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2-Acetyl-3-methylpyrazine phenylsulfonylhydrazone

Xi-Shi Tai, Yi-Min Feng and Fan-Yuan Kong*

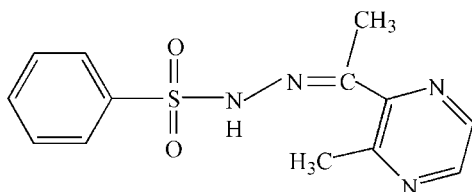
 Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China
 Correspondence e-mail: taixishi@lzu.edu.cn

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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.046; wR factor = 0.104; data-to-parameter ratio = 13.1.

In the title compound, $\text{C}_{13}\text{H}_{14}\text{N}_4\text{O}_2\text{S}$, the dihedral angle between the aromatic rings is $55.42(14)^\circ$. In the crystal structure, an $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond leads to chains of molecules along $[001]$.

Related literature

 For related literature, see: Tai *et al.* (2008).


Experimental

Crystal data

 $\text{C}_{13}\text{H}_{14}\text{N}_4\text{O}_2\text{S}$
 $M_r = 290.34$
 Monoclinic, $P2_1/c$
 $a = 10.9848(15)$ Å

 $b = 16.7921(18)$ Å
 $c = 7.4817(10)$ Å
 $\beta = 97.264(1)^\circ$
 $V = 1369.0(3)$ Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹
 $T = 298(2)$ K
 $0.50 \times 0.28 \times 0.14$ mm

Data collection

 Bruker SMART CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.888$, $T_{\max} = 0.967$

 6807 measured reflections
 2402 independent reflections
 1499 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.061$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.103$
 $S = 1.02$
 2402 reflections

 183 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.32$ e Å⁻³
 $\Delta\rho_{\min} = -0.31$ e Å⁻³

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O2}^i$	0.86	2.34	3.027 (3)	137

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2717).

References

- Bruker (2000). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
 Tai, X.-S., Feng, Y.-M. & Kong, F.-Y. (2008). Acta Cryst. E64, o750.

supporting information

Acta Cryst. (2008). E64, o827 [doi:10.1107/S1600536808009331]

2-Acetyl-3-methylpyrazine phenylsulfonylhydrazone

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S1. Comment

As part of our ongoing studies of aroylhydrazones as possible ligands (Tai *et al.*, 2008), we now report the synthesis and structure of the title compound, (I), (Fig. 1).

The dihedral angle between the aromatic ring planes is 55.42 (14)°. Otherwise, the geometrical parameters for (I) are normal. In the crystal of (I), an N-H...O hydrogen bond (Table 1) leads to [001] chains.

S2. Experimental

1 mmol of 2-Acetyl-3-methylpyrazine (1 mmol) was added to a solution of benzenesulfonyl hydrazide (1 mmol) in 5 ml of 95% ethanol. The mixture was continuously stirred for 4 h at refluxing temperature, evaporating some ethanol, then, upon cooling, the solid product was collected by filtration and dried *in vacuo* (yield 67%). Clear blocks of (I) were obtained by evaporation from a methanol solution after 3 days.

S3. Refinement

The H atoms were placed geometrically (C—H = 0.93–0.96 Å, N—H = 0.86 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

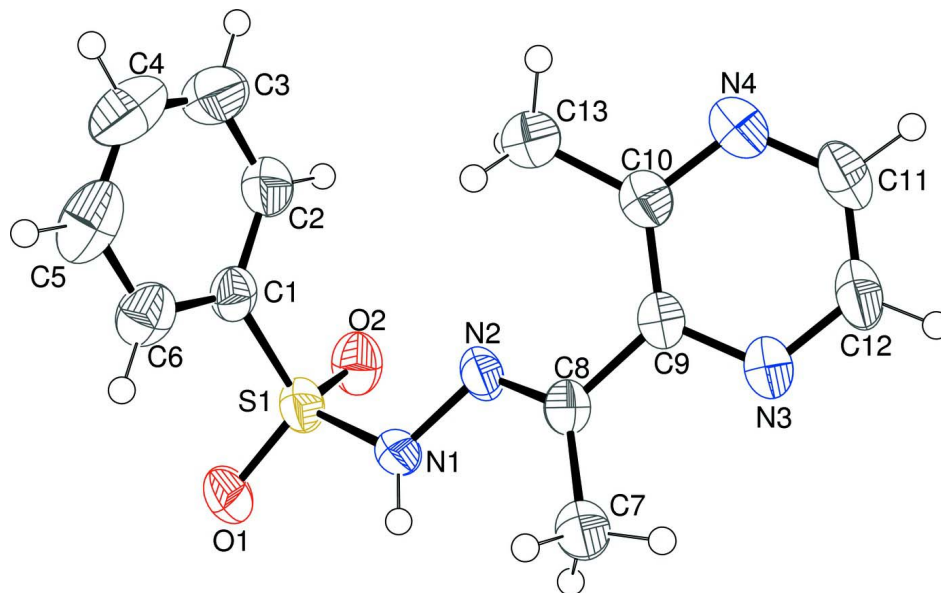


Figure 1

The molecular structure of (I) showing 30% displacement ellipsoids for the non-hydrogen atoms.

2-Acetyl-3-methylpyrazine phenylsulfonylhydrazone

Crystal data

C₁₃H₁₄N₄O₂S $M_r = 290.34$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 10.9848$ (15) Å $b = 16.7921$ (18) Å $c = 7.4817$ (10) Å $\beta = 97.264$ (1)° $V = 1369.0$ (3) Å³ $Z = 4$ $F(000) = 608$ $D_x = 1.409$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1439 reflections

 $\theta = 3.0$ – 22.9 ° $\mu = 0.24$ mm⁻¹ $T = 298$ K

Block, colourless

 $0.50 \times 0.28 \times 0.14$ mm

Data collection

Bruker SMART CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2000)

 $T_{\min} = 0.888$, $T_{\max} = 0.967$

6807 measured reflections

2402 independent reflections

1499 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.061$ $\theta_{\text{max}} = 25.0$ °, $\theta_{\text{min}} = 1.9$ ° $h = -13 \rightarrow 8$ $k = -19 \rightarrow 17$ $l = -8 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.046$ $wR(F^2) = 0.103$ $S = 1.03$

2402 reflections

183 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.036P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.76385 (18)	0.24028 (12)	0.7039 (3)	0.0401 (6)
H1	0.7785	0.2885	0.7389	0.048*
N2	0.64583 (19)	0.21106 (12)	0.6591 (3)	0.0386 (6)
N3	0.3511 (2)	0.28276 (13)	0.5461 (3)	0.0504 (7)

N4	0.2852 (2)	0.12465 (14)	0.5560 (3)	0.0552 (7)
O1	0.98156 (16)	0.21805 (11)	0.7451 (3)	0.0528 (6)
O2	0.85153 (17)	0.14561 (10)	0.5061 (2)	0.0508 (5)
S1	0.87236 (6)	0.17593 (4)	0.68486 (10)	0.0412 (2)
C1	0.8548 (2)	0.09773 (15)	0.8320 (4)	0.0385 (7)
C2	0.7922 (3)	0.03023 (16)	0.7692 (4)	0.0539 (8)
H2	0.7631	0.0249	0.6475	0.065*
C3	0.7734 (3)	-0.02929 (19)	0.8895 (6)	0.0704 (10)
H3A	0.7318	-0.0754	0.8494	0.084*
C4	0.8160 (3)	-0.0204 (2)	1.0670 (6)	0.0723 (11)
H4	0.8010	-0.0602	1.1479	0.087*
C5	0.8807 (3)	0.0460 (2)	1.1294 (4)	0.0660 (10)
H5	0.9114	0.0505	1.2506	0.079*
C6	0.8996 (3)	0.10584 (17)	1.0104 (4)	0.0516 (8)
H6	0.9425	0.1515	1.0508	0.062*
C7	0.5726 (3)	0.34525 (15)	0.7167 (4)	0.0500 (8)
H7A	0.6031	0.3497	0.8423	0.075*
H7B	0.4948	0.3718	0.6935	0.075*
H7C	0.6298	0.3694	0.6462	0.075*
C8	0.5571 (2)	0.25952 (15)	0.6673 (3)	0.0361 (7)
C9	0.4326 (2)	0.22753 (15)	0.6112 (3)	0.0370 (7)
C10	0.3985 (3)	0.14763 (16)	0.6207 (4)	0.0427 (7)
C11	0.2074 (3)	0.17989 (19)	0.4865 (4)	0.0581 (9)
H11	0.1286	0.1650	0.4380	0.070*
C12	0.2400 (3)	0.2581 (2)	0.4845 (4)	0.0588 (9)
H12	0.1817	0.2953	0.4381	0.071*
C13	0.4793 (3)	0.08310 (16)	0.7054 (4)	0.0641 (10)
H13A	0.4296	0.0395	0.7368	0.096*
H13B	0.5272	0.1031	0.8121	0.096*
H13C	0.5329	0.0651	0.6220	0.096*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0289 (14)	0.0326 (12)	0.0566 (16)	-0.0015 (10)	-0.0026 (11)	-0.0017 (11)
N2	0.0279 (14)	0.0445 (13)	0.0413 (15)	-0.0002 (11)	-0.0037 (11)	-0.0015 (11)
N3	0.0346 (16)	0.0528 (15)	0.0610 (18)	0.0029 (12)	-0.0043 (13)	0.0027 (13)
N4	0.0402 (17)	0.0594 (16)	0.0645 (18)	-0.0082 (13)	0.0006 (13)	-0.0014 (14)
O1	0.0291 (12)	0.0601 (13)	0.0667 (14)	-0.0069 (9)	-0.0033 (10)	0.0065 (11)
O2	0.0515 (13)	0.0615 (13)	0.0383 (12)	0.0083 (10)	0.0008 (9)	-0.0039 (10)
S1	0.0308 (4)	0.0479 (4)	0.0439 (5)	0.0027 (3)	0.0003 (3)	0.0018 (4)
C1	0.0321 (17)	0.0405 (16)	0.0429 (19)	0.0085 (12)	0.0042 (13)	-0.0010 (14)
C2	0.044 (2)	0.0517 (19)	0.063 (2)	0.0042 (15)	-0.0036 (16)	0.0007 (18)
C3	0.065 (2)	0.049 (2)	0.096 (3)	-0.0003 (16)	0.007 (2)	0.013 (2)
C4	0.080 (3)	0.057 (2)	0.086 (3)	0.019 (2)	0.037 (2)	0.025 (2)
C5	0.085 (3)	0.069 (2)	0.047 (2)	0.034 (2)	0.0166 (19)	0.004 (2)
C6	0.061 (2)	0.0464 (18)	0.048 (2)	0.0134 (15)	0.0079 (16)	-0.0043 (16)
C7	0.0441 (19)	0.0471 (18)	0.056 (2)	0.0026 (13)	-0.0027 (15)	-0.0038 (15)

C8	0.0340 (17)	0.0413 (16)	0.0323 (17)	0.0031 (13)	0.0014 (13)	0.0011 (13)
C9	0.0316 (17)	0.0447 (17)	0.0342 (17)	0.0030 (13)	0.0019 (13)	-0.0008 (14)
C10	0.0374 (18)	0.0484 (17)	0.0422 (18)	-0.0012 (14)	0.0045 (14)	-0.0009 (15)
C11	0.0324 (19)	0.073 (2)	0.066 (2)	-0.0047 (17)	-0.0047 (16)	-0.0056 (19)
C12	0.032 (2)	0.069 (2)	0.071 (2)	0.0059 (16)	-0.0084 (16)	0.0003 (18)
C13	0.050 (2)	0.0490 (19)	0.090 (3)	-0.0034 (15)	-0.0032 (18)	0.0134 (19)

Geometric parameters (Å, °)

N1—N2	1.387 (3)	C4—H4	0.9300
N1—S1	1.628 (2)	C5—C6	1.376 (4)
N1—H1	0.8600	C5—H5	0.9300
N2—C8	1.277 (3)	C6—H6	0.9300
N3—C12	1.316 (3)	C7—C8	1.491 (3)
N3—C9	1.337 (3)	C7—H7A	0.9600
N4—C11	1.323 (3)	C7—H7B	0.9600
N4—C10	1.334 (3)	C7—H7C	0.9600
O1—S1	1.4163 (18)	C8—C9	1.480 (3)
O2—S1	1.4220 (19)	C9—C10	1.397 (3)
S1—C1	1.740 (3)	C10—C13	1.490 (3)
C1—C6	1.370 (4)	C11—C12	1.361 (4)
C1—C2	1.377 (3)	C11—H11	0.9300
C2—C3	1.378 (4)	C12—H12	0.9300
C2—H2	0.9300	C13—H13A	0.9600
C3—C4	1.359 (5)	C13—H13B	0.9600
C3—H3A	0.9300	C13—H13C	0.9600
C4—C5	1.372 (4)		
N2—N1—S1	114.60 (16)	C5—C6—H6	120.2
N2—N1—H1	122.7	C8—C7—H7A	109.5
S1—N1—H1	122.7	C8—C7—H7B	109.5
C8—N2—N1	117.3 (2)	H7A—C7—H7B	109.5
C12—N3—C9	117.2 (2)	C8—C7—H7C	109.5
C11—N4—C10	117.8 (3)	H7A—C7—H7C	109.5
O1—S1—O2	120.49 (13)	H7B—C7—H7C	109.5
O1—S1—N1	103.92 (12)	N2—C8—C9	116.0 (2)
O2—S1—N1	106.70 (11)	N2—C8—C7	124.3 (2)
O1—S1—C1	109.44 (12)	C9—C8—C7	119.5 (2)
O2—S1—C1	107.89 (12)	N3—C9—C10	120.9 (2)
N1—S1—C1	107.71 (12)	N3—C9—C8	113.8 (2)
C6—C1—C2	121.1 (3)	C10—C9—C8	125.3 (2)
C6—C1—S1	119.1 (2)	N4—C10—C9	120.2 (2)
C2—C1—S1	119.7 (2)	N4—C10—C13	115.0 (2)
C1—C2—C3	118.9 (3)	C9—C10—C13	124.8 (2)
C1—C2—H2	120.6	N4—C11—C12	121.5 (3)
C3—C2—H2	120.6	N4—C11—H11	119.2
C4—C3—C2	119.8 (3)	C12—C11—H11	119.2
C4—C3—H3A	120.1	N3—C12—C11	122.3 (3)

C2—C3—H3A	120.1	N3—C12—H12	118.9
C3—C4—C5	121.4 (3)	C11—C12—H12	118.9
C3—C4—H4	119.3	C10—C13—H13A	109.5
C5—C4—H4	119.3	C10—C13—H13B	109.5
C4—C5—C6	119.2 (3)	H13A—C13—H13B	109.5
C4—C5—H5	120.4	C10—C13—H13C	109.5
C6—C5—H5	120.4	H13A—C13—H13C	109.5
C1—C6—C5	119.5 (3)	H13B—C13—H13C	109.5
C1—C6—H6	120.2		
S1—N1—N2—C8	-178.3 (2)	N1—N2—C8—C9	177.5 (2)
N2—N1—S1—O1	-177.34 (18)	N1—N2—C8—C7	1.7 (4)
N2—N1—S1—O2	54.3 (2)	C12—N3—C9—C10	-2.9 (4)
N2—N1—S1—C1	-61.3 (2)	C12—N3—C9—C8	176.5 (3)
O1—S1—C1—C6	30.8 (3)	N2—C8—C9—N3	-152.2 (3)
O2—S1—C1—C6	163.6 (2)	C7—C8—C9—N3	23.8 (4)
N1—S1—C1—C6	-81.6 (2)	N2—C8—C9—C10	27.2 (4)
O1—S1—C1—C2	-151.7 (2)	C7—C8—C9—C10	-156.8 (3)
O2—S1—C1—C2	-18.9 (3)	C11—N4—C10—C9	-1.1 (4)
N1—S1—C1—C2	96.0 (2)	C11—N4—C10—C13	177.0 (3)
C6—C1—C2—C3	0.9 (4)	N3—C9—C10—N4	3.5 (4)
S1—C1—C2—C3	-176.5 (2)	C8—C9—C10—N4	-175.9 (3)
C1—C2—C3—C4	0.3 (5)	N3—C9—C10—C13	-174.4 (3)
C2—C3—C4—C5	-1.8 (5)	C8—C9—C10—C13	6.3 (5)
C3—C4—C5—C6	2.0 (5)	C10—N4—C11—C12	-1.6 (5)
C2—C1—C6—C5	-0.7 (4)	C9—N3—C12—C11	0.2 (5)
S1—C1—C6—C5	176.8 (2)	N4—C11—C12—N3	2.1 (5)
C4—C5—C6—C1	-0.7 (4)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O2 ⁱ	0.86	2.34	3.027 (3)	137

Symmetry code: (i) $x, -y+1/2, z+1/2$.