Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Methoxyanilinium nitrate

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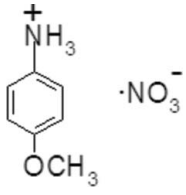
Received 12 July 2011; accepted 6 August 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.069; wR factor = 0.219; data-to-parameter ratio = 35.1.

The title compound, $C_7H_{10}NO^+\cdot NO_3^-$, crystallized with two *p*-ansidinium cations and two nitrate anions in the asymmetric unit. As well as Columbic and van der Waals forces, moleucles interact *via* multiple bifurcated N—H···O hydrogen bonds that help consolidate the crystal packing, resulting in a three-dimensional network.

Related literature

For background to anisidine, see: Li *et al.* (2001). For applications of nitrates, see: Kapoor *et al.* (2008). Association of both entities could lead to new molecular salts with interesting physical and chemical properties, see: Wilkes *et al.* (1985). For related structures, see: Ben Amor *et al.* (1995); Liu *et al.* (2011).



Experimental

Crystal data

$C_7H_{10}NO^+\cdot NO_3^-$
M_r = 186.17
Monoclinic, $P2_1/n$
a = 14.724 (2) Å
b = 7.304 (3) Å
c = 17.509 (2) Å
β = 112.84 (2)°
V = 1735.3 (8) Å^3
Z = 8
Ag $K\alpha$ radiation
λ = 0.56085 Å
μ = 0.07 mm^{-1}
T = 293 K
0.35 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
12462 measured reflections
8244 independent reflections
2756 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
2 standard reflections every 120 min
intensity decay: 5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.219
S = 0.93
8244 reflections
235 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.89	2.25	2.823 (3)	122
N1—H1A···O7^{ii}	0.89	2.52	2.979 (3)	113
N1—H1B···O6^{iii}	0.89	2.26	2.843 (3)	123
N1—H1B···O5^{iv}	0.89	2.12	2.903 (3)	146
N1—H1C···O7^{v}	0.89	2.14	2.935 (3)	148
N2—H2A···O3^{i}	0.89	2.08	2.967 (3)	177
N2—H2A···O4^{i}	0.89	2.55	3.187 (3)	129
N2—H2B···O6^{vi}	0.89	2.46	3.070 (3)	127
N2—H2B···O7^{vi}	0.89	2.22	3.083 (3)	163
N2—H2C···O3	0.89	2.07	2.891 (2)	152
C9—H9···O8^{v}	0.93	2.48	3.223 (3)	137

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2352).

References

Ben Amor, F., Soumhi, E. H., Ould Abdellahi, M. & Jouini, T. (1995). *Acta Cryst.* C**51**, 933–935.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Enraf–Nonius (1994). *CAD-4 EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Harms, K. & Wocadlo, S. (1996). *XCAD4*. University of Marburg, Germany.
Kapoor, I. P. S., Srivastava, P. & Singh, G. (2008). *J. Hazard. Mater.* **150**, 687–694.
Li, X. G., Huang, M. R., Zhi-Liang Zhu, P. P. & Yang, Y. L. (2001). *Polym. Degrad. Stab.* **71**, 333–341.
Liu, J., Tang, X., Lu, Z., Zhang, G. & Liu, W. (2011). *Acta Cryst.* E**67**, o203–o204.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wilkes, G. L., Orler, B. & Huang, H. (1985). *Polym. Prep.* , **26**, 300–302.

supporting information

Acta Cryst. (2011). E**67**, o2334 [doi:10.1107/S1600536811031862]

4-Methoxyanilinium nitrate

Hajer Rahmouni, Wajda Smirani Sta, S. Salem Al-Deyab and Mohamed Rzaigui

S1. Comment

Anisidine is used in various areas such as the production of polymers of high solubility which are interesting materials in electroconductivity and thermostability (Li *et al.*, 2001). Nitrates also have many applications such as explosives and pyrotechnics and they can be powerful oxidizers (Kapoor *et al.*, 2008). Association of both entities could lead to novel hybrid compounds with interesting physical and chemical properties (Wilkes *et al.*, 1985). The exploitation of these materials requires knowledge of not only their electronic properties but also of their atomic arrangements.

In this paper, we report crystal structure of the interaction product of [*p*-ANI] and nitric acid (I). As shown in Fig.1, the asymmetric unit of (I) contains two nitrate anions and two $[p\text{-ANIH}]^+$ cations interconnected by N—H···O and C—H···O hydrogen bonds (Table 1)). Geometrical characteristics of the two independent nitrate anions are slightly different. In one the N—O distances ($N_3/O_3/O_4/O_5$) range from 1.202 (3) to 1.268 (2) Å while in the other one the N—O distances ($N_4/O_6/O_7/O_8$) range from 1.177 (3) to 1.268 (2) Å. Examination of the $[p\text{-ANIH}]^+$ cations shows that the bond distances and angles show no significant difference from those obtained in other structures involving the same organic groups (Ben Amor *et al.*, 1995). The phenyl rings of these cations are planar with a maximum atomic deviation of ±0.00027 Å and a dihedral angle between them of 8.17°.

The crystal packing shows how each nitrate anion is connected to three $[p\text{-ANIH}]^+$ cations by N—H···O hydrogen bonding interactions (Table 1, Fig.2). It is noteworthy that the oxygen atom of the shortest bond (N_4—O_8: 1.177 (3) Å) does not participate to the hydrogen bonding network. A similar situation was observed in $C_{36}H_{40}N_5O_6NO_3.2C_2H_5OH$ (Liu *et al.*, 2011), where the N—O bond length is 1.186 (8) Å.

S2. Experimental

An ethanolic solution of *p*-anisidine [*p*-ANI] (10 mmol, in 10 ml) was added drop wise to a magnetically stirred aqueous solution of nitric acid HNO_3 (10 mmol, 20 ml). The resultant solution was then slowly evaporated at room temperature (295 K). After a few days, colorless crystals ofn(I) appeared that were suitable for *X*– ray diffraction measurements.

S3. Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, [N–H = 0.89, C–H =0.96 Å (CH_3) with with U_{iso}(H) = 1.5Ueq and C–H =0.96 Å (Ar–H), with U_{iso}(H) = 1.5Ueq],.

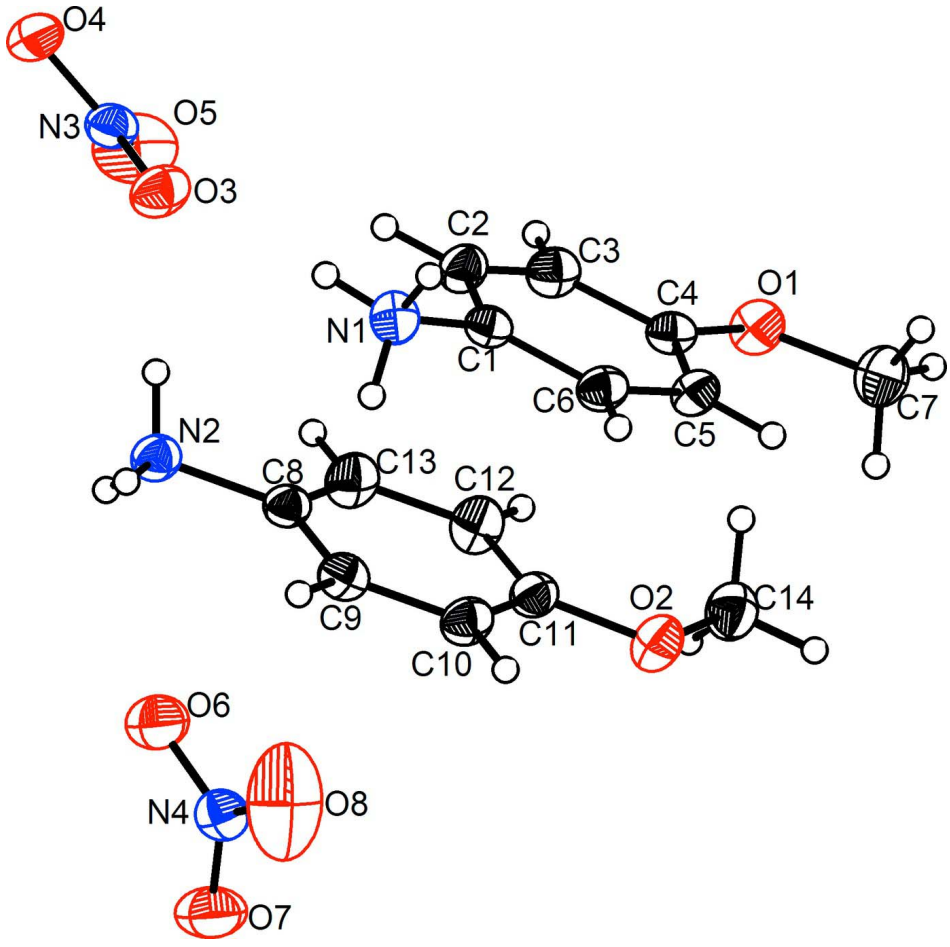


Figure 1

The assymetric unit of the title compound, with the atom numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small sphere of arbitrary radii.

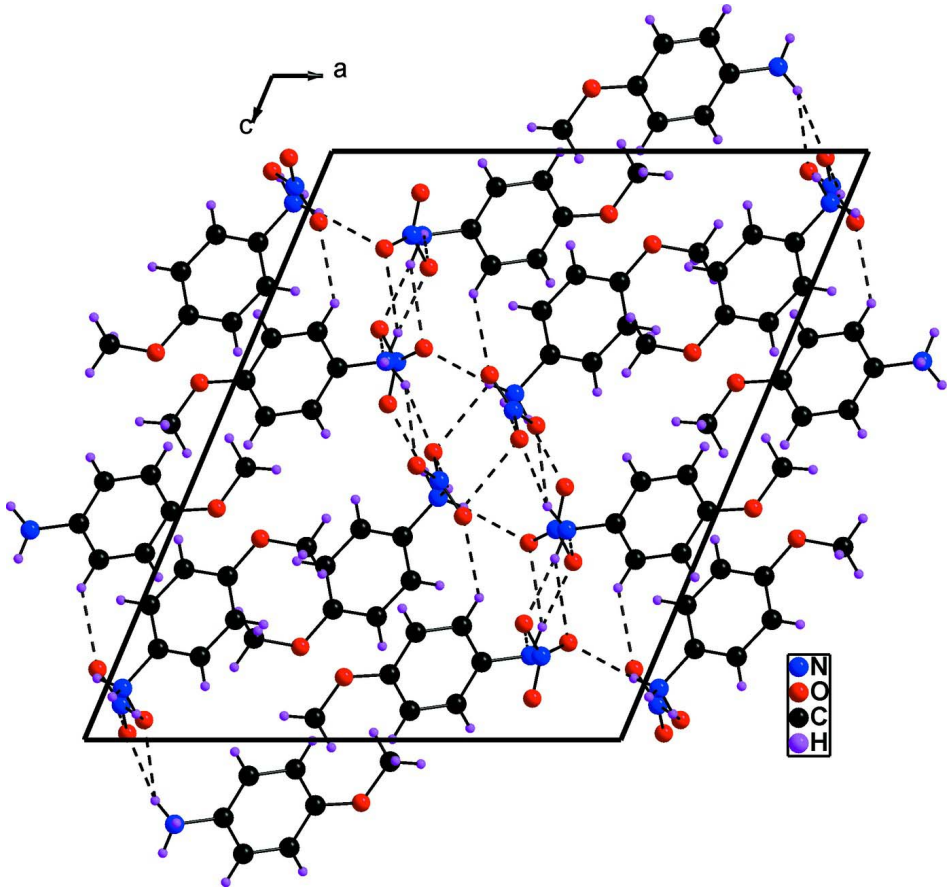


Figure 2

Projection of the crystal packing along the b axis.

4-Methoxyanilinium nitrate

Crystal data

$C_7H_{10}NO^+·NO_3^-$
M_r = 186.17
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 14.724 (2) Å
b = 7.304 (3) Å
c = 17.509 (2) Å
β = 112.84 (2)°
V = 1735.3 (8) Å^3
Z = 8

$F(000)$ = 784
D_x = 1.425 Mg m^{-3}
Ag $K\alpha$ radiation, λ = 0.56085 Å
Cell parameters from 25 reflections
θ = 9–11°
μ = 0.07 mm^{-1}
T = 293 K
Block, brown
0.35 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled ω scans
12462 measured reflections
8244 independent reflections
2756 reflections with $I > 2\sigma(I)$

R_{int} = 0.033
θ_{max} = 28.0°, θ_{min} = 2.4°
h = −24→23
k = −3→12
l = −2→29
2 standard reflections every 120 min
intensity decay: 5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.219$
$S = 0.93$
8244 reflections
235 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-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0981P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
N4	0.45826 (13)	−0.1031 (3)	0.55952 (10)	0.0482 (4)
O7	0.42497 (16)	−0.2382 (2)	0.51123 (13)	0.0797 (6)
O8	0.53055 (17)	−0.0919 (6)	0.61993 (13)	0.1391 (13)
C8	0.32040 (12)	0.3581 (2)	0.13224 (11)	0.0384 (4)
N2	0.23464 (11)	0.4351 (2)	0.14327 (10)	0.0439 (4)
H2A	0.2350	0.4003	0.1921	0.066*
H2B	0.1798	0.3952	0.1030	0.066*
H2C	0.2369	0.5567	0.1414	0.066*
O2	0.56519 (10)	0.1375 (2)	0.10671 (9)	0.0554 (4)
C9	0.38580 (13)	0.2496 (3)	0.19357 (12)	0.0425 (4)
H9	0.3755	0.2252	0.2418	0.051*
C10	0.46634 (14)	0.1778 (3)	0.18263 (12)	0.0460 (4)
H10	0.5105	0.1038	0.2235	0.055*
C11	0.48208 (13)	0.2150 (3)	0.11122 (11)	0.0411 (4)
C12	0.41615 (15)	0.3222 (3)	0.04975 (12)	0.0518 (5)
H12	0.4260	0.3461	0.0014	0.062*
C13	0.33478 (15)	0.3940 (3)	0.06103 (12)	0.0503 (5)
H13	0.2900	0.4667	0.0200	0.060*
C14	0.58718 (17)	0.1818 (4)	0.03694 (15)	0.0594 (6)
H14A	0.6462	0.1195	0.0409	0.089*
H14B	0.5966	0.3116	0.0354	0.089*
H14C	0.5336	0.1444	−0.0127	0.089*
O1	0.67037 (11)	0.5777 (2)	0.15815 (8)	0.0509 (4)
C1	0.56621 (13)	0.5870 (2)	0.34540 (11)	0.0381 (4)
C4	0.63880 (13)	0.5719 (3)	0.22190 (11)	0.0396 (4)

C5	0.68716 (14)	0.4806 (3)	0.29573 (12)	0.0422 (4)
H5	0.7438	0.4135	0.3038	0.051*
N1	0.53081 (12)	0.6024 (3)	0.41235 (11)	0.0482 (4)
H1A	0.4758	0.6687	0.3952	0.072*
H1B	0.5766	0.6567	0.4558	0.072*
H1C	0.5185	0.4912	0.4268	0.072*
C2	0.51623 (14)	0.6757 (3)	0.27082 (12)	0.0459 (5)
H2	0.4588	0.7405	0.2625	0.055*
C6	0.65044 (14)	0.4896 (3)	0.35843 (12)	0.0426 (4)
H6	0.6831	0.4298	0.4086	0.051*
C3	0.55199 (14)	0.6674 (3)	0.20970 (12)	0.0467 (5)
H3	0.5182	0.7258	0.1594	0.056*
C7	0.76321 (18)	0.4981 (4)	0.17243 (15)	0.0588 (6)
H7A	0.7774	0.5105	0.1236	0.088*
H7B	0.7619	0.3706	0.1854	0.088*
H7C	0.8133	0.5591	0.2180	0.088*
N3	0.21447 (12)	0.9114 (2)	0.14231 (11)	0.0462 (4)
O3	0.27158 (11)	0.8115 (2)	0.19742 (10)	0.0577 (4)
O4	0.17418 (11)	1.0419 (2)	0.16528 (12)	0.0642 (5)
O5	0.19753 (18)	0.8822 (3)	0.07051 (12)	0.0925 (7)
O6	0.40423 (13)	0.0358 (3)	0.53394 (14)	0.0787 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N4	0.0599 (10)	0.0482 (10)	0.0424 (9)	0.0005 (9)	0.0262 (8)	0.0083 (8)
O7	0.1130 (15)	0.0479 (10)	0.0942 (14)	−0.0030 (10)	0.0575 (12)	−0.0063 (10)
O8	0.0827 (14)	0.259 (4)	0.0518 (11)	0.0282 (18)	0.0007 (11)	0.0389 (16)
C8	0.0372 (8)	0.0337 (9)	0.0392 (9)	−0.0009 (7)	0.0092 (7)	−0.0012 (7)
N2	0.0417 (8)	0.0402 (9)	0.0448 (8)	0.0024 (7)	0.0112 (7)	0.0001 (7)
O2	0.0528 (8)	0.0581 (10)	0.0563 (9)	0.0137 (7)	0.0223 (7)	0.0056 (7)
C9	0.0422 (9)	0.0421 (10)	0.0370 (9)	−0.0007 (8)	0.0085 (7)	0.0054 (8)
C10	0.0465 (10)	0.0422 (10)	0.0438 (10)	0.0081 (8)	0.0116 (8)	0.0084 (9)
C11	0.0403 (9)	0.0370 (10)	0.0427 (9)	0.0024 (7)	0.0124 (8)	0.0001 (8)
C12	0.0546 (11)	0.0610 (13)	0.0405 (10)	0.0101 (10)	0.0191 (9)	0.0117 (10)
C13	0.0478 (10)	0.0547 (12)	0.0419 (10)	0.0105 (9)	0.0104 (8)	0.0142 (9)
C14	0.0585 (12)	0.0624 (14)	0.0642 (13)	0.0047 (11)	0.0312 (11)	−0.0008 (12)
O1	0.0584 (8)	0.0552 (9)	0.0418 (7)	0.0062 (7)	0.0221 (6)	0.0036 (6)
C1	0.0399 (8)	0.0334 (9)	0.0393 (8)	−0.0047 (7)	0.0136 (7)	−0.0037 (7)
C4	0.0445 (9)	0.0329 (9)	0.0389 (9)	−0.0022 (7)	0.0134 (7)	−0.0021 (7)
C5	0.0430 (9)	0.0385 (10)	0.0444 (10)	0.0074 (8)	0.0161 (8)	0.0035 (8)
N1	0.0446 (8)	0.0523 (10)	0.0484 (9)	−0.0010 (7)	0.0189 (7)	−0.0013 (8)
C2	0.0388 (9)	0.0430 (10)	0.0501 (10)	0.0058 (8)	0.0107 (8)	0.0016 (9)
C6	0.0457 (9)	0.0372 (9)	0.0411 (9)	0.0049 (8)	0.0125 (8)	0.0055 (8)
C3	0.0475 (10)	0.0457 (11)	0.0400 (9)	0.0037 (9)	0.0095 (8)	0.0046 (8)
C7	0.0598 (12)	0.0687 (15)	0.0520 (12)	0.0045 (11)	0.0261 (10)	−0.0026 (11)
N3	0.0418 (8)	0.0362 (8)	0.0506 (9)	−0.0026 (7)	0.0070 (7)	0.0007 (7)
O3	0.0564 (8)	0.0490 (8)	0.0568 (9)	0.0122 (7)	0.0100 (7)	0.0069 (7)

O4	0.0537 (8)	0.0401 (8)	0.0936 (12)	0.0076 (7)	0.0230 (8)	−0.0016 (8)
O5	0.1142 (16)	0.0904 (15)	0.0516 (10)	−0.0027 (13)	0.0089 (10)	−0.0085 (10)
O6	0.0653 (10)	0.0539 (10)	0.1067 (14)	0.0002 (8)	0.0223 (10)	−0.0067 (10)

Geometric parameters (Å, º)

N4—O8	1.177 (3)	O1—C4	1.366 (2)
N4—O6	1.259 (3)	O1—C7	1.416 (3)
N4—O7	1.268 (2)	C1—C6	1.370 (3)
C8—C13	1.369 (3)	C1—C2	1.386 (3)
C8—C9	1.381 (2)	C1—N1	1.460 (3)
C8—N2	1.462 (2)	C4—C5	1.381 (3)
N2—H2A	0.8900	C4—C3	1.398 (3)
N2—H2B	0.8900	C5—C6	1.401 (3)
N2—H2C	0.8900	C5—H5	0.9300
O2—C11	1.378 (2)	N1—H1A	0.8900
O2—C14	1.417 (3)	N1—H1B	0.8900
C9—C10	1.376 (3)	N1—H1C	0.8900
C9—H9	0.9300	C2—C3	1.365 (3)
C10—C11	1.384 (3)	C2—H2	0.9300
C10—H10	0.9300	C6—H6	0.9300
C11—C12	1.379 (3)	C3—H3	0.9300
C12—C13	1.390 (3)	C7—H7A	0.9600
C12—H12	0.9300	C7—H7B	0.9600
C13—H13	0.9300	C7—H7C	0.9600
C14—H14A	0.9600	N3—O5	1.202 (3)
C14—H14B	0.9600	N3—O3	1.241 (2)
C14—H14C	0.9600	N3—O4	1.268 (2)
O8—N4—O6	119.3 (3)	C4—O1—C7	117.01 (16)
O8—N4—O7	129.4 (3)	C6—C1—C2	121.00 (18)
O6—N4—O7	111.25 (18)	C6—C1—N1	119.25 (16)
C13—C8—C9	120.71 (18)	C2—C1—N1	119.72 (17)
C13—C8—N2	119.57 (16)	O1—C4—C5	124.37 (17)
C9—C8—N2	119.72 (17)	O1—C4—C3	116.13 (17)
C8—N2—H2A	109.5	C5—C4—C3	119.50 (18)
C8—N2—H2B	109.5	C4—C5—C6	119.69 (17)
H2A—N2—H2B	109.5	C4—C5—H5	120.2
C8—N2—H2C	109.5	C6—C5—H5	120.2
H2A—N2—H2C	109.5	C1—N1—H1A	109.5
H2B—N2—H2C	109.5	C1—N1—H1B	109.5
C11—O2—C14	117.42 (16)	H1A—N1—H1B	109.5
C10—C9—C8	119.27 (18)	C1—N1—H1C	109.5
C10—C9—H9	120.4	H1A—N1—H1C	109.5
C8—C9—H9	120.4	H1B—N1—H1C	109.5
C9—C10—C11	120.42 (17)	C3—C2—C1	119.49 (17)
C9—C10—H10	119.8	C3—C2—H2	120.3
C11—C10—H10	119.8	C1—C2—H2	120.3

O2—C11—C12	124.06 (18)	C1—C6—C5	119.58 (17)
O2—C11—C10	115.76 (17)	C1—C6—H6	120.2
C12—C11—C10	120.17 (18)	C5—C6—H6	120.2
C11—C12—C13	119.17 (18)	C2—C3—C4	120.71 (18)
C11—C12—H12	120.4	C2—C3—H3	119.6
C13—C12—H12	120.4	C4—C3—H3	119.6
C8—C13—C12	120.25 (18)	O1—C7—H7A	109.5
C8—C13—H13	119.9	O1—C7—H7B	109.5
C12—C13—H13	119.9	H7A—C7—H7B	109.5
O2—C14—H14A	109.5	O1—C7—H7C	109.5
O2—C14—H14B	109.5	H7A—C7—H7C	109.5
H14A—C14—H14B	109.5	H7B—C7—H7C	109.5
O2—C14—H14C	109.5	O5—N3—O3	120.8 (2)
H14A—C14—H14C	109.5	O5—N3—O4	122.17 (19)
H14B—C14—H14C	109.5	O3—N3—O4	117.03 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.89	2.25	2.823 (3)	122
N1—H1*A*···O7^{ii}	0.89	2.52	2.979 (3)	113
N1—H1*B*···O6^{iii}	0.89	2.26	2.843 (3)	123
N1—H1*B*···O5^{iv}	0.89	2.12	2.903 (3)	146
N1—H1*C*···O7^{v}	0.89	2.14	2.935 (3)	148
N2—H2*A*···O3^{i}	0.89	2.08	2.967 (3)	177
N2—H2*A*···O4^{i}	0.89	2.55	3.187 (3)	129
N2—H2*B*···O6^{vi}	0.89	2.46	3.070 (3)	127
N2—H2*B*···O7^{vi}	0.89	2.22	3.083 (3)	163
N2—H2*C*···O3	0.89	2.07	2.891 (2)	152
C9—H9···O8^{v}	0.93	2.48	3.223 (3)	137

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