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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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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$. H atoms refined isotropically

	x	y	z	B_{eq} or B
K	0.16982 (5)	0.15193 (4)	0.91373 (4)	2.21 (1)
S	0.09065 (5)	0.36290 (4)	0.28726 (3)	1.48 (1)
O(1)	0.1962 (2)	0.3387 (1)	0.1250 (1)	2.02 (3)
O(2)	−0.0310 (2)	0.2379 (2)	0.3759 (1)	2.57 (4)
O(3)	−0.0313 (2)	0.5373 (1)	0.2935 (2)	2.82 (4)
N(1)	0.3507 (2)	0.4822 (2)	0.3933 (2)	1.89 (4)
C(2)	0.4787 (2)	0.5587 (2)	0.2677 (2)	1.61 (4)
O(4)	0.4902 (2)	0.7066 (1)	0.2510 (1)	2.34 (4)
N(3)	0.6009 (2)	0.4607 (1)	0.1557 (1)	1.67 (4)
C(4)	0.6223 (2)	0.2897 (2)	0.1722 (2)	1.64 (4)
O(5)	0.7273 (2)	0.2113 (1)	0.0694 (1)	2.40 (4)
C(5)	0.5089 (2)	0.2058 (2)	0.3350 (2)	1.79 (4)
F	0.4794 (2)	0.0538 (1)	0.3217 (1)	2.75 (4)
C(6)	0.3014 (2)	0.3248 (2)	0.3981 (1)	1.56 (4)
O*W*	0.9247 (2)	0.0531 (2)	0.7712 (2)	3.27 (5)
H(N1)	0.270 (3)	0.544 (3)	0.459 (3)	2.3 (4)
H(N3)	0.694 (2)	0.517 (2)	0.069 (3)	3.1 (4)
H(C5)	0.607 (4)	0.165 (3)	0.423 (3)	3.4 (4)
H(C6)	0.254 (3)	0.265 (2)	0.499 (2)	2.2 (3)
H(*W*1)	0.977 (4)	−0.015 (3)	0.704 (3)	4.0 (4)
H(*W*2)	0.832 (5)	0.124 (3)	0.753 (3)	4.0 (4)

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

S—O(1)	1.456 (1)	S—O(2)	1.451 (1)
S—O(3)	1.448 (1)	S—C(6)	1.838 (1)
C(6)—N(1)	1.440 (2)	N(1)—C(2)	1.347 (2)
C(2)—O(4)	1.229 (2)	C(2)—N(3)	1.386 (2)
N(3)—C(4)	1.364 (2)	C(4)—O(5)	1.204 (2)
C(4)—C(5)	1.526 (2)	C(5)—F	1.385 (2)
C(5)—C(6)	1.512 (2)		
K—O$W^{\rm i}$	2.741 (2)	K—O($4^{\rm ii}$)	2.768 (1)
K—O($3^{\rm iii}$)	2.798 (1)	K—O($1^{\rm iv}$)	2.810 (1)
K—O$W^{\rm v}$	2.863 (1)	K—O($5^{\rm vi}$)	2.875 (1)
K—O($5^{\rm vii}$)	2.875 (1)	K—$F^{\rm vi}$	3.113 (1)
O(1)—S—O(2)	113.24 (7)	O(1)—S—O(3)	113.40 (6)
O(1)—S—C(6)	106.93 (6)	O(2)—S—O(3)	112.48 (7)
O(2)—S—C(6)	105.16 (6)	O(3)—S—C(6)	104.72 (8)
S—C(6)—N(1)	111.55 (9)	S—C(6)—C(5)	113.9 (1)
N(1)—C(6)—C(5)	106.5 (1)	C(6)—N(1)—C(2)	123.5 (1)
N(1)—C(2)—N(3)	116.2 (1)	N(1)—C(2)—O(4)	123.1 (1)
O(4)—C(2)—N(3)	120.7 (1)	C(2)—N(3)—C(4)	125.6 (1)
N(3)—C(4)—C(5)	113.9 (1)	N(3)—C(4)—O(5)	122.9 (1)
O(5)—C(4)—C(5)	123.2 (1)	C(4)—C(5)—C(6)	111.96 (9)
C(4)—C(5)—F	108.9 (1)	F—C(5)—C(6)	112.0 (1)

Symmetry code: (i) $x-1, y, z$; (ii) $1-x, 1-y, 1-z$; (iii) $-x, 1-y, 1-z$; (iv) $x, y, z+1$; (v) $1-x, -y, 2-z$; (vi) $1-x, -y, 1-z$; (vii) $x-1, y, z+1$.

the ring in axial position, whereas the F substituent is equatorially bonded, so determining *gauche* orientation with the C(5)—C(6) bond. The S—C(6)—C(5)—F torsion angle is −51.8 (1)°. Bond distances and bond angles within the anion are consistent with those observed in 5,6-dihydrouracil (Rohrer & Sundaralingam, 1970), and in sodium 5,6-dihydrouracil-6-sulfonate monohydrate (Barnes & Hawkinson, 1980). As expected, the six-membered ring is puckered in such a manner that we have assumed a distorted 'half-chair' conformation. The C(6) atom lies 0.605 Å out of the mean plane through the other five non-H ring atoms, whose deviations from the plane are within ±0.080 Å. Furthermore, our six-membered ring is much more puckered than that of the above cited compounds. In our case, the sum of the torsion angles within the ring is 159.3°, larger than that of 139.7° observed in 5,6-dihydrouracil (Rohrer & Sundaralingam, 1970), and that of 108.4° reported for sodium 5,6-dihydrouracil-6-sulfonate monohydrate (Barnes & Hawkinson, 1980). The anion is bonded to five symmetry-related K$^+$ cations through all but one O atom [O(2)], and the F atom. The K$^+$ ion is coordinated by seven O atoms and one F atom in a severely distorted bicapped-trigonal-prismatic geometry. Six of the bonded atoms belong to five neighbouring anions, and two water O atoms complete the coordination sphere. The K$^+$ ion coordination ties anions and water molecules in an infinite three-dimensional network. Further contributions to the crystal packing arise from hydrogen bonding. Each anion is linked to two adjacent units by two centrosymmetrically related N—H···O(sulfonic) interactions. Moreover, the sulfonic O(2) and the carbonyl O(4) atoms are hydrogen bonded to two water molecules.

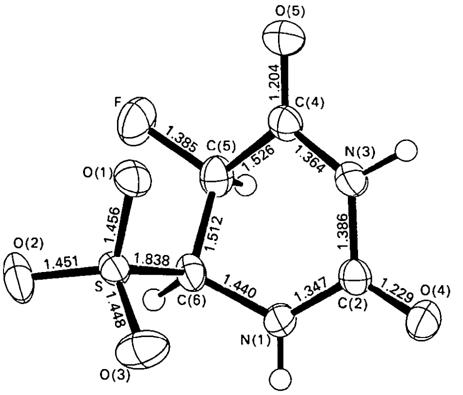

Fig. 1. *ORTEP* (Johnson, 1965) plot of the anion with atom-numbering scheme and bond distances (Å). Thermal ellipsoids for non-H atoms enclose 60% probability.

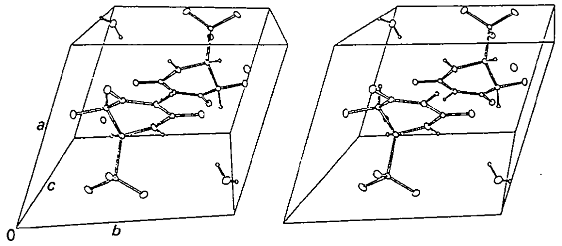

Fig. 2. Stereoview of the unit-cell contents.

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Structure of [*N*-(2-Mercaptophenyl)salicylideneaminato-*N*,*O*,*S*]dimethyltin(IV)

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Abstract. $[Sn(C_{13}H_9NOS)(CH_3)_2]$, $M_r = 376.04$, monoclinic, $P2_1/n$, $a = 9.623\,(2)$, $b = 8.202\,(1)$, $c = 18.757\,(3)$ Å, $\beta = 96.20\,(1)^\circ$, $V = 1471.79\,(7)$ Å^3, $Z = 4$, $D_x = 1.697$ Mg m^{-3}, $D_m = 1.688$ Mg m^{-3} (flotation in chloroform/iodomethane), $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.87$ mm^{-1}, $F(000) = 744$, $T = 298$ K, $R = 0.0456$ for 2505 observed reflections [$I > 2.5\sigma(I)$]. The Sn atom displays a five-coordinate trigonal bipyramidal arrangement in which the tridentate ligand binds to the Sn atom through the O, N and S atoms. The N atom of the ligand and the two methyl groups are located at the equatorial positions. The Sn—O, Sn—N and Sn—S distances are 2.130 (4), 2.176 (4) and 2.554 (1) Å respectively, while the angles O—Sn—N, N—Sn—S and O—Sn—S are 80.6 (1), 77.6 (1) and 158.0 (1)°, respectively.

Experimental. The 2-(2-hydroxyphenyl)benzothiazoline was prepared by a previously reported method (Charles & Freiser, 1953). The complex was obtained from a mixture of dimethyltin(IV) dichloride and the ligand in the presence of sodium methoxide. Preliminary data obtained from Weissenberg and Buerger precession photographs yielded approximate cell dimensions and showed monoclinic symmetry, space group $P2_1/n$. Data collection on a crystal 0.3 × 0.2 × 0.2 mm was performed on a Syntex $P2_1$ diffractometer in $2\theta/\theta$ mode ($0 < h < 12$, $0 < k < 10$, $-20 < l < 20$) out to 2θ(max.) of 52.2°. Variable scan speed of 5.0–29.3° min^{-1} was used. Lattice parameters refined using 30 reflections in the range $35 < 2\theta < 45^\circ$. Standard reflection $31\bar{3}$ checked every 50 reflections: no significant deviation. The data were corrected for Lorentz and polarization effects. 3370 reflections were collected, 2970 unique ($R_{\text{int}} = 0.0243$), of which 2505 observed reflections with $I > 2.5\sigma(I)$ were used for refinement of the structure. Structure solved by direct methods, *SHELXS86* (Sheldrick, 1986), and refined using *SHELX76* (Sheldrick, 1976). Scattering factors for C, H, N, O, S and Sn inlaid in *SHELX76*.

Structure determination and refinement performed on an IBM 4361/4381 computer. Refinement by full-matrix least squares based on F with idealized riding H atoms (C—H 0.96 Å); anisotropic refinement of non-H atoms, isotropic refinement for H atoms (187 scale, positional and thermal parameters of all the atoms refined in the final cycle). In order to correct the observed structure factors for absorption effects, the program *ABSORB* (Ugozzoli, 1987) was

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