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S,*S*-Diphenyl-*S*-pyrrolidinoiminosulfonium perchlorate

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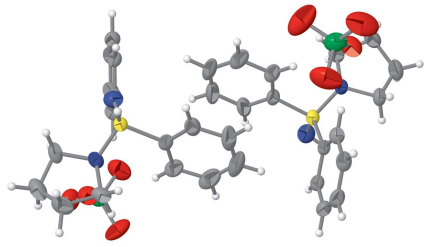
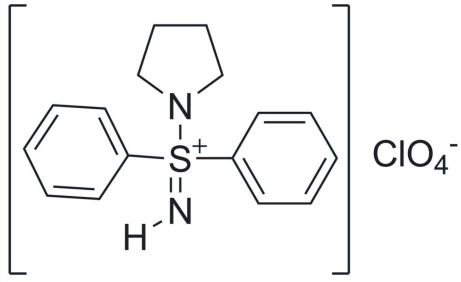
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title salt, $C_{16}H_{19}N_2S^+\cdot ClO_4^-$, consists of two crystallographically independent cations and anions. In the salt, protonation occurs at the nitrile N atom attached to the S atom of the corresponding λ^6-sulfanenitrile. The structures of the two independent cations are almost the same and the configuration around the S atom is a slightly distorted tetrahedral geometry, with two S—N bonds and two S—C bonds. The S—N(pyrrolidine) and S=N bond lengths are 1.6216 (18) and 1.503 (2) Å, respectively, for one cation, and 1.6236 (19) and 1.502 (2) Å, respectively, for the other. The dihedral angles between the two phenyl rings in the cations are 76.61 (9) and 76.42 (9)°. There are N—H···O hydrogen bonds, which link the cation and the anion. The cation–anion pairs are further linked by C—H···O and C—H···N hydrogen bonds, forming a three-dimensional network.

3D view



Chemical scheme



Structure description

The chemistry of heteroatom-substituted sulfonium salts (heterosulfonium salts) is very interesting because of their anomalous reactivity (Oae *et al.*, 1981). Meanwhile, only a few sulfur(VI) sulfonium compounds, such as oxosulfonium salts, have been reported to date (Mori *et al.*, 1990). Furthermore, iminosulfonium salts belong to the isoelectronic compounds of the oxosulfonium salts and are very rare. Previously, we prepared *S*,*S*,*S*-triphenylsulfanenitrile bearing an S≡N triple bond and found that its N atom has a nucleophilic character (Yoshimura *et al.*, 1997) and thus reported the preparation of iminosulfonium salts bearing three carbon ligands by its alkylation or neutralization (Yoshimura *et al.*, 1998). However, iminosulfonium salts bearing two carbons and one amino ligand are also very rare. Furthermore, we reported that the reaction of *S*,*S*-diaryl-

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H19···O2^{i}	0.76 (4)	2.34 (4)	3.057 (4)	159 (4)
N4—H38···O8^{ii}	0.79 (3)	2.16 (3)	2.952 (4)	173 (3)
C2—H1···O1	0.95	2.58	3.531 (3)	178
C10—H8···O7^{iii}	0.95	2.56	3.453 (4)	157
C12—H10···O1	0.95	2.43	3.379 (4)	174
C16—H18···O4^{i}	0.99	2.58	3.392 (4)	139
C18—H20···O6	0.95	2.47	3.362 (3)	156
C25—H26···O3^{iv}	0.95	2.57	3.512 (5)	171
C29—H30···O5^{ii}	0.99	2.50	3.344 (3)	142
C28—H29···N4^{v}	0.95	2.58	3.401 (4)	145

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

S-fluorosulfanenitrile with cyclic secondary amines afforded the corresponding aminosulfanenitriles (Yoshimura, Kita *et al.*, 1992; Yoshimura, Takata *et al.*, 1992), though the corresponding pyrrolidinosulfanenitrile was an impure oily material and thus its identification remained ambiguous. The crystal structure of the title compound, which is now successfully resolved, is a precursor of the sulfanenitrile.

The molecular structure of the title compound is illustrated in Fig. 1. The S1—N1(pyrrolidine) and S1—N2(NH) bond lengths in one cation are 1.6216 (18) and 1.503 (2) Å, respectively. The corresponding bond lengths of the other cation, S2—N3(pyrrolidine) and S2—N4(NH), are 1.6236 (19) and 1.502 (2) Å, respectively. The S1—N2 and S2—N4 bond lengths are significantly longer than the S≡N triple bond of triphenylsulfanenitrile [1.462 (3) Å; Yoshimura *et al.*, 1997] and close to the double bond of *S*,*S*-dimethylsulfonediimine [1.533 (2) Å, electron diffraction study; Oberhammer & Zeil, 1969] and those of S1—N1 and S2—N3 are close to the S—N single bond of a sulfonediiminosulfonium salt [1.599 (3) Å; Ohkubo *et al.*, 1997]. There are N—H···O hydrogen bonds involving the perchlorate counter-anion. In the extended structure, the anions are linked through weak C—H···O and C—H···N hydrogen bonds, forming a three-dimensional network (Table 1 and Fig. 2).

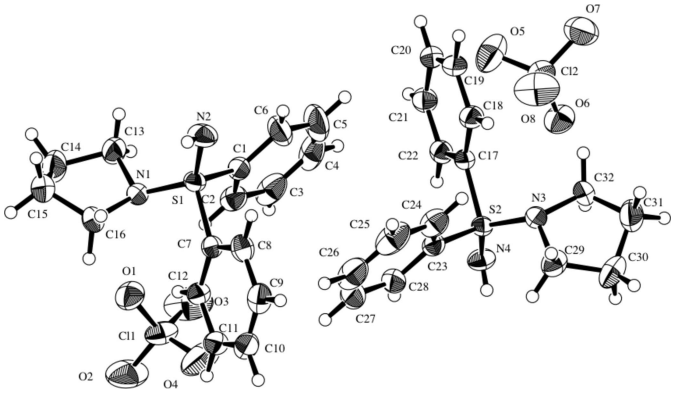


Figure 1
The asymmetric unit of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{19}N_2S^+\cdot ClO_4^-$
M_r	370.85
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, b, c (Å)	13.4430 (3), 15.9737 (3), 17.6478 (4)
β (°)	113.0241 (7)
V (Å^3)	3487.71 (12)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	3.26
Crystal size (mm)	0.55 × 0.47 × 0.12
Data collection	
Diffractometer	Rigaku R-AXIS RAPID
Absorption correction	Multi-scan (*ABSCOR*; Higashi, 1995)
T_{min}, T_{max}	0.549, 0.676
No. of measured, independent and observed [$F^2 > 2.0\sigma(F^2)$] reflections	40289, 6302, 5448
R_{int}	0.088
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.149, 1.12
No. of reflections	6302
No. of parameters	441
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.32

Computer programs: *RAPID-AUTO* (Rigaku, 2001), *SIR92* (Altomare *et al.*, 1993), *SHELXL97* (Sheldrick, 2008) and *CrystalStructure* (Rigaku, 2010).

Synthesis and crystallization

Fluorodiphenyl-λ^6-sulfanenitrile (Yoshimura *et al.*, 1998) (219 mg, 0.91 mmol) was treated with an excess of pyrrolidine

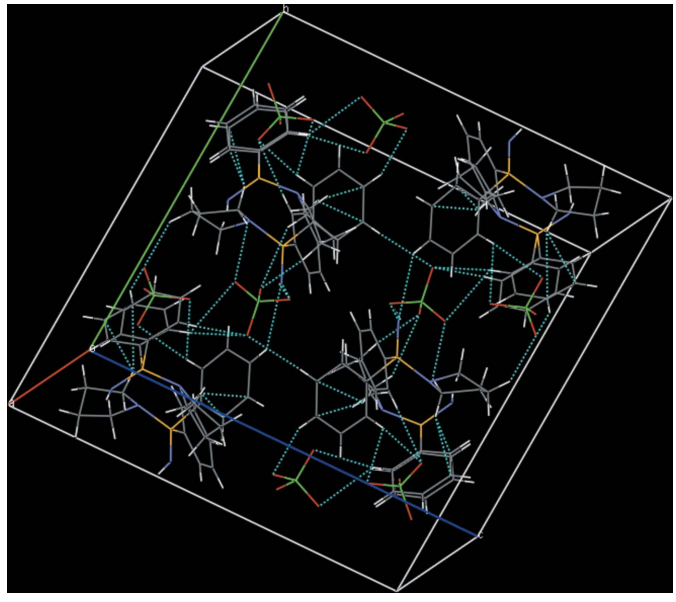


Figure 2
A packing view of the title compound, showing N—H···O, C—H···O, and C—H···N hydrogen bonds (dashed lines).

(512 mg, 7.2 mmol) at 303 K for 8 h. The excess pyrrolidine was removed in a vacuum and the residue was dissolved in $CHCl_3$ (20 ml). The solution was then washed with water (3 × 30 ml), dried over anhydrous $MgSO_4$ and concentrated under reduced pressure to give the corresponding *S*,*S*-diphenyl-*S*-pyrrolidino-λ^6-sulfanenitrile (yield: 226 mg, 92%) as a colourless oil. $HClO_4$ (183 mg, 1.8 mmol) was added to the ice-cooled oily pyrrolidino-λ^6-sulfanenitrile to give the corresponding title compound (yield: 250 mg, 74%) as a solid. Single crystals were obtained from a THF/hexane (1:1 *v*/*v*) solution (m.p. 414–415 K).

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171251 [https://doi.org/10.1107/S2414314617012512]

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Crystal data

$C_{16}H_{19}N_2S^+\cdot ClO_4^-$
M_r = 370.85
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.4430 (3) Å
b = 15.9737 (3) Å
c = 17.6478 (4) Å
β = 113.0241 (7)°
V = 3487.71 (12) Å^3
Z = 8
$F(000)$ = 1552.00
D_x = 1.412 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54187 Å
Cell parameters from 32602 reflections
θ = 3.5–68.3°
μ = 3.26 mm^{-1}
T = 173 K
Prism, colorless
0.55 × 0.47 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.000 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
T_{min} = 0.549, T_{max} = 0.676
40289 measured reflections
6302 independent reflections
5448 reflections with $F^2 > 2.0\sigma(F^2)$
R_{int} = 0.088
θ_{max} = 68.3°
h = −16→16
k = −19→19
l = −21→21

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.149
S = 1.12
6302 reflections
441 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0943P)^2 + 0.1549P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY
Refinement. Refinement was performed using all reflections. The weighted R-factor (wR) and goodness of fit (S) are based on F^2. R-factor (gt) are based on F. The threshold expression of $F^2 > 2.0$ sigma(F^2) is used only for calculating R-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.38944 (5)	0.11435 (3)	0.82405 (4)	0.05096 (19)
Cl2	−0.14167 (4)	0.38569 (3)	0.15363 (3)	0.04201 (17)
S1	0.36462 (4)	0.40529 (3)	0.66582 (3)	0.03120 (16)
S2	−0.08538 (4)	0.10611 (3)	0.34805 (3)	0.02793 (15)
O1	0.42043 (15)	0.19964 (10)	0.84419 (10)	0.0561 (5)
O2	0.4479 (2)	0.06208 (12)	0.89219 (13)	0.0890 (7)
O3	0.2754 (2)	0.10696 (12)	0.80241 (19)	0.1026 (9)
O4	0.4133 (3)	0.08960 (13)	0.75582 (14)	0.0900 (7)
O5	−0.13271 (18)	0.41880 (12)	0.23112 (12)	0.0768 (7)
O6	−0.14848 (14)	0.29633 (9)	0.15370 (10)	0.0536 (5)
O7	−0.23833 (14)	0.41859 (12)	0.09070 (12)	0.0683 (6)
O8	−0.05282 (16)	0.40968 (12)	0.13368 (15)	0.0802 (7)
N1	0.40788 (14)	0.39352 (10)	0.76479 (10)	0.0328 (4)
N2	0.35139 (18)	0.48934 (12)	0.62469 (13)	0.0479 (5)
N3	−0.11238 (14)	0.11995 (10)	0.25085 (10)	0.0342 (4)
N4	−0.10848 (16)	0.02588 (11)	0.38252 (11)	0.0390 (5)
C1	0.23451 (16)	0.35996 (13)	0.63014 (13)	0.0370 (5)
C2	0.21724 (18)	0.28907 (13)	0.66878 (15)	0.0438 (6)
C3	0.1160 (2)	0.25251 (17)	0.63550 (18)	0.0596 (8)
C4	0.0359 (2)	0.2863 (3)	0.56709 (19)	0.0720 (10)
C5	0.0552 (2)	0.3564 (3)	0.53012 (18)	0.0753 (10)
C6	0.1549 (2)	0.39426 (17)	0.56089 (15)	0.0561 (7)
C7	0.44895 (15)	0.33417 (12)	0.64093 (11)	0.0310 (5)
C8	0.47267 (17)	0.35375 (14)	0.57329 (13)	0.0396 (5)
C9	0.53268 (17)	0.29701 (15)	0.54894 (14)	0.0462 (6)
C10	0.56734 (17)	0.22232 (15)	0.59166 (14)	0.0466 (6)
C11	0.54229 (17)	0.20434 (13)	0.65898 (14)	0.0431 (6)
C12	0.48232 (16)	0.25963 (12)	0.68421 (12)	0.0359 (5)
C13	0.34319 (18)	0.43232 (16)	0.80815 (13)	0.0477 (6)
C14	0.4255 (2)	0.43849 (19)	0.89608 (14)	0.0585 (7)
C15	0.5300 (2)	0.45794 (16)	0.88720 (15)	0.0546 (7)
C16	0.52663 (17)	0.40682 (14)	0.81456 (13)	0.0417 (6)
C17	−0.16429 (15)	0.18334 (11)	0.37007 (11)	0.0303 (5)
C18	−0.17600 (17)	0.26117 (12)	0.33235 (12)	0.0378 (5)
C19	−0.23279 (17)	0.32256 (13)	0.35414 (13)	0.0418 (6)
C20	−0.27669 (17)	0.30589 (13)	0.41160 (13)	0.0412 (5)
C21	−0.26307 (17)	0.22830 (13)	0.44834 (13)	0.0403 (5)
C22	−0.20674 (15)	0.16573 (12)	0.42794 (12)	0.0341 (5)
C23	0.05042 (16)	0.14216 (12)	0.39332 (12)	0.0334 (5)
C24	0.0867 (2)	0.20851 (14)	0.36060 (15)	0.0479 (6)
C25	0.1929 (3)	0.23469 (18)	0.4032 (2)	0.0669 (9)
C26	0.2576 (2)	0.1960 (2)	0.4756 (2)	0.0732 (10)
C27	0.2200 (2)	0.13014 (19)	0.50700 (17)	0.0636 (8)
C28	0.11512 (18)	0.10252 (14)	0.46578 (14)	0.0439 (6)
C29	−0.03949 (19)	0.08011 (14)	0.21546 (13)	0.0443 (6)

C30	−0.1147 (3)	0.03031 (15)	0.14368 (15)	0.0570 (7)
C31	−0.2202 (3)	0.07948 (17)	0.11491 (15)	0.0598 (7)
C32	−0.22891 (19)	0.10621 (14)	0.19396 (13)	0.0452 (6)
H1	0.2729	0.2665	0.7163	0.0525*
H2	0.1017	0.2036	0.6602	0.0716*
H3	−0.0335	0.2607	0.5453	0.0864*
H4	−0.0008	0.3790	0.4828	0.0904*
H5	0.1688	0.4427	0.5353	0.0673*
H6	0.4485	0.4048	0.5443	0.0476*
H7	0.5501	0.3093	0.5029	0.0554*
H8	0.6082	0.1836	0.5747	0.0559*
H9	0.5666	0.1534	0.6881	0.0517*
H10	0.4643	0.2471	0.7299	0.0430*
H11	0.2815	0.3963	0.8044	0.0572*
H12	0.3158	0.4883	0.7854	0.0572*
H13	0.4061	0.4837	0.9262	0.0702*
H14	0.4307	0.3850	0.9257	0.0702*
H15	0.5927	0.4418	0.9375	0.0655*
H16	0.5350	0.5184	0.8769	0.0655*
H17	0.5643	0.3527	0.8327	0.0500*
H18	0.5607	0.4374	0.7822	0.0500*
H19	0.407 (3)	0.5079 (19)	0.633 (2)	0.077 (12)*
H20	−0.1459	0.2720	0.2928	0.0454*
H21	−0.2416	0.3764	0.3295	0.0502*
H22	−0.3163	0.3480	0.4257	0.0495*
H23	−0.2927	0.2176	0.4882	0.0484*
H24	−0.1976	0.1121	0.4531	0.0409*
H25	0.0409	0.2351	0.3111	0.0574*
H26	0.2210	0.2795	0.3821	0.0803*
H27	0.3295	0.2153	0.5043	0.0879*
H28	0.2657	0.1038	0.5567	0.0764*
H29	0.0878	0.0571	0.4867	0.0527*
H30	0.0141	0.0432	0.2564	0.0532*
H31	−0.0007	0.1231	0.1971	0.0532*
H32	−0.1247	−0.0270	0.1609	0.0684*
H33	−0.0868	0.0266	0.0995	0.0684*
H34	−0.2172	0.1287	0.0817	0.0717*
H35	−0.2821	0.0438	0.0817	0.0717*
H36	−0.2627	0.0619	0.2152	0.0542*
H37	−0.2714	0.1584	0.1863	0.0542*
H38	−0.069 (3)	−0.0082 (16)	0.3762 (17)	0.065 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0651 (5)	0.0311 (3)	0.0662 (4)	0.0069 (3)	0.0360 (4)	0.0044 (3)
Cl2	0.0461 (4)	0.0330 (3)	0.0509 (4)	−0.0072 (2)	0.0232 (3)	0.0009 (2)
S1	0.0299 (3)	0.0265 (3)	0.0337 (3)	0.00179 (17)	0.0086 (2)	0.00152 (17)

S2	0.0299 (3)	0.0242 (3)	0.0285 (3)	0.00118 (17)	0.0102 (2)	0.00214 (16)
O1	0.0770 (13)	0.0362 (9)	0.0530 (10)	−0.0018 (8)	0.0232 (9)	0.0014 (7)
O2	0.138 (2)	0.0565 (12)	0.0865 (15)	0.0370 (13)	0.0589 (15)	0.0311 (11)
O3	0.0711 (16)	0.0634 (14)	0.182 (3)	−0.0075 (11)	0.0584 (17)	−0.0055 (15)
O4	0.150 (3)	0.0563 (13)	0.0881 (16)	0.0058 (14)	0.0734 (16)	−0.0106 (11)
O5	0.1163 (19)	0.0560 (12)	0.0627 (13)	−0.0213 (12)	0.0402 (13)	−0.0161 (10)
O6	0.0725 (12)	0.0332 (9)	0.0582 (10)	−0.0063 (8)	0.0289 (9)	0.0039 (7)
O7	0.0571 (12)	0.0597 (12)	0.0802 (14)	0.0104 (9)	0.0182 (10)	0.0063 (10)
O8	0.0650 (13)	0.0598 (12)	0.136 (2)	−0.0101 (10)	0.0613 (14)	0.0111 (12)
N1	0.0285 (9)	0.0340 (9)	0.0330 (9)	0.0021 (7)	0.0089 (8)	−0.0029 (7)
N2	0.0505 (13)	0.0327 (10)	0.0570 (13)	0.0058 (9)	0.0172 (10)	0.0099 (9)
N3	0.0407 (10)	0.0302 (9)	0.0289 (9)	0.0043 (7)	0.0104 (8)	0.0018 (7)
N4	0.0488 (11)	0.0260 (9)	0.0473 (11)	0.0029 (8)	0.0241 (9)	0.0044 (8)
C1	0.0271 (11)	0.0416 (12)	0.0392 (11)	0.0017 (9)	0.0097 (9)	−0.0094 (9)
C2	0.0368 (12)	0.0384 (12)	0.0561 (14)	−0.0049 (9)	0.0182 (11)	−0.0091 (10)
C3	0.0466 (15)	0.0581 (16)	0.083 (2)	−0.0187 (12)	0.0352 (15)	−0.0302 (14)
C4	0.0325 (14)	0.103 (3)	0.078 (2)	−0.0193 (15)	0.0188 (14)	−0.0517 (19)
C5	0.0339 (15)	0.120 (3)	0.0557 (18)	0.0016 (16)	−0.0003 (13)	−0.0209 (18)
C6	0.0379 (14)	0.0797 (19)	0.0396 (13)	0.0098 (12)	0.0032 (11)	−0.0011 (12)
C7	0.0259 (10)	0.0314 (10)	0.0327 (10)	−0.0020 (8)	0.0081 (8)	−0.0032 (8)
C8	0.0318 (11)	0.0461 (12)	0.0380 (11)	−0.0065 (9)	0.0104 (9)	−0.0006 (9)
C9	0.0337 (12)	0.0652 (16)	0.0421 (12)	−0.0069 (11)	0.0175 (10)	−0.0103 (11)
C10	0.0297 (11)	0.0565 (15)	0.0523 (14)	−0.0005 (10)	0.0146 (10)	−0.0183 (11)
C11	0.0368 (12)	0.0379 (12)	0.0470 (13)	0.0034 (9)	0.0083 (10)	−0.0055 (10)
C12	0.0356 (11)	0.0313 (10)	0.0383 (11)	0.0013 (9)	0.0118 (9)	−0.0017 (8)
C13	0.0424 (13)	0.0579 (15)	0.0415 (13)	0.0049 (11)	0.0150 (10)	−0.0121 (11)
C14	0.0555 (16)	0.0770 (19)	0.0398 (13)	0.0022 (14)	0.0152 (12)	−0.0139 (12)
C15	0.0484 (14)	0.0570 (15)	0.0456 (13)	0.0034 (12)	0.0045 (11)	−0.0115 (11)
C16	0.0314 (12)	0.0464 (13)	0.0388 (12)	−0.0005 (9)	0.0046 (10)	−0.0048 (9)
C17	0.0291 (10)	0.0257 (9)	0.0306 (10)	0.0017 (8)	0.0057 (8)	−0.0018 (8)
C18	0.0467 (13)	0.0309 (10)	0.0354 (11)	0.0039 (9)	0.0157 (10)	0.0029 (8)
C19	0.0496 (13)	0.0303 (11)	0.0396 (12)	0.0101 (9)	0.0109 (10)	0.0023 (9)
C20	0.0375 (12)	0.0427 (12)	0.0379 (11)	0.0098 (10)	0.0087 (9)	−0.0072 (9)
C21	0.0349 (11)	0.0460 (13)	0.0404 (12)	0.0034 (10)	0.0151 (10)	−0.0017 (9)
C22	0.0291 (10)	0.0350 (11)	0.0360 (11)	−0.0015 (8)	0.0105 (9)	0.0008 (8)
C23	0.0294 (11)	0.0324 (10)	0.0381 (11)	0.0004 (8)	0.0129 (9)	−0.0062 (8)
C24	0.0490 (14)	0.0411 (12)	0.0591 (15)	−0.0107 (11)	0.0273 (12)	−0.0083 (11)
C25	0.0617 (18)	0.0554 (17)	0.098 (3)	−0.0209 (14)	0.0467 (18)	−0.0279 (16)
C26	0.0333 (14)	0.080 (2)	0.099 (3)	−0.0082 (14)	0.0181 (15)	−0.0472 (19)
C27	0.0354 (14)	0.0767 (19)	0.0609 (17)	0.0126 (13)	−0.0005 (12)	−0.0243 (14)
C28	0.0369 (12)	0.0514 (14)	0.0396 (13)	0.0092 (10)	0.0108 (10)	−0.0048 (10)
C29	0.0582 (15)	0.0432 (13)	0.0377 (12)	0.0010 (11)	0.0255 (11)	−0.0006 (10)
C30	0.0808 (19)	0.0502 (14)	0.0477 (14)	−0.0105 (13)	0.0333 (14)	−0.0167 (11)
C31	0.0714 (18)	0.0594 (16)	0.0364 (13)	−0.0020 (14)	0.0079 (12)	−0.0033 (11)
C32	0.0417 (13)	0.0435 (13)	0.0359 (12)	0.0060 (10)	−0.0005 (10)	−0.0037 (9)

Geometric parameters (Å, º)

Cl1—O1	1.4283 (17)	C25—C26	1.380 (5)
Cl1—O2	1.422 (2)	C26—C27	1.375 (5)
Cl1—O3	1.432 (3)	C27—C28	1.381 (4)
Cl1—O4	1.418 (3)	C29—C30	1.503 (3)
Cl2—O5	1.427 (3)	C30—C31	1.524 (4)
Cl2—O6	1.4304 (16)	C31—C32	1.507 (4)
Cl2—O7	1.4385 (17)	N2—H19	0.76 (4)
Cl2—O8	1.424 (3)	N4—H38	0.79 (3)
S1—N1	1.6216 (18)	C2—H1	0.950
S1—N2	1.503 (2)	C3—H2	0.950
S1—C1	1.766 (2)	C4—H3	0.950
S1—C7	1.777 (3)	C5—H4	0.950
S2—N3	1.6236 (19)	C6—H5	0.950
S2—N4	1.502 (2)	C8—H6	0.950
S2—C17	1.767 (3)	C9—H7	0.950
S2—C23	1.778 (2)	C10—H8	0.950
N1—C13	1.499 (4)	C11—H9	0.950
N1—C16	1.506 (3)	C12—H10	0.950
N3—C29	1.495 (4)	C13—H11	0.990
N3—C32	1.510 (3)	C13—H12	0.990
C1—C2	1.387 (4)	C14—H13	0.990
C1—C6	1.384 (3)	C14—H14	0.990
C2—C3	1.383 (4)	C15—H15	0.990
C3—C4	1.375 (4)	C15—H16	0.990
C4—C5	1.371 (6)	C16—H17	0.990
C5—C6	1.374 (4)	C16—H18	0.990
C7—C8	1.386 (4)	C18—H20	0.950
C7—C12	1.391 (3)	C19—H21	0.950
C8—C9	1.389 (4)	C20—H22	0.950
C9—C10	1.391 (4)	C21—H23	0.950
C10—C11	1.386 (4)	C22—H24	0.950
C11—C12	1.382 (4)	C24—H25	0.950
C13—C14	1.516 (3)	C25—H26	0.950
C14—C15	1.506 (5)	C26—H27	0.950
C15—C16	1.506 (4)	C27—H28	0.950
C17—C18	1.390 (3)	C28—H29	0.950
C17—C22	1.380 (4)	C29—H30	0.990
C18—C19	1.386 (4)	C29—H31	0.990
C19—C20	1.384 (4)	C30—H32	0.990
C20—C21	1.377 (3)	C30—H33	0.990
C21—C22	1.384 (4)	C31—H34	0.990
C23—C24	1.383 (4)	C31—H35	0.990
C23—C28	1.387 (3)	C32—H36	0.990
C24—C25	1.392 (4)	C32—H37	0.990
O1—Cl1—O2	109.80 (11)	C3—C4—H3	119.715

O1—Cl1—O3	108.82 (12)	C5—C4—H3	119.711
O1—Cl1—O4	109.67 (14)	C4—C5—H4	119.717
O2—Cl1—O3	110.92 (17)	C6—C5—H4	119.703
O2—Cl1—O4	108.71 (15)	C1—C6—H5	120.907
O3—Cl1—O4	108.91 (18)	C5—C6—H5	120.905
O5—Cl2—O6	110.52 (12)	C7—C8—H6	120.776
O5—Cl2—O7	108.46 (13)	C9—C8—H6	120.779
O5—Cl2—O8	111.89 (14)	C8—C9—H7	119.853
O6—Cl2—O7	109.05 (10)	C10—C9—H7	119.868
O6—Cl2—O8	109.21 (13)	C9—C10—H8	119.996
O7—Cl2—O8	107.62 (13)	C11—C10—H8	119.983
N1—S1—N2	123.26 (11)	C10—C11—H9	119.605
N1—S1—C1	102.97 (11)	C12—C11—H9	119.606
N1—S1—C7	101.38 (9)	C7—C12—H10	120.855
N2—S1—C1	106.28 (11)	C11—C12—H10	120.865
N2—S1—C7	114.71 (13)	N1—C13—H11	111.310
C1—S1—C7	106.76 (10)	N1—C13—H12	111.306
N3—S2—N4	123.22 (10)	C14—C13—H11	111.321
N3—S2—C17	103.41 (9)	C14—C13—H12	111.316
N3—S2—C23	101.34 (11)	H11—C13—H12	109.199
N4—S2—C17	105.64 (12)	C13—C14—H13	110.935
N4—S2—C23	115.65 (10)	C13—C14—H14	110.930
C17—S2—C23	105.92 (10)	C15—C14—H13	110.942
S1—N1—C13	117.82 (13)	C15—C14—H14	110.930
S1—N1—C16	117.62 (17)	H13—C14—H14	108.947
C13—N1—C16	109.77 (16)	C14—C15—H15	110.755
S2—N3—C29	118.12 (13)	C14—C15—H16	110.747
S2—N3—C32	115.33 (17)	C16—C15—H15	110.760
C29—N3—C32	109.85 (17)	C16—C15—H16	110.748
S1—C1—C2	119.48 (15)	H15—C15—H16	108.838
S1—C1—C6	117.93 (19)	N1—C16—H17	110.914
C2—C1—C6	122.5 (2)	N1—C16—H18	110.917
C1—C2—C3	117.5 (2)	C15—C16—H17	110.915
C2—C3—C4	120.7 (3)	C15—C16—H18	110.925
C3—C4—C5	120.6 (3)	H17—C16—H18	108.947
C4—C5—C6	120.6 (3)	C17—C18—H20	120.936
C1—C6—C5	118.2 (3)	C19—C18—H20	120.940
S1—C7—C8	116.90 (15)	C18—C19—H21	119.803
S1—C7—C12	120.76 (18)	C20—C19—H21	119.799
C8—C7—C12	122.2 (2)	C19—C20—H22	119.944
C7—C8—C9	118.4 (2)	C21—C20—H22	119.940
C8—C9—C10	120.3 (3)	C20—C21—H23	119.571
C9—C10—C11	120.0 (3)	C22—C21—H23	119.571
C10—C11—C12	120.8 (2)	C17—C22—H24	120.910
C7—C12—C11	118.3 (3)	C21—C22—H24	120.918
N1—C13—C14	102.28 (19)	C23—C24—H25	121.334
C13—C14—C15	104.1 (3)	C25—C24—H25	121.334
C14—C15—C16	105.0 (2)	C24—C25—H26	119.756

N1—C16—C15	104.2 (2)	C26—C25—H26	119.751
S2—C17—C18	118.99 (19)	C25—C26—H27	119.399
S2—C17—C22	118.57 (14)	C27—C26—H27	119.403
C18—C17—C22	122.3 (2)	C26—C27—H28	120.233
C17—C18—C19	118.1 (3)	C28—C27—H28	120.236
C18—C19—C20	120.4 (2)	C23—C28—H29	120.572
C19—C20—C21	120.1 (3)	C27—C28—H29	120.574
C20—C21—C22	120.9 (3)	N3—C29—H30	110.938
C17—C22—C21	118.17 (19)	N3—C29—H31	110.938
S2—C23—C24	121.85 (15)	C30—C29—H30	110.950
S2—C23—C28	115.46 (18)	C30—C29—H31	110.951
C24—C23—C28	122.58 (19)	H30—C29—H31	108.967
C23—C24—C25	117.3 (3)	C29—C30—H32	110.984
C24—C25—C26	120.5 (3)	C29—C30—H33	110.979
C25—C26—C27	121.2 (3)	C31—C30—H32	110.982
C26—C27—C28	119.5 (3)	C31—C30—H33	110.986
C23—C28—C27	118.9 (3)	H32—C30—H33	108.994
N3—C29—C30	104.0 (2)	C30—C31—H34	111.013
C29—C30—C31	103.9 (2)	C30—C31—H35	111.012
C30—C31—C32	103.70 (19)	C32—C31—H34	111.020
N3—C32—C31	102.7 (3)	C32—C31—H35	111.031
S1—N2—H19	109 (3)	H34—C31—H35	109.004
S2—N4—H38	106 (3)	N3—C32—H36	111.219
C1—C2—H1	121.247	N3—C32—H37	111.222
C3—C2—H1	121.241	C31—C32—H36	111.218
C2—C3—H2	119.661	C31—C32—H37	111.218
C4—C3—H2	119.660	H36—C32—H37	109.134

N2—S1—N1—C13	60.74 (19)	S2—N3—C32—C31	−152.09 (12)
N2—S1—N1—C16	−74.31 (18)	C29—N3—C32—C31	−15.6 (2)
N1—S1—C1—C2	−37.16 (19)	C32—N3—C29—C30	−9.1 (2)
N1—S1—C1—C6	146.68 (16)	S1—C1—C2—C3	−175.87 (16)
C1—S1—N1—C13	−58.94 (14)	S1—C1—C6—C5	176.40 (17)
C1—S1—N1—C16	166.01 (12)	C2—C1—C6—C5	0.4 (4)
N1—S1—C7—C8	−150.09 (12)	C6—C1—C2—C3	0.1 (4)
N1—S1—C7—C12	34.47 (14)	C1—C2—C3—C4	−0.6 (5)
C7—S1—N1—C13	−169.32 (12)	C2—C3—C4—C5	0.6 (5)
C7—S1—N1—C16	55.63 (13)	C3—C4—C5—C6	−0.2 (6)
N2—S1—C1—C2	−167.98 (17)	C4—C5—C6—C1	−0.3 (5)
N2—S1—C1—C6	15.9 (2)	S1—C7—C8—C9	−176.01 (12)
N2—S1—C7—C8	−14.98 (16)	S1—C7—C12—C11	176.09 (11)
N2—S1—C7—C12	169.58 (12)	C8—C7—C12—C11	0.9 (3)
C1—S1—C7—C8	102.46 (14)	C12—C7—C8—C9	−0.6 (3)
C1—S1—C7—C12	−72.97 (14)	C7—C8—C9—C10	0.3 (3)
C7—S1—C1—C2	69.15 (19)	C8—C9—C10—C11	−0.2 (3)
C7—S1—C1—C6	−107.00 (16)	C9—C10—C11—C12	0.4 (3)
N4—S2—N3—C29	−75.95 (17)	C10—C11—C12—C7	−0.8 (3)
N4—S2—N3—C32	56.79 (17)	N1—C13—C14—C15	35.6 (3)

N3—S2—C17—C18	−37.29 (14)	C13—C14—C15—C16	−38.2 (3)
N3—S2—C17—C22	146.53 (12)	C14—C15—C16—N1	24.7 (3)
C17—S2—N3—C29	164.87 (11)	S2—C17—C18—C19	−176.20 (11)
C17—S2—N3—C32	−62.38 (13)	S2—C17—C22—C21	176.26 (10)
N3—S2—C23—C24	35.34 (18)	C18—C17—C22—C21	0.2 (3)
N3—S2—C23—C28	−148.21 (14)	C22—C17—C18—C19	−0.2 (3)
C23—S2—N3—C29	55.27 (13)	C17—C18—C19—C20	−0.3 (3)
C23—S2—N3—C32	−171.98 (11)	C18—C19—C20—C21	0.8 (3)
N4—S2—C17—C18	−167.96 (11)	C19—C20—C21—C22	−0.8 (3)
N4—S2—C17—C22	15.86 (14)	C20—C21—C22—C17	0.3 (3)
N4—S2—C23—C24	171.07 (16)	S2—C23—C24—C25	176.69 (16)
N4—S2—C23—C28	−12.5 (2)	S2—C23—C28—C27	−176.52 (16)
C17—S2—C23—C24	−72.31 (18)	C24—C23—C28—C27	−0.1 (4)
C17—S2—C23—C28	104.14 (15)	C28—C23—C24—C25	0.5 (4)
C23—S2—C17—C18	68.86 (14)	C23—C24—C25—C26	−0.9 (5)
C23—S2—C17—C22	−107.32 (13)	C24—C25—C26—C27	1.0 (6)
S1—N1—C13—C14	−158.92 (12)	C25—C26—C27—C28	−0.6 (6)
S1—N1—C16—C15	136.13 (13)	C26—C27—C28—C23	0.1 (5)
C13—N1—C16—C15	−2.3 (2)	N3—C29—C30—C31	30.1 (3)
C16—N1—C13—C14	−20.6 (2)	C29—C30—C31—C32	−40.5 (3)
S2—N3—C29—C30	126.00 (13)	C30—C31—C32—N3	33.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H19···O2^{i}	0.76 (4)	2.34 (4)	3.057 (4)	159 (4)
N4—H38···O8^{ii}	0.79 (3)	2.16 (3)	2.952 (4)	173 (3)
C2—H1···O1	0.95	2.58	3.531 (3)	178
C10—H8···O7^{iii}	0.95	2.56	3.453 (4)	157
C12—H10···O1	0.95	2.43	3.379 (4)	174
C16—H18···O4^{i}	0.99	2.58	3.392 (4)	139
C18—H20···O6	0.95	2.47	3.362 (3)	156
C25—H26···O3^{iv}	0.95	2.57	3.512 (5)	171
C29—H30···O5^{ii}	0.99	2.50	3.344 (3)	142
C28—H29···N4^{v}	0.95	2.58	3.401 (4)	145

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