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2,2′-Bipyridinium fluorotrioxochromate(VI)

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Key indicators

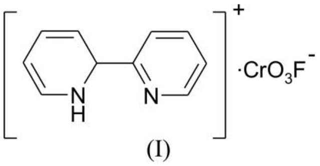
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.086
Data-to-parameter ratio = 6.8

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

A facile synthesis of 2,2′-bipyridinium fluorochromate(VI), $(C_{10}H_9N_2)[CrO_3F]$, was developed by reacting CrO_3 with HF in the presence of 2,2′-bipyridine. The structure of the title compound consists of discrete cations and anions. The coordination around Cr is distorted tetrahedral.

Comment

One of the main thrusts of research in chromium(VI) chemistry is the synthesis of halochromate(VI) compounds suitable for the effective and selective oxidation of organic substrates, particularly alcohols, under mild conditions. Two such species, *viz.* pyridinium chlorochromate (PCC; Corey & Suggs, 1975) and pyridinium fluorochromate (PFC; Bhattacharjee *et al.*, 1987), have emerged as very useful reagents for the oxidation of organic substrates. Many structures of chromates and dichromates have been reported, as well as a few cases of trichromates, tetrachromates and polychromates (Pressprich *et al.*, 1988; Chaudhuri *et al.*, 1997). We report here the synthesis and structure determination of the title compound, (I), and compare its structure with that of other halochromates.



(I)

The molecular structure of (I) is shown in Fig. 1, with the atom-numbering scheme. The structure of (I) consists of 2,2′-bipyridinium cations and fluorochromate(VI) anions. Selected gometric parameters are given in Table 1. The coordination geometry formed by the three O atoms and the F atom around the 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; Chaudhuri *et al.*, 1997; Özyürek *et al.*, 2006). The dihedral angle between the two rings of the cation is 14.6 (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 while stirring at room temperature. Within 5 min, a clear solution resulted. To this solution, 2,2′-bipyridine (31.2 g, 0.2 mol) was added slowly with stirring. The solution was kept at room temperature for crystallization and after 1 d orange-coloured single crystals of (I) suitable for X-ray diffraction were obtained.

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Crystal data

$(C_{10}H_9N_2)[CrFO_3]$
$M_r = 276.19$
Monoclinic, *Pc*
$a = 5.9765$ (5) Å
$b = 13.1069$ (10) Å
$c = 7.2760$ (6) Å
$\beta = 99.829$ (7)°
$V = 561.59$ (8) Å^3
$Z = 2$
$D_x = 1.633$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.03$ mm^{-1}
$T = 293$ (2) K
Prism, orange
0.75 × 0.62 × 0.53 mm

Data collection

Stoe IPDS II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.299$, $T_{max} = 0.478$
6730 measured reflections
1118 independent reflections
1109 reflections with $I > 2\sigma(I)$
$R_{int} = 0.082$
$\theta_{max} = 26.0$°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.086$
$S = 1.06$
1118 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 0.0279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}
Absolute structure: Flack (1983), with 1118 Friedel pairs
Flack parameter: 0.48 (7)

Table 1
Selected geometric parameters (Å, °).

Cr1—F1	1.647 (3)	Cr1—O3*A*	1.568 (11)
Cr1—O1	1.677 (4)	Cr1—O3*B*	1.68 (2)
Cr1—O2	1.623 (4)		
O3*A*—Cr1—O2	110.4 (6)	F1—Cr1—O1	106.2 (2)
O3*A*—Cr1—F1	113.4 (11)	O2—Cr1—O3*B*	103.4 (11)
O2—Cr1—F1	109.5 (2)	F1—Cr1—O3*B*	102.3 (8)
O3*A*—Cr1—O1	109.6 (13)	O1—Cr1—O3*B*	127.1 (17)
O2—Cr1—O1	107.4 (2)		
N1—C5—C6—N2	−14.4 (4)	N1—C5—C6—C7	165.5 (3)
C4—C5—C6—N2	165.1 (3)	C4—C5—C6—C7	−15.0 (5)

The site-occupation factors of the disordered atoms O3*A* and O3*B* refined to 0.62 (7) and 0.38 (7). H atoms were positioned geometrically, with N—H = 0.86 Å and C—H = 0.93 Å, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$. The value of the Flack parameter indicates inversion twinning.

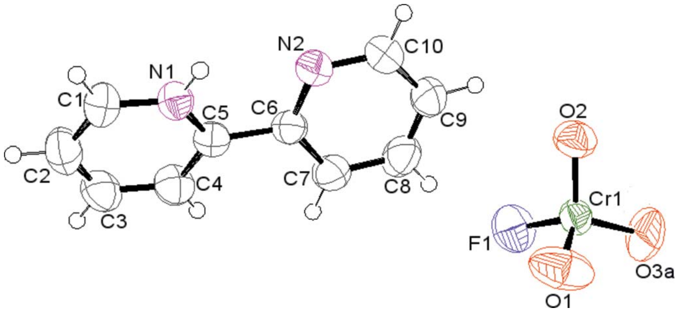

Figure 1
The structure of the ions in (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The minor disorder component has been omitted.

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* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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