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N,*N*′-Bis(3,4-methenedioxybenzyl)-ethane-1,2-diammonium dinitrate: hydrogen-bonded chains of rings

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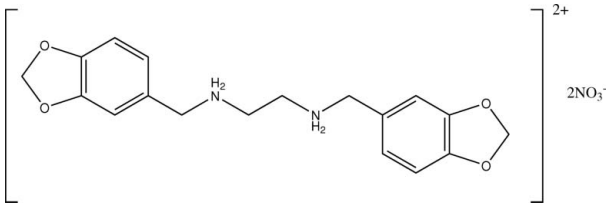
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.053; wR factor = 0.165; data-to-parameter ratio = 12.5.

In the title compound, $C_{18}H_{22}N_2O_4^{2+}\cdot 2NO_3^-$, the cation is centrosymmetric. The asymmetric unit consists of one half-cation and one anion. The molecules are linked by N—H···O and C—H···O hydrogen bonds and by dipolar O—C···O—C interactions, forming a chain of edge-fused $R_4^4(18)$ rings and a chain of $R_2^2(4)$ rings.

Related literature

For related literature, see: Bernstein *et al.* (1995); Bondi (1964).



Experimental

Crystal data

$C_{18}H_{22}N_2O_4^{2+}\cdot 2NO_3^-$
M_r = 454.40
Monoclinic, $C2/c$
a = 33.590 (3) Å
b = 5.6421 (8) Å
c = 11.4747 (15) Å
β = 107.326 (2)°
V = 2076.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.35 × 0.18 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.959, T_{max} = 0.989
4705 measured reflections
1822 independent reflections
1018 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.165
S = 1.07
1822 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1a···O3	0.90	1.89	2.787 (3)	175
N1—H1b···O4^{i}	0.90	1.98	2.876 (4)	173
N1—H1a···O5	0.90	2.56	3.180 (4)	127
C1—H1d···O5^{ii}	0.97	2.61	3.291 (4)	128
N1—H1b···O3^{i}	0.90	2.47	3.032 (4)	121
C1—H1d···O3^{i}	0.97	2.63	3.170 (4)	115
C2—H2b···O4^{iii}	0.97	2.42	3.302 (4)	151
C1—H1c···O4^{iii}	0.97	2.51	3.340 (4)	144

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2368).

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supplementary materials

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N,*N*'-Bis(3,4-methenedioxybenzyl)ethane-1,2-diammonium dinitrate: hydrogen-bonded chains of rings

S.-P. Yang, D.-Q. Wang, L.-J. Han and H.-T. Xia

Comment

The crystal of the compound (I), was obtained unexpectedly in the prepartion of a lanthanum complex with *N*,*N*'-bis (3,4-methenedioxybenzyl)ethane-1,2-diamine. Here, we report the crystal structure and supramolecular arrangement of (I), (Fig.1).

In the supramolecular arrangement of (I), the reference molecule is selected to lie across a center of inversion (1/2, 0, 0) in monoclinic space group *C*2/*c*, the asymmetric unit contains of one half–cation and one anion, cation and anion are linked by N—H···O [H1···O3 = 1.89 (4) Å, N1—H1A···O3 =175 °] hydrogen bonds (Table1).

In the crystal structure of (I), the molecules are linked by N—H···O [H1b···O4^{ii} = 1.98 Å, N1—H1b···O4^{ii} = 173°, symmetry codes: (ii) x, $-1+y$, z] hydrogen bonds, the amino N1 atoms at (x, y, z) and ($1-x$, $-y$, $-z$) in the molecules centred at (1/2, 0, 0) act as hydrogen-bond donors, *via* H1b, to the nitrate O4 atoms at (x, $-1+y$, z) and ($1-x$, $1-y$, $-z$), respectively, which themselves are parts of the molecules centred at (1/2, −1, 0) and (1/2, 1, 0), respectively (Fig. 2). Propagation by translation of this two hydrogen bonds along [010] direction then generates a chain of edge-fused $R_4^4(18)$ rings (Bernstein *et al.*, 1995) running along (1/2,*y*,0) axis (Fig. 2).

The molecules are linked by dipolar O—C···O—C [C9—O2^{i} = 3.101 (5) Å, O2—C9···O2^{i} = 77.5 (2)°; symmetry codes: (i) $1/2-x$, $3/2-y$, $-z$] interactions (Bondi, 1964) into a chain of $R_2^2(4)$ rings, the atom C9 at (x, y, z) in the cation centred at (1/2, 0, 0) act as donor to O2 at ($1/2-x$, $3/2-y$, $-z$) in the cation centred at (0, 3/2, 0), generating a chain of $R_2^2(4)$ rings along [1 $\bar{3}$ 0] (Fig. 3). The combination of the [0 1 0] chain and the [1 $\bar{3}$ 0] chain generates a [0 0 1] sheet. Adjacent sheets were linked by the further N—H···O and C—H···O hydrogen bonds and the crystal sturecture was stabilized (Table. 1).

Experimental

To a solution containing *N*,*N*'-bis(4-chlorobenzyl)ethane-1,2-diamine (1.71 g, 5 mmol) and ethanol 30 ml, a solution of lanthanum nitrate (1.08 g, 5 mmol) and methanol (10 ml) was added with stirring for 3 h at 333 K, and then the white solid obtained was filtered off, washed with ethanol in proper order and dried at room temperture. Colourless crystal of (I) suitatble for X-ray structure analysis were obtained by slow evaporation from the solution of DMF-EtOH(1:3) over a period of one month (m.p. 501 − 503 K).

Refinement

All H atoms were positioned geometrically and refined as riding on their parent atoms, with N—H = 0.90 Å, C—H = 0.93 − 0.97Å and U_{iso}(H) = 1.2U_{eq}(C, N) for all H atoms.

Figures

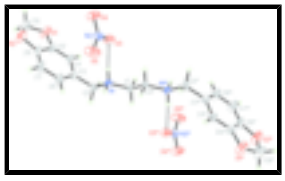

Fig. 1. The molecule of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry position: (*) 1 − *x*, −*y*, −*z*].

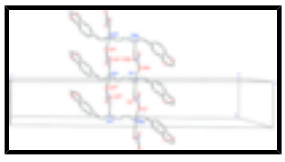

Fig. 2. A view of part of the crystal structure of (I), showing the formation of a chain of edge-fused $R_4^4(18)$ rings along [010]. For the sake of clarity, H atoms not involved in the motif shown have been omitted·[Symmetry position: (*) 1 − *x*, −*y*, −*z*; (#) 1 − *x*, 1 − *y*, −*z*; ($) 1 − *x*, −1 − *y*, −*z*; (&) *x*, −1 + *y*, *z*; (@) *z*, 1 + *y*, *z*].

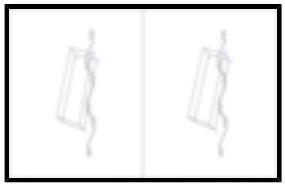

Fig. 3. A stereoview of part of the crystal structure of (I), showing the formation of a chain $R_2^2(4)$ rings along [1 $\bar{3}$ 0] by dipolar O—C···O—C interactions. For the sake of clarity, H atoms and nitrate not involved in the motif shown have been omitted.

N,*N*'-Bis(3,4-methenedioxybenzyl)ethane-1,2-diammonium dinitrate

Crystal data

$C_{18}H_{22}N_2O_4^{2+}\cdot 2NO_3^-$	F_{000} = 952
M_r = 454.40	D_x = 1.454 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 501 K
Hall symbol: -C 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 33.590 (3) Å	Cell parameters from 1077 reflections
b = 5.6421 (8) Å	θ = 2.5–21.3º
c = 11.4747 (15) Å	μ = 0.12 mm^{-1}
β = 107.326 (2)º	*T* = 298 (2) K
V = 2076.0 (4) Å^3	Needle, colourless
Z = 4	0.35 × 0.18 × 0.09 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer	1822 independent reflections
Radiation source: fine-focus sealed tube	1018 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −39→38
T_{min} = 0.959, T_{max} = 0.989	*k* = −6→5
4705 measured reflections	*l* = −10→13

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.053$	$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 2.9489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.165$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
1822 reflections	$\Delta\rho_{min} = -0.21$ e Å^{-3}
146 parameters	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0061 (10)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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}
N1	0.44318 (7)	0.0350 (5)	−0.0179 (2)	0.0396 (7)
H1A	0.4439	0.1890	0.0031	0.048*
H1B	0.4436	−0.0512	0.0485	0.048*
O1	0.29689 (9)	0.6277 (5)	−0.1979 (3)	0.0838 (10)
O2	0.27695 (9)	0.5442 (6)	−0.0269 (3)	0.0860 (10)
C1	0.48103 (9)	−0.0207 (6)	−0.0538 (3)	0.0411 (8)
H1C	0.4822	0.0796	−0.1214	0.049*
H1D	0.4802	−0.1846	−0.0799	0.049*
C2	0.40307 (10)	−0.0125 (6)	−0.1155 (3)	0.0484 (9)
H2A	0.3969	−0.1807	−0.1183	0.058*
H2B	0.4057	0.0339	−0.1943	0.058*
C3	0.36803 (10)	0.1246 (6)	−0.0901 (3)	0.0452 (9)
C4	0.35020 (11)	0.3134 (6)	−0.1661 (3)	0.0512 (9)
H4	0.3588	0.3530	−0.2336	0.061*
C5	0.31951 (11)	0.4381 (7)	−0.1368 (3)	0.0539 (10)
C6	0.30722 (11)	0.3863 (8)	−0.0360 (4)	0.0582 (10)

C7	0.32454 (13)	0.2067 (8)	0.0397 (4)	0.0753 (13)
H7	0.3163	0.1731	0.1085	0.090*
C8	0.35531 (12)	0.0730 (8)	0.0106 (4)	0.0667 (12)
H8	0.3675	−0.0536	0.0604	0.080*
C9	0.26771 (15)	0.6847 (10)	−0.1333 (4)	0.0871 (15)
H9A	0.2698	0.8514	−0.1117	0.105*
H9B	0.2395	0.6532	−0.1843	0.105*
N2	0.44342 (9)	0.5341 (5)	0.1477 (3)	0.0540 (8)
O4	0.43900 (10)	0.7387 (5)	0.1821 (2)	0.0763 (9)
O3	0.45031 (9)	0.5127 (4)	0.0471 (2)	0.0678 (8)
O5	0.44070 (12)	0.3594 (6)	0.2065 (3)	0.0978 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0423 (15)	0.0344 (15)	0.0444 (15)	0.0047 (12)	0.0163 (12)	−0.0011 (12)
O1	0.096 (2)	0.084 (2)	0.080 (2)	0.0439 (18)	0.0395 (17)	0.0151 (17)
O2	0.0779 (19)	0.104 (2)	0.088 (2)	0.0396 (19)	0.0425 (17)	0.0074 (19)
C1	0.0415 (17)	0.0417 (19)	0.0437 (19)	0.0037 (15)	0.0182 (14)	−0.0014 (16)
C2	0.0429 (19)	0.052 (2)	0.049 (2)	0.0025 (17)	0.0108 (16)	−0.0046 (17)
C3	0.0398 (18)	0.053 (2)	0.0442 (19)	0.0016 (17)	0.0147 (15)	−0.0011 (17)
C4	0.055 (2)	0.052 (2)	0.051 (2)	0.0077 (19)	0.0211 (17)	0.0013 (18)
C5	0.050 (2)	0.054 (2)	0.054 (2)	0.0115 (19)	0.0100 (18)	0.0003 (19)
C6	0.045 (2)	0.074 (3)	0.061 (2)	0.014 (2)	0.0248 (18)	0.000 (2)
C7	0.069 (3)	0.095 (3)	0.075 (3)	0.022 (3)	0.042 (2)	0.021 (3)
C8	0.062 (2)	0.083 (3)	0.063 (3)	0.020 (2)	0.030 (2)	0.023 (2)
C9	0.082 (3)	0.101 (4)	0.083 (3)	0.039 (3)	0.032 (3)	0.002 (3)
N2	0.068 (2)	0.0422 (19)	0.0523 (19)	−0.0006 (16)	0.0184 (16)	0.0017 (16)
O4	0.122 (2)	0.0514 (18)	0.0619 (17)	0.0111 (17)	0.0375 (17)	−0.0130 (14)
O3	0.109 (2)	0.0419 (15)	0.0667 (17)	−0.0004 (15)	0.0480 (16)	−0.0029 (13)
O5	0.164 (3)	0.060 (2)	0.074 (2)	−0.010 (2)	0.043 (2)	0.0233 (16)

Geometric parameters (Å, °)

N1—C1	1.483 (4)	C3—C4	1.394 (5)
N1—C2	1.498 (4)	C4—C5	1.370 (5)
N1—H1A	0.9000	C4—H4	0.9300
N1—H1B	0.9000	C5—C6	1.371 (5)
O1—C5	1.377 (4)	C6—C7	1.348 (5)
O1—C9	1.430 (5)	C7—C8	1.398 (5)
O2—C6	1.379 (4)	C7—H7	0.9300
O2—C9	1.411 (5)	C8—H8	0.9300
C1—C1^{i}	1.506 (6)	C9—O2^{ii}	3.101 (5)
C1—H1C	0.9700	C9—H9A	0.9700
C1—H1D	0.9700	C9—H9B	0.9700
C2—C3	1.508 (4)	N2—O5	1.213 (4)
C2—H2A	0.9700	N2—O4	1.243 (4)
C2—H2B	0.9700	N2—O3	1.250 (3)

C3—C8	1.377 (5)		
C1—N1—C2	114.1 (2)	C4—C5—C6	122.4 (4)
C1—N1—H1A	108.7	C4—C5—O1	128.0 (3)
C2—N1—H1A	108.7	C6—C5—O1	109.6 (3)
C1—N1—H1B	108.7	C7—C6—C5	121.5 (3)
C2—N1—H1B	108.7	C7—C6—O2	128.6 (4)
H1A—N1—H1B	107.6	C5—C6—O2	109.9 (4)
C5—O1—C9	105.7 (3)	C6—C7—C8	117.4 (4)
C6—O2—C9	106.0 (3)	C6—C7—H7	121.3
N1—C1—C1^{i}	109.0 (3)	C8—C7—H7	121.3
N1—C1—H1C	109.9	C3—C8—C7	121.5 (4)
C1^{i}—C1—H1C	109.9	C3—C8—H8	119.3
N1—C1—H1D	109.9	C7—C8—H8	119.3
C1^{i}—C1—H1D	109.9	O2—C9—O1	108.3 (3)
H1C—C1—H1D	108.3	O2—C9—O2^{ii}	77.5 (2)
N1—C2—C3	110.1 (3)	O1—C9—O2^{ii}	161.0 (4)
N1—C2—H2A	109.6	O2—C9—H9A	110.0
C3—C2—H2A	109.6	O1—C9—H9A	110.0
N1—C2—H2B	109.6	O2^{ii}—C9—H9A	51.7
C3—C2—H2B	109.6	O2—C9—H9B	110.0
H2A—C2—H2B	108.2	O1—C9—H9B	110.0
C8—C3—C4	120.3 (3)	O2^{ii}—C9—H9B	83.7
C8—C3—C2	120.6 (3)	H9A—C9—H9B	108.4
C4—C3—C2	119.0 (3)	O5—N2—O4	122.9 (3)
C5—C4—C3	116.9 (3)	O5—N2—O3	120.0 (3)
C5—C4—H4	121.5	O4—N2—O3	117.1 (3)
C3—C4—H4	121.5		
C2—N1—C1—C1^{i}	−176.7 (3)	O1—C5—C6—O2	−1.4 (5)
C1—N1—C2—C3	−160.1 (3)	C9—O2—C6—C7	−175.8 (5)
N1—C2—C3—C8	−65.9 (4)	C9—O2—C6—C5	5.5 (5)
N1—C2—C3—C4	110.5 (3)	C5—C6—C7—C8	−0.7 (6)
C8—C3—C4—C5	−1.3 (5)	O2—C6—C7—C8	−179.2 (4)
C2—C3—C4—C5	−177.7 (3)	C4—C3—C8—C7	−0.1 (6)
C3—C4—C5—C6	1.7 (5)	C2—C3—C8—C7	176.3 (4)
C3—C4—C5—O1	−179.0 (4)	C6—C7—C8—C3	1.1 (6)
C9—O1—C5—C4	177.3 (4)	C6—O2—C9—O1	−7.5 (5)
C9—O1—C5—C6	−3.3 (4)	C6—O2—C9—O2^{ii}	−168.7 (3)
C4—C5—C6—C7	−0.7 (6)	C5—O1—C9—O2	6.6 (5)
O1—C5—C6—C7	179.8 (4)	C5—O1—C9—O2^{ii}	111.8 (9)
C4—C5—C6—O2	178.0 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1a···O3	0.90	1.89	2.787 (3)	175.1

N1—H1b···O4^{iii}	0.90	1.98	2.876 (4)	173.3
N1—H1a···O5	0.90	2.56	3.180 (4)	127.1
C1—H1d···O5^{iv}	0.97	2.61	3.291 (4)	127.6
N1—H1b···O3^{iii}	0.90	2.47	3.032 (4)	120.8
C1—H1d···O3^{iii}	0.97	2.63	3.170 (4)	115.4
C2—H2b···O4^{v}	0.97	2.42	3.302 (4)	151.2
C1—H1c···O4^{v}	0.97	2.51	3.340 (4)	143.8

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

Fig. 1

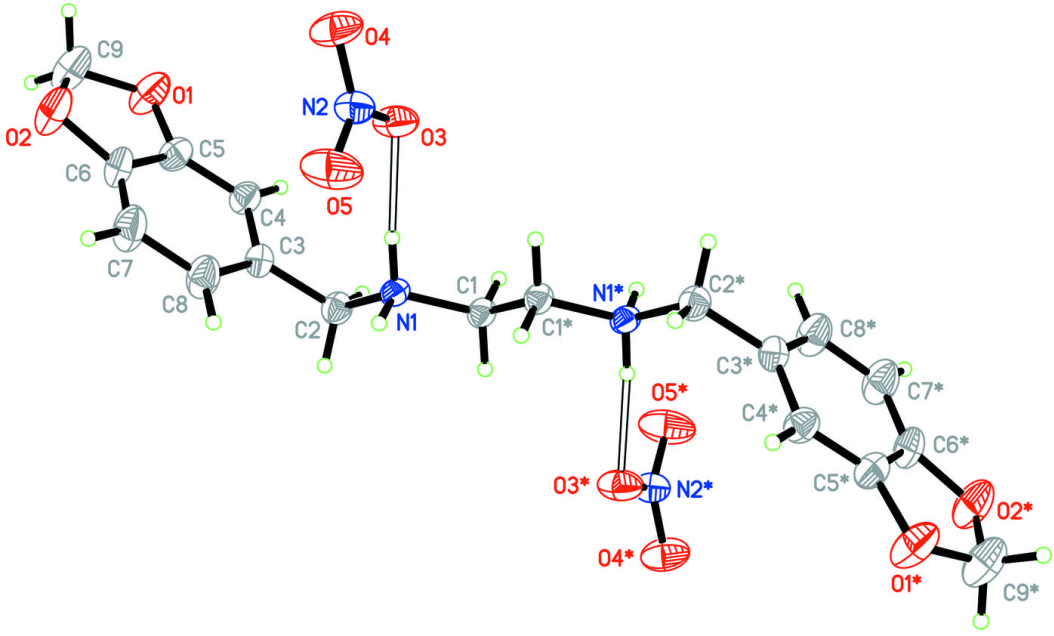
C9
O1
O2
C6
C5
C4
C3
C7
C8
C2
N1
C1
C1*
N1*
C2*
C3*
C8*
C7*
C4*
C6*
C5*
O2*
O1*
C9*
O4
N2
O3
O5
O5*
O3*
N2*
O4*

Fig. 2

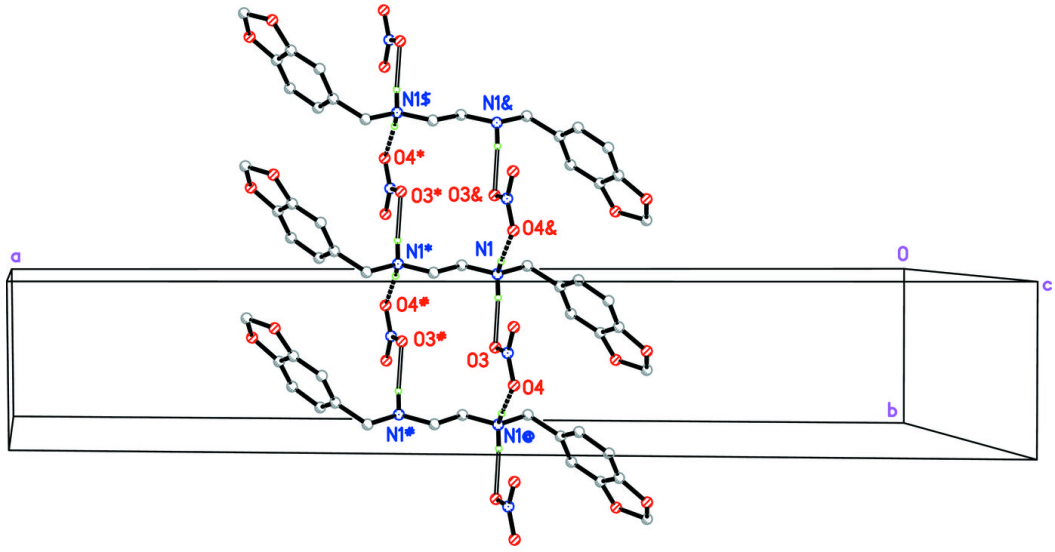

Fig. 3