towards a more tetrahedral geometry. The distortion present in this salt lies well towards the tetrahedral end of the range of geometries normally observed, and is consistent with the limited extent of hydrogen bonding observed. However, it is not unknown for a CuX_4^{2-} anion to assume two or more geometries in the same structure (Bloomquist & Willett, 1981) and it is known that pressure can cause significant distortion (Willett, Ferraro & Choca, 1974; Wang & Drickamer, 1973). Thus, the observed disorder and distortion when the brominated cation is replaced by the unbrominated one are readily rationalized.

The anions and cations are held together in the crystal lattice with hydrogen bonding between the Br atoms on the anion and the N atoms of the cations. The pyridinium N(1) atom is hydrogen bonded to Br(2) with N(11) hydrogen bonded to Br(1). There is also evidence of hydrogen bonding between the amine groups and the anion. There is a bifurcated hydrogen bond from N(7) to Br(1) and Br(5). N(17) shows hydrogen bonding to Br(1).

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Structure of Dimethylformamide(dioxo)[*N,N'*-(1,3-propanediyl)bis(salicylideneiminato)]uranium(IV)

BY D. MENTZAFOS, A. HOUNTAS, H. A. TAJMIR-RIAHI AND A. TERZIS*

Nuclear Research Center 'Demokritos', Physics Department, 15310 *Aghia Paraskevi, Athens, Greece*

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Abstract. $[UO_2(C_{17}H_{16}N_2O_2)(C_3H_7NO)]$, $M_r = 623{\cdot}45$, orthorhombic, *Pbca*, $a = 12{\cdot}664$ (2), $b = 9{\cdot}563$ (2), $c = 35{\cdot}024$ (6) Å, $V = 4242$ (1) Å^3, $Z = 8$, D_m(by flotation) = 1·94, $D_x = 1{\cdot}952$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 73{\cdot}35$ cm^{-1}, $F(000) = 2368$, $T = 296$ (2) K, $R = 0{\cdot}037$ for 1600 observed reflections with $F_o \geq 6{\cdot}0\sigma(F_o)$. The U atom has a pentagonal bipyramidal coordination and the Schiff base the 'stepped' conformation. U—N distances [2·573 (2), 2·569 (8) Å] are much longer than U—O distances [2·240 (8), 2·253 (8), 2·417 (8) Å]. Bond angles at U are 71·2 (3), 74·0 (3) and 75·5 (3)° for N(1)—U—N(2), O(3)—U—O(5) and O(4)—U—O(5) respectively.

Introduction. Uranyl complexes with Schiff bases have been the subject of many investigations (Casellato, Vigato, Tamburini, Sitran & Graziani, 1984). The greatest attention has been given to polyhedra generated by bidentate or polydentate ligands with oxygen or nitrogen donor atoms (Casellato, Vidali & Vigato, 1976). A coordination number of 7 for the U atom seems to be the most common case (Cattalini, Croato, Degetto & Tondello, 1971). Most complexes exhibit a pentagonal bipyramidal geometry, with the uranyl oxygens at the axial position (Casellato, Vidali & Vigato 1976; Cattalini, Croato, Degetto & Tondello, 1971). In this report we present our results on the structure of such a complex of U with the Schiff base *N,N'*-(1,3-propanediyl)bis(salicylideneimine).

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* To whom all correspondence should be addressed.

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Experimental. Preparation of the compound is described elsewhere (Tajmir-Riahi & Sarkheil, 1981). Crystals were obtained from a dimethylformamide (DMF) solution. Prisms 0·36 × 0·11 × 0·09 mm. Syntex $P2_1$ computer-controlled diffractometer. Cell dimensions from 15 automatically-centred reflections with $18 \leq 2\theta \leq 22°$. Data collected/unique/$R_{\rm int}$, 5926/2616/0·019, $2\theta \leq 44{\cdot}0°$, $-13 \leq h \leq 13$, $0 \leq k \leq 10$, $0 \leq l \leq 36$. Three standard reflections monitored periodically (fluctuation <3%), θ–2θ scan model, scan speed 1·5–10·0° (2θ) min^{-1}, scan range 0·9° below $K\alpha_1$, 0·9° above $K\alpha_2$. Lp and numerical absorption corrections (transmission factors 0·504–0·718) applied. The U-atom position deduced from a Patterson map, the other non-H atoms by ΔF calculations. Refinement by blocked-matrix least squares minimizing $\sum w\Delta^2$, *SHELX*76 (Sheldrick, 1976). H (calculated) isotropic riding on carbons at 0·98 Å. Final refinement for 1600 observed reflections $[F_o \geq 6\sigma(F_o)]$: $R = 0{\cdot}037$, $wR = 0{\cdot}038$, $w = 2{\cdot}6245[\sigma^2(F_o) + 0{\cdot}0017 F_o^2]^{-1}$. Ten reflections showing strong extinction effects were given zero weights during final refinement cycles. 289 parameters refined. $\Delta/\sigma = 0{\cdot}105$. $\Delta\rho_{\rm max} = 0{\cdot}93$, $\Delta\rho_{\rm min} = -0{\cdot}71$ e Å^{-3}, in the vicinity of the U atom. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* bond lengths and angles in Fig. 1 and an *ORTEP* (Johnson, 1976) view in Fig. 2.

Discussion. The U atom has the pentagonal bipyramidal coordination which seems to be the preferred geometry for seven-coordinated uranyl compounds (Lintredt, Heed, Ahmad & Glick, 1982). The equatorial pentagon, determined by the O and N atoms of the ligand and the O atom of the DMF, is slightly puckered [maximum

Table 1. *Positional* (× 10^4) *and equivalent isotropic thermal* (× 10^3) *parameters for the non-hydrogen atoms*

$$U_{\rm eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	$U_{\rm eq}$(Å^2)
U	136 (1)	956 (1)	1240 (1)	53·8 (3)
O(1)	321 (6)	−803 (9)	1395 (3)	73 (3)
O(2)	62 (7)	2678 (9)	1067 (3)	82 (3)
O(3)	337 (6)	1750 (10)	1838 (2)	73 (3)
O(4)	−928 (6)	110 (10)	779 (3)	72 (4)
O(5)	−1626 (6)	1200 (10)	1497 (3)	91 (4)
N	−3367 (7)	1230 (10)	1557 (3)	69 (4)
N(1)	2108 (6)	1230 (10)	1394 (2)	49 (3)
N(2)	1212 (7)	440 (10)	633 (3)	52 (3)
C(1)	890 (9)	2750 (10)	2001 (3)	59 (4)
C(2)	420 (10)	3640 (20)	2265 (4)	83 (4)
C(3)	1050 (10)	4650 (20)	2452 (4)	83 (4)
C(4)	2070 (10)	4720 (20)	2382 (4)	85 (4)
C(5)	2580 (10)	3860 (10)	2130 (4)	79 (4)
C(6)	1970 (10)	2900 (10)	1923 (3)	58 (4)
C(7)	2510 (10)	1940 (10)	1656 (3)	55 (4)
C(8)	2807 (8)	250 (10)	1187 (3)	59 (4)
C(9)	3118 (9)	850 (10)	805 (3)	72 (4)
C(10)	2136 (8)	1310 (10)	561 (3)	73 (4)
C(11)	950 (10)	−450 (10)	367 (3)	63 (4)
C(12)	50 (10)	−1360 (10)	369 (3)	66 (4)
C(13)	70 (10)	−2550 (10)	135 (4)	86 (4)
C(14)	790 (10)	−3410 (20)	128 (4)	95 (4)
C(15)	−1650 (10)	−3160 (20)	342 (4)	95 (4)
C(16)	−1690 (10)	−1990 (20)	568 (4)	83 (4)
C(17)	−868 (9)	−1040 (10)	580 (3)	61 (4)
C(18)	−2490 (10)	830 (20)	1419 (4)	91 (4)
C(19)	−4340 (10)	760 (20)	1399 (4)	96 (4)
C(20)	−3410 (10)	2220 (20)	1858 (4)	99 (4)

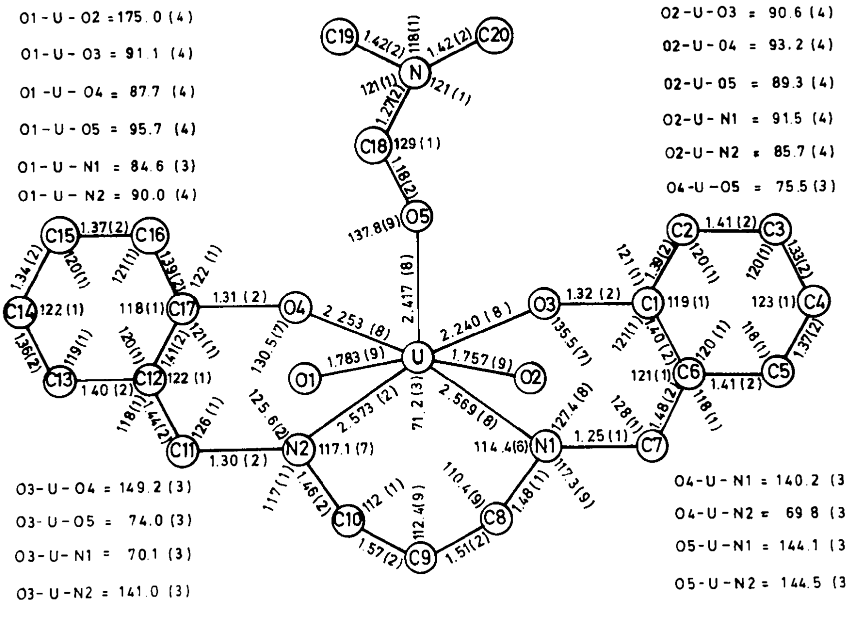

Fig. 1. Interatomic bond distances and angles with e.s.d.'s in parentheses.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43894 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

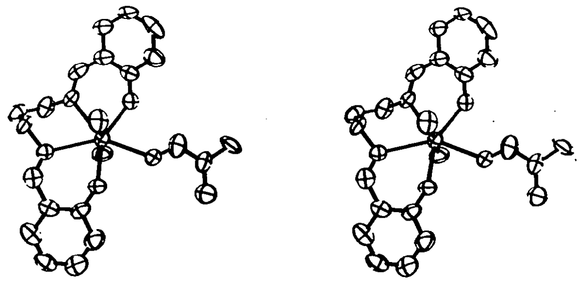

Fig. 2. An *ORTEP* (Johnson, 1976) drawing of the molecule.

deviation from the least-squares plane 0·141 (3) Å for O(4)]. The uranyl group is not considered linear, the U=O bonds deviating 5° from 180°, greater than that observed elsewhere (Lintredt *et al.* 1982; Bandoli, Clemente, Croato, Vidali & Vigato, 1973; Graziani, Casellato, Vigato, Rajan & Chakravorty, 1981). The U—N distances are found to be much longer than the U—O, more so than would be suggested by the difference in covalent radii between O and N. This is testimony to the 'hardness' (Pearson, 1963) of UO_2^{2+} which would be expected to be bonded less strongly to the 'softer' nitrogen. The U—O distance to the monodentate ligand (DMF) is the same as in the case of water (Bandoli, Cattalani, Clemento, Vidali & Vigato, 1972), also a polar ligand, and slightly shorter than in the case of methanol (Bandoli *et al.*, 1973) and ethanol (Bandoli, Clemente, Croato, Vidali & Vigato, 1971).

The propane chain that links the N(1) and N(2) imine atoms causes an increase of the N(1)—U—N(2) angle from a value of 62·4 (Bandoli *et al.*, 1972) or 66·4 (Lintredt *et al.*, 1982) to 71·2° and a decrease of the O(3)—U—O(5) and O(4)—U—O(5) angles from a mean value of 78·6 (Bandoli *et al.*, 1972, 1973) to 74·7°.

The C(9) atom of the propane group has a deviation of 0·15 (5) Å from the equatorial pentagon and defines with U and O(5) a twofold pseudosymmetry axis, so the molecule has the 'stepped' conformation (Casellato *et al.*, 1976; Bandoli *et al.*, 1972).

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Structure of a Seven-Coordinated Tin(IV)–Hydrazone Complex

By Daniele Delledonne and Giancarlo Pelizzi*

Istituto di Chimica Generale, Università di Parma, Centro di Studio per la Strutturistica Diffrattometrica del CNR, Viale delle Scienze, 43100 *Parma, Italy*

and Corrado Pelizzi

Istituto di Chimica Biologica, Facoltà di Farmacia, Università di Sassari, Via Muroni 23/*a*, 07100 *Sassari, Italy*

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Abstract. Dichloro[2,6-diacetylpyridine bis(picolinoylhydrazonato)]tin(IV) monohydrate, $[Sn(C_{21}H_{17}N_7O_2)Cl_2].H_2O$, $M_r = 607{\cdot}02$, monoclinic, $P2_1/c$, $a = 8{\cdot}485\ (5)$, $b = 14{\cdot}476\ (6)$, $c = 19{\cdot}533\ (12)$ Å, $\beta = 95{\cdot}67\ (8)°$, $V = 2387\ (2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}689$ g cm^{-3}, D_m not measured, $\lambda(\mathrm{Cu}\ K\alpha) =$

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* To whom correspondence should be addressed.

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