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1,1′,2,2′-Tetramethyl-3,3′-(*p*-phenylenedimethylene)diimidazol-1-ium bis(tetrafluoridoborate)

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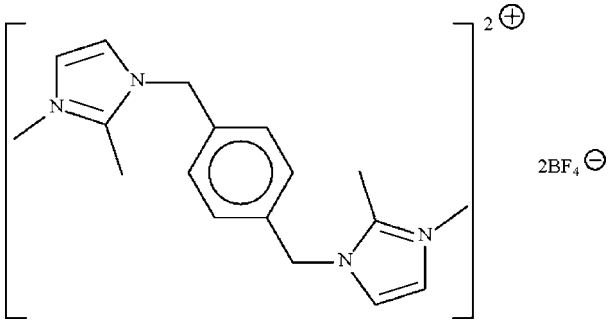
Key indicators: single-crystal X-ray study; T = 140 K; mean σ(C–C) = 0.002 Å; disorder in solvent or counterion; R factor = 0.047; wR factor = 0.140; data-to-parameter ratio = 12.5.

The title imidazolium-based ionic-liquid salt, $C_{18}H_{24}N_4^{2+}\cdot 2BF_4^-$, has the cation lying about a center of inversion. The five-membered imidazole ring is approximately perpendicular to the six-membered phenylene ring [dihedral angle = 86.9 (1)°]. The tetrafluoroborate anion is disordered over two sites in a 0.722 (3):0.278 (3) ratio.

Related literature

For background to imidazolium-based ionic liquid salts, see: Ganesan *et al.* (2008).



Experimental

Crystal data

$C_{18}H_{24}N_4^{2+}\cdot 2BF_4^-$
M_r = 470.03
Monoclinic, $P2_1/n$
a = 8.9095 (2) Å
b = 10.2254 (2) Å
c = 11.7113 (3) Å
β = 93.024 (1)°
V = 1065.45 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 140 K
0.40 × 0.35 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.993
7256 measured reflections
2418 independent reflections
2063 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.140
S = 1.03
2418 reflections
193 parameters
124 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2495).

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supporting information

Acta Cryst. (2009). E**65**, o1829 [doi:10.1107/S1600536809026312]

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S1. Experimental

α,*α*-Dibromo-*p*-xylene (0.78 g, 3 mmol) and 1,2-dimethylimidazole (0.58 g, 6 mmol) were refluxed in DMF (50 ml) for 3 h. The product that separated from solution was collected and washed with ether. Crystals were grown from its solution in water.

The bromide salt (0.46 g, 1 mmol) and sodium tetrafluoroborate (0.11 g, 1 mol) were stirred in water (100 ml) for 24 h. The product that separated from solution was collected and washed with ethanol. Crystals were grown from its solution in DMF.

S2. Refinement

The $[BF_4]^-$ anion is disordered in both the boron and fluorine atoms. The B–F distances were restrained to within 0.01 Å as were the F···F distances. The disorder refined to a 0.722 (3):0.278 (3) ratio. The anisotropic displacement parameters of the minor component atoms were restrained to be nearly isotropic. The F–B–F angles, although not ideal, are regarded as being satisfactory.

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with U(H) set to 1.2 to 1.5U_{eq}(C).

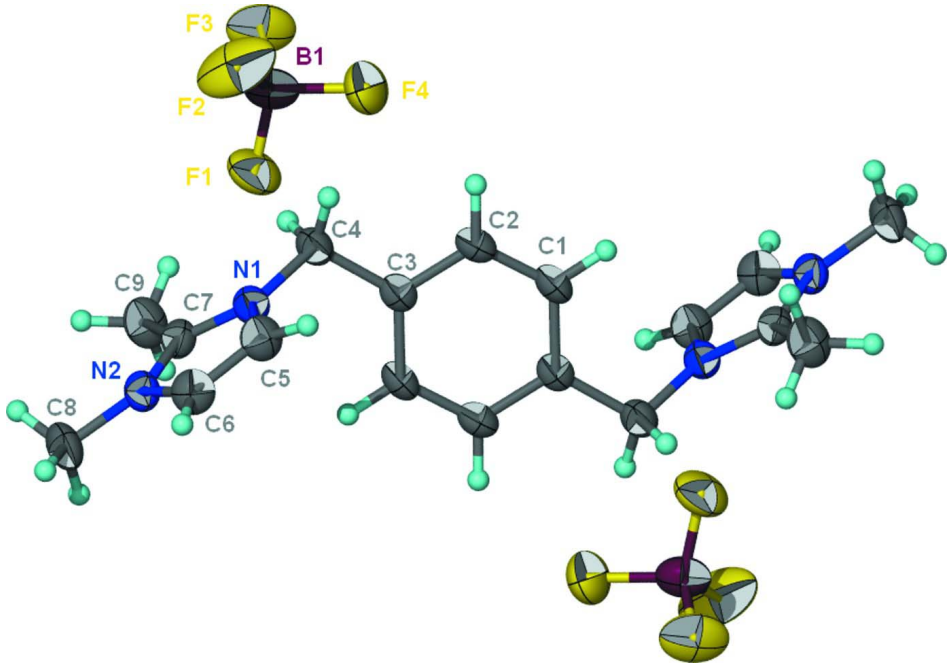

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $[C_{18}H_{24}N_4]^{2+}$ $2[BF_4]^-$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The $[BF_4]^-$ anion is disordered; the minor component of the disorder is not shown. The non-H atoms comprising the asymmetric unit are labelled and the unlabelled atoms are related by 1-x, 1-y, -z.

1,1′,2,2′-Tetramethyl-3,3′-(*p*-phenylenedimethylene)diimidazol-1-ium bis(tetrafluoridoborate)

Crystal data

$C_{18}H_{24}N_4^{2+}\cdot 2BF_4^-$
$M_r = 470.03$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.9095$ (2) Å
$b = 10.2254$ (2) Å
$c = 11.7113$ (3) Å
$\beta = 93.024$ (1)°
$V = 1065.45$ (4) Å^3
$Z = 2$
$F(000) = 484$
$D_x = 1.465$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3223 reflections
$\theta = 2.6$–$28.2°$
$\mu = 0.14$ mm^{-1}
$T = 140$ K
Irregular block, colorless
0.40 × 0.35 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.948$, $T_{max} = 0.993$
7256 measured reflections
2418 independent reflections
2063 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.7°$
$h = -11\rightarrow 11$
$k = -12\rightarrow 13$
$l = -15\rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.140$
$S = 1.03$
2418 reflections
193 parameters
124 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0793P)^2 + 0.4801P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
F1	0.5880 (4)	0.1389 (5)	0.7595 (4)	0.0560 (16)	0.722 (3)
F2	0.3734 (4)	0.1621 (3)	0.8509 (2)	0.0798 (12)	0.722 (3)
F3	0.4339 (3)	−0.0345 (2)	0.7799 (3)	0.0564 (7)	0.722 (3)
F4	0.3648 (2)	0.1294 (2)	0.66391 (16)	0.0572 (6)	0.722 (3)
F1′	0.5870 (8)	0.1500 (9)	0.7551 (6)	0.025 (2)	0.278 (3)
F2′	0.3434 (6)	0.1720 (6)	0.7957 (7)	0.065 (2)	0.278 (3)
F3′	0.4968 (6)	0.0503 (6)	0.9079 (4)	0.0664 (17)	0.278 (3)
F4′	0.4287 (12)	−0.0201 (7)	0.7334 (7)	0.099 (4)	0.278 (3)
N1	0.53432 (15)	0.55200 (13)	0.81766 (11)	0.0262 (3)	
N2	0.69734 (16)	0.64335 (14)	0.93417 (11)	0.0278 (3)	
C1	0.38756 (18)	0.46412 (16)	0.41995 (14)	0.0277 (4)	
H1	0.3101	0.4392	0.3655	0.033*	
C2	0.36751 (18)	0.44717 (16)	0.53553 (14)	0.0275 (4)	
H2	0.2761	0.4110	0.5595	0.033*	
C3	0.47960 (17)	0.48256 (14)	0.61695 (13)	0.0247 (3)	
C4	0.4550 (2)	0.45769 (16)	0.74175 (14)	0.0291 (4)	
H4A	0.3460	0.4621	0.7541	0.035*	
H4B	0.4900	0.3683	0.7619	0.035*	
C7	0.66006 (18)	0.53075 (16)	0.88270 (13)	0.0264 (4)	
C9	0.7447 (2)	0.40622 (18)	0.89444 (17)	0.0382 (4)	
H9A	0.6774	0.3329	0.8749	0.057*	
H9B	0.7850	0.3966	0.9735	0.057*	
H9C	0.8277	0.4069	0.8427	0.057*	
C8	0.8332 (2)	0.66710 (19)	1.00765 (15)	0.0368 (4)	
H8A	0.8437	0.5982	1.0657	0.055*	
H8B	0.8252	0.7522	1.0453	0.055*	
H8C	0.9214	0.6667	0.9611	0.055*	
C6	0.5949 (2)	0.73843 (17)	0.89965 (15)	0.0324 (4)	

H6	0.5962	0.8274	0.9229	0.039*	
C5	0.4932 (2)	0.68179 (17)	0.82714 (15)	0.0322 (4)	
H5	0.4089	0.7230	0.7894	0.039*	
B1	0.4413 (4)	0.0981 (4)	0.7654 (3)	0.0246 (8)	0.722 (3)
B1′	0.4639 (8)	0.0891 (8)	0.7968 (6)	0.026 (3)	0.278 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.033 (3)	0.057 (2)	0.077 (3)	−0.0039 (17)	−0.0074 (19)	0.0112 (18)
F2	0.123 (3)	0.0547 (16)	0.0673 (16)	−0.0125 (16)	0.0595 (18)	−0.0215 (14)
F3	0.0532 (13)	0.0217 (9)	0.095 (2)	0.0044 (8)	0.0083 (13)	0.0100 (11)
F4	0.0433 (10)	0.0755 (13)	0.0509 (11)	−0.0056 (9)	−0.0156 (8)	0.0165 (9)
F1′	0.024 (5)	0.027 (3)	0.025 (3)	−0.003 (3)	0.005 (3)	−0.006 (2)
F2′	0.0158 (19)	0.035 (2)	0.146 (7)	0.0079 (17)	0.016 (3)	0.011 (4)
F3′	0.068 (3)	0.086 (4)	0.047 (3)	0.011 (3)	0.009 (2)	0.009 (2)
F4′	0.092 (5)	0.082 (6)	0.123 (7)	−0.031 (5)	0.011 (5)	−0.072 (5)
N1	0.0254 (7)	0.0273 (7)	0.0257 (7)	0.0001 (5)	−0.0009 (5)	0.0001 (5)
N2	0.0284 (7)	0.0327 (7)	0.0223 (6)	−0.0002 (5)	0.0007 (5)	−0.0026 (5)
C1	0.0228 (8)	0.0289 (8)	0.0305 (8)	−0.0023 (6)	−0.0060 (6)	−0.0037 (6)
C2	0.0214 (8)	0.0275 (8)	0.0334 (8)	−0.0034 (6)	−0.0007 (6)	−0.0019 (6)
C3	0.0241 (7)	0.0201 (7)	0.0294 (8)	0.0014 (6)	−0.0028 (6)	−0.0015 (6)
C4	0.0296 (8)	0.0268 (8)	0.0303 (8)	−0.0052 (6)	−0.0047 (6)	0.0003 (6)
C7	0.0279 (8)	0.0297 (8)	0.0215 (7)	0.0009 (6)	0.0011 (6)	0.0010 (6)
C9	0.0391 (10)	0.0313 (9)	0.0432 (10)	0.0078 (7)	−0.0079 (8)	0.0001 (7)
C8	0.0362 (9)	0.0441 (10)	0.0291 (8)	−0.0030 (8)	−0.0075 (7)	−0.0070 (7)
C6	0.0351 (9)	0.0283 (8)	0.0338 (9)	0.0043 (7)	0.0031 (7)	−0.0039 (7)
C5	0.0320 (9)	0.0290 (8)	0.0353 (9)	0.0061 (7)	−0.0013 (7)	0.0007 (7)
B1	0.0270 (16)	0.0229 (14)	0.0234 (18)	0.0008 (11)	−0.0033 (14)	−0.0002 (12)
B1′	0.022 (4)	0.032 (4)	0.023 (5)	0.005 (3)	−0.012 (3)	−0.006 (3)

Geometric parameters (Å, º)

F1—B1	1.377 (4)	C2—C3	1.392 (2)
F2—B1	1.364 (4)	C2—H2	0.9500
F3—B1	1.368 (4)	C3—C1^{i}	1.392 (2)
F4—B1	1.376 (4)	C3—C4	1.511 (2)
F1′—B1′	1.373 (7)	C4—H4A	0.9900
F2′—B1′	1.368 (6)	C4—H4B	0.9900
F3′—B1′	1.377 (6)	C7—C9	1.483 (2)
F4′—B1′	1.368 (7)	C9—H9A	0.9800
N1—C7	1.339 (2)	C9—H9B	0.9800
N1—C5	1.383 (2)	C9—H9C	0.9800
N1—C4	1.467 (2)	C8—H8A	0.9800
N2—C7	1.333 (2)	C8—H8B	0.9800
N2—C6	1.379 (2)	C8—H8C	0.9800
N2—C8	1.468 (2)	C6—C5	1.340 (2)
C1—C2	1.386 (2)	C6—H6	0.9500

C1—C3^{i}	1.392 (2)	C5—H5	0.9500
C1—H1	0.9500		
C7—N1—C5	109.11 (14)	C7—C9—H9C	109.5
C7—N1—C4	126.85 (14)	H9A—C9—H9C	109.5
C5—N1—C4	123.94 (14)	H9B—C9—H9C	109.5
C7—N2—C6	109.33 (14)	N2—C8—H8A	109.5
C7—N2—C8	125.87 (15)	N2—C8—H8B	109.5
C6—N2—C8	124.58 (15)	H8A—C8—H8B	109.5
C2—C1—C3^{i}	120.51 (14)	N2—C8—H8C	109.5
C2—C1—H1	119.7	H8A—C8—H8C	109.5
C3^{i}—C1—H1	119.7	H8B—C8—H8C	109.5
C1—C2—C3	120.79 (15)	C5—C6—N2	107.20 (15)
C1—C2—H2	119.6	C5—C6—H6	126.4
C3—C2—H2	119.6	N2—C6—H6	126.4
C2—C3—C1^{i}	118.69 (15)	C6—C5—N1	107.02 (15)
C2—C3—C4	118.92 (14)	C6—C5—H5	126.5
C1^{i}—C3—C4	122.36 (14)	N1—C5—H5	126.5
N1—C4—C3	112.68 (13)	F2—B1—F3	111.0 (3)
N1—C4—H4A	109.1	F2—B1—F4	107.7 (3)
C3—C4—H4A	109.1	F3—B1—F4	108.2 (3)
N1—C4—H4B	109.1	F2—B1—F1	110.5 (3)
C3—C4—H4B	109.1	F3—B1—F1	111.0 (3)
H4A—C4—H4B	107.8	F4—B1—F1	108.3 (3)
N2—C7—N1	107.34 (14)	F2′—B1′—F4′	110.1 (6)
N2—C7—C9	125.89 (15)	F2′—B1′—F1′	110.9 (6)
N1—C7—C9	126.76 (15)	F4′—B1′—F1′	110.0 (6)
C7—C9—H9A	109.5	F2′—B1′—F3′	108.4 (6)
C7—C9—H9B	109.5	F4′—B1′—F3′	108.0 (6)
H9A—C9—H9B	109.5	F1′—B1′—F3′	109.5 (6)
C3^{i}—C1—C2—C3	−0.2 (3)	C8—N2—C7—C9	3.3 (3)
C1—C2—C3—C1^{i}	0.2 (3)	C5—N1—C7—N2	1.03 (18)
C1—C2—C3—C4	−177.78 (15)	C4—N1—C7—N2	177.39 (14)
C7—N1—C4—C3	−104.65 (18)	C5—N1—C7—C9	−177.97 (17)
C5—N1—C4—C3	71.2 (2)	C4—N1—C7—C9	−1.6 (3)
C2—C3—C4—N1	−150.75 (15)	C7—N2—C6—C5	0.69 (19)
C1^{i}—C3—C4—N1	31.3 (2)	C8—N2—C6—C5	175.47 (16)
C6—N2—C7—N1	−1.06 (18)	N2—C6—C5—N1	−0.05 (19)
C8—N2—C7—N1	−175.76 (15)	C7—N1—C5—C6	−0.61 (19)
C6—N2—C7—C9	177.95 (17)	C4—N1—C5—C6	−177.10 (15)

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