Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)benzenesulfonamide

Yun-Fa Zheng, Chun-Niu Zhang and Ming-Hua Yang*

Department of Chemistry, Lishui University, Lishui 323000, People's Republic of China
Correspondence e-mail: xraylab@hotmail.com

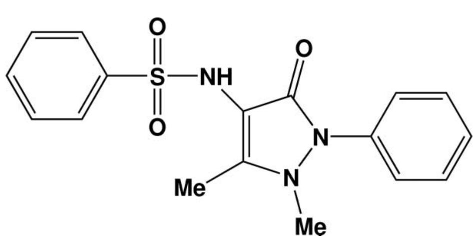
Received 17 June 2007; accepted 19 June 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.140; data-to-parameter ratio = 14.1.

Molecules of the title compound, $C_{17}H_{17}N_3O_3S$, form centrosymmetric hydrogen-bonded dimers *via* N—H···O hydrogen bonds.

Related literature

For related literature, see: El-Naggar *et al.* (1981); Lenarcik *et al.* (1980); Talley *et al.* (2000).



Experimental

Crystal data

$C_{17}H_{17}N_3O_3S$
M_r = 343.40
Triclinic, $P\overline{1}$
a = 9.085 (3) Å
b = 9.799 (3) Å
c = 9.809 (4) Å
α = 70.333 (4)°
β = 76.301 (2)°
γ = 87.991 (5)°
V = 798.0 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 291 (2) K
0.30 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.941, T_{max} = 0.952
8297 measured reflections
3125 independent reflections
2676 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.140
S = 1.05
3125 reflections
222 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1^{i}	0.97 (3)	1.86 (3)	2.789 (2)	160 (2)

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

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

The authors are grateful to the Natural Science Foundation of Zhejiang Province (No. M203052) and the Research Foundation of Lishui University (No. FC06002) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2397).

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Lenarcik, B., Wisniewski, M. & Gabryszewski, M. (1980). *Pol. J. Chem.* **54**, 1869–1874.
Talley, J. J., Brown, D. L., Carter, J. S., Graneto, M. J., Koboldt, C. M., Masferrer, J. L., Perkins, W. E., Rogers, R. S., Shaffer, A. F., Zhang, Y. Y., Zweifel, B. S. & Seibert, K. (2000). *J. Med. Chem.* **43**, 775–777.

 doi:10.1107/S1600536807029881

supplementary materials

Acta Cryst. (2007). E**63**, o3293 [**doi:10.1107/S1600536807029881**]

N-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)benzenesulfonamide

Y.-F. Zheng, C.-N. Zhang and M.-H. Yang

Comment

Benzenesulfonamides are very important intermediates in the organic synthesis and were widely used for the synthesis of medicinal and pesticidal compounds (Talley *et al.*, 2000). 4-Aminophenazone and its derivatives are also very important compounds in pharmacology and biochemistry (El-Naggar *et al.*, 1981; Lenarcik *et al.*, 1980). Recently we have synthesized the title compound, a new benzenesulfonamide containing the aminophenazone component and report here its crystal structure.

All bond lengths and angles have normal values. The dihedral angle between the pyrazol and the two phenyl rings are 51.23 (7)° and 30.21 (12)°, respectively. In the structure there is a N—H···O hyrogen bond (N3—H3A···O1^{i}, i: $2 - x$, $1 - y$, $2 - z$) it links two molecules to form a dimer (Fig. 2). It should be indicated that weak intermolecular C—H···O interactions (C3—H3···O2^{ii}, C6—H6···O3^{iii}, C10—H10A···O2^{iv} and C13—H13···O2^{iv}, ii: $2 - x$, $1 - y$, $1 - z$; iii: $-1 + x$, y, z; iv: $2 - x$, $-y$, $2 - z$) further connect the dimers.

Experimental

Under nitrogen, 4-amino-1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (2.03 g, 10 mmol) was dissolved in 50 ml CH_2Cl_2, and then Et_3N (10 mmol) and benzenesulfonyl chloride (1.77 g, 10 mmol) were added dropwise to the above solution. The resulting mixture was refluxed for 6 h. 15 ml hydrochloric acid (0.1 *M*) was added to the reaction mixture and then the organic layer was separated. The aqueous layer was extracted with ethyl acetate (3 *X* 5 ml). The combined organic layer was washed with the 10% $NaHCO_3$ and water. The crude product was obtained by removing the solvent *in vacuo*. The crude product was further purified by washing it with a solution of CH_2Cl_2 and hexane (1:1). A white solid was obtained in 92% yield (3.30 g). Colourless single crystals suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol (4:1) by slow evaporation of the solvent at room temperature over a period of about a week.

Refinement

H atoms bonded to N atoms were located in a difference map and refined with distance restraints of N—H = 0.97 (3) Å, and with U_{iso}(H) = 1.2U_{eq}(N). Other H atoms were positioned geometrically and refined using a riding model (including free rotation about the methyl C—C bond), with C—H = 0.93–0.96 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures

Fig. 1. The molecular structure of (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

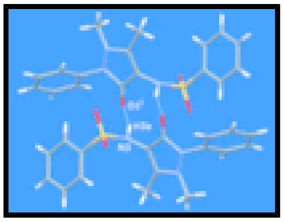

Fig. 2. View of the dimer of (I) (symmetry code: (i) $2 - x$, $1 - y$, $2 - z$)

N-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)benzenesulfonamide

Crystal data

$C_{17}H_{17}N_3O_3S$	$Z = 2$
$M_r = 343.40$	$F_{000} = 360$
Triclinic, $P\bar{1}$	$D_x = 1.429$ Mg m^{-3}
Hall symbol: -P 1	Mo *K*α radiation λ = 0.71073 Å
$a = 9.085$ (3) Å	Cell parameters from 3641 reflections
$b = 9.799$ (3) Å	θ = 2.6–28.0°
$c = 9.809$ (4) Å	$\mu = 0.22$ mm^{-1}
α = 70.333 (4)°	$T = 291$ (2) K
β = 76.301 (2)°	Block, colourless
γ = 87.991 (5)°	0.30 × 0.26 × 0.24 mm
$V = 798.0$ (5) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3125 independent reflections
Radiation source: sealed tube	2676 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 291(2)$ K	$\theta_{max} = 26.0°$
φ and ω scans	$\theta_{min} = 2.2°$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -10 \rightarrow 11$
$T_{min} = 0.941$, $T_{max} = 0.952$	$k = -12 \rightarrow 12$
8297 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

$wR(F^2) = 0.140$

$S = 1.05$

3125 reflections

222 parameters

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0837P)^2 + 0.1646P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.56$ e Å^{-3}

$\Delta\rho_{min} = -0.46$ e Å^{-3}

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.

Least-squares planes (*x*,*y*,*z* in crystal coordinates) and deviations from them (* indicates atom used to define plane)

4.5464 (0.0079) x + 8.5127 (0.0055) y + 5.2985 (0.0085) z = 11.0166 (0.0059)

* 0.0041 (0.0014) C1 * −0.0073 (0.0017) C2 * 0.0008 (0.0018) C3 * 0.0089 (0.0018) C4 * −0.0120 (0.0018) C5 * 0.0055 (0.0016) C6

Rms deviation of fitted atoms = 0.0073

− 2.9653 (0.0085) x + 8.2368 (0.0059) y + 5.6491 (0.0083) z = 5.5778 (0.0128)

Angle to previous plane (with approximate e.s.d.) = 51.23 (0.07)

* 0.0418 (0.0012) N2 * −0.0347 (0.0012) N1 * 0.0147 (0.0012) C7 * 0.0113 (0.0012) C8 * −0.0331 (0.0013) C9

Rms deviation of fitted atoms = 0.0296

6.4582 (0.0077) x − 4.7433 (0.0095) y − 4.3399 (0.0100) z = 1.7354 (0.0208)

Angle to previous plane (with approximate e.s.d.) = 30.21 (0.12)

* −0.0014 (0.0016) C12 * 0.0066 (0.0017) C13 * −0.0061 (0.0019) C14 * 0.0004 (0.0020) C15 * 0.0048 (0.0021) C16 * −0.0042 (0.0018) C17

Rms deviation of fitted atoms = 0.0045

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 > 2sigma(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	0.6807 (2)	0.4957 (2)	0.6995 (2)	0.0302 (4)
C2	0.7550 (3)	0.5468 (3)	0.5516 (3)	0.0413 (5)
H2	0.8501	0.5146	0.5192	0.050*
C3	0.6865 (3)	0.6467 (3)	0.4512 (3)	0.0526 (7)
H3	0.7363	0.6826	0.3508	0.063*
C4	0.5465 (3)	0.6932 (3)	0.4983 (3)	0.0538 (7)
H4	0.5021	0.7615	0.4302	0.065*
C5	0.4707 (3)	0.6393 (3)	0.6459 (3)	0.0490 (6)
H5	0.3738	0.6688	0.6768	0.059*
C6	0.5384 (3)	0.5413 (2)	0.7487 (3)	0.0383 (5)
H6	0.4889	0.5066	0.8492	0.046*
C7	0.8830 (2)	0.4196 (2)	0.8417 (2)	0.0288 (4)
C8	0.8730 (2)	0.3155 (2)	0.9876 (2)	0.0296 (4)
C9	0.7415 (2)	0.2332 (2)	1.0308 (2)	0.0305 (4)
C10	0.6867 (3)	0.1048 (2)	1.1652 (3)	0.0421 (5)
H10A	0.6986	0.0187	1.1385	0.063*
H10B	0.5815	0.1132	1.2074	0.063*
H10C	0.7445	0.0992	1.2371	0.063*
C11	0.5614 (3)	0.1922 (2)	0.8902 (3)	0.0388 (5)
H11A	0.6226	0.1302	0.8445	0.058*
H11B	0.5046	0.2516	0.8229	0.058*
H11C	0.4927	0.1340	0.9804	0.058*
C12	1.1464 (2)	0.0924 (2)	1.2053 (2)	0.0309 (4)
C13	1.0703 (3)	−0.0376 (2)	1.2324 (3)	0.0417 (5)
H13	1.0193	−0.0476	1.1643	0.050*
C14	1.0708 (3)	−0.1510 (3)	1.3601 (3)	0.0546 (7)
H14	1.0183	−0.2380	1.3799	0.065*
C15	1.1490 (4)	−0.1368 (3)	1.4594 (3)	0.0628 (8)
H15	1.1499	−0.2147	1.5455	0.075*
C16	1.2256 (4)	−0.0086 (4)	1.4322 (3)	0.0618 (8)
H16	1.2785	−0.0002	1.4996	0.074*
C17	1.2245 (3)	0.1080 (3)	1.3052 (3)	0.0432 (5)
H17	1.2753	0.1955	1.2869	0.052*
N1	0.7521 (2)	0.39254 (18)	0.80364 (19)	0.0310 (4)
N2	0.6594 (2)	0.28519 (18)	0.9251 (2)	0.0328 (4)
N3	0.9785 (2)	0.3078 (2)	1.0760 (2)	0.0338 (4)
H3A	0.981 (3)	0.384 (3)	1.117 (3)	0.041*
O1	0.98010 (17)	0.51793 (16)	0.76107 (17)	0.0365 (4)
O2	1.1496 (2)	0.18380 (19)	0.92410 (18)	0.0470 (4)
O3	1.25871 (18)	0.34497 (18)	1.0265 (2)	0.0458 (4)
S1	1.14382 (6)	0.24086 (5)	1.04167 (5)	0.03147 (18)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0369 (11)	0.0247 (9)	0.0314 (10)	−0.0027 (7)	−0.0159 (8)	−0.0069 (8)
C2	0.0375 (12)	0.0478 (13)	0.0339 (12)	−0.0046 (10)	−0.0065 (9)	−0.0086 (10)
C3	0.0635 (17)	0.0549 (15)	0.0322 (12)	−0.0076 (13)	−0.0170 (12)	−0.0008 (11)
C4	0.0664 (18)	0.0478 (14)	0.0497 (15)	0.0082 (12)	−0.0342 (14)	−0.0065 (12)
C5	0.0490 (14)	0.0466 (13)	0.0613 (16)	0.0158 (11)	−0.0271 (12)	−0.0227 (12)
C6	0.0403 (12)	0.0391 (11)	0.0371 (12)	0.0031 (9)	−0.0128 (9)	−0.0126 (9)
C7	0.0273 (10)	0.0279 (9)	0.0341 (10)	0.0018 (7)	−0.0091 (8)	−0.0129 (8)
C8	0.0300 (10)	0.0321 (10)	0.0287 (10)	0.0028 (8)	−0.0089 (8)	−0.0116 (8)
C9	0.0317 (10)	0.0293 (10)	0.0298 (10)	0.0040 (8)	−0.0078 (8)	−0.0093 (8)
C10	0.0444 (13)	0.0346 (11)	0.0381 (12)	−0.0008 (10)	−0.0065 (10)	−0.0025 (9)
C11	0.0360 (11)	0.0341 (11)	0.0458 (13)	−0.0061 (9)	−0.0106 (9)	−0.0115 (9)
C12	0.0262 (10)	0.0336 (10)	0.0327 (10)	0.0049 (8)	−0.0089 (8)	−0.0104 (8)
C13	0.0342 (12)	0.0386 (12)	0.0526 (14)	0.0019 (9)	−0.0129 (10)	−0.0140 (10)
C14	0.0431 (14)	0.0345 (12)	0.0687 (18)	0.0035 (10)	−0.0008 (12)	−0.0038 (12)
C15	0.0652 (19)	0.0595 (17)	0.0444 (15)	0.0221 (14)	−0.0079 (13)	0.0018 (13)
C16	0.073 (2)	0.075 (2)	0.0406 (14)	0.0200 (16)	−0.0292 (14)	−0.0141 (14)
C17	0.0441 (13)	0.0488 (13)	0.0391 (12)	0.0042 (10)	−0.0168 (10)	−0.0134 (10)
N1	0.0319 (9)	0.0278 (8)	0.0302 (9)	−0.0056 (7)	−0.0101 (7)	−0.0031 (7)
N2	0.0298 (9)	0.0278 (8)	0.0357 (10)	−0.0060 (7)	−0.0097 (7)	−0.0020 (7)
N3	0.0360 (10)	0.0370 (9)	0.0362 (10)	0.0110 (7)	−0.0164 (8)	−0.0181 (8)
O1	0.0312 (8)	0.0389 (8)	0.0364 (8)	−0.0087 (6)	−0.0061 (6)	−0.0091 (6)
O2	0.0589 (11)	0.0560 (10)	0.0328 (9)	0.0158 (8)	−0.0136 (8)	−0.0228 (8)
O3	0.0339 (9)	0.0447 (9)	0.0508 (10)	−0.0080 (7)	−0.0027 (7)	−0.0100 (7)
S1	0.0311 (3)	0.0340 (3)	0.0288 (3)	0.0027 (2)	−0.0073 (2)	−0.0100 (2)

Geometric parameters (Å, °)

C1—C2	1.372 (3)	C11—N2	1.467 (3)
C1—C6	1.383 (3)	C11—H11A	0.9600
C1—N1	1.435 (2)	C11—H11B	0.9600
C2—C3	1.383 (3)	C11—H11C	0.9600
C2—H2	0.9300	C12—C13	1.384 (3)
C3—C4	1.364 (4)	C12—C17	1.388 (3)
C3—H3	0.9300	C12—S1	1.771 (2)
C4—C5	1.374 (4)	C13—C14	1.367 (4)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.385 (3)	C14—C15	1.378 (5)
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C15—C16	1.372 (5)
C7—O1	1.244 (2)	C15—H15	0.9300
C7—N1	1.387 (3)	C16—C17	1.379 (4)
C7—C8	1.434 (3)	C16—H16	0.9300
C8—C9	1.362 (3)	C17—H17	0.9300
C8—N3	1.422 (3)	N1—N2	1.406 (2)
C9—N2	1.369 (3)	N3—S1	1.6246 (19)

C9—C10	1.478 (3)	N3—H3A	0.97 (3)
C10—H10A	0.9600	O2—S1	1.4313 (17)
C10—H10B	0.9600	O3—S1	1.4320 (17)
C10—H10C	0.9600		
C2—C1—C6	121.0 (2)	N2—C11—H11C	109.5
C2—C1—N1	119.1 (2)	H11A—C11—H11C	109.5
C6—C1—N1	119.93 (19)	H11B—C11—H11C	109.5
C1—C2—C3	119.1 (2)	C13—C12—C17	120.7 (2)
C1—C2—H2	120.4	C13—C12—S1	119.79 (17)
C3—C2—H2	120.4	C17—C12—S1	119.47 (17)
C4—C3—C2	120.5 (2)	C14—C13—C12	119.5 (2)
C4—C3—H3	119.8	C14—C13—H13	120.2
C2—C3—H3	119.8	C12—C13—H13	120.2
C3—C4—C5	120.3 (2)	C13—C14—C15	120.1 (3)
C3—C4—H4	119.8	C13—C14—H14	119.9
C5—C4—H4	119.8	C15—C14—H14	119.9
C4—C5—C6	120.1 (3)	C16—C15—C14	120.5 (3)
C4—C5—H5	120.0	C16—C15—H15	119.8
C6—C5—H5	120.0	C14—C15—H15	119.8
C1—C6—C5	119.0 (2)	C15—C16—C17	120.3 (3)
C1—C6—H6	120.5	C15—C16—H16	119.8
C5—C6—H6	120.5	C17—C16—H16	119.8
O1—C7—N1	123.91 (19)	C16—C17—C12	118.8 (3)
O1—C7—C8	131.31 (18)	C16—C17—H17	120.6
N1—C7—C8	104.75 (16)	C12—C17—H17	120.6
C9—C8—N3	125.92 (19)	C7—N1—N2	109.51 (16)
C9—C8—C7	109.00 (17)	C7—N1—C1	125.59 (16)
N3—C8—C7	124.93 (18)	N2—N1—C1	118.18 (16)
C8—C9—N2	109.17 (17)	C9—N2—N1	107.01 (16)
C8—C9—C10	129.3 (2)	C9—N2—C11	123.24 (17)
N2—C9—C10	121.52 (19)	N1—N2—C11	116.90 (17)
C9—C10—H10A	109.5	C8—N3—S1	121.88 (14)
C9—C10—H10B	109.5	C8—N3—H3A	118.0 (15)
H10A—C10—H10B	109.5	S1—N3—H3A	111.9 (15)
C9—C10—H10C	109.5	O2—S1—O3	119.74 (11)
H10A—C10—H10C	109.5	O2—S1—N3	107.71 (10)
H10B—C10—H10C	109.5	O3—S1—N3	109.03 (10)
N2—C11—H11A	109.5	O2—S1—C12	107.54 (10)
N2—C11—H11B	109.5	O3—S1—C12	106.77 (10)
H11A—C11—H11B	109.5	N3—S1—C12	105.13 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···O1^{i}	0.97 (3)	1.86 (3)	2.789 (2)	160 (2)

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

Fig. 1

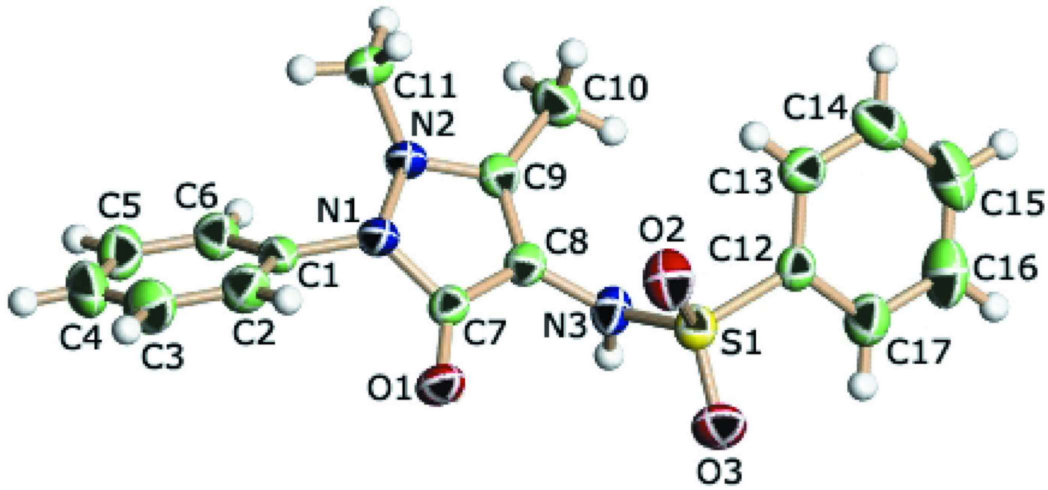
C11
N2
C10
C9
C14
C13
C15
C5
C6
N1
C8
O2
C12
C16
C1
C4
C3
C2
C7
N3
S1
C17
O1
O3

Fig. 2