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3-(4-Methyl-1,3-thiazol-5-yl)-1-[1′-(4-methyl-1,3-thiazol-5-yl)-2-oxo-2,3,2′,3′,5′,6′,7′,7a′-octahydro-1*H*-indole-3-spiro-3′-1*H*-pyrrolizin-2′-yl]-prop-2-en-1-one

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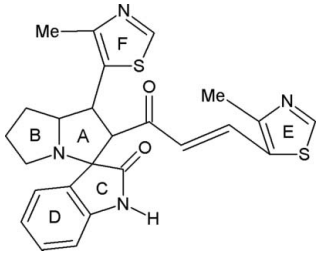
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.051; wR factor = 0.160; data-to-parameter ratio = 22.8.

In the title compound, $C_{25}H_{24}N_4O_2S_2$, one of the pyrrolidine rings in the pyrrolizine ring system adopts an envelope conformation, whereas the other ring adopts a twist conformation. The five-membered ring in the indolone ring system also adopts a twist conformation. The crystal packing is stabilized by N—H···N and C—H···O intermolecular hydrogen bonds, and C—H···π interactions. The N—H···N hydrogen bonds link the molecules into cyclic centrosymmetric $R_2^2(22)$ dimers.

Related literature

For general background, see: Chimirri *et al.* (1994); Farhanullah *et al.* (2004); Köysal *et al.* (2004); Kondo *et al.* (1990); Stylianakis *et al.* (2003). For ring conformations, see: Cremer & Pople (1975); Nardelli (1983). For related structures, see: Beddoes *et al.* (1986); Seshadri *et al.* (2003); Usha, Selvanayagam, Velmurugan, Ravikumar, Durga & Raghunathan (2005); Usha, Selvanayagam, Velmurugan, Ravikumar & Poornachandran (2005).



Experimental

Crystal data

$C_{25}H_{24}N_4O_2S_2$
M_r = 476.60
Monoclinic, $P2_1/c$
a = 10.8362 (3) Å
b = 9.0056 (2) Å
c = 24.7698 (5) Å
β = 95.272 (1)°
V = 2406.97 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 293 (2) K
0.26 × 0.21 × 0.17 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.939, T_{max} = 0.958
29274 measured reflections
6839 independent reflections
4872 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.160
S = 1.01
6839 reflections
300 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

Cg is the centroid of the S1/C25/C26/N27/C28 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N15—H15···N22^{i}	0.86	2.15	2.978 (2)	162
C7—H7···O1	0.98	2.56	3.005 (2)	108
C19—H19···O1	0.93	2.51	3.301 (2)	143
C28—H28···O1^{ii}	0.93	2.47	3.016 (3)	118
C24—H24*C*···*Cg*^{ii}	0.96	2.77	3.631 (3)	149

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2460).

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supplementary materials

Acta Cryst. (2007). E**63**, o4106-o4107 [**doi:10.1107/S1600536807045072**]

3-(4-Methyl-1,3-thiazol-5-yl)-1-[1'-(4-methyl-1,3-thiazol-5-yl)-2-oxo-2,3,2',3',5',6',7',7a'-octahydro-1*H*-indole-3-spiro-3'-1*H*-pyrrolizin-2'-yl]prop-2-en-1-one

P. Ramesh, S. Murugavel, A. SubbiahPandi, R. Murugan and S. S. Narayanan

Comment

The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids. Synthetic spiro-pyrrolidine derivatives have activity against aldose reductase enzyme which controls influenza (Stylianakis *et al.*, 2003). The pyrrolizidine alkaloids are well documented for their mutagenic, antineoplastic, carcinogenic, hepatoxic, and many pharmacological activities (Usha, Selvanayagam, Velmurugan, Ravikumar, Durga & Raghunathan, 2005); Usha, Selvanayagam, Velmurugan, Ravikumar & Poornachandran, 2005). Thiazole derivatives possess anti-inflammatory properties (Köysal *et al.*, 2004) and thiazole napthyridine derivatives exibit good antibactrial activity (Kondo *et al.*, 1990). A series of thiazole[3,4–9]benzimidazole derivatives have been evaluated *in vitro* as antitumor agents against 60 human tumor cell-lines (Chimirri *et al.*, 1994). Indole, being an integral part of many natural products of therapeutic importance, possesses potentially reactive sites for a variety of chemical reactions to generate molecular diversity (Farhanullah *et al.*, 2004). In view of this biological importance, the crystal structure of the title compound has been determined and the results are presented here.

In the molecule of the title compound (Fig.1), the C—C and C—N bond lengths in the pyrrolizine ring system are slightly longer than the values reported for similar structures (Seshadri *et al.*, 2003). This may be due to steric forces caused by the bulky substituents on the pyrrolizine ring system. The sum of angles at N1 of the pyrrolizine ring system (341.9°) is in accordance with sp^3 hybridization (Beddoes *et al.*, 1986), and the sum of the angles at N15 of the indole moiety (359.9°) is in accordance with sp^2 hybridization.

In the pyrrolizine ring system, the pyrrolidine ring A adopts an envelope conformation whereas the ring B adopts a twist conformation. The puckering parameters (q_2 and φ; Cremer & Pople, 1975) and the smallest displacement asymmetry parameter (Δ; Nardelli, 1983) are, for the ring A, q_2 = 0.371 (2) Å, φ = 79.0 (3)° and Δ_s(C6) = 4.6 (2)°; for the ring B, q_2 = 0.334 (3) Å, φ = 241.9 (4)°, Δ_2(C5)=6.7 (3)° and Δ_s(C3) = 8.5 (3)°. The pyrrolizine ring system is folded about the bridging N1—C5 bond, as observed in related structures (Usha, Selvanayagam, Velmurugan, Ravikumar, Durga & Raghunathan, 2005); Usha, Selvanayagam, Velmurugan, Ravikumar & Poornachandran, 2005). The five-membered ring C in the indolone ring system adopts a twist conformation, with puckering parameters q_2 = 0.074 (2) Å, φ = 133.5 (2)°, Δ_2(C14) = 2.4 (2)° and Δ_s(C16) = 1.0 (2)°.

The molecular structure is stabilized by intramolecular C—H···O interactions and the crystal packing is stabilized by C—H···O and N—H···N intermolecular hydrogen bonds. The molecules at (*x*, *y*, *z*) and (1 − *x*, 2 − *y*, 1 − *z*) are linked by N15—H15···N22 hydrogen bonds into cyclic centrosymmetric $R_2^2(22)$ dimers. The dimers are cross-linked *via* C—H···π interactions involving the S1/C25/C26/N27/C28 ring (centroid *Cg*).

Experimental

A solution of (1E,4E,6*Z*)-1,7-bis(methylthiazol-5-yl)-4-[(4-methylthiazol −5yl)methylene]hepta-1,6-diene-3,5-dione (1 mmol), isatin (1 mmol) and *L*-proline (1 mmol) in aqueous methonal (20 ml) was refluxed until the disappearence of starting materials as evidenced by TLC. The solvent was removed under reduced pressure and the crude product was purified by column-chromatography using petroleum ether/ethyl acetate (5:1) as eluent. The final product was recrystallized in ethanol and chloroform (2:8).

Refinement

H atoms were positioned geometrically (N—H = 0.86 Å and C—H = 0.93–0.98 Å) and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). *PLATON* (Spek, 2003) detected a solvent accessible void of approximately 75 Å^3 including the position of this peak. This void could have initially contained solvent molecules but these molecules have since evaporated from the structure without degradation of the crystal.

Figures

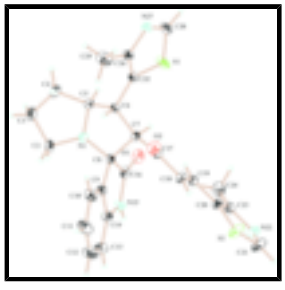


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

3-(4-methyl-1,3-thiazol-5-yl)-1-[1'-(4-methyl-1,3-thiazol-5-yl)-2-oxo- 2,3,2',3',5',6',7',7a'-octahydro-1*H*-indole-3-spiro-3'-1*H*-pyrrolizin-2'-yl]prop-2-en-1-one

Crystal data

$C_{25}H_{24}N_4O_2S_2$	F_{000} = 1000
M_r = 476.60	D_x = 1.315 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2850 reflections
a = 10.8362 (3) Å	θ = 2.0–25.5º
b = 9.0056 (2) Å	μ = 0.25 mm^{-1}
c = 24.7698 (5) Å	T = 293 (2) K
β = 95.272 (1)º	Block, colourless
V = 2406.97 (10) Å^3	0.26 × 0.21 × 0.17 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	6839 independent reflections
Radiation source: fine-focus sealed tube	4872 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 293(2)$ K	$\theta_{max} = 29.8°$
ω scans	$\theta_{min} = 1.7°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	$h = -15\rightarrow15$
$T_{min} = 0.939$, $T_{max} = 0.958$	$k = -12\rightarrow11$
29274 measured reflections	$l = -34\rightarrow34$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.160$	$w = 1/[\sigma^2(F_o^2) + (0.0799P)^2 + 0.9857P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.005$
6839 reflections	$\Delta\rho_{max} = 0.56$ e Å^{-3}
300 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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.

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 $F^2 > 2$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	0.40437 (6)	0.92038 (8)	0.18158 (2)	0.0692 (2)
S2	0.25147 (6)	1.26490 (8)	0.44224 (2)	0.0696 (2)
O1	0.48783 (12)	0.74268 (16)	0.36378 (6)	0.0506 (3)
O2	0.08715 (13)	0.96110 (17)	0.26782 (6)	0.0546 (4)
N1	0.30422 (15)	0.55545 (16)	0.29597 (6)	0.0408 (3)

C2	0.2387 (3)	0.4160 (3)	0.30022 (10)	0.0762 (8)
H2A	0.1512	0.4330	0.3031	0.091*
H2B	0.2725	0.3601	0.3316	0.091*
C3	0.2585 (3)	0.3356 (3)	0.24902 (10)	0.0665 (6)
H3A	0.3374	0.2838	0.2523	0.080*
H3B	0.1927	0.2645	0.2399	0.080*
C4	0.2573 (3)	0.4542 (3)	0.20783 (9)	0.0723 (8)
H4A	0.1736	0.4726	0.1919	0.087*
H4B	0.3086	0.4273	0.1793	0.087*
C5	0.30954 (19)	0.5913 (2)	0.23858 (7)	0.0445 (4)
H5	0.3960	0.6064	0.2312	0.053*
C6	0.23813 (16)	0.73706 (19)	0.23011 (6)	0.0344 (3)
H6	0.1492	0.7148	0.2264	0.041*
C7	0.26998 (14)	0.81582 (18)	0.28429 (6)	0.0316 (3)
H7	0.3536	0.8572	0.2843	0.038*
C8	0.27434 (15)	0.68763 (18)	0.32670 (6)	0.0330 (3)
C9	0.16479 (16)	0.6752 (2)	0.35981 (7)	0.0375 (4)
C10	0.03935 (18)	0.6653 (3)	0.34481 (9)	0.0525 (5)
H10	0.0095	0.6637	0.3084	0.063*
C11	−0.0415 (2)	0.6579 (3)	0.38492 (12)	0.0742 (7)
H11	−0.1265	0.6532	0.3755	0.089*
C12	0.0038 (3)	0.6573 (4)	0.43879 (12)	0.0817 (8)
H12	−0.0518	0.6503	0.4652	0.098*
C13	0.1288 (3)	0.6668 (3)	0.45480 (9)	0.0674 (7)
H13	0.1584	0.6659	0.4912	0.081*
C14	0.20798 (19)	0.6779 (2)	0.41451 (7)	0.0441 (4)
N15	0.33653 (16)	0.69457 (19)	0.42016 (6)	0.0472 (4)
H15	0.3812	0.6932	0.4507	0.057*
C16	0.38215 (16)	0.71317 (19)	0.37152 (7)	0.0379 (4)
C17	0.18303 (16)	0.94061 (19)	0.29627 (6)	0.0358 (3)
C18	0.21351 (17)	1.0351 (2)	0.34393 (7)	0.0415 (4)
H18	0.1489	1.0887	0.3568	0.050*
C19	0.32534 (17)	1.05114 (19)	0.37045 (7)	0.0391 (4)
H19	0.3895	0.9957	0.3581	0.047*
C20	0.35632 (18)	1.1463 (2)	0.41653 (7)	0.0423 (4)
C21	0.3649 (3)	1.3260 (3)	0.48850 (9)	0.0733 (7)
H21	0.3506	1.3987	0.5139	0.088*
N22	0.47284 (19)	1.2656 (2)	0.48645 (7)	0.0596 (5)
C23	0.46987 (19)	1.1618 (2)	0.44532 (7)	0.0450 (4)
C24	0.5839 (2)	1.0779 (3)	0.43622 (11)	0.0623 (6)
H24A	0.5622	0.9784	0.4252	0.093*
H24B	0.6378	1.0753	0.4692	0.093*
H24C	0.6255	1.1258	0.4084	0.093*
C25	0.26903 (16)	0.8213 (2)	0.18115 (6)	0.0374 (4)
C26	0.20859 (17)	0.8262 (2)	0.13094 (7)	0.0416 (4)
N27	0.26748 (17)	0.9047 (2)	0.09272 (6)	0.0494 (4)
C28	0.3701 (2)	0.9577 (3)	0.11439 (8)	0.0604 (6)
H28	0.4227	1.0132	0.0947	0.073*
C29	0.0868 (2)	0.7560 (4)	0.11396 (10)	0.0760 (8)

H29A	0.0998	0.6660	0.0945	0.114*
H29B	0.0373	0.8230	0.0910	0.114*
H29C	0.0448	0.7336	0.1455	0.114*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0752 (4)	0.0939 (5)	0.0364 (2)	−0.0519 (3)	−0.0055 (2)	0.0098 (3)
S2	0.0681 (4)	0.0850 (5)	0.0526 (3)	0.0197 (3)	−0.0103 (3)	−0.0331 (3)
O1	0.0369 (7)	0.0500 (8)	0.0625 (9)	0.0037 (6)	−0.0084 (6)	−0.0038 (6)
O2	0.0535 (8)	0.0560 (9)	0.0501 (8)	0.0122 (7)	−0.0174 (6)	−0.0026 (6)
N1	0.0555 (9)	0.0313 (7)	0.0350 (7)	0.0002 (6)	0.0006 (6)	−0.0035 (6)
C2	0.144 (3)	0.0380 (12)	0.0490 (12)	−0.0259 (14)	0.0224 (14)	−0.0036 (9)
C3	0.0973 (18)	0.0446 (12)	0.0562 (13)	−0.0152 (12)	−0.0011 (12)	−0.0089 (10)
C4	0.135 (2)	0.0408 (12)	0.0410 (10)	−0.0058 (13)	0.0063 (13)	−0.0106 (9)
C5	0.0597 (11)	0.0361 (9)	0.0395 (9)	−0.0037 (8)	0.0139 (8)	−0.0043 (7)
C6	0.0407 (8)	0.0358 (9)	0.0265 (7)	−0.0085 (6)	0.0022 (6)	−0.0019 (6)
C7	0.0360 (7)	0.0313 (8)	0.0267 (7)	−0.0039 (6)	−0.0012 (5)	−0.0002 (6)
C8	0.0382 (8)	0.0319 (8)	0.0277 (7)	−0.0008 (6)	−0.0026 (6)	0.0000 (6)
C9	0.0433 (9)	0.0366 (9)	0.0322 (8)	−0.0021 (7)	0.0009 (6)	0.0048 (6)
C10	0.0453 (10)	0.0609 (13)	0.0509 (11)	−0.0075 (9)	0.0009 (8)	0.0108 (9)
C11	0.0484 (12)	0.091 (2)	0.0858 (19)	−0.0083 (12)	0.0182 (12)	0.0186 (15)
C12	0.0846 (19)	0.097 (2)	0.0703 (17)	−0.0054 (16)	0.0413 (15)	0.0091 (15)
C13	0.0881 (18)	0.0782 (17)	0.0384 (10)	−0.0047 (14)	0.0195 (11)	0.0017 (10)
C14	0.0579 (11)	0.0431 (10)	0.0311 (8)	0.0001 (8)	0.0029 (7)	0.0014 (7)
N15	0.0570 (9)	0.0519 (10)	0.0297 (7)	0.0009 (7)	−0.0119 (6)	−0.0001 (6)
C16	0.0428 (9)	0.0313 (8)	0.0372 (8)	0.0062 (7)	−0.0082 (7)	−0.0017 (6)
C17	0.0416 (8)	0.0331 (8)	0.0316 (7)	−0.0004 (6)	−0.0030 (6)	0.0020 (6)
C18	0.0472 (9)	0.0393 (9)	0.0371 (8)	0.0074 (7)	−0.0012 (7)	−0.0059 (7)
C19	0.0495 (9)	0.0327 (9)	0.0343 (8)	0.0018 (7)	−0.0009 (7)	−0.0015 (6)
C20	0.0539 (10)	0.0396 (10)	0.0321 (8)	0.0018 (8)	−0.0024 (7)	−0.0027 (7)
C21	0.0852 (18)	0.0872 (19)	0.0449 (11)	0.0044 (15)	−0.0087 (11)	−0.0311 (12)
N22	0.0730 (12)	0.0670 (12)	0.0359 (8)	−0.0090 (10)	−0.0105 (8)	−0.0081 (8)
C23	0.0558 (10)	0.0442 (10)	0.0337 (8)	−0.0071 (8)	−0.0035 (7)	0.0028 (7)
C24	0.0506 (12)	0.0630 (14)	0.0715 (14)	−0.0059 (10)	−0.0036 (10)	0.0011 (11)
C25	0.0436 (9)	0.0387 (9)	0.0298 (7)	−0.0099 (7)	0.0026 (6)	−0.0010 (6)
C26	0.0438 (9)	0.0488 (11)	0.0318 (8)	−0.0044 (8)	0.0012 (6)	0.0007 (7)
N27	0.0607 (10)	0.0569 (10)	0.0304 (7)	−0.0069 (8)	0.0038 (7)	0.0047 (7)
C28	0.0780 (15)	0.0678 (14)	0.0359 (9)	−0.0287 (12)	0.0075 (9)	0.0076 (9)
C29	0.0569 (13)	0.118 (2)	0.0493 (12)	−0.0302 (14)	−0.0145 (10)	0.0133 (13)

Geometric parameters (Å, °)

S1—C28	1.705 (2)	C11—H11	0.93
S1—C25	1.7157 (17)	C12—C13	1.379 (4)
S2—C21	1.694 (2)	C12—H12	0.93
S2—C20	1.723 (2)	C13—C14	1.378 (3)
O1—C16	1.208 (2)	C13—H13	0.93
O2—C17	1.215 (2)	C14—N15	1.395 (3)

N1—C2	1.451 (3)	N15—C16	1.354 (2)
N1—C5	1.464 (2)	N15—H15	0.86
N1—C8	1.465 (2)	C16—O1	1.208 (2)
C2—C3	1.493 (3)	C16—O1	1.208 (2)
C3—C4	1.476 (3)	C17—C18	1.468 (2)
C3—H3A	0.97	C18—C19	1.332 (2)
C3—H3B	0.97	C18—H18	0.93
C4—C5	1.532 (3)	C19—C20	1.442 (2)
C4—H4A	0.97	C19—H19	0.93
C4—H4B	0.97	C20—C23	1.371 (3)
C5—C6	1.529 (3)	C21—N22	1.295 (3)
C5—H5	0.98	C21—H21	0.93
C6—C25	1.494 (2)	N22—C23	1.381 (3)
C6—C7	1.529 (2)	C23—C24	1.483 (3)
C6—H6	0.98	C24—H24A	0.96
C7—C17	1.513 (2)	C24—H24B	0.96
C7—C8	1.559 (2)	C24—H24C	0.96
C7—H7	0.98	C25—C26	1.352 (2)
C8—C9	1.508 (2)	C26—N27	1.384 (2)
C8—C16	1.553 (2)	C26—C29	1.489 (3)
C9—C10	1.379 (3)	N27—C28	1.282 (3)
C9—C14	1.392 (2)	C28—H28	0.93
C10—C11	1.386 (3)	C29—H29A	0.96
C10—H10	0.93	C29—H29B	0.96
C11—C12	1.379 (4)	C29—H29C	0.96
C28—S1—C25	89.25 (9)	C11—C12—H12	119.0
C21—S2—C20	89.37 (11)	C12—C13—C14	117.2 (2)
C2—N1—C5	108.9 (2)	C12—C13—H13	121.4
C2—N1—C8	122.2 (2)	C14—C13—H13	121.4
C5—N1—C8	110.8 (1)	C13—C14—C9	121.8 (2)
N1—C2—C3	104.38 (19)	C13—C14—N15	128.1 (2)
N1—C2—H2A	110.9	C9—C14—N15	110.04 (16)
C3—C2—H2A	110.9	C16—N15—C14	111.5 (1)
N1—C2—H2B	110.9	C16—N15—H15	124.2
C3—C2—H2B	110.9	C14—N15—H15	124.2
H2A—C2—H2B	108.9	O1—C16—N15	126.69 (16)
C4—C3—C2	104.09 (19)	O1—C16—N15	126.69 (16)
C4—C3—H3A	110.9	O1—C16—N15	126.69 (16)
C2—C3—H3A	110.9	O1—C16—C8	125.51 (16)
C4—C3—H3B	110.9	O1—C16—C8	125.51 (16)
C2—C3—H3B	110.9	O1—C16—C8	125.51 (16)
H3A—C3—H3B	109.0	N15—C16—C8	107.80 (15)
C3—C4—C5	105.07 (18)	O2—C17—C18	119.71 (16)
C3—C4—H4A	110.7	O2—C17—C7	120.92 (15)
C5—C4—H4A	110.7	C18—C17—C7	119.35 (14)
C3—C4—H4B	110.7	C19—C18—C17	125.91 (17)
C5—C4—H4B	110.7	C19—C18—H18	117.0
H4A—C4—H4B	108.8	C17—C18—H18	117.0
N1—C5—C6	104.95 (14)	C18—C19—C20	126.13 (18)

N1—C5—C4	105.04 (15)	C18—C19—H19	116.9
C6—C5—C4	117.69 (18)	C20—C19—H19	116.9
N1—C5—H5	109.6	C23—C20—C19	127.18 (18)
C6—C5—H5	109.6	C23—C20—S2	109.60 (14)
C4—C5—H5	109.6	C19—C20—S2	123.19 (14)
C25—C6—C7	115.30 (14)	N22—C21—S2	116.02 (17)
C25—C6—C5	113.53 (14)	N22—C21—H21	122.0
C7—C6—C5	101.96 (13)	S2—C21—H21	122.0
C25—C6—H6	108.6	C21—N22—C23	110.71 (18)
C7—C6—H6	108.6	C20—C23—N22	114.30 (19)
C5—C6—H6	108.6	C20—C23—C24	126.11 (18)
C17—C7—C6	114.97 (13)	N22—C23—C24	119.58 (19)
C17—C7—C8	113.49 (13)	C23—C24—H24A	109.5
C6—C7—C8	103.79 (13)	C23—C24—H24B	109.5
C17—C7—H7	108.1	H24A—C24—H24B	109.5
C6—C7—H7	108.1	C23—C24—H24C	109.5
C8—C7—H7	108.1	H24A—C24—H24C	109.5
N1—C8—C9	116.42 (14)	H24B—C24—H24C	109.5
N1—C8—C16	107.70 (13)	C26—C25—C6	129.59 (16)
C9—C8—C16	101.55 (13)	C26—C25—S1	109.29 (13)
N1—C8—C7	104.24 (12)	C6—C25—S1	120.93 (12)
C9—C8—C7	116.43 (14)	C25—C26—N27	115.54 (16)
C16—C8—C7	110.26 (13)	C25—C26—C29	126.01 (18)
C10—C9—C14	119.89 (17)	N27—C26—C29	118.44 (17)
C10—C9—C8	131.62 (16)	C28—N27—C26	109.91 (16)
C14—C9—C8	108.48 (15)	N27—C28—S1	115.99 (15)
C9—C10—C11	118.9 (2)	N27—C28—H28	122.0
C9—C10—H10	120.6	S1—C28—H28	122.0
C11—C10—H10	120.6	C26—C29—H29A	109.5
C12—C11—C10	120.1 (2)	C26—C29—H29B	109.5
C12—C11—H11	119.9	H29A—C29—H29B	109.5
C10—C11—H11	119.9	C26—C29—H29C	109.5
C13—C12—C11	122.1 (2)	H29A—C29—H29C	109.5
C13—C12—H12	119.0	H29B—C29—H29C	109.5
C5—N1—C2—C3	26.5 (3)	O1—O1—C16—N15	0.0 (2)
C8—N1—C2—C3	157.8 (2)	O1—O1—C16—C8	0.0 (2)
N1—C2—C3—C4	−35.7 (3)	O1—O1—C16—C8	0.0 (2)
C2—C3—C4—C5	31.2 (3)	C14—N15—C16—O1	173.29 (18)
C2—N1—C5—C6	117.7 (2)	C14—N15—C16—O1	173.29 (18)
C8—N1—C5—C6	−19.43 (19)	C14—N15—C16—O1	173.29 (18)
C2—N1—C5—C4	−7.0 (3)	C14—N15—C16—C8	−7.5 (2)
C8—N1—C5—C4	−144.16 (18)	N1—C8—C16—O1	64.1 (2)
C3—C4—C5—N1	−15.3 (3)	C9—C8—C16—O1	−173.06 (17)
C3—C4—C5—C6	−131.6 (2)	C7—C8—C16—O1	−49.0 (2)
N1—C5—C6—C25	159.30 (14)	N1—C8—C16—O1	64.1 (2)
C4—C5—C6—C25	−84.4 (2)	C9—C8—C16—O1	−173.06 (17)
N1—C5—C6—C7	34.64 (17)	C7—C8—C16—O1	−49.0 (2)
C4—C5—C6—C7	150.96 (17)	N1—C8—C16—O1	64.1 (2)
C25—C6—C7—C17	75.27 (19)	C9—C8—C16—O1	−173.06 (17)

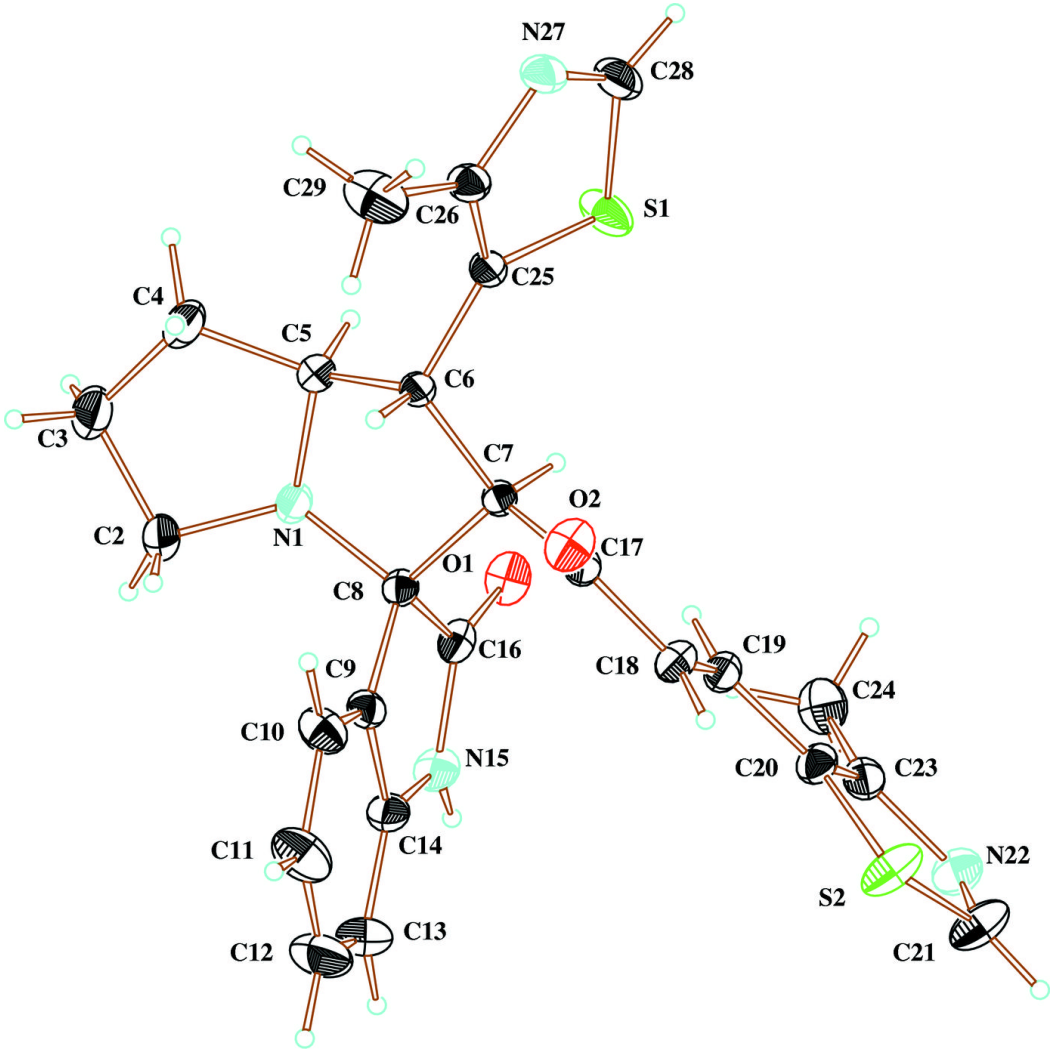
C5—C6—C7—C17	−161.26 (14)	C7—C8—C16—O1	−49.0 (2)
C25—C6—C7—C8	−160.18 (14)	N1—C8—C16—N15	−115.12 (15)
C5—C6—C7—C8	−36.71 (16)	C9—C8—C16—N15	7.70 (18)
C2—N1—C8—C9	−4.7 (3)	C7—C8—C16—N15	131.73 (15)
C5—N1—C8—C9	125.79 (16)	C6—C7—C17—O2	9.2 (2)
C2—N1—C8—C16	108.4 (2)	C8—C7—C17—O2	−110.07 (18)
C5—N1—C8—C16	−121.05 (16)	C6—C7—C17—C18	−172.64 (15)
C2—N1—C8—C7	−134.4 (2)	C8—C7—C17—C18	68.1 (2)
C5—N1—C8—C7	−3.91 (18)	O2—C17—C18—C19	−162.21 (19)
C17—C7—C8—N1	151.14 (13)	C7—C17—C18—C19	19.6 (3)
C6—C7—C8—N1	25.64 (16)	C17—C18—C19—C20	178.51 (18)
C17—C7—C8—C9	21.44 (19)	C18—C19—C20—C23	178.2 (2)
C6—C7—C8—C9	−104.06 (15)	C18—C19—C20—S2	−4.0 (3)
C17—C7—C8—C16	−93.52 (16)	C21—S2—C20—C23	0.26 (18)
C6—C7—C8—C16	140.98 (14)	C21—S2—C20—C19	−177.86 (19)
N1—C8—C9—C10	−70.0 (3)	C20—S2—C21—N22	−0.3 (2)
C16—C8—C9—C10	173.3 (2)	S2—C21—N22—C23	0.2 (3)
C7—C8—C9—C10	53.6 (3)	C19—C20—C23—N22	177.81 (19)
N1—C8—C9—C14	111.27 (17)	S2—C20—C23—N22	−0.2 (2)
C16—C8—C9—C14	−5.35 (18)	C19—C20—C23—C24	−2.9 (3)
C7—C8—C9—C14	−125.11 (16)	S2—C20—C23—C24	179.10 (18)
C14—C9—C10—C11	−0.2 (3)	C21—N22—C23—C20	0.0 (3)
C8—C9—C10—C11	−178.7 (2)	C21—N22—C23—C24	−179.3 (2)
C9—C10—C11—C12	−1.3 (4)	C7—C6—C25—C26	−145.1 (2)
C10—C11—C12—C13	1.3 (5)	C5—C6—C25—C26	97.8 (2)
C11—C12—C13—C14	0.3 (5)	C7—C6—C25—S1	40.4 (2)
C12—C13—C14—C9	−1.9 (4)	C5—C6—C25—S1	−76.68 (18)
C12—C13—C14—N15	177.3 (2)	C28—S1—C25—C26	−0.87 (17)
C10—C9—C14—C13	1.8 (3)	C28—S1—C25—C6	174.65 (17)
C8—C9—C14—C13	−179.3 (2)	C6—C25—C26—N27	−174.20 (18)
C10—C9—C14—N15	−177.48 (18)	S1—C25—C26—N27	0.8 (2)
C8—C9—C14—N15	1.4 (2)	C6—C25—C26—C29	5.9 (4)
C13—C14—N15—C16	−175.2 (2)	S1—C25—C26—C29	−179.1 (2)
C9—C14—N15—C16	4.0 (2)	C25—C26—N27—C28	−0.2 (3)
O1—O1—C16—O1	0.0 (2)	C29—C26—N27—C28	179.7 (2)
O1—O1—C16—O1	0.0 (2)	C26—N27—C28—S1	−0.5 (3)
O1—O1—C16—N15	0.0 (2)	C25—S1—C28—N27	0.8 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N15—H15···N22^{i}	0.86	2.15	2.978 (2)	162
C7—H7···O1	0.98	2.56	3.005 (2)	108
C19—H19···O1	0.93	2.51	3.301 (2)	143
C28—H28···O1^{ii}	0.93	2.47	3.016 (3)	118
C24—H24C···Cg^{ii}	0.96	2.77	3.631 (3)	149

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

Fig. 1


N27
C28
C29
C26
S1
C25
C4
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C6
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C7
O2
C2
N1
C17
O1
C8
C16
C18
C19
C9
C24
C10
N15
C20
C23
C11
C14
N22
S2
C13
C12
C21