organic compounds

4-(3,7-Dimethyl-4-oxo-4,5-dihydro-isoxazolo[4,5-*d*]pyridazin-5-yl)benzene-sulfonamide

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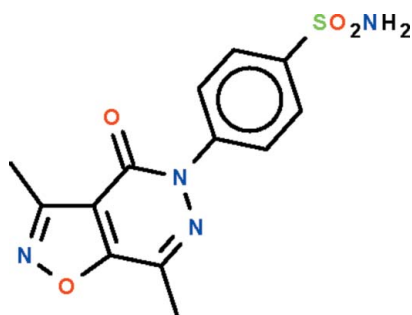Correspondence e-mail: seikweng@um.edu.my

The nine-membered fused-ring system of the title pyridazine derivative, $C_{13}H_{12}N_4O_4S$, is approximately planar (r.m.s. deviation 0.027 Å), and the benzene ring of the phenyl-sulfamide substituent is aligned at 43.5 (1)° to the fused-ring system. The amine group of the sulfonamide substituent forms an N—H···O hydrogen bond to the ketonic O atom of two neigboring molecules to generate a chain running along the *c* axis.

Related literature

For a related structure, see: Abdel-Aziz *et al.* (2010). For the biological activity of the class of pyridazines, see: Faid-Allah *et al.* (2011); Makki & Faid-Allah (1996).

Experimental

Crystal data

$C_{13}H_{12}N_4O_4S$	V = 5305.1 (2) Å^3
M_r = 320.33	Z = 16
Orthorhombic, $Fdd2$	Cu $K\alpha$ radiation
a = 18.0113 (4) Å	μ = 2.43 mm^{-1}
b = 35.5302 (11) Å	T = 100 K
c = 8.2900 (2) Å	$0.30 \times 0.20 \times 0.05$ mm

Data collection

Agilent Technologies SuperNova Dual diffractometer with Atlas detector	7699 measured reflections
	1886 independent reflections
	1870 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010) T_{min} = 0.529, T_{max} = 0.888	R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.096	
S = 1.08	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
1886 reflections	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
207 parameters	Absolute structure: Flack (1983), 441 Friedel pairs
1 restraint	Flack parameter: 0.026 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1···O2^i	0.95 (3)	2.09 (4)	3.012 (3)	163 (3)
N4—H2···O2^{ii}	0.85 (5)	2.11 (5)	2.933 (3)	162 (4)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

References

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supplementary materials

4-(3,7-Dimethyl-4-oxo-4,5-dihydroisoxazolo[4,5-*d*]pyridazin-5-yl)benzenesulfonamide

A. M. Asiri, H. M. Faidallah, A. O. Al-Youbi, A. Y. Obaid and **S. W. Ng**

Comment

We have reported the synthesis of some pyridazines, which exhibit biological activity (Faid-Allah *et al.*, 2011; Makki & Faid-Allah, 1996). There are few crystal structure reports of such systems; recently, we reported the crystal structure of 3-methyl-2-(4-methyl)-2*H*-pyrazolo[3,4-*d*]pyridazin-5-ium thiocyanate, a salt (Abdel-Aziz *et al.*, 2010).

The nine-membered fused-ring system of $C_{13}H_{12}N_4O_4S$ (Scheme I), is planar and the benzene ring of the phenylsufamido substitutent is aligned at 43.5 (1) ° (Fig. 1). The amino group the substitutent forms a hydrogen bond to the ketonic O atom of two neigboring molecules to generate a chain running along the *c*-axis of the orthorhombic unit cell (Table 1).

Experimental

A solution of ethyl 5-acetyl-3-methylisoxazole-4-carboxylate (0.39 g, 0.002 mol) in ethanol (25 ml) was refluxed with *p*-sulfonamidophenyl hydrazine hydrochloride (0.49 g, 0.002 mol) for 2 h. The pyridazine which separated after concentration of the reaction mixture was filtered off, washed with ethanol and recrystallized from the same solvent to give long thin prisms in 90% yield, m.p. 488 K.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, $U_{iso}(H)$ 1.2 to $1.5U_{eq}(C)$] and were included in the refinement in the riding model approximation.

The amino H-atoms were located in a difference Fourier map, and were refined freely.

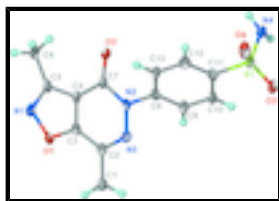The Flack (Flack, 1983) parameter was refined from 441 Friedel pairs.

Figures

Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{13}H_{12}N_4O_4S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-(3,7-Dimethyl-4-oxo-4,5- dihydroisoxazolo[4,5-*d*]pyridazin-5-yl)benzenesulfonamide

Crystal data

$C_{13}H_{12}N_4O_4S$	$F(000) = 2656$
$M_r = 320.33$	$D_x = 1.604$ Mg m^{-3}
Orthorhombic, *Fdd*2	Cu *K*α radiation, λ = 1.54184 Å
Hall symbol: F 2 -2d	Cell parameters from 5872 reflections
$a = 18.0113$ (4) Å	θ = 4.9–74.4°
$b = 35.5302$ (11) Å	μ = 2.43 mm^{-1}
$c = 8.2900$ (2) Å	$T = 100$ K
$V = 5305.1$ (2) Å^3	Plate, colorless
$Z = 16$	0.30 × 0.20 × 0.05 mm

Data collection

Agilent Technologies SuperNova Dual diffractometer with Atlas detector	1886 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	1870 reflections with $I > 2\sigma(I)$
Mirror	$R_{int} = 0.032$
Detector resolution: 10.4041 pixels mm^{-1}	$\theta_{max} = 74.5°$, $\theta_{min} = 5.0°$
ω scan	$h = -42 \rightarrow 44$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	$k = -22 \rightarrow 22$
$T_{min} = 0.529$, $T_{max} = 0.888$	$l = -10 \rightarrow 7$
7699 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 4.3892P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
1886 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
207 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
1 restraint	Absolute structure: Flack (1983), 441 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.026 (18)

Special details

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

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

	x	y	z	U_{iso}*/U_{eq}
S1	0.12413 (3)	0.212823 (15)	0.43880 (7)	0.01769 (17)
O1	0.21513 (9)	0.37146 (4)	−0.5296 (2)	0.0193 (4)
O2	0.05983 (9)	0.28881 (4)	−0.2671 (2)	0.0188 (4)
O3	0.17529 (10)	0.22599 (5)	0.5595 (2)	0.0235 (4)
N4	0.04217 (11)	0.22369 (5)	0.5034 (3)	0.0213 (4)
N1	0.14841 (11)	0.36838 (5)	−0.6234 (3)	0.0200 (4)
N2	0.17467 (10)	0.30278 (5)	−0.1592 (3)	0.0154 (4)
N3	0.24011 (10)	0.32316 (5)	−0.1526 (3)	0.0163 (4)
O4	0.12339 (11)	0.17367 (5)	0.3966 (3)	0.0277 (5)
C1	0.32798 (13)	0.36781 (6)	−0.2608 (3)	0.0204 (5)
H1A	0.3547	0.3612	−0.1618	0.031*
H1B	0.3168	0.3948	−0.2603	0.031*
H1C	0.3589	0.3618	−0.3546	0.031*
C2	0.25679 (12)	0.34585 (6)	−0.2694 (3)	0.0170 (5)
C3	0.20621 (12)	0.34980 (6)	−0.3997 (3)	0.0169 (5)
C4	0.13880 (13)	0.33186 (6)	−0.4030 (3)	0.0156 (5)
C5	0.10469 (13)	0.34528 (6)	−0.5465 (3)	0.0177 (5)
C6	0.02971 (13)	0.33542 (7)	−0.6145 (3)	0.0213 (5)
H6A	0.0218	0.3492	−0.7153	0.032*
H6B	−0.0090	0.3423	−0.5367	0.032*
H6C	0.0275	0.3083	−0.6357	0.032*
C7	0.11926 (12)	0.30620 (6)	−0.2766 (3)	0.0155 (5)
C8	0.16360 (12)	0.28011 (6)	−0.0177 (3)	0.0159 (5)
C9	0.18337 (13)	0.29491 (6)	0.1314 (3)	0.0175 (5)
H9	0.2040	0.3195	0.1383	0.021*
C10	0.17290 (12)	0.27382 (6)	0.2696 (3)	0.0175 (5)
H10	0.1876	0.2835	0.3715	0.021*
C11	0.14063 (12)	0.23826 (6)	0.2583 (3)	0.0171 (5)
C12	0.12297 (12)	0.22293 (7)	0.1099 (4)	0.0189 (5)
H12	0.1026	0.1983	0.1037	0.023*
C13	0.13496 (12)	0.24347 (6)	−0.0300 (3)	0.0174 (5)
H13	0.1240	0.2329	−0.1325	0.021*
H1	0.0433 (17)	0.2472 (9)	0.559 (4)	0.025 (8)*
H2	0.008 (2)	0.2165 (10)	0.439 (6)	0.043 (10)*

supplementary materials

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0235 (3)	0.0179 (3)	0.0117 (3)	−0.00015 (18)	−0.0010 (2)	0.0027 (2)
O1	0.0226 (8)	0.0227 (7)	0.0124 (9)	−0.0015 (6)	0.0005 (7)	0.0035 (7)
O2	0.0198 (8)	0.0231 (7)	0.0135 (9)	−0.0028 (5)	−0.0009 (7)	0.0002 (7)
O3	0.0273 (9)	0.0289 (8)	0.0143 (10)	−0.0008 (7)	−0.0034 (8)	0.0049 (7)
N4	0.0219 (10)	0.0270 (10)	0.0150 (11)	−0.0041 (8)	−0.0011 (9)	0.0002 (9)
N1	0.0230 (9)	0.0225 (9)	0.0145 (11)	0.0025 (7)	−0.0033 (9)	0.0000 (8)
N2	0.0190 (8)	0.0174 (8)	0.0098 (10)	−0.0005 (7)	0.0018 (8)	0.0000 (8)
N3	0.0183 (9)	0.0169 (7)	0.0136 (10)	0.0005 (7)	0.0002 (8)	−0.0033 (8)
O4	0.0464 (11)	0.0194 (8)	0.0172 (11)	0.0006 (7)	−0.0003 (8)	0.0042 (8)
C1	0.0236 (10)	0.0242 (10)	0.0134 (13)	−0.0058 (8)	0.0020 (11)	−0.0016 (9)
C2	0.0208 (10)	0.0163 (8)	0.0139 (12)	0.0009 (8)	0.0016 (10)	−0.0015 (8)
C3	0.0219 (10)	0.0166 (9)	0.0123 (12)	0.0003 (8)	0.0025 (10)	−0.0015 (9)
C4	0.0201 (10)	0.0171 (9)	0.0096 (12)	0.0017 (8)	0.0007 (9)	−0.0015 (9)
C5	0.0220 (10)	0.0202 (9)	0.0107 (12)	0.0031 (8)	0.0020 (10)	0.0003 (9)
C6	0.0220 (11)	0.0291 (11)	0.0128 (12)	0.0017 (8)	−0.0028 (10)	0.0024 (10)
C7	0.0195 (10)	0.0164 (9)	0.0107 (13)	0.0032 (7)	0.0005 (9)	−0.0007 (9)
C8	0.0170 (9)	0.0176 (9)	0.0130 (13)	0.0022 (7)	0.0013 (10)	0.0015 (9)
C9	0.0220 (10)	0.0176 (10)	0.0127 (12)	−0.0001 (8)	0.0001 (10)	−0.0016 (9)
C10	0.0223 (10)	0.0195 (10)	0.0107 (11)	0.0017 (8)	0.0003 (10)	−0.0007 (9)
C11	0.0179 (9)	0.0186 (10)	0.0147 (13)	0.0027 (8)	0.0016 (10)	0.0030 (10)
C12	0.0210 (11)	0.0171 (9)	0.0186 (14)	0.0000 (8)	−0.0009 (10)	−0.0022 (10)
C13	0.0212 (10)	0.0189 (10)	0.0122 (13)	0.0004 (8)	−0.0022 (9)	−0.0023 (9)

Geometric parameters (Å, °)

S1—O4	1.4345 (19)	C2—C3	1.420 (3)
S1—O3	1.4385 (19)	C3—C4	1.372 (3)
S1—N4	1.617 (2)	C4—C5	1.422 (4)
S1—C11	1.773 (3)	C4—C7	1.433 (3)
O1—C3	1.333 (3)	C5—C6	1.505 (3)
O1—N1	1.435 (3)	C6—H6A	0.9800
O2—C7	1.238 (3)	C6—H6B	0.9800
N4—H1	0.95 (3)	C6—H6C	0.9800
N4—H2	0.85 (5)	C8—C9	1.389 (4)
N1—C5	1.304 (3)	C8—C13	1.404 (3)
N2—N3	1.384 (3)	C9—C10	1.382 (3)
N2—C7	1.399 (3)	C9—H9	0.9500
N2—C8	1.437 (3)	C10—C11	1.394 (3)
N3—C2	1.296 (3)	C10—H10	0.9500
C1—C2	1.502 (3)	C11—C12	1.383 (4)
C1—H1A	0.9800	C12—C13	1.387 (4)
C1—H1B	0.9800	C12—H12	0.9500
C1—H1C	0.9800	C13—H13	0.9500
O4—S1—O3	119.41 (11)	N1—C5—C4	111.0 (2)

O4—S1—N4	107.69 (11)	N1—C5—C6	120.4 (2)
O3—S1—N4	106.07 (12)	C4—C5—C6	128.5 (2)
O4—S1—C11	106.85 (12)	C5—C6—H6A	109.5
O3—S1—C11	108.30 (11)	C5—C6—H6B	109.5
N4—S1—C11	108.10 (11)	H6A—C6—H6B	109.5
C3—O1—N1	107.01 (16)	C5—C6—H6C	109.5
S1—N4—H1	110.4 (18)	H6A—C6—H6C	109.5
S1—N4—H2	112 (3)	H6B—C6—H6C	109.5
H1—N4—H2	125 (3)	O2—C7—N2	121.9 (2)
C5—N1—O1	106.8 (2)	O2—C7—C4	125.2 (2)
N3—N2—C7	126.1 (2)	N2—C7—C4	112.9 (2)
N3—N2—C8	112.27 (18)	C9—C8—C13	120.6 (2)
C7—N2—C8	121.16 (18)	C9—C8—N2	118.59 (18)
C2—N3—N2	119.6 (2)	C13—C8—N2	120.8 (2)
C2—C1—H1A	109.5	C10—C9—C8	119.8 (2)
C2—C1—H1B	109.5	C10—C9—H9	120.1
H1A—C1—H1B	109.5	C8—C9—H9	120.1
C2—C1—H1C	109.5	C9—C10—C11	119.5 (2)
H1A—C1—H1C	109.5	C9—C10—H10	120.2
H1B—C1—H1C	109.5	C11—C10—H10	120.2
N3—C2—C3	118.8 (2)	C12—C11—C10	120.8 (2)
N3—C2—C1	119.0 (2)	C12—C11—S1	120.77 (17)
C3—C2—C1	122.2 (2)	C10—C11—S1	118.4 (2)
O1—C3—C4	111.0 (2)	C11—C12—C13	120.0 (2)
O1—C3—C2	126.5 (2)	C11—C12—H12	120.0
C4—C3—C2	122.5 (2)	C13—C12—H12	120.0
C3—C4—C5	104.1 (2)	C12—C13—C8	119.0 (2)
C3—C4—C7	119.9 (2)	C12—C13—H13	120.5
C5—C4—C7	136.0 (2)	C8—C13—H13	120.5
C3—O1—N1—C5	−0.6 (2)	C3—C4—C7—O2	179.4 (2)
C7—N2—N3—C2	−5.6 (3)	C5—C4—C7—O2	−1.1 (4)
C8—N2—N3—C2	−177.91 (19)	C3—C4—C7—N2	0.3 (3)
N2—N3—C2—C3	1.5 (3)	C5—C4—C7—N2	179.9 (2)
N2—N3—C2—C1	179.99 (19)	N3—N2—C8—C9	38.9 (3)
N1—O1—C3—C4	1.5 (2)	C7—N2—C8—C9	−133.9 (2)
N1—O1—C3—C2	−176.5 (2)	N3—N2—C8—C13	−139.7 (2)
N3—C2—C3—O1	−179.1 (2)	C7—N2—C8—C13	47.5 (3)
C1—C2—C3—O1	2.4 (4)	C13—C8—C9—C10	−1.8 (3)
N3—C2—C3—C4	3.2 (3)	N2—C8—C9—C10	179.60 (19)
C1—C2—C3—C4	−175.3 (2)	C8—C9—C10—C11	−1.8 (3)
O1—C3—C4—C5	−1.7 (2)	C9—C10—C11—C12	3.8 (3)
C2—C3—C4—C5	176.3 (2)	C9—C10—C11—S1	−177.18 (17)
O1—C3—C4—C7	177.95 (19)	O4—S1—C11—C12	24.3 (2)
C2—C3—C4—C7	−4.0 (3)	O3—S1—C11—C12	154.11 (18)
O1—N1—C5—C4	−0.5 (3)	N4—S1—C11—C12	−91.4 (2)
O1—N1—C5—C6	−179.15 (19)	O4—S1—C11—C10	−154.73 (18)
C3—C4—C5—N1	1.3 (3)	O3—S1—C11—C10	−24.9 (2)
C7—C4—C5—N1	−178.2 (2)	N4—S1—C11—C10	89.6 (2)
C3—C4—C5—C6	179.9 (2)	C10—C11—C12—C13	−2.2 (3)

C7—C4—C5—C6	0.3 (4)	S1—C11—C12—C13	178.81 (17)
N3—N2—C7—O2	−174.6 (2)	C11—C12—C13—C8	−1.4 (3)
C8—N2—C7—O2	−2.9 (3)	C9—C8—C13—C12	3.4 (3)
N3—N2—C7—C4	4.5 (3)	N2—C8—C13—C12	−178.04 (19)
C8—N2—C7—C4	176.19 (19)		

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1···O2^i	0.95 (3)	2.09 (4)	3.012 (3)	163 (3)
N4—H2···O2^{ii}	0.85 (5)	2.11 (5)	2.933 (3)	162 (4)

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

Fig. 1