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Tris(L-argininium) hexafluorochromate(III) trihydrate

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Key indicators

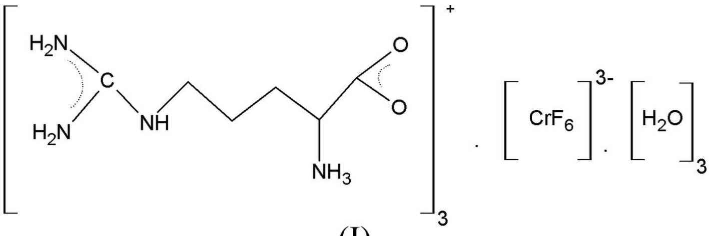
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.028
wR factor = 0.076
Data-to-parameter ratio = 14.2

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

The title compound, $(C_6H_{15}N_4O_2)_3\cdot CrF_6\cdot 3H_2O$, is isostructural with the vanadium analogue. One of the argininium cations is partially disordered over two different conformations. A complex network of N—H···O, N—H···F, O—H···O and O—H···F hydrogen bonds supports the structure.

Comment

The title compound, (I) (Fig. 1), is isostructural with tris(L-argininium) hexafluorovanadate(III) trihydrate, (II) (Ramos Silva, Matos Beja, Paixão & Alte da Veiga, 2000). The bond lengths and angles for (I) (Table 1) and the molecular conformations are all very similar to those of the equivalent species in (II). The argininium species in (I) are positively charged, with the guanidyl and amino groups protonated and the carboxyl groups deprotonated. One of the argininium cations is partially disordered, adopting two alternative conformations (Fig. 1). In the two ordered argininium cations (containing atoms C1 and C7), Cγ (*i.e.* atoms C3 and C9, respectively) is in a staggered *trans* position with respect to to the carboxyl C atom, while in the disordered C13-containing molecule, Cγ (atom C15) is in a *gauche* position with respect to both the amino and carboxyl C atoms (Table 1).



(I)

There is an extended and complex three-dimensional network of hydrogen bonds in the crystal structure of (I), three of which are bifurcated, involving 15 donors and 31 H atoms (Table 2). Every F atom accepts at least two hydrogen bonds (Fig. 2), as also seen in a related compound (Ramos Silva, Matos Beja, Costa *et al.*, 2000). Isomorphous compounds of hexafluorovanadate(III) and hexafluorochromate(III) have already been found, for instance trihydrazinium hexafluorochromate(III) and trihydrazinium hexafluorovanadate(III) (Kojić-Prodić *et al.*, 1972).

Magnetic susceptibility measurements on (I) were performed down to 5 K in an applied field of 50 Oe using a powdered sample. The variation of the induced magnetic moment with temperature shows paramagnetic behaviour down to the lowest temperature with no sign of magnetic ordering.

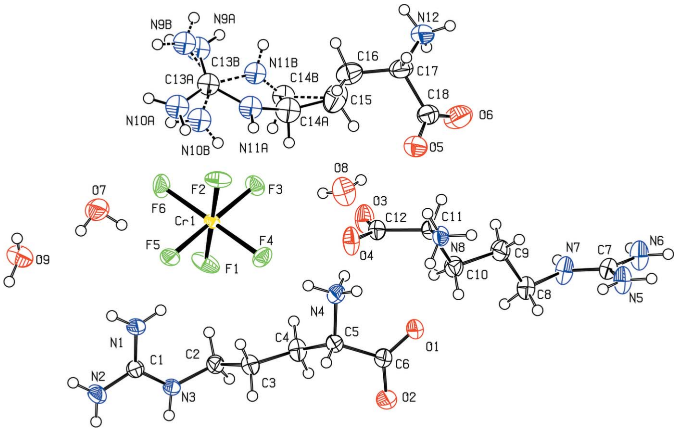


Figure 1
A view of (I), showing 50% probability displacement ellipsoids. Both disorder components are shown.

Experimental

Hydrofluoric acid (Merck, 40%) was reacted with powdered chromium (99.7%) in a plastic flask. The resulting solution was then added to an aqueous solution of L-arginine (Aldrich, 98%) in a 1:3 Cr–arginine molar proportion. Several recrystallizations were carried out using water and ethanol as solvent over a period of several weeks, eventually yielding green prisms of (I).

Crystal data

$(C_6H_{15}N_4O_2)_3[CrF_6]\cdot 3H_2O$
$M_r = 745.71$
Monoclinic, $P2_1$
$a = 10.8194$ (15) Å
$b = 9.0434$ (15) Å
$c = 16.998$ (2) Å
$\beta = 96.890$ (12)°
$V = 1651.2$ (4) Å^3
$Z = 2$
$D_x = 1.500$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.8$–15.7°
$\mu = 0.44$ mm^{-1}
$T = 293$ (2) K
Prism, green
0.34 × 0.22 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Profile-fitted $\omega/2\theta$ scans
Absorption correction: ψ scan North *et al.* (1968)
$T_{\min} = 0.884$, $T_{\max} = 0.984$
12219 measured reflections
6118 independent reflections
5397 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 25.5°$
$h = -13 \rightarrow 13$
$k = -10 \rightarrow 10$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 180 min
intensity decay: 7%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.076$
$S = 1.05$
6118 reflections
432 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.3634P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Absolute structure: Flack (1983), with 2845 Friedel pairs
Flack parameter: 0.010 (14)

Table 1
Selected geometric parameters (Å, °).

Cr1—F2	1.8855 (14)	Cr1—F3	1.9010 (13)
Cr1—F1	1.8857 (14)	Cr1—F5	1.9020 (13)
Cr1—F6	1.8902 (13)	Cr1—F4	1.9096 (13)
C15—C16—C17—N12	72.1 (3)	C3—C4—C5—C6	144.5 (2)
C15—C16—C17—C18	−52.0 (4)	C9—C10—C11—C12	142.9 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N12—H12*A*···O5$^{\rm i}$	0.89	2.20	3.034 (3)	157
N12—H12*B*···O3$^{\rm ii}$	0.89	2.23	2.948 (3)	137
N12—H12*C*···O7$^{\rm iii}$	0.89	1.92	2.756 (3)	156
N10*A*—H10*B*···F6	0.86	1.89	2.620 (5)	141
N10*A*—H10*B*···F3	0.86	2.60	3.302 (5)	140
N10*B*—H10*D*···F6	0.86	2.32	2.748 (6)	111
N10*B*—H10*C*···F3	0.86	2.03	2.803 (6)	150
N10*A*—H10*A*···O6$^{\rm iv}$	0.86	1.89	2.740 (5)	169
N11*B*—H11*B*···F2$^{\rm iii}$	0.86	1.91	2.744 (5)	163
N11*A*—H11*A*···F3	0.86	1.98	2.823 (4)	165
N9*A*—H9*E*···O8$^{\rm iii}$	0.86	2.43	3.102 (5)	136
N9*B*—H9*H*···O8$^{\rm iii}$	0.86	2.41	2.855 (6)	113
N9*B*—H9*G*···O6$^{\rm iv}$	0.86	2.29	3.117 (6)	160
N9*A*—H9*F*···F2$^{\rm iii}$	0.86	2.13	2.793 (5)	133
N9*B*—H9*H*···F2$^{\rm iii}$	0.86	2.33	3.006 (6)	136
N9*A*—H9*F*···F3$^{\rm iii}$	0.86	2.48	3.072 (5)	127
N9*B*—H9*H*···F3$^{\rm iii}$	0.86	2.64	3.384 (6)	146
N1—H1*A*···O1$^{\rm iv}$	0.86	2.23	3.080 (2)	171
N1—H1*B*···F1	0.86	1.89	2.732 (2)	167
N2—H21···O2$^{\rm iv}$	0.86	1.96	2.818 (3)	176
N2—H22···F4$^{\rm v}$	0.86	2.00	2.820 (2)	158
N3—H3···F5$^{\rm v}$	0.86	1.88	2.726 (2)	168
N4—H4*A*···O4	0.89	2.03	2.901 (3)	168
N4—H4*B*···O9$^{\rm vi}$	0.89	2.09	2.946 (3)	161
N4—H4*C*···F4	0.89	1.84	2.696 (2)	161
N5—H5*B*···O4$^{\rm vi}$	0.86	2.07	2.929 (3)	173
N5—H5*A*···F1$^{\rm vi}$	0.86	1.92	2.747 (2)	162
N6—H6*A*···F4$^{\rm vii}$	0.86	2.42	3.028 (3)	128
N6—H6*A*···F2$^{\rm vii}$	0.86	2.63	3.256 (3)	131
N6—H6*B*···O3$^{\rm vi}$	0.86	1.99	2.846 (3)	171
N7—H7···F2$^{\rm vii}$	0.86	1.89	2.723 (2)	162
N8—H8*A*···O5	0.89	2.03	2.860 (3)	154
N8—H8*B*···F6$^{\rm vi}$	0.89	1.75	2.640 (2)	176
N8—H8*C*···O1	0.89	2.00	2.884 (2)	171
O7—H71···O9	0.79 (4)	2.17 (4)	2.875 (3)	148 (4)
O7—H72···F5	0.88 (4)	1.81 (4)	2.687 (2)	175 (4)
O8—H81···O5	0.78 (4)	2.05 (4)	2.804 (3)	165 (5)
O8—H82···F3	0.79 (4)	2.06 (4)	2.787 (3)	154 (4)
O9—H91···O2$^{\rm viii}$	0.89 (4)	1.85 (4)	2.718 (3)	166 (4)
O9—H92···O3$^{\rm ix}$	0.80 (4)	2.23 (4)	2.996 (3)	160 (4)

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

The amino acid H atoms were placed in calculated positions and refined as riding on their parent atoms, with C—H = 0.97 Å and N—H = 0.86–0.89 Å, and with $U_{\rm iso}$(H) = 1.2 or 1.5$U_{\rm eq}$(C,N). Water H atoms were located in a Fourier difference map and their positions were freely refined, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O). Atoms N11, C14, N9 and N10 and their attached H atoms were found to be disordered over two positions in the ratio 0.547 (4):0.453 (4) and were refined with isotropic displacement factors. Examination of the crystal structure with *PLATON* (Spek, 2003) showed that there are no solvent-accessible voids in the crystal structure.

m2460 Ramos Silva *et al.* • $(C_6H_{15}N_4O_2)_3\cdot CrF_6\cdot 3H_2O$ *Acta Cryst.* (2005). E**61**, m2459–m2461

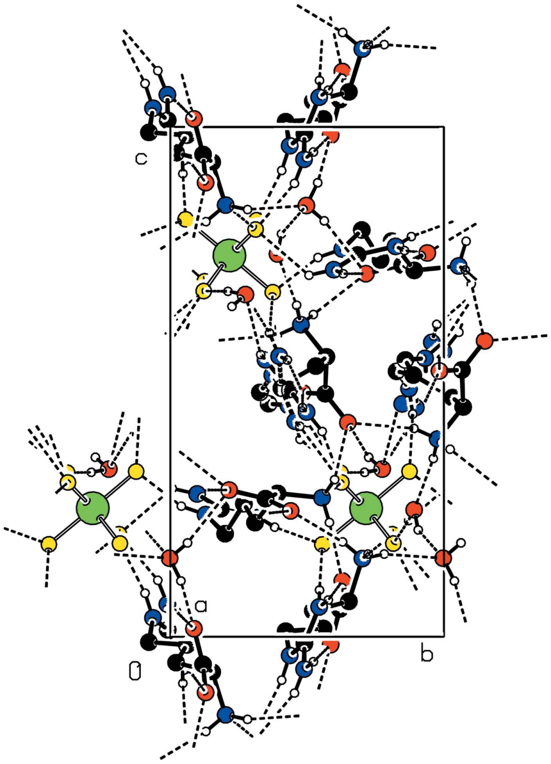


Figure 2
The packing of (I), projected along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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