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Potassium trifluoro[(*E*)-2-phenyl-1-ethenyl]borate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.026
wR factor = 0.067
Data-to-parameter ratio = 13.2

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

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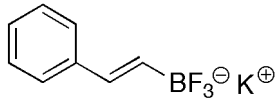
In the title compound, $K^+ \cdot C_8H_7BF_3^-$, the closest distance between the K^+ and $C_8H_7BF_3^-$ moieties is a K···F distance of 2.1619 (17) Å. The overall structure consists of two-dimensional sheets in which $C_8H_7BF_3^-$ moieties coordinate to both sides of layers of K^+ ions.

Comment

While (*E*)-2-phenyl-1-ethenylboronic acid, along with other aryl- and alkenylboronic acids, has been successfully employed in rhodium(I)-catalyzed 1,4-additions to α,β-unsaturated carbonyl compounds (Sakai *et al.*, 1997) and additions to aldehydes (Sakai *et al.*, 1998), the title compound, (I), has shown greater reactivity in analogous processes (Batey *et al.*, 1999). The potassium trifluoroborate salt, (I), is also considerably more stable towards air and water than the corresponding boronic acid, and consequently facile to prepare, isolate, store, and handle.



(I)

Compound (I) crystallizes as discrete K^+ and $C_8H_7BF_3^-$ moieties (Fig. 1). The closest distance between a cation and an anion is 2.1619 (17) Å, which is between K1 and F1. In the structure, each K^+ is surrounded by five anions making close contacts with seven F atoms (Fig. 2) with distances ranging between 2.6169 (17) and 2.8924 (16) Å (see Table 1). The overall structure consists of two-dimensional sheets in which the $-BF_3$ groups of $C_8H_7BF_3^-$ are coordinated above and below layers of K^+, and the terminal phenyl groups point away from the centre on either side of the layers. Hence, each two-dimensional sheet is separated by normal van der Waals interactions between terminal phenyl groups (Fig. 3).

A search of the April 2001 release of the Cambridge Structural Database (Allen & Kennard, 1993) revealed only three other structures of potassium trifluoroborate salts, namely potassium methyltrifluoroborate (Brauer *et al.*, 1982), potassium trifluoromethyltrifluoroborate (Brauer *et al.*, 1977), and potassium phenyltrifluoroborate (Conole *et al.*, 1995).

Experimental

Crystals of (I) were obtained by treatment of (E)-2-phenyl-1-ethenylboronic acid in a minimal amount of methanol with aqueous potassium hydrogen fluoride. The precipitate was subsequently collected by filtration, and recrystallized from acetonitrile.

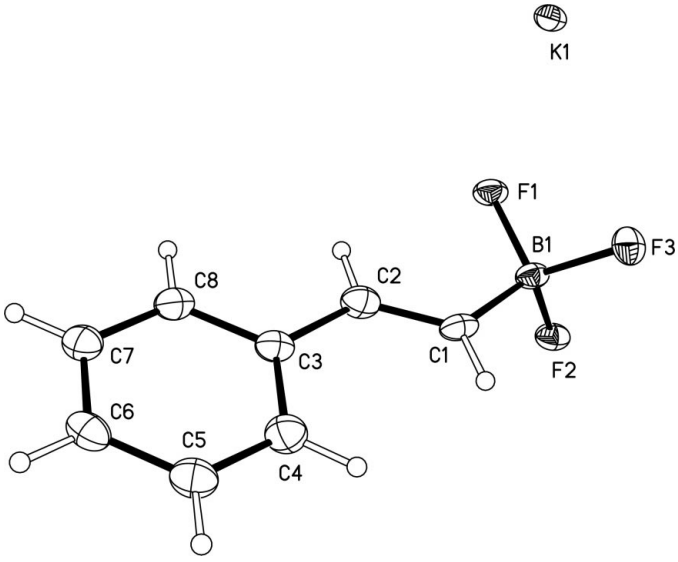


Figure 1
View of (I) showing the atom-labelling scheme. Ellipsoids are shown at the 50% probability level.

Crystal data

$K^+ \cdot C_8H_7BF_3^-$
M_r = 210.05
Orthorhombic, $Pca2_1$
a = 7.0265 (4) Å
b = 18.9749 (10) Å
c = 7.1157 (4) Å
V = 948.72 (9) Å^3
Z = 4
D_x = 1.471 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5135 reflections
θ = 4.2–25.0°
μ = 0.55 mm^{-1}
T = 100 (1) K
Plate, colourless
0.30 × 0.30 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ scans, and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.852, T_{max} = 0.973
5135 measured reflections
1576 independent reflections
1488 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 25.0°
$h = -8 \rightarrow 8$
$k = -22 \rightarrow 22$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.067
S = 1.06
1576 reflections
119 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0117P)^2 + 0.3254P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.50 (4)

Table 1
Selected bond lengths (Å).

K1—F1	2.6169 (17)	K1—F2^{iii}	2.8275 (14)
K1—F3^{i}	2.6235 (15)	K1—F3^{iv}	2.8934 (16)
K1—F2^{ii}	2.6573 (16)	B1—C1	1.583 (4)
K1—F1^{iii}	2.6993 (15)	C1—C2	1.330 (4)
K1—F2^{iv}	2.7106 (17)		

Symmetry codes: (i) $\frac{3}{2}-x, y, z-\frac{1}{2}$; (ii) $x, y, z-1$; (iii) $\frac{1}{2}-x, y, z-\frac{1}{2}$; (iv) $1-x, -y, z-\frac{1}{2}$.

H atoms were included in calculated positions with C—H distances of 0.95 Å. 668 Friedel pairs were used to determine the absolute stereochemistry and the Flack parameter refined to 0.50 (4) indicating the crystal was a racemic twin (Flack, 1983).

Data collection: *COLLECT* (Nonius, 1997–2001); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

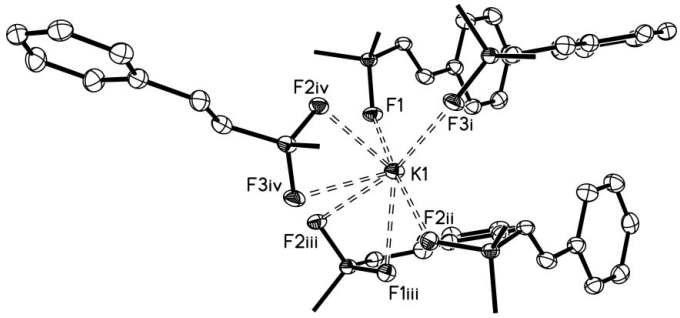


Figure 2
View of K^+ cation environment with the five closest anions. Only F atoms involved in close contacts are labelled. Ellipsoids are shown at the 50% probability level.

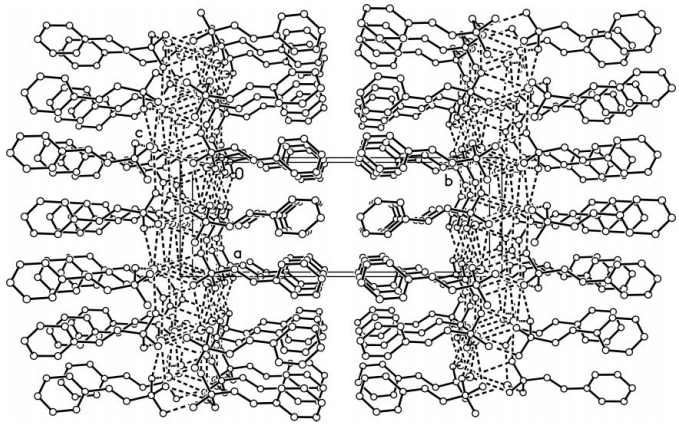


Figure 3
Diagram viewed approximately perpendicular to the *ab* plane showing the packing of two-dimensional sheets.

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