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Morpholine-4-carboxamidinium sulfate

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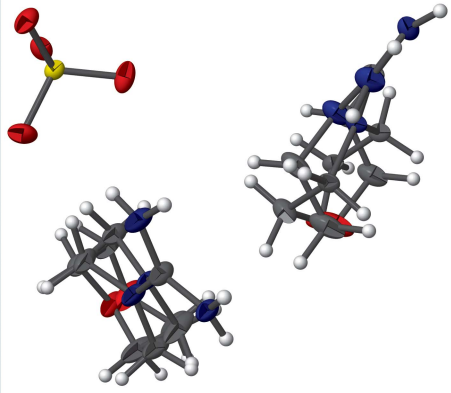
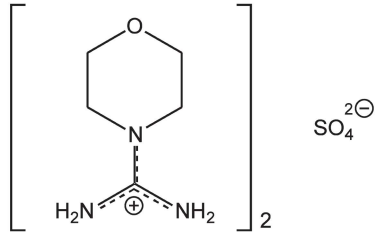
The asymmetric unit of the title salt, $2C_5H_{12}N_3O^+\cdot SO_4^{2-}$, comprises two cations and one sulfate ion. In both cations, the C, N and O atoms of the morpholine rings are disordered over two sets of sites, with refined occupancies of 0.849 (3):0.151 (3) for cation I and 0.684 (4):0.316 (4) for cation II. The C—N bond lengths in both central C_3N units of the carboxamidinium ions range between 1.253 (12) and 1.362 (5) Å, indicating a degree of double-bond character. The central C atoms are bonded to the three N atoms in a nearly ideal trigonal–planar geometry and the positive charges are delocalized in both CN_3 planes. The crystal structure is stabilized by a three-dimensional network of N—H···O hydrogen bonds between the cations and the sulfate ion.

3D view



Chemical scheme



Structure description

The title salt is the second structurally characterized compound in a series of morpholine-4-carboxamidinium derivatives. The asymmetric unit comprises two carboxamidinium ions and one sulfate ion (Fig. 1). In both cations, the carbon, nitrogen and oxygen atoms of the morpholine rings are disordered over two alternative chair conformations (Fig. 2). The C—N bonds of the CN_3 units range from 1.253 (12) to 1.362 (5) Å, showing partial double-bond character. The N—C1—N and N—C6—N angles range from 117.14 (12)° to 122.2 (4)°, indicating that the carbon atoms C1 and C6 adopt nearly ideal trigonal–planar environments. The positive charges are completely delocalized on the CN_3 planes. The structural parameters of the morpholine-4-carboxamidinium ions in the title compound agree very well with the data obtained from the X-ray analysis of the compound morpholine-4-carboxamidinium ethyl carbonate (Tiritiris, 2012*b*). The crystal structure of the related 4-morpholinecarboxamidine, has also been reported (Tiritiris, 2012*a*).

The crystal structure is stabilized by N—H···O hydrogen bonds (Table 1), forming a three-dimensional network (Figs. 3 and 4).

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11···O2^{i}	0.87 (2)	2.07 (2)	2.866 (2)	153 (2)
N1—H12···O2^{ii}	0.89 (2)	1.95 (2)	2.820 (2)	164 (2)
N2—H21···O4^{ii}	0.91 (2)	1.99 (2)	2.881 (2)	174 (2)
N2—H22···O3^{iii}	0.88 (2)	2.56 (2)	3.309 (2)	144 (2)
N2—H22···O4^{iii}	0.88 (2)	2.21 (2)	2.956 (2)	143 (2)
N4—H41···O1^{iii}	0.89 (2)	1.89 (2)	2.779 (2)	170 (2)
N4—H42···O3^{iv}	0.83 (2)	2.06 (2)	2.799 (2)	150 (2)
N5—H51···O3^{iii}	0.86 (2)	2.04 (2)	2.894 (2)	168 (2)
N5—H52···O1	0.86 (2)	2.48 (2)	3.113 (2)	131 (2)
N5—H52···O4	0.86 (2)	2.28 (2)	3.121 (2)	159 (2)

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

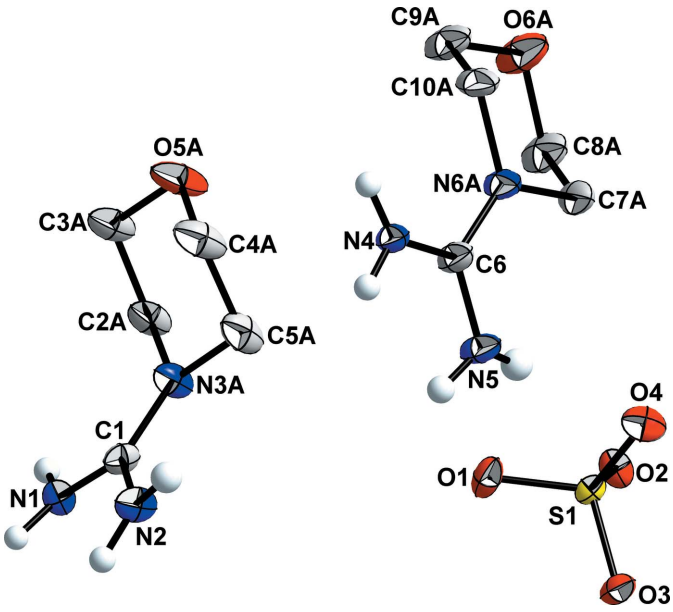


Figure 1
The structure of the title compound with displacement ellipsoids at the 50% probability level. All carbon-bonded hydrogen atoms are omitted for the sake of clarity. Only the disordered carbon, nitrogen and oxygen atoms of the morpholine rings with the major population are shown.

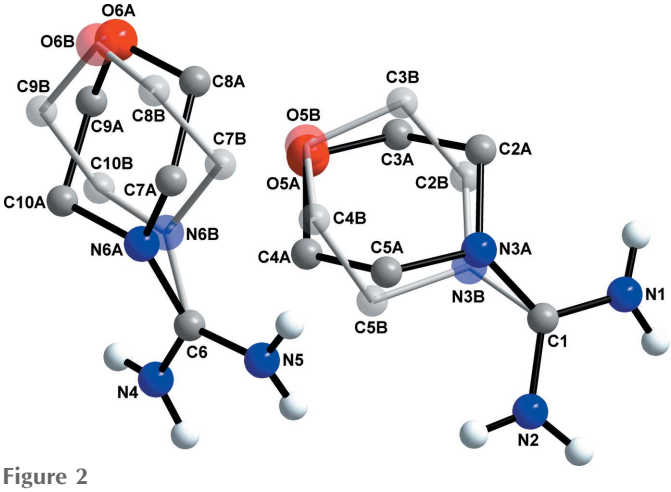


Figure 2
The structures of the orientationally disordered cations I and II. The carbon, nitrogen and oxygen atoms of the morpholine ring are disordered between the opaque and dark positions.

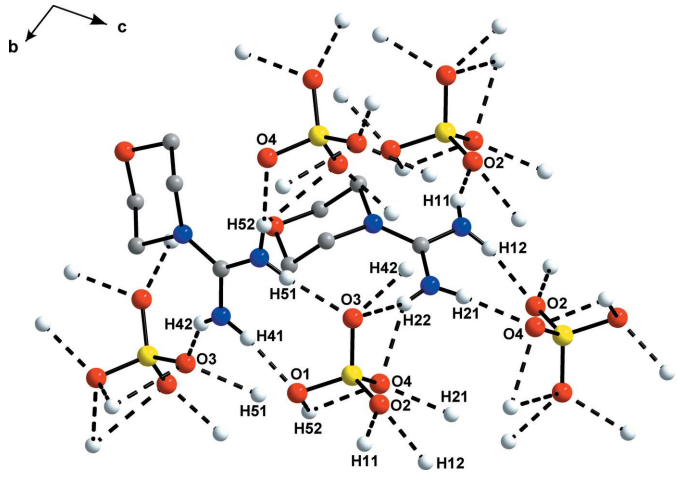


Figure 3
N—H···O hydrogen bonds (black dashed lines) in the crystal structure of the title compound (view normal to the *bc* plane).

Synthesis and crystallization

By heating *O*-methylisourea sulfate with two equivalents of morpholine, the title salt morpholine-4-carboxamidinium sulfate was obtained in nearly quantitative yield. The crude product was crystallized from a saturated acetonitrile–water solution. After slow evaporation of the solvent at ambient temperature, colorless single crystals suitable for X-ray analysis were obtained.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Atoms C2–C5, N3, O5 of cation I and C7–C10, N6, O6 of cation II are disordered over two sets of sites [C2*A*/C2*B*–C5*A*/C5*B*, N3*A*/N3*B*, O3*A*/O3*B* (cation I);

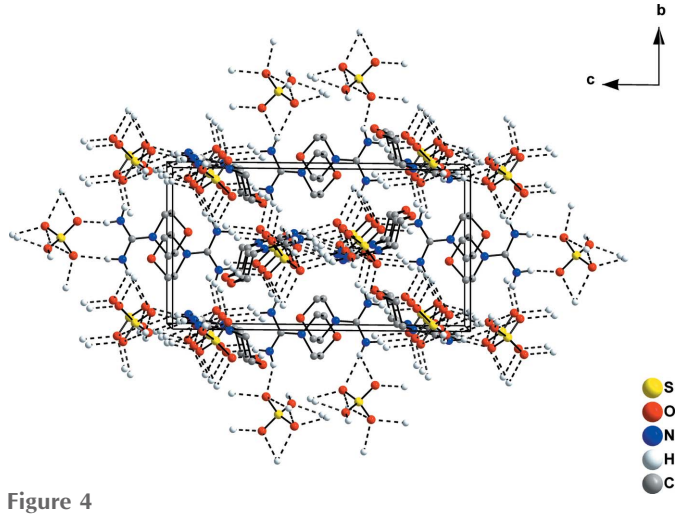


Figure 4
Molecular packing of the title compound. The N—H···O hydrogen bonds are depicted by black dashed lines (view along *bc*).

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_5H_{12}N_3O^+\cdot O_4S^{2-}$
M_r	356.41
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, b, c (Å)	9.7194 (3), 9.4737 (3), 17.9416 (8)
β (°)	105.386 (1)
V (Å^3)	1592.8 (1)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.25
Crystal size (mm)	0.30 × 0.22 × 0.09
Data collection	
Diffractometer	Bruker–Nonius KappaCCD diffractometer
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7346, 3791, 3284
R_{int}	0.016
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.658
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.031, 0.086, 1.04
No. of reflections	3791
No. of parameters	351
No. of restraints	96
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.45

Computer programs: *COLLECT* (Hooft, 2004), *DENZO-SMN* (Otwinowski & Minor, 1997), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Brandenburg & Putz, 2005).

C7*A*/C7*B*–C10*A*/C10*B*, N6*A*/N6*B*, O6*A*/O6*B* (cation II)] with refined occupancy ratios of 0.849 (3):0.151 (3) (cation I) and 0.684 (4):0.316 (4) (cation II). The major and minor disordered components of both cations were each restrained to have similar geometries (e.s.d. 0.02 Å).

Acknowledgements

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full crystallographic data

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Morpholine-4-carboxamidinium sulfate

Ioannis Tiritiris and Willi Kantlehner

Bis(morpholine-4-carboxamidinium) sulfate

Crystal data

$2C_5H_{12}N_3O^+\cdot O_4S^{2-}$
M_r = 356.41
Monoclinic, $P2_1/c$
a = 9.7194 (3) Å
b = 9.4737 (3) Å
c = 17.9416 (8) Å
β = 105.386 (1)°
V = 1592.8 (1) Å^3
Z = 4
$F(000)$ = 760
D_x = 1.486 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 16137 reflections
θ = 0.4–27.9°
μ = 0.25 mm^{-1}
T = 100 K
Block, colorless
0.30 × 0.22 × 0.09 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ scans, and ω scans
7346 measured reflections
3791 independent reflections
3284 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 27.9°, θ_{min} = 2.2°
h = −12→12
k = −12→12
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.086
S = 1.04
3791 reflections
351 parameters
96 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 0.5482P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0169 (19)

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}	Occ. (<1)
S1	0.34109 (3)	0.04700 (3)	0.13172 (2)	0.01678 (10)	
O1	0.31289 (9)	0.15120 (10)	0.18623 (5)	0.0287 (2)	
O2	0.20703 (8)	−0.01540 (10)	0.08510 (5)	0.0228 (2)	
O3	0.43384 (9)	−0.06576 (9)	0.17503 (5)	0.0245 (2)	
O4	0.41605 (9)	0.11615 (11)	0.07973 (5)	0.0290 (2)	
N1	0.09034 (11)	0.43956 (12)	0.42911 (6)	0.0209 (2)	
H11	−0.0017 (19)	0.4426 (18)	0.4099 (10)	0.034 (4)*	
H12	0.1265 (17)	0.4464 (17)	0.4800 (10)	0.029 (4)*	
N2	0.31799 (11)	0.46406 (13)	0.41952 (7)	0.0276 (3)	
H21	0.3434 (18)	0.4373 (18)	0.4698 (11)	0.034 (4)*	
H22	0.381 (2)	0.500 (2)	0.3976 (11)	0.048 (5)*	
C1	0.17786 (12)	0.47050 (14)	0.38565 (8)	0.0226 (3)	
N3A	0.1259 (2)	0.4973 (3)	0.30900 (13)	0.0273 (5)	0.849 (3)
C2A	−0.02492 (16)	0.5293 (2)	0.27179 (10)	0.0287 (4)	0.849 (3)
H2A1	−0.0837	0.5078	0.3078	0.034*	0.849 (3)
H2A2	−0.0592	0.4702	0.2250	0.034*	0.849 (3)
C3A	−0.03936 (16)	0.6826 (2)	0.24999 (10)	0.0326 (5)	0.849 (3)
H3A1	−0.1402	0.7036	0.2233	0.039*	0.849 (3)
H3A2	−0.0119	0.7411	0.2974	0.039*	0.849 (3)
O5A	0.0492 (3)	0.7181 (5)	0.2004 (2)	0.0429 (11)	0.849 (3)
C4A	0.19575 (17)	0.6888 (2)	0.23798 (12)	0.0395 (5)	0.849 (3)
H4A1	0.2263	0.7462	0.2856	0.047*	0.849 (3)
H4A2	0.2557	0.7154	0.2034	0.047*	0.849 (3)
C5A	0.21679 (19)	0.5350 (3)	0.25823 (12)	0.0353 (5)	0.849 (3)
H5A1	0.1907	0.4771	0.2106	0.042*	0.849 (3)
H5A2	0.3182	0.5167	0.2850	0.042*	0.849 (3)
N3B	0.1385 (11)	0.5417 (12)	0.3248 (7)	0.016 (2)	0.151 (3)
C2B	−0.0061 (8)	0.6053 (10)	0.2992 (5)	0.0194 (19)	0.151 (3)
H2B1	−0.0036	0.7025	0.3198	0.023*	0.151 (3)
H2B2	−0.0737	0.5494	0.3200	0.023*	0.151 (3)
C3B	−0.0569 (8)	0.6089 (9)	0.2122 (5)	0.0203 (18)	0.151 (3)
H3B1	−0.0608	0.5123	0.1908	0.024*	0.151 (3)
H3B2	−0.1534	0.6512	0.1953	0.024*	0.151 (3)
O5B	0.0459 (16)	0.695 (2)	0.1858 (13)	0.027 (3)	0.151 (3)
C4B	0.1742 (8)	0.6091 (11)	0.1992 (5)	0.026 (2)	0.151 (3)
H4B1	0.2419	0.6510	0.1728	0.032*	0.151 (3)
H4B2	0.1500	0.5125	0.1789	0.032*	0.151 (3)
C5B	0.2399 (8)	0.6047 (11)	0.2852 (5)	0.0212 (19)	0.151 (3)
H5B1	0.3286	0.5480	0.2965	0.025*	0.151 (3)
H5B2	0.2650	0.7016	0.3048	0.025*	0.151 (3)

N4	0.50660 (12)	0.65021 (12)	0.16492 (6)	0.0237 (2)	
H41	0.5630 (18)	0.6385 (18)	0.2122 (10)	0.034 (4)*	
H42	0.4752 (17)	0.729 (2)	0.1504 (10)	0.033 (4)*	
N5	0.48910 (13)	0.41075 (12)	0.15820 (7)	0.0285 (3)	
H51	0.5246 (19)	0.4134 (19)	0.2073 (11)	0.038 (5)*	
H52	0.448 (2)	0.335 (2)	0.1367 (12)	0.051 (5)*	
C6	0.45058 (14)	0.53623 (14)	0.12572 (8)	0.0263 (3)	
N6A	0.3734 (5)	0.5485 (5)	0.0507 (3)	0.0306 (9)	0.684 (4)
C7A	0.3111 (3)	0.4280 (2)	0.00101 (13)	0.0337 (6)	0.684 (4)
H7A1	0.3378	0.3388	0.0299	0.040*	0.684 (4)
H7A2	0.3494	0.4256	−0.0449	0.040*	0.684 (4)
C8A	0.1510 (3)	0.4412 (2)	−0.02445 (18)	0.0425 (7)	0.684 (4)
H8A1	0.1110	0.3621	−0.0595	0.051*	0.684 (4)
H8A2	0.1126	0.4346	0.0213	0.051*	0.684 (4)
O6A	0.1069 (6)	0.5720 (5)	−0.0634 (3)	0.0470 (12)	0.684 (4)
C9A	0.1645 (3)	0.6870 (2)	−0.01292 (18)	0.0436 (7)	0.684 (4)
H9A1	0.1277	0.6824	0.0335	0.052*	0.684 (4)
H9A2	0.1323	0.7773	−0.0397	0.052*	0.684 (4)
C10A	0.3258 (4)	0.6835 (3)	0.01176 (19)	0.0366 (7)	0.684 (4)
H10A	0.3636	0.6937	−0.0340	0.044*	0.684 (4)
H10B	0.3624	0.7628	0.0474	0.044*	0.684 (4)
N6B	0.3402 (9)	0.5432 (9)	0.0630 (5)	0.0207 (15)	0.316 (4)
C7B	0.2509 (5)	0.4213 (4)	0.0306 (3)	0.0261 (11)	0.316 (4)
H7B1	0.1683	0.4162	0.0529	0.031*	0.316 (4)
H7B2	0.3070	0.3334	0.0441	0.031*	0.316 (4)
C8B	0.1994 (6)	0.4346 (5)	−0.0553 (3)	0.0281 (11)	0.316 (4)
H8B1	0.2816	0.4246	−0.0777	0.034*	0.316 (4)
H8B2	0.1320	0.3567	−0.0756	0.034*	0.316 (4)
O6B	0.1311 (12)	0.5645 (10)	−0.0790 (5)	0.0351 (19)	0.316 (4)
C9B	0.2220 (8)	0.6785 (5)	−0.0515 (3)	0.0489 (19)	0.316 (4)
H9B1	0.1693	0.7674	−0.0681	0.059*	0.316 (4)
H9B2	0.3029	0.6751	−0.0753	0.059*	0.316 (4)
C10B	0.2793 (10)	0.6805 (7)	0.0341 (4)	0.048 (2)	0.316 (4)
H10C	0.3538	0.7542	0.0489	0.057*	0.316 (4)
H10D	0.2016	0.7045	0.0581	0.057*	0.316 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01160 (14)	0.01619 (16)	0.02146 (16)	0.00006 (10)	0.00249 (10)	−0.00071 (10)
O1	0.0219 (4)	0.0287 (5)	0.0314 (5)	0.0078 (4)	−0.0001 (4)	−0.0108 (4)
O2	0.0141 (4)	0.0334 (5)	0.0202 (4)	−0.0053 (3)	0.0032 (3)	−0.0035 (4)
O3	0.0253 (4)	0.0175 (4)	0.0255 (4)	0.0043 (3)	−0.0025 (3)	−0.0013 (3)
O4	0.0245 (4)	0.0302 (5)	0.0327 (5)	−0.0082 (4)	0.0085 (4)	0.0063 (4)
N1	0.0130 (5)	0.0290 (6)	0.0196 (5)	−0.0017 (4)	0.0026 (4)	−0.0021 (4)
N2	0.0139 (5)	0.0363 (7)	0.0329 (6)	0.0019 (4)	0.0067 (4)	0.0081 (5)
C1	0.0158 (5)	0.0236 (6)	0.0290 (6)	0.0026 (4)	0.0070 (5)	0.0044 (5)
N3A	0.0146 (7)	0.0434 (15)	0.0246 (10)	0.0027 (8)	0.0062 (7)	0.0090 (9)

C2A	0.0150 (7)	0.0459 (12)	0.0238 (8)	−0.0001 (7)	0.0025 (6)	0.0080 (8)
C3A	0.0185 (7)	0.0497 (12)	0.0292 (9)	0.0021 (7)	0.0058 (6)	0.0152 (9)
O5A	0.0224 (9)	0.070 (2)	0.0365 (13)	0.0032 (10)	0.0083 (7)	0.0290 (15)
C4A	0.0201 (8)	0.0636 (14)	0.0359 (10)	−0.0010 (8)	0.0094 (7)	0.0204 (10)
C5A	0.0217 (8)	0.0574 (14)	0.0311 (9)	0.0047 (9)	0.0145 (7)	0.0114 (9)
N3B	0.006 (3)	0.022 (5)	0.021 (5)	0.000 (3)	0.004 (3)	0.003 (4)
C2B	0.011 (3)	0.021 (4)	0.026 (4)	0.003 (3)	0.005 (3)	0.005 (3)
C3B	0.017 (3)	0.020 (4)	0.024 (4)	−0.002 (3)	0.004 (3)	0.002 (3)
O5B	0.015 (4)	0.023 (4)	0.045 (8)	0.004 (3)	0.012 (4)	0.020 (5)
C4B	0.020 (3)	0.038 (5)	0.021 (4)	0.006 (3)	0.006 (3)	0.006 (4)
C5B	0.016 (3)	0.022 (4)	0.028 (4)	0.000 (3)	0.011 (3)	0.003 (3)
N4	0.0312 (6)	0.0162 (5)	0.0210 (5)	0.0026 (4)	0.0023 (4)	0.0009 (4)
N5	0.0329 (6)	0.0162 (5)	0.0298 (6)	0.0009 (5)	−0.0029 (5)	0.0009 (5)
C6	0.0304 (7)	0.0182 (6)	0.0265 (6)	0.0024 (5)	0.0009 (5)	−0.0006 (5)
N6A	0.049 (2)	0.0147 (11)	0.0230 (15)	0.0008 (13)	0.0011 (12)	−0.0002 (9)
C7A	0.0560 (15)	0.0171 (10)	0.0232 (10)	0.0012 (9)	0.0019 (10)	−0.0044 (8)
C8A	0.0561 (17)	0.0193 (11)	0.0445 (16)	−0.0003 (10)	0.0001 (13)	−0.0064 (10)
O6A	0.0515 (17)	0.0242 (12)	0.051 (3)	0.0039 (11)	−0.0113 (16)	−0.0065 (14)
C9A	0.0610 (16)	0.0181 (10)	0.0407 (15)	0.0059 (10)	−0.0058 (12)	−0.0027 (10)
C10A	0.0622 (19)	0.0160 (11)	0.0255 (14)	0.0002 (11)	0.0010 (11)	0.0037 (10)
N6B	0.029 (3)	0.012 (2)	0.017 (3)	0.0015 (19)	−0.002 (2)	0.0013 (18)
C7B	0.031 (2)	0.0183 (19)	0.024 (2)	−0.0029 (16)	−0.0022 (17)	−0.0002 (16)
C8B	0.035 (2)	0.020 (2)	0.026 (2)	0.0007 (17)	0.0026 (18)	−0.0024 (17)
O6B	0.060 (4)	0.020 (2)	0.015 (2)	0.008 (2)	−0.007 (2)	0.0001 (16)
C9B	0.077 (4)	0.020 (2)	0.030 (3)	−0.004 (2)	−0.019 (3)	0.0046 (19)
C10B	0.080 (6)	0.017 (2)	0.025 (3)	0.012 (3)	−0.023 (3)	0.000 (2)

Geometric parameters (Å, º)

S1—O1	1.4657 (9)	C5B—H5B1	0.9900
S1—O2	1.4726 (8)	C5B—H5B2	0.9900
S1—O3	1.4784 (9)	N4—C6	1.3244 (16)
S1—O4	1.4801 (9)	N4—H41	0.886 (17)
N1—C1	1.3301 (16)	N4—H42	0.826 (18)
N1—H11	0.869 (18)	N5—C6	1.3331 (17)
N1—H12	0.890 (17)	N5—H51	0.857 (18)
N2—C1	1.3375 (15)	N5—H52	0.86 (2)
N2—H21	0.905 (18)	C6—N6B	1.335 (9)
N2—H22	0.88 (2)	C6—N6A	1.362 (5)
C1—N3B	1.253 (12)	N6A—C10A	1.471 (5)
C1—N3A	1.357 (3)	N6A—C7A	1.475 (5)
N3A—C5A	1.472 (3)	C7A—C8A	1.506 (4)
N3A—C2A	1.472 (3)	C7A—H7A1	0.9900
C2A—C3A	1.501 (3)	C7A—H7A2	0.9900
C2A—H2A1	0.9900	C8A—O6A	1.431 (5)
C2A—H2A2	0.9900	C8A—H8A1	0.9900
C3A—O5A	1.433 (3)	C8A—H8A2	0.9900
C3A—H3A1	0.9900	O6A—C9A	1.432 (5)

C3A—H3A2	0.9900	C9A—C10A	1.513 (4)
O5A—C4A	1.432 (4)	C9A—H9A1	0.9900
C4A—C5A	1.502 (3)	C9A—H9A2	0.9900
C4A—H4A1	0.9900	C10A—H10A	0.9900
C4A—H4A2	0.9900	C10A—H10B	0.9900
C5A—H5A1	0.9900	N6B—C10B	1.466 (9)
C5A—H5A2	0.9900	N6B—C7B	1.469 (9)
N3B—C5B	1.485 (12)	C7B—C8B	1.492 (7)
N3B—C2B	1.486 (12)	C7B—H7B1	0.9900
C2B—C3B	1.508 (11)	C7B—H7B2	0.9900
C2B—H2B1	0.9900	C8B—O6B	1.410 (9)
C2B—H2B2	0.9900	C8B—H8B1	0.9900
C3B—O5B	1.462 (15)	C8B—H8B2	0.9900
C3B—H3B1	0.9900	O6B—C9B	1.400 (11)
C3B—H3B2	0.9900	C9B—C10B	1.488 (8)
O5B—C4B	1.454 (15)	C9B—H9B1	0.9900
C4B—C5B	1.506 (11)	C9B—H9B2	0.9900
C4B—H4B1	0.9900	C10B—H10C	0.9900
C4B—H4B2	0.9900	C10B—H10D	0.9900

O1—S1—O2	110.82 (5)	N3B—C5B—H5B2	109.6
O1—S1—O3	109.38 (5)	C4B—C5B—H5B2	109.6
O2—S1—O3	109.43 (5)	H5B1—C5B—H5B2	108.1
O1—S1—O4	109.44 (6)	C6—N4—H41	118.0 (11)
O2—S1—O4	109.21 (5)	C6—N4—H42	120.5 (12)
O3—S1—O4	108.52 (6)	H41—N4—H42	119.9 (16)
C1—N1—H11	120.7 (11)	C6—N5—H51	114.4 (12)
C1—N1—H12	116.5 (10)	C6—N5—H52	120.5 (13)
H11—N1—H12	119.2 (15)	H51—N5—H52	119.8 (18)
C1—N2—H21	116.2 (11)	N4—C6—N5	117.82 (12)
C1—N2—H22	121.7 (12)	N4—C6—N6B	122.2 (4)
H21—N2—H22	121.0 (16)	N5—C6—N6B	118.9 (4)
N3B—C1—N1	122.3 (5)	N4—C6—N6A	119.8 (2)
N3B—C1—N2	117.3 (5)	N5—C6—N6A	121.7 (2)
N1—C1—N2	117.14 (12)	C6—N6A—C10A	124.4 (4)
N1—C1—N3A	120.73 (14)	C6—N6A—C7A	124.2 (3)
N2—C1—N3A	121.97 (14)	C10A—N6A—C7A	111.0 (4)
C1—N3A—C5A	123.31 (18)	N6A—C7A—C8A	110.1 (2)
C1—N3A—C2A	123.81 (18)	N6A—C7A—H7A1	109.6
C5A—N3A—C2A	111.00 (18)	C8A—C7A—H7A1	109.6
N3A—C2A—C3A	109.32 (16)	N6A—C7A—H7A2	109.6
N3A—C2A—H2A1	109.8	C8A—C7A—H7A2	109.6
C3A—C2A—H2A1	109.8	H7A1—C7A—H7A2	108.1
N3A—C2A—H2A2	109.8	O6A—C8A—C7A	111.8 (3)
C3A—C2A—H2A2	109.8	O6A—C8A—H8A1	109.3
H2A1—C2A—H2A2	108.3	C7A—C8A—H8A1	109.3
O5A—C3A—C2A	111.0 (2)	O6A—C8A—H8A2	109.3
O5A—C3A—H3A1	109.4	C7A—C8A—H8A2	109.3

C2A—C3A—H3A1	109.4	H8A1—C8A—H8A2	107.9
O5A—C3A—H3A2	109.4	C8A—O6A—C9A	109.5 (3)
C2A—C3A—H3A2	109.4	O6A—C9A—C10A	111.6 (3)
H3A1—C3A—H3A2	108.0	O6A—C9A—H9A1	109.3
C4A—O5A—C3A	110.4 (2)	C10A—C9A—H9A1	109.3
O5A—C4A—C5A	110.9 (2)	O6A—C9A—H9A2	109.3
O5A—C4A—H4A1	109.5	C10A—C9A—H9A2	109.3
C5A—C4A—H4A1	109.5	H9A1—C9A—H9A2	108.0
O5A—C4A—H4A2	109.5	N6A—C10A—C9A	109.2 (3)
C5A—C4A—H4A2	109.5	N6A—C10A—H10A	109.8
H4A1—C4A—H4A2	108.0	C9A—C10A—H10A	109.8
N3A—C5A—C4A	108.65 (17)	N6A—C10A—H10B	109.8
N3A—C5A—H5A1	110.0	C9A—C10A—H10B	109.8
C4A—C5A—H5A1	110.0	H10A—C10A—H10B	108.3
N3A—C5A—H5A2	110.0	C6—N6B—C10B	120.1 (7)
C4A—C5A—H5A2	110.0	C6—N6B—C7B	123.6 (7)
H5A1—C5A—H5A2	108.3	C10B—N6B—C7B	114.6 (7)
C1—N3B—C5B	123.1 (8)	N6B—C7B—C8B	110.1 (5)
C1—N3B—C2B	122.2 (9)	N6B—C7B—H7B1	109.6
C5B—N3B—C2B	112.5 (9)	C8B—C7B—H7B1	109.6
N3B—C2B—C3B	110.6 (7)	N6B—C7B—H7B2	109.6
N3B—C2B—H2B1	109.5	C8B—C7B—H7B2	109.6
C3B—C2B—H2B1	109.5	H7B1—C7B—H7B2	108.2
N3B—C2B—H2B2	109.5	O6B—C8B—C7B	112.8 (5)
C3B—C2B—H2B2	109.5	O6B—C8B—H8B1	109.0
H2B1—C2B—H2B2	108.1	C7B—C8B—H8B1	109.0
O5B—C3B—C2B	106.5 (11)	O6B—C8B—H8B2	109.0
O5B—C3B—H3B1	110.4	C7B—C8B—H8B2	109.0
C2B—C3B—H3B1	110.4	H8B1—C8B—H8B2	107.8
O5B—C3B—H3B2	110.4	C9B—O6B—C8B	111.4 (8)
C2B—C3B—H3B2	110.4	O6B—C9B—C10B	113.8 (7)
H3B1—C3B—H3B2	108.6	O6B—C9B—H9B1	108.8
C4B—O5B—C3B	105.4 (11)	C10B—C9B—H9B1	108.8
O5B—C4B—C5B	107.3 (11)	O6B—C9B—H9B2	108.8
O5B—C4B—H4B1	110.3	C10B—C9B—H9B2	108.8
C5B—C4B—H4B1	110.3	H9B1—C9B—H9B2	107.7
O5B—C4B—H4B2	110.3	N6B—C10B—C9B	111.0 (6)
C5B—C4B—H4B2	110.3	N6B—C10B—H10C	109.4
H4B1—C4B—H4B2	108.5	C9B—C10B—H10C	109.4
N3B—C5B—C4B	110.3 (8)	N6B—C10B—H10D	109.4
N3B—C5B—H5B1	109.6	C9B—C10B—H10D	109.4
C4B—C5B—H5B1	109.6	H10C—C10B—H10D	108.0

N1—C1—N3A—C5A	178.66 (18)	N4—C6—N6A—C10A	−10.6 (4)
N2—C1—N3A—C5A	−6.2 (3)	N5—C6—N6A—C10A	179.1 (3)
N1—C1—N3A—C2A	15.7 (3)	N4—C6—N6A—C7A	177.6 (2)
N2—C1—N3A—C2A	−169.13 (17)	N5—C6—N6A—C7A	7.3 (4)
C1—N3A—C2A—C3A	108.2 (2)	C6—N6A—C7A—C8A	118.2 (4)

C5A—N3A—C2A—C3A	−56.6 (2)	C10A—N6A—C7A—C8A	−54.5 (4)
N3A—C2A—C3A—O5A	57.1 (3)	N6A—C7A—C8A—O6A	56.5 (4)
C2A—C3A—O5A—C4A	−59.2 (4)	C7A—C8A—O6A—C9A	−58.9 (4)
C3A—O5A—C4A—C5A	60.1 (4)	C8A—O6A—C9A—C10A	60.0 (4)
C1—N3A—C5A—C4A	−107.7 (2)	C6—N6A—C10A—C9A	−117.7 (4)
C2A—N3A—C5A—C4A	57.2 (2)	C7A—N6A—C10A—C9A	55.0 (4)
O5A—C4A—C5A—N3A	−58.6 (3)	O6A—C9A—C10A—N6A	−58.2 (4)
N1—C1—N3B—C5B	165.8 (7)	N4—C6—N6B—C10B	1.6 (8)
N2—C1—N3B—C5B	6.7 (11)	N5—C6—N6B—C10B	169.1 (6)
N1—C1—N3B—C2B	4.2 (11)	N4—C6—N6B—C7B	−162.7 (5)
N2—C1—N3B—C2B	−154.8 (7)	N5—C6—N6B—C7B	4.8 (8)
C1—N3B—C2B—C3B	−147.3 (9)	C6—N6B—C7B—C8B	−147.4 (6)
C5B—N3B—C2B—C3B	49.3 (11)	C10B—N6B—C7B—C8B	47.6 (8)
N3B—C2B—C3B—O5B	−60.2 (12)	N6B—C7B—C8B—O6B	−53.2 (8)
C2B—C3B—O5B—C4B	71.8 (16)	C7B—C8B—O6B—C9B	58.5 (9)
C3B—O5B—C4B—C5B	−72.0 (17)	C8B—O6B—C9B—C10B	−56.9 (11)
C1—N3B—C5B—C4B	148.0 (9)	C6—N6B—C10B—C9B	148.3 (8)
C2B—N3B—C5B—C4B	−48.9 (11)	C7B—N6B—C10B—C9B	−46.1 (10)
O5B—C4B—C5B—N3B	60.0 (13)	O6B—C9B—C10B—N6B	50.2 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H11···O2^{i}	0.87 (2)	2.07 (2)	2.866 (2)	153 (2)
N1—H12···O2^{ii}	0.89 (2)	1.95 (2)	2.820 (2)	164 (2)
N2—H21···O4^{ii}	0.91 (2)	1.99 (2)	2.881 (2)	174 (2)
N2—H22···O3^{iii}	0.88 (2)	2.56 (2)	3.309 (2)	144 (2)
N2—H22···O4^{iii}	0.88 (2)	2.21 (2)	2.956 (2)	143 (2)
N4—H41···O1^{iii}	0.89 (2)	1.89 (2)	2.779 (2)	170 (2)
N4—H42···O3^{iv}	0.83 (2)	2.06 (2)	2.799 (2)	150 (2)
N5—H51···O3^{iii}	0.86 (2)	2.04 (2)	2.894 (2)	168 (2)
N5—H52···O1	0.86 (2)	2.48 (2)	3.113 (2)	131 (2)
N5—H52···O4	0.86 (2)	2.28 (2)	3.121 (2)	159 (2)

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