

Potassium trifluoro[(Z)-3-methoxyprop-1-enyl]borate

Julio Zukerman-Schpector,^{a*} Rafael C. Guadagnin,^b
Hélio A. Stefani^b and Lorenzo do Canto Visentin^c

^aDepartment of Chemistry, Universidade Federal de São Carlos, 13565-905 São Carlos, SP, Brazil, ^bDepartamento de Farmácia, Faculdade de Ciências Farmacêuticas, Universidade de São Paulo, São Paulo, SP, Brazil, and ^cInstituto de Química, Universidade Federal do Rio de Janeiro, RJ, Brazil

Correspondence e-mail: julio@power.ufscar.br

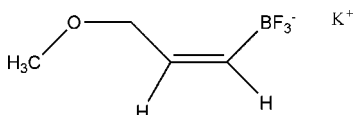
Received 30 October 2008; accepted 6 November 2008

Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.027; wR factor = 0.072; data-to-parameter ratio = 14.4.

In the title salt, $\text{K}^+\cdot\text{C}_4\text{H}_7\text{BF}_3\text{O}^-$, the K atom is surrounded by six anions making close contacts through seven F [$\text{K}\cdots\text{F} = 2.779(1)\text{--}3.048(1)$ Å] and two O [$\text{K}\cdots\text{O} = 2.953(2)$ and $3.127(2)$ Å] atoms in a trivacant *fac*-vIC-9 icosahedral coordination geometry.

Related literature

For related structures, see: Caracelli *et al.* (2007); Stefani *et al.* (2006); For related literature, see: Ruiz-Martínez *et al.* (2008); Vieira *et al.* (2008).



Experimental

Crystal data

$\text{K}^+\cdot\text{C}_4\text{H}_7\text{BF}_3\text{O}^-$
 $M_r = 178.01$
Monoclinic, $P2_1/c$
 $a = 10.882(2)$ Å
 $b = 7.2668(15)$ Å
 $c = 9.2317(18)$ Å
 $\beta = 101.52(3)^\circ$

$V = 715.3(3)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.72$ mm⁻¹
 $T = 291(2)$ K
 $0.31 \times 0.22 \times 0.11$ mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2006)
 $T_{\min} = 0.804$, $T_{\max} = 0.924$
16605 measured reflections
1327 independent reflections
1182 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.059$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.072$
 $S = 1.00$
1327 reflections
92 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.25$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *PHICHI* (Duisenberg *et al.*, 2000); data reduction: *EVAL-14* (CCD) (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We thank FAPESP (07/59404-2 to HAS and 08/02531-5 to JZS), CNPq (300613/2007 to HAS and 307121/2006-0 to JZS) and CAPES for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2510).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Bruker (2006). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Caracelli, I., Stefani, H. A., Vieira, A. S., Machado, M. M. P. & Zukerman-Schpector, J. (2007). *Z. Kristallogr. New Cryst. Struct.* **222**, 345–346.
- Duisenberg, A. J. M., Hooft, R. W. W., Schreurs, A. M. M. & Kroon, J. (2000). *J. Appl. Cryst.* **33**, 893–898.
- Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Ruiz-Martínez, A., Casanova, D. & Alvarez, S. (2008). *Dalton Trans.* pp. 2583–2591.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Stefani, H. A., Cella, R., Zukerman-Schpector, J. & Caracelli, I. (2006). *Z. Kristallogr. New Cryst. Struct.* **221**, 167–168.
- Vieira, A. S., Fiorante, P. F., Zukerman-Schpector, J., Alves, D., Botteselle, G. V. & Stefani, H. A. (2008). *Tetrahedron*, **64**, 7234–7241.

supplementary materials

Acta Cryst. (2008). E64, m1525 [doi:10.1107/S1600536808036428]

Potassium trifluoro[(*Z*)-3-methoxyprop-1-enyl]borate

J. Zukerman-Schpector, R. C. Guadagnin, H. A. Stefani and L. do C. Visentin

Comment

Organic compounds of tellurium, such as *Z*-vinylic tellurides, are important synthetic precursors of organometallic molecules and organic salts and can be useful in the synthesis of new potassium vinyl trifluoroborate salts. Organotrifluoroborates represent an alternative to boronic acids, boronate esters, and organoboranes for use in the Suzuki-Miyaura reaction and other transition-metal-catalyzed cross-coupling reactions (Vieira *et al.* 2008). Following the ideas of Ruiz-Martínez *et al.* (2008) the geometry around the K^+ ion can be described as a trivacant icosahedron, *fac*-vIC-9, a non spherical shape, as shown in Figure 2. The independent molecules in (I) are connected *via* $C3 \cdots F2^i = 3.214(2) \text{ \AA}$, $C3-H3B \cdots F2^i = 137^\circ$ ($i = x - 1/2, -y + 3/2, z$).

Experimental

nBuLi (0.8 mmol) was added dropwise at 203 K to a solution of the appropriated *Z*-vinylic telluride (1 mmol) in Et₂O (6 ml). The bath temperature was raised to 253 K. After 20 minutes B(OiPr)₃ (1.0 mmol) was added at 233 K. After 1 h, a aqueous solution of KHF₂ (4 mmol in 10 ml of water) was added to the reaction mixture. Then, the solvent and water were eliminated by evaporation. To the obtained solid hot acetone was added and the bulk reactional was filtered and dried, yielding 67% of (*Z*)-potassium vinyltrifluoroborate salt. Single crystals were obtained by slow evaporation from Et₂O.

Refinement

The H atoms were refined in the riding-model approximation, with C—H = 0.93–0.97 Å and $U_{iso}(H) = 1.5U_{eq}(\text{methyl C})$ or $1.2U_{eq}(\text{remaining C})$.

Figures

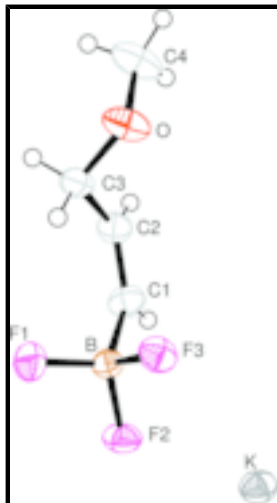


Fig. 1. The molecular structure of the title compound showing atom labelling scheme and displacement ellipsoids at the 50% probability level (arbitrary spheres for the H atoms).

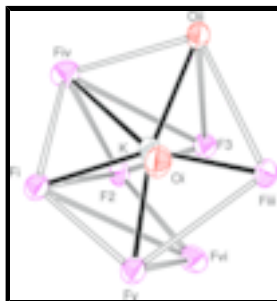


Fig. 2. The trivalent icosahedron, *fac*-vIC-9, around the K^+ ion. Symmetry operations: i = $1 - x, y - 1/2, 1/2 - z$; ii = $1 - x, y - 1, -z$; iii = $1 - x, y + 1/2, 1/2 - z$; iv = $1 - x, -2 - y, -z$; v = $x, -3/2 - y, z - 1/2$; vi = $x, -3/2 - y, 1/2 + z$.

Potassium trifluoro[(*Z*)-3-methoxyprop-1-enyl]borate

Crystal data

$K^+ \cdot C_4H_7BF_3O^-$

$M_r = 178.01$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 10.882\ (2)\ \text{\AA}$

$b = 7.2668\ (15)\ \text{\AA}$

$c = 9.2317\ (18)\ \text{\AA}$

$\beta = 101.52\ (3)^\circ$

$V = 715.3\ (3)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 360$

$D_x = 1.653\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 9536 reflections

$\theta = 2.3\text{--}21.8^\circ$

$\mu = 0.72\ \text{mm}^{-1}$

$T = 291\ (2)\ \text{K}$

Block, colourless

$0.31 \times 0.22 \times 0.11\ \text{mm}$

Data collection

Enraf–Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

1327 independent reflections

1182 reflections with $I > 2\sigma(I)$

Monochromator: graphite $R_{\text{int}} = 0.059$
 $T = 290(2)$ K $\theta_{\text{max}} = 25.5^\circ$
 φ and ω scans $\theta_{\text{min}} = 4.3^\circ$
 Absorption correction: multi-scan
 (SADABS; Bruker, 2006) $h = -13 \rightarrow 13$
 $T_{\text{min}} = 0.804$, $T_{\text{max}} = 0.924$ $k = -8 \rightarrow 8$
 16605 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
 $R[F^2 > 2\sigma(F^2)] = 0.027$ H-atom parameters constrained
 $wR(F^2) = 0.072$ $w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.2723P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.00$ $(\Delta/\sigma)_{\text{max}} < 0.001$
 1327 reflections $\Delta\rho_{\text{max}} = 0.25 \text{ e } \text{\AA}^{-3}$
 92 parameters $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$
 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
B	0.64509 (18)	-0.7960 (2)	0.0837 (2)	0.0305 (4)
C1	0.77652 (16)	-0.7044 (2)	0.16327 (19)	0.0371 (4)
H1	0.8143	-0.7569	0.253	0.045*
C2	0.83849 (15)	-0.5637 (2)	0.11809 (19)	0.0352 (4)
H2	0.9123	-0.5254	0.1798	0.042*
C3	0.79730 (17)	-0.4641 (2)	-0.02438 (18)	0.0379 (4)
H3A	0.863	-0.4722	-0.0814	0.045*
H3B	0.7233	-0.5242	-0.0806	0.045*
C4	0.8776 (2)	-0.1624 (3)	0.0361 (3)	0.0588 (6)
H4A	0.8539	-0.0349	0.0304	0.088*
H4B	0.9337	-0.1857	-0.0299	0.088*

supplementary materials

H4C	0.9186	-0.1916	0.1354	0.088*
F1	0.63816 (10)	-0.84405 (14)	-0.06632 (11)	0.0438 (3)
F2	0.61847 (9)	-0.95573 (12)	0.16043 (10)	0.0396 (3)
F3	0.54238 (9)	-0.67152 (13)	0.08481 (11)	0.0384 (3)
K	0.40701 (3)	-0.83116 (5)	0.27917 (4)	0.03619 (15)
O	0.76899 (12)	-0.27319 (17)	-0.00483 (16)	0.0469 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
B	0.0374 (9)	0.0264 (9)	0.0288 (9)	-0.0009 (7)	0.0095 (7)	0.0035 (7)
C1	0.0362 (9)	0.0389 (9)	0.0344 (8)	-0.0009 (7)	0.0030 (7)	0.0085 (7)
C2	0.0304 (8)	0.0362 (9)	0.0376 (9)	-0.0027 (7)	0.0034 (7)	0.0001 (7)
C3	0.0488 (10)	0.0295 (9)	0.0353 (9)	-0.0089 (7)	0.0080 (7)	-0.0018 (7)
C4	0.0504 (12)	0.0335 (10)	0.0867 (17)	-0.0095 (9)	-0.0005 (11)	-0.0073 (10)
F1	0.0508 (6)	0.0504 (6)	0.0312 (5)	-0.0058 (5)	0.0104 (4)	-0.0054 (4)
F2	0.0477 (6)	0.0291 (5)	0.0412 (6)	-0.0057 (4)	0.0064 (4)	0.0077 (4)
F3	0.0359 (5)	0.0332 (5)	0.0454 (6)	0.0032 (4)	0.0059 (4)	0.0007 (4)
K	0.0380 (2)	0.0316 (2)	0.0396 (2)	0.00022 (14)	0.00919 (16)	0.00628 (15)
O	0.0390 (7)	0.0293 (6)	0.0682 (9)	-0.0014 (5)	0.0007 (6)	0.0024 (6)

Geometric parameters (\AA , $^\circ$)

B—F1	1.415 (2)	C2—H2	0.9300
B—F2	1.4198 (19)	C3—O	1.440 (2)
B—F3	1.440 (2)	C3—H3A	0.9700
B—C1	1.615 (3)	C3—H3B	0.9700
B—K	3.450 (2)	C4—O	1.417 (2)
C1—C2	1.337 (2)	C4—H4A	0.9600
C1—H1	0.9300	C4—H4B	0.9600
C2—C3	1.490 (2)	C4—H4C	0.9600
F1—B—F2	108.08 (13)	O—C3—H3A	109.0
F1—B—F3	105.82 (13)	C2—C3—H3A	109.0
F2—B—F3	105.89 (13)	O—C3—H3B	109.0
F1—B—C1	114.59 (15)	C2—C3—H3B	109.0
F2—B—C1	111.11 (13)	H3A—C3—H3B	107.8
F3—B—C1	110.86 (13)	O—C4—H4A	109.5
C2—C1—B	129.00 (15)	O—C4—H4B	109.5
C2—C1—H1	115.5	H4A—C4—H4B	109.5
B—C1—H1	115.5	O—C4—H4C	109.5
C1—C2—C3	124.44 (15)	H4A—C4—H4C	109.5
C1—C2—H2	117.8	H4B—C4—H4C	109.5
C3—C2—H2	117.8	C4—O—C3	113.13 (14)
O—C3—C2	113.05 (14)		
F1—B—C1—C2	50.8 (3)	B—C1—C2—C3	-2.2 (3)
F2—B—C1—C2	173.64 (17)	C1—C2—C3—O	116.83 (19)
F3—B—C1—C2	-68.9 (2)	C2—C3—O—C4	76.3 (2)

Fig. 1

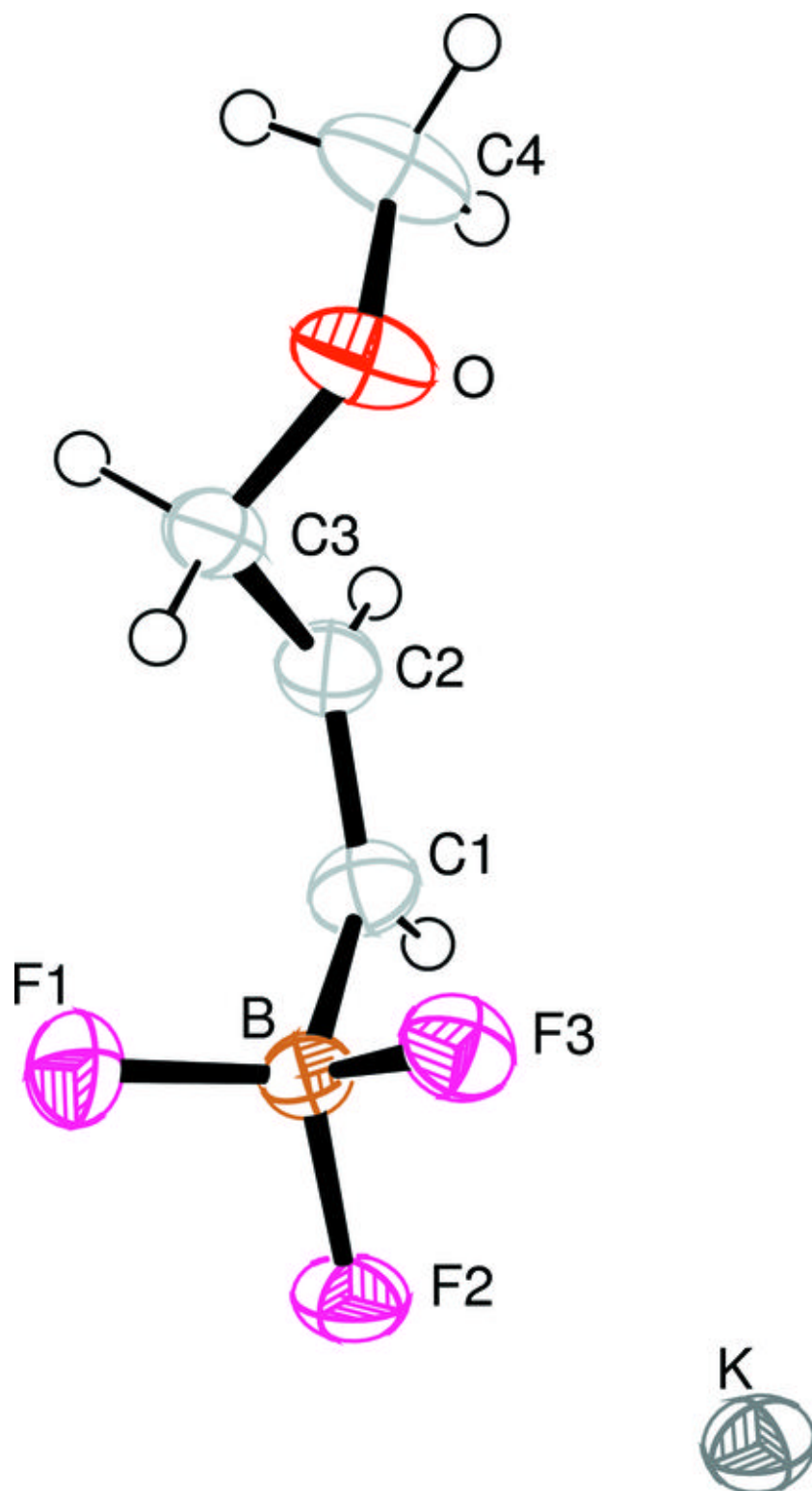


Fig. 2

