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4-(3-Methyl-5-phenyl-1*H*-pyrazol-1-yl)-benzenesulfonamide

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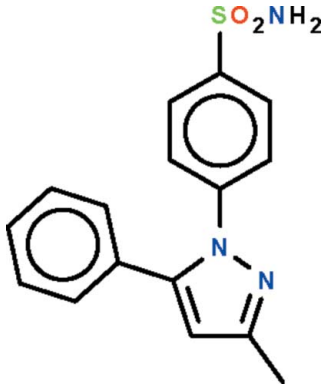
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.115; data-to-parameter ratio = 15.1.

With respect to the planar five-membered ring of the title compound, $C_{16}H_{15}N_3O_2S$, the phenyl ring is aligned at 47.0 (1)° and the phenylene ring at 37.6 (1)°. The amino group has the N atom in a pyramidal geometry; the group is a hydrogen-bond donor to the sulfonyl O atom of one molecule and to the pyrazole N atom of another molecule, resulting in the formation of a layer parallel to the *bc* plane.

Related literature

For the synthesis, see: Gosselin *et al.* (2006); Organ & Mayer (2003).



Experimental

Crystal data

$C_{16}H_{15}N_3O_2S$
M_r = 313.37
Monoclinic, $C2/c$
a = 28.2545 (8) Å
b = 11.9135 (4) Å
c = 9.3739 (3) Å
β = 91.016 (3)°
V = 3154.85 (17) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 1.91 mm^{-1}
T = 100 K
0.30 × 0.03 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.598, T_{max} = 0.945
6579 measured reflections
3137 independent reflections
2689 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.115
S = 1.03
3137 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3^{i}	0.92 (2)	1.98 (2)	2.878 (2)	164 (2)
N1—H2···O1^{ii}	0.86 (2)	2.07 (2)	2.930 (2)	177 (2)

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x, -y+1, z-\frac{1}{2}$.

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).

We thank King Abdulaziz University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5609).

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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supplementary materials

Acta Cryst. (2011). E**67**, o2426 [**doi:10.1107/S1600536811032855**]

4-(3-Methyl-5-phenyl-1*H*-pyrazol-1-yl)benzenesulfonamide

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Comment

We are examining the medicinal properties of phenylpyrazolones of which the 4-benzenesulfamide derivative (Scheme I) is expected to show enhanced activity. As the inhibitory activity against cyclooxygenase-1 and cyclooxygenase-2 of the title compound (Scheme I) has been claimed in a number of patents, other researchers have attempted its synthesis in order to increase yield (Gosselin *et al.*, 2006; Organ & Mayer, 2003). With respect to the planar five-membered ring, the phenyl ring is aligned at 47.0 (1) ° and the phenylene ring at 37.6 (1)°. The amino group is hydrogen bond donor to the sulfonyl O atom of one molecule and to the pyrazolyl N atom of another molecule to result in the formation of a layer parallel to the bc plane.

Experimental

1-Phenylbutan-1,3-dione (10 mmol) and 4-hydrazinobenzenesulfonamide hydrochloride (10 mmol) were heated in ethanol (50 ml) for 4 h; water was added to precipitate the product, which was collected and recrystallized from ethanol as light yellow crystals; m.p. 471–472 K.

Refinement

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

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

Figures

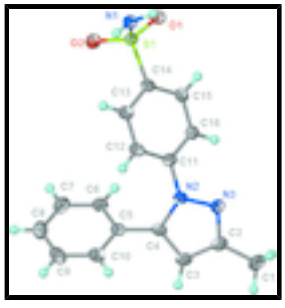


Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{16}H_{15}N_3O_2S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-(3-Methyl-5-phenyl-1*H*-pyrazol-1-yl)benzenesulfonamide

Crystal data

$C_{16}H_{15}N_3O_2S$ $F(000) = 1312$

M_r = 313.37	D_x = 1.320 Mg m^{-3}
Monoclinic, *C*2/*c*	Cu *K*α radiation, λ = 1.54184 Å
Hall symbol: -C 2yc	Cell parameters from 2773 reflections
a = 28.2545 (8) Å	θ = 3.1–74.2°
b = 11.9135 (4) Å	μ = 1.91 mm^{-1}
c = 9.3739 (3) Å	*T* = 100 K
β = 91.016 (3)°	Prism, light-yellow
V = 3154.85 (17) Å^3	0.30 × 0.03 × 0.03 mm
Z = 8	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	3137 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	2689 reflections with $I > 2\sigma(I)$
Mirror	R_{int} = 0.035
Detector resolution: 10.4041 pixels mm^{-1}	θ_{max} = 74.4°, θ_{min} = 3.1°
ω scans	*h* = −35→31
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	*k* = −12→14
T_{min} = 0.598, T_{max} = 0.945	*l* = −11→9
6579 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.041	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.115	H atoms treated by a mixture of independent and constrained refinement
S = 1.03	$w = 1/[\sigma^2(F_o^2) + (0.0629P)^2 + 1.4812P]$ where $P = (F_o^2 + 2F_c^2)/3$
3137 reflections	$(\Delta/\sigma)_{max}$ = 0.001
208 parameters	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.690362 (14)	0.48353 (4)	0.65383 (4)	0.01752 (14)
O1	0.67646 (4)	0.41033 (11)	0.76803 (13)	0.0220 (3)
O2	0.69504 (5)	0.60135 (11)	0.68142 (14)	0.0252 (3)
N1	0.65210 (5)	0.46753 (14)	0.52785 (16)	0.0191 (3)
H1	0.6414 (8)	0.395 (2)	0.516 (2)	0.026 (6)*
H2	0.6583 (8)	0.5054 (18)	0.452 (2)	0.017 (5)*
N2	0.88441 (5)	0.35478 (13)	0.48169 (15)	0.0189 (3)

N3	0.89694 (5)	0.24415 (13)	0.47817 (17)	0.0217 (3)
C1	0.96762 (7)	0.13366 (18)	0.4272 (3)	0.0341 (5)
H1A	0.9686	0.0956	0.5199	0.051*
H1B	1.0000	0.1470	0.3954	0.051*
H1C	0.9508	0.0864	0.3573	0.051*
C2	0.94240 (6)	0.24326 (17)	0.4411 (2)	0.0234 (4)
C3	0.95933 (6)	0.35272 (17)	0.42301 (19)	0.0225 (4)
H3	0.9904	0.3741	0.3974	0.027*
C4	0.92187 (6)	0.42307 (16)	0.44972 (17)	0.0193 (4)
C5	0.91994 (6)	0.54696 (16)	0.44973 (18)	0.0192 (4)
C6	0.89996 (6)	0.60729 (16)	0.5616 (2)	0.0229 (4)
H6	0.8878	0.5683	0.6413	0.028*
C7	0.89779 (7)	0.72353 (17)	0.5573 (2)	0.0284 (4)
H7	0.8840	0.7639	0.6335	0.034*
C8	0.91585 (7)	0.78079 (17)	0.4409 (2)	0.0307 (5)
H8	0.9138	0.8603	0.4365	0.037*
C9	0.93685 (7)	0.72183 (18)	0.3313 (2)	0.0292 (4)
H9	0.9499	0.7612	0.2531	0.035*
C10	0.93889 (6)	0.60550 (17)	0.3355 (2)	0.0235 (4)
H10	0.9533	0.5656	0.2600	0.028*
C11	0.83692 (6)	0.38175 (15)	0.51674 (18)	0.0188 (4)
C12	0.81362 (6)	0.47000 (16)	0.44808 (19)	0.0214 (4)
H12	0.8285	0.5097	0.3733	0.026*
C13	0.76862 (6)	0.49917 (16)	0.48990 (19)	0.0213 (4)
H13	0.7529	0.5611	0.4465	0.026*
C14	0.74629 (6)	0.43750 (15)	0.59607 (18)	0.0183 (4)
C15	0.76849 (6)	0.34505 (15)	0.65805 (18)	0.0193 (4)
H15	0.7523	0.3004	0.7256	0.023*
C16	0.81447 (6)	0.31858 (15)	0.62039 (19)	0.0194 (4)
H16	0.8305	0.2576	0.6652	0.023*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0180 (2)	0.0196 (2)	0.0150 (2)	−0.00041 (15)	0.00220 (16)	−0.00277 (15)
O1	0.0219 (6)	0.0280 (7)	0.0161 (6)	0.0002 (5)	0.0038 (5)	−0.0002 (5)
O2	0.0254 (7)	0.0217 (7)	0.0287 (7)	−0.0010 (5)	0.0032 (5)	−0.0067 (5)
N1	0.0192 (7)	0.0221 (8)	0.0159 (7)	−0.0009 (6)	0.0013 (6)	0.0001 (6)
N2	0.0169 (7)	0.0212 (8)	0.0187 (7)	0.0003 (6)	0.0012 (6)	−0.0010 (6)
N3	0.0201 (7)	0.0195 (8)	0.0256 (8)	0.0009 (6)	0.0010 (6)	−0.0027 (6)
C1	0.0271 (10)	0.0280 (11)	0.0474 (13)	0.0041 (8)	0.0067 (9)	−0.0085 (9)
C2	0.0198 (9)	0.0270 (10)	0.0234 (9)	0.0012 (7)	0.0000 (7)	−0.0044 (7)
C3	0.0179 (8)	0.0283 (10)	0.0212 (9)	−0.0012 (7)	0.0017 (7)	−0.0037 (7)
C4	0.0180 (8)	0.0249 (9)	0.0149 (8)	−0.0034 (7)	0.0001 (6)	−0.0002 (7)
C5	0.0148 (8)	0.0236 (9)	0.0190 (9)	−0.0015 (7)	−0.0018 (6)	0.0007 (7)
C6	0.0207 (9)	0.0264 (10)	0.0218 (9)	−0.0014 (7)	0.0010 (7)	0.0004 (7)
C7	0.0248 (9)	0.0269 (10)	0.0335 (11)	0.0031 (8)	−0.0015 (8)	−0.0050 (8)
C8	0.0288 (10)	0.0219 (10)	0.0410 (12)	0.0007 (8)	−0.0067 (8)	0.0047 (9)

C9	0.0278 (10)	0.0302 (10)	0.0293 (10)	−0.0067 (8)	−0.0037 (8)	0.0098 (8)
C10	0.0206 (9)	0.0289 (10)	0.0211 (9)	−0.0038 (7)	0.0004 (7)	0.0009 (7)
C11	0.0161 (8)	0.0223 (9)	0.0179 (8)	−0.0009 (7)	−0.0001 (6)	−0.0023 (7)
C12	0.0189 (9)	0.0279 (10)	0.0175 (9)	−0.0022 (7)	0.0014 (7)	0.0054 (7)
C13	0.0186 (8)	0.0254 (9)	0.0199 (9)	−0.0003 (7)	−0.0003 (7)	0.0036 (7)
C14	0.0171 (8)	0.0210 (9)	0.0169 (8)	−0.0025 (7)	0.0005 (6)	−0.0031 (7)
C15	0.0220 (9)	0.0189 (9)	0.0171 (8)	−0.0028 (7)	0.0028 (7)	−0.0016 (7)
C16	0.0209 (9)	0.0192 (9)	0.0182 (8)	0.0003 (7)	0.0010 (6)	−0.0004 (7)

Geometric parameters (Å, °)

S1—O2	1.4330 (13)	C6—C7	1.387 (3)
S1—O1	1.4407 (13)	C6—H6	0.9500
S1—N1	1.5983 (15)	C7—C8	1.391 (3)
S1—C14	1.7666 (18)	C7—H7	0.9500
N1—H1	0.92 (2)	C8—C9	1.387 (3)
N1—H2	0.86 (2)	C8—H8	0.9500
N2—N3	1.365 (2)	C9—C10	1.388 (3)
N2—C4	1.372 (2)	C9—H9	0.9500
N2—C11	1.424 (2)	C10—H10	0.9500
N3—C2	1.337 (2)	C11—C16	1.391 (2)
C1—C2	1.494 (3)	C11—C12	1.392 (3)
C1—H1A	0.9800	C12—C13	1.382 (3)
C1—H1B	0.9800	C12—H12	0.9500
C1—H1C	0.9800	C13—C14	1.397 (2)
C2—C3	1.400 (3)	C13—H13	0.9500
C3—C4	1.376 (3)	C14—C15	1.390 (3)
C3—H3	0.9500	C15—C16	1.389 (2)
C4—C5	1.477 (3)	C15—H15	0.9500
C5—C10	1.393 (3)	C16—H16	0.9500
C5—C6	1.398 (3)		
O2—S1—O1	118.97 (8)	C7—C6—H6	119.7
O2—S1—N1	108.01 (8)	C5—C6—H6	119.7
O1—S1—N1	106.68 (8)	C6—C7—C8	119.71 (19)
O2—S1—C14	106.21 (8)	C6—C7—H7	120.1
O1—S1—C14	107.27 (8)	C8—C7—H7	120.1
N1—S1—C14	109.49 (8)	C9—C8—C7	120.03 (19)
S1—N1—H1	114.6 (14)	C9—C8—H8	120.0
S1—N1—H2	113.6 (14)	C7—C8—H8	120.0
H1—N1—H2	117.8 (19)	C8—C9—C10	120.20 (18)
N3—N2—C4	111.46 (14)	C8—C9—H9	119.9
N3—N2—C11	117.98 (14)	C10—C9—H9	119.9
C4—N2—C11	130.56 (16)	C9—C10—C5	120.36 (18)
C2—N3—N2	105.35 (15)	C9—C10—H10	119.8
C2—C1—H1A	109.5	C5—C10—H10	119.8
C2—C1—H1B	109.5	C16—C11—C12	120.91 (16)
H1A—C1—H1B	109.5	C16—C11—N2	118.85 (16)
C2—C1—H1C	109.5	C12—C11—N2	120.24 (16)
H1A—C1—H1C	109.5	C13—C12—C11	119.26 (16)

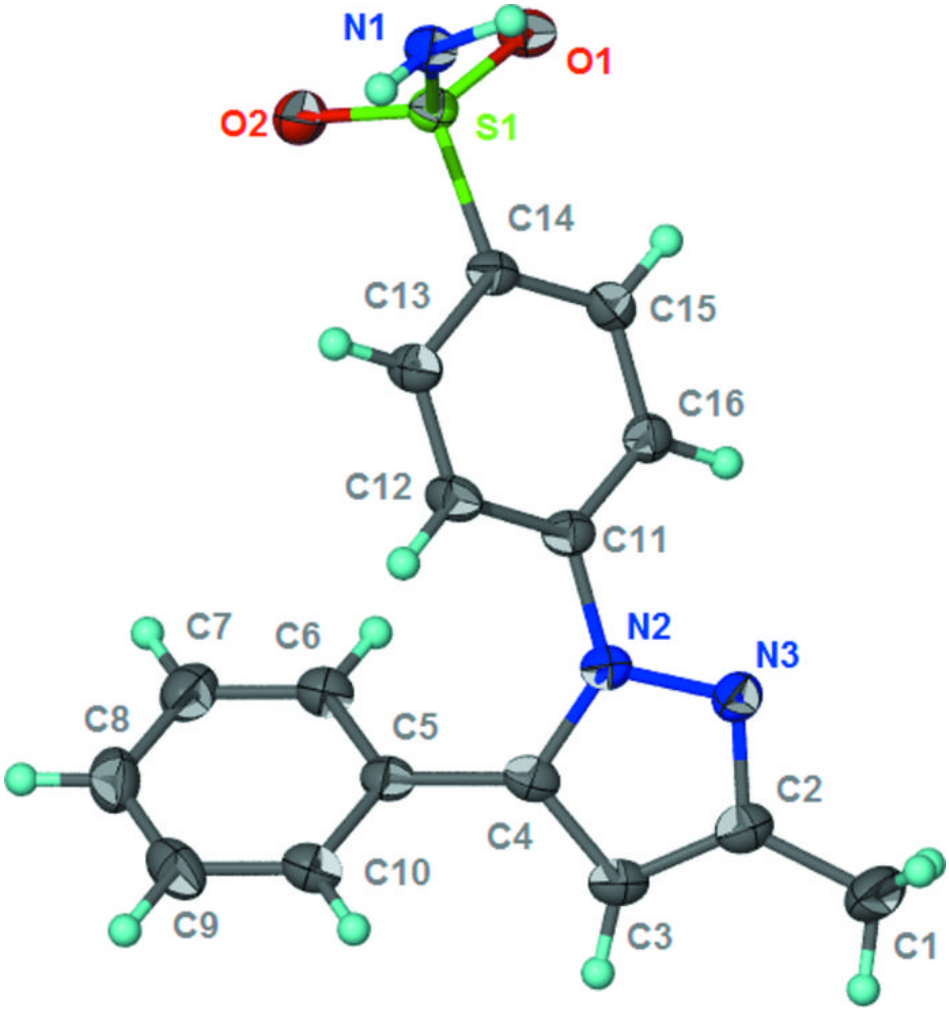
H1B—C1—H1C	109.5	C13—C12—H12	120.4
N3—C2—C3	110.85 (16)	C11—C12—H12	120.4
N3—C2—C1	119.44 (18)	C12—C13—C14	119.97 (17)
C3—C2—C1	129.70 (17)	C12—C13—H13	120.0
C4—C3—C2	106.24 (16)	C14—C13—H13	120.0
C4—C3—H3	126.9	C15—C14—C13	120.57 (16)
C2—C3—H3	126.9	C15—C14—S1	121.16 (13)
N2—C4—C3	106.09 (16)	C13—C14—S1	118.22 (14)
N2—C4—C5	124.32 (16)	C16—C15—C14	119.42 (16)
C3—C4—C5	129.57 (16)	C16—C15—H15	120.3
C10—C5—C6	119.01 (18)	C14—C15—H15	120.3
C10—C5—C4	119.05 (16)	C15—C16—C11	119.67 (16)
C6—C5—C4	121.94 (16)	C15—C16—H16	120.2
C7—C6—C5	120.64 (18)	C11—C16—H16	120.2
C4—N2—N3—C2	−1.13 (19)	C6—C5—C10—C9	−1.6 (3)
C11—N2—N3—C2	179.13 (15)	C4—C5—C10—C9	178.87 (16)
N2—N3—C2—C3	1.0 (2)	N3—N2—C11—C16	37.9 (2)
N2—N3—C2—C1	179.95 (17)	C4—N2—C11—C16	−141.76 (18)
N3—C2—C3—C4	−0.5 (2)	N3—N2—C11—C12	−142.14 (17)
C1—C2—C3—C4	−179.3 (2)	C4—N2—C11—C12	38.2 (3)
N3—N2—C4—C3	0.86 (19)	C16—C11—C12—C13	3.9 (3)
C11—N2—C4—C3	−179.44 (16)	N2—C11—C12—C13	−176.02 (16)
N3—N2—C4—C5	−177.79 (15)	C11—C12—C13—C14	−2.5 (3)
C11—N2—C4—C5	1.9 (3)	C12—C13—C14—C15	−1.5 (3)
C2—C3—C4—N2	−0.24 (19)	C12—C13—C14—S1	176.01 (14)
C2—C3—C4—C5	178.31 (17)	O2—S1—C14—C15	128.99 (15)
N2—C4—C5—C10	−133.78 (18)	O1—S1—C14—C15	0.77 (17)
C3—C4—C5—C10	47.9 (3)	N1—S1—C14—C15	−114.63 (15)
N2—C4—C5—C6	46.7 (2)	O2—S1—C14—C13	−48.55 (16)
C3—C4—C5—C6	−131.6 (2)	O1—S1—C14—C13	−176.76 (14)
C10—C5—C6—C7	1.8 (3)	N1—S1—C14—C13	67.84 (16)
C4—C5—C6—C7	−178.70 (16)	C13—C14—C15—C16	4.2 (3)
C5—C6—C7—C8	−0.3 (3)	S1—C14—C15—C16	−173.24 (13)
C6—C7—C8—C9	−1.4 (3)	C14—C15—C16—C11	−2.8 (3)
C7—C8—C9—C10	1.6 (3)	C12—C11—C16—C15	−1.2 (3)
C8—C9—C10—C5	−0.1 (3)	N2—C11—C16—C15	178.71 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3^{i}	0.92 (2)	1.98 (2)	2.878 (2)	164 (2)
N1—H2···O1^{ii}	0.86 (2)	2.07 (2)	2.930 (2)	177 (2)

Symmetry codes: (i) −*x*+3/2, −*y*+1/2, −*z*+1; (ii) *x*, −*y*+1, *z*−1/2.

Fig. 1


N1
O1
O2
S1
C14
C13
C15
C16
C12
C11
N2
N3
C7
C6
C8
C5
C2
C4
C10
C9
C3
C1