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Crystal structure of bis(1,4-diazabicyclo[2.2.2]-octan-1-ium) thiosulfate dihydrate

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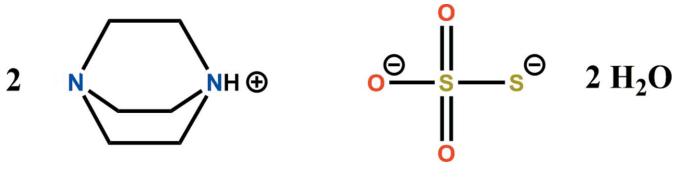
The crystal structure of the hydrated title salt, $2C_6H_{13}N_2^+\cdot S_2O_3^{2-}\cdot 2H_2O$, contains a centrosymmetric cyclic motif of eight hydrogen-bonded molecular subunits. Two $DABCOH^+$ cations (DABCO = 1,4-diazabicyclo[2.2.2]octane) are linked to two water molecules and two thiosulfate anions *via* O—H···N and O—H···O hydrogen bonds, respectively. Two other water molecules close the cyclic motif through O—H···O contacts to the first two water molecules and to the two thiosulfate anions. A second pair of $DABCOH^+$ cations is N—H···O hydrogen bonded to the two anions and is pendant to the ring. Adjacent cyclic motifs are bridged into a block-like arrangement extending along [100] through O—H···O interactions involving the second pair of water molecules and neighbouring thiosulfate anions.

1. Chemical context

The title thiosulfate was isolated accidentally when thioacetamide was mixed in ethanol with DABCO (1,4-diazabicyclo[2.2.2]octane), leading to the formation of the thiosulfate anion *in situ*.

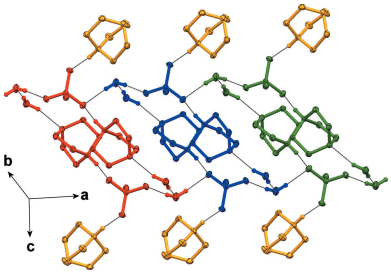


2. Structural commentary

The asymmetric unit (Fig. 1) consists of one thiosulfate anion, two monoprotonated $DABCOH^+$ cations and two water molecules. The thiosulfate anion exhibits approximate C_{3v} symmetry. However, in the crystal it has C_1 symmetry with S—O distances in the range 1.4688 (8) to 1.4898 (8) Å and an S—S bond length of 2.0047 (4) Å, and O—S—O and S—S—O angles ranging from 107.47 (4) to 110.48 (5)°. In both $DABCOH^+$ cations, the three N—C bonds involving the protonated N atom are elongated [mean value 1.499 (2) Å] compared to the three N—C bonds involving the non-protonated N atoms [mean value 1.472 (4) Å].

3. Supramolecular features

The thiosulfate anion is linked *via* charge-assisted N—H···O hydrogen bonds to two $DABCOH^+$ cations. The third oxygen atom (O2) of the anion acts as a hydrogen-bond acceptor for



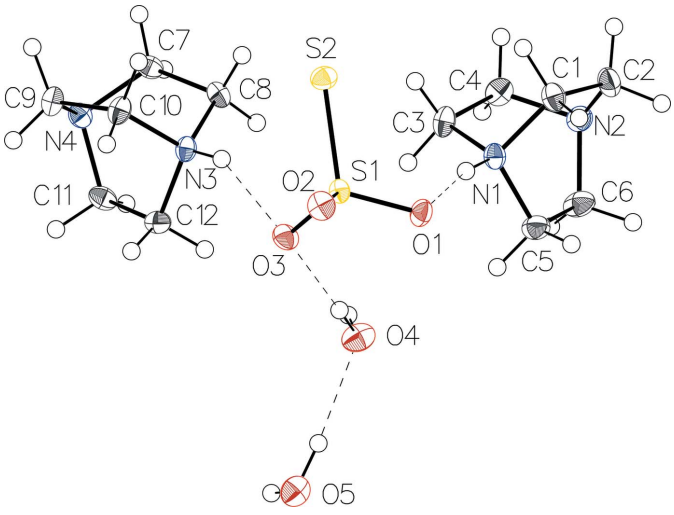


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

one of the water molecules (O4). The second hydrogen bond involving this water molecule is directed towards a symmetry-related thiosulfate anion. The second water molecule (O5) is the donor of one O—H···O hydrogen bond to the other water molecule and of one N—H···O hydrogen bond to one of the $DABCOH^+$ cations. Numerical details of the hydrogen-bonding interactions are given in Table 1. This arrangement leads to the formation of a centrosymmetric cyclic motif consisting of eight hydrogen-bonded molecules with two pendant $DABCOH^+$ cations (Fig. 2). Adjacent cyclic motifs are bridged through O4—H44···O3 contacts into supramolecular blocks running along [100] (Fig. 3).

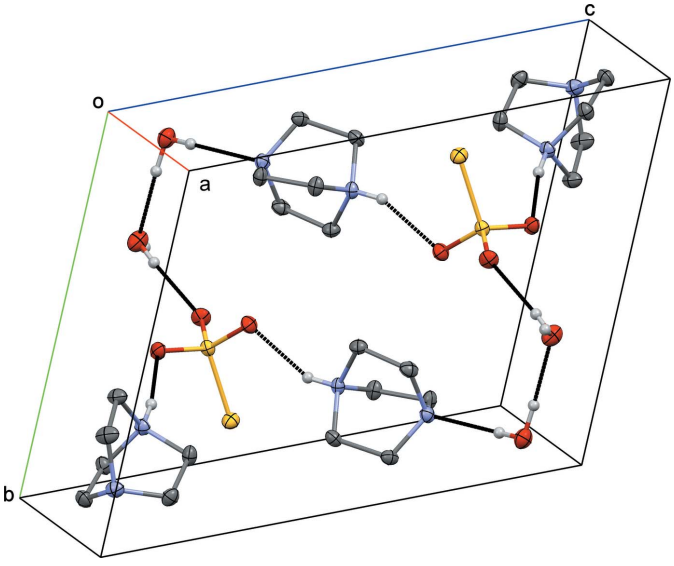


Figure 2
View of the content of one unit cell, showing the hydrogen-bonded macrocycle made up from the asymmetric unit and its inversion-symmetry-related counterpart. H atoms not involved in hydrogen bonding (black dotted lines) are omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.887 (17)	1.861 (17)	2.7380 (12)	169.6 (15)
N3—H3···O3	0.850 (18)	2.030 (17)	2.8003 (12)	150.4 (15)
O4—H4*C*···O2^{i}	0.79 (2)	2.02 (2)	2.8121 (13)	173.4 (18)
O4—H4*D*···O3	0.81 (2)	2.04 (2)	2.8511 (13)	175.1 (18)
O5—H5*C*···N4^{ii}	0.85 (2)	2.10 (2)	2.9273 (13)	163.9 (19)
O5—H5*D*···O4	0.91 (2)	1.94 (2)	2.8449 (14)	173.2 (19)

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

4. Database survey

A search in the Cambridge Structural Database (CSD Version 5.36 with three updates; Groom & Allen, 2014) for salts with isolated thiosulfate anions returned 25 records with ten of them featuring a metal complex for the cationic part. Entries with simple protonated amine functionalities include structures with *tert*-butylammonium (Okuniewski *et al.*, 2013) and its hydrate (Dabrowska & Chojnacki, 2014), cyclohexylammonium (Dabrowska & Chojnacki, 2014), tetramethylammonium tetrahydrate (Yang & Ng, 2011), tetraethylammonium dihydrate (Leyten *et al.*, 1988), isopropylammonium (Okuniewski *et al.*, 2013), piperazinium (Srinivasan *et al.*, 2011) and adamantanaminium (Jiang *et al.*, 1998). The thiosulfate anion has also been encapsulated in protonated azacryptands ligands (Maubert *et al.*, 2001; Nelson *et al.*, 2004).

5. Synthesis and crystallization

Crystals suitable for a single-crystal X-ray diffraction study were isolated from a clear ethanolic solution of thioacetamide and DABCO in an equimolar ratio.

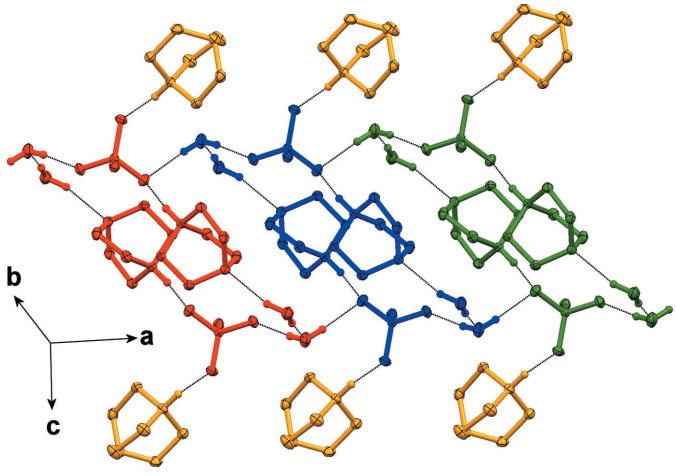


Figure 3
View of three successive hydrogen-bonded cycles displayed in red, blue and green. Pendant $DABCOH^+$ cations are shown in orange. H atoms not involved in hydrogen bonding (black dotted lines) are omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$2C_6H_{13}N_2^+\cdot S_2O_3^{2-}\cdot 2H_2O$
M_r	374.52
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	6.5063 (2), 10.5966 (3), 13.2066 (4)
α, β, γ (°)	105.951 (1), 92.065 (1), 96.550 (1)
V (Å^3)	867.59 (5)
Z	2
Radiation type	Ga $K\alpha$, λ = 1.34139 Å
μ (mm^{-1})	1.98
Crystal size (mm)	0.51 × 0.18 × 0.06
Data collection	
Diffractometer	Bruker Venture Metaljet
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.170, 0.311
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24479, 3965, 3865
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.650
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.079, 1.07
No. of reflections	3965
No. of parameters	328
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.31

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were located from Fourier difference maps and freely refined.

Acknowledgements

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Crystal structure of bis(1,4-diazabicyclo[2.2.2]octan-1-ium) thiosulfate dihydrate

Gorgui Awa Seck, Aboubacary Sene, Libasse Diop and Thierry Maris

Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

Bis(1,4-diazabicyclo[2.2.2]octan-1-ium) thiosulfate dihydrate

Crystal data

$2C_6H_{13}N_2^+\cdot S_2O_3^{2-}\cdot 2H_2O$
M_r = 374.52
Triclinic, $P\overline{1}$
a = 6.5063 (2) Å
b = 10.5966 (3) Å
c = 13.2066 (4) Å
α = 105.951 (1)°
β = 92.065 (1)°
γ = 96.550 (1)°
V = 867.59 (5) Å^3
Z = 2
$F(000)$ = 404
D_x = 1.434 Mg m^{-3}
Ga $K\alpha$ radiation, λ = 1.34139 Å
Cell parameters from 9855 reflections
θ = 3.0–60.7°
μ = 1.98 mm^{-1}
T = 100 K
Platelet, clear light colourless
0.51 × 0.18 × 0.06 mm

Data collection

Bruker Venture Metaljet diffractometer
Radiation source: Metal Jet, Gallium Liquid Metal Jet Source
Helios MX Mirror Optics monochromator
Detector resolution: 10.24 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.170, T_{max} = 0.311
24479 measured reflections
3965 independent reflections
3865 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 60.6°, θ_{min} = 3.0°
h = −8→8
k = −13→13
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.079
S = 1.07
3965 reflections
328 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.2876P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

Experimental. X-ray crystallographic data for I were collected from a single-crystal sample, which was mounted on a loop fiber. Data were collected using a Bruker Venture diffractometer equipped with a Photon 100 CMOS Detector, a Helios MX optics and a Kappa goniometer. The crystal-to-detector distance was 4.0 cm, and the data collection was carried out in 1024 *x* 1024 pixel mode.

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.37120 (14)	0.25575 (9)	0.89793 (7)	0.01709 (19)
H1	0.474 (3)	0.2918 (16)	0.8688 (13)	0.027 (4)*
N2	0.07772 (15)	0.16056 (10)	0.98667 (8)	0.0204 (2)
C1	0.44755 (18)	0.15612 (12)	0.94669 (9)	0.0204 (2)
H1A	0.477 (2)	0.0796 (16)	0.8860 (13)	0.026 (4)*
H1B	0.572 (3)	0.1986 (16)	0.9896 (13)	0.026 (4)*
C2	0.27163 (19)	0.11153 (12)	1.00935 (9)	0.0227 (2)
H2A	0.308 (3)	0.1446 (18)	1.0861 (15)	0.036 (4)*
H2B	0.250 (3)	0.0161 (17)	0.9902 (13)	0.029 (4)*
C3	0.19715 (18)	0.18911 (13)	0.81671 (9)	0.0211 (2)
H3A	0.254 (2)	0.1287 (16)	0.7618 (13)	0.025 (4)*
H3B	0.149 (3)	0.2574 (17)	0.7886 (13)	0.032 (4)*
C4	0.03175 (18)	0.12017 (12)	0.87190 (9)	0.0215 (2)
H4A	−0.102 (3)	0.1438 (16)	0.8578 (13)	0.028 (4)*
H4B	0.027 (2)	0.0273 (17)	0.8492 (13)	0.028 (4)*
C5	0.29855 (19)	0.36527 (12)	0.98198 (10)	0.0226 (2)
H5A	0.268 (3)	0.4332 (17)	0.9503 (13)	0.029 (4)*
H5B	0.417 (3)	0.3988 (17)	1.0355 (13)	0.030 (4)*
C6	0.1044 (2)	0.30577 (12)	1.02479 (11)	0.0263 (3)
H6A	0.118 (3)	0.3316 (18)	1.1015 (15)	0.037 (4)*
H6B	−0.020 (3)	0.3362 (17)	0.9978 (13)	0.032 (4)*
N3	0.32970 (14)	0.27267 (9)	0.50138 (7)	0.01556 (18)
H3	0.423 (3)	0.2931 (16)	0.5520 (13)	0.028 (4)*
N4	0.05427 (14)	0.20524 (9)	0.34935 (7)	0.01749 (19)
C7	0.02619 (17)	0.11478 (11)	0.41613 (9)	0.0180 (2)
H7A	0.050 (2)	0.0289 (16)	0.3785 (12)	0.022 (4)*
H7B	−0.117 (2)	0.1056 (15)	0.4338 (11)	0.020 (3)*
C8	0.17326 (17)	0.16570 (11)	0.51711 (9)	0.0182 (2)
H8A	0.107 (2)	0.2033 (15)	0.5776 (12)	0.020 (3)*
H8B	0.248 (2)	0.1029 (16)	0.5297 (12)	0.024 (4)*
C9	0.26331 (18)	0.20134 (12)	0.30931 (9)	0.0197 (2)
H9A	0.265 (2)	0.1127 (16)	0.2591 (12)	0.024 (4)*
H9B	0.287 (2)	0.2726 (16)	0.2771 (13)	0.026 (4)*

C10	0.43012 (17)	0.22308 (12)	0.40030 (8)	0.0185 (2)
H10A	0.482 (2)	0.1435 (16)	0.4030 (12)	0.024 (4)*
H10B	0.540 (2)	0.2897 (15)	0.3999 (12)	0.022 (4)*
C11	0.03966 (18)	0.34059 (11)	0.41524 (9)	0.0202 (2)
H11A	−0.091 (2)	0.3368 (14)	0.4464 (11)	0.019 (3)*
H11B	0.039 (2)	0.3977 (16)	0.3696 (13)	0.026 (4)*
C12	0.22526 (17)	0.39014 (11)	0.49767 (9)	0.0188 (2)
H12A	0.328 (3)	0.4497 (16)	0.4780 (13)	0.027 (4)*
H12B	0.184 (2)	0.4274 (15)	0.5693 (12)	0.023 (4)*
S1	0.73999 (4)	0.36034 (2)	0.71916 (2)	0.01430 (8)
S2	0.66847 (4)	0.16633 (3)	0.64755 (2)	0.01940 (9)
O1	0.70661 (12)	0.38533 (8)	0.83256 (6)	0.01974 (17)
O2	0.95658 (12)	0.40399 (8)	0.70450 (6)	0.02108 (18)
O3	0.59842 (12)	0.43146 (8)	0.67012 (6)	0.02058 (17)
O4	0.31661 (15)	0.56983 (9)	0.80581 (8)	0.02666 (19)
H4C	0.211 (3)	0.5246 (19)	0.7817 (15)	0.035 (5)*
H4D	0.402 (3)	0.5326 (18)	0.7700 (15)	0.036 (5)*
O5	0.31448 (14)	0.83414 (9)	0.79500 (7)	0.02522 (19)
H5C	0.196 (3)	0.830 (2)	0.7642 (16)	0.048 (5)*
H5D	0.308 (3)	0.751 (2)	0.8025 (16)	0.049 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0159 (4)	0.0205 (4)	0.0158 (4)	0.0012 (3)	0.0031 (3)	0.0069 (3)
N2	0.0205 (5)	0.0199 (5)	0.0201 (5)	0.0000 (4)	0.0066 (4)	0.0045 (4)
C1	0.0186 (5)	0.0246 (6)	0.0204 (5)	0.0038 (4)	0.0005 (4)	0.0097 (4)
C2	0.0251 (6)	0.0253 (6)	0.0199 (5)	−0.0001 (5)	0.0018 (4)	0.0115 (4)
C3	0.0180 (5)	0.0295 (6)	0.0151 (5)	0.0022 (4)	−0.0008 (4)	0.0059 (4)
C4	0.0161 (5)	0.0236 (6)	0.0226 (5)	−0.0003 (4)	−0.0005 (4)	0.0042 (4)
C5	0.0256 (6)	0.0174 (5)	0.0236 (5)	0.0005 (4)	0.0064 (5)	0.0040 (4)
C6	0.0272 (6)	0.0209 (6)	0.0284 (6)	0.0019 (5)	0.0132 (5)	0.0020 (5)
N3	0.0135 (4)	0.0200 (4)	0.0134 (4)	0.0025 (3)	0.0005 (3)	0.0049 (3)
N4	0.0154 (4)	0.0190 (4)	0.0191 (4)	0.0021 (3)	−0.0008 (3)	0.0072 (3)
C7	0.0156 (5)	0.0160 (5)	0.0230 (5)	0.0014 (4)	0.0014 (4)	0.0069 (4)
C8	0.0175 (5)	0.0201 (5)	0.0201 (5)	0.0033 (4)	0.0031 (4)	0.0104 (4)
C9	0.0178 (5)	0.0263 (6)	0.0143 (5)	0.0013 (4)	0.0009 (4)	0.0052 (4)
C10	0.0138 (5)	0.0256 (5)	0.0154 (5)	0.0041 (4)	0.0030 (4)	0.0039 (4)
C11	0.0178 (5)	0.0181 (5)	0.0269 (6)	0.0047 (4)	0.0001 (4)	0.0091 (4)
C12	0.0194 (5)	0.0156 (5)	0.0211 (5)	0.0029 (4)	0.0029 (4)	0.0043 (4)
S1	0.01214 (13)	0.01792 (14)	0.01419 (13)	0.00319 (9)	0.00261 (9)	0.00605 (9)
S2	0.01877 (14)	0.01794 (14)	0.02064 (14)	0.00461 (10)	0.00074 (10)	0.00317 (10)
O1	0.0211 (4)	0.0228 (4)	0.0144 (4)	0.0000 (3)	0.0044 (3)	0.0044 (3)
O2	0.0139 (4)	0.0256 (4)	0.0247 (4)	0.0016 (3)	0.0051 (3)	0.0085 (3)
O3	0.0199 (4)	0.0209 (4)	0.0228 (4)	0.0063 (3)	−0.0010 (3)	0.0081 (3)
O4	0.0197 (4)	0.0269 (5)	0.0302 (5)	0.0071 (4)	−0.0005 (4)	0.0015 (4)
O5	0.0196 (4)	0.0290 (5)	0.0251 (4)	0.0034 (3)	−0.0016 (3)	0.0047 (3)

Geometric parameters (Å, º)

N1—H1	0.887 (17)	N4—C7	1.4720 (14)
N1—C1	1.4988 (14)	N4—C9	1.4778 (14)
N1—C3	1.4984 (14)	N4—C11	1.4746 (14)
N1—C5	1.5025 (14)	C7—H7A	0.942 (16)
N2—C2	1.4694 (16)	C7—H7B	0.967 (15)
N2—C4	1.4668 (15)	C7—C8	1.5428 (15)
N2—C6	1.4691 (15)	C8—H8A	0.933 (15)
C1—H1A	1.013 (16)	C8—H8B	0.916 (16)
C1—H1B	0.959 (17)	C9—H9A	0.991 (16)
C1—C2	1.5421 (16)	C9—H9B	0.963 (17)
C2—H2A	0.988 (18)	C9—C10	1.5401 (15)
C2—H2B	0.964 (17)	C10—H10A	0.952 (16)
C3—H3A	0.942 (16)	C10—H10B	0.947 (16)
C3—H3B	0.975 (18)	C11—H11A	0.960 (15)
C3—C4	1.5469 (16)	C11—H11B	0.964 (17)
C4—H4A	0.960 (17)	C11—C12	1.5380 (16)
C4—H4B	0.943 (17)	C12—H12A	0.958 (17)
C5—H5A	0.962 (17)	C12—H12B	0.979 (16)
C5—H5B	0.992 (17)	S1—S2	2.0047 (4)
C5—C6	1.5424 (16)	S1—O1	1.4761 (8)
C6—H6A	0.972 (18)	S1—O2	1.4688 (8)
C6—H6B	0.993 (17)	S1—O3	1.4898 (8)
N3—H3	0.850 (18)	O4—H4C	0.79 (2)
N3—C8	1.4985 (14)	O4—H4D	0.81 (2)
N3—C10	1.4969 (13)	O5—H5C	0.85 (2)
N3—C12	1.4958 (14)	O5—H5D	0.91 (2)
C1—N1—H1	109.6 (10)	C12—N3—C8	109.71 (8)
C1—N1—C5	109.73 (9)	C12—N3—C10	109.52 (8)
C3—N1—H1	110.5 (11)	C7—N4—C9	108.87 (9)
C3—N1—C1	109.37 (9)	C7—N4—C11	108.26 (9)
C3—N1—C5	110.23 (9)	C11—N4—C9	108.68 (9)
C5—N1—H1	107.4 (11)	N4—C7—H7A	110.5 (9)
C4—N2—C2	108.23 (9)	N4—C7—H7B	109.8 (9)
C4—N2—C6	108.88 (10)	N4—C7—C8	110.60 (9)
C6—N2—C2	109.51 (10)	H7A—C7—H7B	105.1 (13)
N1—C1—H1A	106.3 (9)	C8—C7—H7A	110.1 (9)
N1—C1—H1B	106.9 (10)	C8—C7—H7B	110.6 (9)
N1—C1—C2	107.31 (9)	N3—C8—C7	107.51 (8)
H1A—C1—H1B	111.4 (13)	N3—C8—H8A	106.7 (9)
C2—C1—H1A	110.7 (9)	N3—C8—H8B	105.6 (10)
C2—C1—H1B	113.7 (10)	C7—C8—H8A	113.8 (9)
N2—C2—C1	111.18 (9)	C7—C8—H8B	113.4 (10)
N2—C2—H2A	108.2 (10)	H8A—C8—H8B	109.2 (13)
N2—C2—H2B	109.3 (10)	N4—C9—H9A	107.4 (9)
C1—C2—H2A	111.0 (11)	N4—C9—H9B	106.9 (9)

C1—C2—H2B	108.9 (10)	N4—C9—C10	110.83 (9)
H2A—C2—H2B	108.2 (14)	H9A—C9—H9B	113.4 (13)
N1—C3—H3A	107.1 (10)	C10—C9—H9A	109.4 (9)
N1—C3—H3B	106.8 (10)	C10—C9—H9B	108.9 (10)
N1—C3—C4	107.21 (9)	N3—C10—C9	107.32 (9)
H3A—C3—H3B	108.1 (14)	N3—C10—H10A	106.7 (9)
C4—C3—H3A	112.5 (10)	N3—C10—H10B	105.8 (9)
C4—C3—H3B	114.6 (10)	C9—C10—H10A	112.9 (9)
N2—C4—C3	111.11 (9)	C9—C10—H10B	112.7 (9)
N2—C4—H4A	107.9 (10)	H10A—C10—H10B	111.0 (13)
N2—C4—H4B	106.8 (10)	N4—C11—H11A	106.2 (9)
C3—C4—H4A	110.0 (10)	N4—C11—H11B	108.1 (10)
C3—C4—H4B	111.5 (10)	N4—C11—C12	110.79 (9)
H4A—C4—H4B	109.5 (14)	H11A—C11—H11B	109.1 (13)
N1—C5—H5A	108.0 (10)	C12—C11—H11A	113.0 (9)
N1—C5—H5B	105.4 (10)	C12—C11—H11B	109.4 (10)
N1—C5—C6	107.54 (9)	N3—C12—C11	107.39 (9)
H5A—C5—H5B	110.3 (14)	N3—C12—H12A	105.8 (10)
C6—C5—H5A	111.7 (10)	N3—C12—H12B	105.9 (9)
C6—C5—H5B	113.5 (10)	C11—C12—H12A	112.4 (10)
N2—C6—C5	110.75 (9)	C11—C12—H12B	113.0 (9)
N2—C6—H6A	108.4 (11)	H12A—C12—H12B	111.7 (13)
N2—C6—H6B	107.4 (10)	O1—S1—S2	109.01 (3)
C5—C6—H6A	109.9 (11)	O1—S1—O3	109.73 (5)
C5—C6—H6B	108.8 (10)	O2—S1—S2	110.12 (4)
H6A—C6—H6B	111.6 (14)	O2—S1—O1	110.48 (5)
C8—N3—H3	108.5 (11)	O2—S1—O3	109.98 (5)
C10—N3—H3	108.3 (11)	O3—S1—S2	107.47 (4)
C10—N3—C8	109.88 (9)	H4C—O4—H4D	103.3 (18)
C12—N3—H3	110.8 (11)	H5C—O5—H5D	100.6 (18)

N1—C1—C2—N2	10.05 (13)	N4—C7—C8—N3	−13.10 (12)
N1—C3—C4—N2	10.16 (13)	N4—C9—C10—N3	−13.22 (13)
N1—C5—C6—N2	11.29 (14)	N4—C11—C12—N3	−14.10 (12)
C1—N1—C3—C4	55.01 (12)	C7—N4—C9—C10	−51.09 (12)
C1—N1—C5—C6	−66.86 (12)	C7—N4—C11—C12	67.80 (11)
C2—N2—C4—C3	−65.61 (12)	C8—N3—C10—C9	67.72 (11)
C2—N2—C6—C5	52.04 (13)	C8—N3—C12—C11	−52.05 (11)
C3—N1—C1—C2	−66.56 (11)	C9—N4—C7—C8	66.64 (11)
C3—N1—C5—C6	53.66 (12)	C9—N4—C11—C12	−50.30 (12)
C4—N2—C2—C1	53.54 (12)	C10—N3—C8—C7	−52.78 (11)
C4—N2—C6—C5	−66.11 (13)	C10—N3—C12—C11	68.63 (11)
C5—N1—C1—C2	54.47 (12)	C11—N4—C7—C8	−51.34 (11)
C5—N1—C3—C4	−65.72 (12)	C11—N4—C9—C10	66.62 (12)
C6—N2—C2—C1	−65.02 (12)	C12—N3—C8—C7	67.68 (11)
C6—N2—C4—C3	53.35 (12)	C12—N3—C10—C9	−52.85 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.887 (17)	1.861 (17)	2.7380 (12)	169.6 (15)
N3—H3···O3	0.850 (18)	2.030 (17)	2.8003 (12)	150.4 (15)
O4—H4*C*···O2^{i}	0.79 (2)	2.02 (2)	2.8121 (13)	173.4 (18)
O4—H4*D*···O3	0.81 (2)	2.04 (2)	2.8511 (13)	175.1 (18)
O5—H5*C*···N4^{ii}	0.85 (2)	2.10 (2)	2.9273 (13)	163.9 (19)
O5—H5*D*···O4	0.91 (2)	1.94 (2)	2.8449 (14)	173.2 (19)

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