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1,1′-(Butane-1,4-diyl)bis(imidazolium) bis(perchlorate)

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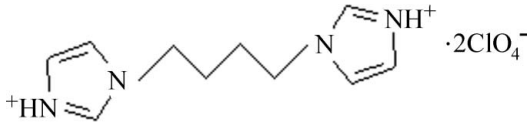
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.095; data-to-parameter ratio = 13.5.

The asymmetric unit of the title compound, $C_{10}H_{16}N_4^{2+}\cdot 2ClO_4^-$, consists of half of a centrosymmetric 1,1′-(butane-1,4-diyl)bis(imidazolium) cation and one perchlorate anion. These ions are linked to each other by intermolecular bifurcated N—H···O hydrogen bonds to form infinite chains, which are further connected to each other by weak C—H···O hydrogen bonds to build up a three-dimensional network.

Related literature

For related literature, see: Dhal & Arnold (1992); Ding *et al.* (2006); Fan *et al.* (2006); Hoskins *et al.* (1997); Krolikowska & Garbarczyk (2005); Ma *et al.* (2000, 2003); Moulton & Zaworotko (2001); Sato *et al.* (1999); Yang *et al.* (2006); Yao *et al.* (2003).



Experimental

Crystal data

$C_{10}H_{16}N_4^{2+}\cdot 2ClO_4^-$
M_r = 391.17
Triclinic, $P\overline{1}$
a = 5.557 (1) Å
b = 7.6280 (14) Å
c = 9.6139 (17) Å
α = 95.273 (2)°
β = 96.074 (2)°
γ = 95.853 (2)°
V = 400.93 (13) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 291 (2) K
0.48 × 0.38 × 0.37 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.813, T_{max} = 0.849
2949 measured reflections
1475 independent reflections
1396 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.14
1475 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3	0.86	2.23	3.019 (3)	153
N2—H2···O3^{i}	0.86	2.42	3.032 (3)	129
C1—H1···O4	0.93	2.51	3.228 (3)	134
C3—H3···O1^{i}	0.93	2.49	3.363 (3)	156
C2—H2A···O2^{ii}	0.93	2.55	3.464 (3)	169
C4—H4B···O4^{iii}	0.97	2.55	3.329 (3)	138

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2176).

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supplementary materials

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1,1'-(Butane-1,4-diyl)bis(imidazolium) bis(perchlorate)

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Comment

In recent years, metal coordination polymers with flexible bis(imidazole) ligands has been a rapidly developing area of research, because of its intriguing structures and potential applications in functional materials(Moulton & Zaworotko, 2001), such as 1,4'-bis(imidazol-1-ylmethyl)benzene (Fan *et al.*, 2006; Hoskins *et al.*, 1997), 1,1'-(1,2-ethanediyl)bis(imidazole) (Ding *et al.*, 2006), 1,1'-(butane-1,4-diyl)bis(imidazole) (bim; Yang *et al.*, 2006). Some others have been widely studied on their properties of bifunctional di(imidazole) templates of varying geometry (Dhal & Arnold, 1992) and imidazolium salts (Sato *et al.*,1999). In our previous studies, we have synthesized coordination polymer and imidazolium with 1,3'-bis(imidazol-1-ylmethyl)-5-methylbenzene (Ma *et al.*, 2003; Yao *et al.*, 2003).

In order to understand the influence of protonation of imidazole ring on the configuration of the bim molecule, the title compound (I) has been structurally characterized. The cation of (I) lies on an inversion center(Fig. 1). The protonation of imidazole groups have resulted in the changes of the bond parameters and configurations in comparison to known compounds (Krolikowska & Garbarczyk, 2005). The C—N bond lengths are in the range 1.325 (3)–1.478 (3) Å. The bimH_2 and perchlorate are linked to each other by intermolecular bifurcated N—H···O hydrogen bonds to form chains wich are further connected by weak C—H···O hydrogen bonds to build up a three-dimensional supramolecular structure (table 1).

Experimental

All reagents were of AR grade and were used without further purification. 1,1'-(butane-1,4-diyl)bis(imidazole) was prepared following the literature method (Ma, *et al.*, 2000). An ethanol solution of bim (1 mmol) was reacted with an aqueous solution of perchloric acid (2 mmol) that had been neutralized with sodium hydroxide (2 mmol). The mixture was treated with 0.05 *M* perchloric acid to a pH of 1–2 and was refluxed for 8 h, colorless crystals of the title compound separated from the filtrate after several days. Analysis calculated for C10H16Cl2N4O8: C 30.71, H 4.12, N 14.32%; found: C 31.21, H 4.14, N 14.98%.

Refinement

All H atoms were treated as riding on their parent atoms, with C—H = 0.930 Å (aromatic), 0.970Å (CH_2) and N—H= 0.86Å and with U_{iso}(H) = 1.2U_{eq}(C,*N*).

Figures

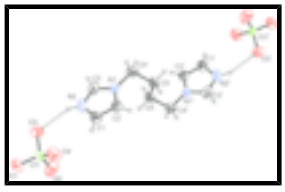

Fig. 1. Molecular view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii. N—H···O hydrogen bonds are shown as dashed lines. [Symmetry code: (i) 1 − *x*, 1 − *y*, −*z*]

1,1'-(Butane-1,4-diyl)bis(imidazolium) bis(perchlorate)

Crystal data

$C_{10}H_{16}N_4^{2+}\cdot 2ClO_4^-$	$Z = 1$
$M_r = 391.17$	$F_{000} = 202$
Triclinic, $P\overline{1}$	$D_x = 1.620$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.557$ (1) Å	Cell parameters from 2535 reflections
$b = 7.6280$ (14) Å	$\theta = 2.7$–28.2º
$c = 9.6139$ (17) Å	$\mu = 0.45$ mm^{-1}
$\alpha = 95.273$ (2)º	$T = 291$ (2) K
$\beta = 96.074$ (2)º	BLOCK, yellow
$\gamma = 95.853$ (2)º	0.48 × 0.38 × 0.37 mm
$V = 400.93$ (13) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1475 independent reflections
Radiation source: fine-focus sealed tube	1396 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.014$
$T = 291(2)$ K	$\theta_{max} = 25.5$º
φ and ω scans	$\theta_{min} = 2.7$º
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -6\rightarrow 6$
$T_{min} = 0.813$, $T_{max} = 0.849$	$k = -9\rightarrow 9$
2949 measured reflections	$l = -11\rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	H-atom parameters constrained
$wR(F^2) = 0.096$	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.2168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.14$	$(\Delta/\sigma)_{max} = 0.001$
1475 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
109 parameters	$\Delta\rho_{min} = -0.35$ 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}
Cl1	0.08600 (9)	0.78746 (6)	0.73164 (5)	0.03864 (19)
O1	0.0172 (4)	0.8598 (2)	0.86226 (18)	0.0652 (5)
O2	−0.0516 (4)	0.6196 (2)	0.6882 (2)	0.0732 (6)
O3	0.0428 (4)	0.9096 (2)	0.62750 (18)	0.0639 (5)
O4	0.3407 (3)	0.7663 (3)	0.74746 (18)	0.0616 (5)
N1	0.5280 (3)	0.7551 (2)	0.21145 (17)	0.0378 (4)
N2	0.3307 (4)	0.8272 (3)	0.3850 (2)	0.0569 (5)
H2	0.2264	0.8723	0.4335	0.068*
C1	0.5040 (5)	0.7278 (3)	0.4347 (2)	0.0488 (5)
H1	0.5315	0.6969	0.5259	0.059*
C2	0.6280 (4)	0.6827 (3)	0.3267 (2)	0.0435 (5)
H2A	0.7579	0.6149	0.3295	0.052*
C3	0.3476 (4)	0.8438 (3)	0.2504 (3)	0.0502 (6)
H3	0.2504	0.9066	0.1929	0.060*
C4	0.6119 (5)	0.7460 (3)	0.0705 (2)	0.0499 (6)
H4A	0.7726	0.8112	0.0768	0.060*
H4B	0.5024	0.8024	0.0079	0.060*
C5	0.6226 (4)	0.5566 (3)	0.0085 (2)	0.0431 (5)
H5A	0.7381	0.5024	0.0690	0.052*
H5B	0.6820	0.5587	−0.0827	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0467 (3)	0.0361 (3)	0.0359 (3)	0.0107 (2)	0.0101 (2)	0.00608 (19)
O1	0.0902 (14)	0.0611 (11)	0.0498 (10)	0.0131 (10)	0.0330 (9)	0.0013 (8)
O2	0.0756 (13)	0.0462 (10)	0.0915 (15)	−0.0008 (9)	0.0017 (10)	−0.0088 (9)
O3	0.0812 (13)	0.0673 (11)	0.0549 (10)	0.0354 (10)	0.0171 (9)	0.0279 (9)
O4	0.0482 (10)	0.0834 (13)	0.0590 (11)	0.0187 (9)	0.0095 (8)	0.0211 (9)
N1	0.0487 (10)	0.0310 (8)	0.0332 (9)	0.0046 (7)	0.0051 (7)	−0.0002 (6)
N2	0.0592 (13)	0.0520 (12)	0.0606 (13)	0.0085 (10)	0.0234 (10)	−0.0106 (10)
C1	0.0647 (15)	0.0474 (12)	0.0339 (11)	0.0038 (11)	0.0096 (10)	0.0001 (9)

C2	0.0515 (12)	0.0423 (11)	0.0379 (11)	0.0122 (9)	0.0040 (9)	0.0035 (9)
C3	0.0490 (13)	0.0399 (11)	0.0610 (15)	0.0126 (10)	0.0006 (10)	0.0012 (10)
C4	0.0732 (16)	0.0398 (11)	0.0352 (11)	−0.0069 (10)	0.0129 (10)	0.0034 (9)
C5	0.0541 (13)	0.0428 (11)	0.0326 (10)	−0.0015 (9)	0.0155 (9)	0.0002 (8)

Geometric parameters (Å, °)

Cl1—O2	1.4262 (19)	C1—C2	1.343 (3)
Cl1—O1	1.4301 (17)	C1—H1	0.9300
Cl1—O4	1.4346 (18)	C2—H2A	0.9300
Cl1—O3	1.4471 (17)	C3—H3	0.9300
N1—C3	1.332 (3)	C4—C5	1.520 (3)
N1—C2	1.373 (3)	C4—H4A	0.9700
N1—C4	1.478 (3)	C4—H4B	0.9700
N2—C3	1.325 (3)	C5—C5^{i}	1.522 (4)
N2—C1	1.360 (3)	C5—H5A	0.9700
N2—H2	0.8600	C5—H5B	0.9700
O2—Cl1—O1	109.83 (12)	N1—C2—H2A	126.2
O2—Cl1—O4	109.42 (12)	N2—C3—N1	108.2 (2)
O1—Cl1—O4	109.85 (12)	N2—C3—H3	125.9
O2—Cl1—O3	110.53 (13)	N1—C3—H3	125.9
O1—Cl1—O3	108.80 (11)	N1—C4—C5	112.46 (17)
O4—Cl1—O3	108.39 (11)	N1—C4—H4A	109.1
C3—N1—C2	108.00 (18)	C5—C4—H4A	109.1
C3—N1—C4	125.27 (19)	N1—C4—H4B	109.1
C2—N1—C4	126.65 (19)	C5—C4—H4B	109.1
C3—N2—C1	109.38 (19)	H4A—C4—H4B	107.8
C3—N2—H2	125.3	C4—C5—C5^{i}	113.3 (2)
C1—N2—H2	125.3	C4—C5—H5A	108.9
C2—C1—N2	106.8 (2)	C5^{i}—C5—H5A	108.9
C2—C1—H1	126.6	C4—C5—H5B	108.9
N2—C1—H1	126.6	C5^{i}—C5—H5B	108.9
C1—C2—N1	107.6 (2)	H5A—C5—H5B	107.7
C1—C2—H2A	126.2		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3	0.86	2.23	3.019 (3)	153
N2—H2···O3^{ii}	0.86	2.42	3.032 (3)	129
C1—H1···O4	0.93	2.51	3.228 (3)	134
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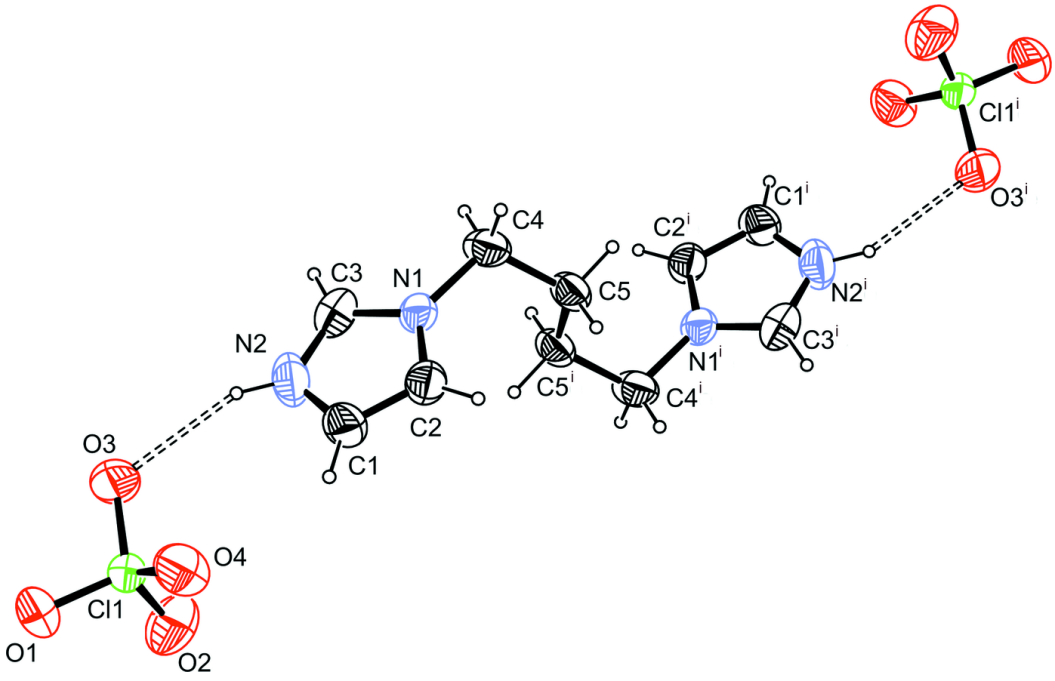
C4—H4B···O4^{iv}	0.97	2.55	3.329 (3)	138

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

Fig. 1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C1i
C2i
C4
C3
N1
C5
N2i
N2
C3i
N1i
C5i
C4i
C2
O3
C1
O4
Cl1
O1
O2