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Reinvestigation of hydrazinium tetrafluoroborate

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Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(F–B) = 0.003 Å
R factor = 0.050
wR factor = 0.121
Data-to-parameter ratio = 11.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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The single-crystal X-ray study of hydrazinium tetrafluoroborate, $N_2H_5^+ \cdot BF_4^-$, confirms the previous structure determinations in the space group $C2/c$ [Conant, Corrigan & Sparks (1964). *Acta Cryst.* **17**, 1085; Conant & Roof (1970). *Acta Cryst.* B**26**, 1928–1932]. The present study includes the determination of the H-atom parameters, which reveal an extensively hydrogen-bonded structure, as expected. Several interionic hydrogen bonds are found, involving short N···F distances [2.929 (3)–2.989 (3) Å] and a short N···N distance [2.916 (3) Å].

Comment

The unit cell of the title compound derived from a two-dimensional model was first reported by Conant *et al.* (1964) and a corrected crystal structure was published by the same authors six years later (Conant & Roof, 1970). As we are especially interested in interionic electrostatic interactions and the published data were not sufficiently precise for the H atoms to be clearly resolved in the difference electron-density map, we have reinvestigated the crystal structure to locate the H atoms and refine them freely.

The hydrazinium cation adopts a staggered conformation in the solid state (Fig. 1), with an N—N bond distance of 1.445 (3) Å [*cf.* 1.425 (19) Å (Conant & Roof, 1970) and 1.438 (2) Å in hydrazinium oxalate (Thomas, 1973)]. Other bond distances are given in Table 1.

In the tetrafluoroborate anion, the coordination of the B atom by the four F atoms can be considered as slightly distorted tetrahedral, with F—B—F bond angles in the range 107.81 (18)–111.2 (2)°.

In the crystal structure, the $N_2H_5^+$ cations and BF_4^- anions are linked by N—H···F and N—H···N hydrogen bonds with relatively short donor···acceptor distances. Furthermore, serveral N—H···F hydrogen bonds with slightly longer donor···acceptor distances exist between the ionic pairs (Table 2). Selected bond lengths and angles, along with the symmetry codes of the hydrogen bonds, are summarized in Tables 1 and 2.

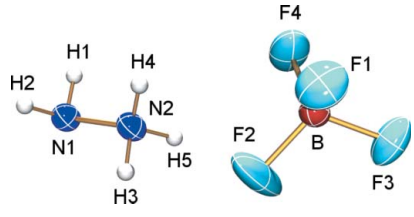

Figure 1
The molecular structure of (I), showing the atomic numbering scheme and with displacement ellipsoids drawn at the 50% probability level.

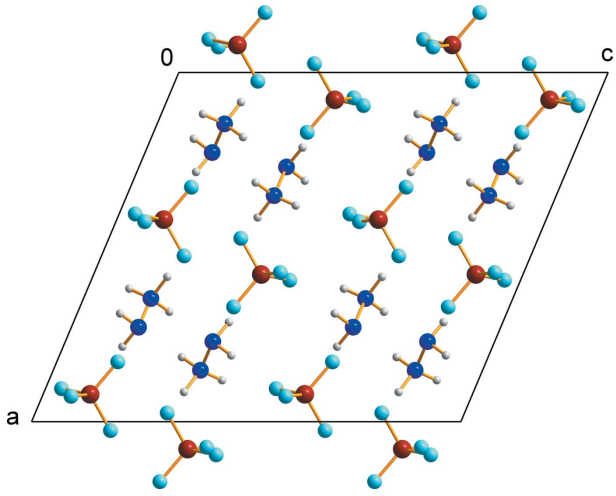

Figure 2
A packing diagram for (I), viewed down the *b* axis.

Experimental

An aqueous solution (5 ml) of silver tetrafluoroborate (2 mmol) was added dropwise, with constant stirring, to an aqueous solution (10 ml) of hydrazinium chloride (2 mmol), with exclusion of light at ambient temperature. The precipitated AgCl was filtered off, and the resulting clear solution was then evaporated to dryness *in vacuo*. Analysis, found: N 23.18, H 3.97%; calculated: N 23.37, H 4.20%. Crystallization from a saturated aqueous solution at 273 K gave colourless X-ray quality crystals.

Crystal data

$N_2H_5^+\cdot BF_4^-$
M_r = 119.87
Monoclinic, $C2/c$
a = 13.8644 (5) Å
b = 5.3233 (2) Å
c = 12.2334 (4) Å
β = 112.8848 (16)°
V = 831.81 (5) Å^3
Z = 8
D_x = 1.914 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 200 (2) K
Square prism, colourless
0.24 × 0.08 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: none
1779 measured reflections
953 independent reflections
752 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.121
S = 1.07
953 reflections
84 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0411P)^2 + 1.8993P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

B—F2	1.361 (3)	N1—H1	0.91 (3)
B—F3	1.377 (3)	N1—H2	0.91 (3)
B—F4	1.379 (3)	N2—H3	0.93 (3)
B—F1	1.384 (3)	N2—H4	0.89 (3)
N1—N2	1.445 (2)	N2—H5	0.97 (4)
N2—N1—H1	106.0 (19)	H3—N2—H4	111 (3)
N2—N1—H2	106.3 (18)	N1—N2—H5	108 (2)
H1—N1—H2	103 (2)	H3—N2—H5	104 (3)
N1—N2—H3	109.4 (19)	H4—N2—H5	112 (3)
N1—N2—H4	111.3 (18)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F4^{i}	0.91 (3)	2.08 (3)	2.989 (2)	175 (3)
N1—H2···F3^{ii}	0.91 (3)	2.37 (3)	3.019 (2)	128 (2)
N1—H2···F1^{ii}	0.91 (3)	2.56 (3)	3.467 (3)	174 (2)
N2—H3···N1^{iii}	0.93 (3)	2.00 (3)	2.929 (3)	173 (3)
N2—H4···F1^{iv}	0.89 (3)	2.12 (3)	2.916 (3)	150 (3)
N2—H4···F4	0.89 (3)	2.63 (3)	3.135 (2)	118 (2)
N2—H5···F1^{v}	0.97 (4)	2.27 (4)	2.969 (3)	128 (3)

Symmetry codes: (i) $-x, -y-1, -z+2$; (ii) $x, -y, z+\frac{1}{2}$; (iii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{5}{2}$; (iv) $-x, -y, -z+2$; (v) $x, y-1, z$.

H atoms were visible in a difference synthesis and were refined freely.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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References

Conant, J. W., Corrigan, L. I. & Sparks, R. A. (1964). *Acta Cryst.* **17**, 1085.
Conant, J. W. & Roof, R. B. (1970). *Acta Cryst.* B**26**, 1928–1932.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Thomas, J. O. (1973). *Acta Cryst.* B**29**, 1767–1776.