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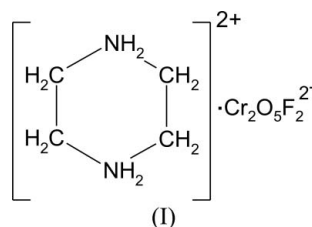Correspondence e-mail: kkara@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.031
wR factor = 0.092
Data-to-parameter ratio = 14.3

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

Piperazinediium μ-oxo-bis(fluorodioxochromate)

In the molecule of the title compound, $(C_4H_{12}N_2)[Cr_2F_2O_5]$, the two crystallographically independent piperazinium dications are both located on centers of inversion, while the anions are located in general positions. Intermolecular N—H⋯F and N—H⋯O and intramolecular N—H⋯O hydrogen bonds may be effective in the stabilization of the crystal structure.

Comment

Many structures of chromates and dichromates, including a few tri-, tetra- and polychromates (Pressprich *et al.*, 1988), have been reported. Chromates may exist in mono-, di-, tri-, tetra- and polymeric forms (Wang *et al.*, 2003; Fouada *et al.*, 1999). We report here the crystal structure of the title compound, (I).

(I)

In the molecule of (I) (Fig. 1), the bond lengths and angles are in normal ranges (Allen *et al.*, 1987). It consists of di(fluorotrioxochromate) dianions and piperazinium dications, where the two crystallographically independent cations are both located on centers of inversion, while the anions are located in general positions.

The rings *A* (C1/N1/C2/C1a/N1a/C2a) and *B* (C3/N2/C4/C3b/N2b/C4b) are not planar, having total puckering amplitudes, Q_T of 1.456 (3) and 1.448 (4) Å, respectively, and chair conformations [φ = −96.82 (5)°, θ = 180.00 (3)° and φ = 40.15 (4)°, θ = 180.00 (3)°; Cremer & Pople, 1975], as usually observed (Srinivasan *et al.*, 2003, 2004). The O—Cr—O bond angles range from 106.73 (11)° to 111.27 (17)° (Table 1). In the anion, the bridging Cr1—O3 [1.782 (2) Å] and Cr2—O3 [1.790 (2) Å] bonds are longer than the terminal Cr—O [average 1.604 (2) Å] bonds. These values are in good agreement with those reported for other dihalochromates (Özyürek *et al.*, 2006; Ding *et al.*, 2004; Chaudhuri *et al.*, 1997).

As can be seen from the packing diagram (Fig. 2), intermolecular N—H⋯F and N—H⋯O hydrogen bonds (Table 2) link the molecules; intramolecular N—H⋯O and intermolecular hydrogen bonds may be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are also effective in the molecular packing.
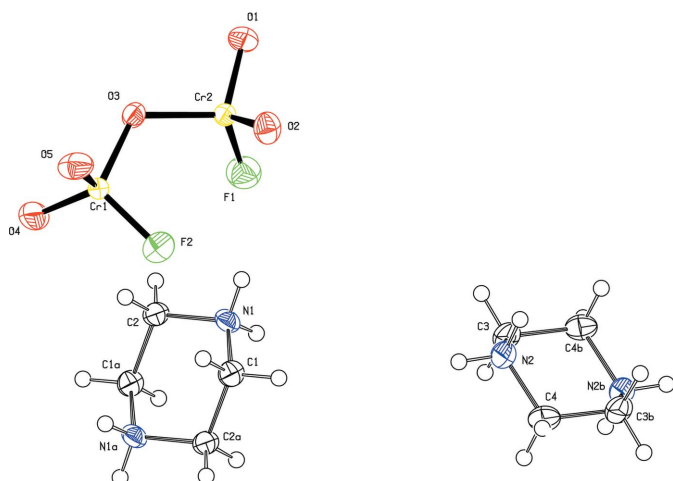
Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (a) $2 - x, -y - 1, 1 - z$; (b) $1 - x, -y, -z$.]
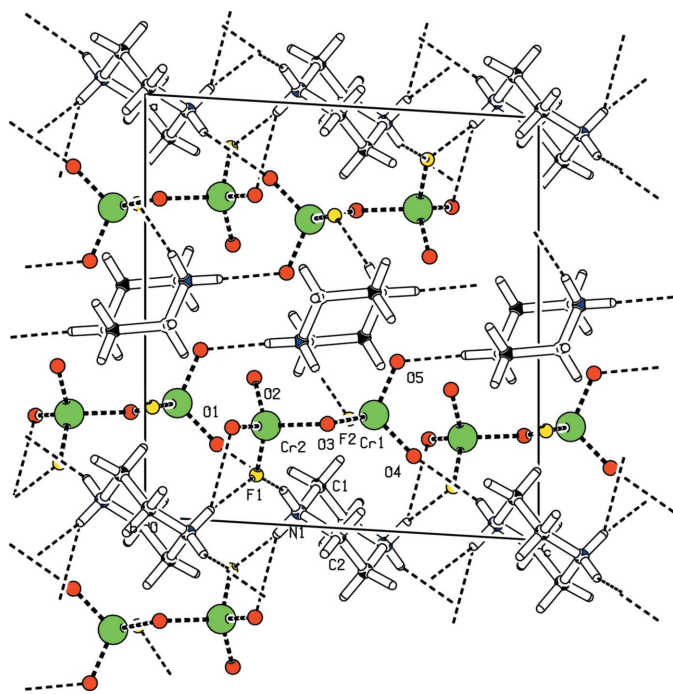

Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

Experimental

Chromium(VI) oxide (20 g, 0.2 mol) was dissolved in water (25 ml) in a polythene beaker, and hydrofluoric acid (11.3 ml, 0.23 mol, 40%) was added to it, with stirring at room temperature. Within 5 min, a clear solution resulted. To this solution, piperazine (17.2 g, 0.2 mol) was added slowly with stirring. The compound obtained was kept at room temperature for crystallization, and after 1 d, orange single crystals suitable for X-ray diffraction were obtained.

Crystal data

$(C_4H_{12}N_2)[Cr_2F_2O_5]$	$Z = 4$
$M_r = 310.16$	$D_x = 2.073$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 11.6467$ (11) Å	$\mu = 2.22$ mm^{-1}
$b = 7.8702$ (5) Å	$T = 294$ (2) K
$c = 10.8605$ (11) Å	Prism, orange
$\beta = 93.452$ (8)°	$0.34 \times 0.29 \times 0.20$ mm
$V = 993.69$ (15) Å^3	

Data collection

Stoe IPDS-II diffractometer	13786 measured reflections
ω scans	1957 independent reflections
Absorption correction: integration	1785 reflections with $I > 2\sigma(I)$
(X-RED32; Stoe & Cie, 2002)	$R_{int} = 0.063$
$T_{min} = 0.562$, $T_{max} = 0.702$	$\theta_{max} = 26.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.031$	$+ 1.7431P]$
$wR(F^2) = 0.092$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.001$
1957 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
137 parameters	$\Delta\rho_{min} = -0.56$ e Å^{-3}
H-atom parameters constrained	Extinction correction: SHELXL97
	Extinction coefficient: 0.059 (3)

Table 1
Selected geometric parameters (Å, °).

Cr1—O4	1.588 (3)	Cr2—O1	1.607 (2)
Cr1—O5	1.615 (2)	Cr2—O2	1.607 (2)
Cr1—F2	1.623 (2)	Cr2—F1	1.630 (2)
Cr1—O3	1.782 (2)	Cr2—O3	1.790 (2)
Cr1—O3—Cr2	117.90 (11)	O2—Cr2—O3	109.72 (11)
F1—Cr2—O3	110.27 (11)	O4—Cr1—O3	108.40 (13)
F2—Cr1—O3	107.51 (10)	O4—Cr1—O5	111.27 (17)
O1—Cr2—O2	110.00 (13)	O4—Cr1—F2	110.57 (13)
O1—Cr2—F1	108.39 (12)	O5—Cr1—F2	110.01 (13)
O1—Cr2—O3	106.73 (11)	O5—Cr1—O3	108.97 (12)
O2—Cr2—F1	111.59 (13)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H5$A\cdots$O5	0.90	1.91	2.779 (4)	162
N2—H5$B\cdots$F2^i	0.90	1.93	2.831 (4)	174
N1—H1$A\cdots$F1^{ii}	0.90	2.04	2.895 (3)	158
N1—H1$A\cdots$O1^{ii}	0.90	2.55	3.219 (4)	132
N1—H1$B\cdots$F1^{iii}	0.90	2.08	2.879 (3)	148
N1—H1$B\cdots$O4^{iv}	0.90	2.37	2.939 (3)	121

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

H atoms were positioned geometrically, with N—H = 0.90 Å and C—H = 0.97 Å, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: X-AREA (Stoe & Cie, 2002); cell refinement: X-AREA; data reduction: X-RED32 (Stoe & Cie, 2002); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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