Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-Methyl-2-[4-(trifluoromethyl)phenyl]-8*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]-pyrimidin-5-amine methanol disolvate

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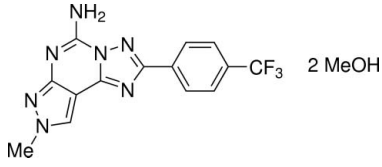
Received 22 June 2010; accepted 23 June 2010

Key indicators: single-crystal X-ray study; T = 223 K; mean σ(C–C) = 0.002 Å; R factor = 0.054; wR factor = 0.145; data-to-parameter ratio = 15.3.

In the title compound, $C_{14}H_{10}F_3N_7\cdot 2CH_4O$, the heterocyclic ring system is essentially planar (r.m.s. deviation = 0.009 Å) and makes a dihedral angle of 6.91 (8)° with the attached benzene ring. In the crystal, the main molecules form centrosymmetric $R_2^2(8)$ dimers *via* pairs of N—H···N hydrogen bonds between the amino groups and pyrimidine N atoms. One of the independent methanol molecules and its inversion equivalent are linked to the dimers *via* O—H···N and N—H···O hydrogen bonds, forming $R_4^4(16)$ graph-set motifs. The dimers along with the hydrogen-bonded methanol molecules are stacked along the *a* axis, with π–π interactions between the pyrazole and triazole rings [centroid–centroid distance = 3.4953 (10) Å].

Related literature

For reviews on pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine adenosine receptor antagonists, see: Baraldi *et al.* (2006); Cacciari *et al.* (2007). For the general method used for the synthesis of the title compound, see: Dolzhenko *et al.* (2009); Cheong *et al.* (2010). For the crystal structures of related pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidines, see: Ferretti *et al.* (2006); Mezheritsky *et al.* (2004); Tyurin *et al.* (2005); Xiao & Shi (2007). For graph-set analysis of hydrogen bonding, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{10}F_3N_7\cdot 2CH_4O$
M_r = 397.37
Monoclinic, $P2_1/n$
a = 4.6179 (3) Å
b = 17.1149 (10) Å
c = 22.7627 (13) Å
β = 94.323 (1)°
V = 1793.93 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 223 K
0.58 × 0.32 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.932, T_{max} = 0.985
12385 measured reflections
4076 independent reflections
3538 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.145
S = 1.05
4076 reflections
266 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*S*—H1*S*···N2^{i}	0.83	2.05	2.877 (2)	175
O2*S*—H2*S*···N6	0.83	2.04	2.853 (2)	165
N7—H7*A*···O1*S*	0.85 (2)	2.46 (2)	3.050 (2)	128 (2)
N7—H7*B*···N3^{i}	0.89 (2)	2.09 (3)	2.979 (2)	179 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Medical Research Council, Singapore (grant No. NMRC/NIG/0020/2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5111).

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supporting information

Acta Cryst. (2010). E**66**, o1835–o1836 [doi:10.1107/S1600536810024591]

8-Methyl-2-[4-(trifluoromethyl)phenyl]-8*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidin-5-amine methanol disolvate

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S1. Comment

Pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine system has been recognized as an excellent template for the construction of new adenosine receptor antagonists (Baraldi *et al.*, 2006; Cacciari *et al.*, 2007). However, information on the structure of this heterocyclic system is limited (Ferretti *et al.*, 2006; Mezheritsky *et al.*, 2004; Tyurin *et al.*, 2005; Xiao & Shi, 2007). In continuation of our works on the development of new pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine adenosine receptor antagonists (Dolzhenko *et al.*, 2009; Cheong *et al.*, 2010), we report here the molecular and crystal structure of 8-methyl-2-(4-trifluoromethylphenyl)-8*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidin-5-amine.

The compound crystallizes with two methanol solvent molecules. The heterocyclic ring system is essentially planar with an r.m.s. deviation of 0.009 Å. The phenyl ring makes a dihedral angle of 6.91 (8)° with the pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidine core. The trifluoromethyl group C atom, C13, is located 0.130 (3) Å above the C7—C12 mean plane.

In the crystal, molecules of 8-methyl-2-(4-trifluoromethylphenyl)-8*H*-pyrazolo[4,3-*e*][1,2,4]triazolo[1,5-*c*]pyrimidin-5-amine form centrosymmetric inversion dimers (Fig. 2). The pyrimidine N3 atom is connected with amino group N7—H7B of the pair molecule by intermolecular N···H—N hydrogen bond making $R_2^2(8)$ graph-set motif (Bernstein *et al.*,1995). Methanol hydroxy group O1S—H1S also links the heterocyclic molecules in the dimer by the N—H···O—H···N hydrogen bond array with amino group N7—H7A and N2 of the pyrazole ring making $R_4^4(16)$ graph-set motif. Another methanol molecule forms the O—H···N hydrogen bond with N6 of the triazole ring. The dimers are stacked along the *a* axis, with π–π interactions between pyrazole and triazole rings [centroid-to-centroid distance = 3.4953 (10) Å] (Fig. 2).

S2. Experimental

8-Methyl-2-(4-trifluoromethylphenyl)-8*H*-pyrazolo[4,3-*e*][1,2,4]triazolo][1,5-*c*]pyrimidin-5-amine was prepared from 8-methyl-2-(4-trifluoromethylphenyl)-8*H*-pyrazolo[4,3-*e*][1,2,4]triazolo][1,5-*c*]pyrimidine (Dolzhenko *et al.*, 2009) similarly to the described method (Cheong *et al.*, 2010). The detail procedure will be reported elsewhere. The crystals suitable for crystallographic analysis were grown by recrystallization from methanol. m.p. 573 K.

S3. Refinement

All C-bound H atoms were positioned geometrically and included in the refinement in riding-motion approximation [0.94 Å for CH of aromatic systems, 0.97 Å for methyl groups, and 0.83 Å for hydroxyl groups; $U_{iso}(H) = 1.2U_{eq}(C_{Ar})$ and $U_{iso}(H) = 1.5U_{eq}(O,C_{Me})$] while the amino group H atoms were located in a difference map and refined freely.

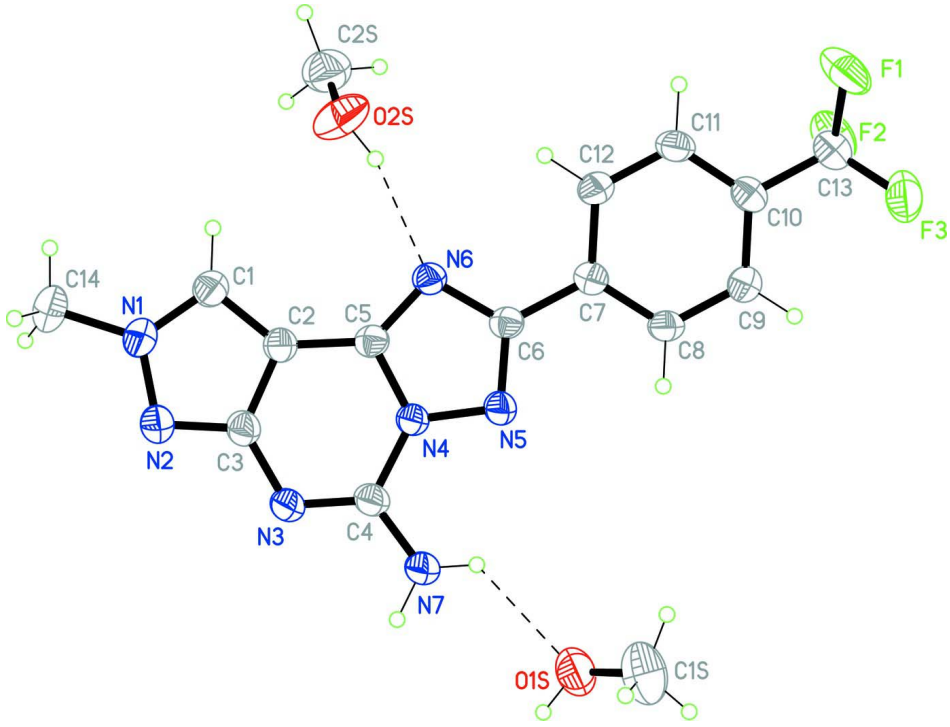

Figure 1

The asymmetric unit of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

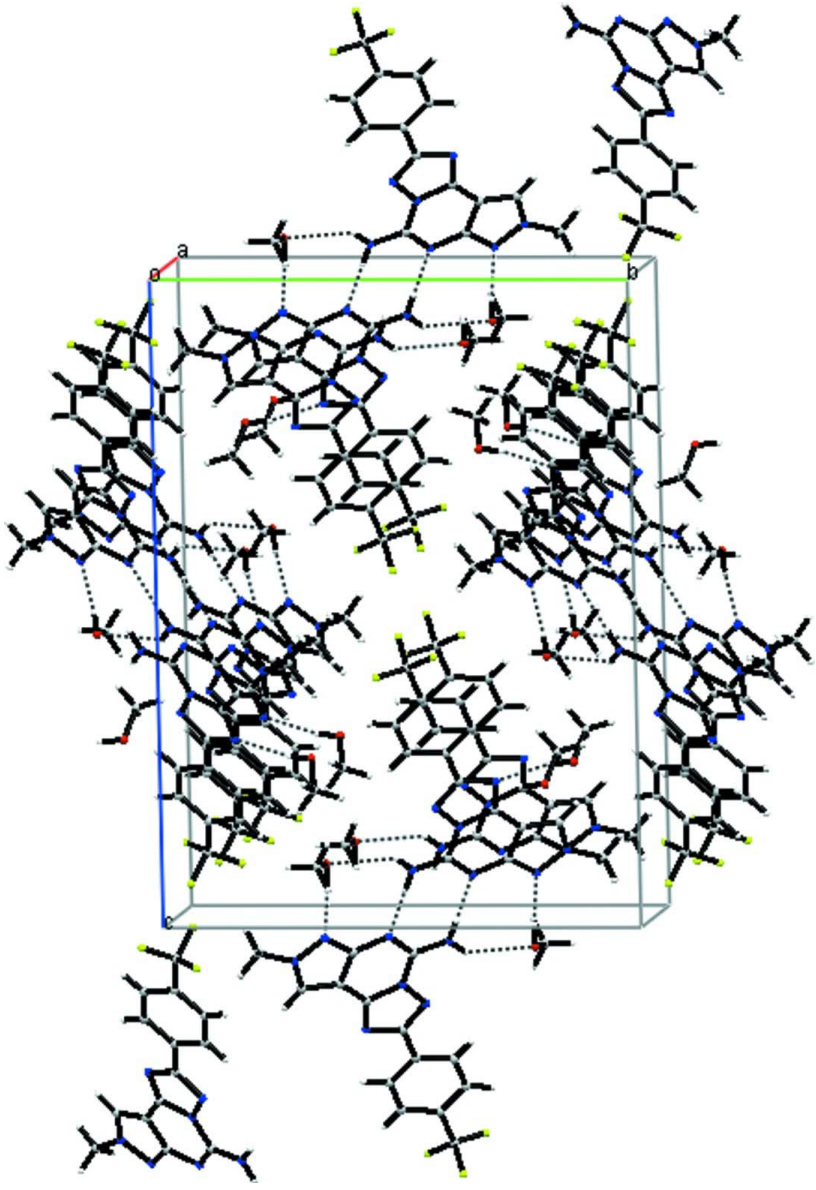

Figure 2

Crystal packing of the title compound, viewed along the *a* axis.

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Crystal data

$C_{14}H_{10}F_3N_7 \cdot 2CH_4O$
$M_r = 397.37$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.6179\ (3)$ Å
$b = 17.1149\ (10)$ Å
$c = 22.7627\ (13)$ Å
$\beta = 94.323\ (1)°$
$V = 1793.93\ (19)$ Å^3
$Z = 4$
$F(000) = 824$
$D_x = 1.471$ Mg m^{-3}
Melting point: 573 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4421 reflections
$\theta = 2.4–27.2°$

μ = 0.12 mm^{-1}
T = 223 K

Block, colourless
0.58 × 0.32 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.932, T_{max} = 0.985

12385 measured reflections
4076 independent reflections
3538 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 27.5°, θ_{min} = 1.5°
h = −5→5
k = −21→22
l = −23→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.145
S = 1.05
4076 reflections
266 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
$w = 1/[\sigma^2(F_o^2) + (0.0746P)^2 + 0.7421P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1S	0.5063 (3)	0.68580 (11)	0.08136 (7)	0.0581 (4)
H1S	0.4507	0.6882	0.0459	0.087*
C1S	0.8079 (5)	0.68768 (18)	0.08766 (13)	0.0684 (7)
H1S1	0.8735	0.7415	0.0887	0.103*
H1S2	0.8852	0.6611	0.0546	0.103*
H1S3	0.8756	0.6617	0.1240	0.103*
C2S	0.1594 (6)	0.15803 (13)	0.27649 (11)	0.0581 (6)
H2S1	0.2028	0.1917	0.3103	0.087*
H2S2	0.1779	0.1038	0.2887	0.087*
H2S3	−0.0374	0.1678	0.2602	0.087*
F1	1.4952 (3)	0.39342 (8)	0.43457 (6)	0.0616 (4)
F2	1.2039 (3)	0.48312 (8)	0.45616 (5)	0.0550 (4)
F3	1.5562 (3)	0.50993 (9)	0.40469 (6)	0.0642 (4)

N1	−0.3122 (3)	0.23144 (9)	0.07561 (7)	0.0362 (3)
N2	−0.2997 (3)	0.29563 (9)	0.04026 (7)	0.0375 (4)
N3	−0.0193 (3)	0.41222 (8)	0.04749 (6)	0.0365 (4)
N4	0.3019 (3)	0.41895 (8)	0.13254 (6)	0.0296 (3)
N5	0.5146 (3)	0.45421 (8)	0.16847 (6)	0.0309 (3)
N6	0.3934 (3)	0.33542 (8)	0.20487 (6)	0.0286 (3)
N7	0.2815 (4)	0.51915 (10)	0.06445 (8)	0.0461 (4)
H7A	0.402 (5)	0.5454 (13)	0.0863 (10)	0.044 (6)*
H7B	0.204 (5)	0.5402 (14)	0.0312 (11)	0.052 (6)*
C1	−0.1317 (4)	0.23497 (10)	0.12400 (8)	0.0343 (4)
H1	−0.1081	0.1971	0.1539	0.041*
C2	0.0145 (4)	0.30557 (9)	0.12153 (7)	0.0301 (4)
C3	−0.0985 (4)	0.34097 (10)	0.06864 (7)	0.0322 (4)
C4	0.1813 (4)	0.45034 (10)	0.07964 (7)	0.0336 (4)
C5	0.2331 (4)	0.34778 (9)	0.15530 (7)	0.0276 (3)
C6	0.5597 (4)	0.40191 (9)	0.21105 (7)	0.0269 (3)
C7	0.7738 (3)	0.41460 (9)	0.26155 (7)	0.0274 (3)
C8	0.9154 (4)	0.48630 (10)	0.26875 (7)	0.0326 (4)
H8	0.8786	0.5261	0.2407	0.039*
C9	1.1098 (4)	0.49892 (10)	0.31706 (8)	0.0343 (4)
H9	1.2041	0.5474	0.3221	0.041*
C10	1.1650 (4)	0.43965 (10)	0.35808 (7)	0.0307 (4)
C11	1.0299 (4)	0.36786 (10)	0.35067 (8)	0.0343 (4)
H11	1.0710	0.3277	0.3782	0.041*
C12	0.8337 (4)	0.35550 (10)	0.30243 (8)	0.0331 (4)
H12	0.7407	0.3068	0.2974	0.040*
C13	1.3561 (4)	0.45625 (11)	0.41268 (8)	0.0368 (4)
C14	−0.5043 (5)	0.16731 (12)	0.05670 (10)	0.0464 (5)
H14A	−0.4241	0.1396	0.0245	0.070*
H14B	−0.6940	0.1879	0.0436	0.070*
H14C	−0.5229	0.1317	0.0894	0.070*
O2S	0.3553 (4)	0.17366 (9)	0.23347 (8)	0.0626 (5)
H2S	0.3819	0.2215	0.2314	0.094*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1S	0.0507 (9)	0.0792 (12)	0.0434 (8)	−0.0125 (8)	−0.0029 (7)	−0.0038 (8)
C1S	0.0505 (14)	0.0805 (18)	0.0727 (17)	0.0068 (12)	−0.0050 (12)	−0.0207 (14)
C2S	0.0740 (16)	0.0445 (12)	0.0554 (13)	0.0039 (11)	0.0027 (12)	0.0023 (10)
F1	0.0664 (9)	0.0601 (8)	0.0537 (8)	0.0231 (7)	−0.0252 (6)	−0.0033 (6)
F2	0.0506 (7)	0.0806 (9)	0.0333 (6)	0.0123 (6)	0.0006 (5)	−0.0145 (6)
F3	0.0544 (8)	0.0857 (10)	0.0502 (7)	−0.0294 (7)	−0.0117 (6)	0.0062 (7)
N1	0.0395 (8)	0.0307 (8)	0.0381 (8)	−0.0050 (6)	0.0010 (6)	−0.0035 (6)
N2	0.0428 (9)	0.0336 (8)	0.0348 (8)	−0.0033 (6)	−0.0047 (6)	−0.0025 (6)
N3	0.0491 (9)	0.0286 (7)	0.0300 (7)	−0.0014 (6)	−0.0081 (6)	0.0023 (6)
N4	0.0391 (8)	0.0237 (7)	0.0254 (7)	−0.0008 (5)	−0.0027 (5)	−0.0003 (5)
N5	0.0385 (8)	0.0260 (7)	0.0273 (7)	−0.0014 (6)	−0.0036 (6)	−0.0018 (5)

N6	0.0343 (7)	0.0250 (7)	0.0264 (7)	0.0006 (5)	0.0011 (5)	0.0003 (5)
N7	0.0665 (12)	0.0316 (8)	0.0369 (9)	−0.0117 (8)	−0.0177 (8)	0.0086 (7)
C1	0.0389 (9)	0.0310 (9)	0.0332 (9)	−0.0026 (7)	0.0030 (7)	−0.0004 (7)
C2	0.0350 (9)	0.0278 (8)	0.0274 (8)	0.0001 (6)	0.0019 (6)	−0.0020 (6)
C3	0.0382 (9)	0.0286 (8)	0.0292 (8)	0.0018 (7)	−0.0014 (7)	−0.0029 (6)
C4	0.0441 (10)	0.0277 (8)	0.0279 (8)	0.0018 (7)	−0.0037 (7)	0.0018 (6)
C5	0.0335 (8)	0.0232 (7)	0.0264 (7)	0.0026 (6)	0.0040 (6)	−0.0013 (6)
C6	0.0322 (8)	0.0234 (7)	0.0253 (7)	0.0024 (6)	0.0031 (6)	−0.0008 (6)
C7	0.0295 (8)	0.0277 (8)	0.0252 (7)	0.0032 (6)	0.0029 (6)	−0.0008 (6)
C8	0.0402 (9)	0.0272 (8)	0.0299 (8)	−0.0004 (7)	−0.0010 (7)	0.0052 (6)
C9	0.0374 (9)	0.0300 (8)	0.0349 (9)	−0.0041 (7)	−0.0011 (7)	−0.0002 (7)
C10	0.0281 (8)	0.0361 (9)	0.0279 (8)	0.0051 (7)	0.0022 (6)	−0.0005 (7)
C11	0.0398 (9)	0.0312 (9)	0.0314 (8)	0.0042 (7)	−0.0016 (7)	0.0068 (7)
C12	0.0394 (9)	0.0252 (8)	0.0344 (9)	−0.0010 (7)	0.0000 (7)	0.0021 (6)
C13	0.0350 (9)	0.0426 (10)	0.0323 (9)	0.0037 (7)	0.0002 (7)	−0.0005 (7)
C14	0.0476 (11)	0.0386 (10)	0.0526 (12)	−0.0119 (9)	0.0009 (9)	−0.0091 (8)
O2S	0.0839 (12)	0.0294 (7)	0.0764 (11)	0.0002 (8)	0.0186 (9)	0.0034 (7)

Geometric parameters (Å, º)

O1S—C1S	1.390 (3)	N7—C4	1.321 (2)
O1S—H1S	0.83	N7—H7A	0.85 (2)
C1S—H1S1	0.97	N7—H7B	0.89 (2)
C1S—H1S2	0.97	C1—C2	1.387 (2)
C1S—H1S3	0.97	C1—H1	0.94
C2S—O2S	1.408 (3)	C2—C3	1.412 (2)
C2S—H2S1	0.97	C2—C5	1.419 (2)
C2S—H2S2	0.97	C6—C7	1.475 (2)
C2S—H2S3	0.97	C7—C12	1.388 (2)
F1—C13	1.330 (2)	C7—C8	1.394 (2)
F2—C13	1.338 (2)	C8—C9	1.383 (2)
F3—C13	1.325 (2)	C8—H8	0.94
N1—C1	1.331 (2)	C9—C10	1.389 (2)
N1—N2	1.366 (2)	C9—H9	0.94
N1—C14	1.456 (2)	C10—C11	1.382 (3)
N2—C3	1.339 (2)	C10—C13	1.496 (2)
N3—C4	1.310 (2)	C11—C12	1.386 (2)
N3—C3	1.371 (2)	C11—H11	0.94
N4—N5	1.3698 (19)	C12—H12	0.94
N4—C5	1.370 (2)	C14—H14A	0.97
N4—C4	1.396 (2)	C14—H14B	0.97
N5—C6	1.324 (2)	C14—H14C	0.97
N6—C5	1.319 (2)	O2S—H2S	0.83
N6—C6	1.374 (2)		
C1S—O1S—H1S	109.5	N6—C5—N4	109.56 (14)
O1S—C1S—H1S1	109.5	N6—C5—C2	135.39 (15)
O1S—C1S—H1S2	109.5	N4—C5—C2	115.05 (14)

H1S1—C1S—H1S2	109.5	N5—C6—N6	115.41 (14)
O1S—C1S—H1S3	109.5	N5—C6—C7	122.01 (14)
H1S1—C1S—H1S3	109.5	N6—C6—C7	122.58 (14)
H1S2—C1S—H1S3	109.5	C12—C7—C8	119.65 (15)
O2S—C2S—H2S1	109.5	C12—C7—C6	120.21 (15)
O2S—C2S—H2S2	109.5	C8—C7—C6	120.14 (14)
H2S1—C2S—H2S2	109.5	C9—C8—C7	120.09 (15)
O2S—C2S—H2S3	109.5	C9—C8—H8	120.0
H2S1—C2S—H2S3	109.5	C7—C8—H8	120.0
H2S2—C2S—H2S3	109.5	C8—C9—C10	119.71 (16)
C1—N1—N2	113.54 (14)	C8—C9—H9	120.1
C1—N1—C14	127.47 (16)	C10—C9—H9	120.1
N2—N1—C14	118.95 (15)	C11—C10—C9	120.58 (16)
C3—N2—N1	103.90 (14)	C11—C10—C13	120.16 (16)
C4—N3—C3	116.28 (14)	C9—C10—C13	119.11 (16)
N5—N4—C5	110.01 (13)	C10—C11—C12	119.62 (15)
N5—N4—C4	124.55 (14)	C10—C11—H11	120.2
C5—N4—C4	125.41 (14)	C12—C11—H11	120.2
C6—N5—N4	101.84 (13)	C11—C12—C7	120.34 (16)
C5—N6—C6	103.18 (13)	C11—C12—H12	119.8
C4—N7—H7A	123.1 (15)	C7—C12—H12	119.8
C4—N7—H7B	117.2 (15)	F3—C13—F1	106.84 (16)
H7A—N7—H7B	119 (2)	F3—C13—F2	105.89 (16)
N1—C1—C2	106.40 (15)	F1—C13—F2	105.45 (15)
N1—C1—H1	126.8	F3—C13—C10	113.04 (15)
C2—C1—H1	126.8	F1—C13—C10	113.31 (15)
C1—C2—C3	104.97 (15)	F2—C13—C10	111.71 (14)
C1—C2—C5	138.58 (16)	N1—C14—H14A	109.5
C3—C2—C5	116.45 (15)	N1—C14—H14B	109.5
N2—C3—N3	122.67 (15)	H14A—C14—H14B	109.5
N2—C3—C2	111.18 (15)	N1—C14—H14C	109.5
N3—C3—C2	126.15 (15)	H14A—C14—H14C	109.5
N3—C4—N7	122.99 (16)	H14B—C14—H14C	109.5
N3—C4—N4	120.64 (15)	C2S—O2S—H2S	109.5
N7—C4—N4	116.37 (16)		

C1—N1—N2—C3	0.3 (2)	C1—C2—C5—N6	0.6 (4)
C14—N1—N2—C3	−177.53 (16)	C3—C2—C5—N6	−178.44 (18)
C5—N4—N5—C6	−0.34 (17)	C1—C2—C5—N4	−179.5 (2)
C4—N4—N5—C6	−178.42 (15)	C3—C2—C5—N4	1.4 (2)
N2—N1—C1—C2	−0.4 (2)	N4—N5—C6—N6	0.51 (18)
C14—N1—C1—C2	177.22 (17)	N4—N5—C6—C7	−179.41 (14)
N1—C1—C2—C3	0.29 (19)	C5—N6—C6—N5	−0.49 (19)
N1—C1—C2—C5	−178.9 (2)	C5—N6—C6—C7	179.44 (14)
N1—N2—C3—N3	179.97 (16)	N5—C6—C7—C12	−174.11 (16)
N1—N2—C3—C2	−0.1 (2)	N6—C6—C7—C12	6.0 (2)
C4—N3—C3—N2	−179.75 (17)	N5—C6—C7—C8	6.4 (2)
C4—N3—C3—C2	0.3 (3)	N6—C6—C7—C8	−173.47 (15)

C1—C2—C3—N2	−0.1 (2)	C12—C7—C8—C9	−1.4 (3)
C5—C2—C3—N2	179.25 (15)	C6—C7—C8—C9	178.09 (16)
C1—C2—C3—N3	179.81 (17)	C7—C8—C9—C10	0.5 (3)
C5—C2—C3—N3	−0.8 (3)	C8—C9—C10—C11	0.8 (3)
C3—N3—C4—N7	179.18 (18)	C8—C9—C10—C13	−174.85 (16)
C3—N3—C4—N4	−0.5 (3)	C9—C10—C11—C12	−1.2 (3)
N5—N4—C4—N3	179.14 (16)	C13—C10—C11—C12	174.37 (16)
C5—N4—C4—N3	1.3 (3)	C10—C11—C12—C7	0.4 (3)
N5—N4—C4—N7	−0.6 (3)	C8—C7—C12—C11	0.9 (3)
C5—N4—C4—N7	−178.36 (17)	C6—C7—C12—C11	−178.53 (16)
C6—N6—C5—N4	0.23 (17)	C11—C10—C13—F3	154.07 (17)
C6—N6—C5—C2	−179.89 (18)	C9—C10—C13—F3	−30.3 (2)
N5—N4—C5—N6	0.06 (19)	C11—C10—C13—F1	32.3 (2)
C4—N4—C5—N6	178.13 (15)	C9—C10—C13—F1	−152.04 (17)
N5—N4—C5—C2	−179.84 (14)	C11—C10—C13—F2	−86.6 (2)
C4—N4—C5—C2	−1.8 (2)	C9—C10—C13—F2	89.0 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*S*—H1*S*···N2^{i}	0.83	2.05	2.877 (2)	175
O2*S*—H2*S*···N6	0.83	2.04	2.853 (2)	165
N7—H7*A*···O1*S*	0.85 (2)	2.46 (2)	3.050 (2)	128 (2)
N7—H7*B*···N3^{i}	0.89 (2)	2.09 (3)	2.979 (2)	179 (2)

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