.54 (2)	3.334 (2)	170 (2)
C9—H9 \cdots N2 ^{iv}	0.93	2.60	3.521 (2)	169

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

H atoms bonded to N atoms were located in a difference map and refined freely. All other H atoms were placed in calculated positions, with $C\text{—}H = 0.93\text{--}0.96 \text{ \AA}$, and refined using a riding model, with $U_{\text{iso}}(H) = 1.2$ or $1.5 U_{\text{eq}}(C)$. The methyl group was found to be disordered; C5 and attached H atoms were refined over two positions with occupancies of 0.80 (3) and 0.20 (3).

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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References

- Bruker (1998). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1999). SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
 Li, Z. M., Chen, H. S., Zhao, W. G. & Huang, X. S. (1997). *Chem. J. Chin. Univ.* **18**, 1794–1799.
 Sheldrick, G. M. (1996). SADABS, University of Göttingen, Germany.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Wen, L. R., Wang, S. W., Li, M. & Yang, H. Z. (2005). *Chin. J. Chem.* **23**, 1231–1235.
 Zhao, W. G., Chen, H. S., Li, Z. M., Han, Y. F., Yan, H., Lai, J. Y. & Wang, S. H. (2001). *Chem. J. Chin. Univ.* **22**, 939–942.