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ISSN 2414-3146

1-Allyl-3′-phenyl-6′*H*-spiro[indoline-3,4′-isoxazolo-[4′,5′:5,6]pyrido[2,3-*d*]pyrimidine]-2,5′,7′(8′*H*,9′*H*)-trione dimethyl sulfoxide monosolvate

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Received 25 October 2016
Accepted 23 December 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

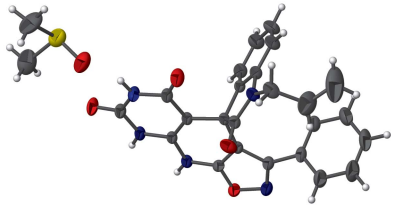
Keywords: crystal structure; spiro; indoline; isooxazolo; pyrimidine; hydrogen bonding; C—H···π interactions.

CCDC reference: 1419465

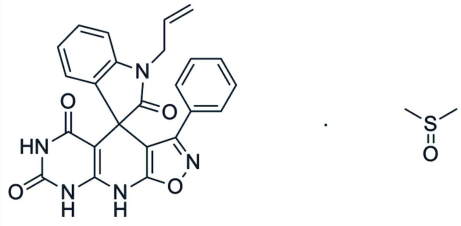
Structural data: full structural data are available from iucrdata.iucr.org

In the title solvated compound, $C_{24}H_{17}N_5O_4\cdot C_2H_6OS$, the solvent molecule, dimethyl sulfoxide, is linked to the title molecule by an N—H···O hydrogen bond. The pyridine ring adopts a twist-boat conformation. The isoxazole ring is inclined to the indoline ring system, the pyrimidine ring, and the phenyl ring by 82.31 (7), 10.41 (8) and 53.77 (10)°, respectively. There is an intramolecular C—H···π interaction present involving the phenyl ring and the indoline ring system. In the crystal, molecules are connected by two pairs of N—H···O hydrogen bonds, forming chains along the *b*-axis direction, and enclosing $R_2^2(8)$ and $R_2^2(14)$ ring motifs. The chains are linked by C—H···O and C—H···N hydrogen bonds and offset π–π interactions, between the pyrimidine and isoxazole rings of inversion-related molecules [centroid–centroid distance = 3.7140 (9) Å], forming a three-dimensional structure.

3D view



Chemical scheme



Structure description

Pyrrolidine derivatives are used as norepinephrine reuptake inhibitors and 5-HT(1A) partial agonists for treating neuropsychiatric disorders including depression and anxiety (Pettersson *et al.*, 2011). These derivatives are also used as α-mannosidase inhibitors and have antitumor activities against hematological and solid malignancies (Bello *et al.*, 2010). In view of their importance, we have undertaken the synthesis and crystal structure determination of the title compound and the results are presented herein.

The molecular structure of the title compound is illustrated in Fig. 1. The solvent molecule is attached to the title molecule *via* an N4—H4*A*···O5 hydrogen bond and there is an intramolecular C—H···π interaction present involving the phenyl ring (C1–C6) and the indoline (N5/C13/C15–C21) ring system (Fig. 1 and Table 1). The sum of the

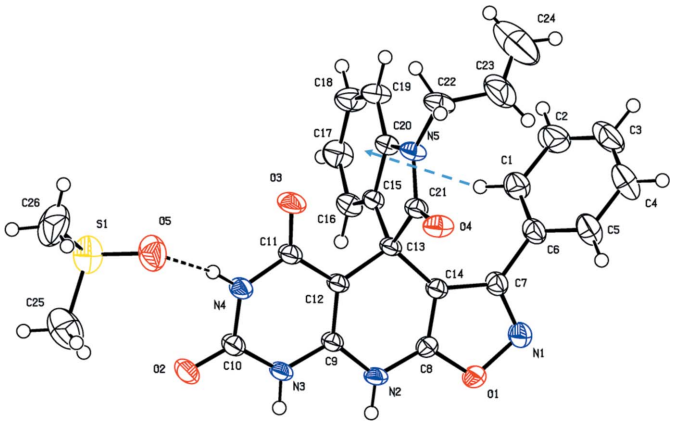


Figure 1
The molecular structure of the title compound, showing the atom labelling and 30% probability displacement ellipsoids. The N—H···O hydrogen bond is shown as a dashed line and the intramolecular C—H···π interaction by a blue dashed arrow (see Table 1).

angles at N3 and N4 of the pyrimidine ring (360 and 360.09°, respectively) is in accordance with sp^2 hybridization. The pyridine ring (N2/C8/C9/C12–C14) adopts a twist-boat conformation [puckering parameters: Q, θ, φ = 0.1030 (15) Å, 98.1 (8)° and 34.1 (9)°, respectively]. The isoxazole ring (O1/N1/C7/C8/C14) makes dihedral angles of 82.31 (7), 10.41 (8) and 53.77 (10)° with the mean plane of the indoline (N5/C13/C15—C21) ring system and the pyrimidine (N3/N4/C9–C12) and the phenyl (C1–C6) rings, respectively.

In the crystal, pairs of N2—H2*A*···O4^{i} and N3—H3*A*···O2^{ii} hydrogen bonds form centrosymmetric loops, with $R_2^2(8)$ and $R_2^2(14)$ ring motifs. These combine to form chains which propagate in the *b*-axis direction (Fig. 2 and Table 1). The crystal packing is further stabilized by C—H···O and C—H···N hydrogen bonds and offset π–π stacking interactions, forming a three-dimensional structure (Table 1 and Fig. 3). The offset π–π interactions involve inversion-related isoxazolo and pyrimidine rings: *Cg*1···*Cg*4($-x$ + 1, $-y$ + 2, $-z$) = 3.7140 (9) Å, α = 10.41 (8) °, interplanar distances 3.518 (1)/3.247 (1) Å, slippage = 1.803 Å, where *Cg*1 and *Cg*4 are the centroids of rings (O1/N1/C7/C8/C14) and (N3/N4/C9–C12), respectively.

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

Cg is the centroid of the C15–C20 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O5	0.86	2.03	2.862 (2)	163
C1—H1···*Cg*	0.93	2.88	3.624 (2)	138
N2—H2*A*···O4^{i}	0.86	2.11	2.7631 (16)	132
N3—H3*A*···O2^{ii}	0.86	1.99	2.7887 (18)	155
C17—H17···N1^{iii}	0.93	2.53	3.412 (2)	159
C19—H19···O3^{iv}	0.93	2.42	3.324 (2)	165
C26—H26*C*···O1^{v}	0.96	2.59	3.322 (3)	133

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

Synthesis and crystallization

A mixture of *N*-allyl isatin (1 mmol), 6-aminouracil (1 mmol), isoxazole (1 mmol) and *p*-TSA·H_2O (0.20 mmol) in water (3 ml) were placed in a 25 ml round-bottomed flask and the mixture was heated at reflux with stirring for 4 h. The consumption of the starting material was monitored by TLC. The precipitated solid was filtered and washed with ethanol (5–7 ml), and dried under vacuum to obtain pure title product in good yield (87%). Colourless block-like crystals were obtained by slow evaporation of a solution in dimethyl sulfoxide.

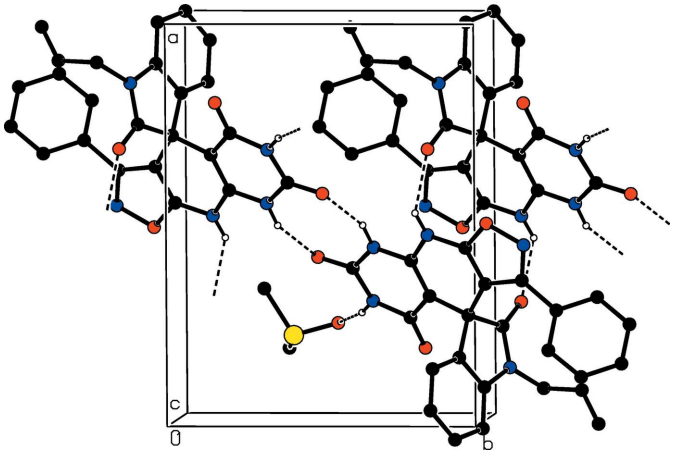


Figure 2
A partial view along the *c* axis of the crystal packing of the title compound. The N—H···O hydrogen bonds, enclosing $R_2^2(8)$ and $R_2^2(14)$ ring motifs (see Table 1), are shown as dashed lines, and H atoms not involved in these interaction have been omitted.

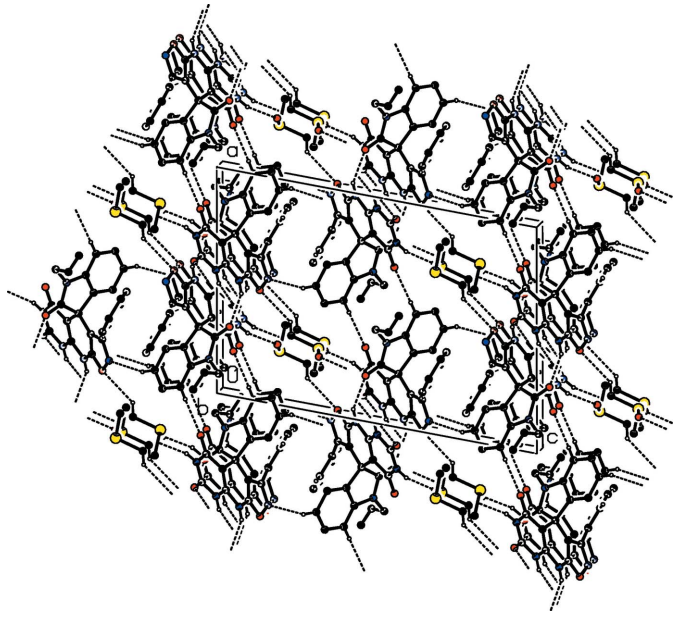


Figure 3
A view along the *b* axis of the crystal packing of the title compound. Hydrogen bonds (see Table 1), are shown as dashed lines, and for clarity, H atoms not involved in these interaction have been omitted.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{24}H_{17}N_5O_4{\cdot}C_2H_6OS$
M_r	517.55
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	13.2381 (3), 9.9265 (2), 19.0348 (4)
β (°)	100.693 (1)
V (Å^3)	2457.89 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.18
Crystal size (mm)	0.2 × 0.17 × 0.16
Data collection	
Diffractometer	Bruker *SMART* APEXII area-detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2008)
T_{min}, T_{max}	0.625, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22793, 6183, 4931
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.671
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.143, 0.99
No. of reflections	6183
No. of parameters	336
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.46, −0.52

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS2014* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors thank the Department of chemistry, IIT, Chennai, India, for the X-ray intensity data collection.

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full crystallographic data

IUCrData (2017). **2**, x162044 [https://doi.org/10.1107/S2414314616020447]

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Crystal data

$C_{24}H_{17}N_5O_4·C_2H_6OS$
M_r = 517.55
Monoclinic, $P2_1/n$
a = 13.2381 (3) Å
b = 9.9265 (2) Å
c = 19.0348 (4) Å
β = 100.693 (1)°
V = 2457.89 (9) Å^3
Z = 4
$F(000)$ = 1080
D_x = 1.399 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6183 reflections
θ = 1.7–28.5°
μ = 0.18 mm^{-1}
T = 293 K
Block, colourless
0.2 × 0.17 × 0.16 mm

Data collection

Bruker SMART APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2008)
T_{min} = 0.625, T_{max} = 0.746
22793 measured reflections
6183 independent reflections
4931 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.5°, θ_{min} = 1.7°
h = −17→17
k = −13→13
l = −24→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.143
S = 0.99
6183 reflections
336 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0787P)^2 + 1.1801P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C25	0.3362 (2)	0.3045 (3)	0.20062 (16)	0.0773 (8)
H25A	0.3697	0.3120	0.1602	0.116*
H25B	0.3203	0.2117	0.2077	0.116*
H25C	0.3810	0.3381	0.2425	0.116*
C26	0.1865 (2)	0.3806 (3)	0.26996 (16)	0.0801 (8)
H26A	0.2416	0.4121	0.3063	0.120*
H26B	0.1737	0.2872	0.2779	0.120*
H26C	0.1255	0.4319	0.2717	0.120*
O5	0.25162 (13)	0.54474 (15)	0.18055 (8)	0.0582 (4)
S1	0.22081 (5)	0.40028 (6)	0.18485 (3)	0.05503 (16)
C1	0.19635 (14)	1.24982 (19)	−0.19265 (10)	0.0416 (4)
H1	0.1742	1.1619	−0.2031	0.050*
C2	0.13102 (16)	1.3570 (2)	−0.21489 (12)	0.0541 (5)
H2	0.0652	1.3410	−0.2405	0.065*
C3	0.16352 (19)	1.4871 (2)	−0.19911 (13)	0.0605 (6)
H3	0.1193	1.5587	−0.2138	0.073*
C4	0.2602 (2)	1.5114 (2)	−0.16207 (14)	0.0619 (6)
H4	0.2815	1.5996	−0.1515	0.074*
C5	0.32702 (17)	1.40555 (18)	−0.14015 (11)	0.0484 (5)
H5	0.3933	1.4226	−0.1157	0.058*
C6	0.29440 (13)	1.27389 (16)	−0.15493 (8)	0.0339 (3)
C7	0.36787 (12)	1.16141 (15)	−0.13492 (8)	0.0295 (3)
C8	0.44759 (11)	0.97633 (15)	−0.09886 (8)	0.0263 (3)
C9	0.41376 (10)	0.79624 (14)	−0.03097 (7)	0.0247 (3)
C10	0.39271 (12)	0.60233 (15)	0.04044 (8)	0.0313 (3)
C11	0.27034 (11)	0.78910 (16)	0.02992 (8)	0.0304 (3)
C12	0.32515 (10)	0.85463 (14)	−0.01953 (8)	0.0251 (3)
C13	0.27903 (10)	0.98222 (14)	−0.05754 (7)	0.0239 (3)
C14	0.35792 (11)	1.03975 (14)	−0.09694 (8)	0.0253 (3)
C15	0.17513 (11)	0.95515 (14)	−0.10448 (8)	0.0252 (3)
C16	0.14866 (13)	0.87217 (16)	−0.16264 (9)	0.0338 (3)
H16	0.1982	0.8205	−0.1789	0.041*
C17	0.04632 (14)	0.86675 (19)	−0.19675 (10)	0.0428 (4)
H17	0.0270	0.8112	−0.2363	0.051*
C18	−0.02682 (13)	0.9440 (2)	−0.17197 (10)	0.0435 (4)
H18	−0.0952	0.9376	−0.1947	0.052*
C19	−0.00087 (12)	1.03074 (18)	−0.11404 (10)	0.0375 (4)
H19	−0.0500	1.0837	−0.0981	0.045*
C20	0.10113 (11)	1.03445 (15)	−0.08129 (8)	0.0274 (3)

C21	0.24980 (11)	1.08978 (15)	−0.00520 (8)	0.0265 (3)
C22	0.09541 (13)	1.22087 (18)	0.00906 (9)	0.0382 (4)
H22A	0.1311	1.2371	0.0576	0.046*
H22B	0.0259	1.1924	0.0110	0.046*
C23	0.0922 (2)	1.3473 (2)	−0.03306 (16)	0.0697 (7)
H23	0.1544	1.3893	−0.0350	0.084*
C24	0.0094 (4)	1.4031 (4)	−0.0671 (2)	0.1144 (15)
H24A	−0.0543	1.3640	−0.0663	0.137*
H24B	0.0133	1.4825	−0.0924	0.137*
N1	0.45659 (11)	1.16924 (14)	−0.15619 (8)	0.0363 (3)
N2	0.47848 (9)	0.85237 (13)	−0.07198 (7)	0.0290 (3)
H2A	0.5332	0.8130	−0.0799	0.035*
N3	0.44539 (10)	0.67292 (13)	−0.00266 (7)	0.0297 (3)
H3A	0.5008	0.6386	−0.0125	0.036*
N4	0.31052 (10)	0.66641 (14)	0.05790 (7)	0.0338 (3)
H4A	0.2805	0.6281	0.0890	0.041*
N5	0.14801 (9)	1.11418 (13)	−0.02309 (7)	0.0287 (3)
O1	0.50932 (8)	1.04704 (11)	−0.13302 (6)	0.0336 (3)
O2	0.41907 (10)	0.48822 (12)	0.06147 (7)	0.0432 (3)
O3	0.19414 (10)	0.83580 (14)	0.04842 (7)	0.0461 (3)
O4	0.30914 (8)	1.14745 (12)	0.04212 (6)	0.0366 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C25	0.109 (2)	0.0547 (14)	0.0711 (16)	0.0167 (14)	0.0239 (15)	−0.0052 (12)
C26	0.097 (2)	0.0754 (17)	0.0801 (18)	−0.0189 (15)	0.0486 (16)	0.0116 (14)
O5	0.0783 (10)	0.0463 (8)	0.0560 (9)	0.0017 (7)	0.0277 (8)	0.0116 (7)
S1	0.0697 (4)	0.0498 (3)	0.0427 (3)	−0.0102 (2)	0.0032 (2)	−0.0009 (2)
C1	0.0425 (9)	0.0396 (9)	0.0430 (9)	0.0061 (7)	0.0086 (7)	0.0101 (7)
C2	0.0458 (11)	0.0600 (13)	0.0553 (12)	0.0156 (9)	0.0057 (9)	0.0193 (10)
C3	0.0697 (14)	0.0474 (12)	0.0646 (14)	0.0281 (11)	0.0131 (11)	0.0207 (10)
C4	0.0783 (16)	0.0326 (10)	0.0723 (15)	0.0155 (10)	0.0077 (12)	0.0102 (9)
C5	0.0567 (11)	0.0323 (9)	0.0535 (11)	0.0065 (8)	0.0031 (9)	0.0061 (8)
C6	0.0408 (8)	0.0297 (7)	0.0326 (7)	0.0080 (6)	0.0106 (6)	0.0086 (6)
C7	0.0330 (7)	0.0275 (7)	0.0288 (7)	0.0031 (6)	0.0077 (6)	0.0034 (6)
C8	0.0256 (6)	0.0284 (7)	0.0267 (7)	0.0013 (5)	0.0098 (5)	0.0025 (5)
C9	0.0244 (6)	0.0246 (6)	0.0263 (6)	0.0057 (5)	0.0080 (5)	0.0023 (5)
C10	0.0334 (7)	0.0287 (7)	0.0345 (7)	0.0091 (6)	0.0130 (6)	0.0057 (6)
C11	0.0291 (7)	0.0301 (7)	0.0350 (7)	0.0099 (6)	0.0138 (6)	0.0066 (6)
C12	0.0238 (6)	0.0250 (7)	0.0282 (7)	0.0072 (5)	0.0093 (5)	0.0034 (5)
C13	0.0227 (6)	0.0232 (6)	0.0271 (6)	0.0066 (5)	0.0079 (5)	0.0012 (5)
C14	0.0246 (6)	0.0250 (6)	0.0273 (6)	0.0044 (5)	0.0072 (5)	0.0017 (5)
C15	0.0245 (6)	0.0237 (6)	0.0281 (7)	0.0046 (5)	0.0070 (5)	0.0019 (5)
C16	0.0360 (8)	0.0302 (7)	0.0364 (8)	0.0067 (6)	0.0097 (6)	−0.0050 (6)
C17	0.0430 (9)	0.0414 (9)	0.0416 (9)	0.0012 (7)	0.0011 (7)	−0.0134 (7)
C18	0.0282 (8)	0.0488 (10)	0.0499 (10)	0.0040 (7)	−0.0025 (7)	−0.0090 (8)
C19	0.0255 (7)	0.0423 (9)	0.0442 (9)	0.0095 (6)	0.0050 (6)	−0.0057 (7)

C20	0.0257 (7)	0.0269 (7)	0.0300 (7)	0.0055 (5)	0.0062 (5)	−0.0014 (5)
C21	0.0252 (7)	0.0270 (7)	0.0283 (7)	0.0056 (5)	0.0078 (5)	0.0007 (5)
C22	0.0344 (8)	0.0400 (9)	0.0411 (9)	0.0130 (7)	0.0097 (7)	−0.0122 (7)
C23	0.0858 (17)	0.0399 (11)	0.0924 (18)	0.0233 (11)	0.0403 (15)	−0.0006 (11)
C24	0.162 (4)	0.089 (2)	0.104 (3)	0.074 (2)	0.054 (2)	0.0295 (19)
N1	0.0377 (7)	0.0332 (7)	0.0402 (7)	0.0045 (6)	0.0130 (6)	0.0106 (6)
N2	0.0237 (6)	0.0305 (6)	0.0363 (7)	0.0098 (5)	0.0149 (5)	0.0062 (5)
N3	0.0275 (6)	0.0278 (6)	0.0373 (7)	0.0118 (5)	0.0150 (5)	0.0073 (5)
N4	0.0348 (7)	0.0313 (7)	0.0406 (7)	0.0110 (5)	0.0210 (6)	0.0117 (6)
N5	0.0243 (6)	0.0300 (6)	0.0321 (6)	0.0091 (5)	0.0061 (5)	−0.0056 (5)
O1	0.0296 (5)	0.0353 (6)	0.0393 (6)	0.0044 (4)	0.0148 (5)	0.0107 (5)
O2	0.0505 (7)	0.0316 (6)	0.0543 (8)	0.0182 (5)	0.0275 (6)	0.0157 (5)
O3	0.0425 (7)	0.0466 (7)	0.0584 (8)	0.0215 (6)	0.0330 (6)	0.0186 (6)
O4	0.0273 (5)	0.0441 (7)	0.0376 (6)	0.0044 (5)	0.0040 (4)	−0.0118 (5)

Geometric parameters (Å, º)

C25—S1	1.777 (3)	C11—O3	1.2200 (18)
C25—H25A	0.9600	C11—N4	1.3945 (19)
C25—H25B	0.9600	C11—C12	1.4454 (19)
C25—H25C	0.9600	C12—C13	1.5285 (18)
C26—S1	1.773 (3)	C13—C14	1.5066 (19)
C26—H26A	0.9600	C13—C15	1.5187 (19)
C26—H26B	0.9600	C13—C21	1.5574 (19)
C26—H26C	0.9600	C15—C16	1.372 (2)
O5—S1	1.4972 (16)	C15—C20	1.3909 (19)
C1—C6	1.383 (3)	C16—C17	1.391 (2)
C1—C2	1.387 (3)	C16—H16	0.9300
C1—H1	0.9300	C17—C18	1.384 (3)
C2—C3	1.376 (3)	C17—H17	0.9300
C2—H2	0.9300	C18—C19	1.391 (2)
C3—C4	1.364 (4)	C18—H18	0.9300
C3—H3	0.9300	C19—C20	1.379 (2)
C4—C5	1.387 (3)	C19—H19	0.9300
C4—H4	0.9300	C20—N5	1.4085 (19)
C5—C6	1.389 (3)	C21—O4	1.2221 (19)
C5—H5	0.9300	C21—N5	1.3490 (18)
C6—C7	1.483 (2)	C22—N5	1.4626 (18)
C7—N1	1.314 (2)	C22—C23	1.486 (3)
C7—C14	1.426 (2)	C22—H22A	0.9700
C8—O1	1.3343 (17)	C22—H22B	0.9700
C8—C14	1.3501 (19)	C23—C24	1.290 (5)
C8—N2	1.3658 (19)	C23—H23	0.9300
C9—C12	1.3617 (18)	C24—H24A	0.9300
C9—N3	1.3714 (18)	C24—H24B	0.9300
C9—N2	1.3791 (18)	N1—O1	1.4279 (17)
C10—O2	1.2301 (19)	N2—H2A	0.8600
C10—N4	1.3540 (19)	N3—H3A	0.8600

C10—N3	1.3647 (19)	N4—H4A	0.8600
S1—C25—H25A	109.5	C14—C13—C21	109.64 (12)
S1—C25—H25B	109.5	C15—C13—C21	101.08 (10)
H25A—C25—H25B	109.5	C12—C13—C21	112.96 (11)
S1—C25—H25C	109.5	C8—C14—C7	102.52 (12)
H25A—C25—H25C	109.5	C8—C14—C13	122.07 (13)
H25B—C25—H25C	109.5	C7—C14—C13	135.32 (13)
S1—C26—H26A	109.5	C16—C15—C20	120.42 (14)
S1—C26—H26B	109.5	C16—C15—C13	130.44 (13)
H26A—C26—H26B	109.5	C20—C15—C13	109.12 (12)
S1—C26—H26C	109.5	C15—C16—C17	118.74 (14)
H26A—C26—H26C	109.5	C15—C16—H16	120.6
H26B—C26—H26C	109.5	C17—C16—H16	120.6
O5—S1—C26	105.90 (12)	C18—C17—C16	120.12 (16)
O5—S1—C25	106.69 (12)	C18—C17—H17	119.9
C26—S1—C25	98.34 (15)	C16—C17—H17	119.9
C6—C1—C2	119.87 (19)	C17—C18—C19	121.84 (16)
C6—C1—H1	120.1	C17—C18—H18	119.1
C2—C1—H1	120.1	C19—C18—H18	119.1
C3—C2—C1	120.1 (2)	C20—C19—C18	116.87 (14)
C3—C2—H2	120.0	C20—C19—H19	121.6
C1—C2—H2	120.0	C18—C19—H19	121.6
C4—C3—C2	120.31 (19)	C19—C20—C15	121.99 (14)
C4—C3—H3	119.8	C19—C20—N5	128.48 (13)
C2—C3—H3	119.8	C15—C20—N5	109.52 (12)
C3—C4—C5	120.4 (2)	O4—C21—N5	125.26 (13)
C3—C4—H4	119.8	O4—C21—C13	126.19 (12)
C5—C4—H4	119.8	N5—C21—C13	108.47 (12)
C4—C5—C6	119.7 (2)	N5—C22—C23	110.75 (15)
C4—C5—H5	120.2	N5—C22—H22A	109.5
C6—C5—H5	120.2	C23—C22—H22A	109.5
C1—C6—C5	119.64 (16)	N5—C22—H22B	109.5
C1—C6—C7	120.73 (15)	C23—C22—H22B	109.5
C5—C6—C7	119.47 (16)	H22A—C22—H22B	108.1
N1—C7—C14	112.23 (13)	C24—C23—C22	124.8 (3)
N1—C7—C6	117.22 (14)	C24—C23—H23	117.6
C14—C7—C6	130.52 (14)	C22—C23—H23	117.6
O1—C8—C14	112.89 (13)	C23—C24—H24A	120.0
O1—C8—N2	119.25 (12)	C23—C24—H24B	120.0
C14—C8—N2	127.84 (13)	H24A—C24—H24B	120.0
C12—C9—N3	121.76 (13)	C7—N1—O1	105.56 (12)
C12—C9—N2	124.23 (13)	C8—N2—C9	113.69 (11)
N3—C9—N2	114.00 (12)	C8—N2—H2A	123.2
O2—C10—N4	123.02 (14)	C9—N2—H2A	123.2
O2—C10—N3	121.51 (14)	C10—N3—C9	122.80 (12)
N4—C10—N3	115.47 (13)	C10—N3—H3A	118.6
O3—C11—N4	119.92 (14)	C9—N3—H3A	118.6

O3—C11—C12	123.97 (14)	C10—N4—C11	125.69 (13)
N4—C11—C12	116.10 (12)	C10—N4—H4A	117.2
C9—C12—C11	117.65 (13)	C11—N4—H4A	117.2
C9—C12—C13	123.87 (12)	C21—N5—C20	111.80 (12)
C11—C12—C13	118.46 (11)	C21—N5—C22	123.72 (13)
C14—C13—C15	114.20 (12)	C20—N5—C22	124.16 (12)
C14—C13—C12	107.27 (11)	C8—O1—N1	106.79 (11)
C15—C13—C12	111.75 (12)		

C6—C1—C2—C3	−0.3 (3)	C20—C15—C16—C17	−1.4 (2)
C1—C2—C3—C4	0.5 (4)	C13—C15—C16—C17	−179.56 (16)
C2—C3—C4—C5	0.3 (4)	C15—C16—C17—C18	−0.1 (3)
C3—C4—C5—C6	−1.2 (4)	C16—C17—C18—C19	1.5 (3)
C2—C1—C6—C5	−0.6 (3)	C17—C18—C19—C20	−1.3 (3)
C2—C1—C6—C7	−175.83 (17)	C18—C19—C20—C15	−0.3 (3)
C4—C5—C6—C1	1.3 (3)	C18—C19—C20—N5	178.72 (17)
C4—C5—C6—C7	176.65 (19)	C16—C15—C20—C19	1.6 (2)
C1—C6—C7—N1	123.62 (18)	C13—C15—C20—C19	−179.87 (15)
C5—C6—C7—N1	−51.6 (2)	C16—C15—C20—N5	−177.52 (14)
C1—C6—C7—C14	−54.3 (2)	C13—C15—C20—N5	0.96 (16)
C5—C6—C7—C14	130.43 (19)	C14—C13—C21—O4	−56.51 (19)
N3—C9—C12—C11	6.4 (2)	C15—C13—C21—O4	−177.41 (15)
N2—C9—C12—C11	−174.55 (14)	C12—C13—C21—O4	63.1 (2)
N3—C9—C12—C13	−171.57 (13)	C14—C13—C21—N5	120.39 (13)
N2—C9—C12—C13	7.4 (2)	C15—C13—C21—N5	−0.51 (15)
O3—C11—C12—C9	174.82 (17)	C12—C13—C21—N5	−120.04 (13)
N4—C11—C12—C9	−4.0 (2)	N5—C22—C23—C24	115.0 (3)
O3—C11—C12—C13	−7.1 (2)	C14—C7—N1—O1	0.99 (18)
N4—C11—C12—C13	174.10 (13)	C6—C7—N1—O1	−177.31 (13)
C9—C12—C13—C14	−9.96 (19)	O1—C8—N2—C9	172.97 (13)
C11—C12—C13—C14	172.03 (13)	C14—C8—N2—C9	−8.5 (2)
C9—C12—C13—C15	115.92 (15)	C12—C9—N2—C8	2.1 (2)
C11—C12—C13—C15	−62.08 (17)	N3—C9—N2—C8	−178.79 (13)
C9—C12—C13—C21	−130.89 (15)	O2—C10—N3—C9	175.32 (15)
C11—C12—C13—C21	51.10 (18)	N4—C10—N3—C9	−4.6 (2)
O1—C8—C14—C7	0.51 (17)	C12—C9—N3—C10	−2.1 (2)
N2—C8—C14—C7	−178.07 (15)	N2—C9—N3—C10	178.80 (14)
O1—C8—C14—C13	−176.47 (13)	O2—C10—N4—C11	−172.78 (17)
N2—C8—C14—C13	5.0 (2)	N3—C10—N4—C11	7.1 (2)
N1—C7—C14—C8	−0.95 (18)	O3—C11—N4—C10	178.19 (17)
C6—C7—C14—C8	177.06 (16)	C12—C11—N4—C10	−2.9 (2)
N1—C7—C14—C13	175.41 (16)	O4—C21—N5—C20	178.07 (15)
C6—C7—C14—C13	−6.6 (3)	C13—C21—N5—C20	1.14 (17)
C15—C13—C14—C8	−120.20 (15)	O4—C21—N5—C22	4.3 (3)
C12—C13—C14—C8	4.21 (19)	C13—C21—N5—C22	−172.62 (14)
C21—C13—C14—C8	127.21 (15)	C19—C20—N5—C21	179.55 (16)
C15—C13—C14—C7	64.0 (2)	C15—C20—N5—C21	−1.35 (18)
C12—C13—C14—C7	−171.59 (16)	C19—C20—N5—C22	−6.7 (3)

C21—C13—C14—C7	−48.6 (2)	C15—C20—N5—C22	172.37 (14)
C14—C13—C15—C16	60.4 (2)	C23—C22—N5—C21	91.4 (2)
C12—C13—C15—C16	−61.6 (2)	C23—C22—N5—C20	−81.6 (2)
C21—C13—C15—C16	177.99 (15)	C14—C8—O1—N1	0.05 (17)
C14—C13—C15—C20	−117.90 (13)	N2—C8—O1—N1	178.76 (13)
C12—C13—C15—C20	120.11 (13)	C7—N1—O1—C8	−0.64 (17)
C21—C13—C15—C20	−0.29 (15)		

Hydrogen-bond geometry (Å, º)

Cg is the centroid of the C15–C20 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O5	0.86	2.03	2.862 (2)	163
C1—H1···*Cg*	0.93	2.88	3.624 (2)	138
N2—H2*A*···O4^{i}	0.86	2.11	2.7631 (16)	132
N3—H3*A*···O2^{ii}	0.86	1.99	2.7887 (18)	155
C17—H17···N1^{iii}	0.93	2.53	3.412 (2)	159
C19—H19···O3^{iv}	0.93	2.42	3.324 (2)	165
C26—H26*C*···O1^{v}	0.96	2.59	3.322 (3)	133

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