organic compounds

DL-Asparaginium nitrate

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In the title compound, $C_4H_9N_2O_3^+ \cdot NO_3^-$, alternatively called (1RS)-2-carbamoyl-1-carboxyethanaminium nitrate, the asymmetric unit comprises one asparaginium cation and one nitrate anion. The strongest cation–cation O—H···O hydrogen bond in the structure, together with other strong cation–cation N—H···O hydrogen bonds, generates a succession of infinite chains of $R_2^2(8)$ rings along the b axis. Additional cation–cation C—H···O hydrogen bonds link these chains into two-dimensional layers formed by alternating $R_4^4(24)$ and $R_4^2(12)$ rings. Connections between these layers are provided by the strong cation–anion N—H···O hydrogen bonds, as well as by one weak C—H···O interaction, thus forming a three-dimensional network. Some of the cation–anion N—H···O hydrogen bonds are bifurcated of the type D—H···(A_1,A_2).

Related literature

DL-Asparagine has been used in growth media for bacteria, see: Gerhardt & Wilson (1948); Palleroni *et al.* (1973); Wagtendonk *et al.* (1963). For related structures, see Aarthy *et al.* (2005); Anitha *et al.* (2005); Arnold *et al.* (2000); Flaig *et al.* (2002); Kartha & de Vries (1961); Ramanadham *et al.* (1972); Smirnova *et al.* (1990); Verbist *et al.* (1972); Wang *et al.* (1985); Weisinger-Lewin *et al.* (1989); Yamada *et al.* (2007). For hydrogen bonding, see: Desiraju & Steiner (1999). For hydrogen-bond morifs, see: Bernstein *et al.* (1995); Etter *et al.* (1990).
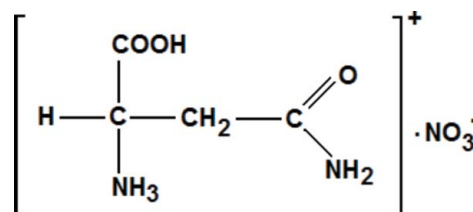
Experimental

Crystal data

$C_4H_9N_2O_3^+ \cdot NO_3^-$	V = 772.2 (3) Å^3
M_r = 195.14	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 7.923 (2) Å	μ = 0.16 mm^{-1}
b = 9.608 (2) Å	T = 100 K
c = 10.613 (3) Å	0.30 × 0.20 × 0.09 mm
β = 107.105 (2)°	

Data collection

Oxford Diffraction Xcalibur–Sapphire2 CCD diffractometer	19446 measured reflections
Absorption correction: gaussian (*CrysAlis RED*; Oxford Diffraction, 2008)	2236 independent reflections
	1804 reflections with $I > 2\sigma(I)$
T_{min} = 0.966, T_{max} = 0.991	R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.087	
S = 1.07	
2236 reflections	$\Delta\rho_{max}$ = 0.45 e Å^{-3}
136 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^i	0.845 (16)	1.736 (16)	2.571 (2)	169.0 (17)
N2—H1N···O4^{ii}	0.899 (15)	1.962 (15)	2.822 (2)	159.6 (13)
N2—H2N···O3	0.907 (15)	2.406 (16)	2.965 (2)	119.9 (12)
N2—H2N···O5	0.907 (15)	2.233 (14)	3.024 (2)	145.4 (13)
N2—H2N···O6	0.907 (15)	2.474 (15)	3.039 (2)	120.7 (11)
N2—H3N···O4^{iii}	0.903 (14)	2.454 (14)	3.157 (2)	135.0 (12)
N2—H3N···O6^{iii}	0.903 (14)	2.068 (15)	2.957 (2)	168.3 (14)
N3—H5N···O2^{iv}	0.893 (16)	2.064 (15)	2.924 (2)	161.4 (15)
C3—H3···O5^v	0.99	2.36	3.086 (2)	130
C3—H4···O2^v	0.99	2.39	3.313 (2)	156

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2157).

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supporting information

DL-**Asparaginium nitrate**

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S1. Comment

DL-asparagine, the racemic melange of the aparagine L and D-enantiomers, has been used in growth-media for bacteria-growth such as Brucellae (Gerhardt & Wilson, 1948), Pseudomonas fluorescens (Palleroni et al., 1973) and lambda particles (van Wagtendonk et al., 1963).

The crystal structures of the L-enantiomer compounds are reported most often, indeed, L-asparagine monohydrate, determined by X-ray or neutron diffraction, has been reported for the first time by Kartha & de Vries (1961) and then reported, i.a., by Verbist et al. (1972); Ramanadham et al. (1972); Wang et al. (1985); Weisinger-Lewin et al. (1989); Smirnova et al. (1990); Arnold et al. (2000); Flaig et al. (2002).

Recently, Yamada et al. (2007) reported the crystal structure of the anhydrous L-asparagine and Aarthy et al. (2005) reported the crystal structure of L-asparaginium nitrate.

In the present study, the single-crystal structure determination of anhydrous DL-asparaginium nitrate as a part of the work of our team is reported. The asymmetric unit is formed by the monoprotonated asparaginium cation $(C_4H_9O_3N_2)^+$ and the nitrate anion $(NO_3)^-$ (Fig. 1). Observation of the build-up of the electron density in the vicinity of O1, the different C-O bond distances [1.3116 (16) and 1.2143 (15) Å] and the pertinent O-C-O bond angle [126.2 (1)°] clearly confirm the protonation of the carboxyl group.

The crystal structure is stabilized by O-H···O, N-H···O and C-H···O, cation-cation (Fig. 2) and cation-anion (Fig. 3) hydrogen bonds. The O1-H1···O3 cation-cation hydrogen bond is the strongest one observed in the title structure at all (Tab. 1).

The O1-H1···O3 and N3-H5N···O2 cation-cation hydrogen bonds generate a succession of infinite chains composed of $R_2^2(8)$ rings that propagate in a zig-zagged way along the axis b (Fig. 4). These chains are interconnected by C3-H4···O2 hydrogen bonds (Tab. 1), giving rise to two-dimensional cationic layers which are formed by a succession of alternating $R_4^2(12)$ and $R_4^4(24)$ rings (Fig. 4). The former ring includes also N3-H5N···O2 while the latter O1-H1···O3 intermolecular hydrogen bonds (see Tab. 1). The cation-anion hydrogen bonds interlink the cationic layers into a three-dimensional network (Fig. 5). Some of the cation-anion N—H···O hydrogen bonds are bifurcated of the type D-H···(A_1,A_2) (Desiraju & Steiner, 1999).

The backbone conformation of the cation asparaginium is stabilized by the intramolecular N2-H2N···O3 interaction, with the S(6) motif (Bernstein et al., 1995), between the O atom of the amide group as an acceptor and one of the H atoms of the -NH3 group. The pertinent angle N2-H2N···O3 is quite acute (Tab. 1). A similiar intramolecular interaction is observed in L-asparaginium picrate (Anitha et al., 2005) and L-asparaginium nitrate (Aarthy et al., 2005).

In this study the graph-set suggested by Etter et al. (1990) and the quantitative graph set descriptor $G^a_d(n)$ (Bernstein et al., 1995) are used in order to describe the hydrogen bonds in the title structure (Tab. 1). The unitary graph set is

composed of ten motifs: N_1 = C(7)C(7)S(6)C(5)DDDDDD where C(7) applies for O1—H···O3 and N3—H5N···O2; S(6) for N2—H2N···O3 and C(5) for C3—H4···O2 while the rest of the motifs D refer to the cation-anion interactions.

S2. Experimental

The title compound was prepared by heating of a mixture of DL-asparagine monohydrate of purity 98 % (Alfa Aesar) and nitric acid. This mixture was obtained by dissolution and agitation for 20 minutes of 0.75 g of the DL-asparagine monohydrate in 15 ml of water at 25°C followed by addition of 15 ml of 1 *M* nitric acid. Colourless needle crystals with approximate dimensions 0.30 x 0.20 x 0.10 mm were obtained by evaporation of the solution at room temperature in the course of a few weeks.

S3. Refinement

All the H atoms were located in the difference electron density maps. All the H atoms attached to C were treated as riding with C-H = 1.00 Å (methine) or 0.99 Å (methylene) with $U_{iso}H = 1.2U_{eq}C$. The coordinate parameters of the H atoms attached to N or O were freely refined with $U_{iso}H = 1.2U_{eq}N$ and $U_{iso}H = 1.5U_{eq}O$.

Figure 1
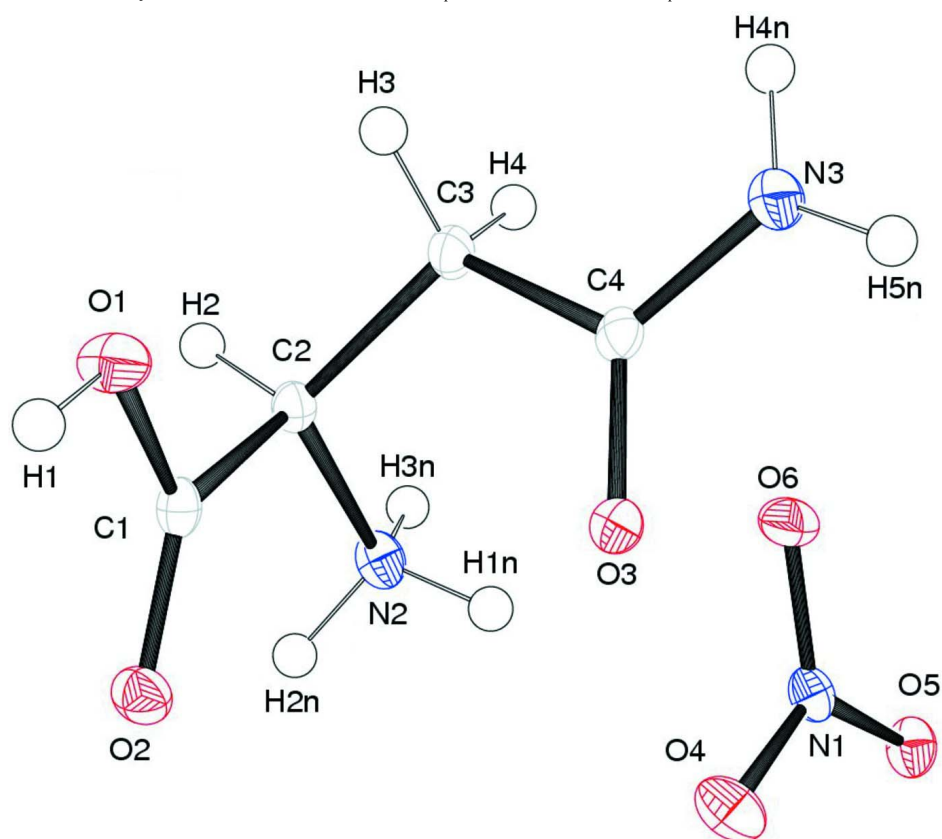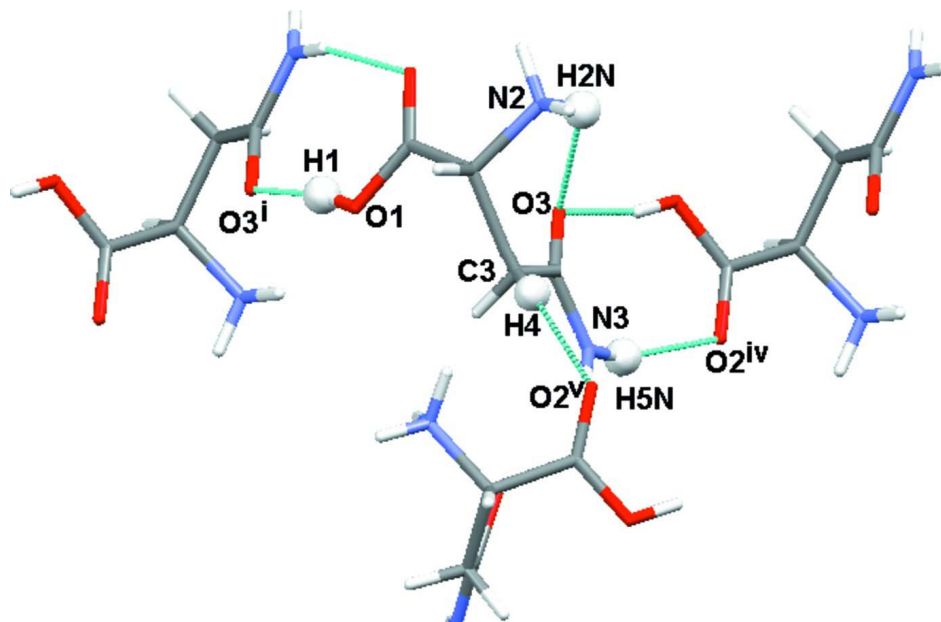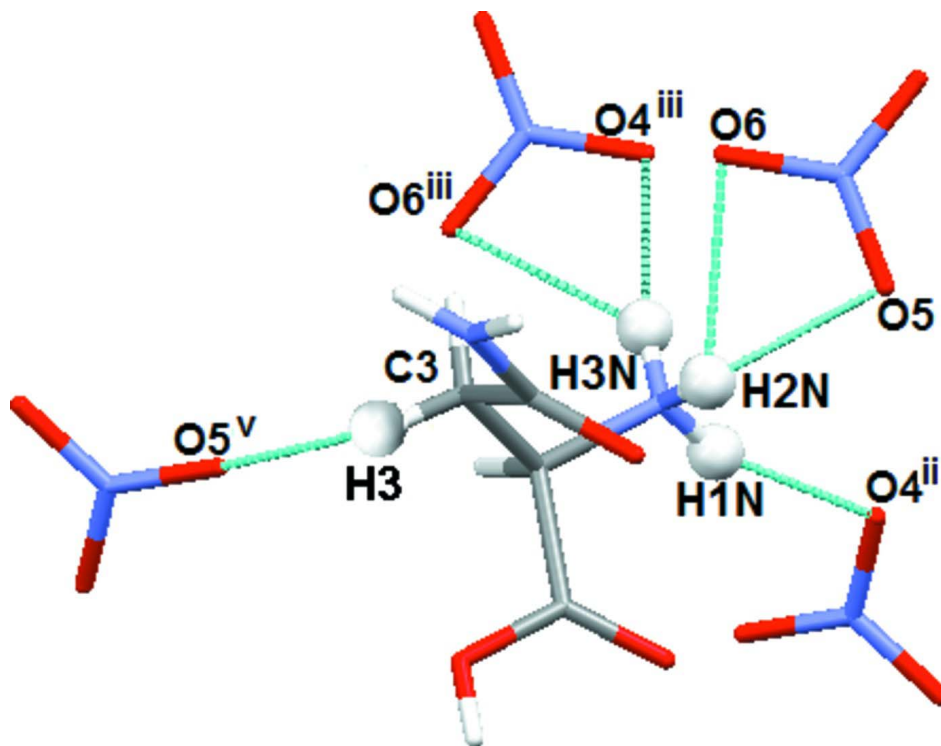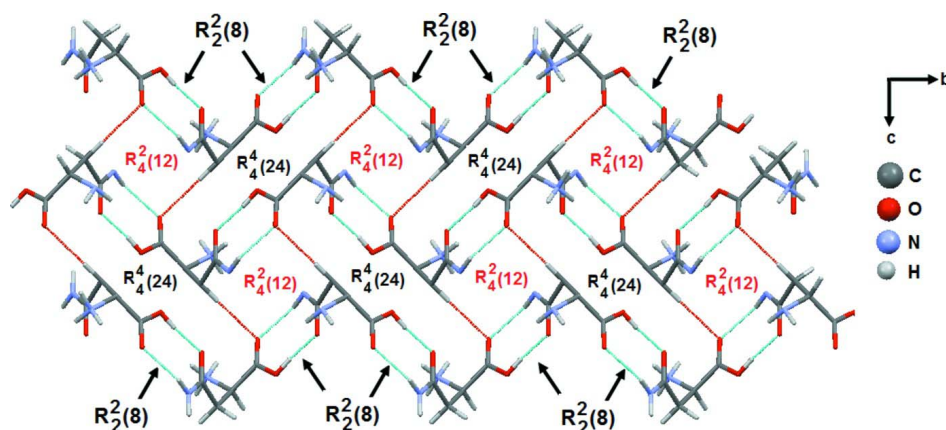
The title molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level

Figure 2

The cation-cation hydrogen bonds. *Symmetry codes: (i) -x+1, y+1/2, -z+3/2; (iv) -x+1, y-1/2, -z+3/2; (v) x, -y+3/2, z-1/2*

Figure 3

The cation-anion hydrogen bonds. *Symmetry codes: (ii) -x, y+1/2, -z+3/2; (iii) -x, -y+1, -z+1; (v) x, -y+3/2, z-1/2*

Figure 4

Hydrogen bonding cation-cation infinit chains within the DL-asparaginium layer. The axis *a* is directed downwards from the projection plane.
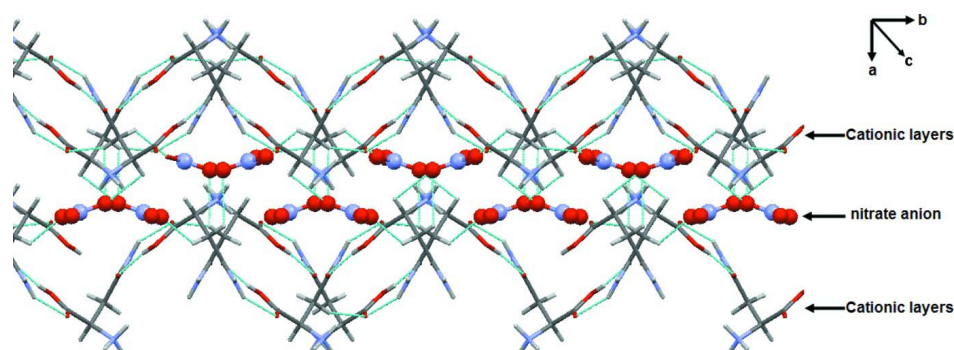
Figure 5

Connection between the cationic layers *via* cation-anion H-bonds

(1*RS*)-2-carbamoyl-1-carboxyethanaminium nitrate

Crystal data

$C_4H_9N_2O_3{}^+\cdot NO_3{}^-$
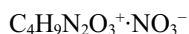
$M_r = 195.14$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.923$ (2) Å

$b = 9.608$ (2) Å

$c = 10.613$ (3) Å

$\beta = 107.105$ (2)°

$V = 772.2$ (3) Å^3

$Z = 4$

$F(000) = 408$

$D_x = 1.679$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 19446 reflections

$\theta = 2.9$–30.0°

$\mu = 0.16$ mm^{-1}

$T = 100$ K

Prism, colorless

$0.3 \times 0.2 \times 0.09$ mm

Data collection

Oxford Diffraction Xcalibur–Sapphire2 CCD diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: gaussian (*CrysAlis RED*; Oxford Diffraction, 2008)

$T_{min} = 0.966$, $T_{max} = 0.991$

19446 measured reflections

2236 independent reflections

1804 reflections with $I > 2\sigma(I)$

$R_{int} = 0.036$

$\theta_{max} = 30.0°$, $\theta_{min} = 2.9°$

$h = -11\rightarrow11$

$k = -13\rightarrow13$

$l = -11\rightarrow14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: difference Fourier map
$wR(F^2) = 0.087$	H atoms treated by a mixture of independent
$S = 1.07$	and constrained refinement
2236 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.1163P]$
136 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant	$\Delta\rho_{max} = 0.45$ e Å^{-3}
direct methods	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of $F^{\wedge}2^{\wedge}$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^{\wedge}2^{\wedge}$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^{\wedge}2^{\wedge}$. The threshold expression of $F^{\wedge}2^{\wedge} > \sigma(F^{\wedge}2^{\wedge})$ is used only for calculating *R*-factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^{\wedge}2^{\wedge}$ 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}
O1	0.35366 (11)	1.04131 (8)	0.62255 (8)	0.0171 (2)
H1	0.403 (2)	1.0991 (17)	0.6820 (16)	0.0256*
O2	0.21545 (11)	0.96178 (8)	0.76602 (8)	0.0150 (2)
O3	0.46509 (10)	0.71971 (8)	0.71175 (8)	0.0144 (2)
N2	0.07508 (13)	0.74096 (10)	0.61109 (10)	0.0133 (3)
H1N	0.0164 (19)	0.7751 (15)	0.6650 (14)	0.0158*
H2N	0.1526 (19)	0.6789 (15)	0.6612 (14)	0.0158*
H3N	0.0026 (19)	0.6923 (15)	0.5440 (14)	0.0158*
N3	0.57418 (14)	0.66187 (11)	0.54436 (11)	0.0172 (3)
H4N	0.5623 (19)	0.6635 (16)	0.4621 (16)	0.0207*
H5N	0.659 (2)	0.6100 (16)	0.5979 (15)	0.0207*
C1	0.24959 (14)	0.95636 (10)	0.66154 (11)	0.0118 (3)
C2	0.16701 (14)	0.84965 (10)	0.55565 (10)	0.0111 (3)
H2	0.07526	0.89888	0.48445	0.0134*
C3	0.29888 (14)	0.78621 (11)	0.49291 (10)	0.0129 (3)
H3	0.34226	0.85986	0.44499	0.0154*
H4	0.23798	0.71521	0.42775	0.0154*
C4	0.45449 (14)	0.71962 (10)	0.59194 (11)	0.0117 (3)
O4	0.10171 (10)	0.28059 (8)	0.69646 (8)	0.0164 (2)
O5	0.18243 (12)	0.48467 (9)	0.78126 (9)	0.0228 (3)
O6	0.17667 (12)	0.44085 (9)	0.57939 (9)	0.0204 (3)
N1	0.15474 (12)	0.40279 (9)	0.68709 (9)	0.0132 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0219 (4)	0.0149 (4)	0.0156 (4)	−0.0083 (3)	0.0074 (3)	−0.0035 (3)
O2	0.0182 (4)	0.0129 (3)	0.0149 (4)	−0.0026 (3)	0.0064 (3)	−0.0031 (3)
O3	0.0147 (4)	0.0153 (4)	0.0131 (4)	0.0045 (3)	0.0040 (3)	0.0021 (3)
N2	0.0129 (4)	0.0116 (4)	0.0153 (5)	−0.0020 (3)	0.0042 (4)	−0.0034 (3)
N3	0.0171 (5)	0.0193 (5)	0.0168 (5)	0.0066 (4)	0.0074 (4)	0.0029 (4)
C1	0.0105 (5)	0.0090 (4)	0.0144 (5)	0.0017 (3)	0.0012 (4)	0.0007 (4)
C2	0.0113 (5)	0.0096 (4)	0.0117 (5)	0.0008 (3)	0.0021 (4)	−0.0004 (3)
C3	0.0139 (5)	0.0125 (4)	0.0117 (5)	0.0029 (4)	0.0030 (4)	0.0000 (4)
C4	0.0119 (5)	0.0083 (4)	0.0151 (5)	−0.0003 (3)	0.0043 (4)	0.0010 (4)
O4	0.0158 (4)	0.0115 (4)	0.0222 (4)	−0.0018 (3)	0.0061 (3)	−0.0004 (3)
O5	0.0249 (5)	0.0228 (4)	0.0197 (4)	−0.0048 (3)	0.0049 (4)	−0.0115 (3)
O6	0.0266 (5)	0.0189 (4)	0.0205 (4)	0.0002 (3)	0.0142 (4)	0.0018 (3)
N1	0.0104 (4)	0.0127 (4)	0.0164 (5)	−0.0004 (3)	0.0037 (3)	−0.0027 (3)

Geometric parameters (Å, º)

O1—C1	1.3107 (16)	N2—H3N	0.903 (14)
O2—C1	1.2173 (16)	N2—H2N	0.907 (15)
O3—C4	1.2494 (16)	N3—H5N	0.893 (16)
O1—H1	0.845 (16)	N3—H4N	0.850 (16)
O4—N1	1.2608 (14)	C1—C2	1.5198 (17)
O5—N1	1.2397 (15)	C2—C3	1.5218 (18)
O6—N1	1.2599 (15)	C3—C4	1.5072 (18)
N2—C2	1.4898 (17)	C2—H2	1.0000
N3—C4	1.3205 (18)	C3—H3	0.9900
N2—H1N	0.899 (15)	C3—H4	0.9900
C1—O1—H1	111.7 (11)	C1—C2—C3	113.09 (9)
H2N—N2—H3N	106.6 (13)	N2—C2—C3	111.74 (8)
C2—N2—H2N	111.5 (10)	N2—C2—C1	109.57 (9)
H1N—N2—H3N	111.3 (14)	C2—C3—C4	113.02 (9)
C2—N2—H1N	113.6 (9)	N3—C4—C3	116.35 (10)
C2—N2—H3N	108.9 (9)	O3—C4—N3	123.27 (11)
H1N—N2—H2N	104.7 (13)	O3—C4—C3	120.38 (10)
C4—N3—H4N	120.7 (11)	N2—C2—H2	107.00
H4N—N3—H5N	119.9 (15)	C1—C2—H2	107.00
C4—N3—H5N	118.8 (10)	C3—C2—H2	107.00
O4—N1—O6	118.68 (9)	C2—C3—H4	109.00
O5—N1—O6	120.55 (9)	H3—C3—H4	108.00
O4—N1—O5	120.76 (9)	C4—C3—H3	109.00
O1—C1—C2	111.14 (9)	C4—C3—H4	109.00
O1—C1—O2	126.15 (10)	C2—C3—H3	109.00
O2—C1—C2	122.67 (10)		
O1—C1—C2—N2	170.37 (9)	N2—C2—C3—C4	−67.79 (11)

O1—C1—C2—C3	45.00 (12)	C1—C2—C3—C4	56.40 (11)
O2—C1—C2—N2	−11.93 (15)	C2—C3—C4—O3	0.35 (14)
O2—C1—C2—C3	−137.30 (11)	C2—C3—C4—N3	179.75 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3[i]	0.845 (16)	1.736 (16)	2.571 (2)	169.0 (17)
N2—H1*N*···O4[ii]	0.899 (15)	1.962 (15)	2.822 (2)	159.6 (13)
N2—H2*N*···O3	0.907 (15)	2.406 (16)	2.965 (2)	119.9 (12)
N2—H2*N*···O5	0.907 (15)	2.233 (14)	3.024 (2)	145.4 (13)
N2—H2*N*···O6	0.907 (15)	2.474 (15)	3.039 (2)	120.7 (11)
N2—H3*N*···O4[iii]	0.903 (14)	2.454 (14)	3.157 (2)	135.0 (12)
N2—H3*N*···O6[iii]	0.903 (14)	2.068 (15)	2.957 (2)	168.3 (14)
N3—H5*N*···O2[iv]	0.893 (16)	2.064 (15)	2.924 (2)	161.4 (15)
C3—H3···O5[v]	0.9900	2.3600	3.086 (2)	130.00
C3—H4···O2[v]	0.9900	2.3900	3.313 (2)	156.00

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