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m-Xylylenediaminium dinitrate

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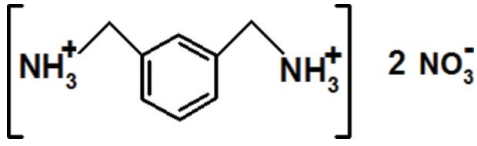
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.129; data-to-parameter ratio = 16.6.

The asymmetric unit of the title salt, $C_8H_{14}N_2^{2+}\cdot 2NO_3^-$, contains two independent dications and four independent nitrate anions. The crystal structure consists of discrete nitrate ions, three of which stack in layers parallel to (001) at z = 0 and 0.5. These layers are connected *via* *m*-xylylenediaminium dications. The fourth anion is sandwiched by the two independent organic cations in the asymmetric unit. In the crystal, the ions are connected by a large number of bifurcated and non-bifurcated N—H···O(O) hydrogen bonds, forming sheets parallel to (100). These sheets are connected by C—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For related nitrate salts, see: Gatfaoui *et al.* (2013, 2014); Marouani *et al.* (2012); Kefi *et al.* (2013). For the dichloride salt of the title cation, see: Cheng & Li (2008). For background to hydrogen bonding and aromatic π–π stacking interactions, see: Brown (1976); Blessing (1986); Janiak (2000).



Experimental

Crystal data

$C_8H_{14}N_2^{2+}\cdot 2NO_3^-$
M_r = 262.23
Monoclinic, $P2_1/c$
a = 21.4308 (7) Å
b = 5.7255 (2) Å
c = 20.4476 (5) Å
β = 108.502 (1)°
V = 2379.28 (13) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 150 K
0.47 × 0.24 × 0.17 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.889, T_{max} = 0.979
19108 measured reflections
5457 independent reflections
4429 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.129
S = 1.04
5457 reflections
329 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3	0.91	2.06	2.9545 (16)	168
N1—H1B···O6	0.91	2.34	2.9872 (18)	128
N1—H1B···O9^{i}	0.91	2.23	2.9554 (17)	137
N1—H1C···O2^{i}	0.91	2.29	3.197 (2)	173
N1—H1C···O3^{i}	0.91	2.42	3.0837 (16)	130
N2—H2B···O10^{ii}	0.91	2.48	3.032 (2)	119
N2—H2B···O12^{ii}	0.91	1.96	2.8391 (19)	162
N2—H2C···O10^{iii}	0.91	1.92	2.829 (2)	173
N2—H2C···O11^{iii}	0.91	2.51	3.074 (2)	120
N3—H3A···O5	0.91	2.42	3.0008 (17)	122
N3—H3A···O6	0.91	1.91	2.8155 (16)	175
N3—H3B···O4^{iv}	0.91	1.93	2.7974 (17)	159
N3—H3B···O6^{iv}	0.91	2.50	2.9910 (16)	114
N3—H3C···O3	0.91	2.03	2.8897 (19)	157
N4—H4A···O1	0.91	2.35	3.079 (2)	137
N4—H4A···O2	0.91	2.15	2.9967 (19)	155
N4—H4B···O8^{v}	0.91	2.42	3.0673 (16)	128
N4—H4B···O9^{v}	0.91	2.12	2.9368 (17)	150
N4—H4C···O7^{iii}	0.91	2.52	3.2192 (18)	134
N4—H4C···O8^{iii}	0.91	1.99	2.8810 (16)	165
C7—H7···O2^{i}	0.95	2.53	3.309 (2)	140
C8—H8A···O12^{vi}	0.99	2.46	3.373 (2)	153
C9—H9B···O4^{vii}	0.99	2.31	3.267 (2)	163
C16—H16A···O4^{iii}	0.99	2.33	3.267 (2)	157

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CRYSCAL* (T. Roisnel, local program).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: BH2495).

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supplementary materials

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m-Xylylenediaminium dinitrate

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1. Experimental

1.1. Synthesis and crystallization

An aqueous solution containing 4 mmol of HNO_3 in 10 ml of water was added to 2 mmol of *m*-xylylenediamine in 10 ml of water. The obtained solution was stirred for 1 h and then left to stand at room temperature. Colorless single crystals of the title compound were obtained after one week.

1.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. All H atoms bonded to C and N atoms were treated as riding with C—H = 0.99 Å (methylene) or 0.95 Å (aromatic CH), N—H = 0.91 Å (NH_3), and with U_{iso}(H) = 1.2U_{eq}(C) and U_{iso}(H) = 1.5U_{eq}(N).

2. Results and discussion

As a part of our study of crystal packing containing the nitrate anion (Marouani *et al.*, 2012; Gatfaoui *et al.*, 2013, 2014; Kefi *et al.*, 2013), we report here the preparation and the structural investigation of a new organic nitrate, $(C_8H_{14}N_2)\cdot(NO_3)_2$ (I). The asymmetric unit of (I) is composed of two *m*-xylylenediaminium dications and four nitrate anions (Fig. 1). The structure of the compound consists of discrete nitrate ions stacked in layers parallel to the (001) plane at z = 0 and 1/2, for N6/O4/O5/O6, N7/O7/O8/O9, N8/O10/O11/O12 and at z = 0.25 and 0.75 for N5/O1/O2/O3, separated by organic cations. Each organic entity is connected to different nitrate layers at z = 0 and 1/2. At the same time, the $N5O_3$ nitrate groups ensure the cohesion between the organic groups (Fig. 2). The structural cohesion is established by a three-dimensional network of N—H···O and weak C—H···O hydrogen bonds.

Interatomic bond lengths and angles of the nitrate anions spread respectively within the ranges 1.2314 (19)–1.2719 (17) Å and 118.69 (15)–121.48 (13)°. These geometrical features have also been noticed in other related crystal structures (Gatfaoui *et al.*, 2013, 2014).

In this crystal arrangement two independent *m*-xylylediaminium cations are present. Both ammonium groups in each cation adopt a *cis* conformation with respect to the benzene ring. The *trans* conformation has been observed in $C_8H_{14}N_2^{2+}\cdot 2Cl^-$ (Cheng & Li, 2008). Thus, the cation conformation is modified when substituting nitrate anions by chlorides. Examination of the organic cations shows that the bond distances and angles show no significant differences from those obtained in other compounds involving the same organic groups (Cheng & Li, 2008). The aromatic rings are planar with an average deviation of 0.0022 Å and form a dihedral angle of 3.85° in the asymmetric unit. The inter-planar distance between nearby benzene rings in the crystal structure is in the vicinity of 4.59 Å, which is much longer than 3.80 Å, value required for the formation of π–π interactions (Janiak, 2000).

The established weak H-bonds (Brown, 1976; Blessing, 1986) of types N—H···O and C—H···O involve oxygen atoms of the nitrate anions as acceptors, and the protonated nitrogen atoms and carbon atoms of *m*-xylylenediaminium as

donors.

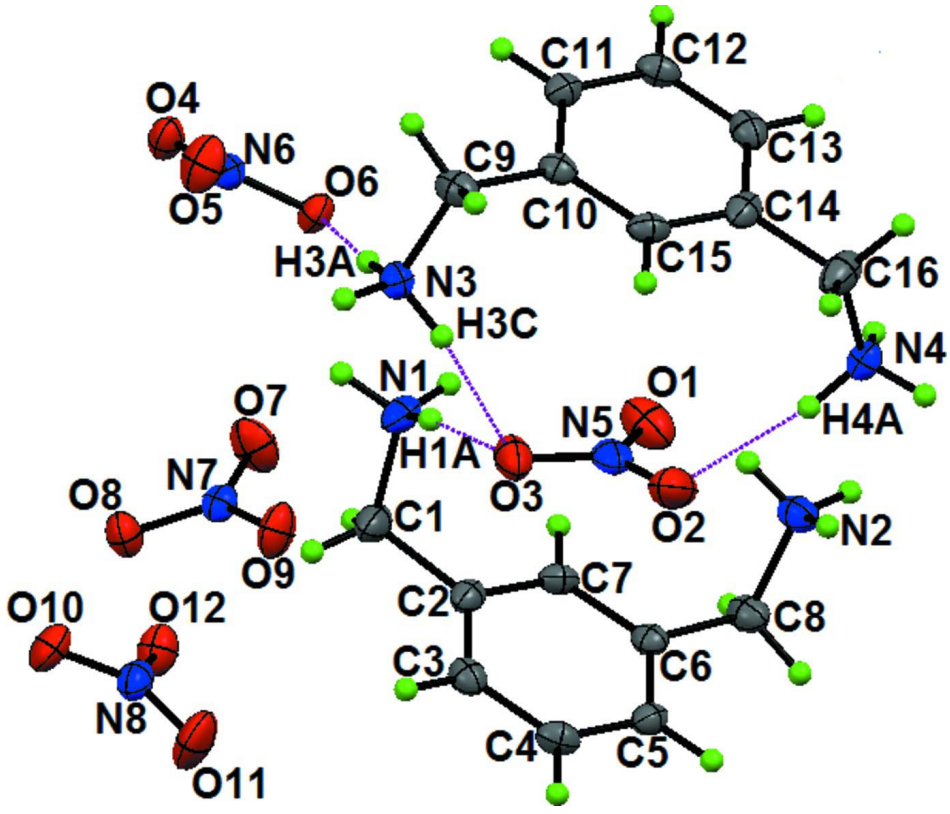

Figure 1

An *ORTEP* view of (I) with displacement ellipsoids drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bonds are shown as dotted lines.

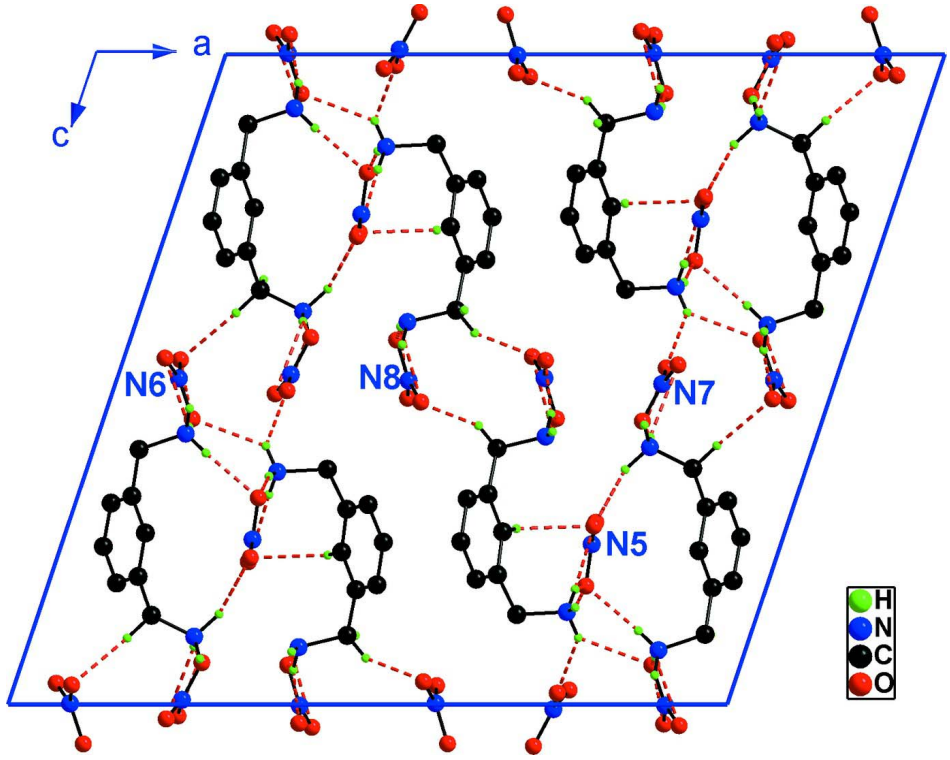

Figure 2

Projection of (I) along the *b* axis. The H-atoms not involved in H-bonding are omitted.

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Crystal data

$C_8H_{14}N_2^{2+}\cdot 2NO_3^-$
$M_r = 262.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 21.4308\ (7)$ Å
$b = 5.7255\ (2)$ Å
$c = 20.4476\ (5)$ Å
$\beta = 108.502\ (1)°$
$V = 2379.28\ (13)$ Å^3
$Z = 8$
$F(000) = 1104$
$D_x = 1.464$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6173 reflections
$\theta = 3.0–27.3°$
$\mu = 0.13$ mm^{-1}
$T = 150$ K
Prism, colourless
0.47 × 0.24 × 0.17 mm

Data collection

Bruker APEXII diffractometer
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min} = 0.889$, $T_{max} = 0.979$
19108 measured reflections
5457 independent reflections
4429 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -26\rightarrow22$
$k = -6\rightarrow7$
$l = -27\rightarrow27$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.129$
$S = 1.04$
5457 reflections
329 parameters
0 restraints
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 1.0374P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.26601 (6)	0.0292 (2)	0.14220 (7)	0.0272 (3)
H1A	0.2554	−0.1199	0.1498	0.041*
H1B	0.2374	0.0818	0.1018	0.041*
H1C	0.2635	0.1223	0.1774	0.041*
C1	0.33450 (8)	0.0351 (3)	0.13828 (9)	0.0329 (4)
H1D	0.3490	0.1991	0.1376	0.040*
H1E	0.3356	−0.0416	0.0953	0.040*
C2	0.38017 (7)	−0.0900 (3)	0.19976 (8)	0.0221 (3)
C3	0.40757 (7)	−0.3050 (3)	0.19264 (8)	0.0236 (3)
H3	0.3998	−0.3709	0.1481	0.028*
C4	0.44632 (7)	−0.4234 (3)	0.25056 (9)	0.0229 (3)
H4	0.4656	−0.5688	0.2453	0.027*
C5	0.45719 (7)	−0.3312 (3)	0.31598 (8)	0.0215 (3)
H5	0.4827	−0.4153	0.3555	0.026*
C6	0.43055 (7)	−0.1149 (3)	0.32352 (8)	0.0198 (3)
C7	0.39272 (7)	0.0044 (3)	0.26535 (8)	0.0208 (3)
H7	0.3751	0.1530	0.2704	0.025*
C8	0.43934 (8)	−0.0113 (3)	0.39380 (9)	0.0278 (4)
H8A	0.4758	−0.0919	0.4288	0.033*
H8B	0.4508	0.1561	0.3938	0.033*
N2	0.37725 (7)	−0.0370 (3)	0.41183 (7)	0.0270 (3)
H2A	0.3426	0.0137	0.3760	0.041*
H2B	0.3802	0.0495	0.4500	0.041*
H2C	0.3713	−0.1899	0.4204	0.041*
N3	0.12089 (6)	−0.4473 (2)	0.08345 (7)	0.0215 (3)
H3A	0.1231	−0.2923	0.0746	0.032*
H3B	0.1162	−0.5304	0.0442	0.032*
H3C	0.1585	−0.4920	0.1166	0.032*
C9	0.06349 (8)	−0.4918 (3)	0.10789 (9)	0.0267 (4)
H9A	0.0552	−0.6620	0.1076	0.032*
H9B	0.0239	−0.4169	0.0759	0.032*
C10	0.07523 (7)	−0.3980 (3)	0.17964 (8)	0.0195 (3)
C11	0.04955 (7)	−0.1832 (3)	0.19040 (8)	0.0205 (3)
H11	0.0256	−0.0906	0.1521	0.025*
C12	0.05893 (7)	−0.1046 (3)	0.25702 (9)	0.0234 (3)

H12	0.0410	0.0413	0.2642	0.028*
C13	0.09441 (8)	−0.2383 (3)	0.31318 (8)	0.0248 (3)
H13	0.1005	−0.1838	0.3587	0.030*
C14	0.12113 (7)	−0.4520 (3)	0.30315 (8)	0.0242 (3)
C15	0.11106 (7)	−0.5290 (3)	0.23635 (8)	0.0216 (3)
H15	0.1291	−0.6747	0.2291	0.026*
C16	0.15782 (8)	−0.6046 (4)	0.36295 (10)	0.0368 (4)
H16A	0.1348	−0.6021	0.3979	0.044*
H16B	0.1575	−0.7675	0.3466	0.044*
N4	0.22723 (6)	−0.5286 (2)	0.39575 (7)	0.0227 (3)
H4A	0.2492	−0.5372	0.3644	0.034*
H4B	0.2470	−0.6234	0.4322	0.034*
H4C	0.2279	−0.3787	0.4108	0.034*
N5	0.26305 (6)	−0.4703 (3)	0.24641 (7)	0.0287 (3)
O1	0.26758 (7)	−0.2898 (3)	0.28036 (7)	0.0436 (4)
O2	0.26855 (6)	−0.6677 (3)	0.27431 (7)	0.0415 (3)
O3	0.25175 (6)	−0.4589 (2)	0.18215 (6)	0.0350 (3)
N6	0.08581 (6)	0.0558 (2)	−0.00143 (7)	0.0236 (3)
O4	0.07924 (6)	0.2525 (2)	−0.03009 (6)	0.0283 (3)
O5	0.05713 (7)	−0.1176 (2)	−0.03262 (6)	0.0391 (3)
O6	0.12308 (5)	0.03715 (19)	0.06066 (6)	0.0246 (3)
N7	0.23961 (6)	−0.4784 (2)	−0.00724 (7)	0.0219 (3)
O7	0.22667 (7)	−0.3051 (2)	0.02227 (7)	0.0402 (3)
O8	0.24994 (6)	−0.4545 (2)	−0.06438 (6)	0.0290 (3)
O9	0.24162 (6)	−0.6782 (2)	0.01751 (6)	0.0346 (3)
N8	0.40614 (7)	0.0407 (3)	0.00192 (7)	0.0279 (3)
O10	0.37062 (6)	0.0189 (2)	−0.06046 (6)	0.0332 (3)
O11	0.43303 (8)	−0.1319 (3)	0.03493 (7)	0.0481 (4)
O12	0.41292 (6)	0.2396 (2)	0.02899 (6)	0.0347 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0203 (6)	0.0296 (8)	0.0274 (7)	0.0020 (5)	0.0012 (5)	0.0040 (6)
C1	0.0228 (8)	0.0458 (11)	0.0301 (9)	0.0040 (7)	0.0082 (7)	0.0153 (8)
C2	0.0159 (7)	0.0263 (8)	0.0237 (8)	−0.0021 (6)	0.0056 (6)	0.0068 (6)
C3	0.0215 (7)	0.0271 (8)	0.0233 (8)	−0.0056 (6)	0.0086 (6)	−0.0042 (7)
C4	0.0222 (7)	0.0159 (7)	0.0332 (9)	−0.0007 (6)	0.0126 (6)	−0.0017 (6)
C5	0.0173 (7)	0.0213 (8)	0.0257 (8)	0.0015 (6)	0.0063 (6)	0.0059 (6)
C6	0.0152 (6)	0.0218 (7)	0.0226 (7)	−0.0026 (6)	0.0062 (5)	−0.0019 (6)
C7	0.0167 (7)	0.0163 (7)	0.0307 (8)	0.0012 (5)	0.0093 (6)	0.0023 (6)
C8	0.0214 (7)	0.0342 (9)	0.0277 (8)	−0.0031 (7)	0.0076 (6)	−0.0080 (7)
N2	0.0245 (7)	0.0318 (8)	0.0266 (7)	0.0001 (6)	0.0106 (6)	−0.0073 (6)
N3	0.0225 (6)	0.0219 (7)	0.0205 (6)	−0.0001 (5)	0.0073 (5)	−0.0005 (5)
C9	0.0220 (7)	0.0303 (9)	0.0290 (9)	−0.0045 (6)	0.0100 (6)	−0.0094 (7)
C10	0.0167 (7)	0.0191 (7)	0.0240 (8)	−0.0035 (5)	0.0083 (6)	−0.0023 (6)
C11	0.0177 (7)	0.0185 (7)	0.0247 (8)	−0.0015 (5)	0.0061 (6)	0.0030 (6)
C12	0.0213 (7)	0.0187 (7)	0.0328 (9)	−0.0016 (6)	0.0121 (6)	−0.0045 (7)
C13	0.0224 (7)	0.0298 (8)	0.0232 (8)	−0.0072 (6)	0.0089 (6)	−0.0041 (7)
C14	0.0167 (7)	0.0282 (8)	0.0266 (8)	−0.0044 (6)	0.0053 (6)	0.0089 (7)

C15	0.0169 (7)	0.0161 (7)	0.0329 (9)	0.0000 (5)	0.0095 (6)	0.0016 (6)
C16	0.0233 (8)	0.0447 (11)	0.0379 (10)	−0.0055 (8)	0.0032 (7)	0.0207 (9)
N4	0.0243 (6)	0.0205 (6)	0.0205 (7)	0.0010 (5)	0.0033 (5)	0.0015 (5)
N5	0.0175 (6)	0.0420 (9)	0.0270 (7)	−0.0025 (6)	0.0077 (5)	−0.0016 (7)
O1	0.0410 (8)	0.0502 (9)	0.0449 (8)	−0.0155 (6)	0.0210 (6)	−0.0169 (7)
O2	0.0359 (7)	0.0463 (8)	0.0471 (8)	0.0113 (6)	0.0200 (6)	0.0128 (7)
O3	0.0266 (6)	0.0554 (8)	0.0228 (6)	−0.0075 (6)	0.0077 (5)	−0.0010 (6)
N6	0.0241 (7)	0.0254 (7)	0.0209 (7)	0.0023 (5)	0.0067 (5)	−0.0019 (6)
O4	0.0364 (6)	0.0253 (6)	0.0220 (6)	0.0069 (5)	0.0073 (5)	0.0029 (5)
O5	0.0484 (8)	0.0304 (7)	0.0296 (7)	−0.0083 (6)	−0.0002 (6)	−0.0073 (6)
O6	0.0269 (6)	0.0243 (6)	0.0186 (5)	0.0021 (4)	0.0013 (4)	−0.0001 (5)
N7	0.0217 (6)	0.0213 (7)	0.0212 (7)	−0.0024 (5)	0.0046 (5)	−0.0005 (5)
O7	0.0586 (9)	0.0290 (7)	0.0412 (7)	−0.0008 (6)	0.0275 (7)	−0.0099 (6)
O8	0.0389 (7)	0.0267 (6)	0.0250 (6)	−0.0006 (5)	0.0152 (5)	0.0014 (5)
O9	0.0478 (8)	0.0246 (6)	0.0266 (6)	−0.0043 (5)	0.0051 (5)	0.0072 (5)
N8	0.0237 (7)	0.0338 (8)	0.0251 (7)	−0.0016 (6)	0.0063 (6)	0.0087 (6)
O10	0.0302 (6)	0.0347 (7)	0.0261 (6)	−0.0003 (5)	−0.0034 (5)	0.0037 (5)
O11	0.0600 (9)	0.0416 (8)	0.0322 (7)	0.0123 (7)	−0.0005 (6)	0.0127 (6)
O12	0.0406 (7)	0.0324 (7)	0.0282 (6)	−0.0058 (5)	0.0070 (5)	0.0025 (5)

Geometric parameters (Å, º)

N1—C1	1.495 (2)	C9—H9B	0.9900
N1—H1A	0.9100	C10—C15	1.390 (2)
N1—H1B	0.9100	C10—C11	1.393 (2)
N1—H1C	0.9100	C11—C12	1.388 (2)
C1—C2	1.507 (2)	C11—H11	0.9500
C1—H1D	0.9900	C12—C13	1.389 (2)
C1—H1E	0.9900	C12—H12	0.9500
C2—C7	1.391 (2)	C13—C14	1.393 (2)
C2—C3	1.392 (2)	C13—H13	0.9500
C3—C4	1.388 (2)	C14—C15	1.386 (2)
C3—H3	0.9500	C14—C16	1.506 (2)
C4—C5	1.387 (2)	C15—H15	0.9500
C4—H4	0.9500	C16—N4	1.489 (2)
C5—C6	1.393 (2)	C16—H16A	0.9900
C5—H5	0.9500	C16—H16B	0.9900
C6—C7	1.388 (2)	N4—H4A	0.9100
C6—C8	1.510 (2)	N4—H4B	0.9100
C7—H7	0.9500	N4—H4C	0.9100
C8—N2	1.496 (2)	N5—O1	1.2314 (19)
C8—H8A	0.9900	N5—O2	1.255 (2)
C8—H8B	0.9900	N5—O3	1.2599 (18)
N2—H2A	0.9100	N6—O4	1.2566 (17)
N2—H2B	0.9100	N6—O5	1.2334 (18)
N2—H2C	0.9100	N6—O6	1.2719 (17)
N3—C9	1.489 (2)	N7—O7	1.2379 (18)
N3—H3A	0.9100	N7—O8	1.2637 (17)
N3—H3B	0.9100	N7—O9	1.2459 (17)
N3—H3C	0.9100	N8—O10	1.2671 (18)

C9—C10	1.507 (2)	N8—O11	1.2321 (19)
C9—H9A	0.9900	N8—O12	1.2542 (19)
C1—N1—H1A	109.5	N3—C9—C10	111.42 (12)
C1—N1—H1B	109.5	N3—C9—H9A	109.3
H1A—N1—H1B	109.5	C10—C9—H9A	109.3
C1—N1—H1C	109.5	N3—C9—H9B	109.3
H1A—N1—H1C	109.5	C10—C9—H9B	109.3
H1B—N1—H1C	109.5	H9A—C9—H9B	108.0
N1—C1—C2	109.48 (13)	C15—C10—C11	119.09 (14)
N1—C1—H1D	109.8	C15—C10—C9	119.82 (14)
C2—C1—H1D	109.8	C11—C10—C9	121.08 (14)
N1—C1—H1E	109.8	C12—C11—C10	120.02 (14)
C2—C1—H1E	109.8	C12—C11—H11	120.0
H1D—C1—H1E	108.2	C10—C11—H11	120.0
C7—C2—C3	119.06 (14)	C11—C12—C13	120.23 (15)
C7—C2—C1	119.72 (15)	C11—C12—H12	119.9
C3—C2—C1	121.14 (15)	C13—C12—H12	119.9
C4—C3—C2	120.05 (14)	C12—C13—C14	120.34 (15)
C4—C3—H3	120.0	C12—C13—H13	119.8
C2—C3—H3	120.0	C14—C13—H13	119.8
C5—C4—C3	120.59 (15)	C15—C14—C13	118.81 (14)
C5—C4—H4	119.7	C15—C14—C16	119.58 (16)
C3—C4—H4	119.7	C13—C14—C16	121.55 (16)
C4—C5—C6	119.72 (14)	C14—C15—C10	121.51 (14)
C4—C5—H5	120.1	C14—C15—H15	119.2
C6—C5—H5	120.1	C10—C15—H15	119.2
C7—C6—C5	119.42 (14)	N4—C16—C14	112.69 (13)
C7—C6—C8	119.01 (14)	N4—C16—H16A	109.1
C5—C6—C8	121.52 (14)	C14—C16—H16A	109.1
C6—C7—C2	121.13 (14)	N4—C16—H16B	109.1
C6—C7—H7	119.4	C14—C16—H16B	109.1
C2—C7—H7	119.4	H16A—C16—H16B	107.8
N2—C8—C6	110.10 (12)	C16—N4—H4A	109.5
N2—C8—H8A	109.6	C16—N4—H4B	109.5
C6—C8—H8A	109.6	H4A—N4—H4B	109.5
N2—C8—H8B	109.6	C16—N4—H4C	109.5
C6—C8—H8B	109.6	H4A—N4—H4C	109.5
H8A—C8—H8B	108.2	H4B—N4—H4C	109.5
C8—N2—H2A	109.5	O1—N5—O2	121.35 (15)
C8—N2—H2B	109.5	O1—N5—O3	119.95 (15)
H2A—N2—H2B	109.5	O2—N5—O3	118.69 (15)
C8—N2—H2C	109.5	O4—N6—O6	118.86 (13)
H2A—N2—H2C	109.5	O5—N6—O4	121.10 (13)
H2B—N2—H2C	109.5	O5—N6—O6	120.04 (13)
C9—N3—H3A	109.5	O7—N7—O9	121.48 (13)
C9—N3—H3B	109.5	O7—N7—O8	119.70 (13)
H3A—N3—H3B	109.5	O9—N7—O8	118.80 (13)
C9—N3—H3C	109.5	O11—N8—O12	121.20 (14)

H3A—N3—H3C	109.5	O11—N8—O10	119.97 (15)
H3B—N3—H3C	109.5	O12—N8—O10	118.83 (14)
N1—C1—C2—C7	−67.34 (19)	N3—C9—C10—C15	−82.48 (18)
N1—C1—C2—C3	109.35 (17)	N3—C9—C10—C11	98.88 (16)
C7—C2—C3—C4	0.6 (2)	C15—C10—C11—C12	−1.1 (2)
C1—C2—C3—C4	−176.12 (14)	C9—C10—C11—C12	177.56 (14)
C2—C3—C4—C5	1.2 (2)	C10—C11—C12—C13	0.6 (2)
C3—C4—C5—C6	−1.8 (2)	C11—C12—C13—C14	0.3 (2)
C4—C5—C6—C7	0.8 (2)	C12—C13—C14—C15	−0.7 (2)
C4—C5—C6—C8	178.08 (14)	C12—C13—C14—C16	−177.88 (14)
C5—C6—C7—C2	1.0 (2)	C13—C14—C15—C10	0.1 (2)
C8—C6—C7—C2	−176.39 (13)	C16—C14—C15—C10	177.42 (14)
C3—C2—C7—C6	−1.7 (2)	C11—C10—C15—C14	0.7 (2)
C1—C2—C7—C6	175.10 (14)	C9—C10—C15—C14	−177.94 (13)
C7—C6—C8—N2	73.72 (19)	C15—C14—C16—N4	102.05 (19)
C5—C6—C8—N2	−103.59 (16)	C13—C14—C16—N4	−80.8 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O3	0.91	2.06	2.9545 (16)	168
N1—H1*B*···O6	0.91	2.34	2.9872 (18)	128
N1—H1*B*···O9^{i}	0.91	2.23	2.9554 (17)	137
N1—H1*C*···O2^{i}	0.91	2.29	3.197 (2)	173
N1—H1*C*···O3^{i}	0.91	2.42	3.0837 (16)	130
N2—H2*B*···O10^{ii}	0.91	2.48	3.032 (2)	119
N2—H2*B*···O12^{ii}	0.91	1.96	2.8391 (19)	162
N2—H2*C*···O10^{iii}	0.91	1.92	2.829 (2)	173
N2—H2*C*···O11^{iii}	0.91	2.51	3.074 (2)	120
N3—H3*A*···O5	0.91	2.42	3.0008 (17)	122
N3—H3*A*···O6	0.91	1.91	2.8155 (16)	175
N3—H3*B*···O4^{iv}	0.91	1.93	2.7974 (17)	159
N3—H3*B*···O6^{iv}	0.91	2.50	2.9910 (16)	114
N3—H3*C*···O3	0.91	2.03	2.8897 (19)	157
N4—H4*A*···O1	0.91	2.35	3.079 (2)	137
N4—H4*A*···O2	0.91	2.15	2.9967 (19)	155
N4—H4*B*···O8^{v}	0.91	2.42	3.0673 (16)	128
N4—H4*B*···O9^{v}	0.91	2.12	2.9368 (17)	150
N4—H4*C*···O7^{iii}	0.91	2.52	3.2192 (18)	134
N4—H4*C*···O8^{iii}	0.91	1.99	2.8810 (16)	165
C7—H7···O2^{i}	0.95	2.53	3.309 (2)	140
C8—H8*A*···O12^{vi}	0.99	2.46	3.373 (2)	153
C9—H9*B*···O4^{vii}	0.99	2.31	3.267 (2)	163
C16—H16*A*···O4^{iii}	0.99	2.33	3.267 (2)	157

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