Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Ethyl 5-(3,5-dimethyl-1*H*-pyrazol-1-ylsulfonyl)-1-methyl-1*H*-pyrazole-4-carboxylate

Xiao-Mao Zou, Huan-Le Shi, Li-Min Yu, Hua-Zheng Yang,* Jiang Pei and Pei Liu

State Key Laboratory of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail:
shihuanle@mail.nankai.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.124
Data-to-parameter ratio = 16.2

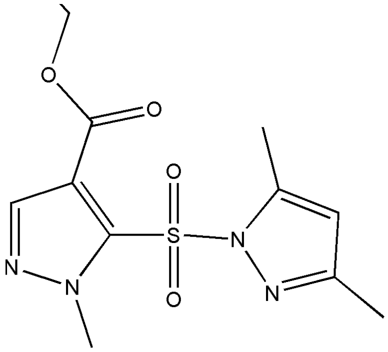
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 September 2004
Accepted 13 September 2004
Online 18 September 2004

The title compound, $C_{12}H_{16}N_4O_4S$, is composed of two substituted pyrazole rings, which are connected through a sulfone group. The dihedral angle between the two pyrazole rings is 79.1 (1)°. The ethoxycarbonyl substituent is planar and is slightly twisted away from the attached pyrazole ring.

Comment

Many pyrazolesulfonylureas are excellent herbicides (Okajima *et al.*, 1992; Suzuki, 1994; Yamamoto, 1994). Mostly, they are one of the inhibitors of acetolactate synthase (ALS) (Hidharm, 1991; Schloss *et al.*, 1998; Abell *et al.*, 1995). Triazinylsulfonyl and triazolylsulfonylpyrazoles also show good herbicidal activities (Ishikawa, 1993, 1994). They are composed of two heterocycles and a urea bridge. We have designed and synthesized a series of pyrazolylsulfonylpyrazoles to find some lead compounds with good biological activity. In the process of synthesis, we obtained the title compound, (I).



(I)

In (I), the dihedral angle between the two pyrazole rings which are linked through the sulfone group is 79.1 (1)° (Fig. 1). The ethoxycarbonyl substituent at atom C7 is nearly planar, with the r.m.s deviation of fitted atoms being 0.042 Å; the dihedral angle between this plane and the N3-pyrazole plane is 14.7 (2)°. The crystal structure is stabilized by van der Waals forces.

Experimental

The title compound was prepared by refluxing 4-ethoxycarbonyl-1-methylpyrazole-5-sulfonohydrazide (2 mmol, 0.49 g) and pentane-2,4-dione (2 mmol, 0.20 g) in tetrahydrofuran (20 ml) for about 8 h. After cooling, the mixture was evaporated on a rotary evaporator to remove the tetrahydrofuran and crystallized from a mixture of $CHCl_3$/n-C_6H_{14} (1:3).

Crystal data

$C_{12}H_{16}N_4O_4S$
$M_r = 312.35$
Monoclinic, $P2_1/n$
$a = 7.604$ (3) Å
$b = 12.868$ (5) Å
$c = 15.732$ (6) Å
$\beta = 93.765$ (7)°
$V = 1536.0$ (10) Å^3
$Z = 4$

$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1024 reflections
$\theta = 2.6$–23.9°
$\mu = 0.23$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.40 × 0.30 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.800$, $T_{\max} = 0.944$
8642 measured reflections

3156 independent reflections
1929 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.050$
$\theta_{\max} = 26.4°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 13$
$l = -19 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.124$
$S = 1.01$
3156 reflections
195 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 0.0685P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.116 (5)

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4135 (19)	N3—C6	1.353 (3)
S1—O2	1.4171 (18)	N3—C9	1.469 (3)
S1—N1	1.658 (2)	N4—C8	1.323 (3)
S1—C6	1.753 (2)	C1—C2	1.351 (4)
O3—C10	1.195 (3)	C1—C4	1.487 (4)
O4—C10	1.332 (3)	C2—C3	1.402 (4)
O4—C11	1.453 (3)	C3—C5	1.500 (5)
N1—C1	1.378 (3)	C6—C7	1.385 (3)
N1—N2	1.382 (3)	C7—C8	1.392 (3)
N2—C3	1.314 (3)	C7—C10	1.474 (3)
N3—N4	1.347 (3)	C11—C12	1.477 (5)
O1—S1—O2	120.17 (11)	N1—C1—C4	124.6 (3)
O1—S1—N1	107.25 (11)	C1—C2—C3	108.5 (3)
O2—S1—N1	108.14 (11)	N2—C3—C2	110.9 (3)
O1—S1—C6	109.90 (11)	N2—C3—C5	119.2 (3)
O2—S1—C6	107.74 (11)	C2—C3—C5	130.0 (3)
N1—S1—C6	102.16 (10)	N3—C6—C7	107.3 (2)
C10—O4—C11	116.5 (2)	N3—C6—S1	122.59 (18)
C1—N1—N2	112.8 (2)	C7—C6—S1	129.90 (18)
C1—N1—S1	132.5 (2)	C6—C7—C8	104.0 (2)
N2—N1—S1	114.45 (15)	C6—C7—C10	129.6 (2)
C3—N2—N1	104.0 (2)	C8—C7—C10	126.2 (2)
N4—N3—C6	111.2 (2)	N4—C8—C7	112.1 (2)
N4—N3—C9	117.5 (2)	O3—C10—O4	124.4 (3)
C6—N3—C9	131.2 (2)	O3—C10—C7	125.8 (3)
C8—N4—N3	105.4 (2)	O4—C10—C7	109.7 (2)
C2—C1—N1	103.8 (3)	O4—C11—C12	108.1 (3)
C2—C1—C4	131.6 (3)		

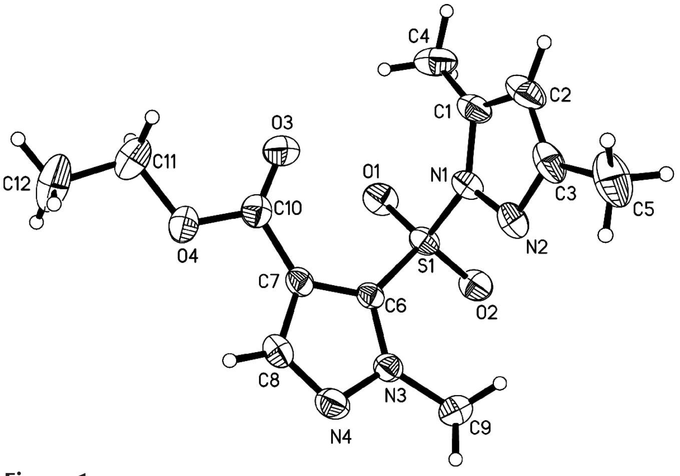


Figure 1
Displacement ellipsoid (40% probability) plot of (I), showing the atom-numbering scheme.

H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.97 Å, and refined using a riding model, with $U_{\rm iso}$(H) = 1.2 or 1.5(methyl) times $U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXL*97.

The project was supported by the Natural Science Foundation of Shandong Province (grant No. Y2003B01).

References

Abell, L. M., Hanna, W. S., Kunitsky, K. J. & Kerschen, J. A. (1995). *Pestic. Sci.* **44**, 89–92.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hidharm, M. A. (1991). *Weed Sci.* **39**, 428–434.
Ishikawa, H. (1993). Japan Patent No. 05 194 494.
Ishikawa, H. (1994). Japan Patent No. 06 116 263.
Okajima, N., Masamoto, K. & Uekodo, T. (1992). Japan Patent No. 0311065.
Schloss, J. V., Ciskanik, L. M. & Van, D. E. (1998). *Nature (London)*, **331**, 360–363.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Suzuki, F. (1994). Japan Patent No. 06 56 792.
Yamamoto, S. (1994). Japan Patent. No. 06 56 793.

Acta Cryst. (2004). E**60**, o1812–o1813 Xiao-Mao Zou *et al.* • $C_{12}H_{16}N_4O_4S$ **o1813**