Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis(4-chlorobenzyl)ethane-1,2-diammonium dinitrate

Shu-Ping Yang,[a]* Li-Jun Han,[b] Da-Qi Wang[c] and Hai-Tao Xia[a]

[a]Department of Chemical Engineering, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, [b]Department of Mathematics and Science, Huaihai Institute of Technology, Lianyungang 222005, People's Republic of China, and [c]College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: yangshuping@hhit.edu.cn

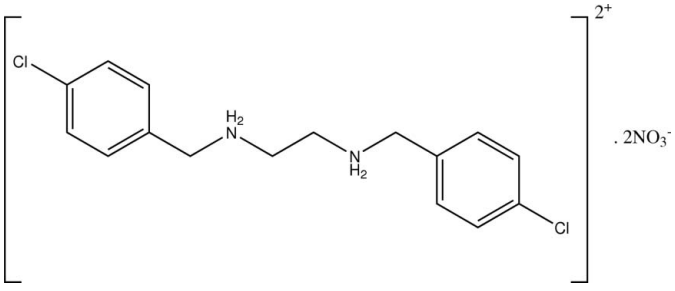
Received 13 August 2007; accepted 19 August 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.056; wR factor = 0.169; data-to-parameter ratio = 13.8.

In the title compound, $C_{16}H_{20}Cl_2N_2^{2+}\cdot 2NO_3^-$, the cation is located on an inversion center and links to the anions *via* N—H···O and C—H···O hydrogen bonding.

Related literature

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



Experimental

Crystal data

$C_{16}H_{20}Cl_2N_2^{2+}\cdot 2NO_3^-$
M_r = 435.26
Monoclinic, $P2_1/n$
a = 5.6168 (7) Å
b = 31.132 (3) Å
c = 5.7361 (8) Å
β = 91.533 (2)°
V = 1002.7 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 298 (2) K
0.56 × 0.49 × 0.45 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.822, T_{max} = 0.853
5098 measured reflections
1768 independent reflections
1486 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.169
S = 1.00
1768 reflections
128 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1	0.90	1.92	2.818 (3)	177
N1—H1B···O3^{i}	0.90	1.98	2.870 (3)	172
C2—H2B···O2^{ii}	0.97	2.42	3.265 (4)	145

Symmetry codes: (i) $x, y, z-1$; (ii) $x+1, y, z$.

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*.

The authors acknowledge the financial support of the Huaihai Institute of Technology Science Foundation.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2312).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

  doi:10.1107/S1600536807040949

supplementary materials

Acta Cryst. (2007). E**63**, o3880 [**doi:10.1107/S1600536807040949**]

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

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

Comment

We intended to prepare a lanthanum(III) complex with *N*,*N*'-bis(4-chlorobenzyl)ethane-1,2-diamine. However, we obtained the crystal of the title compound. Here we report its structure (Fig. 1).

In the crystal, the cation is located across on an inversion center, the asymmetric unit contains one half-cation and one anion, the cation and the anion are linked by N—H···O hydrogen bonding (Table 1 and Fig. 1).

In the crystal structure, the molecules are linked by two N—H···O hydrogen bonds, the atoms N1(amine) at (*x*,*y*,*z*) and (1 − *x*,1 − *y*,1 − *z*) in the molecule centred at (1/2,1/2,1/2) act as hydrogen-bond donors to the atom O3 (nitrate) at (*x*,*y*,-1 + *z*) in the molecule centred at(1/2,1/2,-1/2) and the atom O3 (nitrate) at (1 − *x*,1 − *y*,2 − *z*) in the molecule centred at(1/2,1/2, 3/2), respectively. Propagation by translation of the two hydrogen bonds generates a chain of $R_4^4(18)$ rings (Bernstein *et al.*, 1995) (Table 1 and Fig. 2).

The molecules are linked by two C—H···O and two N—H···O hydrogen bonds, so forming a [1 0 0] chain of $R_4^4(20)$ ring, the atoms C2 at (*x*,*y*,*z*) and (1 − *x*,1 − *y*,1 − *z*) in the molecule centred at (1/2,1/2,1/2) act as hydrogen-bond donors, *via* H2b, to the atom O2 (nitrate)at (1 + *x*,*y*,*z*) in the molecule centred at(3/2,1/2,1/2) and the atom O2 (nitrate)at (−*x*,1 − *y*,1 − *z*) in the molecule centred at(−1/2,1/2, 1/2), respectively, and the atoms N1(amino) at (*x*,*y*,*z*) and (1 − *x*,1 − *y*,1 − *z*) in the molecule centred at (1/2,1/2,1/2) act as hydrogen-bond donors, *via* H1a, to the atom O1 (nitrate) at (*x*,*y*,*z*) in the molecule centred at(1/2,1/2,1/2) and the atom O1 (nitrate) at (1 − *x*,1 − *y*,1 − *z*) in the molecule centred at(3/2,1/2,1/2), respectively. Propagation by translation of the two pairs of hydrogen bonds generates a chain of $R_4^4(20)$ rings along [1 0 0]direction (Table 1 and Fig. 3).

The combination of the [0 0 1] chain and the [1 0 0] chain generates a [0 1 0] stack, the stack lies in a domain of 0.260 < *y* < 0.740. Neighbouring stacks are linked by the intermolecular Cl···C weak interactions [$Cl1\cdots C6^{i}$ = 3.441 (3) Å, symmetry codes: (i)1/2 + *x*,3/2 − *y*,-1/2 + *z*], resulting in a three-dimensional structure.

Experimental

To a methanol solution (30 ml) of *N*,*N*'-bis(4-chlorobenzyl)ethane-1,2-diamine (3.42 g, 10 mmol) a methanol solution (10 ml) of lanthanum nitrate (2.16 g, 10 mmol) was added, and the mixture was stirred for 3 h at 333 k. The white solid was filtered off, washed with ethanol and dried at room temperature. Colourless crystals of the title compound suitaible for X-ray structure analysis were obtained by slow evaporation of a DMF solution containing the crude solid product over a period of two months.

Refinement

H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic), 0.97 Å (methylene) and N—H = 0.90 Å, and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C,*N*).

Figures

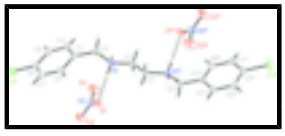


Fig. 1. The molecular stucture of compound (I), with displacement ellipsoids drawn at the 30% probability level. Doubled lines indicate hydrogen bonds. [Symmetry codes: (*)1 − *x*,1 − *y*,1 − *z*]

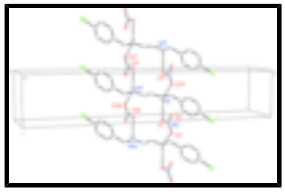


Fig. 2. Part of the crystal structure of (I), showing the formation of a chain of $R_4^4(18)$ rings along [0 0 1] direction. For the sake of clarity, H atoms bonded to C atoms have been omitted. Dashed lines and doubled lines indicate hydrogen bonds. [Symmetry codes: (*) 1 − *x*,1 − *y*,1 − *z*; (#) *x*,*y*,-1 + *z*; (&)1 − *x*,1 − *y*,2 − *z*].

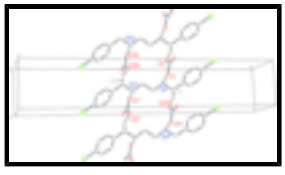


Fig. 3. Part of the crystal structure of (I), showing the formation of a chain of $R_4^4(20)$ rings along [1 0 0]direction. For the sake of clarity, H atoms bonded to C atoms have been omitted. Dashed lines and doubled lines indicate hydrogen bonds. [Symmetry codes: (*)1 − *x*,1 − *y*,1 − *z*; (#)1 + *x*,*y*,*z*; (&)-*x*,1 − *y*,1 − *z*].

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

Crystal data

$C_{16}H_{20}Cl_2N_2^{2+}\cdot 2NO_3^-$	F_{000} = 452
M_r = 435.26	D_x = 1.442 Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 523-525 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 5.6168 (7) Å	Cell parameters from 2811 reflections
b = 31.132 (3) Å	θ = 2.5–27.9º
c = 5.7361 (8) Å	μ = 0.36 mm^{-1}
β = 91.533 (2)º	*T* = 298 (2) K
V = 1002.7 (2) Å^3	Block, colourless
Z = 2	0.56 × 0.49 × 0.45 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1768 independent reflections
Radiation source: fine-focus sealed tube	1486 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 298(2) K	θ_{max} = 25.0º

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

T_{min} = 0.822, T_{max} = 0.853

5098 measured reflections

θ_{min} = 1.3º

h = −6→6

k = −37→19

l = −6→6

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.056

$wR(F^2)$ = 0.169

S = 1.00

1768 reflections

128 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0938P)^2 + 1.0094P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.22 e Å^{-3}

$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.40 (3)

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 $F^2 > 2\text{sigma}(F^2)$ 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}
Cl1	0.0520 (2)	0.73850 (3)	1.0413 (2)	0.0699 (5)
N1	0.4390 (4)	0.55809 (7)	0.5597 (4)	0.0316 (6)
H1A	0.3978	0.5555	0.7096	0.038*
H1B	0.3039	0.5588	0.4718	0.038*
N2	0.1099 (5)	0.55806 (9)	1.0499 (4)	0.0411 (7)
O1	0.3261 (4)	0.55003 (9)	1.0333 (4)	0.0567 (8)
O3	0.0322 (4)	0.56523 (10)	1.2485 (4)	0.0619 (8)
O2	−0.0175 (5)	0.55903 (12)	0.8745 (5)	0.0843 (11)
C1	0.5788 (5)	0.51959 (9)	0.4944 (5)	0.0325 (7)
H1C	0.7148	0.5163	0.6006	0.039*
H1D	0.6371	0.5230	0.3378	0.039*

C2	0.5679 (5)	0.59956 (10)	0.5305 (5)	0.0395 (8)
H2A	0.5778	0.6062	0.3659	0.047*
H2B	0.7288	0.5969	0.5947	0.047*
C3	0.4409 (5)	0.63547 (9)	0.6525 (5)	0.0357 (7)
C4	0.2310 (6)	0.65296 (10)	0.5608 (6)	0.0424 (8)
H4	0.1703	0.6433	0.4178	0.051*
C5	0.1111 (6)	0.68469 (10)	0.6801 (6)	0.0461 (8)
H5	−0.0299	0.6962	0.6189	0.055*
C6	0.2042 (6)	0.69886 (10)	0.8906 (6)	0.0437 (8)
C7	0.4130 (6)	0.68286 (11)	0.9844 (6)	0.0481 (9)
H7	0.4747	0.6934	1.1253	0.058*
C8	0.5312 (6)	0.65058 (10)	0.8650 (6)	0.0442 (8)
H8	0.6715	0.6391	0.9280	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0765 (8)	0.0546 (7)	0.0796 (8)	0.0153 (5)	0.0193 (5)	−0.0206 (5)
N1	0.0344 (13)	0.0332 (13)	0.0274 (12)	0.0030 (10)	0.0051 (9)	−0.0039 (9)
N2	0.0415 (15)	0.0510 (16)	0.0309 (14)	−0.0042 (12)	−0.0003 (11)	−0.0007 (11)
O1	0.0449 (14)	0.093 (2)	0.0330 (12)	0.0156 (13)	0.0078 (10)	0.0070 (12)
O3	0.0445 (14)	0.097 (2)	0.0453 (14)	0.0002 (13)	0.0147 (11)	−0.0157 (13)
O2	0.0629 (18)	0.138 (3)	0.0511 (17)	−0.0002 (18)	−0.0231 (14)	−0.0028 (18)
C1	0.0302 (14)	0.0373 (16)	0.0303 (15)	0.0041 (12)	0.0031 (11)	−0.0023 (11)
C2	0.0383 (16)	0.0395 (17)	0.0411 (17)	−0.0016 (13)	0.0099 (13)	−0.0036 (13)
C3	0.0379 (16)	0.0282 (15)	0.0412 (16)	−0.0001 (12)	0.0079 (12)	0.0008 (12)
C4	0.0434 (18)	0.0396 (17)	0.0440 (18)	0.0002 (13)	−0.0017 (14)	−0.0048 (13)
C5	0.0437 (18)	0.0382 (18)	0.056 (2)	0.0070 (14)	0.0019 (15)	0.0008 (14)
C6	0.0523 (19)	0.0291 (16)	0.0503 (19)	0.0004 (13)	0.0156 (15)	−0.0047 (13)
C7	0.057 (2)	0.049 (2)	0.0389 (18)	−0.0035 (16)	0.0033 (15)	−0.0092 (14)
C8	0.0450 (18)	0.0441 (18)	0.0437 (18)	0.0034 (14)	0.0030 (14)	0.0012 (14)

Geometric parameters (Å, °)

Cl1—C6	1.744 (3)	C2—H2A	0.9700
Cl1—C6^{i}	3.441 (3)	C2—H2B	0.9700
N1—C1	1.486 (3)	C3—C4	1.389 (4)
N1—C2	1.492 (4)	C3—C8	1.389 (5)
N1—H1A	0.9000	C4—C5	1.387 (4)
N1—H1B	0.9000	C4—H4	0.9300
N2—O2	1.219 (3)	C5—C6	1.375 (5)
N2—O1	1.246 (3)	C5—H5	0.9300
N2—O3	1.251 (3)	C6—C7	1.371 (5)
C1—C1^{ii}	1.509 (6)	C7—C8	1.394 (4)
C1—H1C	0.9700	C7—H7	0.9300
C1—H1D	0.9700	C8—H8	0.9300
C2—C3	1.508 (4)		
C6—Cl1—C6^{i}	169.47 (12)	C3—C2—H2B	109.5

C1—N1—C2	114.1 (2)	H2A—C2—H2B	108.1
C1—N1—H1A	108.7	C4—C3—C8	119.1 (3)
C2—N1—H1A	108.7	C4—C3—C2	121.5 (3)
C1—N1—H1B	108.7	C8—C3—C2	119.5 (3)
C2—N1—H1B	108.7	C5—C4—C3	120.7 (3)
H1A—N1—H1B	107.6	C5—C4—H4	119.6
O2—N2—O1	119.6 (3)	C3—C4—H4	119.6
O2—N2—O3	122.4 (3)	C6—C5—C4	118.9 (3)
O1—N2—O3	118.0 (2)	C6—C5—H5	120.5
N1—C1—C1^{ii}	109.1 (3)	C4—C5—H5	120.5
N1—C1—H1C	109.9	C7—C6—C5	121.9 (3)
C1^{ii}—C1—H1C	109.9	C7—C6—Cl1	119.2 (3)
N1—C1—H1D	109.9	C5—C6—Cl1	118.8 (3)
C1^{ii}—C1—H1D	109.9	C6—C7—C8	118.9 (3)
H1C—C1—H1D	108.3	C6—C7—H7	120.6
N1—C2—C3	110.6 (2)	C8—C7—H7	120.6
N1—C2—H2A	109.5	C3—C8—C7	120.5 (3)
C3—C2—H2A	109.5	C3—C8—H8	119.8
N1—C2—H2B	109.5	C7—C8—H8	119.8
C2—N1—C1—C1^{ii}	−173.8 (3)	C4—C5—C6—Cl1	−179.5 (2)
C1—N1—C2—C3	−166.4 (2)	C6^{i}—Cl1—C6—C7	−86.6 (11)
N1—C2—C3—C4	−74.6 (4)	C6^{i}—Cl1—C6—C5	93.6 (10)
N1—C2—C3—C8	103.4 (3)	C5—C6—C7—C8	−1.5 (5)
C8—C3—C4—C5	−0.8 (5)	Cl1—C6—C7—C8	178.8 (2)
C2—C3—C4—C5	177.2 (3)	C4—C3—C8—C7	0.0 (5)
C3—C4—C5—C6	0.5 (5)	C2—C3—C8—C7	−178.1 (3)
C4—C5—C6—C7	0.7 (5)	C6—C7—C8—C3	1.1 (5)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O1	0.90	1.92	2.818 (3)	177
N1—H1B···O3^{iii}	0.90	1.98	2.870 (3)	172
C2—H2B···O2^{iv}	0.97	2.42	3.265 (4)	145

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

Fig. 1

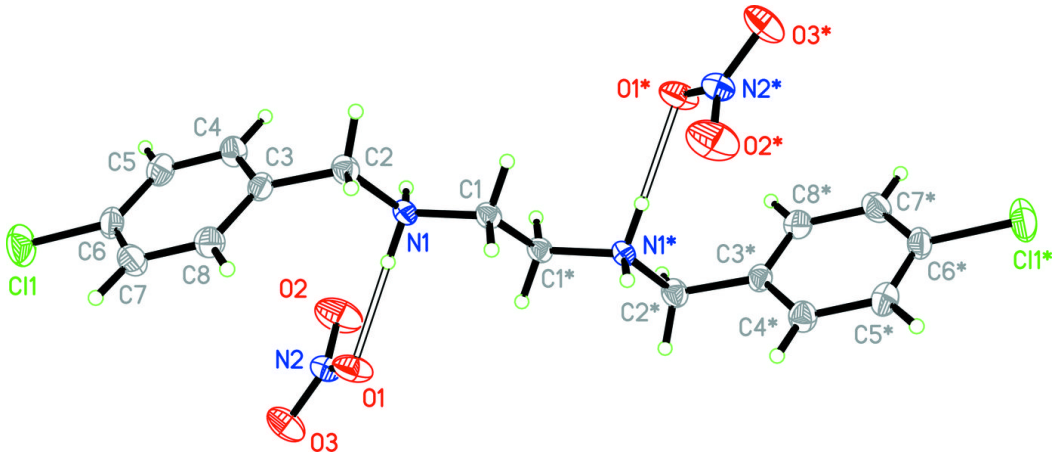

O3*
O1*
N2*
O2*
C4
C5
C3
C2
C1
C8*
C7*
N1
N1*
C3*
Cl1*
C6
C8
C7
Cl1
C1*
C6*
C2*
C4*
C5*
O2
N2
O1
O3

Fig. 2

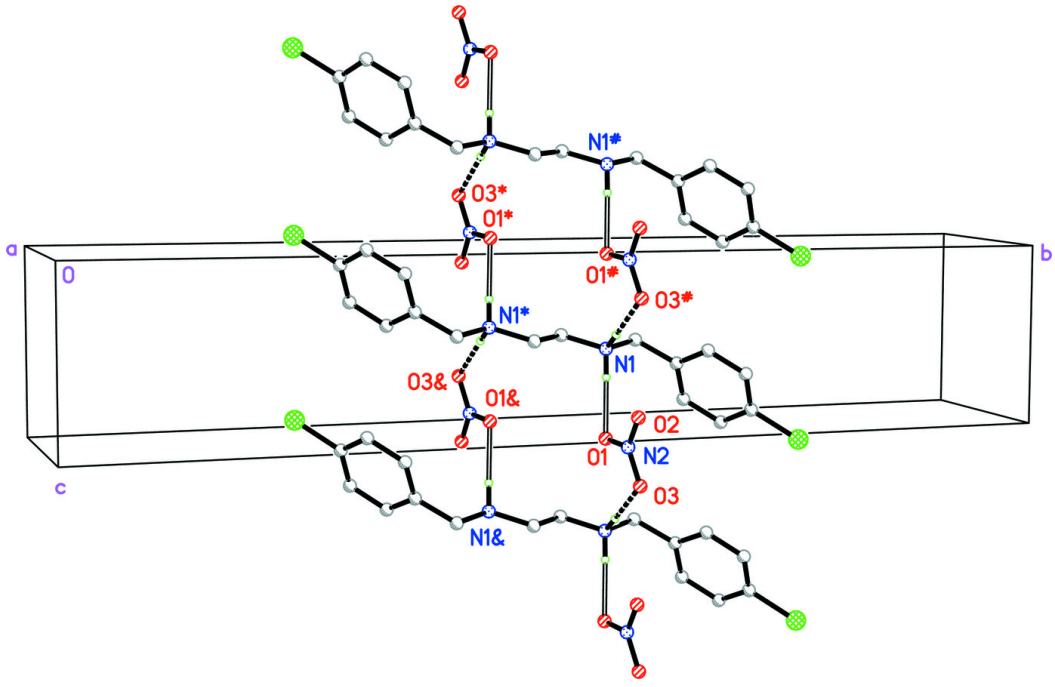


Fig. 3