

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for (2)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^*$$

	x	y	z	U_{eq}
Mg1	0	0	0	0.0224 (3)
O1	-0.0551 (3)	-0.1321 (3)	0.06030 (5)	0.0306 (4)
O2	-0.4100 (3)	-0.0685 (3)	0.07154 (6)	0.0354 (4)
O3	0.3435 (3)	-0.7882 (3)	0.11978 (6)	0.0366 (4)
O4	-0.1922 (3)	-0.2839 (3)	-0.02889 (6)	0.0336 (4)
O5	-0.2916 (3)	0.2246 (3)	-0.00191 (7)	0.0287 (4)
C1	-0.2264 (3)	-0.1805 (4)	0.07987 (7)	0.0247 (5)
C2	-0.2154 (4)	-0.3834 (5)	0.11325 (8)	0.0311 (5)
C3	-0.0287 (4)	-0.5042 (4)	0.12352 (7)	0.0259 (5)
C4	0.0128 (4)	-0.7119 (4)	0.15430 (7)	0.0271 (5)
C5	0.2057 (4)	-0.8527 (4)	0.15210 (7)	0.0281 (5)
C6	0.2544 (4)	-1.0472 (5)	0.18146 (9)	0.0375 (6)
C7	0.1084 (5)	-1.1059 (5)	0.21307 (9)	0.0436 (7)
C8	-0.0852 (5)	-0.9716 (5)	0.21513 (9)	0.0460 (7)
C9	-0.1329 (5)	-0.7774 (5)	0.18624 (8)	0.0379 (6)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (2)

Mg1—O1	1.995 (2)	C2—C3	1.315 (3)
Mg1—O4	2.067 (2)	C3—C4	1.460 (3)
Mg1—O5	2.130 (2)	O2...O5	2.846 (3)
O1—C1	1.253 (3)	O2...O5 ⁱ	2.777 (3)
O2—C1	1.268 (3)	O2...O3 ⁱⁱ	2.628 (3)
O3—C5	1.369 (3)	O3...O4 ⁱⁱⁱ	2.833 (3)
C1—C2	1.480 (3)	O4...O5 ^{iv}	2.845 (3)
O1—Mg1—O4	89.37 (8)	O2—C1—C2	118.5 (2)
O1—Mg1—O5	91.81 (8)	C3—C2—C1	121.0 (2)
O4—Mg1—O5	88.54 (8)	C2—C3—C4	128.9 (2)
O1—C1—O2	123.1 (2)	C9—C4—C3	122.9 (2)
O1—C1—C2	118.4 (2)		

Symmetry codes: (i) $-1 - x, -y, -z$; (ii) $x - 1, 1 + y, z$; (iii) $-x, -1 - y, -z$; (iv) $x, y - 1, z$.

All non-H atoms were assigned anisotropic displacement parameters. Water and hydroxyl H atoms were located in difference Fourier synthesis maps while the remainder were placed in calculated positions.

For both compounds, data collection: *CAD-4 Software* (Enraf-Nonius, 1989); cell refinement: *CAD-4 Software*; program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *SHELXL92* (Sheldrick, 1992); molecular graphics: *DTMM* (Crabbe & Appleyard, 1991); software used to prepare material for publication: *SHELXL92*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monosodium L-Glutamate Pentahydrate

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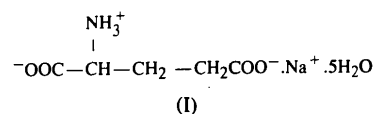
(Received 6 January 1994; accepted 12 December 1994)

Abstract

The title compound, $\text{Na}^+ \cdot \text{C}_5\text{H}_8\text{NO}_4^- \cdot 5\text{H}_2\text{O}$, crystallizes with two independent chemical units, composed of two L-glutamate anions (Glu A and Glu B), two Na^+ ions and ten water molecules, per asymmetric unit. Each Na^+ ion is coordinated by six water molecules in an octahedral arrangement. Strands of coordination octahedra sharing edges with neighbouring octahedra along the *a* axis are linked to one another through hydrogen bonds, forming extended layers parallel to the *ac* plane. Each of the Glu A and Glu B anions also forms hydrogen-bonded layers parallel to the *ac* plane on either side of the Na^+ -water coordination layer. The crystal packing is dominated by stacking of this triple-layer structure.

Comment

Monosodium L-glutamate (MSG) pentahydrate was found in 1936 and reported by Ogawa (1949). It crystallizes from a concentrated aqueous solution at a temperature between 264.5 and 272.2 K. At room temperature it transforms immediately to the MSG monohydrate crystal. Compared with the monohydrate crystal, the pentahydrate crystal has a higher selectivity for impurities such as other L-amino acids and colouring substances, *i.e.* these impurities are incorporated less in the pentahydrate crystal than in the monohydrate crystal when crystals are grown from an impure solution. The structure analysis of the pentahydrate crystal (I) was undertaken as a part of an investigation of these phenomena.



The unit cell contains two independent chemical units. Each Na⁺ ion coordinates in an octahedral fashion only with water O atoms at distances of 2.34–2.51 Å, with O(W)··Na⁺··O(W) angles in the range 82.1–101.6°. In the monohydrate crystal (Sano, Nagashima, Kawakita & Iitaka, 1989), which also has two chemical units per asymmetric unit, two independent Na⁺ ions are octahedrally coordinated to four carboxyl O atoms (three α and one γ) and two water molecules. The coordination octahedra of Na(1) and Na(2) share edges [O(WB)–O(WE) and O(WC)–O(WD)] to form stranded chains along the *a* axis. Thus, the number of coordinated water molecules in the unit cell is eight. Chains of coordination octahedra are connected to neighbouring chains by hydrogen bonds, forming an extended layer structure parallel to the *ac* plane. The Na⁺–water coordination chains seen in this crystal structure are also present in inorganic acid salt polyhydrates such as sodium sulfate decahydrate (Glauger's Salt) and sodium tetraborate decahydrate (Borax) (Levy & Lisensky, 1978).

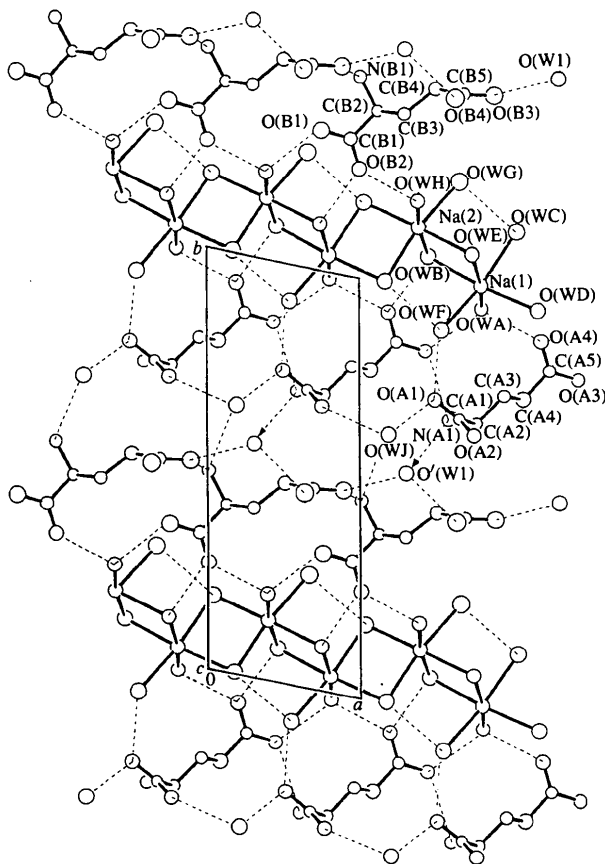


Fig. 1. The molecular packing in the crystal viewed along the *c* axis, showing the atomic numbering scheme. H atoms are omitted for clarity. Selected hydrogen bonds are shown by dashed lines and the arrows indicate hydrogen bonds to atoms related by translational symmetry in the minus direction of the *c* axis.

Both the Glu *A* and Glu *B* anions are zwitterionic (the α- and γ-carboxyl groups are ionized and the amino group is protonated) and exhibit very similar conformations (Table 2). The torsion angles χ^1 [N(1)—C(2)—C(3)—C(4)], χ^2 [C(2)—C(3)—C(4)—C(5)] and χ^3 [C(3)—C(4)—C(5)—O(4)] of the two glutamate ions are *-gauche* (\bar{g}), *trans* (*t*) and *-gauche* (\bar{g}), respectively, while in the monohydrate crystal, the torsion angles of the two ions are \bar{g} , *t*, *g* and *g*, *t*, \bar{g} .

Each Glu *A* and water molecule *J* pair, and Glu *B* and water molecule *I* pair, form hydrogen-bonded layers in which the molecules are related only by translational symmetry, and lie parallel to the *ac* plane on either side of the Na⁺–water coordination layer. The non-coordinated water molecules *J* and *I* play an important role in the formation of the hydrogen-bond network. Water molecule *J* (or *I*) participates in four hydrogen bonds and bridges four glutamate ions of which three are Glu *A* (or Glu *B*) in intralayer bonds and one is Glu *B* (or Glu *A*).

The number of hydrogen bonds between layers indicates the tightness of the interlayer binding of these three layers (Table 3). The Na⁺–water coordination layer and the Glu *A* (or Glu *B*) anion layer are interlinked tightly by six (or five) interlayer hydrogen bonds per asymmetric unit forming a triple-layer structure. On the other hand, the Glu *A* and Glu *B* layers are bound weakly with only two hydrogen bonds, through water molecules *I* and *J*, per asymmetric unit. A notable feature of the crystal structure is the triple-layer stacking which consists of a layer of Glu *A* anions, a layer of Na⁺–water coordination octahedra and a layer of Glu *B* anions (Fig. 1). This triple-layer structure should be important when considering the crystal-growth mechanism.

Experimental

The title compound is a product of the purification process of the manufacture of monosodium L-glutamate monohydrate.

Crystal data

Na⁺·C₃H₈NO₄⁻·5H₂O

M_r = 259.19

Triclinic

*P*1

a = 6.116 (2) Å

b = 16.656 (4) Å

c = 6.007 (3) Å

α = 99.29 (3)°

β = 100.94 (3)°

γ = 99.05 (2)°

V = 581.7 (4) Å³

Z = 2

D_x = 1.480 Mg m⁻³

Cu Kα radiation

λ = 1.5418 Å

Cell parameters from 25 reflections

θ = 25.4–35.2°

μ = 1.51 mm⁻¹

T = 243 K

Plate

0.5 × 0.4 × 0.2 mm

Colourless

Data collection

Rigaku AFC-5S diffractometer

R_{int} = 0.065

θ_{max} = 60.05°

Profile data from ω - 2θ scans $h = 0 \rightarrow 6$
 Absorption correction: $k = -18 \rightarrow 18$
 ψ scan (North, Phillips
 & Mathews, 1968) $l = -6 \rightarrow 6$
 $T_{\min} = 0.93$, $T_{\max} = 1.00$
 1910 measured reflections 3 standard reflections
 1722 independent reflections monitored every 150
 1343 observed reflections reflections
 $[I > 3.0\sigma(I)]$ intensity decay: 2.2%

Refinement

Refinement on F $R = 0.056$ $wR = 0.076$ $S = 3.06$

1343 reflections

286 parameters

H-atom parameters not
refined $w = 1/(2|F_{\min}| + |F|$
 $+ 2|F|^2/|F_{\max}|)$ $(\Delta/\sigma)_{\max} = 0.10$ $\Delta\rho_{\max} = 0.38 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -0.52 \text{ e } \text{Å}^{-3}$ Atomic scattering factors
from *International Tables*
for *X-ray Crystallography*
(1974, Vol. IV, Table
2.2A)Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	U_{eq}
Na(1)	0.7996	0.0382	0.0644	0.032 (1)
Na(2)	0.3923 (7)	0.1432 (3)	0.2404 (7)	0.032 (1)
O(A1)	0.496 (1)	-0.2547 (4)	-0.102 (1)	0.032 (3)
O(A2)	0.752 (1)	-0.3224 (4)	0.058 (1)	0.030 (3)
O(A3)	1.434 (1)	-0.1424 (4)	-0.499 (1)	0.028 (3)
O(A4)	1.191 (1)	-0.0668 (4)	-0.390 (1)	0.033 (3)
N(A1)	0.587 (1)	-0.2971 (5)	-0.521 (1)	0.024 (3)
C(A1)	0.657 (1)	-0.2899 (5)	-0.099 (1)	0.019 (3)
C(A2)	0.768 (1)	-0.2929 (5)	-0.314 (1)	0.022 (4)
C(A3)	0.950 (2)	-0.2141 (5)	-0.273 (2)	0.024 (4)
C(A4)	1.073 (2)	-0.2132 (7)	-0.472 (2)	0.035 (4)
C(A5)	1.246 (1)	-0.1346 (6)	-0.448 (2)	0.023 (4)
O(B1)	-0.248 (1)	0.3289 (5)	0.802 (1)	0.043 (4)
O(B2)	0.005 (1)	0.2546 (4)	0.707 (1)	0.033 (3)
O(B3)	0.891 (1)	0.4888 (5)	1.244 (1)	0.044 (4)
O(B4)	0.635 (1)	0.4650 (6)	1.450 (1)	0.049 (4)
N(B1)	0.010 (1)	0.4729 (5)	0.820 (1)	0.025 (3)
C(B1)	-0.054 (1)	0.3220 (6)	0.764 (2)	0.028 (4)
C(B2)	0.123 (1)	0.4007 (5)	0.803 (2)	0.020 (4)
C(B3)	0.301 (1)	0.4059 (5)	1.017 (2)	0.025 (4)
C(B4)	0.506 (1)	0.4784 (6)	1.059 (2)	0.029 (4)
C(B5)	0.690 (1)	0.4782 (5)	1.260 (2)	0.024 (4)
O(WA)	0.802 (1)	-0.0157 (4)	-0.320 (1)	0.029 (3)
O(WB)	0.455 (1)	0.0816 (4)	-0.121 (1)	0.035 (3)
O(WC)	1.033 (1)	0.1775 (4)	0.096 (1)	0.039 (3)
O(WD)	1.169 (1)	0.0051 (5)	0.208 (1)	0.037 (3)
O(WE)	0.742 (1)	0.1058 (4)	0.423 (1)	0.030 (3)
O(WF)	0.540 (1)	-0.0854 (5)	0.098 (1)	0.038 (3)
O(WG)	0.662 (1)	0.2706 (5)	0.220 (1)	0.046 (4)
O(WH)	0.388 (1)	0.2052 (4)	0.617 (1)	0.032 (3)
O(WI)	1.307 (1)	0.5575 (4)	1.544 (1)	0.033 (3)
O(WJ)	0.192 (1)	-0.3588 (5)	0.068 (1)	0.037 (3)

Table 2. Selected geometric parameters (Å , $^\circ$)

O(A1)—C(A1)	1.22 (1)	O(B1)—C(B1)	1.27 (1)
O(A2)—C(A1)	1.259 (9)	O(B2)—C(B1)	1.25 (1)
O(A3)—C(A5)	1.26 (1)	O(B3)—C(B5)	1.24 (1)
O(A4)—C(A5)	1.24 (1)	O(B4)—C(B5)	1.29 (1)
N(A1)—C(A2)	1.48 (1)	N(B1)—C(B2)	1.48 (1)
C(A1)—C(A2)	1.57 (1)	C(B1)—C(B2)	1.52 (1)
C(A2)—C(A3)	1.54 (1)	C(B2)—C(B3)	1.50 (1)
C(A3)—C(A4)	1.52 (1)	C(B3)—C(B4)	1.55 (1)
C(A4)—C(A5)	1.52 (1)	C(B4)—C(B5)	1.49 (1)

O(A1)—C(A1)—O(A2)	128.3 (7)	O(B1)—C(B1)—O(B2)	124.3 (8)
O(A1)—C(A1)—C(A2)	116.9 (6)	O(B1)—C(B1)—C(B2)	117.9 (8)
O(A2)—C(A1)—C(A2)	114.7 (6)	O(B2)—C(B1)—C(B2)	117.7 (7)
N(A1)—C(A2)—C(A1)	108.0 (6)	N(B1)—C(B2)—C(B1)	109.2 (6)
N(A1)—C(A2)—C(A3)	110.9 (6)	N(B1)—C(B2)—C(B3)	111.5 (7)
C(A1)—C(A2)—C(A3)	108.5 (6)	C(B1)—C(B2)—C(B3)	109.6 (7)
C(A2)—C(A3)—C(A4)	112.0 (7)	C(B2)—C(B3)—C(B4)	114.4 (7)
C(A3)—C(A4)—C(A5)	114.5 (8)	C(B3)—C(B4)—C(B5)	113.6 (7)
O(A3)—C(A5)—O(A4)	124.2 (8)	O(B3)—C(B5)—O(B4)	120.7 (8)
O(A3)—C(A5)—C(A4)	117.5 (8)	O(B3)—C(B5)—C(B4)	120.6 (7)
O(A4)—C(A5)—C(A4)	118.2 (7)	O(B4)—C(B5)—C(B4)	118.6 (7)
O(A1)—C(A1)—C(A2)—N(A1)	-32.6 (9)		
O(A1)—C(A1)—C(A2)—C(A3)	87.7 (9)		
O(A2)—C(A1)—C(A2)—N(A1)	149.6 (7)		
O(A2)—C(A1)—C(A2)—C(A3)	-90.1 (8)		
O(A3)—C(A5)—C(A4)—C(A3)	137.3 (8)		
O(A4)—C(A5)—C(A4)—C(A3)	-46 (1)		
N(A1)—C(A2)—C(A3)—C(A4)	-63.3 (9)		
C(A1)—C(A2)—C(A3)—C(A4)	178.3 (7)		
C(A2)—C(A3)—C(A4)—C(A5)	176.6 (7)		
O(B1)—C(B1)—C(B2)—N(B1)	-15 (1)		
O(B1)—C(B1)—C(B2)—C(B3)	107.5 (9)		
O(B2)—C(B1)—C(B2)—N(B1)	168.2 (7)		
O(B2)—C(B1)—C(B2)—C(B3)	-69.4 (9)		
O(B3)—C(B5)—C(B4)—C(B3)	133.7 (8)		
O(B4)—C(B5)—C(B4)—C(B3)	-45 (1)		
N(B1)—C(B2)—C(B3)—C(B4)	-65.7 (8)		
C(B1)—C(B2)—C(B3)—C(B4)	173.3 (6)		
C(B2)—C(B3)—C(B4)—C(B5)	-174.8 (7)		

Table 3. Intermolecular hydrogen bonds (Å)

D	A	$D \cdots A$	D	A	$D \cdots A$
Within the L-Glu A layer (including water molecule J)			Within the L-Glu B layer (including water molecule I)		
N(A1)	O(A2 ⁱ)	2.888 (8)	N(B1)	O(B3 ⁱⁱ)	2.762 (9)
N(A1)	O(A3 ⁱⁱ)	2.868 (9)	N(B1)	O(B4 ⁱⁱⁱ)	2.845 (9)
N(A1)	O(WJ ^{iv})	3.021 (9)	N(B1)	O(WI ^v)	3.020 (8)
O(WJ)	O(A1)	2.792 (8)	O(WI)	O(B3)	2.779 (8)
O(WJ)	O(A2 ⁱⁱ)	2.841 (8)	O(WI)	O(B4 ⁱⁱⁱ)	2.80 (1)
Within the layer of Na^+ - H_2O coordination octahedra			Between the L-Glu A and B layers		
O(WB)	O(WH ⁱ)	2.819 (9)	N(A1)	O(WI ^{vi})	2.864 (8)
O(WC)	O(WG)	3.08 (1)	N(B1)	O(WJ ^{vii})	2.89 (1)
O(WD)	O(WF ⁱⁱⁱ)	3.03 (1)			
O(WE)	O(WA ^{iv})	2.765 (8)			
Between the L-Glu A layer and Na^+ - H_2O coordination octahedra			Between the L-Glu B layer and Na^+ - H_2O coordination octahedra		
O(WA)	O(A3 ⁱⁱ)	2.745 (8)	O(WC)	O(B2 ^{ix})	2.829 (9)
O(WA)	O(A4)	2.728 (9)	O(WE)	O(B2 ⁱⁱⁱ)	2.824 (9)
O(WB)	O(A4 ⁱⁱ)	2.790 (9)	O(WG)	O(B1 ^{ix})	2.95 (1)
O(WD)	O(A4 ^{iv})	2.855 (9)	O(WH)	O(B1 ⁱⁱⁱ)	2.706 (9)
O(WF)	O(A1)	2.831 (9)	O(WH)	O(B2)	2.721 (8)
O(WF)	O(A3 ^v)	2.889 (8)			

Symmetry codes: (i) $x, y, z - 1$; (ii) $x - 1, y, z$; (iii) $x + 1, y, z$; (iv) $x, y, z + 1$; (v) $x - 1, y, z + 1$; (vi) $x - 1, y, z - 1$; (vii) $x - 1, y - 1, z - 2$; (viii) $x, y + 1, z + 1$; (ix) $x + 1, y, z - 1$.

The mounted crystal was cooled at 243 K in an N_2 gas stream from liquid N_2 using a low-temperature system (Molecular Structure Corporation).

Most H atoms were located on difference Fourier maps and the six remaining atoms were placed in calculated positions, but because of the poor ratio of the number of reflections to the number of parameters, the H-atom parameters were not included in the refinement.

Data collection: *MSC/AFSC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1984)

and *DIRDIF* (Beurskens, 1984). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1110). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Potassium Trifluorophenylborate

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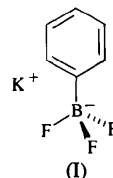
Abstract

X-ray structural analysis confirms that the title compound, potassium trifluorophenylborate, K⁺.C₆H₅BF₃⁻, contains the first structurally characterized example of a trifluorophenylborate anion. The C—C bond lengths of the phenyl ring lie within the range 1.38 (2)–1.41 (2) Å, consistent with π-electron delocalization; the three B—F bond lengths are 1.41 (1), 1.41 (1) and 1.43 (1) Å. The

anion can be considered as a Lewis acid complex of BF₃ and Ph⁻, and as expected from a simple VSEPR model, the geometry around the B atom is tetrahedral, although the presence of two different substituents around the B atom results in two distinct sets of bond angles, with significantly smaller values for the three F—B—F angles [104.4 (7), 106.0 (7) and 106.7 (9)°] compared with the remaining three C(phenyl)—B—F angles [112.1 (7), 112.9 and 114.1 (7)°].

Comment

This work is part of a general investigation into the structures of Lewis acids and their complexes, as an aid to efficient parameterization of Lewis acid complexes for molecular modelling (Allinger, 1977; Eliel, Allinger, Angyal & Morrison, 1965; Burkert & Allinger, 1982). The tetrafluoroborate and tetraphenylborate anions are widely used as counterions in both organic and inorganic chemistry, and the number of reports of crystal structures containing such anions run into the thousands. The crystal structure determination of the title compound, (I), is the first of an intermediate between these two anions, and thus, the effect of substitution of one of the F atoms by a phenyl group on the molecular geometry of the anion can be assessed. This asymmetric anion may also prove to be a better counter-ion than the tetrafluoroborate anion, which is frequently prone to crystallographic disorder.



X-ray structural analysis shows that potassium trifluorophenylborate consists of discrete K⁺ cations and BF₃Ph⁻ anions. The structure crystallizes in the non-centrosymmetric orthorhombic space group *Pca*2₁ with one independent molecule in the asymmetric unit. A perspective *ORTEPII* (Johnson, 1976) drawing of the trifluorophenylborate anion in the asymmetric unit is shown in Fig. 1.

The phenyl ring bond distances in the trifluorophenylborate anions are consistent with π-electron delocalization and lie within the range 1.38 (2)–1.41 (2) Å; similarly, the C—C—C bond angles are consistent with *sp*² hybridization and lie within the range 117.7(8)–121(1)°. The phenyl ring is essentially planar with only slight deviations from planarity (maximum deviation 0.01 Å).

The three B—F bond distances of 1.41 (1), 1.41 (1) and 1.43 (1) Å are within the range observed for compounds containing the related tetrafluoroborate anion. Recent literature examples include the selenium compound [(C₁₀H₁₂S₂Se₂)₂][BF₄], which has a