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4-(3,4-Diacetyl-5-methyl-1*H*-pyrazol-1-yl)benzenesulfonamide

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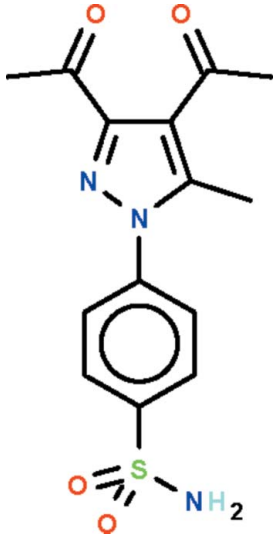
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.095; data-to-parameter ratio = 14.7.

In the title molecule, $C_{14}H_{15}N_3O_4S$, the pyrazole ring is aligned at a dihedral angle of 55.5 (1)° with respect to the benzene ring; the mean planes of the acetyl substituents are twisted by 13.4 (3) and 30.1 (3)° with respect to the pyrazole ring. Intermolecular classical N—H···O and weak C—H···O hydrogen bonding links the molecules, forming a three-dimensional network architecture in the crystal structure.

Related literature

For background to the biological properties of aryl-substituted pyrazoles, see: Abdel-Aziz *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{15}N_3O_4S$
M_r = 321.35
Orthorhombic, $Pna2_1$
a = 8.3716 (3) Å
b = 21.7722 (8) Å
c = 7.8915 (3) Å
V = 1438.37 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.952, T_{max} = 0.988
10477 measured reflections
3087 independent reflections
2634 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.095
S = 1.05
3087 reflections
210 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983), 1337 Friedel pairs
Flack parameter: 0.08 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H31···O2^{i}	0.88 (1)	2.03 (1)	2.864 (3)	159 (3)
N3—H32···O4^{ii}	0.88 (1)	2.06 (1)	2.933 (3)	170 (3)
C1—H1*C*···O3^{i}	0.98	2.55	3.446 (3)	151
C10—H10···O1^{iii}	0.95	2.51	3.314 (3)	142
C14—H14···O1^{iv}	0.95	2.54	3.414 (3)	153

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5161).

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supporting information

Acta Cryst. (2011). E**67**, o693 [doi:10.1107/S1600536811005733]

4-(3,4-Diacetyl-5-methyl-1*H*-pyrazol-1-yl)benzenesulfonamide

Hatem A. Abdel-Aziz, Ahmed Bari and Seik Weng Ng

S1. Comment

We have reported the antitumor activity of aryl-pyrazoles against CaCo-2 and HEP-2 cell lines (Abdel-Aziz *et al.*, 2010). These compounds were synthesized by a cycloaddition under microwave conditions. The present study involves the synthesis of an aryl-pyrazole having a sulfonamide $–SO_2NH_2$ substituent (Scheme I) that is expected to improve aqueous solubility. The $C_{14}H_{15}N_3O_4$ molecule has two acetyl substitutents on the pyrazolyl ring along with a benzenesulfanomide group. The sulfonamido unit interacts with an adjacent acetyl and sulfonamido O-atoms to generate a three-dimensional network (Table 1).

S2. Experimental

1-Phenyl-2-(phenylsulfonyl)ethanone (0.26 g, 10 mmol) was dissolved in a sodium ethoxide solution (prepared by dissolving 0.23 g sodium metal in 50 ml absolute ethanol). To the solution was added (*Z*)-2-oxo-*N*′-(4-sulfamoylphenyl)-propanehydrazonoyl chloride (0.28 g, 10 mmol). The mixture was stirred for 12 h. The mixuture was then poured into cold water; the solid product was collected and recrystallized from an ethanol-water (4:1) mixture.

S3. Refinement

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

The amino H-atoms were located in a difference Fourier map, and were refined with a distance restraint of N–H 0.88±0.01 Å; their temperature factors were refined.

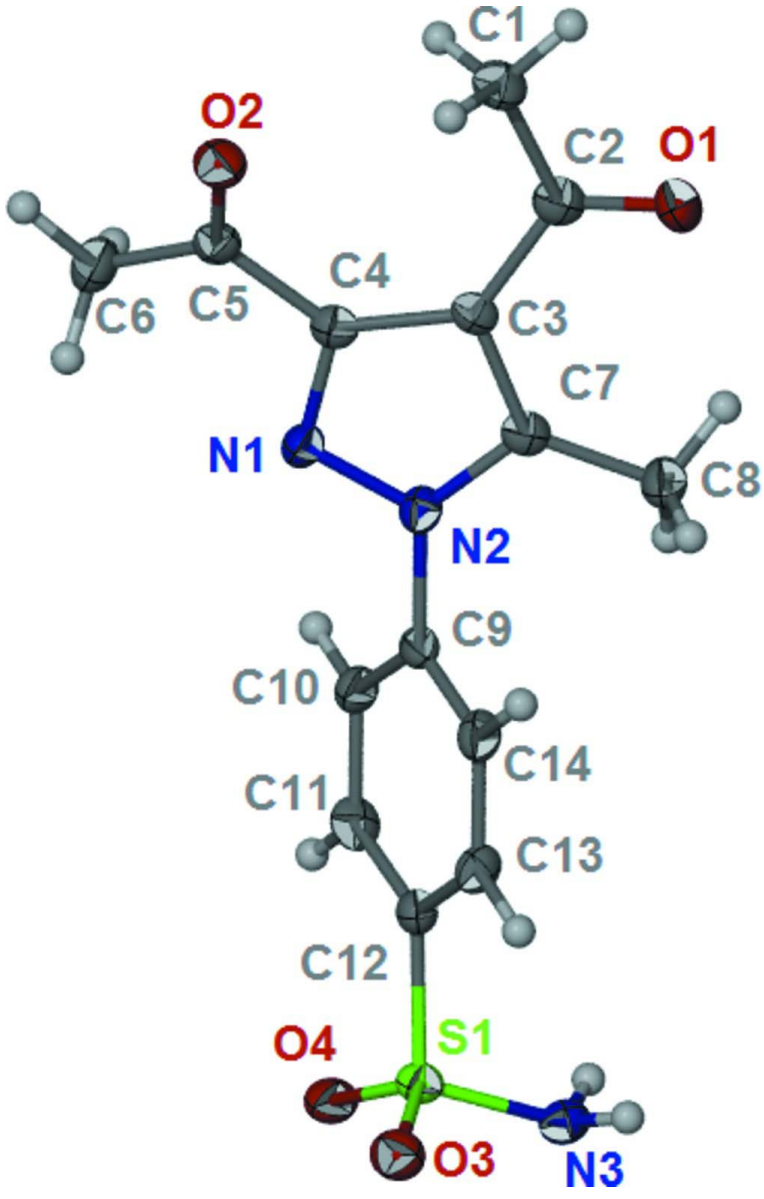

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{15}N_3O_4S$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-(3,4-Diacetyl-5-methyl-1*H*-pyrazol-1-yl)benzenesulfonamide

Crystal data

$C_{14}H_{15}N_3O_4S$
$M_r = 321.35$
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a = 8.3716\ (3)$ Å
$b = 21.7722\ (8)$ Å
$c = 7.8915\ (3)$ Å
$V = 1438.37\ (9)$ Å^3
$Z = 4$
$F(000) = 672$
$D_x = 1.484$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3291 reflections
$\theta = 2.4$–$29.2°$
$\mu = 0.25$ mm^{-1}
$T = 100$ K
Prism, colorless
0.20 × 0.15 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.952, T_{max} = 0.988
10477 measured reflections
3087 independent reflections
2634 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 27.5°, θ_{min} = 2.6°
h = −10→10
k = −28→27
l = −10→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.095
S = 1.05
3087 reflections
210 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.31 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.39 e $Å^{-3}$
Absolute structure: Flack (1983), 1337 Friedel pairs
Absolute structure parameter: 0.08 (8)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.69022 (7)	0.29885 (3)	0.49994 (9)	0.01726 (16)
O1	1.5767 (2)	0.55022 (9)	0.1918 (3)	0.0278 (5)
O2	1.3205 (2)	0.68881 (8)	0.5594 (3)	0.0229 (5)
O3	0.7216 (2)	0.27511 (9)	0.6664 (3)	0.0234 (5)
O4	0.5323 (2)	0.31899 (7)	0.4576 (3)	0.0226 (5)
N1	1.0863 (2)	0.56565 (9)	0.4491 (3)	0.0164 (5)
N2	1.1261 (2)	0.50985 (9)	0.3847 (3)	0.0160 (5)
N3	0.7369 (3)	0.24582 (10)	0.3684 (3)	0.0182 (5)
H31	0.726 (3)	0.2569 (13)	0.2624 (17)	0.017 (8)*
H32	0.8310 (18)	0.2289 (11)	0.385 (4)	0.021 (8)*
C1	1.5821 (3)	0.64151 (12)	0.3508 (4)	0.0235 (7)
H1A	1.6916	0.6438	0.3072	0.035*
H1B	1.5845	0.6411	0.4750	0.035*
H1C	1.5215	0.6773	0.3115	0.035*
C2	1.5041 (3)	0.58395 (12)	0.2877 (4)	0.0196 (6)
C3	1.3404 (3)	0.56637 (11)	0.3441 (3)	0.0149 (6)
C4	1.2165 (3)	0.60007 (11)	0.4274 (3)	0.0157 (6)
C5	1.2032 (3)	0.66253 (11)	0.5028 (4)	0.0179 (5)
C6	1.0404 (3)	0.68993 (11)	0.5136 (5)	0.0252 (6)
H6A	1.0466	0.7299	0.5704	0.038*
H6B	0.9704	0.6625	0.5783	0.038*
H6C	0.9972	0.6954	0.3992	0.038*
C7	1.2751 (3)	0.50868 (12)	0.3166 (3)	0.0170 (6)

C8	1.3336 (3)	0.45309 (12)	0.2257 (4)	0.0236 (7)
H8A	1.2448	0.4339	0.1646	0.035*
H8B	1.3777	0.4238	0.3077	0.035*
H8C	1.4170	0.4650	0.1449	0.035*
C9	1.0197 (3)	0.45957 (11)	0.4118 (4)	0.0160 (6)
C10	0.8629 (3)	0.46375 (12)	0.3568 (3)	0.0176 (6)
H10	0.8261	0.4996	0.3002	0.021*
C11	0.7607 (3)	0.41477 (12)	0.3856 (4)	0.0196 (6)
H11	0.6525	0.4168	0.3498	0.024*
C12	0.8176 (3)	0.36269 (11)	0.4670 (3)	0.0172 (6)
C13	0.9744 (3)	0.35924 (11)	0.5236 (4)	0.0178 (6)
H13	1.0112	0.3236	0.5810	0.021*
C14	1.0763 (3)	0.40801 (11)	0.4958 (4)	0.0190 (6)
H14	1.1839	0.4063	0.5338	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0148 (3)	0.0163 (3)	0.0207 (4)	−0.0022 (2)	0.0012 (3)	0.0006 (3)
O1	0.0200 (10)	0.0241 (10)	0.0392 (13)	0.0012 (8)	0.0106 (10)	−0.0016 (10)
O2	0.0238 (11)	0.0223 (10)	0.0226 (12)	−0.0058 (8)	0.0029 (8)	−0.0047 (9)
O3	0.0263 (10)	0.0229 (10)	0.0211 (12)	−0.0025 (9)	0.0023 (9)	0.0001 (9)
O4	0.0157 (9)	0.0188 (9)	0.0334 (14)	−0.0020 (7)	0.0016 (8)	0.0017 (9)
N1	0.0164 (11)	0.0135 (10)	0.0192 (13)	0.0009 (8)	0.0006 (9)	−0.0009 (9)
N2	0.0147 (11)	0.0159 (10)	0.0175 (12)	0.0008 (9)	−0.0015 (9)	−0.0025 (10)
N3	0.0192 (12)	0.0160 (11)	0.0192 (14)	−0.0004 (9)	−0.0017 (10)	0.0022 (11)
C1	0.0171 (13)	0.0244 (14)	0.0289 (18)	−0.0047 (11)	0.0023 (12)	−0.0023 (14)
C2	0.0170 (13)	0.0203 (14)	0.0215 (16)	0.0011 (11)	−0.0017 (12)	0.0043 (14)
C3	0.0143 (12)	0.0174 (13)	0.0132 (15)	−0.0004 (10)	−0.0010 (10)	0.0031 (11)
C4	0.0165 (12)	0.0173 (12)	0.0134 (14)	−0.0015 (10)	−0.0011 (11)	0.0011 (12)
C5	0.0242 (13)	0.0162 (12)	0.0134 (13)	−0.0016 (10)	0.0032 (13)	0.0016 (14)
C6	0.0255 (14)	0.0191 (13)	0.0312 (18)	0.0020 (11)	0.0024 (15)	−0.0052 (14)
C7	0.0155 (13)	0.0180 (13)	0.0176 (15)	−0.0002 (10)	−0.0017 (11)	0.0026 (12)
C8	0.0190 (13)	0.0197 (14)	0.0321 (19)	−0.0007 (11)	0.0051 (12)	−0.0050 (13)
C9	0.0163 (12)	0.0147 (12)	0.0170 (15)	−0.0027 (10)	0.0032 (11)	−0.0048 (11)
C10	0.0173 (13)	0.0151 (12)	0.0204 (16)	0.0021 (10)	0.0002 (11)	−0.0003 (12)
C11	0.0118 (12)	0.0241 (13)	0.0229 (16)	0.0013 (11)	−0.0031 (11)	−0.0012 (14)
C12	0.0165 (12)	0.0158 (12)	0.0193 (17)	0.0001 (10)	0.0031 (11)	−0.0017 (12)
C13	0.0179 (12)	0.0150 (12)	0.0207 (16)	0.0032 (10)	−0.0021 (11)	0.0007 (12)
C14	0.0157 (12)	0.0189 (12)	0.0224 (15)	0.0016 (10)	−0.0001 (13)	−0.0040 (13)

Geometric parameters (Å, º)

S1—O4	1.4325 (18)	C4—C5	1.489 (3)
S1—O3	1.436 (2)	C5—C6	1.490 (3)
S1—N3	1.601 (3)	C6—H6A	0.9800
S1—C12	1.771 (2)	C6—H6B	0.9800
O1—C2	1.218 (3)	C6—H6C	0.9800

O2—C5	1.221 (3)	C7—C8	1.490 (4)
N1—C4	1.334 (3)	C8—H8A	0.9800
N1—N2	1.358 (3)	C8—H8B	0.9800
N2—C7	1.358 (3)	C8—H8C	0.9800
N2—C9	1.427 (3)	C9—C10	1.385 (3)
N3—H31	0.875 (10)	C9—C14	1.387 (4)
N3—H32	0.879 (10)	C10—C11	1.386 (4)
C1—C2	1.498 (4)	C10—H10	0.9500
C1—H1A	0.9800	C11—C12	1.388 (4)
C1—H1B	0.9800	C11—H11	0.9500
C1—H1C	0.9800	C12—C13	1.388 (3)
C2—C3	1.491 (4)	C13—C14	1.380 (3)
C3—C7	1.387 (3)	C13—H13	0.9500
C3—C4	1.430 (4)	C14—H14	0.9500

O4—S1—O3	119.48 (11)	C5—C6—H6B	109.5
O4—S1—N3	107.16 (12)	H6A—C6—H6B	109.5
O3—S1—N3	106.79 (13)	C5—C6—H6C	109.5
O4—S1—C12	106.35 (11)	H6A—C6—H6C	109.5
O3—S1—C12	107.84 (12)	H6B—C6—H6C	109.5
N3—S1—C12	108.90 (12)	N2—C7—C3	106.5 (2)
C4—N1—N2	104.7 (2)	N2—C7—C8	120.5 (2)
N1—N2—C7	112.94 (19)	C3—C7—C8	132.9 (2)
N1—N2—C9	118.5 (2)	C7—C8—H8A	109.5
C7—N2—C9	128.2 (2)	C7—C8—H8B	109.5
S1—N3—H31	113 (2)	H8A—C8—H8B	109.5
S1—N3—H32	115.2 (19)	C7—C8—H8C	109.5
H31—N3—H32	110 (3)	H8A—C8—H8C	109.5
C2—C1—H1A	109.5	H8B—C8—H8C	109.5
C2—C1—H1B	109.5	C10—C9—C14	121.8 (2)
H1A—C1—H1B	109.5	C10—C9—N2	119.6 (2)
C2—C1—H1C	109.5	C14—C9—N2	118.6 (2)
H1A—C1—H1C	109.5	C9—C10—C11	118.9 (2)
H1B—C1—H1C	109.5	C9—C10—H10	120.6
O1—C2—C3	119.3 (2)	C11—C10—H10	120.6
O1—C2—C1	119.6 (2)	C10—C11—C12	119.5 (2)
C3—C2—C1	121.1 (2)	C10—C11—H11	120.2
C7—C3—C4	104.5 (2)	C12—C11—H11	120.2
C7—C3—C2	123.3 (2)	C11—C12—C13	121.2 (2)
C4—C3—C2	132.2 (2)	C11—C12—S1	120.17 (19)
N1—C4—C3	111.3 (2)	C13—C12—S1	118.66 (19)
N1—C4—C5	113.6 (2)	C14—C13—C12	119.5 (2)
C3—C4—C5	135.0 (2)	C14—C13—H13	120.3
O2—C5—C4	120.9 (2)	C12—C13—H13	120.3
O2—C5—C6	121.8 (2)	C13—C14—C9	119.2 (2)
C4—C5—C6	117.2 (2)	C13—C14—H14	120.4
C5—C6—H6A	109.5	C9—C14—H14	120.4

C4—N1—N2—C7	−2.5 (3)	C4—C3—C7—C8	174.8 (3)
C4—N1—N2—C9	170.7 (2)	C2—C3—C7—C8	−3.5 (5)
O1—C2—C3—C7	11.2 (4)	N1—N2—C9—C10	57.7 (3)
C1—C2—C3—C7	−167.1 (3)	C7—N2—C9—C10	−130.3 (3)
O1—C2—C3—C4	−166.6 (3)	N1—N2—C9—C14	−121.2 (3)
C1—C2—C3—C4	15.0 (5)	C7—N2—C9—C14	50.9 (4)
N2—N1—C4—C3	1.4 (3)	C14—C9—C10—C11	−0.5 (4)
N2—N1—C4—C5	−175.3 (2)	N2—C9—C10—C11	−179.4 (2)
C7—C3—C4—N1	0.1 (3)	C9—C10—C11—C12	−0.6 (4)
C2—C3—C4—N1	178.3 (3)	C10—C11—C12—C13	1.4 (4)
C7—C3—C4—C5	175.9 (3)	C10—C11—C12—S1	−178.3 (2)
C2—C3—C4—C5	−6.0 (5)	O4—S1—C12—C11	−10.3 (3)
N1—C4—C5—O2	148.1 (3)	O3—S1—C12—C11	−139.6 (2)
C3—C4—C5—O2	−27.6 (5)	N3—S1—C12—C11	104.9 (2)
N1—C4—C5—C6	−29.0 (4)	O4—S1—C12—C13	169.9 (2)
C3—C4—C5—C6	155.3 (3)	O3—S1—C12—C13	40.7 (3)
N1—N2—C7—C3	2.7 (3)	N3—S1—C12—C13	−74.9 (2)
C9—N2—C7—C3	−169.8 (2)	C11—C12—C13—C14	−1.1 (4)
N1—N2—C7—C8	−174.3 (2)	S1—C12—C13—C14	178.6 (2)
C9—N2—C7—C8	13.2 (4)	C12—C13—C14—C9	0.0 (4)
C4—C3—C7—N2	−1.6 (3)	C10—C9—C14—C13	0.8 (4)
C2—C3—C7—N2	−179.9 (2)	N2—C9—C14—C13	179.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H31···O2^{i}	0.88 (1)	2.03 (1)	2.864 (3)	159 (3)
N3—H32···O4^{ii}	0.88 (1)	2.06 (1)	2.933 (3)	170 (3)
C1—H1*C*···O3^{i}	0.98	2.55	3.446 (3)	151
C10—H10···O1^{iii}	0.95	2.51	3.314 (3)	142
C14—H14···O1^{iv}	0.95	2.54	3.414 (3)	153

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