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Crystal structure of penoxsulam

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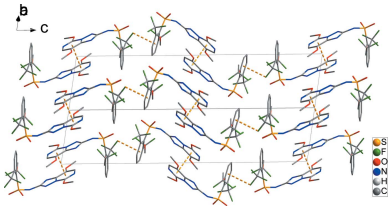
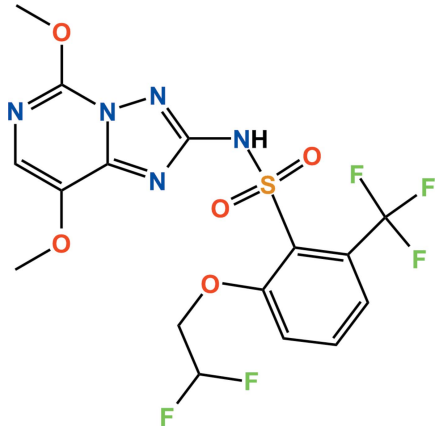
The title compound, $C_{16}H_{14}F_5N_5O_5S$ [systematic name: 2-(2,2-difluoroethoxy)-*N*-(5,8-dimethoxy-1,2,4-triazolo[1,5-*c*]pyrimidin-2-yl)-6-(trifluoromethyl)-benzenesulfonamide], is used as a herbicide. The asymmetric unit of this structure comprises two independent molecules, *A* and *B*. The dihedral angles between the ring planes of the triazolopyrimidine ring systems and the benzene rings are 68.84 (7)° for *A* and 68.05 (6)° for *B*. In the crystal, weak intermolecular π–π interactions, with centroid–centroid separations of 3.4456 (17) and 3.5289 (15) Å and C—F$\cdots\pi$ [3.5335 (17) Å and 107.92 (13)°] contacts link adjacent molecules into chains along [001]. C—H$\cdots$O and C—H$\cdots$F hydrogen bonds link type *B* molecules into chains parallel to (100). Additional C—H$\cdots$F hydrogen bonds together with short F$\cdots$F contacts further aggregate the structure into a three-dimensional network.

1. Chemical context

Penxosulam is a triazolopyrimidine sulfonamide herbicide, which is used to control the growth of annual grasses, sedges, and broadleaf weeds in rice agriculture. The compound inhibits the synthesis of acetolactate and targets the biosynthesis of branch-chained amino acids, a metabolic pathway found in plants, fungi, and microorganisms. Acetolactate synthase (ALS) inhibitors are present in most effective herbicides. They are used in agriculture because they show a broad weed control spectrum, crop selectivity, are safe to humans, and can be applied at relatively low usage rates (Jabusch *et al.*, 2005; Yasuor *et al.*, 2009). Moreover, penoxsulam controls a number of troublesome weeds including northern jointvetch, alligatorweed, Texasweed/Mexicanweed, annual sedge, ducksalad, smartweed, and hemp sesbania (Willingham *et al.*, 2008). We now report here the crystal structure of penoxsulam, 2-(2,2-difluoroethoxy)-*N*-(5,8-dimethoxy[1,2,4]triazolo[1,5-*c*]-pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide.





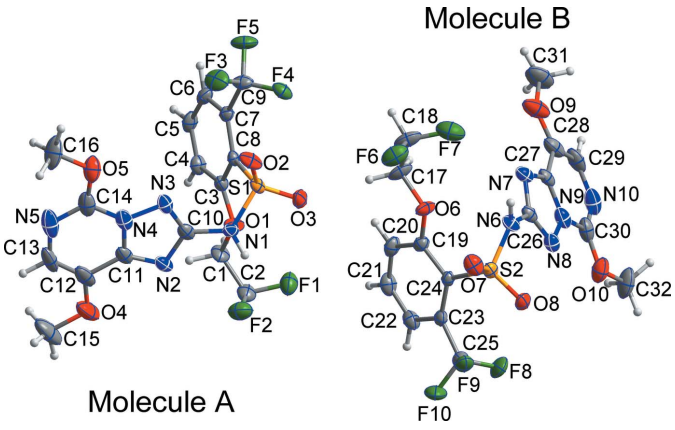


Figure 1
The molecular structures of the title compound with the atom labelling and displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radius.

2. Structural commentary

The penoxsulam molecule crystallizes with two independent molecules, *A* and *B* in the asymmetric unit, Fig. 1. The triazolopyrimidin unit caries methoxy substituents while the benzene ring of the benzenesulfonamide segment of the molecule carries trifluoro methyl and the unusual difluoroethoxy substituents. The dihedral angles between the planes of the triazolopyrimidine ring systems and the benzene ring planes are 68.84 (7)° in molecule *A* and 68.05 (6)° in *B*. All bond lengths and bond angles are normal and comparable to those observed in similar crystal structures for triazolopyrimidine (AboulWafa *et al.*, 2014) and triazolopyrimidine sulfonamide herbicides (Kumar *et al.*, 2012).

3. Supramolecular features

In the crystal, there are weak π–π interactions between the pyrimidine rings of neighbouring molecules of type *A* with $Cg1\cdots Cg1^{v}$ = 3.4456 (17), and type *B* with $Cg2\cdots Cg2^{vi}$ = 3.5289 (15) Å [*Cg*1 and *Cg*2 are the centroids of the N4/N5/C11–C14 and N9/N10/C28–C30 rings, respectively; symmetry codes: (v) $-x, -y, -z+1$; (vi) $-x+2, -y, -z$]. These combine with C25—F9$\cdots Cg3^{vii}$ interactions involving the C3–C8 benzene ring to form chains along [001] (Fig. 2). C17—H17*B*$\cdots$O8^{i} and C20—H20$\cdots$F9^{i} hydrogen bonds form chains along the *a*-axis direction, forming a two-dimensional network in the *ab* plane (Fig. 3 and Table 1). In addition, short intermolecular F1$\cdots$F5^{iii} and F6$\cdots$F10^{iii} contacts [2.846 (2) and 2.794 (2) Å] together with C1—H1*A*$\cdots$F10^{i}, C16—H16*C*$\cdots$F3^{ii}, C18—H18$\cdots$F10^{iii} and C32—H32*C*$\cdots$F8^{iv} hydrogen bonds generate a three-dimensional network with molecules stacked along the *a*-axis direction (Fig. 4) and Table 1).

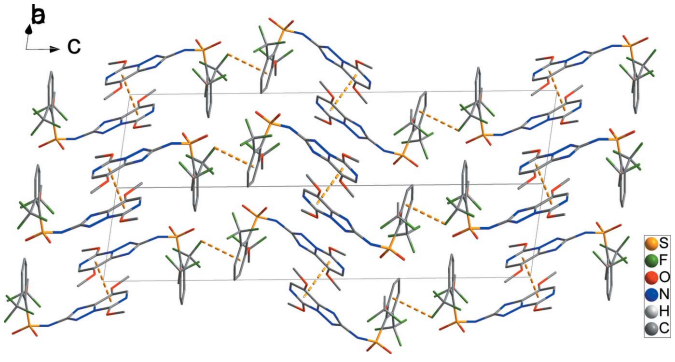


Figure 2
C—F$\cdots\pi$ and weak π–π interactions, yellow dashed lines, form chain along [001] in the crystal packing. H atoms have been omitted for clarity.

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C1—H1*A*$\cdots$F10^{i}	0.99	2.48	3.293 (3)	139
C16—H16*C*$\cdots$F3^{ii}	0.98	2.40	3.185 (3)	136
C17—H17*B*$\cdots$O8^{i}	0.99	2.40	3.102 (3)	127
C18—H18$\cdots$F10^{iii}	1.00	2.60	3.104 (3)	111
C20—H20$\cdots$F9^{i}	0.95	2.55	3.466 (3)	162
C32—H32*C*$\cdots$F8^{iv}	0.98	2.36	3.146 (3)	137

Symmetry codes: (i) $x-1, y, z$; (ii) $-x, -y+1, -z+1$; (iii) $x-1, y+1, z$; (iv) $-x+3, -y, -z$.

4. Database survey

Crystal structures of sulfonamide (Kang *et al.*, 2015; Chen, Wu *et al.*, 2005) and triazolopyrimidine (Chen, Li *et al.*, 2005) herbicides have been reported previously. Moreover, the crystal structures of compounds with a triazolopyrimidine ring system and a benzene ring in the molecule, ethyl 2-(5,7-

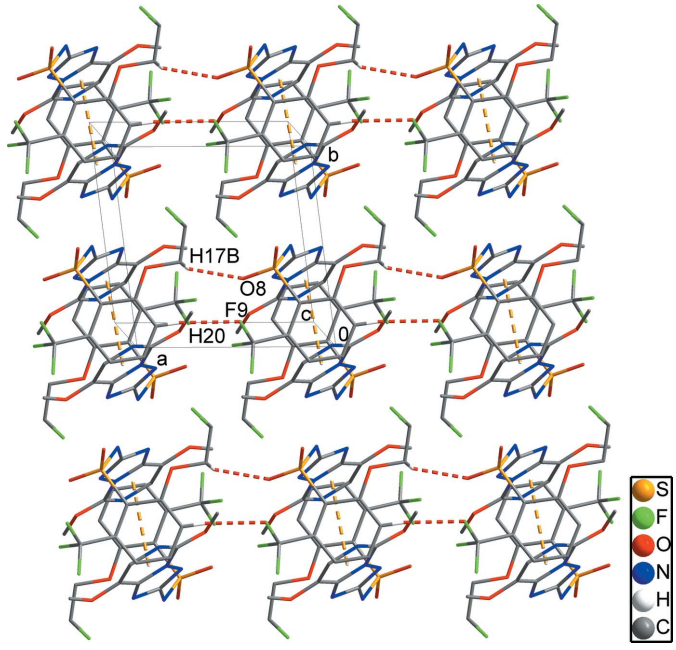


Figure 3
A two-dimensional network in the *ab* plane. Yellow dashed lines indicate weak intermolecular π–π interactions. Red dashed lines indicate C—F$\cdots\pi$ interactions and C—H$\cdots$O hydrogen bonds. H atoms have been omitted for clarity.

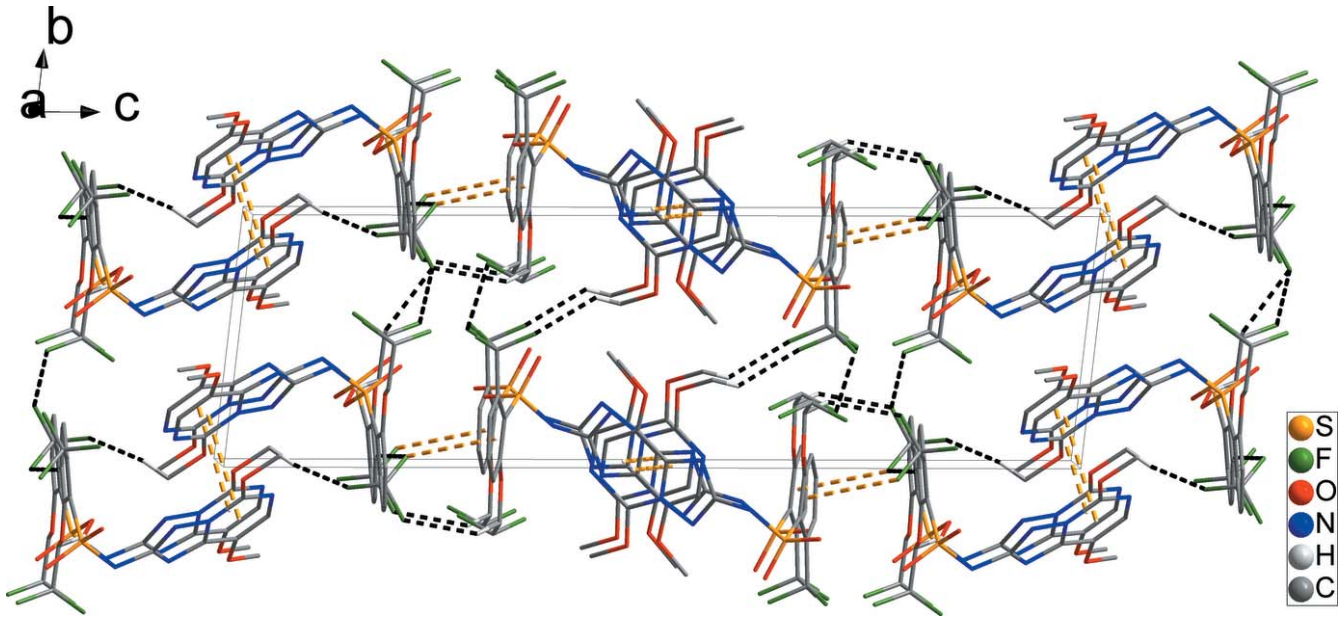


Figure 4
Overall packing showing the three-dimensional network viewed along the *a*-axis direction, C—H···F hydrogen bonds and F···F intermolecular short contacts are shown as black dashed lines. H atoms have been omitted for clarity.

dimethyl-1,2,4-triazolo[1,5-*a*]-pyrimidin-2-yloxy)benzoate (Chen, Li *et al.*, 2005) and 5-(4-Chlorophenoxy)-6-isopropyl-3-phenyl-3*H*-1,2,3-triazolo[4,5-*d*]-pyrimidin-7(6*H*)-one (Zeng *et al.*, 2009) have also been reported.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{14}F_5N_5O_5S$
M_r	483.38
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	173
a, b, c (Å)	8.1945 (3), 8.3733 (3), 28.3277 (9)
α, β, γ (°)	82.698 (2), 84.183 (2), 81.814 (2)
V (Å^3)	1901.43 (12)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.26
Crystal size (mm)	0.40 × 0.19 × 0.11
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.646, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24336, 6571, 5503
R_{int}	0.032
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.044, 0.106, 1.05
No. of reflections	6571
No. of parameters	581
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.52

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg, 2010) and *publCIF* (Westrip, 2010).

5. Synthesis and crystallization

The title compound was purchased from the Dr. Ehrenstorfer GmbH Company. Single crystals were obtained by slow evaporation of an acetonitrile solution.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were positioned geometrically and refined using a riding model with d(N—H) = 0.88 Å, U_{iso}(H) = 1.2U_{eq}(C) for the N—H group, d(C—H) = 0.95 Å, U_{iso}(H) = 1.2U_{eq}(C) for aromatic C—H, d(C—H) = 0.98 Å, U_{iso}(H) = 1.5U_{eq}(C) for methyl group, d(C—H) = 0.99 Å, U_{iso}(H) = 1.2U_{eq}(C) for Csp^3—H, and d(C—H) = 1.00 Å, U_{iso}(H) = 1.5U_{eq}(C) for Csp^3—H.

Funding information

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Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *publCIF* (Westrip, 2010).

2-(2,2-Difluoroethoxy)-*N*-(5,8-dimethoxy-1,2,4-triazolo[1,5-*c*]pyrimidin-2-yl)-6-(trifluoromethyl)benzenesulfonamide

Crystal data

$C_{16}H_{14}F_5N_5O_5S$
$M_r = 483.38$
Triclinic, $P\overline{1}$
$a = 8.1945$ (3) Å
$b = 8.3733$ (3) Å
$c = 28.3277$ (9) Å
$\alpha = 82.698$ (2)°
$\beta = 84.183$ (2)°
$\gamma = 81.814$ (2)°
$V = 1901.43$ (12) Å^3
$Z = 4$
$F(000) = 984$
$D_x = 1.689$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9990 reflections
$\theta = 2.5$–$27.3°$
$\mu = 0.26$ mm^{-1}
$T = 173$ K
Plate, colourless
0.40 × 0.19 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
$T_{min} = 0.646$, $T_{max} = 0.746$
24336 measured reflections
6571 independent reflections
5503 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.5°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -32 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.106$
$S = 1.05$
6571 reflections
581 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 1.5689P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.47$ 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}
S1	0.40491 (7)	0.27322 (7)	0.33986 (2)	0.02327 (15)
S2	1.17808 (7)	0.29094 (7)	0.15763 (2)	0.02404 (15)
F1	0.6784 (2)	−0.21313 (19)	0.28932 (6)	0.0473 (4)
F2	0.6798 (2)	−0.2261 (2)	0.36604 (6)	0.0526 (5)
F3	0.0164 (2)	0.53003 (19)	0.34514 (6)	0.0477 (4)
F4	0.18482 (19)	0.53143 (16)	0.28142 (5)	0.0326 (3)
F5	−0.0713 (2)	0.51888 (19)	0.27738 (6)	0.0465 (4)
F6	0.6485 (2)	0.5512 (2)	0.21727 (6)	0.0501 (4)
F7	0.6787 (3)	0.5868 (2)	0.13991 (7)	0.0629 (5)
F8	1.4342 (2)	−0.0947 (2)	0.15363 (6)	0.0505 (5)
F9	1.39010 (18)	0.03048 (19)	0.21686 (5)	0.0381 (4)
F10	1.3647 (2)	−0.22070 (19)	0.22130 (6)	0.0523 (5)
O1	0.4016 (2)	−0.06137 (18)	0.33033 (6)	0.0292 (4)
O2	0.3455 (2)	0.42426 (19)	0.35764 (6)	0.0343 (4)
O3	0.5333 (2)	0.2632 (2)	0.30221 (6)	0.0356 (4)
O4	0.3555 (3)	−0.3031 (3)	0.52168 (8)	0.0591 (6)
O5	0.0298 (3)	0.3079 (3)	0.51945 (7)	0.0460 (5)
O6	0.8254 (2)	0.2966 (2)	0.17644 (6)	0.0309 (4)
O7	1.1568 (2)	0.3944 (2)	0.19444 (6)	0.0349 (4)
O8	1.3393 (2)	0.2352 (2)	0.13777 (6)	0.0334 (4)
O9	0.7015 (3)	0.3776 (3)	−0.02223 (8)	0.0535 (6)
O10	1.3171 (3)	0.0395 (3)	−0.02043 (7)	0.0449 (5)
N1	0.4795 (2)	0.1502 (2)	0.38497 (7)	0.0250 (4)
H1N	0.5842	0.1083	0.3822	0.030*
N2	0.4113 (3)	−0.0440 (2)	0.44874 (8)	0.0315 (5)
N3	0.2718 (3)	0.2139 (2)	0.44832 (7)	0.0293 (5)
N4	0.2201 (3)	0.1158 (3)	0.48799 (7)	0.0360 (5)
N5	0.0657 (4)	0.0425 (4)	0.55905 (8)	0.0537 (7)
N6	1.0775 (3)	0.3946 (2)	0.11402 (7)	0.0273 (5)
H6N	1.0315	0.4945	0.1175	0.033*
N7	0.9191 (3)	0.3767 (2)	0.05042 (7)	0.0313 (5)
N8	1.1790 (3)	0.2324 (3)	0.05044 (7)	0.0303 (5)
N9	1.1034 (3)	0.2116 (3)	0.01107 (7)	0.0354 (5)
N10	1.0719 (3)	0.1088 (3)	−0.05969 (8)	0.0483 (6)
C1	0.4275 (3)	−0.2344 (3)	0.33482 (10)	0.0319 (6)
H1A	0.3768	−0.2749	0.3091	0.038*
H1B	0.3776	−0.2797	0.3661	0.038*
C2	0.6090 (4)	−0.2821 (3)	0.33091 (10)	0.0366 (7)
H2	0.6349	−0.4030	0.3329	0.044*

C3	0.2487 (3)	0.0195 (3)	0.32428 (8)	0.0226 (5)
C4	0.1131 (3)	−0.0560 (3)	0.31745 (9)	0.0283 (6)
H4	0.1223	−0.1710	0.3198	0.034*
C5	−0.0334 (3)	0.0372 (3)	0.30731 (9)	0.0319 (6)
H5	−0.1263	−0.0142	0.3032	0.038*
C6	−0.0480 (3)	0.2044 (3)	0.30301 (9)	0.0291 (6)
H6	−0.1488	0.2671	0.2944	0.035*
C7	0.0837 (3)	0.2816 (3)	0.31124 (8)	0.0219 (5)
C8	0.2322 (3)	0.1891 (3)	0.32403 (7)	0.0188 (5)
C9	0.0556 (3)	0.4652 (3)	0.30368 (9)	0.0303 (6)
C10	0.3842 (3)	0.1093 (3)	0.42737 (8)	0.0264 (5)
C11	0.3046 (3)	−0.0355 (3)	0.48755 (9)	0.0303 (6)
C12	0.2670 (4)	−0.1556 (4)	0.52547 (11)	0.0451 (8)
C13	0.1508 (4)	−0.1123 (4)	0.55917 (10)	0.0472 (8)
H13	0.1246	−0.1919	0.5848	0.057*
C14	0.1007 (4)	0.1546 (4)	0.52415 (9)	0.0400 (7)
C15	0.3152 (5)	−0.4323 (4)	0.55754 (13)	0.0761 (13)
H15A	0.3362	−0.4064	0.5890	0.114*
H15B	0.3837	−0.5338	0.5506	0.114*
H15C	0.1981	−0.4448	0.5575	0.114*
C16	−0.0932 (4)	0.3529 (4)	0.55773 (11)	0.0618 (10)
H16A	−0.1876	0.2927	0.5580	0.093*
H16B	−0.1309	0.4697	0.5526	0.093*
H16C	−0.0443	0.3266	0.5884	0.093*
C17	0.6501 (3)	0.3241 (3)	0.17727 (11)	0.0388 (7)
H17A	0.5996	0.2640	0.2062	0.047*
H17B	0.6132	0.2872	0.1487	0.047*
C18	0.6008 (4)	0.5018 (4)	0.17763 (12)	0.0460 (8)
H18	0.4782	0.5284	0.1767	0.055*
C19	0.8998 (3)	0.1400 (3)	0.17952 (8)	0.0244 (5)
C20	0.8126 (3)	0.0072 (3)	0.18630 (9)	0.0326 (6)
H20	0.6953	0.0231	0.1871	0.039*
C21	0.8963 (3)	−0.1464 (3)	0.19184 (9)	0.0350 (6)
H21	0.8369	−0.2372	0.1948	0.042*
C22	1.0651 (3)	−0.1711 (3)	0.19321 (9)	0.0321 (6)
H22	1.1208	−0.2783	0.1991	0.039*
C23	1.1547 (3)	−0.0404 (3)	0.18604 (8)	0.0238 (5)
C24	1.0729 (3)	0.1177 (3)	0.17622 (8)	0.0203 (5)
C25	1.3353 (3)	−0.0779 (3)	0.19369 (9)	0.0344 (6)
C26	1.0613 (3)	0.3314 (3)	0.07141 (8)	0.0257 (5)
C27	0.9510 (3)	0.2979 (3)	0.01135 (8)	0.0275 (6)
C28	0.8526 (4)	0.2892 (3)	−0.02617 (10)	0.0407 (7)
C29	0.9154 (4)	0.1956 (4)	−0.05974 (9)	0.0422 (7)
H29	0.8498	0.1880	−0.0850	0.051*
C30	1.1653 (4)	0.1154 (3)	−0.02503 (9)	0.0369 (7)
C31	0.5908 (5)	0.3640 (5)	−0.05730 (12)	0.0651 (10)
H31A	0.6383	0.4036	−0.0891	0.098*
H31B	0.4841	0.4291	−0.0501	0.098*

H31C	0.5747	0.2500	−0.0567	0.098*
C32	1.3861 (4)	−0.0568 (4)	−0.05853 (11)	0.0574 (9)
H32A	1.3238	−0.1487	−0.0580	0.086*
H32B	1.5023	−0.0976	−0.0540	0.086*
H32C	1.3789	0.0106	−0.0894	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0248 (3)	0.0191 (3)	0.0273 (3)	−0.0096 (2)	−0.0074 (2)	0.0035 (2)
S2	0.0227 (3)	0.0273 (3)	0.0243 (3)	−0.0104 (3)	−0.0056 (2)	−0.0001 (2)
F1	0.0495 (11)	0.0363 (9)	0.0517 (10)	−0.0063 (8)	0.0157 (8)	−0.0038 (8)
F2	0.0434 (11)	0.0549 (11)	0.0572 (11)	0.0058 (8)	−0.0152 (9)	−0.0032 (9)
F3	0.0608 (12)	0.0387 (9)	0.0427 (9)	0.0094 (8)	−0.0013 (8)	−0.0212 (7)
F4	0.0448 (9)	0.0202 (7)	0.0334 (8)	−0.0065 (6)	−0.0109 (7)	0.0034 (6)
F5	0.0383 (10)	0.0389 (9)	0.0598 (11)	0.0152 (7)	−0.0215 (8)	−0.0052 (8)
F6	0.0374 (10)	0.0509 (10)	0.0675 (12)	−0.0021 (8)	−0.0103 (8)	−0.0271 (9)
F7	0.0708 (14)	0.0459 (10)	0.0689 (13)	0.0095 (10)	−0.0230 (11)	0.0004 (9)
F8	0.0353 (10)	0.0617 (11)	0.0460 (10)	0.0086 (8)	0.0167 (8)	−0.0063 (8)
F9	0.0239 (8)	0.0547 (10)	0.0349 (8)	−0.0035 (7)	−0.0082 (6)	0.0007 (7)
F10	0.0421 (11)	0.0414 (9)	0.0616 (11)	0.0160 (8)	−0.0023 (8)	0.0140 (8)
O1	0.0291 (10)	0.0136 (8)	0.0455 (10)	−0.0016 (7)	−0.0089 (8)	−0.0020 (7)
O2	0.0486 (12)	0.0161 (8)	0.0417 (10)	−0.0081 (8)	−0.0193 (9)	−0.0008 (7)
O3	0.0253 (10)	0.0465 (11)	0.0347 (10)	−0.0149 (8)	−0.0016 (8)	0.0073 (8)
O4	0.0559 (15)	0.0501 (14)	0.0696 (15)	−0.0188 (12)	−0.0251 (12)	0.0290 (11)
O5	0.0544 (14)	0.0528 (13)	0.0349 (11)	−0.0212 (11)	0.0097 (9)	−0.0158 (9)
O6	0.0160 (9)	0.0283 (9)	0.0490 (11)	−0.0019 (7)	−0.0057 (8)	−0.0056 (8)
O7	0.0458 (12)	0.0342 (10)	0.0298 (10)	−0.0159 (9)	−0.0095 (8)	−0.0062 (8)
O8	0.0191 (9)	0.0468 (11)	0.0346 (10)	−0.0121 (8)	−0.0033 (7)	0.0041 (8)
O9	0.0629 (16)	0.0460 (12)	0.0560 (13)	−0.0108 (11)	−0.0342 (11)	0.0043 (10)
O10	0.0409 (13)	0.0634 (13)	0.0343 (11)	−0.0178 (11)	0.0065 (9)	−0.0158 (10)
N1	0.0177 (11)	0.0273 (11)	0.0302 (11)	−0.0045 (8)	−0.0094 (8)	0.0041 (9)
N2	0.0320 (13)	0.0284 (11)	0.0357 (12)	−0.0117 (10)	−0.0146 (10)	0.0086 (9)
N3	0.0346 (13)	0.0310 (11)	0.0249 (11)	−0.0144 (10)	−0.0046 (9)	−0.0004 (9)
N4	0.0387 (14)	0.0455 (14)	0.0273 (12)	−0.0188 (11)	−0.0086 (10)	0.0014 (10)
N5	0.0623 (19)	0.079 (2)	0.0285 (13)	−0.0423 (16)	−0.0065 (12)	0.0003 (13)
N6	0.0355 (13)	0.0201 (10)	0.0276 (11)	−0.0067 (9)	−0.0086 (9)	0.0003 (8)
N7	0.0346 (13)	0.0272 (11)	0.0332 (12)	−0.0091 (10)	−0.0120 (10)	0.0055 (9)
N8	0.0332 (13)	0.0358 (12)	0.0242 (11)	−0.0126 (10)	−0.0043 (9)	−0.0018 (9)
N9	0.0446 (15)	0.0384 (13)	0.0262 (12)	−0.0190 (11)	−0.0053 (10)	0.0026 (10)
N10	0.0610 (15)	0.0614 (17)	0.0280 (12)	−0.0318 (12)	−0.0021 (12)	−0.0012 (11)
C1	0.0404 (16)	0.0120 (11)	0.0417 (15)	−0.0024 (11)	−0.0001 (12)	−0.0003 (10)
C2	0.0409 (17)	0.0201 (13)	0.0451 (17)	0.0000 (12)	0.0027 (13)	0.0004 (12)
C3	0.0243 (14)	0.0218 (12)	0.0212 (12)	−0.0040 (10)	−0.0013 (10)	0.0005 (9)
C4	0.0325 (15)	0.0232 (12)	0.0317 (14)	−0.0128 (11)	−0.0013 (11)	−0.0039 (10)
C5	0.0253 (14)	0.0408 (15)	0.0337 (14)	−0.0175 (12)	0.0000 (11)	−0.0069 (12)
C6	0.0192 (13)	0.0387 (15)	0.0300 (13)	−0.0029 (11)	−0.0016 (10)	−0.0075 (11)
C7	0.0218 (13)	0.0239 (12)	0.0199 (11)	−0.0014 (10)	−0.0008 (9)	−0.0049 (9)

C8	0.0214 (13)	0.0177 (11)	0.0180 (11)	−0.0064 (9)	−0.0005 (9)	−0.0011 (9)
C9	0.0319 (15)	0.0264 (13)	0.0319 (14)	0.0070 (11)	−0.0075 (11)	−0.0090 (11)
C10	0.0281 (14)	0.0276 (13)	0.0268 (13)	−0.0126 (11)	−0.0119 (11)	0.0021 (10)
C11	0.0300 (15)	0.0359 (15)	0.0285 (14)	−0.0172 (12)	−0.0143 (11)	0.0068 (11)
C12	0.0468 (19)	0.0523 (19)	0.0403 (17)	−0.0251 (16)	−0.0251 (15)	0.0175 (14)
C13	0.056 (2)	0.065 (2)	0.0266 (15)	−0.0373 (18)	−0.0174 (14)	0.0175 (14)
C14	0.0433 (18)	0.059 (2)	0.0240 (14)	−0.0256 (15)	−0.0038 (12)	−0.0066 (13)
C15	0.099 (3)	0.062 (2)	0.072 (2)	−0.043 (2)	−0.039 (2)	0.0379 (19)
C16	0.071 (2)	0.078 (2)	0.0443 (19)	−0.036 (2)	0.0250 (17)	−0.0330 (18)
C17	0.0167 (14)	0.0439 (16)	0.0591 (18)	−0.0001 (12)	−0.0096 (12)	−0.0178 (14)
C18	0.0244 (16)	0.0511 (18)	0.067 (2)	0.0050 (14)	−0.0176 (14)	−0.0227 (16)
C19	0.0220 (13)	0.0267 (13)	0.0252 (12)	−0.0033 (10)	−0.0033 (10)	−0.0045 (10)
C20	0.0242 (14)	0.0402 (15)	0.0362 (15)	−0.0136 (12)	−0.0006 (11)	−0.0065 (12)
C21	0.0431 (18)	0.0301 (14)	0.0355 (15)	−0.0191 (13)	0.0012 (12)	−0.0059 (11)
C22	0.0437 (17)	0.0238 (13)	0.0280 (13)	−0.0046 (12)	0.0018 (12)	−0.0036 (10)
C23	0.0251 (13)	0.0263 (13)	0.0185 (11)	−0.0025 (10)	0.0038 (10)	−0.0020 (10)
C24	0.0182 (12)	0.0238 (12)	0.0195 (11)	−0.0059 (10)	−0.0009 (9)	−0.0022 (9)
C25	0.0301 (15)	0.0367 (15)	0.0312 (14)	0.0040 (12)	0.0026 (12)	0.0019 (12)
C26	0.0300 (14)	0.0230 (12)	0.0254 (13)	−0.0114 (11)	−0.0061 (11)	0.0043 (10)
C27	0.0373 (16)	0.0241 (13)	0.0230 (13)	−0.0144 (11)	−0.0087 (11)	0.0072 (10)
C28	0.053 (2)	0.0354 (15)	0.0367 (16)	−0.0185 (14)	−0.0211 (14)	0.0123 (13)
C29	0.0613 (17)	0.0504 (18)	0.0212 (13)	−0.0282 (13)	−0.0155 (13)	0.0046 (12)
C30	0.0489 (19)	0.0424 (16)	0.0233 (14)	−0.0242 (14)	0.0030 (12)	−0.0027 (12)
C31	0.066 (3)	0.073 (2)	0.062 (2)	−0.022 (2)	−0.0397 (19)	0.0113 (18)
C32	0.056 (2)	0.081 (2)	0.0413 (18)	−0.0272 (19)	0.0199 (16)	−0.0260 (17)

Geometric parameters (Å, º)

S1—O3	1.4217 (18)	N10—C30	1.316 (4)
S1—O2	1.4238 (18)	N10—C29	1.381 (4)
S1—N1	1.6466 (19)	C1—C2	1.481 (4)
S1—C8	1.787 (2)	C1—H1A	0.9900
S2—O7	1.4207 (18)	C1—H1B	0.9900
S2—O8	1.4210 (18)	C2—H2	1.0000
S2—N6	1.640 (2)	C3—C4	1.395 (3)
S2—C24	1.788 (2)	C3—C8	1.406 (3)
F1—C2	1.353 (3)	C4—C5	1.371 (4)
F2—C2	1.364 (3)	C4—H4	0.9500
F3—C9	1.346 (3)	C5—C6	1.379 (4)
F4—C9	1.330 (3)	C5—H5	0.9500
F5—C9	1.333 (3)	C6—C7	1.388 (3)
F6—C18	1.355 (3)	C6—H6	0.9500
F7—C18	1.361 (4)	C7—C8	1.401 (3)
F8—C25	1.336 (3)	C7—C9	1.513 (3)
F9—C25	1.335 (3)	C11—C12	1.417 (4)
F10—C25	1.348 (3)	C12—C13	1.324 (5)
O1—C3	1.353 (3)	C13—H13	0.9500
O1—C1	1.425 (3)	C15—H15A	0.9800

O4—C12	1.351 (4)	C15—H15B	0.9800
O4—C15	1.438 (4)	C15—H15C	0.9800
O5—C14	1.328 (4)	C16—H16A	0.9800
O5—C16	1.452 (3)	C16—H16B	0.9800
O6—C19	1.361 (3)	C16—H16C	0.9800
O6—C17	1.420 (3)	C17—C18	1.487 (4)
O9—C28	1.351 (4)	C17—H17A	0.9900
O9—C31	1.436 (4)	C17—H17B	0.9900
O10—C30	1.324 (4)	C18—H18	1.0000
O10—C32	1.450 (3)	C19—C20	1.389 (3)
N1—C10	1.397 (3)	C19—C24	1.399 (3)
N1—H1N	0.8800	C20—C21	1.367 (4)
N2—C11	1.336 (3)	C20—H20	0.9500
N2—C10	1.346 (3)	C21—C22	1.373 (4)
N3—C10	1.326 (3)	C21—H21	0.9500
N3—N4	1.373 (3)	C22—C23	1.386 (3)
N4—C11	1.355 (3)	C22—H22	0.9500
N4—C14	1.380 (4)	C23—C24	1.403 (3)
N5—C14	1.313 (4)	C23—C25	1.501 (4)
N5—C13	1.382 (4)	C27—C28	1.413 (4)
N6—C26	1.403 (3)	C28—C29	1.326 (4)
N6—H6N	0.8800	C29—H29	0.9500
N7—C27	1.344 (3)	C31—H31A	0.9800
N7—C26	1.346 (3)	C31—H31B	0.9800
N8—C26	1.323 (3)	C31—H31C	0.9800
N8—N9	1.370 (3)	C32—H32A	0.9800
N9—C27	1.350 (3)	C32—H32B	0.9800
N9—C30	1.393 (3)	C32—H32C	0.9800

O3—S1—O2	120.60 (11)	C12—C13—N5	123.4 (3)
O3—S1—N1	104.84 (11)	C12—C13—H13	118.3
O2—S1—N1	106.54 (10)	N5—C13—H13	118.3
O3—S1—C8	109.19 (10)	N5—C14—O5	125.9 (3)
O2—S1—C8	108.35 (11)	N5—C14—N4	119.6 (3)
N1—S1—C8	106.43 (10)	O5—C14—N4	114.5 (2)
O7—S2—O8	120.38 (11)	O4—C15—H15A	109.5
O7—S2—N6	104.97 (11)	O4—C15—H15B	109.5
O8—S2—N6	107.16 (11)	H15A—C15—H15B	109.5
O7—S2—C24	109.88 (10)	O4—C15—H15C	109.5
O8—S2—C24	107.92 (11)	H15A—C15—H15C	109.5
N6—S2—C24	105.54 (10)	H15B—C15—H15C	109.5
C3—O1—C1	120.10 (19)	O5—C16—H16A	109.5
C12—O4—C15	116.5 (3)	O5—C16—H16B	109.5
C14—O5—C16	115.5 (2)	H16A—C16—H16B	109.5
C19—O6—C17	117.77 (19)	O5—C16—H16C	109.5
C28—O9—C31	116.7 (3)	H16A—C16—H16C	109.5
C30—O10—C32	115.5 (2)	H16B—C16—H16C	109.5
C10—N1—S1	123.11 (17)	O6—C17—C18	106.7 (2)

C10—N1—H1N	118.4	O6—C17—H17A	110.4
S1—N1—H1N	118.4	C18—C17—H17A	110.4
C11—N2—C10	100.9 (2)	O6—C17—H17B	110.4
C10—N3—N4	100.3 (2)	C18—C17—H17B	110.4
C11—N4—N3	109.7 (2)	H17A—C17—H17B	108.6
C11—N4—C14	121.8 (2)	F6—C18—F7	105.9 (2)
N3—N4—C14	128.4 (2)	F6—C18—C17	110.2 (3)
C14—N5—C13	119.3 (3)	F7—C18—C17	111.1 (2)
C26—N6—S2	123.03 (17)	F6—C18—H18	109.9
C26—N6—H6N	118.5	F7—C18—H18	109.9
S2—N6—H6N	118.5	C17—C18—H18	109.9
C27—N7—C26	100.8 (2)	O6—C19—C20	123.2 (2)
C26—N8—N9	100.1 (2)	O6—C19—C24	116.2 (2)
C27—N9—N8	110.4 (2)	C20—C19—C24	120.5 (2)
C27—N9—C30	121.9 (2)	C21—C20—C19	119.7 (2)
N8—N9—C30	127.7 (2)	C21—C20—H20	120.1
C30—N10—C29	120.1 (3)	C19—C20—H20	120.1
O1—C1—C2	106.3 (2)	C20—C21—C22	120.8 (2)
O1—C1—H1A	110.5	C20—C21—H21	119.6
C2—C1—H1A	110.5	C22—C21—H21	119.6
O1—C1—H1B	110.5	C21—C22—C23	120.3 (2)
C2—C1—H1B	110.5	C21—C22—H22	119.8
H1A—C1—H1B	108.7	C23—C22—H22	119.8
F1—C2—F2	105.6 (2)	C22—C23—C24	119.8 (2)
F1—C2—C1	110.6 (2)	C22—C23—C25	116.3 (2)
F2—C2—C1	110.8 (2)	C24—C23—C25	123.7 (2)
F1—C2—H2	109.9	C19—C24—C23	118.3 (2)
F2—C2—H2	109.9	C19—C24—S2	118.21 (17)
C1—C2—H2	109.9	C23—C24—S2	123.49 (18)
O1—C3—C4	123.6 (2)	F9—C25—F8	108.1 (2)
O1—C3—C8	116.1 (2)	F9—C25—F10	105.0 (2)
C4—C3—C8	120.3 (2)	F8—C25—F10	105.2 (2)
C5—C4—C3	119.5 (2)	F9—C25—C23	113.5 (2)
C5—C4—H4	120.3	F8—C25—C23	114.2 (2)
C3—C4—H4	120.3	F10—C25—C23	110.2 (2)
C4—C5—C6	121.0 (2)	N8—C26—N7	118.5 (2)
C4—C5—H5	119.5	N8—C26—N6	123.6 (2)
C6—C5—H5	119.5	N7—C26—N6	117.9 (2)
C5—C6—C7	120.4 (2)	N7—C27—N9	110.3 (2)
C5—C6—H6	119.8	N7—C27—C28	131.1 (3)
C7—C6—H6	119.8	N9—C27—C28	118.6 (2)
C6—C7—C8	119.8 (2)	C29—C28—O9	128.1 (3)
C6—C7—C9	115.7 (2)	C29—C28—C27	117.8 (3)
C8—C7—C9	124.5 (2)	O9—C28—C27	114.0 (3)
C7—C8—C3	118.7 (2)	C28—C29—N10	122.9 (3)
C7—C8—S1	124.01 (17)	C28—C29—H29	118.5
C3—C8—S1	117.28 (18)	N10—C29—H29	118.5
F4—C9—F5	106.3 (2)	N10—C30—O10	126.7 (3)

F4—C9—F3	107.5 (2)	N10—C30—N9	118.7 (3)
F5—C9—F3	105.6 (2)	O10—C30—N9	114.6 (2)
F4—C9—C7	113.7 (2)	O9—C31—H31A	109.5
F5—C9—C7	111.2 (2)	O9—C31—H31B	109.5
F3—C9—C7	112.0 (2)	H31A—C31—H31B	109.5
N3—C10—N2	118.3 (2)	O9—C31—H31C	109.5
N3—C10—N1	123.7 (2)	H31A—C31—H31C	109.5
N2—C10—N1	118.0 (2)	H31B—C31—H31C	109.5
N2—C11—N4	110.8 (2)	O10—C32—H32A	109.5
N2—C11—C12	130.9 (3)	O10—C32—H32B	109.5
N4—C11—C12	118.2 (3)	H32A—C32—H32B	109.5
C13—C12—O4	128.3 (3)	O10—C32—H32C	109.5
C13—C12—C11	117.6 (3)	H32A—C32—H32C	109.5
O4—C12—C11	114.1 (3)	H32B—C32—H32C	109.5
O3—S1—N1—C10	−171.86 (18)	C16—O5—C14—N4	−178.4 (2)
O2—S1—N1—C10	59.2 (2)	C11—N4—C14—N5	−0.7 (4)
C8—S1—N1—C10	−56.2 (2)	N3—N4—C14—N5	178.7 (2)
C10—N3—N4—C11	0.3 (2)	C11—N4—C14—O5	179.8 (2)
C10—N3—N4—C14	−179.1 (2)	N3—N4—C14—O5	−0.9 (4)
O7—S2—N6—C26	−176.81 (19)	C19—O6—C17—C18	175.3 (2)
O8—S2—N6—C26	54.1 (2)	O6—C17—C18—F6	−61.3 (3)
C24—S2—N6—C26	−60.7 (2)	O6—C17—C18—F7	55.7 (3)
C26—N8—N9—C27	0.3 (2)	C17—O6—C19—C20	−4.0 (3)
C26—N8—N9—C30	−178.6 (2)	C17—O6—C19—C24	177.0 (2)
C3—O1—C1—C2	168.7 (2)	O6—C19—C20—C21	−176.2 (2)
O1—C1—C2—F1	−56.8 (3)	C24—C19—C20—C21	2.8 (4)
O1—C1—C2—F2	59.8 (3)	C19—C20—C21—C22	3.3 (4)
C1—O1—C3—C4	−6.4 (3)	C20—C21—C22—C23	−4.0 (4)
C1—O1—C3—C8	175.1 (2)	C21—C22—C23—C24	−1.5 (4)
O1—C3—C4—C5	−174.1 (2)	C21—C22—C23—C25	173.3 (2)
C8—C3—C4—C5	4.3 (3)	O6—C19—C24—C23	171.0 (2)
C3—C4—C5—C6	1.0 (4)	C20—C19—C24—C23	−8.1 (3)
C4—C5—C6—C7	−3.2 (4)	O6—C19—C24—S2	−11.3 (3)
C5—C6—C7—C8	0.0 (3)	C20—C19—C24—S2	169.70 (19)
C5—C6—C7—C9	177.8 (2)	C22—C23—C24—C19	7.4 (3)
C6—C7—C8—C3	5.3 (3)	C25—C23—C24—C19	−167.0 (2)
C9—C7—C8—C3	−172.4 (2)	C22—C23—C24—S2	−170.27 (18)
C6—C7—C8—S1	−175.15 (17)	C25—C23—C24—S2	15.3 (3)
C9—C7—C8—S1	7.2 (3)	O7—S2—C24—C19	71.2 (2)
O1—C3—C8—C7	171.1 (2)	O8—S2—C24—C19	−155.77 (18)
C4—C3—C8—C7	−7.5 (3)	N6—S2—C24—C19	−41.5 (2)
O1—C3—C8—S1	−8.5 (3)	O7—S2—C24—C23	−111.1 (2)
C4—C3—C8—S1	172.95 (18)	O8—S2—C24—C23	21.9 (2)
O3—S1—C8—C7	−107.6 (2)	N6—S2—C24—C23	136.22 (19)
O2—S1—C8—C7	25.5 (2)	C22—C23—C25—F9	−138.3 (2)
N1—S1—C8—C7	139.78 (19)	C24—C23—C25—F9	36.3 (3)
O3—S1—C8—C3	72.01 (19)	C22—C23—C25—F8	97.3 (3)

O2—S1—C8—C3	−154.89 (17)	C24—C23—C25—F8	−88.1 (3)
N1—S1—C8—C3	−40.7 (2)	C22—C23—C25—F10	−20.8 (3)
C6—C7—C9—F4	−138.2 (2)	C24—C23—C25—F10	153.8 (2)
C8—C7—C9—F4	39.5 (3)	N9—N8—C26—N7	0.1 (3)
C6—C7—C9—F5	−18.2 (3)	N9—N8—C26—N6	−178.7 (2)
C8—C7—C9—F5	159.5 (2)	C27—N7—C26—N8	−0.5 (3)
C6—C7—C9—F3	99.7 (3)	C27—N7—C26—N6	178.4 (2)
C8—C7—C9—F3	−82.6 (3)	S2—N6—C26—N8	−36.0 (3)
N4—N3—C10—N2	0.0 (3)	S2—N6—C26—N7	145.19 (18)
N4—N3—C10—N1	−178.3 (2)	C26—N7—C27—N9	0.6 (2)
C11—N2—C10—N3	−0.2 (3)	C26—N7—C27—C28	178.6 (2)
C11—N2—C10—N1	178.2 (2)	N8—N9—C27—N7	−0.7 (3)
S1—N1—C10—N3	−39.4 (3)	C30—N9—C27—N7	178.4 (2)
S1—N1—C10—N2	142.28 (19)	N8—N9—C27—C28	−178.9 (2)
C10—N2—C11—N4	0.4 (3)	C30—N9—C27—C28	0.2 (3)
C10—N2—C11—C12	178.9 (3)	C31—O9—C28—C29	3.1 (4)
N3—N4—C11—N2	−0.4 (3)	C31—O9—C28—C27	−175.4 (2)
C14—N4—C11—N2	179.0 (2)	N7—C27—C28—C29	−177.5 (2)
N3—N4—C11—C12	−179.2 (2)	N9—C27—C28—C29	0.2 (4)
C14—N4—C11—C12	0.3 (4)	N7—C27—C28—O9	1.2 (4)
C15—O4—C12—C13	2.7 (4)	N9—C27—C28—O9	178.9 (2)
C15—O4—C12—C11	−176.1 (2)	O9—C28—C29—N10	−179.1 (2)
N2—C11—C12—C13	−178.4 (3)	C27—C28—C29—N10	−0.7 (4)
N4—C11—C12—C13	0.1 (4)	C30—N10—C29—C28	0.7 (4)
N2—C11—C12—O4	0.6 (4)	C29—N10—C30—O10	−179.7 (2)
N4—C11—C12—O4	179.1 (2)	C29—N10—C30—N9	−0.3 (4)
O4—C12—C13—N5	−178.9 (3)	C32—O10—C30—N10	1.5 (4)
C11—C12—C13—N5	0.0 (4)	C32—O10—C30—N9	−178.0 (2)
C14—N5—C13—C12	−0.4 (4)	C27—N9—C30—N10	−0.2 (4)
C13—N5—C14—O5	−179.8 (3)	N8—N9—C30—N10	178.7 (2)
C13—N5—C14—N4	0.7 (4)	C27—N9—C30—O10	179.4 (2)
C16—O5—C14—N5	2.0 (4)	N8—N9—C30—O10	−1.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···F10^{i}	0.99	2.48	3.293 (3)	139
C16—H16*C*···F3^{ii}	0.98	2.40	3.185 (3)	136
C17—H17*B*···O8^{i}	0.99	2.40	3.102 (3)	127
C18—H18···F10^{iii}	1.00	2.60	3.104 (3)	111
C20—H20···F9^{i}	0.95	2.55	3.466 (3)	162
C32—H32*C*···F8^{iv}	0.98	2.36	3.146 (3)	137

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