Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(5*S*)-1,4-Bis{[(1*E*)-4-methylbenzylidene]-amino}-5-(thien-2-yl)pyrrolidin-2-one

Deepak Chopra,[a]* K. N. Venugopala[b] and G. K. Rao[b]

[a]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, Karnataka, India, and [b]Department of Pharmaceutical Chemistry, Al-Ameen College of Pharmacy, Bangalore 560 027, Karnataka, India
Correspondence e-mail: deepak@sscu.iisc.ernet.in

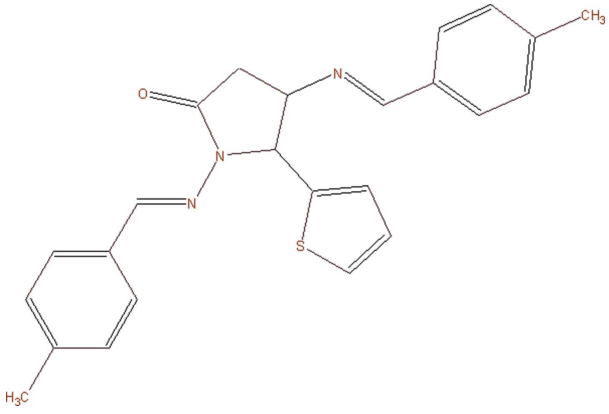
Received 25 April 2007; accepted 1 May 2007

Key indicators: single-crystal X-ray study; T = 290 K; mean σ(C–C) = 0.004 Å; R factor = 0.062; wR factor = 0.140; data-to-parameter ratio = 15.5.

In the crystal structure of the title compound, $C_{25}H_{24}SN_4O_2$, the pyrrole ring adopts an envelope conformation. Each of the 4-methylbenzylidene units is planar and they make angles of 12.26 (8) and 85.46 (10)° with the four coplanar atoms of the pyrrole ring. The thiophene ring makes an angle of 75.06 (9)° with this plane. The crystal structure is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds.

Related literature

For the pharmaceutical applications of pyrrolidinone derivatives, see: Malawska *et al.* (1982, 1988); Stadnicka *et al.* (1991).



Experimental

Crystal data

$C_{25}H_{24}N_4O_2S$
M_r = 444.55
Monoclinic, $P2_1/n$
a = 13.777 (3) Å
b = 7.6072 (14) Å
c = 23.431 (4) Å
β = 104.167 (3)°
V = 2381.0 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.16 mm^{-1}
T = 290 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.920, T_{max} = 0.968
16794 measured reflections
4510 independent reflections
2601 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.140
S = 1.04
4510 reflections
291 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O1^{i}	0.98	2.33	3.299 (3)	168
N3—H14···O2^{i}	0.86	2.13	2.922 (2)	152
C1—H1···O2^{ii}	0.93	2.42	3.337 (4)	168

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *PLATON* (Spek, 2003).

We thank Professor T. N. Guru Row and the Department of Science and Technology for data collection on the CCD facility under the IRHPA–DST scheme. DC thanks CSIR, India, and IISc for a Senior Research Fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2306).

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Bruker (2000). *SMART* (Version 5.628) and *SAINT* (Version 6.45a). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Malawska, B., Gorczyca, M., Cebo, B. & Krupinska, J. (1988). *Pol. J. Pharmacol. Pharm.* **40**, 173–181.
Malawska, B., Gorczyca, M., Chojnacka-Wojcik, E. & Tatarczynska, E. (1982). *Pol. J. Pharmacol. Pharm.* **34**, 373–382.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stadnicka, K., Ciechanowicz-Rutkowska, M. & Malawska, B. (1991). *Acta Cryst.* B**47**, 267–272.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

  doi:10.1107/S1600536807021587

supplementary materials

Acta Cryst. (2007). E**63**, o2840 [**doi:10.1107/S1600536807021587**]

(5*S*)-1,4-Bis{[(1*E*)-4-methylbenzylidene]amino}-5-(thien-2-yl)pyrrolidin-2-one

D. Chopra, K. N. Venugopala and G. K. Rao

Comment

2-Pyrrolidinone and its derivatives find widespread applications in drugs. These exhibit antidepressant or analeptic activity which affects the central nervous system. The alkyl-, aryl- and *N*-methylamine derivatives of 2-pyrrolidinone have been found to possess analgesic and anti-inflammatory activities (Malawska *et al.*, 1982). Further, a series of N-(β-hydroxy-γ-aminopropyl)-2-pyrrolidinones provided treatment for cardiovascular problems (Malawska *et al.*, 1988). One such important compound is the anti-arrhythmic and hypotensive agent 1-[2-Hydroxy-3-(4-phenyl-l- piperazinyl)propyl]pyrrolidin-2-one (Stadnicka *et al.*, 1991).

In this paper we report the crystal structure of the pyrrolidinone derivative, (I). The five membered pyrrole ring exists in an envelope conformation with the atom C11 deviating by -0.379 (2)Å from the mean plane passing through the atoms C9—C10—C12—N4. The dihedral angle between the planes N1—C7—C1/C6—C26 and C13—O1—N3—N2—C15—C21—C27 with the thiophene ring are 87.2 (1)° and 58.3 (1)° respectively. The C—N bond lengths are different, indicating that the nitrogen atoms exist in a different electronic environment. The crystal structure is stabilized by N—H···O and C—H···O hydrogen bonds (involving H14 and H12 connected to chiral carbon C12) along the crystallographic screw 'b' axis, further stablilized by a C—H···O interaction (involving H1, Figure 2).

Experimental

Methyl 4-acetoxybenzothiophen-6-carboxylate (25 g, 0.1 mol) and hydrazine hydrate 14.2 ml were heated gently under reflux for 10 min. Sufficient quantity of absolute alcohol was added to get a clear solution (about 10 ml). The resulting solution was refluxed for 3 h, ethanol distilled off and the product cooled. Crystals of acid hydrazide were filtered and recrystalised from ethanol. The product was isolated as white fluffy mass with yield of 16.26 g (78.20%). A mixture of 4-hydroxy benzothiophen-6-carboxhydrazide 0.208 g (0.001 mol) and *p*-methyl benzaldehyde 0.24 g (0.002 mol) in 50 ml of alcohol was refluxed for 2 hrs to yield the title compound which is recrystallised from ethanol to obtain crystals of 2-(2-thienyl)-1-(*p*-tolualdimino)-3-[*N*-(*p*-tolualdimino] carboxamido-1(H)-pyrrolidine-5-one 0.372 g (83.78%).

Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.98 Å, U_{iso} = 1.2U_{eq} (C) for CH, 0.97 Å, U_{iso} = 1.2U_{eq} (C) for CH_2, 0.96 Å, U_{iso} = 1.2U_{eq} (C) for CH_3 atoms and 0.82 Å, U_{iso} = 1.2U_{eq} (N) for the NH groups.

Figures

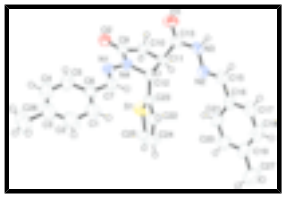


Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids.

Fig. 2. A packing diagram of (I). The dotted lines depict N—H···O and C—H···O hydrogen bonds.

(5*S*)-1,4-Bis{[(1E)-4-methylbenzylidene]amino}-5-(thien-2-yl)pyrrolidin-2-one

Crystal data

$C_{25}H_{24}N_4O_2S$	F_{000} = 936
M_r = 444.55	D_x = 1.240 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 750 reflections
a = 13.777 (3) Å	θ = 1.4–25.8º
b = 7.6072 (14) Å	μ = 0.16 mm^{-1}
c = 23.431 (4) Å	T = 290 (2) K
β = 104.167 (3)º	Blocks, pale yellow
V = 2381.0 (8) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4510 independent reflections
Radiation source: fine-focus sealed tube	2601 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.057
T = 290(2) K	θ_{max} = 25.7º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	h = −16→16
T_{min} = 0.920, T_{max} = 0.968	k = −8→9
16794 measured reflections	l = −28→28

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.062$

$wR(F^2) = 0.140$

$S = 1.04$

4510 reflections

291 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0582P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.21$ e Å^{-3}

$\Delta\rho_{min} = -0.19$ e Å^{-3}

Extinction correction: none

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.06393 (6)	0.02880 (10)	0.06412 (3)	0.0722 (3)
O1	0.21489 (16)	−0.1631 (3)	0.27430 (8)	0.0780 (7)
O2	0.21631 (14)	−0.3454 (2)	0.10967 (7)	0.0625 (5)
N1	0.24290 (15)	−0.0011 (3)	0.08651 (9)	0.0477 (5)
N2	0.05556 (16)	0.1972 (3)	0.25946 (9)	0.0505 (6)
N3	0.12607 (16)	0.0747 (3)	0.28518 (8)	0.0550 (6)
N4	0.18454 (15)	−0.0548 (3)	0.12347 (8)	0.0443 (5)
C1	0.3088 (2)	0.3944 (4)	0.02116 (13)	0.0703 (9)
C2	0.3545 (3)	0.4513 (5)	−0.02215 (14)	0.0854 (10)
C3	0.4016 (3)	0.3363 (6)	−0.05141 (14)	0.0830 (11)
C4	0.4021 (2)	0.1619 (6)	−0.03596 (15)	0.0889 (11)
C5	0.3563 (2)	0.1024 (4)	0.00675 (13)	0.0749 (9)
C6	0.30875 (19)	0.2201 (4)	0.03586 (11)	0.0520 (7)
C7	0.25474 (19)	0.1619 (4)	0.07904 (10)	0.0504 (7)
C9	0.1716 (2)	−0.2311 (4)	0.12955 (10)	0.0481 (7)
C10	0.0960 (2)	−0.2550 (3)	0.16454 (11)	0.0562 (7)

C11	0.08397 (19)	−0.0758 (3)	0.19067 (10)	0.0450 (6)
C12	0.12002 (17)	0.0571 (3)	0.14987 (9)	0.0413 (6)
C13	0.1477 (2)	−0.0613 (4)	0.25350 (11)	0.0504 (7)
C15	0.0506 (2)	0.3362 (4)	0.28833 (11)	0.0539 (7)
C16	−0.0205 (2)	0.4754 (4)	0.26443 (11)	0.0524 (7)
C17	−0.0213 (2)	0.6294 (4)	0.29555 (13)	0.0682 (9)
C18	−0.0852 (3)	0.7654 (4)	0.27262 (15)	0.0757 (9)
C19	−0.1503 (2)	0.7547 (4)	0.21827 (15)	0.0684 (9)
C20	−0.1500 (2)	0.6005 (4)	0.18697 (13)	0.0721 (9)
C21	−0.0874 (2)	0.4634 (4)	0.20952 (12)	0.0625 (8)
C22	0.0261 (2)	0.3155 (4)	0.09161 (13)	0.0642 (8)
C23	0.03622 (18)	0.1436 (3)	0.10518 (10)	0.0426 (6)
C24	−0.0625 (2)	0.3547 (5)	0.04874 (14)	0.0801 (10)
C25	−0.1175 (2)	0.2139 (4)	0.03023 (12)	0.0714 (9)
C26	0.4504 (3)	0.3978 (6)	−0.09953 (15)	0.1286 (16)
C27	−0.2178 (3)	0.9080 (5)	0.19279 (17)	0.1027 (12)
H1	0.2777	0.4757	0.0405	0.084*
H2	0.3532	0.5702	−0.0315	0.102*
H4	0.4342	0.0813	−0.0549	0.107*
H5	0.3575	−0.0166	0.0159	0.090*
H7	0.2292	0.2440	0.1009	0.060*
H10A	0.1188	−0.3410	0.1955	0.067*
H10B	0.0328	−0.2946	0.1396	0.067*
H11	0.0134	−0.0546	0.1897	0.054*
H12	0.1610	0.1480	0.1740	0.050*
H14	0.1566	0.0848	0.3217	0.066*
H15	0.0932	0.3497	0.3255	0.065*
H17	0.0219	0.6416	0.3326	0.082*
H18	−0.0840	0.8673	0.2947	0.091*
H20	−0.1931	0.5895	0.1498	0.086*
H21	−0.0898	0.3607	0.1877	0.075*
H22	0.0729	0.4001	0.1088	0.077*
H24	−0.0804	0.4677	0.0349	0.096*
H25	−0.1782	0.2162	0.0021	0.086*
H26A	0.4080	0.3687	−0.1373	0.193*
H26B	0.5140	0.3408	−0.0949	0.193*
H26C	0.4599	0.5228	−0.0968	0.193*
H27A	−0.2843	0.8860	0.1970	0.154*
H27B	−0.2193	0.9209	0.1518	0.154*
H27C	−0.1924	1.0138	0.2134	0.154*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0638 (5)	0.0668 (6)	0.0699 (5)	−0.0075 (4)	−0.0142 (4)	−0.0071 (4)
O1	0.0810 (16)	0.0805 (15)	0.0621 (13)	0.0295 (13)	−0.0027 (11)	0.0078 (11)
O2	0.0796 (14)	0.0566 (13)	0.0468 (10)	0.0190 (11)	0.0067 (10)	−0.0048 (9)
N1	0.0425 (13)	0.0582 (16)	0.0412 (12)	−0.0001 (11)	0.0083 (10)	−0.0031 (11)

N2	0.0503 (14)	0.0602 (15)	0.0409 (12)	0.0024 (12)	0.0110 (10)	−0.0040 (12)
N3	0.0608 (15)	0.0680 (16)	0.0330 (11)	0.0039 (13)	0.0052 (10)	−0.0007 (11)
N4	0.0441 (13)	0.0470 (14)	0.0417 (11)	0.0007 (11)	0.0105 (10)	−0.0031 (10)
C1	0.081 (2)	0.073 (2)	0.0643 (19)	−0.0034 (18)	0.0315 (17)	−0.0007 (17)
C2	0.105 (3)	0.086 (3)	0.071 (2)	−0.011 (2)	0.033 (2)	0.010 (2)
C3	0.075 (2)	0.121 (3)	0.057 (2)	−0.008 (2)	0.0234 (17)	0.009 (2)
C4	0.082 (3)	0.121 (3)	0.075 (2)	0.015 (2)	0.043 (2)	−0.005 (2)
C5	0.074 (2)	0.083 (2)	0.078 (2)	0.0114 (19)	0.0374 (19)	0.0056 (19)
C6	0.0448 (16)	0.066 (2)	0.0453 (15)	−0.0007 (15)	0.0122 (13)	0.0013 (14)
C7	0.0453 (16)	0.062 (2)	0.0450 (15)	−0.0027 (15)	0.0125 (12)	−0.0058 (14)
C9	0.0558 (18)	0.0473 (19)	0.0354 (14)	0.0046 (15)	−0.0002 (13)	−0.0029 (13)
C10	0.0670 (19)	0.0480 (17)	0.0512 (16)	−0.0044 (15)	0.0100 (14)	0.0001 (13)
C11	0.0456 (15)	0.0468 (16)	0.0426 (13)	−0.0010 (13)	0.0110 (12)	0.0008 (12)
C12	0.0416 (15)	0.0432 (15)	0.0374 (13)	−0.0048 (12)	0.0061 (11)	−0.0075 (11)
C13	0.0520 (17)	0.0547 (18)	0.0440 (15)	0.0000 (15)	0.0110 (13)	0.0049 (14)
C15	0.0521 (17)	0.066 (2)	0.0443 (15)	−0.0070 (16)	0.0134 (13)	−0.0059 (15)
C16	0.0504 (17)	0.0595 (19)	0.0532 (16)	−0.0063 (15)	0.0241 (14)	−0.0115 (14)
C17	0.071 (2)	0.077 (2)	0.0572 (17)	−0.0065 (19)	0.0165 (16)	−0.0166 (17)
C18	0.087 (3)	0.064 (2)	0.082 (2)	0.001 (2)	0.033 (2)	−0.0223 (19)
C19	0.059 (2)	0.071 (2)	0.084 (2)	0.0060 (17)	0.0345 (18)	−0.0041 (19)
C20	0.056 (2)	0.091 (3)	0.0673 (19)	0.0133 (19)	0.0117 (15)	−0.0103 (19)
C21	0.0567 (18)	0.070 (2)	0.0602 (18)	0.0030 (17)	0.0129 (15)	−0.0179 (16)
C22	0.064 (2)	0.0454 (19)	0.076 (2)	0.0028 (15)	0.0023 (16)	−0.0037 (15)
C23	0.0454 (15)	0.0446 (17)	0.0369 (13)	−0.0020 (13)	0.0084 (11)	−0.0084 (12)
C24	0.080 (2)	0.067 (2)	0.083 (2)	0.021 (2)	−0.0014 (19)	0.0165 (19)
C25	0.063 (2)	0.090 (2)	0.0512 (17)	0.0151 (19)	−0.0056 (15)	0.0024 (17)
C26	0.130 (4)	0.200 (5)	0.072 (2)	−0.033 (3)	0.055 (2)	0.013 (3)
C27	0.089 (3)	0.096 (3)	0.130 (3)	0.032 (2)	0.040 (2)	0.003 (2)

Geometric parameters (Å, °)

S1—C25	1.695 (3)	C21—C20	1.374 (4)
S1—C23	1.715 (2)	C21—H21	0.9300
O2—C9	1.222 (3)	C18—C19	1.369 (4)
N2—C15	1.266 (3)	C18—C17	1.379 (4)
N2—N3	1.374 (3)	C18—H18	0.9300
N4—C9	1.365 (3)	C1—C2	1.389 (4)
N4—N1	1.380 (3)	C1—H1	0.9300
N4—C12	1.472 (3)	C20—C19	1.384 (4)
N1—C7	1.268 (3)	C20—H20	0.9300
O1—C13	1.213 (3)	C25—C24	1.322 (4)
N3—C13	1.349 (3)	C25—H25	0.9300
N3—H14	0.8600	C19—C27	1.519 (4)
C15—C16	1.459 (4)	C5—C4	1.383 (4)
C15—H15	0.9300	C5—H5	0.9300
C12—C23	1.507 (3)	C17—H17	0.9300
C12—C11	1.554 (3)	C22—C24	1.410 (4)
C12—H12	0.9800	C22—H22	0.9300
C11—C10	1.520 (3)	C24—H24	0.9300

C11—C13	1.522 (3)	C2—C3	1.369 (4)
C11—H11	0.9800	C2—H2	0.9300
C23—C22	1.345 (3)	C4—C3	1.375 (4)
C6—C1	1.370 (4)	C4—H4	0.9300
C6—C5	1.384 (4)	C27—H27A	0.9600
C6—C7	1.464 (3)	C27—H27B	0.9600
C9—C10	1.486 (4)	C27—H27C	0.9600
C16—C17	1.381 (4)	C3—C26	1.521 (4)
C16—C21	1.390 (4)	C26—H26A	0.9600
C7—H7	0.9300	C26—H26B	0.9600
C10—H10A	0.9700	C26—H26C	0.9600
C10—H10B	0.9700		
C25—S1—C23	92.19 (15)	C16—C21—H21	119.4
C15—N2—N3	117.2 (2)	C19—C18—C17	121.9 (3)
C9—N4—N1	117.9 (2)	C19—C18—H18	119.1
C9—N4—C12	114.6 (2)	C17—C18—H18	119.1
N1—N4—C12	126.6 (2)	C6—C1—C2	120.9 (3)
C7—N1—N4	119.4 (2)	C6—C1—H1	119.6
C13—N3—N2	120.4 (2)	C2—C1—H1	119.6
C13—N3—H14	119.8	C21—C20—C19	121.5 (3)
N2—N3—H14	119.8	C21—C20—H20	119.2
N2—C15—C16	121.3 (2)	C19—C20—H20	119.2
N2—C15—H15	119.3	C24—C25—S1	111.7 (2)
C16—C15—H15	119.3	C24—C25—H25	124.1
N4—C12—C23	113.53 (18)	S1—C25—H25	124.1
N4—C12—C11	101.47 (18)	C18—C19—C20	117.2 (3)
C23—C12—C11	113.93 (19)	C18—C19—C27	121.2 (3)
N4—C12—H12	109.2	C20—C19—C27	121.6 (3)
C23—C12—H12	109.2	C4—C5—C6	120.0 (3)
C11—C12—H12	109.2	C4—C5—H5	120.0
C10—C11—C13	111.1 (2)	C6—C5—H5	120.0
C10—C11—C12	104.66 (19)	C18—C17—C16	121.2 (3)
C13—C11—C12	110.61 (19)	C18—C17—H17	119.4
C10—C11—H11	110.1	C16—C17—H17	119.4
C13—C11—H11	110.1	C23—C22—C24	113.3 (3)
C12—C11—H11	110.1	C23—C22—H22	123.3
C22—C23—C12	127.3 (2)	C24—C22—H22	123.3
C22—C23—S1	109.85 (19)	C25—C24—C22	112.9 (3)
C12—C23—S1	122.81 (19)	C25—C24—H24	123.6
C1—C6—C5	118.2 (3)	C22—C24—H24	123.6
C1—C6—C7	119.9 (3)	C3—C2—C1	121.5 (3)
C5—C6—C7	121.8 (3)	C3—C2—H2	119.2
O1—C13—N3	121.2 (2)	C1—C2—H2	119.2
O1—C13—C11	123.0 (3)	C3—C4—C5	122.2 (3)
N3—C13—C11	115.8 (2)	C3—C4—H4	118.9
O2—C9—N4	124.6 (3)	C5—C4—H4	118.9
O2—C9—C10	127.6 (3)	C19—C27—H27A	109.5
N4—C9—C10	107.8 (2)	C19—C27—H27B	109.5
C17—C16—C21	117.1 (3)	H27A—C27—H27B	109.5

C17—C16—C15	120.3 (3)	C19—C27—H27C	109.5
C21—C16—C15	122.6 (3)	H27A—C27—H27C	109.5
N1—C7—C6	119.7 (3)	H27B—C27—H27C	109.5
N1—C7—H7	120.1	C2—C3—C4	117.2 (3)
C6—C7—H7	120.1	C2—C3—C26	121.6 (4)
C9—C10—C11	105.8 (2)	C4—C3—C26	121.2 (4)
C9—C10—H10A	110.6	C3—C26—H26A	109.5
C11—C10—H10A	110.6	C3—C26—H26B	109.5
C9—C10—H10B	110.6	H26A—C26—H26B	109.5
C11—C10—H10B	110.6	C3—C26—H26C	109.5
H10A—C10—H10B	108.7	H26A—C26—H26C	109.5
C20—C21—C16	121.1 (3)	H26B—C26—H26C	109.5
C20—C21—H21	119.4		
C9—N4—N1—C7	178.6 (2)	C1—C6—C7—N1	170.1 (3)
C12—N4—N1—C7	9.8 (3)	C5—C6—C7—N1	−6.5 (4)
C15—N2—N3—C13	−168.5 (2)	O2—C9—C10—C11	166.2 (2)
N3—N2—C15—C16	178.9 (2)	N4—C9—C10—C11	−13.1 (3)
C9—N4—C12—C23	−106.6 (2)	C13—C11—C10—C9	−97.0 (2)
N1—N4—C12—C23	62.6 (3)	C12—C11—C10—C9	22.4 (3)
C9—N4—C12—C11	16.1 (2)	C17—C16—C21—C20	1.3 (4)
N1—N4—C12—C11	−174.74 (19)	C15—C16—C21—C20	−176.7 (3)
N4—C12—C11—C10	−22.7 (2)	C5—C6—C1—C2	0.5 (4)
C23—C12—C11—C10	99.7 (2)	C7—C6—C1—C2	−176.2 (3)
N4—C12—C11—C13	97.1 (2)	C16—C21—C20—C19	−1.2 (5)
C23—C12—C11—C13	−140.5 (2)	C23—S1—C25—C24	0.3 (3)
N4—C12—C23—C22	−110.8 (3)	C17—C18—C19—C20	0.2 (5)
C11—C12—C23—C22	133.7 (3)	C17—C18—C19—C27	−178.0 (3)
N4—C12—C23—S1	70.2 (3)	C21—C20—C19—C18	0.4 (5)
C11—C12—C23—S1	−45.3 (3)	C21—C20—C19—C27	178.6 (3)
C25—S1—C23—C22	−0.5 (2)	C1—C6—C5—C4	−0.1 (4)
C25—S1—C23—C12	178.6 (2)	C7—C6—C5—C4	176.6 (3)
N2—N3—C13—O1	174.4 (2)	C19—C18—C17—C16	−0.1 (5)
N2—N3—C13—C11	−4.7 (3)	C21—C16—C17—C18	−0.7 (4)
C10—C11—C13—O1	15.3 (4)	C15—C16—C17—C18	177.3 (3)
C12—C11—C13—O1	−100.5 (3)	C12—C23—C22—C24	−178.5 (3)
C10—C11—C13—N3	−165.6 (2)	S1—C23—C22—C24	0.6 (3)
C12—C11—C13—N3	78.6 (3)	S1—C25—C24—C22	0.0 (4)
N1—N4—C9—O2	8.2 (3)	C23—C22—C24—C25	−0.4 (4)
C12—N4—C9—O2	178.4 (2)	C6—C1—C2—C3	−0.4 (5)
N1—N4—C9—C10	−172.49 (19)	C6—C5—C4—C3	−0.6 (5)
C12—N4—C9—C10	−2.3 (3)	C1—C2—C3—C4	−0.2 (5)
N2—C15—C16—C17	−177.0 (3)	C1—C2—C3—C26	179.0 (3)
N2—C15—C16—C21	0.9 (4)	C5—C4—C3—C2	0.7 (5)
N4—N1—C7—C6	−174.4 (2)	C5—C4—C3—C26	−178.5 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12···O1^{i}	0.98	2.33	3.299 (3)	168

N3—H14···O2^{i}	0.86	2.13	2.922 (2)	152
C1—H1···O2^{ii}	0.93	2.42	3.337 (4)	168

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

Fig. 1

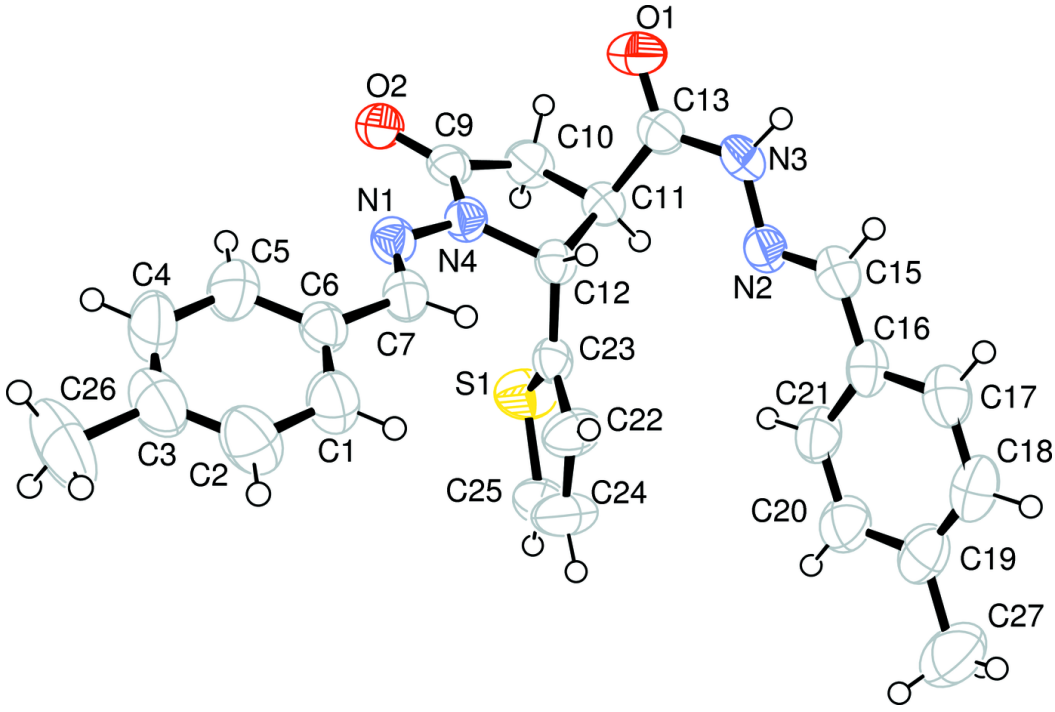

O1
O2
C9
C10
C13
N3
N1
C11
N4
C5
C4
C6
C12
N2
C15
C7
C16
C23
S1
C26
C21
C17
C22
C3
C2
C1
C18
C25
C24
C20
C19
C27

Fig. 2