

Fang-Zhong Hu, Gui-Feng  
Zhang, You-Quan Zhu and  
Hua-Zheng Yang\*State Key Laboratory of Elemento-Organic  
Chemistry, Nankai University, Tianjin 300071,  
People's Republic of ChinaCorrespondence e-mail:  
youquan\_zhu@mail.nankai.edu.cn

## Key indicators

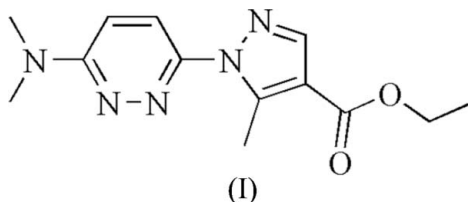
Single-crystal X-ray study  
 $T = 294$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.047  
 $wR$  factor = 0.134  
Data-to-parameter ratio = 15.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Ethyl 1-[6-(dimethylamino)pyridazin-3-yl]-  
5-methyl-1*H*-pyrazole-4-carboxylate

In the title compound,  $\text{C}_{13}\text{H}_{17}\text{N}_5\text{O}_2$ , the dihedral angle between the pyrazole and pyridazine rings is  $14.44(8)^\circ$ . The molecules are linked into a chain along the  $b$  axis by  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds.

Received 16 September 2006  
Accepted 18 September 2006

## Comment

Many pyridazine derivatives have been found to exhibit biological activity, such as insecticidal, fungicidal, herbicidal and plant-growth regulatory activities (Heinisch & Kopelent, 1992). For example, pyridate, credazine and maleic hydrazide (Kolar & Tisler, 1990) have been commercialized as herbicides. In a search for new biologically active pyridazine compounds, the title compound, (I), was synthesized and its structure is reported here.



In the molecule of (I) (Fig. 1), the pyrazole and pyridazine rings make a dihedral angle of  $14.44(8)^\circ$ . The ethyl carboxylate ( $\text{O1}/\text{O2}/\text{C11}-\text{C13}$ ) group is planar and it is almost coplanar with the attached pyrazole ring [dihedral angle  $6.29(11)^\circ$ ]. The dimethylamino substituent is twisted away from the pyridazine ring, with  $\text{C2}-\text{N1}-\text{C3}-\text{N2}$  and  $\text{C1}-\text{N1}-\text{C3}-\text{C4}$  torsion angles of  $8.5(3)$  and  $-5.1(3)^\circ$ , respectively. A weak intramolecular  $\text{C10}-\text{H10B}\cdots\text{O1}$  hydrogen bond is observed.

In the crystal structure of (I),  $\text{C4}-\text{H4}\cdots\text{O1}^i$  (symmetry code is given in Table 1) intermolecular hydrogen bonds link the molecules into chains along the  $b$  axis (Fig. 2).

## Experimental

6-Chloro-3-hydrazinopyridazine (10 mmol) and 3-[(dimethylamino)methylene]hexane-2,4-dione (11 mmol) were mixed in *tert*-butanol (10 mmol) and refluxed for 3 h. The solvent was then evaporated *in vacuo*. The residue was purified by chromatography on silica gel with petroleum ether and ethyl acetate (6:1) to afford compound (I). Single crystals of (I) suitable for X-ray measurements were obtained by recrystallization from a solution in petroleum ether–ethyl acetate (6:1 *v/v*).

## Crystal data

$C_{13}H_{17}N_5O_2$   
 $M_r = 275.32$   
 Monoclinic,  $P2_1/c$   
 $a = 9.870$  (2) Å  
 $b = 10.650$  (2) Å  
 $c = 13.896$  (3) Å  
 $\beta = 107.044$  (4)°  
 $V = 1396.6$  (5) Å<sup>3</sup>

$Z = 4$   
 $D_x = 1.309$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 $\mu = 0.09$  mm<sup>-1</sup>  
 $T = 294$  (2) K  
 Prism, colourless  
 $0.36 \times 0.28 \times 0.20$  mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.969$ ,  $T_{\max} = 0.982$

7702 measured reflections  
 2846 independent reflections  
 1493 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.044$   
 $\theta_{\text{max}} = 26.4^\circ$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.047$   
 $wR(F^2) = 0.134$   
 $S = 0.99$   
 2846 reflections  
 186 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.087P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.17$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>  
 Extinction correction: SHELXL97 (Sheldrick, 1997)  
 Extinction coefficient: 0.0052 (12)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C4-H4\cdots O1^i$	0.93	2.56	3.447 (3)	160
$C10-H10B\cdots O1$	0.96	2.46	3.136 (3)	127

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

All H atoms were placed in calculated positions, with  $C-H = 0.93-0.97$  Å, and included in the final cycles of refinement using a riding model, with  $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$  or  $1.5U_{\text{eq}}(\text{methyl } C)$ . A rotating group model was used for the methyl groups.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve

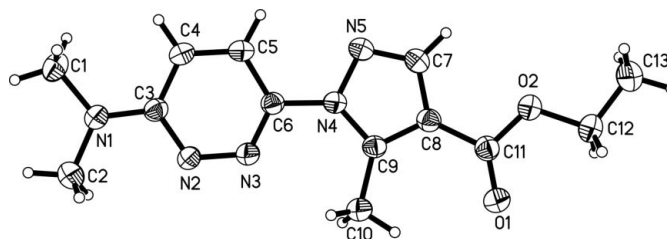


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids.

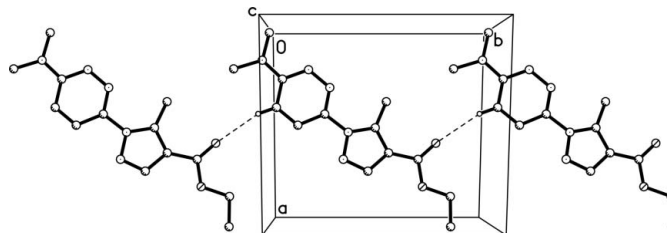


Figure 2

A view of a hydrogen-bonded (dashed lines) chain in (I). H atoms not involved in hydrogen bonding have been omitted.

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: SHELXL97.

## References

- Bruker (1999). SMART (Version 5.618), SAINT (Version 6.45) and SHELXTL (Version 6.1). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Heinisch, G. & Kopelent, H. (1992). *Prog. Med. Chem.* **29**, 141–183.  
 Kolar, P. & Tisler, M. (1990). *Adv. Heterocycl. Chem.* **75**, 167–241.  
 Sheldrick, G. M. (1996). SADABS. Version 4.202. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.