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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$
R factor = 0.036
wR factor = 0.079
Data-to-parameter ratio = 13.7

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

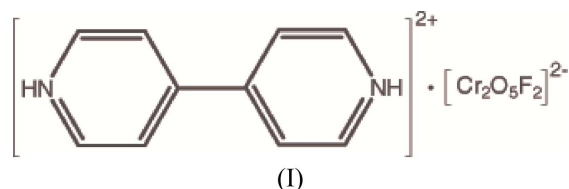
4,4'-Bipyridinium μ -oxo-bis(fluorodioxochromate)

In the cation of the title compound, $(\text{C}_{10}\text{H}_{10}\text{N}_2)[\text{Cr}_2\text{O}_5\text{F}_2]$, a mirror plane passes through the N atoms of the two pyridine rings and the $-\text{C}-\text{C}-$ bridge connecting them, while the two halves of the anion are symmetry-related across a mirror plane passing through the O atom at the centre of the anion. Chiral chains of the title compound are built up *via* $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{F}$ hydrogen bonds.

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Comment

Many structures of chromates and dichromates have been reported, as well as a few cases of trichromates, tetrachromates and polychromates (Pressprich *et al.*, 1988). Chromates may exist as mono-, di-, tri-, tetra- and polymeric forms (Wang *et al.*, 2003; Fouada *et al.*, 1999). It is almost impossible to predict the actual form of these chromium compounds prior to experimental investigation. We report here the structural determination of the title compound (I).



As seen in Fig. 1, the anion and cation of the title compound, (I), have mirror symmetry. In the cation, a mirror plane passes through N1, C3, C4, N2, H11 and H22; in the anion, a mirror plane passes through O3. In the anion of (I), the bridging $\text{Cr}-\text{O}$ bond length [$\text{Cr1}-\text{O3} = 1.7979(16) \text{ \AA}$] is longer than the terminal $\text{Cr}-\text{O}$ bond lengths [$\text{Cr1}-\text{O1} = 1.606(3)$ and $\text{Cr1}-\text{O2} = 1.594(2) \text{ \AA}$]. The average terminal $\text{Cr}-\text{O}$ bond length is $1.600(3) \text{ \AA}$. The $\text{O}-\text{Cr}-\text{O}$ bond angles range from $106.05(15)^\circ$ to $110.59(16)^\circ$. The coordination geometry formed by the three O atoms and F atom around each Cr atom is distorted tetrahedral. The other bond lengths and angles in (I) are within expected ranges, and are similar to those reported in other studies (Jin *et al.*, 2004; Ding *et al.*, 2004; Chaudhuri *et al.*, 1997).

The title compound (I) demonstrates a hydrogen-bonded network of $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{F}$ interactions. There are bifurcated hydrogen bonds between anions and cations (Table 2 and Fig. 2).

Experimental

Chromium (VI) oxide (20 g, 0.2 mol) was dissolved in water (25 ml) in a polythene beaker, and 40% hydrofluoric acid (11.3 ml, 0.23 mol)

was added to it while stirring at room temperature. Within 5 min, a clear solution resulted. To this solution, 4,4'-bipyridine (31.2 g, 0.2 mol) was added slowly with stirring. The compound obtained was left to stand at room temperature for crystallization and, after one day, orange-coloured single crystals suitable of (I) for X-ray diffraction were obtained.

Crystal data

(C₁₀H₁₀N₂)[Cr₂O₅F₂]
M_r = 380.20
 Orthorhombic, *Pnma*
a = 20.883 (3) Å
b = 12.3458 (13) Å
c = 5.0566 (5) Å
V = 1303.7 (3) Å³
Z = 4
D_x = 1.937 Mg m⁻³
 Mo Kα radiation
 μ = 1.71 mm⁻¹
T = 293 (2) K
 Prism, orange
 0.58 × 0.32 × 0.15 mm

Data collection

STOE IPDS 2 diffractometer
 ω scans
 Absorption correction: integration
 (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.524, *T_{max}* = 0.772
 5320 measured reflections
 1495 independent reflections
 819 reflections with *I* > 2σ(*I*)
R_{int} = 0.066
 θ_{max} = 27.1°

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.036
wR (*F*²) = 0.079
S = 0.85
 1495 reflections
 109 parameters
 H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(*F_o*²) + (0.031*P*)²]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} = 0.001
 Δρ_{max} = 0.29 e Å⁻³
 Δρ_{min} = -0.29 e Å⁻³

Table 1 Selected geometric parameters (Å, °).

O1—Cr1	1.606 (3)	O3—Cr1 ⁱ	1.7979 (16)
O2—Cr1	1.594 (2)	O3—Cr1	1.7979 (16)
F1—Cr1	1.610 (2)		
Cr1 ⁱ —O3—Cr1	127.2 (2)	O2—Cr1—O3	109.38 (17)
O2—Cr1—O1	110.59 (16)	O1—Cr1—O3	106.05 (15)
O2—Cr1—F1	109.46 (16)	F1—Cr1—O3	109.98 (17)
O1—Cr1—F1	111.32 (15)		

Symmetry code: (i) *x*, -*y* + ½, *z*.

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

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H11...F1 ⁱⁱ	0.83 (2)	2.380 (14)	2.991 (5)	131.1 (5)
N2—H22...O3 ⁱⁱⁱ	0.83 (2)	1.87 (3)	2.678 (6)	162 (5)
N1—H11...F1 ^{iv}	0.83 (2)	2.380 (14)	2.991 (5)	131.1 (5)

Symmetry codes: (ii) *x* - ½, *y*, -*z* + ½; (iii) *x*, *y*, *z* - 1; (iv) *x* - ½, -*y* + ½, -*z* + ½.

The N-bound H atoms were located in a difference Fourier map and refined freely. The other H atoms were positioned geometrically and refined using a riding model approximation, with C—H = 0.93 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C).

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

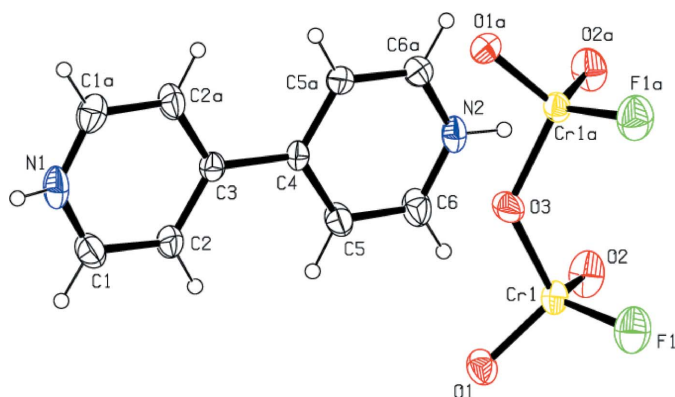


Figure 1 View of structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (a) *x*, ½ - *y*, *z*.]

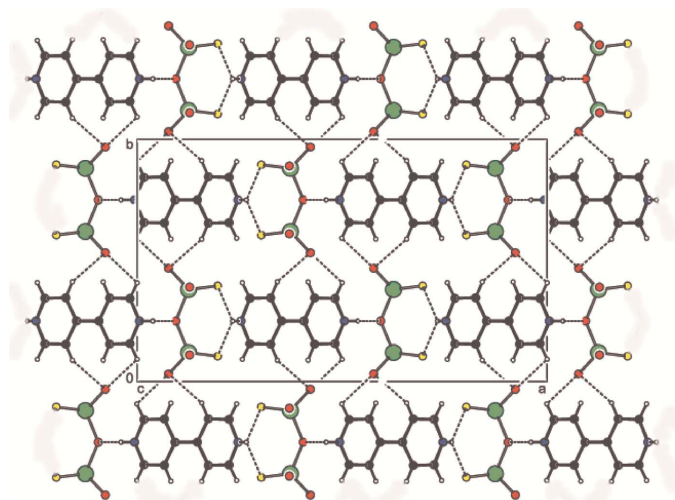


Figure 2 The packing of (I), viewed along the *c* axis. Dashed lines indicate hydrogen bonds.

graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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References

Chaudhuri, M. K., Chettri, S. K., Dey, D., Mandal, G. C., Paul, P. C. & Kharmawphlang, W. (1997). *J. Fluorine Chem.* **81**, 211–213.
 Ding, C.-R., Jin, Z.-M., Wang, H.-B., Hu, M.-L. & Lin, H. (2004). *Acta Cryst. C60*, m203–m204.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Fouada, N. E., Halawyb, S. A., Mohamed, M. A. & Zakid, M. I. (1999). *Thermochim. Acta*, **329**, 23–29.
 Jin, Z.-M., Zhang, H.-M., Wang, H.-B., Hu, M.-L. & Shen, L. (2004). *Acta Cryst. C60*, m572–m574.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

- Pressprich, M. R., Willett, R. D., Poshusta, R. D., Saunders, S. C., Davis, H. B. & Gard, G. L. (1988). *Inorg. Chem.* **27**, 260–264.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
- Wang, Y., Ohishi, Y., Shishido, T., Zhang, Q. H., Yang, W., Guo, Q., Wan, H. & Takehira, K. (2003). *J. Catal.* **220**, 347–357.