Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Ethyl-3-(2,4,6-trimethylphenyl)-imidazolium tetrafluoroborate

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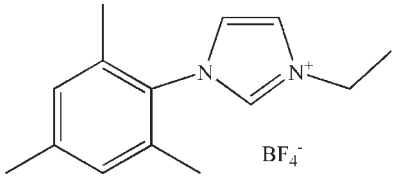
Received 28 June 2010; accepted 11 July 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.067; wR factor = 0.181; data-to-parameter ratio = 15.5.

The title compound, $C_{14}H_{19}N_2^+\cdot BF_4^-$, was obtained by reaction of 1-ethyl-3-(2,4,6-trimethylphenyl)imidazolium tetrafluoroborate with sodium tetrafluoroborate. The imidazole ring makes a dihedral angle of 78.92 (13)° with the benzene ring.

Related literature

For background, reviews and literature related to *N*-heterocyclic carbenes, see: Arduengo *et al.* (1991); Arduengo (1999); Wurtz & Glorius (2008); Haque *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{19}N_2^+\cdot BF_4^-$ M_r = 302.12
Monoclinic, $P2_1/n$
a = 7.7637 (7) Å
b = 9.1625 (9) Å
c = 21.559 (2) Å
β = 91.401 (2)°
V = 1533.2 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 K
0.16 × 0.15 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.983, T_{max} = 0.989
9593 measured reflections
3013 independent reflections
2610 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.181
S = 1.08
3013 reflections
195 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge financial support from the National Natural Science Foundation of China (No. 20572029), the New Century Excellent Talents in Universities (NCET-04–0743) and the Cultivation Fund of the Key Scientific and Technical Innovation Project, Ministry of Education of China (No. 705039).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2177).

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supplementary materials

Acta Cryst. (2010). E**66**, o2036 [**doi:10.1107/S1600536810027431**]

1-Ethyl-3-(2,4,6-trimethylphenyl)imidazolium tetrafluoroborate

J.-T. Guan, J.-G. Hou, Z.-Y. Zhang and S.-Y. Zhao

Comment

N-Heterocyclic carbenes (NHCs) have been playing an important role as ligands in organometallic chemistry and in catalysis ever since their isolation in the free state by Arduengo and coworkers in 1991 (Arduengo *et al.*,1991 and Arduengo *et al.*, 1999). As part of our research,we designed and synthesized an unsymmetrical carbene precursor imidazolinium salt, namely the title complex (I). The molecular structure of the title complex consists of disubstituted imidazolium cation and tetrafluoroborate anion(Fig. 1). The imidazole ring and benzene ring are oriented at 78.92 (13)°, the imidazole and the plane of the atoms of N2 C13 C14 are oriented at 63.8 (2)°, the imidazole ring slightly deviates from planarity as indicated by the torsion angles: N1—C10—C11—N2 = 1.0 (3)and C11—C10—N1—N2 = -1.0 (3), with a maximum deviation of 0.0056 (18)Å for atom N1.The bond lengths of B—F bonds are ranged from 1.343 (3) to 1.385 (3) Å, and the bond angles of F—B—F are ranged from 108.9 (2) to 111.4 (3)°.

Experimental

A mixture of 1-ethyl-3-(2,4,6-trimethylphenyl)imidazolium bromide (295.2 mg, 1 mmol) and sodium tetrafluoroborate (142 mg, 1.3 mmol) in THF(10 ml) was stirred for 4 h. The formed precipitate was separated by filtration and washed with Et_2O and water, dried under vacuum to give an white powder (272 mg). Crystals appropriate for data collection were obtained by slow diffusion of hexane into a solution of the the title compound in dichloromethane at 293 K.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H = 0.93 or 0.97 Å; with U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

Figures

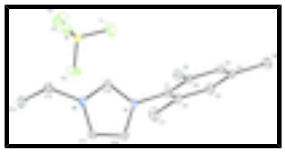

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms. H atoms are represented by circles of arbitrary size.

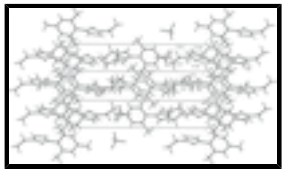

Fig. 2. The packing of (I), viewed down the *c* axis, showing one layer of molecules connected by C—H···F hydrogen bonds (dashed lines).

1-Ethyl-3-(2,4,6-trimethylphenyl)imidazolium tetrafluoroborate

Crystal data

$C_{14}H_{19}N_2^+ \cdot BF_4^-$	$F(000) = 632$
$M_r = 302.12$	$D_x = 1.309$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3305 reflections
$a = 7.7637$ (7) Å	$\theta = 2.4$–$26.5°$
$b = 9.1625$ (9) Å	$\mu = 0.11$ mm^{-1}
$c = 21.559$ (2) Å	$T = 298$ K
$\beta = 91.401$ (2)°	Block, colourless
$V = 1533.2$ (2) Å^3	0.16 × 0.15 × 0.10 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3013 independent reflections
Radiation source: fine-focus sealed tube	2610 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.026$
phi and ω scans	$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	$h = -9 \rightarrow 9$
$T_{min} = 0.983$, $T_{max} = 0.989$	$k = -11 \rightarrow 9$
9593 measured reflections	$l = -25 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.067$	H-atom parameters constrained
$wR(F^2) = 0.181$	$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 0.7775P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.026$
3013 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
195 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
0 restraints	Extinction correction: *SHELXS97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.014 (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 $F^2 > \sigma(F^2)$ 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}
B1	0.3268 (4)	0.6442 (3)	0.16502 (13)	0.0525 (7)
C1	0.7628 (3)	0.8458 (2)	0.04243 (9)	0.0430 (5)
C2	0.7570 (3)	0.7075 (3)	0.01582 (11)	0.0537 (6)
C3	0.7342 (3)	0.7003 (3)	−0.04830 (11)	0.0580 (6)
H3	0.7303	0.6093	−0.0674	0.070*
C4	0.7172 (3)	0.8245 (3)	−0.08467 (10)	0.0506 (6)
C5	0.7239 (3)	0.9592 (3)	−0.05564 (10)	0.0459 (5)
H5	0.7117	1.0429	−0.0797	0.055*
C6	0.7481 (3)	0.9738 (2)	0.00784 (10)	0.0428 (5)
C7	0.7746 (5)	0.5706 (3)	0.05432 (14)	0.0803 (9)
H7A	0.7588	0.4867	0.0281	0.120*
H7B	0.6890	0.5705	0.0857	0.120*
H7C	0.8873	0.5674	0.0736	0.120*
C8	0.6908 (4)	0.8130 (4)	−0.15396 (12)	0.0704 (8)
H8A	0.5704	0.8002	−0.1637	0.106*
H8B	0.7540	0.7308	−0.1691	0.106*
H8C	0.7311	0.9005	−0.1733	0.106*
C9	0.7608 (3)	1.1231 (3)	0.03680 (12)	0.0576 (6)
H9A	0.7485	1.1961	0.0051	0.086*
H9B	0.8709	1.1337	0.0575	0.086*
H9C	0.6710	1.1346	0.0663	0.086*
C10	0.9442 (3)	0.8499 (3)	0.14103 (11)	0.0585 (7)
H10	1.0503	0.8278	0.1243	0.070*
C11	0.9149 (3)	0.8783 (3)	0.20014 (11)	0.0560 (6)
H11	0.9968	0.8806	0.2323	0.067*
C12	0.6688 (3)	0.8903 (2)	0.14954 (10)	0.0432 (5)
H12	0.5519	0.9012	0.1403	0.052*
C13	0.6504 (3)	0.9378 (3)	0.26280 (10)	0.0535 (6)
H13A	0.5305	0.9583	0.2526	0.064*
H13B	0.7004	1.0248	0.2815	0.064*
C14	0.6599 (4)	0.8162 (3)	0.30850 (12)	0.0694 (8)
H14A	0.6102	0.7299	0.2903	0.104*
H14B	0.5974	0.8425	0.3447	0.104*

H14C	0.7781	0.7979	0.3200	0.104*
F1	0.3231 (3)	0.6699 (2)	0.10302 (8)	0.1034 (7)
F2	0.4792 (3)	0.5802 (3)	0.18106 (10)	0.1111 (8)
F3	0.3132 (2)	0.7798 (2)	0.19293 (8)	0.0817 (6)
F4	0.1928 (3)	0.5595 (2)	0.17995 (13)	0.1228 (9)
N1	0.7890 (2)	0.8590 (2)	0.10897 (8)	0.0433 (4)
N2	0.7417 (2)	0.9036 (2)	0.20523 (8)	0.0431 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B1	0.0531 (15)	0.0509 (15)	0.0534 (15)	0.0004 (12)	0.0011 (12)	0.0059 (12)
C1	0.0453 (11)	0.0476 (12)	0.0360 (10)	0.0015 (9)	0.0028 (8)	0.0006 (9)
C2	0.0673 (15)	0.0429 (13)	0.0512 (13)	0.0039 (11)	0.0072 (11)	0.0013 (10)
C3	0.0739 (17)	0.0475 (13)	0.0527 (14)	0.0036 (12)	0.0044 (11)	−0.0124 (11)
C4	0.0488 (12)	0.0596 (14)	0.0434 (12)	0.0042 (10)	0.0019 (9)	−0.0033 (10)
C5	0.0456 (12)	0.0490 (13)	0.0430 (11)	0.0031 (9)	0.0014 (9)	0.0061 (9)
C6	0.0402 (11)	0.0445 (12)	0.0437 (11)	0.0005 (9)	0.0018 (8)	−0.0006 (9)
C7	0.124 (3)	0.0468 (15)	0.0703 (19)	0.0093 (16)	0.0099 (17)	0.0075 (13)
C8	0.0803 (19)	0.083 (2)	0.0475 (14)	0.0079 (15)	−0.0039 (12)	−0.0100 (13)
C9	0.0716 (16)	0.0459 (13)	0.0553 (14)	−0.0023 (11)	0.0002 (11)	−0.0014 (11)
C10	0.0392 (12)	0.0832 (19)	0.0530 (14)	0.0065 (11)	0.0012 (10)	0.0082 (12)
C11	0.0452 (12)	0.0752 (17)	0.0474 (13)	0.0004 (11)	−0.0066 (10)	0.0069 (12)
C12	0.0388 (11)	0.0475 (12)	0.0432 (11)	0.0033 (9)	0.0008 (8)	0.0009 (9)
C13	0.0626 (14)	0.0561 (14)	0.0421 (12)	0.0027 (11)	0.0047 (10)	−0.0088 (10)
C14	0.0866 (19)	0.0722 (18)	0.0500 (14)	0.0049 (15)	0.0156 (13)	0.0078 (13)
F1	0.1536 (19)	0.0988 (14)	0.0573 (11)	0.0246 (13)	−0.0050 (11)	0.0061 (9)
F2	0.0887 (14)	0.1184 (17)	0.1254 (18)	0.0440 (12)	−0.0116 (12)	0.0259 (14)
F3	0.0730 (11)	0.0778 (12)	0.0940 (13)	−0.0010 (8)	−0.0021 (9)	−0.0243 (9)
F4	0.1021 (16)	0.0805 (14)	0.188 (3)	−0.0268 (11)	0.0534 (16)	0.0061 (14)
N1	0.0427 (9)	0.0483 (10)	0.0390 (9)	0.0030 (8)	0.0015 (7)	0.0027 (8)
N2	0.0475 (10)	0.0443 (10)	0.0375 (9)	0.0014 (8)	0.0010 (7)	0.0009 (7)

Geometric parameters (Å, °)

B1—F4	1.343 (3)	C8—H8B	0.9600
B1—F1	1.357 (3)	C8—H8C	0.9600
B1—F2	1.358 (3)	C9—H9A	0.9600
B1—F3	1.385 (3)	C9—H9B	0.9600
C1—C2	1.392 (3)	C9—H9C	0.9600
C1—C6	1.393 (3)	C10—C11	1.326 (3)
C1—N1	1.449 (3)	C10—N1	1.377 (3)
C2—C3	1.391 (3)	C10—H10	0.9300
C2—C7	1.509 (4)	C11—N2	1.371 (3)
C3—C4	1.387 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—N2	1.320 (3)
C4—C5	1.384 (3)	C12—N1	1.326 (3)
C4—C8	1.506 (3)	C12—H12	0.9300
C5—C6	1.383 (3)	C13—N2	1.478 (3)

C5—H5	0.9300	C13—C14	1.488 (4)
C6—C9	1.506 (3)	C13—H13A	0.9700
C7—H7A	0.9600	C13—H13B	0.9700
C7—H7B	0.9600	C14—H14A	0.9600
C7—H7C	0.9600	C14—H14B	0.9600
C8—H8A	0.9600	C14—H14C	0.9600
F4—B1—F1	109.8 (2)	H8B—C8—H8C	109.5
F4—B1—F2	111.4 (3)	C6—C9—H9A	109.5
F1—B1—F2	108.9 (2)	C6—C9—H9B	109.5
F4—B1—F3	110.2 (2)	H9A—C9—H9B	109.5
F1—B1—F3	105.8 (2)	C6—C9—H9C	109.5
F2—B1—F3	110.6 (2)	H9A—C9—H9C	109.5
C2—C1—C6	123.0 (2)	H9B—C9—H9C	109.5
C2—C1—N1	119.11 (19)	C11—C10—N1	107.6 (2)
C6—C1—N1	117.87 (19)	C11—C10—H10	126.2
C3—C2—C1	117.1 (2)	N1—C10—H10	126.2
C3—C2—C7	121.0 (2)	C10—C11—N2	107.6 (2)
C1—C2—C7	121.9 (2)	C10—C11—H11	126.2
C4—C3—C2	122.1 (2)	N2—C11—H11	126.2
C4—C3—H3	119.0	N2—C12—N1	109.08 (18)
C2—C3—H3	119.0	N2—C12—H12	125.5
C5—C4—C3	118.3 (2)	N1—C12—H12	125.5
C5—C4—C8	120.9 (2)	N2—C13—C14	112.4 (2)
C3—C4—C8	120.8 (2)	N2—C13—H13A	109.1
C6—C5—C4	122.5 (2)	C14—C13—H13A	109.1
C6—C5—H5	118.8	N2—C13—H13B	109.1
C4—C5—H5	118.8	C14—C13—H13B	109.1
C5—C6—C1	117.1 (2)	H13A—C13—H13B	107.9
C5—C6—C9	120.3 (2)	C13—C14—H14A	109.5
C1—C6—C9	122.62 (19)	C13—C14—H14B	109.5
C2—C7—H7A	109.5	H14A—C14—H14B	109.5
C2—C7—H7B	109.5	C13—C14—H14C	109.5
H7A—C7—H7B	109.5	H14A—C14—H14C	109.5
C2—C7—H7C	109.5	H14B—C14—H14C	109.5
H7A—C7—H7C	109.5	C12—N1—C10	107.65 (18)
H7B—C7—H7C	109.5	C12—N1—C1	125.94 (18)
C4—C8—H8A	109.5	C10—N1—C1	126.31 (18)
C4—C8—H8B	109.5	C12—N2—C11	108.11 (18)
H8A—C8—H8B	109.5	C12—N2—C13	125.41 (19)
C4—C8—H8C	109.5	C11—N2—C13	126.47 (19)
H8A—C8—H8C	109.5		
C6—C1—C2—C3	−0.4 (4)	N1—C10—C11—N2	0.6 (3)
N1—C1—C2—C3	−179.0 (2)	N2—C12—N1—C10	1.0 (3)
C6—C1—C2—C7	179.5 (3)	N2—C12—N1—C1	−175.53 (19)
N1—C1—C2—C7	0.9 (4)	C11—C10—N1—C12	−1.0 (3)
C1—C2—C3—C4	−0.3 (4)	C11—C10—N1—C1	175.5 (2)
C7—C2—C3—C4	179.8 (3)	C2—C1—N1—C12	−103.7 (3)
C2—C3—C4—C5	0.2 (4)	C6—C1—N1—C12	77.7 (3)

C2—C3—C4—C8	−179.3 (2)	C2—C1—N1—C10	80.4 (3)
C3—C4—C5—C6	0.5 (3)	C6—C1—N1—C10	−98.3 (3)
C8—C4—C5—C6	−179.9 (2)	N1—C12—N2—C11	−0.7 (3)
C4—C5—C6—C1	−1.1 (3)	N1—C12—N2—C13	−179.9 (2)
C4—C5—C6—C9	177.8 (2)	C10—C11—N2—C12	0.0 (3)
C2—C1—C6—C5	1.1 (3)	C10—C11—N2—C13	179.2 (2)
N1—C1—C6—C5	179.73 (18)	C14—C13—N2—C12	115.6 (3)
C2—C1—C6—C9	−177.8 (2)	C14—C13—N2—C11	−63.4 (3)
N1—C1—C6—C9	0.8 (3)		

Fig. 1

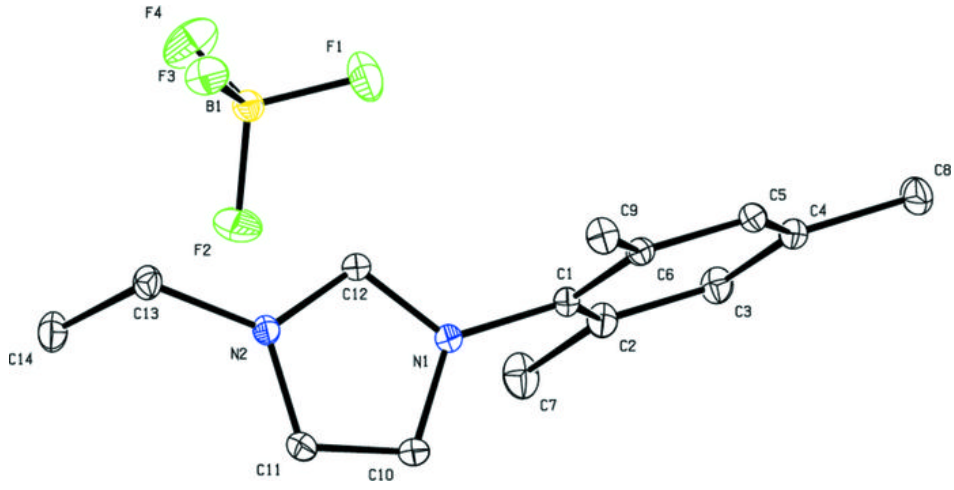

Fig. 2