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Ethyl 5-[(4,6-dimethoxypyrimidin-2-yl)ureidosulfonyl]-1-methyl-1*H*-pyrazole-4-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.159
Data-to-parameter ratio = 13.9

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

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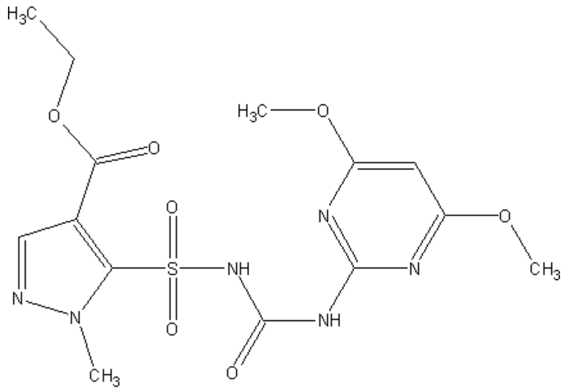
The title compound (also known as pyrazosulfuron-ethyl), $C_{14}H_{18}N_6O_7S$, is a potent agrochemical exhibiting herbicidal activity. There are intramolecular N—H···N and C—H···O hydrogen bonds. Molecules are linked *via* cyclic hydrogen bonds, forming zigzag chains along the *b* axis.

Comment

An important aspect in the rational design of bioactive molecules involves relating chemical structure to biological activity (Lewis *et al.*, 1991). The conformation of the molecule is found to influence the levels of biological activity. Correlation of the results obtained from X-ray crystallography with biological activity has aided in the chemical design of few active agrochemicals. The activity of a series of triazolyl ketone herbicides (Anderson *et al.*, 1983) has been investigated along with the fungicidal activities of *N*-phenylsuccinamides (Zenei *et al.*, 1988). Pyrazosulfuron-ethyl, (I), is a chemical exhibiting herbicidal activity and these herbicides interfere with biochemical processes that are involved in trapping light energy.



(I)

There is an intramolecular N1—H1N···N5 hydrogen bond (Fig. 1 and Table 2), forming a pseudo-six-membered ring [Etter symbol *S*(6); Bernstein *et al.*, 1995]. This interaction essentially locks the molecular conformation. Furthermore, an intramolecular C—H···O interaction is also present, forming a 14-membered ring [Etter symbol *S*(14)].

The molecules are held together by N—H···N and C—H···O hydrogen bonds, forming zigzag chains along the *b* axis (Fig. 2). The Etter symbol for the hetero-dimeric motif of the cyclic hydrogen bond is $R_2^2(7)$.

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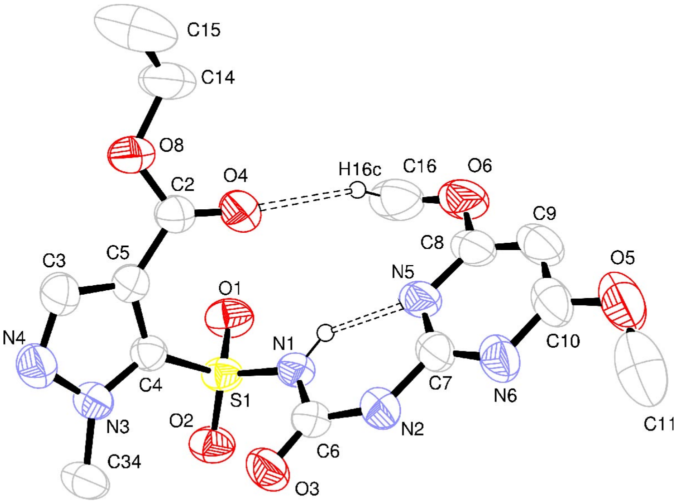

Figure 1
The molecular structure of (I), showing 50% probability ellipsoids. H atoms have been omitted for clarity, except for those involved in the hydrogen bonds (broken lines).

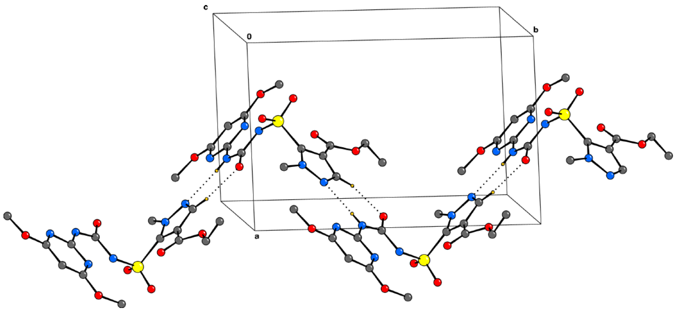

Figure 2
Packing diagram of (I), showing the intermolecular hydrogen bonds (dashed lines).

Experimental

Compound (I) was obtained from Rallis India, Bangalore. Single crystals were grown by slow evaporation of an acetone solution at 278 K.

Crystal data

$C_{14}H_{18}N_6O_7S$
$M_r = 414.41$
Monoclinic, $P2_1/c$
$a = 9.263$ (3) Å
$b = 14.038$ (4) Å
$c = 15.137$ (5) Å
$\beta = 105.294$ (5)°
$V = 1898.4$ (10) Å^3
$Z = 4$

$D_x = 1.450$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 845 reflections
$\theta = 1.6$–25.4°
$\mu = 0.22$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.33 × 0.18 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.925$, $T_{\max} = 0.974$
14374 measured reflections

3797 independent reflections
3155 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 26.4°$
$h = -11 \rightarrow 11$
$k = -17 \rightarrow 16$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.159$
$S = 0.84$
3797 reflections
273 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1226P)^2 + 0.8592P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C6	1.379 (3)	N4—C3	1.325 (3)
N2—C6	1.377 (3)	N5—C7	1.333 (3)
N2—C7	1.393 (3)	N6—C7	1.331 (3)
N3—C4	1.348 (3)		
N1—S1—C4	104.77 (9)		
N4—N3—C4—C5	0.0 (2)	N1—C6—N2—C7	4.0 (4)
C3—C5—C4—N3	−0.2 (2)	C4—N3—N4—C3	0.1 (3)
N1—S1—C4—C5	72.5 (2)	N3—N4—C3—C5	−0.3 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···N5	0.88 (3)	1.94 (3)	2.639 (3)	134 (2)
N2—H2N···N4$^{\rm i}$	0.82 (3)	2.34 (3)	3.141 (3)	168 (3)
C3—H3···O3$^{\rm ii}$	0.94 (3)	2.32 (3)	3.071 (3)	138 (2)
C16—H16C···O4	0.96	2.48	3.388 (4)	158

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

The amino H atoms and ring H atoms (H3 and H9) were located in difference Fourier maps and refined isotropically, the N—H and C—H bond distances being 0.82 (3)–0.88 (3) and 0.93 (3) Å, respectively. The methyl H atoms were constrained to an ideal geometry, with C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$, but were allowed to rotate freely about the C—C bond. The methylene H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H = 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

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