Table 2. *Interatomic bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Fe*a*—C(1*a*)	1.765 (3)	Fe*b*—C(1*b*)	1.776 (3)
Fe*a*—C(2*a*)	1.749 (3)	Fe*b*—C(2*b*)	1.777 (3)
Fe*a*—C(3*a*)	1.773 (4)	Fe*b*—C(3*b*)	1.773 (3)
Fe*a*—C(4*a*)	1.764 (3)	Fe*b*—C(4*b*)	1.762 (3)
Fe*a*—C(5*a*)	2.164 (3)	Fe*b*—C(5*b*)	2.188 (3)
C(1*a*)—O(1*a*)	1.146 (4)	C(1*b*)—O(1*b*)	1.156 (4)
C(2*a*)—O(2*a*)	1.167 (4)	C(2*b*)—O(2*b*)	1.158 (4)
C(3*a*)—O(3*a*)	1.147 (5)	C(3*b*)—O(3*b*)	1.159 (4)
C(4*a*)—O(4*a*)	1.143 (4)	C(4*b*)—O(4*b*)	1.151 (4)
C(5*a*)—C(6*a*)	1.542 (5)	C(5*b*)—C(6*b*)	1.507 (4)
C(5*a*)—C(7*a*)	1.466 (4)	C(5*b*)—C(7*b*)	1.469 (5)
C(5*a*)—H(C5*a*)	0.974 (6)	C(5*b*)—H(C5*b*)	0.97 (2)
C(7*a*)—O(5*a*)	1.202 (4)	C(7*b*)—O(5*b*)	1.211 (4)
C(7*a*)—O(6*a*)	1.367 (4)	C(7*b*)—O(6*b*)	1.353 (4)
O(6*a*)—C(8*a*)	1.447 (4)	O(6*b*)—C(8*b*)	1.433 (5)
C(8*a*)—C(9*a*)	1.451 (6)	C(8*b*)—C(9*b*)	1.476 (6)
N(1)—P(1)	1.581 (2)	N(2)—P(3)	1.579 (2)
N(1)—P(2)	1.570 (2)	N(2)—P(4)	1.576 (2)
P(1)—C(1*c*)	1.796 (2)	P(3)—C(1*d*)	1.781 (2)
P(1)—C(7*c*)	1.794 (2)	P(3)—C(7*d*)	1.796 (2)
P(1)—C(13*c*)	1.781 (1)	P(3)—C(13*d*)	1.790 (2)
P(2)—C(19*c*)	1.793 (2)	P(4)—C(19*d*)	1.799 (2)
P(2)—C(25*c*)	1.802 (2)	P(4)—C(25*d*)	1.791 (2)
P(2)—C(31*c*)	1.798 (1)	P(4)—C(31*d*)	1.789 (2)
C(1*a*)—Fe*a*—C(2*a*)	117.5 (2)	C(1*b*)—Fe*b*—C(2*b*)	117.9 (1)
C(1*a*)—Fe*a*—C(3*a*)	114.3 (1)	C(1*b*)—Fe*b*—C(3*b*)	118.5 (1)
C(1*a*)—Fe*a*—C(4*a*)	95.9 (1)	C(1*b*)—Fe*b*—C(4*b*)	89.7 (1)
C(1*a*)—Fe*a*—C(5*a*)	85.5 (1)	C(1*b*)—Fe*b*—C(5*b*)	93.8 (1)
C(2*a*)—Fe*a*—C(3*a*)	127.3 (1)	C(2*b*)—Fe*b*—C(3*b*)	123.3 (1)
C(2*a*)—Fe*a*—C(4*a*)	93.8 (1)	C(2*b*)—Fe*b*—C(4*b*)	93.6 (1)
C(2*a*)—Fe*a*—C(5*a*)	86.5 (1)	C(2*b*)—Fe*b*—C(5*b*)	87.2 (1)
C(3*a*)—Fe*a*—C(4*a*)	89.6 (2)	C(3*b*)—Fe*b*—C(4*b*)	92.0 (1)
C(3*a*)—Fe*a*—C(5*a*)	88.8 (1)	C(3*b*)—Fe*b*—C(5*b*)	83.8 (1)
C(4*a*)—Fe*a*—C(5*a*)	178.2 (1)	C(4*b*)—Fe*b*—C(5*b*)	175.4 (1)
Fe*a*—C(1*a*)—O(1*a*)	178.9 (4)	Fe*b*—C(1*b*)—O(1*b*)	174.1 (2)
Fe*a*—C(2*a*)—O(2*a*)	177.7 (3)	Fe*b*—C(2*b*)—O(2*b*)	177.7 (2)
Fe*a*—C(3*a*)—O(3*a*)	174.7 (3)	Fe*b*—C(3*b*)—O(3*b*)	178.0 (2)
Fe*a*—C(4*a*)—O(4*a*)	178.2 (3)	Fe*b*—C(4*b*)—O(4*b*)	179.4 (3)
Fe*a*—C(5*a*)—C(6*a*)	113.4 (2)	Fe*b*—C(5*b*)—C(6*b*)	113.5 (2)
Fe*a*—C(5*a*)—C(7*a*)	104.3 (2)	Fe*b*—C(5*b*)—C(7*b*)	105.9 (2)
Fe*a*—C(5*a*)—H(C5*a*)	103. (2)	Fe*b*—C(5*b*)—H(C5*b*)	103. (1)
C(6*a*)—C(5*a*)—C(7*a*)	109.7 (3)	C(6*b*)—C(5*b*)—C(7*b*)	112.2 (3)
C(6*a*)—C(5*a*)—H(C5*a*)	112. (2)	C(6*b*)—C(5*b*)—H(C5*b*)	112. (1)
C(7*a*)—C(5*a*)—H(C5*a*)	114. (2)	C(7*b*)—C(5*b*)—H(C5*b*)	109. (2)
C(5*a*)—C(7*a*)—O(5*a*)	129.4 (3)	C(5*b*)—C(7*b*)—O(5*b*)	127.1 (3)
C(5*a*)—C(7*a*)—O(6*a*)	111.1 (3)	C(5*b*)—C(7*b*)—O(6*b*)	112.2 (3)
O(5*a*)—C(7*a*)—O(6*a*)	119.4 (3)	O(5*b*)—C(7*b*)—O(6*b*)	120.7 (3)
C(7*a*)—O(6*a*)—C(8*a*)	116.8 (3)	C(7*b*)—O(6*b*)—C(8*b*)	117.9 (3)

Table 2 (*cont.*)

O(6*a*)—C(8*a*)—C(9*a*)	111.4 (3)	O(6*b*)—C(8*b*)—C(9*b*)	108.5 (3)
P(1)—N(1)—P(2)	139.9 (1)	P(3)—N(2)—P(4)	140.8 (1)
N(1)—P(1)—C(1*c*)	113.03 (9)	N(2)—P(3)—C(1*d*)	107.7 (1)
N(1)—P(1)—C(7*c*)	109.99 (9)	N(2)—P(3)—C(7*d*)	113.4 (1)
N(1)—P(1)—C(13*c*)	109.75 (8)	N(2)—P(3)—C(13*d*)	114.0 (1)
C(1*c*)—P(1)—C(7*c*)	110.16 (7)	C(1*d*)—P(3)—C(7*d*)	106.04 (8)
C(1*c*)—P(1)—C(13*c*)	105.96 (7)	C(1*d*)—P(3)—C(13*d*)	108.26 (9)
C(7*c*)—P(1)—C(13*c*)	107.74 (7)	C(7*d*)—P(3)—C(13*d*)	107.11 (8)
N(1)—P(2)—C(19*c*)	107.45 (9)	N(2)—P(4)—C(19*d*)	112.0 (1)
N(1)—P(2)—C(25*c*)	110.93 (8)	N(2)—P(4)—C(25*d*)	107.2 (1)
N(1)—P(2)—C(31*c*)	115.66 (9)	N(2)—P(4)—C(31*d*)	114.72 (9)
C(19)—P(2)—C(25*c*)	107.65 (8)	C(19*d*)—P(4)—C(25*d*)	107.95 (7)
C(19*c*)—P(2)—C(31*c*)	106.50 (7)	C(19*d*)—P(4)—C(31*d*)	107.67 (8)
C(25*c*)—P(2)—C(31*c*)	108.28 (8)	C(25*d*)—P(4)—C(31*d*)	107.00 (8)

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Structure of Diaquadichlorobis(hydrazinium)iron(II) Dichloride

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Abstract. $[Fe(N_2H_5)_2(H_2O)_2Cl_2].Cl_2$, $M_r = 299.65$, monoclinic, $P2_1/c$, $a = 8.027\ (1)$, $b = 5.725\ (2)$, $c = 11.430\ (2)$ Å, $\beta = 97.08\ (1)°$, $V = 521.3\ (2)$ Å^3, $Z = 2$, $D_m = 1.92$, $D_x = 1.910$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 24.5$ cm^{-1}, $F(000) = 304$, $T = 295$ K, final $R = 0.0242$ and $wR = 0.0292$ for 1411 significant $[F_o > 5.0\sigma(F_o)]$ reflections. The crystal contains discrete Cl^- ions and complex $[Fe(N_2H_5)_2(H_2O)_2Cl_2]^{2+}$ cations. In the complex cation, the Fe atom is bonded to two hydrazinium cations, two Cl atoms and two water molecules. The coordinated atoms are *trans* to each other. The ions are connected by both N—H···Cl and O—H···Cl type hydrogen bonds.

Experimental. The title compound was prepared by refluxing an aqueous solution of $FeCl_3.6H_2O$ and

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$N_2H_4.HCl$ (1/2 mole ratio) for 0.5 h and concentrating the resulting solution in a desiccator. A colourless crystal of size 0.35 × 0.25 × 0.20 mm was chosen. Intensity data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Cell dimensions were determined by least-squares fit of 25 reflections ($20 < 2\theta < 30°$). A total number of 1759 reflections were measured with ω–2θ scan, 1603 reflections were unique and 1411 were observed with $F_o > 5.0\sigma(F_o)$; $R_{\rm int} = 0.009$; $0 \le h \le 11$, $0 \le k \le 8$, $-11 \le l \le 11$; $(\sin\theta)/\lambda \le 0.7035$ Å^{-1}. The data were corrected for Lorentz and polarization but not for absorption. Three standard reflections ($\bar{3}37$, $\bar{4}\bar{1}\bar{4}$, $\bar{1}45$) were measured every 100 reflections. They showed no significant decay. The structure was solved by direct methods (Sheldrick, 1986). H atoms were located in the $\Delta\rho$ map. Full-matrix least-squares refinement of 81 parameters on $|F_o|$ with anisotropic atomic displacements parameters for non-H atoms and isotropic for H atoms converged to $R = 0.0242$, $wR = 0.0292$, $S = 1.29$; $w = 1.0/[\sigma^2(|F_o|) + 0.000346|F_o|^2]$. Final $(\Delta/\sigma)_{\rm max} = 0.001$, $(\Delta\rho)_{\rm max} = 0.36$ and $(\Delta\rho)_{\rm min} = -0.56$ e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were based on F_o using *SHELX*76 (Sheldrick, 1976) and performed on a VAX 11/780 computer.

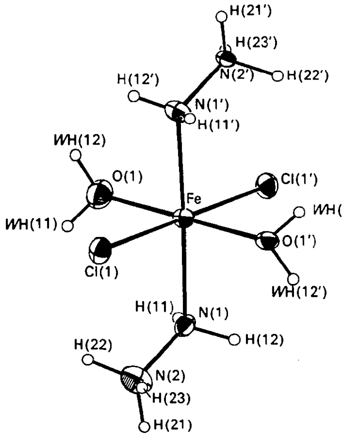

Fig. 1. *ORTEP*II diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 50% probability level. H atoms given arbitrary radii.

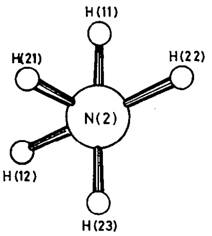

Fig. 2. Configuration of the $N_2H_5^+$ ion viewed down the N—N bond.

Table 1. *Fractional atomic coordinates for the non-*H *atoms and equivalent isotropic temperature factors* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Fe	0.0	0.0	0.0	1.31 (1)
N(1)	0.2098 (2)	−0.1576 (2)	0.1189 (1)	1.60 (3)
N(2)	0.3021 (2)	−0.3565 (2)	0.0831 (1)	2.07 (3)
Cl(1)	−0.00858 (5)	−0.3131 (1)	−0.14494 (3)	1.75 (1)
Cl(2)	0.46301 (5)	−0.3144 (1)	0.35879 (3)	2.11 (1)
O(1)	0.1899 (1)	0.1856 (2)	−0.0837 (1)	1.99 (2)

Table 2. *Bond distances* (Å) *and bond angles* (°)

Fe—N(1)	2.221 (1)	Cl(1)—Fe—O(1)	91.32 (3)
Fe—Cl(1)	2.4360 (7)	N(1)—Fe—O(1)	87.02 (4)
Fe—O(1)	2.174 (1)	N(1)—Fe—Cl(1)	94.18 (4)
N(1)—N(2)	1.445 (2)	Fe—N(1)—N(2)	121.56 (8)

A perspective view of the complex cation showing its conformation and atom-numbering system is presented in Fig. 1. Atomic parameters are given in Table 1* and bond lengths and angles in Table 2. The metal lies on the inversion centre and the coordination geometry in the molecule is an octahedron made up by two N atoms, one from each $N_2H_5^+$ ion, two water O atoms and two Cl atoms. The configuration of the $N_2H_5^+$ ion is staggered, Fig. 2, which is probably the most stable configuration. The ions are linked through O—H···Cl and N—H···Cl type hydrogen bonds. Strong hydrogen bonds are found between —NH_3 of N_2H_5 and Cl^- ions.

Related literature. Shannon & Prewitt (1970), Verbist, Hamilton, Koetzle & Lehmann (1972), Kai, Misawa & Nishimoto (1980), Govindarajan, Patil, Manohar & Werner (1986) and Liminga & Lundgren (1965).

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* Lists of structure factors, anisotropic atomic displacement parameters, H-atom parameters, bond lengths and angles involving H atoms, torsion angles, and intermolecular contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54850 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0163]

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Structure of Potassium 5-Fluoro-5,6-dihydrouracil-6-sulfonate Monohydrate

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Abstract. Potassium 5-fluorohexahydro-2,6-dioxo-4-pyrimidinesulfonate monohydrate, $K^+.C_4H_4FN_2O_5S^-.H_2O$, $M_r = 268.26$, triclinic, $P\bar{1}$, $a = 6.730\ (1)$, $b = 8.366\ (1)$, $c = 8.835\ (1)$ Å, $\alpha = 74.15\ (1)$, $\beta = 74.32\ (1)$, $\gamma = 72.27\ (1)°$, $V = 446.2\ (9)$ Å^3, $Z = 2$, $D_x = 2.00$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0.7107$ Å, $\mu = 8.43$ cm^{-1}, $F(000) = 272$, $T = 293$ K, $R = 0.026$ for 1978 reflections with $I \geq 3\sigma(I)$. The uracil ring shows a distorted 'half-chair' conformation. The SO_3^- group is axially bonded to the ring, the F atom equatorially. The anion is bonded to five symmetry-related K^+ ions, which exhibit severely distorted trigonal prismatic eight coordination. Each anion is involved in six hydrogen-bonding interactions.

Experimental. 5-Fluorouracil, a strong antitumor agent, is known to add covalently the bisulfite ion to yield 5-fluoro-5,6-dihydrouracil-6-sulfonate (I) in a single stereoisomeric form, as shown by ^{1}H NMR spectra (Sander & Deyrup, 1972). By means of ^{1}H NMR analysis of (I) and of its degradation products, Rork & Pitman (1975) demonstrated the *cis* configuration of the F—C(5)—C(6)—S fragment. (I) was prepared according to Rork & Pitman (1975); the colourless crystals were recrystallized from boiling water; the ^{1}H NMR spectrum was consistent with that previously reported. Crystal dimensions 0.35 × 0.25 × 0.25 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, room temperature; 25 ($13 \leq \theta \leq 26°$) reflections for cell-parameter determination; $\omega/2\theta$ scan, scan width $(0.65 + 0.35\tan\theta)°$; two standard reflections ($\bar{3}\bar{5}\bar{2}$ and $\bar{2}\bar{9}0$) measured at two hour intervals showed no significant intensity decay; 2150 unique reflections measured in the θ range 1.5–28°, $-8 \leq h \leq 8$, $-11 \leq k \leq 11$, $0 \leq l \leq 11$, 1978 with $I \geq 3\sigma(I)$ used for structure determination; intensities corrected for Lorentz–polarization effects and for absorption based on empirical ψ scan [$0.949 \leq T$ factor ≤ 0.999]. Structure solved by direct methods (*SHELXS86*; Sheldrick, 1986), and refined by full-matrix least squares with $\sum w\Delta F^2$ being minimized (*SHELX76*; Sheldrick, 1976). All non-H atoms anisotropic, and H atoms, located in ΔF maps, isotropic. $R = 0.026$, $wR = 0.035$, $S = 1.36$, $w = 1.0/(\sigma^2F + 0.00163F^2)$, maximum Δ/σ 0.04, $-0.46 \leq \Delta\rho \leq 0.34$ e Å^{-3}. No corrections for secondary extinction. Scattering factors from *SHELX76*. All calculations were carried out on a VAX 6310 computer. Final atomic coordinates are listed in Table 1, bond lengths and selected bond angles in Table 2.† The anion is depicted in Fig. 1 and the unit cell in Fig. 2.

Related literature. The main interest of this structure determination is the stereochemistry of F and SO_3^- substituents with respect to the uracil ring. Our results confirm the *cis* configuration suggested by Rork & Pitman (1975). The SO_3^- group is bonded to

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† Lists of structure factors, anisotropic thermal parameters for non-H atoms, complete bond distances and angles, selected least-squares planes, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54854 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0289]

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