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4-(2-Chlorobenzylidenehydrazinocarbonyl)-2-methyl-2*H*-pyrazole-3-sulfonamide

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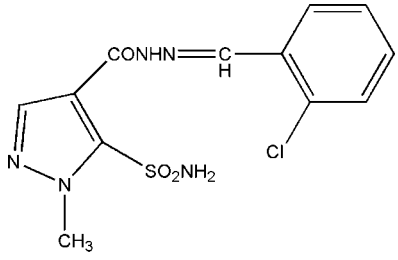
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.136; data-to-parameter ratio = 13.6.

In the title compound, $C_{12}H_{12}ClN_5O_3S$, the pyrazole ring and the benzene ring are approximately coplanar. The mean plane of the pyrazole ring makes a dihedral angle of 12.82 (13)° with the mean plane of the benzene ring. The crystal packing is stabilized by intramolecular N—H···O and intermolecular N—H···O and N—H···N hydrogen bonds.

Related literature

For biological and pharmaceutical activity of pyrazoles, see: Liang & He (2005); Wang *et al.* (2004). For the preparation of derivatives of pyrazoles, see: Zou *et al.* (2005). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{12}H_{12}ClN_5O_3S$
M_r = 341.78
Triclinic, $P\overline{1}$
a = 8.4272 (6) Å
b = 9.0905 (7) Å
c = 10.4381 (7) Å
α = 89.525 (1)°
β = 66.852 (1)°
γ = 87.722 (1)°
V = 734.66 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 292 (2) K
0.10 × 0.10 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
4684 measured reflections
2843 independent reflections
2421 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.136
S = 1.11
2843 reflections
209 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1	0.87 (3)	1.83 (3)	2.685 (3)	166 (3)
N5—H5B···O3^{i}	0.82 (3)	2.05 (3)	2.835 (3)	160 (3)
N5—H5A···N4^{ii}	0.77 (3)	2.42 (3)	3.105 (3)	149 (3)
N5—H5A···O3^{ii}	0.77 (3)	2.58 (3)	3.231 (3)	144 (3)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2001).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2353).

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supplementary materials

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4-(2-Chlorobenzylidenehydrazinocarbonyl)-2-methyl-2*H*-pyrazole-3-sulfonamide

J.-J. Wei, W.-D. Wang, W.-Y. Li and H.-W. He

Comment

Pyrazole derivatives are important compounds with versatile industrial and medical applications (Liang & He, 2005; Wang *et al.*, 2004). We report here the molecular structure of (I). In the title compound, all bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and the molecules are stabilized by intra and intermolecular hydrogen bonds (Table 1). Short intermolecular distances between the centroids, *Cg*1, of two adjacent pyrazole ring suggest π— π stacking interactions [$Cg1\cdots Cg1^{iii}$ = 3.4664 (13) Å, iii = 2 − *x*, 2 − *y*, −*z*].

Experimental

In a 50 mL flask provided with magnetic stirrer were placed 4-hydrazinocarbonyl-2-methyl-2*H*-pyrazole-3-sulfonic acid amide (1.10 g, 5 mmol), 2-chloro-benzaldehyde (0.84 g, 6 mmol) and anhydrous ethanol (10 mL). The reaction mixture was heated to reflux for 3 h, allowed to cool to room temperature. The white solid was filtered and recrystallized from a dimethylformamide-ethanol mixture to give the title compound (yield 94%). Colourless crystals of (I) suitable for X-ray structure analysis were grown from the mixture of tetrahydrofuran and petroleum ether (*v*/*v*, 1:6).

Refinement

The H atom bound to N3 was found in a difference Fourier map and its coordinates were refined with U_{iso}(H) = 1.2U_{eq}(N). All other H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso}=1.2U_{eq} (C) for aromatic and 0.96 Å, U_{iso} = 1.5U_{eq} (C) for CH_3 atoms

Figures

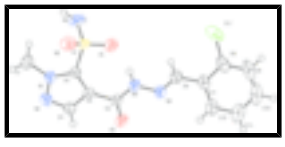

Fig. 1. The structure of (I). showing 50% probability displacement ellipsoids and the atom-numbering scheme.

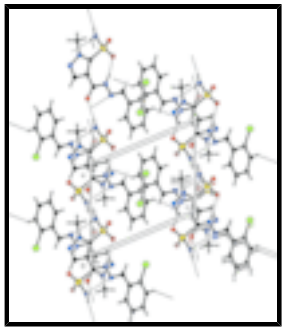

Fig. 2. The crystal packing of (I) with hydrogen bonds drawn as dashed lines.

4-(2-Chlorobenzylidenehydrazinocarbonyl)-2-methyl-2*H*-pyrazole-3-sulfonic acid amide

Crystal data

$C_{12}H_{12}ClN_5O_3S$	$Z = 2$
$M_r = 341.78$	$F_{000} = 352$
Triclinic, $P\overline{1}$	$D_x = 1.545$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.4272$ (6) Å	Cell parameters from 2721 reflections
$b = 9.0905$ (7) Å	$\theta = 2.2$–28.3º
$c = 10.4381$ (7) Å	$\mu = 0.42$ mm^{-1}
$\alpha = 89.525$ (1)º	$T = 292$ (2) K
$\beta = 66.852$ (1)º	Block, colorless
$\gamma = 87.722$ (1)º	0.10 × 0.10 × 0.08 mm
$V = 734.66$ (9) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2421 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.051$
Monochromator: graphite	$\theta_{max} = 26.0$º
$T = 297(2)$ K	$\theta_{min} = 2.1$º
φ and ω scans	$h = -10\rightarrow9$
Absorption correction: None	$k = -11\rightarrow11$
4684 measured reflections	$l = -12\rightarrow8$
2843 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.043$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.136$	$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.1888P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.011$
2843 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
209 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.4090 (3)	0.9541 (3)	−0.1318 (3)	0.0513 (6)
H1A	1.4523	1.0053	−0.2188	0.077*
H1B	1.4896	0.8758	−0.1332	0.077*
H1C	1.3944	1.0214	−0.0572	0.077*
C2	1.1438 (3)	0.8013 (2)	−0.0105 (2)	0.0305 (4)
C3	0.9973 (3)	0.7766 (2)	−0.0376 (2)	0.0324 (5)
C4	1.0236 (3)	0.8610 (3)	−0.1570 (2)	0.0403 (5)
H4	0.9460	0.8673	−0.2004	0.048*
C5	0.8395 (3)	0.6899 (2)	0.0232 (2)	0.0356 (5)
C6	0.6552 (3)	0.4574 (3)	0.3040 (2)	0.0414 (5)
H6	0.7386	0.4629	0.3411	0.050*
C7	0.5040 (3)	0.3680 (3)	0.3721 (2)	0.0425 (5)
C8	0.4960 (4)	0.2636 (3)	0.4734 (3)	0.0520 (7)
C9	0.3514 (5)	0.1801 (3)	0.5337 (3)	0.0724 (10)
H9	0.3483	0.1104	0.6003	0.087*
C10	0.2140 (5)	0.1986 (4)	0.4970 (4)	0.0782 (11)
H10	0.1185	0.1408	0.5373	0.094*
C11	0.2164 (4)	0.3036 (4)	0.3995 (3)	0.0703 (9)
H11	0.1214	0.3182	0.3760	0.084*
C12	0.3606 (3)	0.3868 (3)	0.3372 (3)	0.0521 (6)
H12	0.3620	0.4565	0.2710	0.062*
Cl1	0.66414 (13)	0.23836 (9)	0.52817 (8)	0.0729 (3)
N1	1.2424 (2)	0.8929 (2)	−0.11050 (19)	0.0377 (4)
N2	1.1702 (3)	0.9306 (2)	−0.2012 (2)	0.0451 (5)
N3	0.8156 (3)	0.6116 (2)	0.1381 (2)	0.0413 (5)
H3A	0.894 (4)	0.615 (3)	0.174 (3)	0.050*
N4	0.6715 (2)	0.5276 (2)	0.1936 (2)	0.0393 (4)
N5	1.3822 (3)	0.6394 (3)	0.0437 (3)	0.0551 (6)
H5A	1.371 (4)	0.572 (4)	0.004 (3)	0.066*
H5B	1.475 (4)	0.675 (4)	0.028 (3)	0.066*
O1	1.0830 (2)	0.6507 (2)	0.2113 (2)	0.0637 (6)
O2	1.2641 (2)	0.8658 (2)	0.17484 (19)	0.0550 (5)
O3	0.7372 (2)	0.6923 (2)	−0.03414 (19)	0.0569 (5)

S1	1.21556 (7)	0.74039 (6)	0.12003 (6)	0.0396 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0446 (14)	0.0509 (14)	0.0628 (16)	−0.0168 (11)	−0.0246 (12)	0.0050 (12)
C2	0.0267 (10)	0.0327 (10)	0.0325 (10)	0.0012 (8)	−0.0121 (8)	0.0012 (8)
C3	0.0299 (10)	0.0334 (11)	0.0344 (11)	0.0002 (8)	−0.0134 (9)	0.0011 (9)
C4	0.0391 (12)	0.0452 (13)	0.0420 (12)	−0.0045 (9)	−0.0216 (10)	0.0079 (10)
C5	0.0274 (10)	0.0409 (12)	0.0407 (12)	−0.0031 (9)	−0.0156 (9)	0.0033 (9)
C6	0.0435 (13)	0.0424 (12)	0.0401 (12)	−0.0063 (10)	−0.0180 (10)	0.0042 (10)
C7	0.0501 (14)	0.0370 (12)	0.0351 (12)	−0.0075 (10)	−0.0106 (10)	0.0022 (9)
C8	0.0724 (18)	0.0412 (13)	0.0387 (13)	−0.0043 (12)	−0.0176 (12)	0.0018 (11)
C9	0.104 (3)	0.0474 (16)	0.0473 (16)	−0.0239 (17)	−0.0087 (17)	0.0134 (13)
C10	0.076 (2)	0.069 (2)	0.068 (2)	−0.0373 (18)	−0.0023 (18)	0.0075 (16)
C11	0.0557 (17)	0.077 (2)	0.070 (2)	−0.0231 (15)	−0.0139 (15)	0.0018 (16)
C12	0.0501 (15)	0.0539 (15)	0.0490 (15)	−0.0129 (12)	−0.0151 (12)	0.0061 (12)
Cl1	0.1105 (7)	0.0599 (5)	0.0602 (5)	0.0084 (4)	−0.0475 (5)	0.0067 (3)
N1	0.0360 (10)	0.0388 (10)	0.0397 (10)	−0.0068 (8)	−0.0161 (8)	0.0026 (8)
N2	0.0490 (11)	0.0476 (11)	0.0429 (11)	−0.0113 (9)	−0.0219 (9)	0.0111 (9)
N3	0.0345 (10)	0.0506 (11)	0.0438 (11)	−0.0116 (8)	−0.0200 (9)	0.0111 (9)
N4	0.0362 (10)	0.0415 (10)	0.0409 (10)	−0.0093 (8)	−0.0153 (8)	0.0044 (8)
N5	0.0396 (12)	0.0471 (13)	0.0931 (19)	0.0038 (10)	−0.0418 (13)	−0.0099 (12)
O1	0.0450 (10)	0.0969 (15)	0.0610 (12)	−0.0200 (10)	−0.0326 (9)	0.0362 (11)
O2	0.0611 (11)	0.0611 (11)	0.0556 (11)	0.0016 (9)	−0.0368 (9)	−0.0085 (9)
O3	0.0403 (9)	0.0845 (14)	0.0577 (11)	−0.0222 (9)	−0.0305 (9)	0.0270 (10)
S1	0.0339 (3)	0.0472 (4)	0.0441 (4)	−0.0034 (2)	−0.0224 (3)	0.0067 (3)

Geometric parameters (Å, °)

C1—N1	1.465 (3)	C8—C9	1.386 (4)
C1—H1A	0.9600	C8—Cl1	1.731 (3)
C1—H1B	0.9600	C9—C10	1.360 (5)
C1—H1C	0.9600	C9—H9	0.9300
C2—N1	1.353 (3)	C10—C11	1.384 (5)
C2—C3	1.397 (3)	C10—H10	0.9300
C2—S1	1.772 (2)	C11—C12	1.384 (4)
C3—C4	1.404 (3)	C11—H11	0.9300
C3—C5	1.484 (3)	C12—H12	0.9300
C4—N2	1.323 (3)	N1—N2	1.347 (3)
C4—H4	0.9300	N3—N4	1.381 (3)
C5—O3	1.227 (3)	N3—H3A	0.87 (3)
C5—N3	1.340 (3)	N5—S1	1.575 (2)
C6—N4	1.274 (3)	N5—H5A	0.77 (3)
C6—C7	1.466 (3)	N5—H5B	0.82 (3)
C6—H6	0.9300	O1—S1	1.4292 (19)
C7—C12	1.397 (4)	O2—S1	1.4235 (18)
C7—C8	1.398 (3)		

N1—C1—H1A	109.5	C10—C9—H9	119.5
N1—C1—H1B	109.5	C8—C9—H9	119.5
H1A—C1—H1B	109.5	C9—C10—C11	119.9 (3)
N1—C1—H1C	109.5	C9—C10—H10	120.0
H1A—C1—H1C	109.5	C11—C10—H10	120.0
H1B—C1—H1C	109.5	C12—C11—C10	119.8 (3)
N1—C2—C3	106.98 (18)	C12—C11—H11	120.1
N1—C2—S1	119.13 (15)	C10—C11—H11	120.1
C3—C2—S1	133.89 (16)	C11—C12—C7	121.1 (3)
C2—C3—C4	103.20 (18)	C11—C12—H12	119.5
C2—C3—C5	137.5 (2)	C7—C12—H12	119.5
C4—C3—C5	119.3 (2)	N2—N1—C2	112.22 (17)
N2—C4—C3	112.9 (2)	N2—N1—C1	117.85 (19)
N2—C4—H4	123.6	C2—N1—C1	129.9 (2)
C3—C4—H4	123.6	C4—N2—N1	104.69 (18)
O3—C5—N3	122.8 (2)	C5—N3—N4	118.87 (19)
O3—C5—C3	118.8 (2)	C5—N3—H3A	117.9 (18)
N3—C5—C3	118.38 (19)	N4—N3—H3A	123.2 (18)
N4—C6—C7	119.6 (2)	C6—N4—N3	115.7 (2)
N4—C6—H6	120.2	S1—N5—H5A	116 (2)
C7—C6—H6	120.2	S1—N5—H5B	117 (2)
C12—C7—C8	117.7 (2)	H5A—N5—H5B	124 (3)
C12—C7—C6	119.9 (2)	O2—S1—O1	119.36 (12)
C8—C7—C6	122.4 (3)	O2—S1—N5	108.30 (12)
C9—C8—C7	120.5 (3)	O1—S1—N5	108.40 (14)
C9—C8—Cl1	118.7 (2)	O2—S1—C2	107.99 (10)
C7—C8—Cl1	120.9 (2)	O1—S1—C2	105.91 (10)
C10—C9—C8	120.9 (3)	N5—S1—C2	106.13 (12)
N1—C2—C3—C4	−0.4 (2)	C10—C11—C12—C7	−0.7 (5)
S1—C2—C3—C4	179.24 (18)	C8—C7—C12—C11	−0.9 (4)
N1—C2—C3—C5	178.8 (2)	C6—C7—C12—C11	179.8 (2)
S1—C2—C3—C5	−1.6 (4)	C3—C2—N1—N2	0.3 (2)
C2—C3—C4—N2	0.3 (3)	S1—C2—N1—N2	−179.38 (15)
C5—C3—C4—N2	−178.99 (19)	C3—C2—N1—C1	180.0 (2)
C2—C3—C5—O3	−177.8 (2)	S1—C2—N1—C1	0.3 (3)
C4—C3—C5—O3	1.2 (3)	C3—C4—N2—N1	−0.2 (3)
C2—C3—C5—N3	1.8 (4)	C2—N1—N2—C4	−0.1 (3)
C4—C3—C5—N3	−179.2 (2)	C1—N1—N2—C4	−179.8 (2)
N4—C6—C7—C12	−15.3 (3)	O3—C5—N3—N4	1.2 (4)
N4—C6—C7—C8	165.4 (2)	C3—C5—N3—N4	−178.40 (19)
C12—C7—C8—C9	1.5 (4)	C7—C6—N4—N3	178.6 (2)
C6—C7—C8—C9	−179.2 (2)	C5—N3—N4—C6	−179.1 (2)
C12—C7—C8—Cl1	−177.30 (19)	N1—C2—S1—O2	49.18 (19)
C6—C7—C8—Cl1	2.0 (3)	C3—C2—S1—O2	−130.4 (2)
C7—C8—C9—C10	−0.6 (4)	N1—C2—S1—O1	178.13 (18)
Cl1—C8—C9—C10	178.2 (3)	C3—C2—S1—O1	−1.4 (3)
C8—C9—C10—C11	−1.0 (5)	N1—C2—S1—N5	−66.77 (19)
C9—C10—C11—C12	1.6 (5)	C3—C2—S1—N5	113.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···O1	0.87 (3)	1.83 (3)	2.685 (3)	166 (3)
N5—H5B···O3^{i}	0.82 (3)	2.05 (3)	2.835 (3)	160 (3)
N5—H5A···N4^{ii}	0.77 (3)	2.42 (3)	3.105 (3)	149 (3)
N5—H5A···O3^{ii}	0.77 (3)	2.58 (3)	3.231 (3)	144 (3)

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+2, −*y*+1, −*z*.

Fig. 1

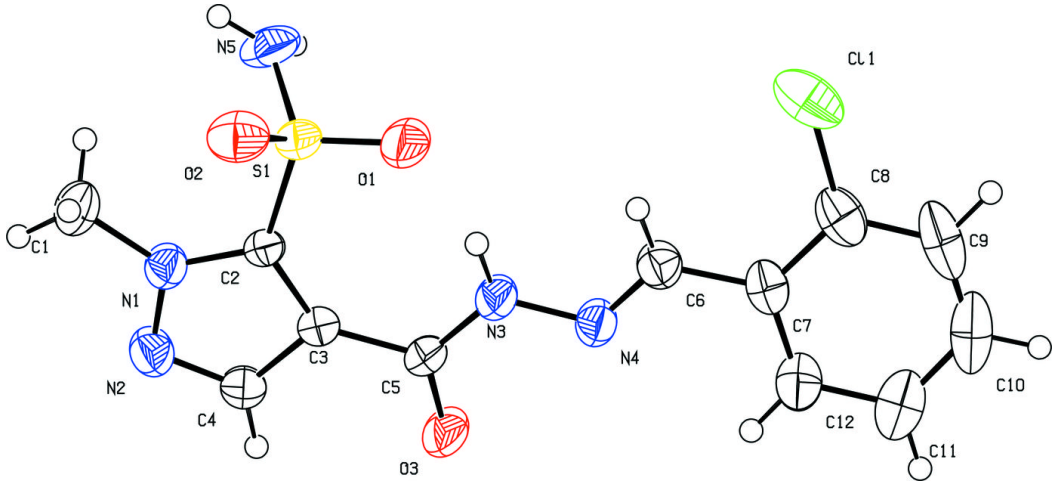

Fig. 2