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Propane-1,3-diaminium pyridine-2,5-dicarboxylate dimethyl sulfoxide monosolvate

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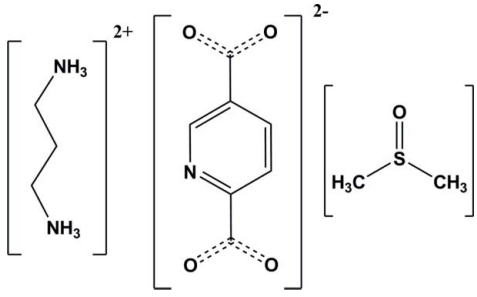
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.095; data-to-parameter ratio = 18.6.

In the crystal structure of the title solvated molecular salt, $C_3H_{12}N_2^{2+}\cdot C_7H_3NO_4^{2-}\cdot C_2H_6OS$, two amine groups of propane-1,3-diamine (pda) are protonated and two carboxylic acid groups of pyridine-2,5-dicarboxylic acid (2,5-pydcH_2) are deprotonated. The crystal packing features N—H···O hydrogen bonds and weak C—H···O intermolecular interactions.

Related literature

Pyridine-2,5-dicarboxylic acid (2,5-pydcH_2) can coordinate to metal centers (Pasdar *et al.*, 2011) or form hydrogen-bonded networks (Zeng *et al.*, 2005). For work by our group on the synthesis of proton-transfer compounds containing different proton donor and acceptor groups, see: Eshtiagh-Hosseini *et al.* (2010*a*,*b*); Aghabozorg *et al.* (2008, 2011).



Experimental

Crystal data

$C_3H_{12}N_2^{2+}\cdot C_7H_3NO_4^{2-}\cdot C_2H_6OS$ M_r = 319.39
Monoclinic, $P2_1/n$
a = 11.984 (2) Å
b = 10.346 (2) Å
c = 12.942 (3) Å
β = 111.63 (3)°
V = 1491.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 K
0.4 × 0.3 × 0.3 mm

Data collection

STOE IPDS 2T diffractometer
12249 measured reflections
4010 independent reflections
3380 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.095
S = 1.07
4010 reflections
216 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4^{i}	0.91 (2)	1.96 (2)	2.8260 (16)	157.8 (17)
N2—H2B···O3^{ii}	0.86 (2)	2.06 (2)	2.8461 (17)	151.0 (18)
N2—H2C···O2^{iii}	0.92 (2)	1.84 (2)	2.7385 (17)	164.4 (18)
N3—H3A···O1^{iv}	0.91 (2)	1.85 (2)	2.7369 (17)	161.6 (18)
N3—H3B···O3	0.890 (19)	2.073 (19)	2.8427 (16)	144.2 (16)
N3—H3C···O4^{v}	0.86 (2)	1.96 (2)	2.7925 (17)	164.2 (18)
C8—H8A···O5^{vi}	0.97	2.50	3.4614 (19)	170
C10—H10A···O5	0.97	2.53	3.4718 (19)	165
C11—H11B···O1^{ii}	0.96	2.46	3.424 (2)	178

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2074).

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supporting information

Acta Cryst. (2011). E**67**, o610 [doi:10.1107/S1600536811004545]

Propane-1,3-diaminium pyridine-2,5-dicarboxylate dimethyl sulfoxide monosolvate

Hossein Aghabozorg, Minoo Bayan, Masoud Mirzaei and Behrouz Notash

S1. Comment

Pyridine-2,5-dicarboxylic acid (2,5-pydcH_2) can coordinate to metal centers (Pasdar *et al.*, 2011) or form hydrogen-bonded networks (Zeng *et al.*, 2005). Our research group has been focused on synthesis of proton transfer compounds containing different proton donor and acceptor groups (Eshtiagh-Hosseini *et al.*, 2010*a*; Eshtiagh-Hosseini *et al.*, 2010*b*; Aghabozorg *et al.*, 2008, 2011).

We report here the synthesis and crystal structure of the title proton transfer compound, $[pdaH2]^{2+}.[2,5\text{-}pydc]^{2-}.(DMSO)$. The asymmetric unit contains deprotonated pyridine-2,5-dicarboxylic acid, diprotonated propane-1,3-diamine, and one DMSO solvent molecule (Fig. 1). Crystal packing is stabilized by N—H···O hydrogen bonds and weak C—H···O intermolecular interactions (Fig. 2 & Table 1).

S2. Experimental

Propane-1,3-diamine (0.07 g, 0.29 ml, 1 mmol) was added to a DMSO/H_2O solution of pyridine-2,5-dicarboxylic acid (0.17 g, 1 mmol) (13 ml) at room temperature. The suitable crystals for X-ray diffraction experiment were isolated by slow evaporation of the solvent after two months.

S3. Refinement

Nitrogen-bound H atoms were found in difference Fourier map and refined isotropically without restraint. Carbon-bound H atoms were positioned geometrically and refined as riding atoms with C—H distances of 0.93 Å (aromatic) and 0.97 Å (CH_2) and were refined with *U*iso(H) = 1.2 *Ueq*(C).

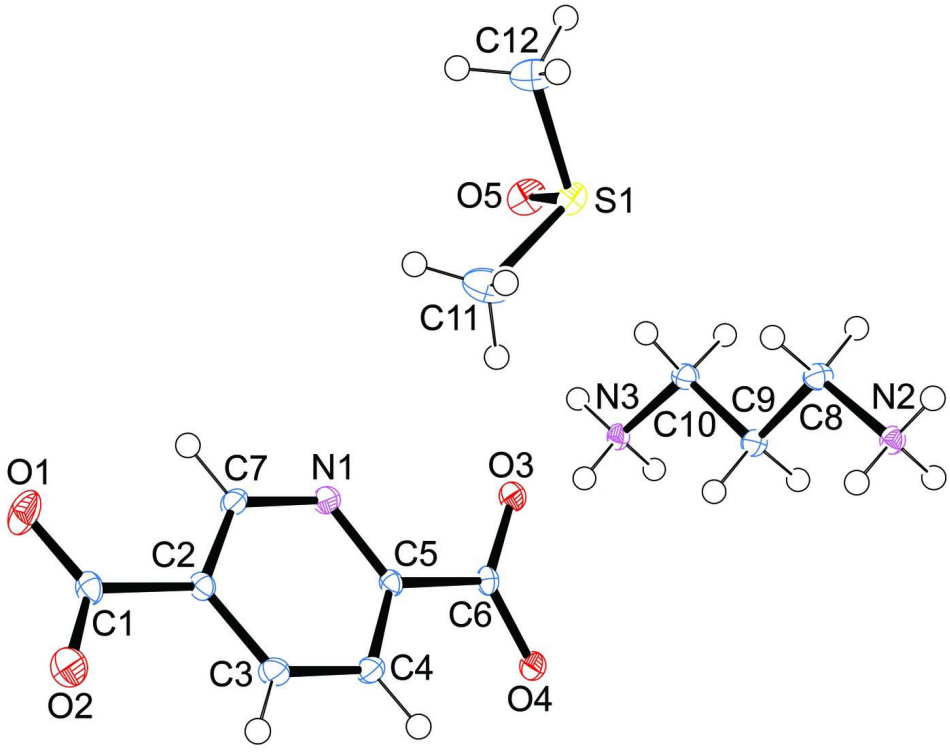

Figure 1

The molecular structure of title compound with displacement ellipsoids drawn at 50% probability level.

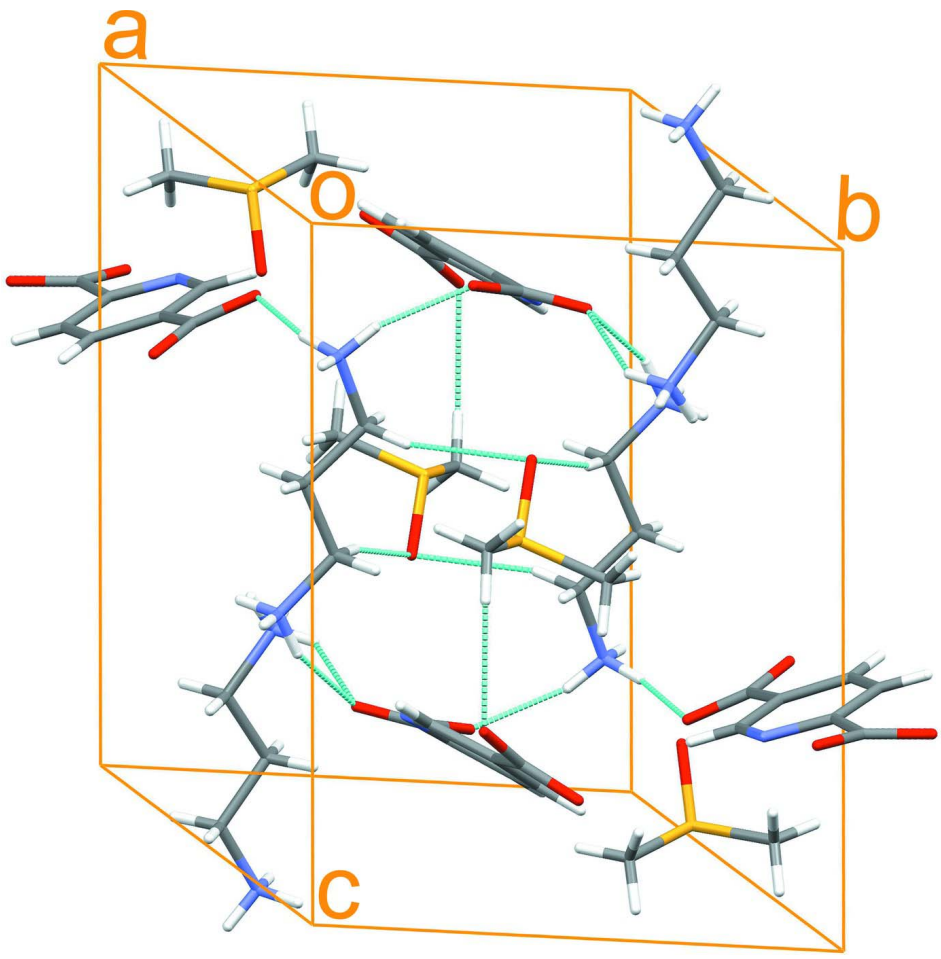

Figure 2

The packing diagram of the title compound, viewed down the *a* axis, showing N—H···O hydrogen bonds and weak C—H···O intermolecular interactions (dashed lines).

Propane-1,3-diaminium pyridine-2,5-dicarboxylate dimethyl sulfoxide monosolvate

Crystal data

$C_3H_{12}N_2^{2+}·C_7H_3NO_4^{2-}·C_2H_6OS$
M_r = 319.39
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 11.984 (2) Å
b = 10.346 (2) Å
c = 12.942 (3) Å
β = 111.63 (3)°
V = 1491.6 (6) Å^3
Z = 4
$F(000)$ = 680
D_x = 1.422 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4010 reflections
θ = 2.6–29.2°
μ = 0.24 mm^{-1}
T = 120 K
Block, colorless
0.4 × 0.3 × 0.3 mm

Data collection

STOE IPDS 2T diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.15 pixels mm^{-1}
rotation method scans
12249 measured reflections
4010 independent reflections

3380 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 29.2°$, $\theta_{min} = 2.6°$
$h = -16 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.095$
$S = 1.07$
4010 reflections
216 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
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2 + 0.6814P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ 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 $F^2 > \sigma(F^2)$ 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}
S1	0.25330 (3)	0.98812 (3)	0.01042 (3)	0.01677 (10)
O1	0.89632 (10)	0.63514 (12)	0.26504 (10)	0.0237 (2)
O2	0.87767 (10)	0.45057 (12)	0.17030 (11)	0.0271 (3)
O3	0.28902 (9)	0.63320 (10)	0.16349 (8)	0.0147 (2)
O4	0.28589 (9)	0.41856 (10)	0.13819 (8)	0.0145 (2)
O5	0.26031 (11)	1.00924 (11)	0.12737 (9)	0.0216 (2)
N1	0.52969 (11)	0.63725 (11)	0.20599 (10)	0.0136 (2)
N2	−0.08584 (11)	0.67039 (12)	−0.18378 (10)	0.0120 (2)
H2A	−0.1484 (18)	0.6233 (18)	−0.1797 (15)	0.018 (5)*
H2B	−0.1114 (17)	0.7160 (19)	−0.2439 (16)	0.019 (5)*
H2C	−0.0253 (19)	0.619 (2)	−0.1876 (16)	0.024 (5)*
N3	0.09589 (11)	0.71239 (12)	0.22640 (10)	0.0122 (2)
H3A	0.0361 (18)	0.6692 (19)	0.2400 (15)	0.020 (5)*
H3B	0.1526 (17)	0.6583 (18)	0.2240 (14)	0.013 (4)*
H3C	0.1289 (18)	0.768 (2)	0.2777 (16)	0.021 (5)*
C1	0.83737 (12)	0.54143 (14)	0.20910 (12)	0.0143 (3)
C2	0.70396 (12)	0.53745 (13)	0.18810 (11)	0.0116 (2)
C3	0.63536 (13)	0.43090 (13)	0.13750 (12)	0.0151 (3)
H3	0.6694	0.3631	0.1122	0.018*
C4	0.51512 (13)	0.42656 (13)	0.12501 (12)	0.0144 (3)
H4	0.4682	0.3550	0.0929	0.017*

C5	0.46609 (11)	0.53089 (13)	0.16125 (11)	0.0108 (2)
C6	0.33590 (12)	0.52826 (13)	0.15352 (10)	0.0111 (2)
C7	0.64639 (12)	0.63845 (13)	0.21994 (11)	0.0133 (3)
H7	0.6915	0.7109	0.2528	0.016*
C8	−0.03989 (13)	0.75898 (13)	−0.08677 (11)	0.0144 (3)
H8A	−0.1022	0.8202	−0.0896	0.017*
H8B	0.0278	0.8073	−0.0907	0.017*
C9	−0.00090 (12)	0.68569 (13)	0.02247 (11)	0.0134 (3)
H9A	0.0622	0.6249	0.0264	0.016*
H9B	−0.0682	0.6374	0.0272	0.016*
C10	0.04461 (13)	0.78090 (13)	0.11828 (11)	0.0139 (3)
H10A	0.1056	0.8357	0.1084	0.017*
H10B	−0.0210	0.8357	0.1183	0.017*
C11	0.37246 (17)	0.88029 (16)	0.02034 (14)	0.0256 (3)
H11A	0.4460	0.9123	0.0744	0.038*
H11B	0.3803	0.8739	−0.0507	0.038*
H11C	0.3553	0.7965	0.0427	0.038*
C12	0.31441 (17)	1.13018 (16)	−0.02737 (15)	0.0264 (3)
H12A	0.2650	1.2033	−0.0275	0.040*
H12B	0.3167	1.1191	−0.1002	0.040*
H12C	0.3943	1.1444	0.0253	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01593 (17)	0.01708 (17)	0.01747 (17)	−0.00372 (13)	0.00635 (13)	−0.00230 (13)
O1	0.0141 (5)	0.0294 (6)	0.0311 (6)	−0.0076 (5)	0.0125 (5)	−0.0101 (5)
O2	0.0145 (5)	0.0236 (6)	0.0466 (7)	0.0026 (5)	0.0151 (5)	−0.0072 (5)
O3	0.0106 (4)	0.0155 (5)	0.0186 (5)	0.0014 (4)	0.0061 (4)	−0.0011 (4)
O4	0.0103 (4)	0.0146 (5)	0.0187 (5)	−0.0015 (4)	0.0054 (4)	0.0018 (4)
O5	0.0245 (6)	0.0222 (5)	0.0223 (5)	−0.0018 (4)	0.0137 (4)	−0.0039 (4)
N1	0.0118 (5)	0.0126 (5)	0.0174 (5)	0.0001 (4)	0.0065 (4)	−0.0009 (4)
N2	0.0098 (5)	0.0133 (5)	0.0133 (5)	−0.0003 (4)	0.0046 (4)	−0.0006 (4)
N3	0.0092 (5)	0.0134 (5)	0.0133 (5)	−0.0004 (5)	0.0035 (4)	0.0003 (4)
C1	0.0103 (6)	0.0176 (6)	0.0164 (6)	0.0004 (5)	0.0064 (5)	0.0038 (5)
C2	0.0099 (6)	0.0131 (6)	0.0131 (6)	0.0005 (5)	0.0057 (5)	0.0017 (5)
C3	0.0139 (6)	0.0126 (6)	0.0208 (6)	0.0015 (5)	0.0087 (5)	−0.0021 (5)
C4	0.0122 (6)	0.0118 (6)	0.0195 (6)	−0.0013 (5)	0.0063 (5)	−0.0021 (5)
C5	0.0085 (6)	0.0121 (6)	0.0124 (5)	0.0006 (5)	0.0044 (5)	0.0022 (5)
C6	0.0082 (6)	0.0152 (6)	0.0101 (5)	0.0001 (5)	0.0035 (4)	0.0008 (5)
C7	0.0116 (6)	0.0114 (6)	0.0172 (6)	−0.0021 (5)	0.0056 (5)	−0.0021 (5)
C8	0.0166 (7)	0.0125 (6)	0.0144 (6)	−0.0006 (5)	0.0059 (5)	−0.0008 (5)
C9	0.0121 (6)	0.0132 (6)	0.0145 (6)	−0.0004 (5)	0.0047 (5)	0.0000 (5)
C10	0.0149 (6)	0.0127 (6)	0.0137 (6)	0.0001 (5)	0.0049 (5)	0.0007 (5)
C11	0.0380 (10)	0.0197 (7)	0.0261 (8)	0.0078 (7)	0.0201 (7)	0.0009 (6)
C12	0.0351 (9)	0.0171 (7)	0.0315 (8)	−0.0015 (7)	0.0175 (7)	0.0028 (6)

Geometric parameters (Å, º)

S1—O5	1.5007 (12)	C3—C4	1.3904 (19)
S1—C11	1.7791 (17)	C3—H3	0.9300
S1—C12	1.7888 (17)	C4—C5	1.3905 (18)
O1—C1	1.2594 (18)	C4—H4	0.9300
O2—C1	1.2442 (19)	C5—C6	1.5265 (18)
O3—C6	1.2508 (17)	C7—H7	0.9300
O4—C6	1.2645 (17)	C8—C9	1.5182 (19)
N1—C5	1.3423 (17)	C8—H8A	0.9700
N1—C7	1.3427 (18)	C8—H8B	0.9700
N2—C8	1.4866 (18)	C9—C10	1.5189 (19)
N2—H2A	0.91 (2)	C9—H9A	0.9700
N2—H2B	0.86 (2)	C9—H9B	0.9700
N2—H2C	0.92 (2)	C10—H10A	0.9700
N3—C10	1.4847 (18)	C10—H10B	0.9700
N3—H3A	0.91 (2)	C11—H11A	0.9600
N3—H3B	0.890 (19)	C11—H11B	0.9600
N3—H3C	0.86 (2)	C11—H11C	0.9600
C1—C2	1.5204 (19)	C12—H12A	0.9600
C2—C3	1.3867 (19)	C12—H12B	0.9600
C2—C7	1.3955 (18)	C12—H12C	0.9600
O5—S1—C11	105.87 (8)	N1—C7—C2	123.87 (13)
O5—S1—C12	106.25 (7)	N1—C7—H7	118.1
C11—S1—C12	97.83 (8)	C2—C7—H7	118.1
C5—N1—C7	117.60 (12)	N2—C8—C9	111.69 (11)
C8—N2—H2A	109.7 (12)	N2—C8—H8A	109.3
C8—N2—H2B	108.8 (13)	C9—C8—H8A	109.3
H2A—N2—H2B	108.5 (17)	N2—C8—H8B	109.3
C8—N2—H2C	110.5 (12)	C9—C8—H8B	109.3
H2A—N2—H2C	112.2 (17)	H8A—C8—H8B	107.9
H2B—N2—H2C	107.1 (17)	C8—C9—C10	109.33 (12)
C10—N3—H3A	109.5 (12)	C8—C9—H9A	109.8
C10—N3—H3B	108.8 (11)	C10—C9—H9A	109.8
H3A—N3—H3B	111.2 (17)	C8—C9—H9B	109.8
C10—N3—H3C	108.6 (13)	C10—C9—H9B	109.8
H3A—N3—H3C	110.5 (17)	H9A—C9—H9B	108.3
H3B—N3—H3C	108.1 (17)	N3—C10—C9	111.05 (11)
O2—C1—O1	126.50 (14)	N3—C10—H10A	109.4
O2—C1—C2	116.52 (13)	C9—C10—H10A	109.4
O1—C1—C2	116.98 (13)	N3—C10—H10B	109.4
C3—C2—C7	117.59 (12)	C9—C10—H10B	109.4
C3—C2—C1	120.48 (12)	H10A—C10—H10B	108.0
C7—C2—C1	121.92 (12)	S1—C11—H11A	109.5
C2—C3—C4	119.29 (13)	S1—C11—H11B	109.5
C2—C3—H3	120.4	H11A—C11—H11B	109.5
C4—C3—H3	120.4	S1—C11—H11C	109.5

C3—C4—C5	118.97 (13)	H11A—C11—H11C	109.5
C3—C4—H4	120.5	H11B—C11—H11C	109.5
C5—C4—H4	120.5	S1—C12—H12A	109.5
N1—C5—C4	122.59 (12)	S1—C12—H12B	109.5
N1—C5—C6	116.55 (11)	H12A—C12—H12B	109.5
C4—C5—C6	120.85 (12)	S1—C12—H12C	109.5
O3—C6—O4	126.18 (12)	H12A—C12—H12C	109.5
O3—C6—C5	117.70 (12)	H12B—C12—H12C	109.5
O4—C6—C5	116.11 (12)		

O2—C1—C2—C3	6.0 (2)	C3—C4—C5—C6	−177.55 (12)
O1—C1—C2—C3	−173.18 (13)	N1—C5—C6—O3	16.67 (17)
O2—C1—C2—C7	−175.04 (14)	C4—C5—C6—O3	−164.35 (13)
O1—C1—C2—C7	5.8 (2)	N1—C5—C6—O4	−162.89 (12)
C7—C2—C3—C4	−2.7 (2)	C4—C5—C6—O4	16.09 (18)
C1—C2—C3—C4	176.36 (13)	C5—N1—C7—C2	1.7 (2)
C2—C3—C4—C5	1.6 (2)	C3—C2—C7—N1	1.1 (2)
C7—N1—C5—C4	−2.97 (19)	C1—C2—C7—N1	−177.95 (13)
C7—N1—C5—C6	175.99 (11)	N2—C8—C9—C10	−179.79 (11)
C3—C4—C5—N1	1.4 (2)	C8—C9—C10—N3	−173.86 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O4^{i}	0.91 (2)	1.96 (2)	2.8260 (16)	157.8 (17)
N2—H2*B*···O3^{ii}	0.86 (2)	2.06 (2)	2.8461 (17)	151.0 (18)
N2—H2*C*···O2^{iii}	0.92 (2)	1.84 (2)	2.7385 (17)	164.4 (18)
N3—H3*A*···O1^{iv}	0.91 (2)	1.85 (2)	2.7369 (17)	161.6 (18)
N3—H3*B*···O3	0.890 (19)	2.073 (19)	2.8427 (16)	144.2 (16)
N3—H3*C*···O4^{v}	0.86 (2)	1.96 (2)	2.7925 (17)	164.2 (18)
C8—H8*A*···O5^{vi}	0.97	2.50	3.4614 (19)	170
C10—H10*A*···O5	0.97	2.53	3.4718 (19)	165
C11—H11*B*···O1^{ii}	0.96	2.46	3.424 (2)	178

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