Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′,2″,2‴-[*N*,*N*,*N*′,*N*′-(3,6-Dioxaoctane-1,8-diyldinitrilo)tetramethylene]tetrakis(benzimidazolium) tetranitrate

Chun-Shan Zhou, Si Hu and Xiang-Gao Meng*

Department of Chemistry, Central China Normal University, Wuhan 430079, People's Republic of China
Correspondence e-mail: mengxianggao@mail.ccnu.edu.cn

Received 23 April 2007; accepted 25 April 2007

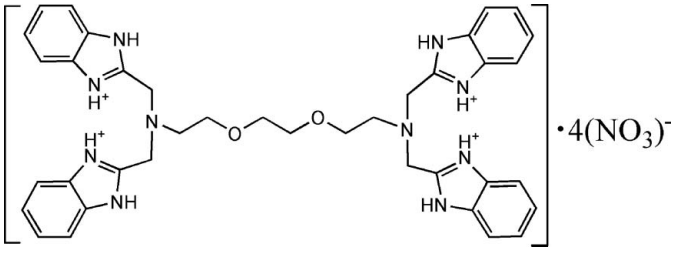
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.065; wR factor = 0.169; data-to-parameter ratio = 15.0.

The cation of the title compound, $C_{38}H_{44}N_{10}O_2^{4+}\cdot 4NO_3^-$, is centrosymmetric. In the crystal structure, cations and anions are linked into a three-dimensional framework by a combination of N—H···O and C—H···O hydrogen bonds and aromatic π–π stacking interactions.

Related literature

We have already published some crystal structures that are related to the title compound (Zhang *et al.*, 2005; Li *et al.*, 2005; Qiu *et al.*, 2005).

For related literature, see: Hendriks *et al.* (1982).



Experimental

Crystal data

$C_{38}H_{44}N_{10}O_2^{4+}\cdot 4NO_3^-$
M_r = 920.87
Monoclinic, $P2_1/c$
a = 8.4296 (6) Å
b = 17.6744 (12) Å
c = 14.279 (1) Å
β = 92.421 (1)°
V = 2125.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 294 (2) K
0.20 × 0.12 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.975, T_{max} = 0.989
23482 measured reflections
4642 independent reflections
2655 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.169
S = 1.03
4642 reflections
310 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H14···O7	0.93	2.58	3.364 (4)	142
C11—H11A···O1	0.97	2.43	3.094 (3)	125
C6—H6···O5	0.93	2.59	3.379 (4)	143
N4—H4···O4	0.82 (3)	2.31 (3)	2.995 (3)	142 (3)
N4—H4···O2	0.82 (3)	2.20 (3)	2.975 (4)	160 (3)
N5—H5···O6	0.86 (3)	1.87 (3)	2.719 (3)	174 (3)
N2—H2···O6	0.91 (3)	1.81 (3)	2.719 (3)	177 (3)
C7—H7···O7^{i}	0.93	2.53	3.443 (4)	166
N3—H3···O4^{ii}	0.87 (3)	1.93 (3)	2.789 (3)	169 (3)
N3—H3···O3^{ii}	0.87 (3)	2.51 (3)	3.180 (3)	134 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

This work was financially supported mostly by Key Fundamental Project No. 2002CCA00500.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2376).

References

Bruker (2001). *SAINT-Plus* (Version 6.45) and *SMART* (Version 5.628). Bruker AXS Inc., Madison, Wisconsin, USA.
Hendriks, H.-M.-J., Birker, J.-M.-W.-L., Van, R.-J., Verschoor, G.-C. & Reedijk, A. (1982). *J. Am. Chem. Soc.* **104**, 3607–3617.
Li, J., Meng, X.-G. & Liao, Z.-R. (2005). *Acta Cryst.* E**61**, o3421–o3423.
Qiu, J.-H., Liao, Z.-R., Meng, X.-G., Zhu, L., Wang, Z.-M. & Yu, K.-B. (2005). *Polyhedron*, **24**, 1617–1623.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Zhang, Y., Liao, Z.-R., Li, D.-F. & Meng, X.-G. (2005). *Acta Cryst.* E**61**, o3396–o3397.

  doi:10.1107/S1600536807020764

supplementary materials

Acta Cryst. (2007). E**63**, o2800 [**doi:10.1107/S1600536807020764**]

2,2',2'',2'''-[*N*,*N*,*N*',*N*'-(3,6-Dioxaoctane-1,8-diyldinitrilo)tetramethylene]tetrakis(benzimidazolium) tetranitrate

C.-S. Zhou, S. Hu and X.-G. Meng

Comment

As part of our continuing studies on the ligands or metal complexes containing multi-benzimidazole groups (Zhang *et al.*, 2005; Li *et al.*, 2005; Qiu *et al.*, 2005), we report here the crystal structure of a related compound, (I), which was obtained unexpectedly by reacting μ$_2$-*N*,*N*, *N*', *N*'-tetrakis(Benzimidazol-2-ylmethyl)- 3,6- dioxaoctane-1,8-diamine (EGTB) with Fe(NO_3).6H_2O in H_2O solution.

In the aymmetric unit of (I) (Fig. 1), the cation lies across an inversion center in space group of $P2_1/c$. The two terminal benzimidazoles rings on one side of the cation are effectively planar with the dihedral angle of only 0.75 (1)°.

All imine N atoms in the benzimidazole groups of are protonated foming a tetracation. Two nitrate anions lie to the inner side of the two end-on benzimidazole groups, forming the four nearly symmetric intra-molecular hydrogen bonds (Table 1). However, the other two anions lie at the outside of the two benzimidazole groups, linking the adjacent cations into a two-dimensional network running parallel the (100) direction. These networks are further joined by the C7–H···O7^{i} (symmetry code as in Table 1) hydrogen bonds, forming a three-dimensional framework (Fig.2). In addition, the supramolecular aggregation is augmented by π-π stacking interactions between the aromatic rings C5—C10 and C13—C18, respectively. The two phenyl rings which lie in the molecules at (*x*,*y*,*z*) and (-*x*,1 - *y*,-*z*), respectively, are almost parallel with the dihedral angle of only 1.36 (1)°, the ring centroid separation of 3.682 (2) Å and the interpalanar spacing of *ca* 3.404 Å.

Experimental

All reagents and solvents were used as obtained without further purification. EGTB [EGTB = (μ$_2$-*N*,*N*,*N*',*N*'-tetrakis(Benzimidazol-2-ylmethyl)-3,6-dioxaoctane-1,8-diamine] was prepared according to literature procedure (Hendriks *et al.*, 1982). The title organic salt was obtained unexpectedly by reacting EGTB with Fe(NO_3).6H_2O (molar ratio: 1/2). The mixture was stirred for half an hour at 70° and then filtered. The resulting pale-yellow solution was kept in air for one week. Crystals of (I) suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation of the solution at the bottom of the vessel.

Refinement

All H atoms bonded to carbon atoms were placed in calculated positions with C–H=0.97 Å(methylene) and 0.93 Å(aromatic), U_{iso}(H) = 1.2U_{eq}(C). H atoms bonded to imine N atoms were located from the difference maps and the N—H distance were refined freely without any constraints, but their U_{iso}(H) values were set 1.2 times U_{eq} of their carrier atoms.

Figures

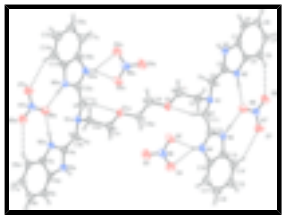


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Atoms marked with suffix 'a' are related by the symmetry operator (1 - *x*, 1 - *y*, 1 - *z*).

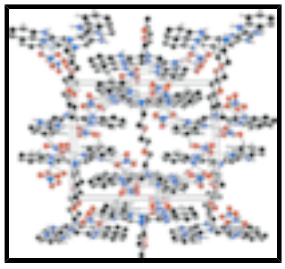


Fig. 2. Part of the crystal structure of (I), showing the formation of the three-dimensional network. Hydrogen bonding are shown as dashed lines. For the sake of clarity, H atoms not involved in the motif have been omitted.

2,2',2'',2'''-[N,*N*,*N*',*N*'-(3,6-Dioxaoctane-1,8- diyldinitrilo)tetramethylene]tetrakis(benzimidazolium) tetranitrate

Crystal data

$C_{38}H_{44}N_{10}O_2^{4+}\cdot 4(NO_3^-)$	F_{000} = 964
M_r = 920.87	D_x = 1.439 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2223 reflections
a = 8.4296 (6) Å	θ = 2.4–19.9º
b = 17.6744 (12) Å	μ = 0.11 mm^{-1}
c = 14.279 (1) Å	*T* = 294 (2) K
β = 92.421 (1)º	Block, colourless
V = 2125.5 (3) Å^3	0.20 × 0.12 × 0.10 mm
Z = 2	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4642 independent reflections
Radiation source: fine focus sealed Siemens Mo tube	2655 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.067
T = 298(2) K	θ_{max} = 27.0º
0.3° wide ω exposures scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	*h* = −10→10
T_{min} = 0.975, T_{max} = 0.989	*k* = −19→22
23482 measured reflections	*l* = −18→18

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0744P)^2 + 0.3238P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.065$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.169$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.03$	$\Delta\rho_{min} = -0.18$ e Å^{-3}
4642 reflections	Extinction correction: none
310 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2$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}
C1	0.2091 (3)	0.53564 (19)	0.38291 (19)	0.0604 (8)
H1A	0.2269	0.5895	0.3759	0.072*
H1B	0.1398	0.5284	0.4347	0.072*
C2	0.1299 (3)	0.50482 (16)	0.29426 (18)	0.0481 (7)
H2A	0.1247	0.4502	0.3006	0.058*
H2B	0.0214	0.5233	0.2911	0.058*
C3	0.2078 (3)	0.60132 (15)	0.1804 (2)	0.0516 (7)
H3A	0.2777	0.6079	0.1287	0.062*
H3B	0.2532	0.6287	0.2339	0.062*
C4	0.0490 (3)	0.63386 (16)	0.15382 (17)	0.0439 (6)
C5	−0.2006 (3)	0.64709 (15)	0.10256 (16)	0.0402 (6)
C6	−0.3564 (3)	0.63661 (16)	0.06912 (18)	0.0482 (7)
H6	−0.3976	0.5887	0.0562	0.058*
C7	−0.4458 (3)	0.70087 (19)	0.05621 (18)	0.0538 (8)
H7	−0.5505	0.6964	0.0337	0.065*

C8	−0.3840 (3)	0.77308 (17)	0.07599 (19)	0.0550 (8)
H8	−0.4491	0.8152	0.0670	0.066*
C9	−0.2306 (3)	0.78310 (16)	0.10818 (18)	0.0510 (7)
H9	−0.1892	0.8310	0.1205	0.061*
C10	−0.1396 (3)	0.71858 (15)	0.12152 (17)	0.0420 (6)
C11	0.3475 (3)	0.48165 (16)	0.1873 (2)	0.0505 (7)
H11A	0.4223	0.4907	0.2395	0.061*
H11B	0.3932	0.5013	0.1310	0.061*
C12	0.3205 (3)	0.39871 (16)	0.17669 (17)	0.0453 (7)
C13	0.2109 (3)	0.28778 (17)	0.14316 (17)	0.0481 (7)
C14	0.1108 (4)	0.22818 (18)	0.11782 (19)	0.0564 (8)
H14	0.0065	0.2361	0.0964	0.068*
C15	0.1746 (4)	0.15673 (19)	0.1262 (2)	0.0666 (9)
H15	0.1120	0.1153	0.1088	0.080*
C16	0.3291 (5)	0.1445 (2)	0.1598 (2)	0.0759 (10)
H16	0.3664	0.0951	0.1657	0.091*
C17	0.4284 (4)	0.2034 (2)	0.1844 (2)	0.0676 (9)
H17	0.5324	0.1952	0.2063	0.081*
C18	0.3663 (3)	0.27573 (17)	0.17529 (18)	0.0508 (7)
C19	0.4248 (3)	0.52156 (19)	0.49117 (19)	0.0605 (8)
H19A	0.3520	0.5113	0.5404	0.073*
H19B	0.4468	0.5754	0.4907	0.073*
N1	0.1992 (2)	0.52134 (12)	0.20352 (14)	0.0427 (5)
N2	−0.0788 (2)	0.59601 (14)	0.12376 (15)	0.0450 (6)
H2	−0.084 (3)	0.5449 (17)	0.1178 (18)	0.054*
N3	0.0169 (3)	0.70765 (14)	0.15369 (15)	0.0465 (6)
H3	0.085 (3)	0.7437 (16)	0.1678 (18)	0.056*
N4	0.4296 (3)	0.34628 (16)	0.19490 (16)	0.0548 (7)
H4	0.516 (4)	0.3587 (17)	0.218 (2)	0.066*
N5	0.1875 (3)	0.36534 (14)	0.14572 (15)	0.0456 (6)
H5	0.106 (3)	0.3912 (16)	0.1281 (19)	0.055*
N6	0.7793 (3)	0.37255 (17)	0.32131 (19)	0.0621 (7)
O1	0.3557 (2)	0.49900 (11)	0.40309 (12)	0.0566 (5)
O2	0.6974 (3)	0.42656 (14)	0.29412 (17)	0.0876 (8)
O3	0.8884 (3)	0.37929 (14)	0.38036 (19)	0.0902 (8)
O4	0.7453 (3)	0.30882 (15)	0.28733 (18)	0.0908 (8)
N7	−0.2022 (3)	0.41060 (15)	0.06511 (16)	0.0546 (6)
O5	−0.3230 (2)	0.44713 (13)	0.04530 (17)	0.0787 (7)
O6	−0.0836 (2)	0.44321 (12)	0.10178 (17)	0.0766 (7)
O7	−0.1903 (3)	0.34285 (14)	0.0487 (2)	0.0942 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0578 (18)	0.075 (2)	0.0474 (17)	0.0142 (16)	−0.0083 (13)	−0.0031 (15)
C2	0.0402 (14)	0.0546 (18)	0.0488 (16)	0.0041 (13)	−0.0059 (12)	0.0025 (13)
C3	0.0405 (14)	0.0479 (18)	0.0652 (18)	−0.0094 (12)	−0.0127 (13)	0.0071 (14)
C4	0.0427 (14)	0.0443 (18)	0.0442 (15)	−0.0071 (12)	−0.0053 (11)	0.0033 (12)

C5	0.0433 (14)	0.0412 (16)	0.0360 (13)	0.0016 (12)	0.0003 (11)	0.0054 (11)
C6	0.0455 (14)	0.0530 (18)	0.0455 (15)	−0.0055 (13)	−0.0057 (12)	0.0012 (13)
C7	0.0441 (15)	0.070 (2)	0.0469 (16)	0.0052 (15)	−0.0040 (12)	0.0087 (14)
C8	0.0589 (18)	0.055 (2)	0.0504 (17)	0.0157 (15)	−0.0009 (14)	0.0098 (14)
C9	0.0621 (18)	0.0461 (18)	0.0447 (16)	0.0003 (14)	0.0032 (13)	−0.0003 (13)
C10	0.0459 (14)	0.0434 (17)	0.0364 (14)	0.0002 (12)	−0.0033 (11)	0.0027 (11)
C11	0.0367 (14)	0.062 (2)	0.0527 (17)	−0.0006 (13)	−0.0044 (12)	0.0033 (14)
C12	0.0409 (14)	0.0554 (19)	0.0395 (14)	0.0031 (13)	−0.0007 (11)	0.0018 (13)
C13	0.0538 (16)	0.055 (2)	0.0356 (14)	0.0063 (14)	0.0035 (12)	0.0021 (13)
C14	0.0645 (18)	0.058 (2)	0.0470 (16)	−0.0045 (16)	0.0040 (14)	−0.0006 (14)
C15	0.092 (2)	0.052 (2)	0.0566 (19)	−0.0009 (18)	0.0123 (17)	0.0033 (15)
C16	0.111 (3)	0.059 (2)	0.058 (2)	0.021 (2)	0.005 (2)	0.0032 (17)
C17	0.075 (2)	0.072 (2)	0.0546 (19)	0.0264 (19)	−0.0056 (16)	0.0036 (17)
C18	0.0571 (17)	0.056 (2)	0.0393 (15)	0.0119 (15)	0.0001 (12)	−0.0011 (13)
C19	0.0645 (19)	0.070 (2)	0.0456 (17)	0.0011 (15)	−0.0152 (14)	−0.0014 (14)
N1	0.0360 (11)	0.0454 (14)	0.0460 (13)	−0.0016 (10)	−0.0062 (9)	0.0050 (10)
N2	0.0433 (12)	0.0399 (13)	0.0507 (13)	−0.0031 (11)	−0.0087 (10)	0.0027 (11)
N3	0.0482 (13)	0.0407 (15)	0.0499 (13)	−0.0080 (10)	−0.0078 (10)	−0.0014 (11)
N4	0.0441 (13)	0.0697 (18)	0.0498 (14)	0.0114 (13)	−0.0078 (11)	0.0008 (12)
N5	0.0409 (12)	0.0483 (16)	0.0469 (13)	0.0074 (11)	−0.0052 (10)	−0.0007 (11)
N6	0.0457 (14)	0.066 (2)	0.0738 (18)	0.0063 (14)	−0.0011 (13)	0.0094 (15)
O1	0.0544 (11)	0.0675 (14)	0.0464 (11)	0.0091 (10)	−0.0150 (9)	−0.0071 (9)
O2	0.0809 (16)	0.0818 (18)	0.0986 (18)	0.0335 (14)	−0.0129 (14)	0.0038 (14)
O3	0.0605 (14)	0.0913 (19)	0.116 (2)	−0.0012 (13)	−0.0352 (14)	0.0062 (15)
O4	0.0764 (16)	0.0726 (18)	0.121 (2)	0.0040 (14)	−0.0302 (15)	−0.0099 (15)
N7	0.0473 (14)	0.0551 (17)	0.0602 (15)	−0.0020 (12)	−0.0134 (11)	−0.0018 (12)
O5	0.0533 (12)	0.0746 (16)	0.1052 (18)	0.0111 (11)	−0.0310 (12)	−0.0085 (13)
O6	0.0480 (12)	0.0495 (13)	0.129 (2)	−0.0029 (10)	−0.0305 (12)	−0.0134 (12)
O7	0.0891 (17)	0.0481 (15)	0.141 (2)	0.0016 (13)	−0.0459 (16)	−0.0181 (15)

Geometric parameters (Å, °)

C1—O1	1.414 (3)	C12—N4	1.323 (3)
C1—C2	1.508 (4)	C12—N5	1.326 (3)
C1—H1A	0.9700	C13—N5	1.386 (4)
C1—H1B	0.9700	C13—C18	1.386 (4)
C2—N1	1.473 (3)	C13—C14	1.388 (4)
C2—H2A	0.9700	C14—C15	1.376 (4)
C2—H2B	0.9700	C14—H14	0.9300
C3—N1	1.454 (3)	C15—C16	1.385 (5)
C3—C4	1.491 (3)	C15—H15	0.9300
C3—H3A	0.9700	C16—C17	1.374 (5)
C3—H3B	0.9700	C16—H16	0.9300
C4—N2	1.324 (3)	C17—C18	1.385 (4)
C4—N3	1.332 (3)	C17—H17	0.9300
C5—C10	1.387 (4)	C18—N4	1.380 (4)
C5—C6	1.391 (3)	C19—O1	1.421 (3)
C5—N2	1.391 (3)	C19—C19^{i}	1.491 (6)
C6—C7	1.371 (4)	C19—H19A	0.9700

C6—H6	0.9300	C19—H19B	0.9700
C7—C8	1.403 (4)	N2—H2	0.91 (3)
C7—H7	0.9300	N3—H3	0.87 (3)
C8—C9	1.366 (4)	N4—H4	0.82 (3)
C8—H8	0.9300	N5—H5	0.86 (3)
C9—C10	1.383 (4)	N6—O3	1.227 (3)
C9—H9	0.9300	N6—O2	1.231 (3)
C10—N3	1.392 (3)	N6—O4	1.255 (3)
C11—N1	1.460 (3)	N7—O7	1.225 (3)
C11—C12	1.490 (4)	N7—O5	1.228 (3)
C11—H11A	0.9700	N7—O6	1.249 (3)
C11—H11B	0.9700		
O1—C1—C2	111.0 (2)	C18—C13—C14	121.7 (3)
O1—C1—H1A	109.4	C15—C14—C13	116.3 (3)
C2—C1—H1A	109.4	C15—C14—H14	121.9
O1—C1—H1B	109.4	C13—C14—H14	121.9
C2—C1—H1B	109.4	C14—C15—C16	122.1 (3)
H1A—C1—H1B	108.0	C14—C15—H15	118.9
N1—C2—C1	119.3 (2)	C16—C15—H15	118.9
N1—C2—H2A	107.5	C17—C16—C15	121.6 (3)
C1—C2—H2A	107.5	C17—C16—H16	119.2
N1—C2—H2B	107.5	C15—C16—H16	119.2
C1—C2—H2B	107.5	C16—C17—C18	116.9 (3)
H2A—C2—H2B	107.0	C16—C17—H17	121.6
N1—C3—C4	112.3 (2)	C18—C17—H17	121.6
N1—C3—H3A	109.1	N4—C18—C17	132.3 (3)
C4—C3—H3A	109.1	N4—C18—C13	106.3 (2)
N1—C3—H3B	109.1	C17—C18—C13	121.4 (3)
C4—C3—H3B	109.1	O1—C19—C19^{i}	108.7 (3)
H3A—C3—H3B	107.9	O1—C19—H19A	110.0
N2—C4—N3	109.4 (2)	C19^{i}—C19—H19A	110.0
N2—C4—C3	126.6 (3)	O1—C19—H19B	110.0
N3—C4—C3	124.0 (2)	C19^{i}—C19—H19B	110.0
C10—C5—C6	121.7 (2)	H19A—C19—H19B	108.3
C10—C5—N2	106.5 (2)	C3—N1—C11	112.3 (2)
C6—C5—N2	131.8 (2)	C3—N1—C2	114.7 (2)
C7—C6—C5	116.2 (3)	C11—N1—C2	114.7 (2)
C7—C6—H6	121.9	C4—N2—C5	109.0 (2)
C5—C6—H6	121.9	C4—N2—H2	124.5 (17)
C6—C7—C8	121.9 (3)	C5—N2—H2	126.5 (17)
C6—C7—H7	119.0	C4—N3—C10	109.0 (2)
C8—C7—H7	119.0	C4—N3—H3	125.6 (18)
C9—C8—C7	121.6 (3)	C10—N3—H3	125.2 (18)
C9—C8—H8	119.2	C12—N4—C18	109.5 (2)
C7—C8—H8	119.2	C12—N4—H4	119 (2)
C8—C9—C10	116.8 (3)	C18—N4—H4	131 (2)
C8—C9—H9	121.6	C12—N5—C13	109.3 (2)
C10—C9—H9	121.6	C12—N5—H5	121.4 (19)

C9—C10—C5	121.7 (2)	C13—N5—H5	129.3 (19)
C9—C10—N3	132.2 (3)	O3—N6—O2	122.4 (3)
C5—C10—N3	106.1 (2)	O3—N6—O4	120.3 (3)
N1—C11—C12	111.2 (2)	O2—N6—O4	117.3 (3)
N1—C11—H11A	109.4	C1—O1—C19	112.0 (2)
C12—C11—H11A	109.4	N6—O2—H4	95.9 (8)
N1—C11—H11B	109.4	N6—O4—H4	89.8 (8)
C12—C11—H11B	109.4	O7—N7—O5	122.9 (2)
H11A—C11—H11B	108.0	O7—N7—O6	117.4 (2)
N4—C12—N5	108.9 (3)	O5—N7—O6	119.6 (3)
N4—C12—C11	124.6 (2)	N7—O6—H2	120.4 (9)
N5—C12—C11	126.5 (2)	N7—O6—H5	121.3 (8)
N5—C13—C18	106.1 (2)	H2—O6—H5	117.9 (12)
N5—C13—C14	132.2 (3)		
O1—C1—C2—N1	−69.8 (3)	C12—C11—N1—C2	67.7 (3)
N1—C3—C4—N2	−21.2 (4)	C1—C2—N1—C3	−60.6 (3)
N1—C3—C4—N3	161.5 (2)	C1—C2—N1—C11	71.7 (3)
C10—C5—C6—C7	−0.1 (4)	N3—C4—N2—C5	0.2 (3)
N2—C5—C6—C7	179.7 (3)	C3—C4—N2—C5	−177.4 (3)
C5—C6—C7—C8	−0.3 (4)	C10—C5—N2—C4	0.0 (3)
C6—C7—C8—C9	0.8 (4)	C6—C5—N2—C4	−179.9 (3)
C7—C8—C9—C10	−0.8 (4)	N2—C4—N3—C10	−0.3 (3)
C8—C9—C10—C5	0.4 (4)	C3—C4—N3—C10	177.4 (2)
C8—C9—C10—N3	−179.2 (3)	C9—C10—N3—C4	179.9 (3)
C6—C5—C10—C9	0.0 (4)	C5—C10—N3—C4	0.3 (3)
N2—C5—C10—C9	−179.9 (2)	N5—C12—N4—C18	0.0 (3)
C6—C5—C10—N3	179.7 (2)	C11—C12—N4—C18	−178.4 (2)
N2—C5—C10—N3	−0.1 (3)	C17—C18—N4—C12	−178.0 (3)
N1—C11—C12—N4	−154.4 (2)	C13—C18—N4—C12	0.3 (3)
N1—C11—C12—N5	27.5 (4)	N4—C12—N5—C13	−0.3 (3)
N5—C13—C14—C15	−178.5 (3)	C11—C12—N5—C13	178.1 (2)
C18—C13—C14—C15	−0.1 (4)	C18—C13—N5—C12	0.5 (3)
C13—C14—C15—C16	1.2 (4)	C14—C13—N5—C12	179.0 (3)
C14—C15—C16—C17	−1.6 (5)	C2—C1—O1—C19	−174.0 (2)
C15—C16—C17—C18	0.8 (5)	$C19^{i}$—C19—O1—C1	177.9 (3)
C16—C17—C18—N4	178.5 (3)	O3—N6—O2—H4	−169.1 (8)
C16—C17—C18—C13	0.3 (4)	O4—N6—O2—H4	9.7 (8)
N5—C13—C18—N4	−0.5 (3)	O3—N6—O4—H4	169.7 (8)
C14—C13—C18—N4	−179.2 (2)	O2—N6—O4—H4	−9.1 (8)
N5—C13—C18—C17	178.1 (2)	O7—N7—O6—H2	173.8 (10)
C14—C13—C18—C17	−0.7 (4)	O5—N7—O6—H2	−4.6 (10)
C4—C3—N1—C11	153.5 (2)	O7—N7—O6—H5	1.3 (10)
C4—C3—N1—C2	−73.1 (3)	O5—N7—O6—H5	−177.1 (10)
C12—C11—N1—C3	−158.9 (2)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

C14—H14···O7	0.93	2.58	3.364 (4)	142
C11—H11A···O1	0.97	2.43	3.094 (3)	125
C6—H6···O5	0.93	2.59	3.379 (4)	143
N4—H4···O4	0.82 (3)	2.31 (3)	2.995 (3)	142 (3)
N4—H4···O2	0.82 (3)	2.20 (3)	2.975 (4)	160 (3)
N5—H5···O6	0.86 (3)	1.87 (3)	2.719 (3)	174 (3)
N2—H2···O6	0.91 (3)	1.81 (3)	2.719 (3)	177 (3)
C7—H7···O7^{ii}	0.93	2.53	3.443 (4)	166
N3—H3···O4^{iii}	0.87 (3)	1.93 (3)	2.789 (3)	169 (3)
N3—H3···O3^{iii}	0.87 (3)	2.51 (3)	3.180 (3)	134 (2)

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

Fig. 1

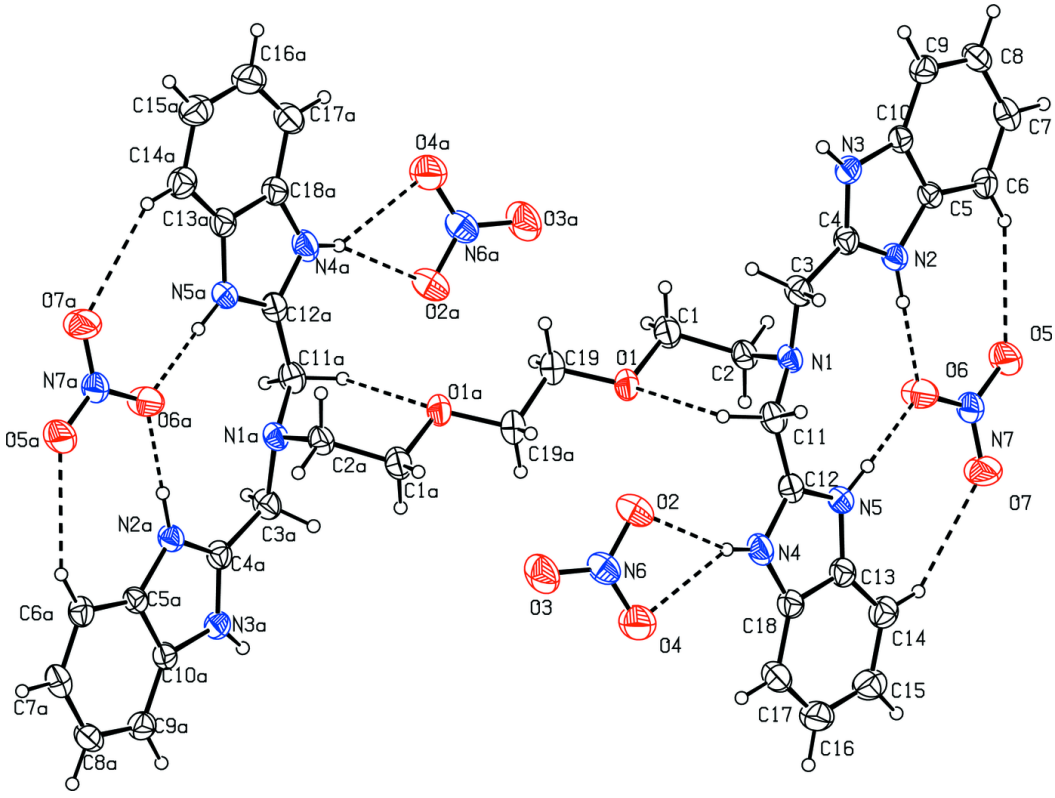

C16a
C15a
C17a
C14a
C18a
C13a
O4a
O3a
N6a
N4a
O2a
O7a
N5a
C12a
C11a
N7a
O1a
C19
O1
C1
C2
N1
O6a
O5a
N1a
C2a
C19a
C1a
C11
C12
O6
O5
N7
O7
N2a
C3a
O2
N5
C3
C4
N2
C4a
N6
N4
C13
C5a
C6a
O3
O4
C18
C14
N3a
C10a
C15
C7a
C9a
C17
C8a
C16
C9
C8
C10
C7
N3
C6
C5

Fig. 2