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2,3,4,6,7,8,9,10-Octahydropyrimido-[1,2-*a*]azepin-1-ium 2-cyano-1-(2,6-dimethylanilino)-2-(phenylsulfonyl)-ethenethiolate

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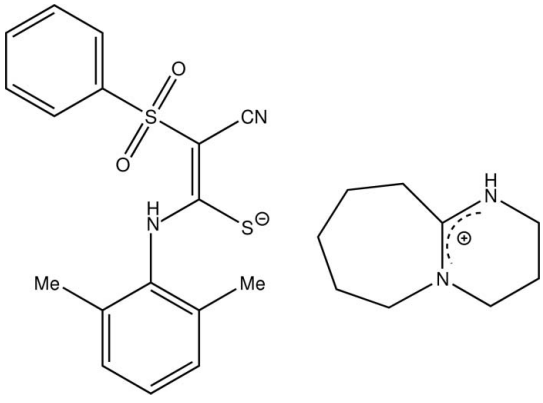
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.090; data-to-parameter ratio = 13.6.

In the title compound, $C_9H_{17}N_2^+\cdot C_{17}H_{15}N_2O_2S_2^-$, the Csp^2—N bonds in the tetrahydropyrimidine ring of the cation are delocalized. The negative charge is localized on the S atom of the thione group. Cations and anions are linked by N—H···S intermolecular hydrogen bonds.

Related literature

For related literature, see: Bürgi & Dunitz (1994); Fadda *et al.* (2000); Dorwald (2000); Lindeman *et al.* (2003); Perez *et al.* (2004); Zefirov *et al.* (1990).



Experimental

Crystal data

$C_9H_{17}N_2^+\cdot C_{17}H_{15}N_2O_2S_2^-$
M_r = 496.68
Triclinic, $P\overline{1}$
a = 8.3278 (5) Å
b = 11.348 (4) Å
c = 13.661 (3) Å
α = 104.03 (2)°
β = 92.517 (9)°
γ = 90.635 (12)°
V = 1250.9 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
0.20 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur3 diffractometer
Absorption correction: none
11732 measured reflections
4319 independent reflections
3564 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.090
S = 1.10
4319 reflections
317 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected bond lengths (Å).

S1—O1	1.4285 (12)	N3—C23	1.308 (2)
S1—O2	1.4413 (13)	N4—C23	1.305 (2)
S1—C1	1.7291 (16)	C1—C2	1.411 (2)
S2—C2	1.7037 (16)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2	0.86 (2)	1.98 (2)	2.7091 (19)	143 (2)
N4—H4*N*···S2^{i}	0.82 (2)	2.45 (2)	3.2335 (19)	161 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL-Plus*; molecular graphics: *XP* (Siemens, 1998); software used to prepare material for publication: *SHELXTL-Plus*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AV2006).

References

Bürgi, H.-B. & Dunitz, J. D. (1994). *Structure Correlation*, Vol. 2, pp. 767–784. Weinheim: VCH.
Fadda, A. A., Refat, H. M. & Zaki, M. E. A. (2000). *Molecules.* **5**, 701–709.
Dorwald, F. Z. (2000). USA Patent 6 136 984.
Lindeman, S. V., Hecht, J. & Kochi, J. K. (2003). *J. Am. Chem. Soc.* **125**, 11597–11606.
Oxford Diffraction (2007). *CrysAlis CCD* and *CrysAlis RED*. Versions 1.171.31.8. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Perez, E. R., Santos, R. H. A., Gambardella, M. T. P., de Macedo, L. G. M., Rogrigues-Filho, U. P., Launay, J.-C. & Franco, D. W. (2004). *J. Org. Chem.* **69**, 8005–8011.
Sheldrick, G. M. (1998). *SHELXTL-Plus*. PC Version 5.1. Bruker AXS, Madison Wisconsin, USA.
Siemens (1998). *XP*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
Zefirov, N. S., Palyulin, V. A. & Dashevskaya, E. E. (1990). *J. Phys. Org. Chem.* **3**, 147–154.

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2,3,4,6,7,8,9,10-Octahydropyrimido[1,2-*a*]azepin-1-ium 2-cyano-1-(2,6-dimethylanilino)-2-(phenylsulfonyl)ethenethiolate

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S1. Comment

The S,*N*-acetaleketenes are very efficient reagents for synthesis of the sulfur-containing heterocycles. However, synthesis and isolation of these compounds from the reaction mixture are difficult enough (Fadda, Refat Hala & Zaki, 2000; Florencio, 2000). Therefore information about crystal structure of these compounds is very limited. In this paper we report the molecular and crystal structure of the salt of the α-nitryl-α-phenylsulfonylketene *S*,*N*-(2,6-dimethylphenyl)-acetal with 1,8-Diaza-bicyclo[5,4,0]undec-7-ene. The N3—C23 and N4—C23 bonds of the cation have very close lengths (Table 1) which significantly differ from their mean values 1.355 Å and 1.279 Å (Bürgi & Dunitz, 1994), respectively. Similar deformation of these bonds was observed earlier in the protonated octahydropyrimidoazepines (Perez *et al.*, 2004; Lindeman *et al.*, 2003). The azepine ring of the cation adopts a chair conformation. The C18, C19, C21 and C22 atoms lie in the plane within 0.01 Å. Deviations of the C20, N3, C23 atoms from this plane are 0.65 Å, -1.08 Å and -1.03 Å, respectively. The tetrahydropyrimidine ring adopts conformation which is an intermediate between sofa and half-chair (the puckering parameters (Zefirov *et al.*, 1990) are S=0.68, θ=42.0 °, ψ=19.8 °). Deviations of the C25 and C26 atoms from the mean-square plane of the remaining atoms of the ring are -0.76 Å and -0.23 Å, respectively. The value of the C2—S2 bond in anion is shorter than the mean value for Csp^2—S bond (1.751 Å) and is longer as compared with Csp^2?S bond (1.671 Å). This indicates that the negative charge is localized on the S2 atom. The C2, C1, N1, S1, S2 and C11 atoms of organic anion lie in the plane within 0.02 Å. The aromatic ring of the phenylsulfoxide substituent is orthogonal to the C1—C2 bond and is turned relatively the C1—S1 bond (the C12—S1—C1—C2 and C1—S1—C12—C17 torsion angles are 105.4 (1) ° and 112.7 (1)°, respectively). Formation of the N1—H1N···O2 intramolecular hydrogen bond (H···O 1.99 Å, N—H···O 144 °) causes almost coplanar arrangement of the S1—O2 and C1—C2 bonds (the O2—S1—C1—C2 torsion angle is -9.3 (2) °) and it leads to some elongation of these bonds as compared to mean values 1.436 Å for the S=O and 1.331 Å for the C=C bonds, and shortening of the C1—S1 bond (mean value is 1.779 Å). In the crystal phase the cations and anions are bonded by the N4—H4n···S2^{i} intermolecular hydrogen bond (i) *x*, 1 + *y*, *z*; H···S 2.41 Å, N—H···S 160°).

S2. Experimental

The reaction was carried out in the methanolic solution of 1,8-diaza-bicyclo[5,4,0]undec-7-ene (1,66 mmol) with heating (about 50 °C), in which arylsulfonylacetonitrile (1,66 mmol) and arylisothiocyanate (1,66 mmol) were dissolved. The solution was mixed during 1.5 h. At the end of synthesis the reacting mixture was cooled and salt was crystallized from a solution.

S3. Refinement

All hydrogen atoms were located from electron density difference maps and included in the refinement in the riding model approximation with U_{iso} constrained to be 1.5 times U_{eq} of the carrier atom for the methyl groups and 1.2 times U_{eq} of the carrier atom for the other atoms.The hydrogen atoms which take part in formation of hydrogen bonds were refined in isotropic approximation.

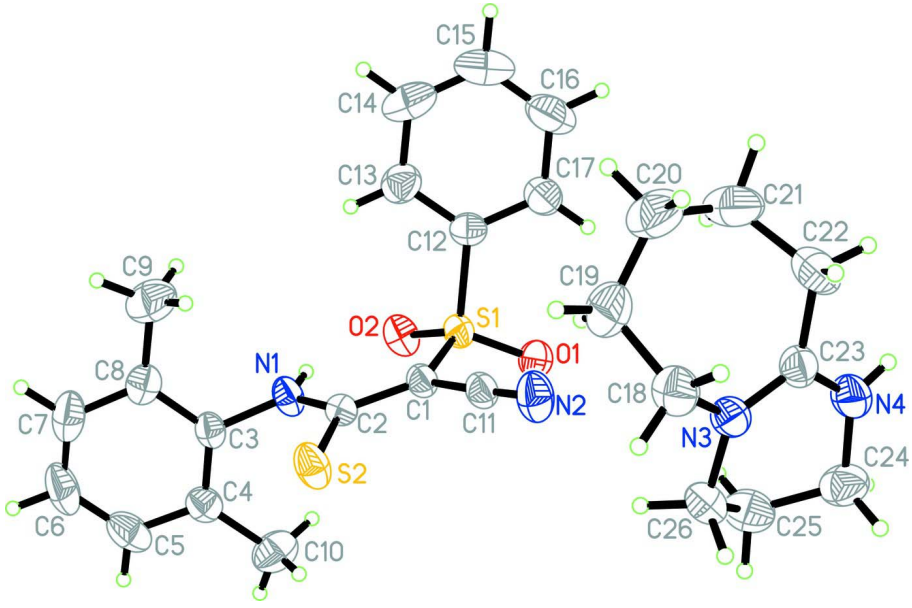

Figure 1

View of the title compound with atomic numbering scheme. All atoms are shown with displacement ellipsoids drawn at the 50% probability level.

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Crystal data

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Triclinic, $P\overline{1}$
$a = 8.3278$ (5) Å
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$c = 13.661$ (3) Å
$\alpha = 104.03$ (2)°
$\beta = 92.517$ (9)°
$\gamma = 90.635$ (12)°
$V = 1250.9$ (5) Å^3
$Z = 2$
$F(000) = 528$
$D_x = 1.319$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9472 reflections
$\theta = 2.8$–$31.9°$
$\mu = 0.24$ mm^{-1}
$T = 293$ K
Block, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.1827 pixels mm^{-1}
ω–scans
11732 measured reflections
4319 independent reflections
3564 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.0°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.090$
$S = 1.10$
4319 reflections
317 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.3755P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.12739 (5)	0.23234 (3)	0.07779 (3)	0.03153 (12)
S2	0.32290 (6)	0.09686 (4)	0.32967 (3)	0.04391 (14)
O1	0.21822 (14)	0.32333 (11)	0.04679 (9)	0.0437 (3)
O2	0.10480 (15)	0.11538 (10)	0.00763 (9)	0.0420 (3)
N1	0.17889 (17)	−0.00391 (12)	0.15248 (11)	0.0365 (3)
H1N	0.136 (2)	0.0021 (17)	0.0957 (16)	0.045 (5)*
N2	0.2919 (2)	0.41319 (14)	0.31254 (12)	0.0506 (4)
N3	0.40259 (16)	0.72438 (12)	0.50300 (10)	0.0363 (3)
N4	0.42004 (19)	0.85309 (15)	0.39899 (13)	0.0459 (4)
H4N	0.376 (3)	0.904 (2)	0.3749 (16)	0.057 (6)*
C1	0.21102 (18)	0.21136 (14)	0.19027 (12)	0.0309 (3)
C2	0.23091 (17)	0.09922 (14)	0.21630 (12)	0.0303 (3)
C3	0.20953 (19)	−0.12349 (14)	0.16308 (12)	0.0323 (3)
C4	0.3526 (2)	−0.17697 (15)	0.12811 (13)	0.0382 (4)
C5	0.3722 (3)	−0.29728 (17)	0.12940 (15)	0.0524 (5)
H5	0.4660	−0.3360	0.1062	0.063*
C6	0.2562 (3)	−0.35999 (17)	0.16407 (17)	0.0586 (6)
H6	0.2713	−0.4412	0.1637	0.070*
C7	0.1188 (3)	−0.30538 (17)	0.19914 (15)	0.0532 (5)
H7	0.0420	−0.3492	0.2239	0.064*
C8	0.0913 (2)	−0.18553 (16)	0.19868 (13)	0.0393 (4)
C9	−0.0614 (2)	−0.1257 (2)	0.23623 (17)	0.0623 (6)
H9C	−0.1219	−0.1788	0.2665	0.093*
H9B	−0.1239	−0.1088	0.1807	0.093*
H9A	−0.0361	−0.0511	0.2855	0.093*

C10	0.4806 (2)	−0.1070 (2)	0.09221 (17)	0.0587 (5)
H10C	0.5594	−0.1619	0.0591	0.088*
H10B	0.5311	−0.0495	0.1488	0.088*
H10A	0.4337	−0.0646	0.0456	0.088*
C11	0.25701 (19)	0.32106 (14)	0.25980 (12)	0.0337 (4)
C12	−0.06628 (19)	0.29086 (15)	0.10429 (12)	0.0343 (4)
C13	−0.1846 (2)	0.21876 (18)	0.12817 (14)	0.0472 (4)
H13	−0.1636	0.1392	0.1306	0.057*
C14	−0.3353 (2)	0.2659 (2)	0.14860 (16)	0.0600 (6)
H14	−0.4158	0.2181	0.1657	0.072*
C15	−0.3663 (3)	0.3824 (2)	0.14384 (17)	0.0665 (6)
H15	−0.4679	0.4136	0.1573	0.080*
C16	−0.2487 (3)	0.4526 (2)	0.11951 (19)	0.0653 (6)
H16	−0.2706	0.5319	0.1166	0.078*
C17	−0.0964 (2)	0.40779 (17)	0.09902 (15)	0.0473 (4)
H17	−0.0163	0.4560	0.0820	0.057*
C18	0.3354 (2)	0.69195 (18)	0.59082 (14)	0.0473 (4)
H18B	0.3227	0.7656	0.6433	0.057*
H18A	0.4110	0.6414	0.6170	0.057*
C19	0.1752 (2)	0.6252 (2)	0.56735 (18)	0.0589 (5)
H19B	0.1787	0.5667	0.5027	0.071*
H19A	0.1577	0.5806	0.6183	0.071*
C20	0.0358 (2)	0.7076 (2)	0.56372 (19)	0.0656 (6)
H20A	0.0282	0.7624	0.6299	0.079*
H20B	−0.0624	0.6585	0.5492	0.079*
C21	0.0472 (2)	0.7822 (2)	0.48615 (18)	0.0639 (6)
H21B	−0.0502	0.8283	0.4865	0.077*
H21A	0.0515	0.7271	0.4199	0.077*
C22	0.1902 (2)	0.86958 (18)	0.50166 (17)	0.0547 (5)
H22B	0.1666	0.9322	0.4662	0.066*
H22A	0.2040	0.9088	0.5730	0.066*
C23	0.3450 (2)	0.81263 (15)	0.46655 (13)	0.0376 (4)
C24	0.5760 (2)	0.80974 (19)	0.36486 (16)	0.0527 (5)
H24B	0.6604	0.8544	0.4105	0.063*
H24A	0.5910	0.8221	0.2980	0.063*
C25	0.5840 (2)	0.67836 (19)	0.36216 (15)	0.0529 (5)
H25B	0.6914	0.6500	0.3460	0.063*
H25A	0.5091	0.6329	0.3098	0.063*
C26	0.5433 (2)	0.65656 (16)	0.46223 (14)	0.0429 (4)
H26B	0.5223	0.5704	0.4545	0.051*
H26A	0.6347	0.6805	0.5097	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0346 (2)	0.0303 (2)	0.0314 (2)	0.00208 (15)	0.00147 (16)	0.01090 (16)
S2	0.0605 (3)	0.0321 (2)	0.0387 (3)	0.0009 (2)	−0.0147 (2)	0.01085 (18)
O1	0.0424 (6)	0.0452 (7)	0.0508 (7)	−0.0001 (5)	0.0073 (6)	0.0248 (6)

O2	0.0575 (7)	0.0361 (6)	0.0312 (6)	0.0053 (5)	−0.0030 (5)	0.0065 (5)
N1	0.0487 (8)	0.0268 (7)	0.0335 (8)	0.0009 (6)	−0.0098 (7)	0.0083 (6)
N2	0.0681 (11)	0.0335 (8)	0.0479 (9)	−0.0050 (7)	−0.0077 (8)	0.0076 (7)
N3	0.0370 (7)	0.0378 (8)	0.0365 (8)	0.0020 (6)	−0.0005 (6)	0.0143 (6)
N4	0.0464 (9)	0.0440 (9)	0.0553 (10)	0.0043 (7)	0.0041 (7)	0.0271 (8)
C1	0.0332 (8)	0.0278 (8)	0.0323 (8)	0.0011 (6)	−0.0026 (6)	0.0093 (7)
C2	0.0283 (7)	0.0293 (8)	0.0338 (8)	0.0014 (6)	0.0003 (6)	0.0085 (7)
C3	0.0404 (9)	0.0257 (8)	0.0303 (8)	−0.0013 (6)	−0.0055 (7)	0.0073 (6)
C4	0.0399 (9)	0.0380 (9)	0.0353 (9)	0.0024 (7)	−0.0031 (7)	0.0067 (7)
C5	0.0583 (12)	0.0405 (10)	0.0526 (12)	0.0158 (9)	−0.0086 (9)	0.0014 (9)
C6	0.0874 (16)	0.0262 (9)	0.0608 (13)	−0.0007 (10)	−0.0164 (12)	0.0114 (9)
C7	0.0717 (13)	0.0409 (10)	0.0488 (11)	−0.0194 (10)	−0.0084 (10)	0.0174 (9)
C8	0.0444 (9)	0.0403 (9)	0.0327 (9)	−0.0070 (7)	−0.0024 (7)	0.0086 (7)
C9	0.0500 (11)	0.0808 (15)	0.0576 (13)	−0.0012 (11)	0.0125 (10)	0.0184 (12)
C10	0.0486 (11)	0.0661 (13)	0.0597 (13)	−0.0044 (10)	0.0123 (10)	0.0107 (11)
C11	0.0370 (8)	0.0302 (9)	0.0356 (9)	0.0019 (7)	−0.0024 (7)	0.0122 (7)
C12	0.0326 (8)	0.0403 (9)	0.0295 (8)	0.0017 (7)	−0.0024 (6)	0.0080 (7)
C13	0.0432 (10)	0.0556 (11)	0.0466 (11)	−0.0027 (8)	0.0015 (8)	0.0199 (9)
C14	0.0379 (10)	0.0952 (18)	0.0475 (11)	−0.0059 (11)	0.0051 (9)	0.0184 (11)
C15	0.0423 (11)	0.0912 (18)	0.0594 (14)	0.0203 (12)	0.0024 (10)	0.0045 (12)
C16	0.0546 (12)	0.0571 (13)	0.0788 (16)	0.0213 (10)	−0.0019 (11)	0.0063 (12)
C17	0.0441 (10)	0.0400 (10)	0.0564 (12)	0.0035 (8)	−0.0029 (9)	0.0103 (9)
C18	0.0533 (11)	0.0528 (11)	0.0413 (10)	0.0029 (9)	0.0040 (8)	0.0220 (9)
C19	0.0607 (12)	0.0579 (13)	0.0649 (13)	−0.0064 (10)	0.0129 (10)	0.0266 (11)
C20	0.0470 (11)	0.0824 (16)	0.0721 (15)	−0.0043 (11)	0.0142 (11)	0.0263 (13)
C21	0.0391 (10)	0.0882 (16)	0.0709 (15)	0.0131 (10)	0.0090 (10)	0.0305 (13)
C22	0.0573 (12)	0.0495 (11)	0.0635 (13)	0.0199 (9)	0.0147 (10)	0.0232 (10)
C23	0.0400 (9)	0.0339 (9)	0.0399 (9)	0.0001 (7)	−0.0008 (7)	0.0113 (7)
C24	0.0401 (10)	0.0677 (13)	0.0576 (12)	0.0005 (9)	0.0076 (9)	0.0284 (11)
C25	0.0489 (11)	0.0609 (12)	0.0489 (11)	0.0122 (9)	0.0076 (9)	0.0121 (10)
C26	0.0446 (10)	0.0385 (9)	0.0465 (10)	0.0094 (8)	0.0007 (8)	0.0122 (8)

Geometric parameters (Å, º)

S1—O1	1.4285 (12)	C12—C17	1.371 (2)
S1—O2	1.4413 (13)	C12—C13	1.373 (2)
S1—C1	1.7291 (16)	C13—C14	1.383 (3)
S1—C12	1.7697 (16)	C13—H13	0.9300
S2—C2	1.7037 (16)	C14—C15	1.366 (3)
N1—C2	1.334 (2)	C14—H14	0.9300
N1—C3	1.424 (2)	C15—C16	1.358 (3)
N1—H1N	0.86 (2)	C15—H15	0.9300
N2—C11	1.143 (2)	C16—C17	1.387 (3)
N3—C23	1.308 (2)	C16—H16	0.9300
N3—C26	1.465 (2)	C17—H17	0.9300
N3—C18	1.469 (2)	C18—C19	1.509 (3)
N4—C23	1.305 (2)	C18—H18B	0.9700
N4—C24	1.450 (2)	C18—H18A	0.9700

N4—H4N	0.82 (2)	C19—C20	1.505 (3)
C1—C11	1.408 (2)	C19—H19B	0.9700
C1—C2	1.411 (2)	C19—H19A	0.9700
C3—C8	1.377 (2)	C20—C21	1.514 (3)
C3—C4	1.393 (2)	C20—H20A	0.9700
C4—C5	1.381 (3)	C20—H20B	0.9700
C4—C10	1.491 (3)	C21—C22	1.515 (3)
C5—C6	1.361 (3)	C21—H21B	0.9700
C5—H5	0.9300	C21—H21A	0.9700
C6—C7	1.358 (3)	C22—C23	1.494 (2)
C6—H6	0.9300	C22—H22B	0.9700
C7—C8	1.383 (3)	C22—H22A	0.9700
C7—H7	0.9300	C24—C25	1.485 (3)
C8—C9	1.500 (3)	C24—H24B	0.9700
C9—H9C	0.9600	C24—H24A	0.9700
C9—H9B	0.9600	C25—C26	1.499 (3)
C9—H9A	0.9600	C25—H25B	0.9700
C10—H10C	0.9600	C25—H25A	0.9700
C10—H10B	0.9600	C26—H26B	0.9700
C10—H10A	0.9600	C26—H26A	0.9700

O1—S1—O2	117.84 (8)	C16—C15—C14	120.04 (19)
O1—S1—C1	109.46 (8)	C16—C15—H15	120.0
O2—S1—C1	108.65 (7)	C14—C15—H15	120.0
O1—S1—C12	107.00 (8)	C15—C16—C17	120.9 (2)
O2—S1—C12	106.90 (8)	C15—C16—H16	119.5
C1—S1—C12	106.39 (8)	C17—C16—H16	119.5
C2—N1—C3	125.95 (14)	C12—C17—C16	118.62 (19)
C2—N1—H1N	116.6 (13)	C12—C17—H17	120.7
C3—N1—H1N	116.7 (13)	C16—C17—H17	120.7
C23—N3—C26	122.01 (14)	N3—C18—C19	113.53 (16)
C23—N3—C18	121.92 (15)	N3—C18—H18B	108.9
C26—N3—C18	115.99 (14)	C19—C18—H18B	108.9
C23—N4—C24	122.62 (16)	N3—C18—H18A	108.9
C23—N4—H4N	118.0 (15)	C19—C18—H18A	108.9
C24—N4—H4N	119.4 (15)	H18B—C18—H18A	107.7
C11—C1—C2	120.44 (14)	C20—C19—C18	113.49 (18)
C11—C1—S1	113.16 (11)	C20—C19—H19B	108.9
C2—C1—S1	126.38 (12)	C18—C19—H19B	108.9
N1—C2—C1	120.26 (14)	C20—C19—H19A	108.9
N1—C2—S2	120.37 (12)	C18—C19—H19A	108.9
C1—C2—S2	119.37 (12)	H19B—C19—H19A	107.7
C8—C3—C4	122.55 (15)	C19—C20—C21	114.37 (18)
C8—C3—N1	118.90 (15)	C19—C20—H20A	108.7
C4—C3—N1	118.28 (15)	C21—C20—H20A	108.7
C5—C4—C3	117.24 (17)	C19—C20—H20B	108.7
C5—C4—C10	121.19 (18)	C21—C20—H20B	108.7
C3—C4—C10	121.56 (16)	H20A—C20—H20B	107.6

C6—C5—C4	120.93 (19)	C20—C21—C22	115.35 (19)
C6—C5—H5	119.5	C20—C21—H21B	108.4
C4—C5—H5	119.5	C22—C21—H21B	108.4
C7—C6—C5	120.74 (18)	C20—C21—H21A	108.4
C7—C6—H6	119.6	C22—C21—H21A	108.4
C5—C6—H6	119.6	H21B—C21—H21A	107.5
C6—C7—C8	121.06 (19)	C23—C22—C21	114.77 (17)
C6—C7—H7	119.5	C23—C22—H22B	108.6
C8—C7—H7	119.5	C21—C22—H22B	108.6
C3—C8—C7	117.46 (17)	C23—C22—H22A	108.6
C3—C8—C9	121.48 (17)	C21—C22—H22A	108.6
C7—C8—C9	121.06 (18)	H22B—C22—H22A	107.6
C8—C9—H9C	109.5	N4—C23—N3	121.38 (16)
C8—C9—H9B	109.5	N4—C23—C22	118.27 (16)
H9C—C9—H9B	109.5	N3—C23—C22	120.34 (16)
C8—C9—H9A	109.5	N4—C24—C25	108.70 (16)
H9C—C9—H9A	109.5	N4—C24—H24B	110.0
H9B—C9—H9A	109.5	C25—C24—H24B	110.0
C4—C10—H10C	109.5	N4—C24—H24A	110.0
C4—C10—H10B	109.5	C25—C24—H24A	110.0
H10C—C10—H10B	109.5	H24B—C24—H24A	108.3
C4—C10—H10A	109.5	C24—C25—C26	110.45 (17)
H10C—C10—H10A	109.5	C24—C25—H25B	109.6
H10B—C10—H10A	109.5	C26—C25—H25B	109.6
N2—C11—C1	176.46 (17)	C24—C25—H25A	109.6
C17—C12—C13	120.95 (17)	C26—C25—H25A	109.6
C17—C12—S1	119.06 (13)	H25B—C25—H25A	108.1
C13—C12—S1	119.98 (14)	N3—C26—C25	112.07 (14)
C12—C13—C14	119.24 (19)	N3—C26—H26B	109.2
C12—C13—H13	120.4	C25—C26—H26B	109.2
C14—C13—H13	120.4	N3—C26—H26A	109.2
C15—C14—C13	120.2 (2)	C25—C26—H26A	109.2
C15—C14—H14	119.9	H26B—C26—H26A	107.9
C13—C14—H14	119.9		

O1—S1—C1—C11	42.43 (14)	O1—S1—C12—C13	174.72 (14)
O2—S1—C1—C11	172.37 (11)	O2—S1—C12—C13	47.62 (16)
C12—S1—C1—C11	−72.86 (13)	C1—S1—C12—C13	−68.34 (16)
O1—S1—C1—C2	−139.31 (14)	C17—C12—C13—C14	−1.0 (3)
O2—S1—C1—C2	−9.36 (17)	S1—C12—C13—C14	−179.96 (14)
C12—S1—C1—C2	105.41 (15)	C12—C13—C14—C15	0.8 (3)
C3—N1—C2—C1	171.24 (15)	C13—C14—C15—C16	−0.4 (3)
C3—N1—C2—S2	−8.4 (2)	C14—C15—C16—C17	0.1 (4)
C11—C1—C2—N1	176.19 (14)	C13—C12—C17—C16	0.8 (3)
S1—C1—C2—N1	−2.0 (2)	S1—C12—C17—C16	179.74 (15)
C11—C1—C2—S2	−4.2 (2)	C15—C16—C17—C12	−0.3 (3)
S1—C1—C2—S2	177.69 (9)	C23—N3—C18—C19	74.2 (2)
C2—N1—C3—C8	101.1 (2)	C26—N3—C18—C19	−109.05 (18)

C2—N1—C3—C4	−84.8 (2)	N3—C18—C19—C20	−80.1 (2)
C8—C3—C4—C5	0.6 (2)	C18—C19—C20—C21	59.1 (3)
N1—C3—C4—C5	−173.36 (15)	C19—C20—C21—C22	−61.7 (3)
C8—C3—C4—C10	−178.51 (17)	C20—C21—C22—C23	79.5 (2)
N1—C3—C4—C10	7.6 (2)	C24—N4—C23—N3	−4.3 (3)
C3—C4—C5—C6	−0.4 (3)	C24—N4—C23—C22	175.81 (18)
C10—C4—C5—C6	178.72 (19)	C26—N3—C23—N4	−7.3 (3)
C4—C5—C6—C7	−0.6 (3)	C18—N3—C23—N4	169.31 (17)
C5—C6—C7—C8	1.4 (3)	C26—N3—C23—C22	172.61 (16)
C4—C3—C8—C7	0.2 (2)	C18—N3—C23—C22	−10.8 (3)
N1—C3—C8—C7	174.06 (16)	C21—C22—C23—N4	122.0 (2)
C4—C3—C8—C9	179.79 (17)	C21—C22—C23—N3	−57.8 (3)
N1—C3—C8—C9	−6.3 (2)	C23—N4—C24—C25	35.4 (3)
C6—C7—C8—C3	−1.2 (3)	N4—C24—C25—C26	−53.5 (2)
C6—C7—C8—C9	179.24 (18)	C23—N3—C26—C25	−14.2 (2)
O1—S1—C12—C17	−4.27 (16)	C18—N3—C26—C25	169.07 (16)
O2—S1—C12—C17	−131.37 (14)	C24—C25—C26—N3	44.5 (2)
C1—S1—C12—C17	112.67 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2	0.86 (2)	1.98 (2)	2.7091 (19)	143 (2)
N4—H4*N*···S2^{i}	0.82 (2)	2.45 (2)	3.2335 (19)	161 (2)

Symmetry code: (i) *x*, *y*+1, *z*.