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1,1′-(Butane-1,4-diyl)diimidazolium dinitrate

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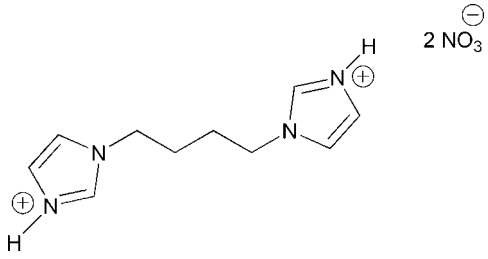
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.115; data-to-parameter ratio = 12.6.

In the title compound, $C_{10}H_{16}N_4^{2+}\cdot 2NO_3^-$, the organic cation is located around an inversion centre. The imidazolium ring forms a dihedral angle of 62.7 (3)° with the plane defined by the C atoms of the $-(CH_2)_4-$ aliphatic linker. Two anions bind to the cation *via* three-centre N—H···O hydrogen bonds and thus discrete hydrogen-bonded ion triples are formed. The nitrate is approximately coplanar with the imidazolium ring to which it binds.

Related literature

For related literature, see: Gould (1986); Holman *et al.* (2001); Jin & Chen (2007*a*,*b*); Jin *et al.* (2007); Królikowska & Garbarczyk (2005).



Experimental

Crystal data

$C_{10}H_{16}N_4^{2+}\cdot 2NO_3^-$
M_r = 316.29
Monoclinic, $P2_1/n$
a = 7.788 (2) Å
b = 10.482 (3) Å
c = 9.363 (3) Å
β = 110.649 (4)°
V = 715.3 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 298 (2) K
0.43 × 0.40 × 0.31 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.949, T_{max} = 0.963
3629 measured reflections
1257 independent reflections
913 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.115
S = 1.06
1257 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	2.59	3.157 (2)	124
N2—H2···O2^{i}	0.86	1.90	2.762 (2)	175

Symmetry code: (i) $x, y, z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); 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: GK2116).

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supplementary materials

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1,1'-(Butane-1,4-diyl)diimidazolium dinitrate

S. Jin and D. Wang

Comment

Intermolecular hydrogen bonds are a well known and efficient tool used to regulate molecular arrangement in crystals (Holman *et al.*, 2001). Salt formation can be driven by hydrogen bond as well (Gould, 1986). As an extension of our study on supramolecular assembly through weak interactions (Jin & Chen, 2007*a*,b; Jin *et al.*, 2007), here we report synthesis and crystal structure of 1,1'-(1,4-butanediyl)bis(imidazolium) dinitrate. The crystal structure of the organic base and its 1:2 salt with hydrochloric acid has been already reported (Królikowska & Garbarczyk, 2005).

The title compound was prepared by reacting ferric nitrate nonahydrate with 1-(4-(1*H*-imidazol-1-yl) butyl)-1*H*-imidazole. The hydrolysis of ferric nitrate nonahydrate led to nitric acid formation, which, in turn, reacted with 1-(4-(1*H*-imidazol-1-yl)butyl)-1*H*-imidazole present in the reaction medium to give 2:1 salt (Fig. 1).

The protonated imidazolium rings interact with the anion *via* a three-center hydrogen bond with one strong and one weak component (Table 1) and the nitrate ion is practically coplanar with the imidazolium ring. On the other hand, the heterocyclic rings and nitrate anions form double-stacks extending along the [100] direction with the alternating anionic and cationic species (Fig. 2).

Experimental

All reagents and solvents were used as obtained without further purification. The CHN elemental analyses were performed on a Perkin-Elmer model 2400 elemental analyzer.

Ferric nitrate nonahydrate (40.4 mg, 0.1 mmol) and 1-(4-(1*H*-imidazol-1-yl) butyl)-1*H*-imidazole (57 mg, 0.3 mmol) in ethanol (10 ml) were mixed and after several minutes a yellow precipitate formed. The precipitate was filtered off to yield colorless solution and the colorless solution was left standing at room temperature. In a few days colorless block crystals appeared. Yield based on 1-(4-(1*H*-imidazol-1-yl) butyl)-1*H*-imidazole: 21.3 mg, 28%. Anal. Calculated for $C_{10}H_{16}N_6O_6$: C, 37.94; H, 5.06; N, 26.56. Found: C, 37.88; H, 5.02; N, 26.51.

Refinement

All H atoms were located in a difference Fourier map. H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms (C—H = 0.93–0.97 Å, N—H = 0.86 Å) with U_{iso}(H) = 1.2U_{eq}(C, N).

Figures

Fig. 1. The structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

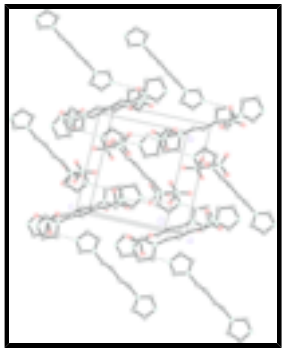

Fig. 2. Crystal packing of the hydrgen-bonded assemblies in the crystal structure of the title compound (dashed lines indicate hydrogen bonds, hydrogen atoms were omitted for clarity).

1,1'-(Butane-1,4-diyl)diimidazolium dinitrate

Crystal data

$C_{10}H_{16}N_4^{2+}\cdot 2NO_3^-$	F_{000} = 332
M_r = 316.29	D_x = 1.469 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 1224 reflections
a = 7.788 (2) Å	θ = 2.8–24.0º
b = 10.482 (3) Å	μ = 0.12 mm^{-1}
c = 9.363 (3) Å	T = 298 (2) K
β = 110.649 (4)º	Block, colourless
V = 715.3 (4) Å^3	0.43 × 0.40 × 0.31 mm
Z = 2	

Data collection

Bruker SMART APEXII CCD diffractometer	1257 independent reflections
Radiation source: fine-focus sealed tube	913 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.036
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 2.9º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9\rightarrow 9$
T_{min} = 0.949, T_{max} = 0.963	$k = -12\rightarrow 10$
3629 measured reflections	$l = -11\rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.1767P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1257 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
100 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
N1	0.6076 (2)	0.66149 (16)	0.79775 (17)	0.0408 (4)
N2	0.5677 (2)	0.72628 (16)	1.00094 (19)	0.0483 (5)
H2	0.5670	0.7281	1.0925	0.058*
N3	0.6173 (2)	0.84419 (17)	0.3487 (2)	0.0458 (5)
O1	0.6683 (3)	0.92237 (15)	0.27403 (18)	0.0673 (5)
O2	0.5460 (2)	0.74148 (15)	0.28855 (17)	0.0623 (5)
O3	0.6379 (3)	0.86529 (19)	0.4822 (2)	0.0934 (7)
C1	0.6268 (3)	0.6310 (2)	0.9390 (2)	0.0465 (5)
H1	0.6744	0.5546	0.9872	0.056*
C2	0.5079 (3)	0.8216 (2)	0.8959 (2)	0.0472 (5)
H2A	0.4590	0.8996	0.9098	0.057*
C3	0.5331 (3)	0.78137 (19)	0.7687 (2)	0.0440 (5)
H3	0.5051	0.8264	0.6778	0.053*
C4	0.6486 (3)	0.5788 (2)	0.6874 (2)	0.0509 (6)
H4A	0.7079	0.5016	0.7385	0.061*
H4B	0.7332	0.6223	0.6488	0.061*
C5	0.4777 (3)	0.54372 (19)	0.5556 (2)	0.0423 (5)
H5A	0.4195	0.6207	0.5030	0.051*
H5B	0.3920	0.5014	0.5941	0.051*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0393 (9)	0.0461 (10)	0.0335 (9)	−0.0006 (8)	0.0086 (7)	−0.0054 (7)
N2	0.0578 (12)	0.0535 (11)	0.0342 (9)	−0.0049 (9)	0.0167 (8)	−0.0053 (8)
N3	0.0479 (10)	0.0514 (11)	0.0390 (10)	0.0031 (9)	0.0168 (8)	0.0011 (9)

O1	0.0972 (14)	0.0527 (10)	0.0581 (10)	−0.0080 (9)	0.0349 (10)	0.0098 (8)
O2	0.0810 (12)	0.0549 (10)	0.0557 (10)	−0.0154 (9)	0.0298 (9)	−0.0085 (8)
O3	0.1370 (18)	0.1073 (16)	0.0490 (11)	−0.0433 (14)	0.0491 (11)	−0.0251 (10)
C1	0.0503 (13)	0.0439 (12)	0.0398 (12)	−0.0001 (10)	0.0090 (10)	−0.0012 (9)
C2	0.0500 (12)	0.0408 (11)	0.0470 (13)	−0.0010 (10)	0.0125 (10)	−0.0054 (10)
C3	0.0470 (12)	0.0419 (12)	0.0390 (11)	−0.0035 (10)	0.0100 (9)	0.0007 (9)
C4	0.0453 (13)	0.0594 (14)	0.0469 (13)	0.0037 (11)	0.0148 (10)	−0.0142 (10)
C5	0.0407 (11)	0.0446 (11)	0.0409 (11)	−0.0020 (9)	0.0136 (9)	−0.0062 (9)

Geometric parameters (Å, °)

N1—C1	1.317 (2)	C2—C3	1.342 (3)
N1—C3	1.371 (3)	C2—H2A	0.9300
N1—C4	1.467 (2)	C3—H3	0.9300
N2—C1	1.316 (3)	C4—C5	1.507 (3)
N2—C2	1.364 (3)	C4—H4A	0.9700
N2—H2	0.8600	C4—H4B	0.9700
N3—O3	1.223 (2)	C5—C5^{i}	1.518 (4)
N3—O1	1.231 (2)	C5—H5A	0.9700
N3—O2	1.251 (2)	C5—H5B	0.9700
C1—H1	0.9300		
C1—N1—C3	108.15 (16)	C2—C3—N1	107.24 (18)
C1—N1—C4	126.02 (18)	C2—C3—H3	126.4
C3—N1—C4	125.73 (17)	N1—C3—H3	126.4
C1—N2—C2	108.78 (18)	N1—C4—C5	111.83 (16)
C1—N2—H2	125.6	N1—C4—H4A	109.3
C2—N2—H2	125.6	C5—C4—H4A	109.3
O3—N3—O1	120.54 (19)	N1—C4—H4B	109.3
O3—N3—O2	119.56 (18)	C5—C4—H4B	109.3
O1—N3—O2	119.90 (18)	H4A—C4—H4B	107.9
N2—C1—N1	108.97 (18)	C4—C5—C5^{i}	111.1 (2)
N2—C1—H1	125.5	C4—C5—H5A	109.4
N1—C1—H1	125.5	C5^{i}—C5—H5A	109.4
C3—C2—N2	106.86 (19)	C4—C5—H5B	109.4
C3—C2—H2A	126.6	C5^{i}—C5—H5B	109.4
N2—C2—H2A	126.6	H5A—C5—H5B	108.0
C2—N2—C1—N1	0.1 (2)	C1—N1—C3—C2	−0.1 (2)
C3—N1—C1—N2	0.0 (2)	C4—N1—C3—C2	176.60 (18)
C4—N1—C1—N2	−176.70 (17)	C1—N1—C4—C5	113.5 (2)
C1—N2—C2—C3	−0.2 (2)	C3—N1—C4—C5	−62.7 (3)
N2—C2—C3—N1	0.2 (2)	N1—C4—C5—C5^{i}	−179.0 (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*
N2—H2···O1^{ii}	0.86	2.59	3.157 (2)	124

N2—H2···O2^{ii}	0.86	1.90	2.762 (2)	175

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

Fig. 1

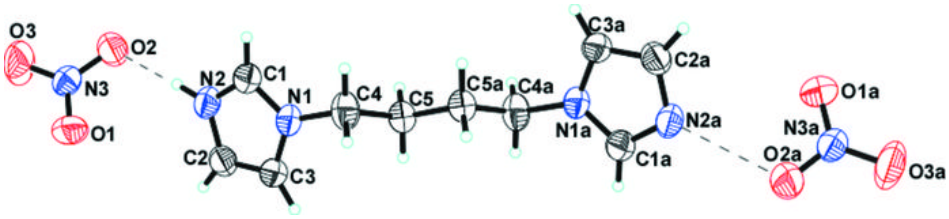
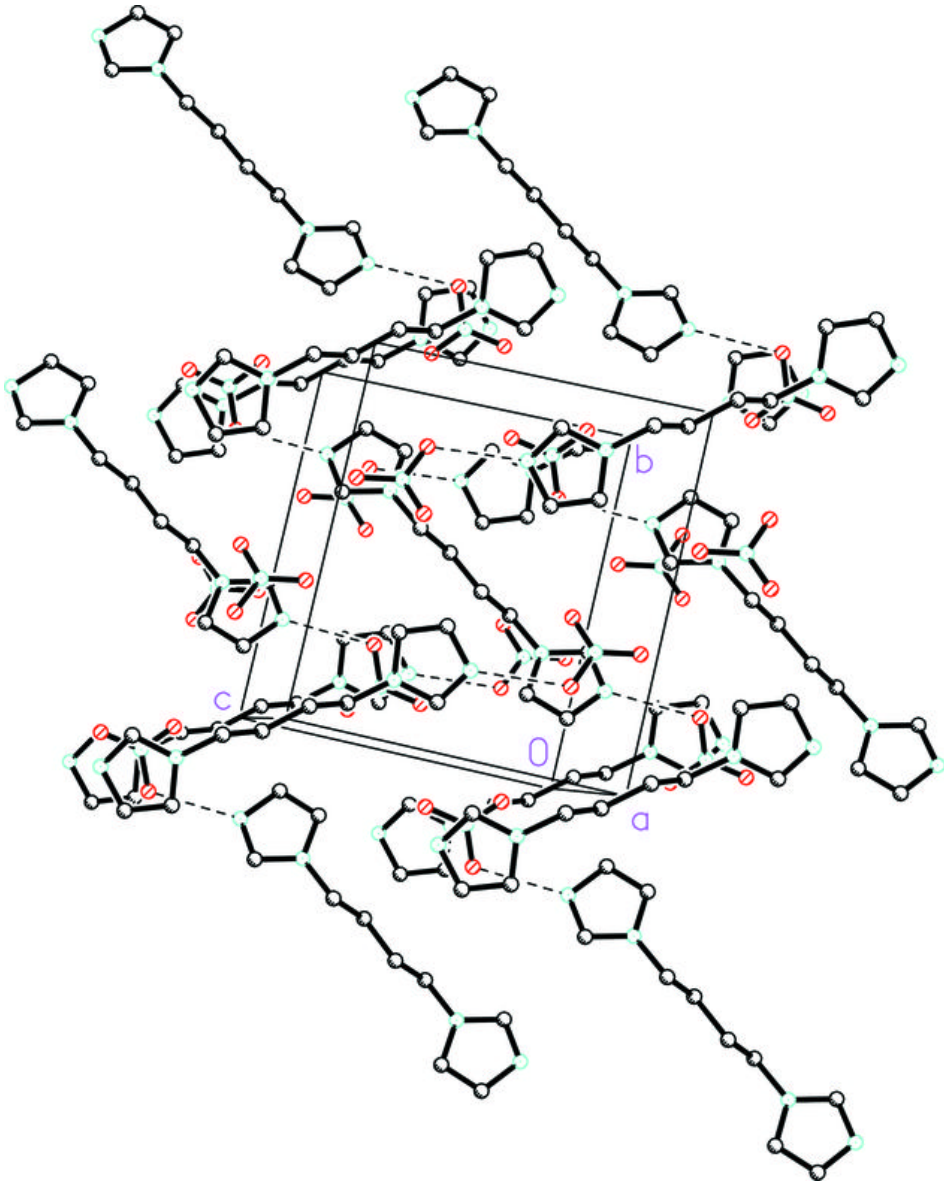
Fig. 2