Ingo Krossing,* Ines Raabe and
Esther Birtalan

Institut für Anorganische und Analytische
Chemie, Universität Freiburg, Albertstr. 21,
D-79104 Freiburg i. Br., Germany

Correspondence e-mail:
krossing@uni-freiburg.de

Key indicators

Single-crystal X-ray study
 $T = 100\text{ K}$
Mean $\sigma(\text{O} - \text{N}) = 0.001\text{ Å}$
 R factor = 0.019
 wR factor = 0.061
Data-to-parameter ratio = 10.5

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

Nitronium tetrafluoroborate

Commercially available nitronium tetrafluoroborate, $\text{NO}_2^+ \cdot \text{BF}_4^-$, has been recrystallized from CH_2Cl_2 as a side-product of a reaction in which it has been used as a starting material. Both the NO_2^+ cations and the BF_4^- anions are packed in considerably distorted cubic environments. The $\text{N}=\text{O}$ distances of 1.1153 (8) and 1.1160 (7) Å as well as the $\text{O}=\text{N}=\text{O}$ bond angle of $179.89 (7)^\circ$ are similar to those found in other NO_2^+ -containing salts described in the literature.

Received 5 December 2006

Accepted 3 January 2007

Comment

The $\text{N}=\text{O}$ distances in $[\text{NO}_2][\text{BF}_4]$ of 1.1153 (8) and 1.1160 (7) Å as well as the $\text{O}=\text{N}=\text{O}$ bond angle of $179.89 (7)^\circ$ are in good agreement with those found for other nitronium salts such as $[\text{NO}_2][\text{Fe}(\text{NO}_3)_4]$ ($d_{\text{av.}} = 1.116\text{ Å}$) or $[\text{NO}_2][\text{Zr}(\text{NO}_3)_5]$ ($d_{\text{av.}} = 1.105\text{ Å}$) (Tikhomirov *et al.*, 2002). In addition, the $\text{B}-\text{F}$ distances (see Table 1) are typical for a tetrafluoroborate anion.

Weak bonding interactions shorter than the sum of the van der Waals radii [$r(\text{O} + \text{F}) = 3.0\text{ Å}$, $r(\text{N} + \text{F}) = 3.1\text{ Å}$; Holleman, 1995] are found between the cations and anions. The shortest are the $\text{N}-\text{F}$ contacts which range from 2.474 (1) to 2.500 (1) Å (Fig. 1). Both the NO_2^+ cations and the BF_4^- anions are packed in considerably distorted cubic environments.

Figure 1
Packing diagram of $[\text{NO}_2][\text{BF}_4]$. Dashed lines represent cation–anion contacts shorter than the sum of the van der Waals radii. Displacement ellipsoids are drawn at the 25% probability level. [Symmetry code: (i) $1 - x, \frac{1}{2} - x, \frac{1}{2} - z$.]

Experimental

Colourless block-shaped crystals of the title salt (Sigma–Aldrich) were fortuitously recrystallized from CH_2Cl_2 as a by-product of an attempted reaction with P_4 .

Crystal data

$\text{NO}_2^+\cdot\text{BF}_4^-$	$Z = 4$
$M_r = 132.82$	$D_x = 2.232 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.5567 (13) \text{ \AA}$	$\mu = 0.30 \text{ mm}^{-1}$
$b = 6.8243 (14) \text{ \AA}$	$T = 100 (2) \text{ K}$
$c = 9.1244 (18) \text{ \AA}$	Block, colourless
$\beta = 104.40 (3)^\circ$	$0.4 \times 0.2 \times 0.2 \text{ mm}$
$V = 395.44 (14) \text{ \AA}^3$	

Data collection

STOE IPDS-II diffractometer	768 independent reflections
φ scans	662 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.027$
2652 measured reflections	$\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 0.0515P]$
$R[F^2 > 2\sigma(F^2)] = 0.019$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{\text{max}} = 0.019$
$S = 1.16$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
768 reflections	$\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$
73 parameters	

Table 1

Selected bond lengths ($\text{\AA}$).

F1—B1	1.3885 (8)	F5—B1	1.3908 (9)
F2—B1	1.3907 (8)	F6—B1	1.3896 (9)

Data collection: *XPOSE* (Stoe, 2003); cell refinement: *CELL* (Stoe, 2003); data reduction: *X-RED32* (SToe, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *SHELXL97*.

References

- Crystal Impact (2006). *DIAMOND*. Version 3.1. Crystal Impact GbR, Bonn, Germany.
- Holleman, A. F. (1995). *Lehrbuch der anorganischen Chemie*, p. 102. Berlin, New York: De Gruyter.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Stoe (2003). *CELL*, *XPOSE* and *X-RED32*. Stoe & Cie GmbH, Darmstadt, Germany.
- Tikhomirov, G., Morozov, I., Znamenkov, K., Kemnitz, E. & Troyanov, S. (2002). *Z. Anorg. Allg. Chem.* **628**, 872–876.

supporting information

Acta Cryst. (2007). E63, i43–i44 [https://doi.org/10.1107/S1600536807000207]

Nitronium tetrafluoroborate

Ingo Krossing, Ines Raabe and Esther Birtalan

Nitronium Tetrafluoroborate

Crystal data

[NO₂][BF₄]

$M_r = 132.82$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 6.5567$ (13) Å

$b = 6.8243$ (14) Å

$c = 9.1244$ (18) Å

$\beta = 104.40$ (3)°

$V = 395.44$ (14) Å³

$Z = 4$

$F(000) = 64$

$D_x = 2.232$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2652 reflections

$\theta = 1.5$ – 26.0°

$\mu = 0.30$ mm⁻¹

$T = 100$ K

Block, colourless

$0.4 \times 0.2 \times 0.2$ mm

Data collection

STOE IPDS-II

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ scans

2652 measured reflections

768 independent reflections

662 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 3.2^\circ$

$h = -8 \rightarrow 8$

$k = -8 \rightarrow 8$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.019$

$wR(F^2) = 0.061$

$S = 1.16$

768 reflections

73 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.0289P)^2 + 0.0515P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.019$

$\Delta\rho_{\text{max}} = 0.13$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.12$ 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.

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.91795 (6)	0.18853 (6)	0.03448 (4)	0.02119 (10)
F2	0.56288 (6)	0.20088 (6)	−0.05137 (4)	0.02109 (10)
F5	0.73340 (6)	0.42075 (6)	0.12365 (5)	0.02709 (11)
F6	0.76422 (6)	0.43746 (6)	−0.11913 (5)	0.02784 (11)
B1	0.74477 (10)	0.31239 (11)	−0.00318 (7)	0.01633 (17)
O2	0.14914 (7)	0.19493 (7)	−0.18388 (5)	0.02076 (12)
O1	0.33678 (7)	0.32845 (7)	−0.33137 (5)	0.02034 (12)
N1	0.24313 (8)	0.26166 (8)	−0.25757 (5)	0.01675 (14)

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.01870 (18)	0.0205 (2)	0.02336 (19)	0.00454 (14)	0.00321 (16)	−0.00039 (16)
F2	0.01816 (18)	0.0210 (2)	0.02302 (19)	−0.00364 (15)	0.00311 (16)	−0.00071 (15)
F5	0.0292 (2)	0.0249 (2)	0.0259 (2)	0.00382 (17)	0.00454 (18)	−0.00991 (17)
F6	0.0297 (2)	0.0236 (2)	0.0296 (2)	−0.00244 (16)	0.00612 (18)	0.01012 (17)
B1	0.0160 (3)	0.0152 (4)	0.0174 (4)	0.0001 (3)	0.0034 (3)	−0.0003 (3)
O2	0.0197 (2)	0.0253 (3)	0.0183 (2)	−0.00407 (19)	0.00657 (18)	−0.00158 (19)
O1	0.0203 (2)	0.0231 (2)	0.0186 (2)	−0.00378 (18)	0.00684 (18)	−0.00141 (19)
N1	0.0163 (3)	0.0168 (3)	0.0157 (3)	0.0001 (2)	0.0013 (2)	−0.0048 (2)

Geometric parameters ($\text{\AA}$, $^\circ$)

F1—B1	1.3885 (8)	F6—B1	1.3896 (9)
F2—B1	1.3907 (8)	O2—N1	1.1160 (7)
F5—B1	1.3908 (9)	O1—N1	1.1153 (8)
F1—B1—F6	109.76 (6)	F6—B1—F5	109.93 (6)
F1—B1—F2	109.23 (6)	F2—B1—F5	109.56 (6)
F6—B1—F2	109.22 (5)	O1—N1—O2	179.89 (7)
F1—B1—F5	109.12 (5)		