and (III) undoubtedly make large contributions, and species with charges on N(2) or N(3) must also be involved.

The intermolecular hydrogen bond N(4)—H(4)··· N(1) is typical of a linear hydrogen bond, and the hydrogen bonding involving the water molecule is typical of distorted tetrahedral hydrogen bonding of water, as discussed in detail by Hamilton & Ibers (1968).

We thank Dr Charles M. Dougherty of Herbert H. Lehman College of the City University of New York for supplying the crystals used.

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X-ray Refinement of the Structure of [N,N'-(3-Aza-1,5-pentanediyl)-bis(salicylideneiminato)]dioxouranium(VI)

BY F. BENETOLLO AND G. BOMBIERI*

Laboratorio di Chimica e Tecnologia dei Radioelementi del CNR, Corso Stati Uniti, Padova, Italy

AND A. J. SMITH

Department of Chemistry, The University, Sheffield S3 7HF, England

Abstract. $C_{18}H_{19}N_3O_4U$, $[U(C_{18}H_{19}N_3O_2)O_2]$, ortho-rhombic, Pnma, a = 10·473 (3), b = 21·803 (10), c = 8·024 (2) Å, V = 1832·2 Å^3, Z = 4, D_c = 2·10 Mg m^{-3}, μ(Mo Kα) = 8·48 mm^{-1}, λ(Mo Kα) = 0·7107 Å. The structure has been refined by full-matrix least-squares methods to an R of 0·036 for 1272 observed reflections. Our results confirm those of an earlier structure determination but give interatomic distances and angles with greater accuracy. Anisotropic temperature factors are introduced for all non-hydrogen atoms.

Introduction. The structure of the title compound has previously been determined by Akhtar & Smith (1973) from visually measured film data, and showed the interesting peculiarity of having asymmetric U—O bonds in the uranyl group. The two bond distances were U—O(1) 1·72 (2) Å and U—O(2) 1·64 (3) Å, differing by 2σ from their mean of 1·68 Å. As this difference is at the limit of significance, we were interested in obtaining more accurate bond distances

and angles; also, we have studied uranyl complexes with a very similar ligand, i.e. [N,N'-(3-oxa-1,5-pentanediyl)bis(salicylideneiminato)]dioxouranium(VI), which showed another peculiarity: a non-lonear uranyl group (Brock, Cook, Fenton, Bombieri, Forsellini & Benetollo, 1978).

A single crystal of prismatic habit was used for data collection. Preliminary Weissenberg and precession photographs showed systematic absences indicating space groups $Pn2_1a$ or Pnma; Pnma was confirmed by the subsequent successful refinement, in agreement with the study of Akhtar & Smith (1973).

Intensities of 3216 reflections with k, l ≥ 0 were collected in the range 3 ≤ θ ≤ 25° with graphite-mono-chromated Mo Kα radiation and the θ/2θ scan mode on a Philips PW 1100 four-circle diffractometer. Two standard reflections registered each hour did not show any change in intensity during data collection.

The intensities of the two sets of independent reflections were corrected for absorption following the North, Phillips & Mathews (1968) method and then averaged to obtain a unique set of reflections. Of these, 1272 were considered observed according to the

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* Author to whom correspondence should be addressed.

criterion $I \geq 2 \cdot 5\sigma(I)$ and used in the subsequent refinement.

The atomic coordinates of the previous paper were refined, initially excluding the H-atom positions. Starting from an R value of $0 \cdot 20$, in the three cycles with $w = 1$ and isotropic parameters convergence was reached at $R = 0 \cdot 06$. The function minimized was $\sum w||F_o| - |F_c||^2$. A Fourier difference map calculated at this stage revealed the positions of some H atoms; these were introduced, and their positional parameters refined; those missing were introduced at calculated positions with $C-H = 0 \cdot 95$ Å (see Table 1); a value of $U = 0 \cdot 06$ Å^2 was attributed to all H atoms. Two further cycles with anisotropic thermal parameters for non-hydrogen atoms lowered the R value to $0 \cdot 036$, which was considered as final. All computations were performed with the *SHELX* 76 (Sheldrick, 1976) system.*

X-ray scattering factors were taken from *International Tables for X-ray Crystallography* (1974); f' and f'' for U and μ/ρ were from Cromer & Liberman (1970).

The atomic parameters are given in Table 1. Interatomic distances and bond angles are reported in Table 2.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34734 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates* $(\times 10^4)$ *and isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

	x	y	z	B
U	333 (1)	2500	659 (1)	2·98 (2)
O(1)	4 (11)	2500	2861 (15)	4·3 (6)
O(2)	549 (11)	2500	−1493 (13)	4·2 (6)
O(3)	1846 (7)	1797 (4)	1077 (10)	4·4 (4)
N(1)	−2110 (12)	2500	167 (17)	3·4 (6)
N(2)	−598 (8)	1417 (5)	165 (11)	3·6 (4)
C(1)	−2533 (11)	1939 (6)	−671 (18)	4·2 (6)
C(2)	−2006 (11)	1382 (7)	156 (19)	4·9 (7)
C(3)	11 (11)	958 (6)	−367 (14)	3·6 (6)
C(4)	1401 (11)	882 (5)	−433 (13)	3·5 (5)
C(5)	1886 (14)	371 (6)	−1224 (17)	4·6 (7)
C(6)	3179 (13)	244 (6)	−1326 (16)	4·6 (6)
C(7)	3988 (13)	663 (6)	−594 (20)	5·2 (7)
C(8)	3589 (11)	1184 (6)	190 (16)	4·2 (6)
C(9)	2267 (11)	1316 (6)	281 (14)	3·6 (5)
H(1)	−2400	2500	1533	
H(2)	−2301	1941	−1829	
*H(3)	−3437	1959	−596	
H(4)	−2372	1007	−534	
*H(5)	−2315	1353	1267	
H(6)	−398	595	−737	
H(7)	1368	64	−1628	
*H(8)	3493	−111	−1880	
H(9)	4842	594	−726	
H(10)	4250	1553	819	

* Introduced at calculated positions.

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

Primed atoms are at $x, \frac{1}{2} - y, z$.

U—O(1)	1·80 (1)	N(2)—C(3)	1·26 (2)
U—O(2)	1·74 (1)	C(3)—C(4)	1·47 (2)
U—O(3)	2·23 (1)	C(4)—C(5)	1·38 (2)
U—N(1)	2·59 (1)	C(4)—C(9)	1·43 (2)
U—N(2)	2·59 (1)	C(5)—C(6)	1·38 (2)
N(1)—C(1)	1·46 (2)	C(6)—C(7)	1·38 (2)
C(1)—C(2)	1·49 (2)	C(7)—C(8)	1·36 (2)
C(2)—N(2)	1·48 (1)	C(8)—C(9)	1·42 (2)
		C(9)—O(3)	1·30 (2)
N(1)—U—N(2)	66·7 (2)	C(4)—C(9)—O(3)	121 (1)
N(2)—U—O(3)	70·3 (3)	C(9)—O(3)—U	136 (1)
O(3)—U—O(3')	86·8 (3)	O(3)—C(9)—C(8)	121 (1)
C(1)—N(1)—C(1')	113 (1)	C(3)—C(4)—C(5)	118 (1)
U—N(1)—C(1)	112 (1)	C(9)—C(4)—C(5)	119 (1)
N(1)—C(1)—C(2)	111 (1)	C(4)—C(5)—C(6)	123 (1)
C(1)—C(2)—N(2)	109 (1)	C(5)—C(6)—C(7)	116 (1)
C(2)—N(2)—U	115 (1)	C(6)—C(7)—C(8)	124 (1)
C(2)—N(2)—C(3)	118 (1)	C(7)—C(8)—C(9)	120 (1)
U—N(2)—C(3)	126 (1)	C(8)—C(9)—C(4)	118 (1)
N(2)—C(3)—C(4)	127 (1)	O(1)—U—O(2)	176·4 (5)
C(3)—C(4)—C(9)	123 (1)		

Discussion. The atomic coordinates of the atoms are close to those previously determined. In Fig. 1 is shown a projection of the molecule along [001]. The ligand is pentadentate in the equatorial plane of the uranyl ion. The five coordinated atoms form a rather puckered pentagon. The uranyl U—O bond lengths differ significantly by 6σ, thus unequivocally confirming the asymmetric U—O(uranyl) bond lengths; the U atom is displaced from the mean plane of the pentagon by 0·05 Å towards the direction of the shorter U—O(2) bond. In addition, O(1), unlike O(2), lies between H(1) [attached to N(1)] of the same molecule and H(1)'' belonging to the molecule at $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} - z$ at distances, respectively, of 2·73 and 2·76 Å [H(1)$\cdots$O(1)$\cdots$H(1)'' 167°]. These contacts could also be responsible for the asymmetry in the U—O(uranyl) bond lengths. The uranyl group is also slightly bent [O(1)—U—O(2) 176·4 (5)°] in the direction of the N—H group [O(1)—U—N(1) 87·7 (5)° and O(2)—U—N(1) 88·7 (5)°]. Bent uranyl groups are present in the α and β isomers [174·2 (6) and 173·8 (5)°] of [N,N'-(3-oxa-1,5-pentanediyl)bis(salicylideneiminato)]dioxouranium(VI) (UO$_2$saloden) (Brock *et al.*, 1978): steric reasons seem to explain this irregularity; in particular, the short contacts between the O atoms of the uranyl groups and the equatorially coordinated atoms (minima in the two cases are 2·78 and 2·80 Å). In the present case the shortest contact is between O(1) and O(3) [2·85 (1) Å] and, consequently, the bending is smaller. There seems to exist a correlation between the intramolecular contacts of the uranyl O, the atoms coordinated in the equatorial plane, and the bending of the uranyl group in
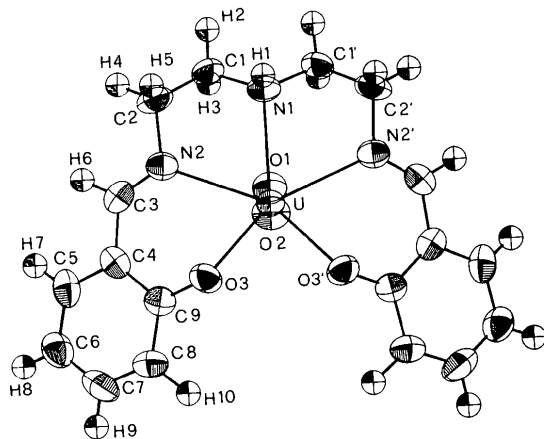
Fig. 1. An *ORTEP* (Johnson, 1965) projection of the molecule along [001].

this class of compounds. In fact, in (UO$_2$saloden).CH-Cl$_3$ (Bombieri, Forsellini, Benetollo & Fenton, 1979) the O(1)—U—O(2) angle is 177·3 (6)° and the shortest

contact is 2·88 Å: the bending diminishes as the contacts lengthen.

In the overall geometry of the molecule, there are no substantial differences from the previous determination.

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Structure of 1,20-Bis(8-quinolyloxy)-3,6,9,12,15,18-hexaoxaicosane–Rubidium Iodide*

By G. WEBER AND W. SAENGER†

Abteilung Chemie, Max-Planck-Institut für Experimentelle Medizin, Hermann-Rein-Strasse 3, D-3400 Göttingen, Federal Republic of Germany

Abstract. C$_{32}$H$_{40}$N$_2$O$_8$.RbI, monoclinic, *C2/c*, *a* = 42·169 (13), *b* = 10·804 (7), *c* = 15·946 (8) Å, *β* = 100·42 (8)°, *M$_r$* = 793·06, *Z* = 8, *d$_c$* = 1·474, *d$_o$* = 1·465 Mg m^{-3} (flotation in bromobenzene/ethyl acetate), *μ* = 2·388 mm^{-1}, *R$_w$* = 0·074 for 4668 unique data. The ligand displays a distorted helical conformation with one and a half turns which provides the cation with tenfold coordination. Eight coordination distances are slightly longer and two are considerably longer than expected for Rb$^+$···O,N. There is no interaction between the shielded cation and the twofold-disordered anion.

Introduction. The title compound was recrystallized from a mixture of methanol and ethyl acetate (Weber, Saenger, Vögtle & Sieger, 1979). Data were collected from a crystal 0·5 × 0·5 × 0·5 mm with a four-circle diffractometer, Mo *K*α radiation, a graphite monochromator and the *θ–2θ* step-scan mode up to sin *θ* = 0·3817. They were corrected for polarization effects.

The structure was solved by direct methods and subsequent Fourier syntheses (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refined by full-matrix least squares (Sheldrick, 1976), omitting four reflexions because of obvious strong secondary extinction. The weighting scheme was based on counting statistics (Stout & Jensen, 1968). H-atom positions were located from difference Fourier syntheses and recalculated according to stereochemical criteria. H atoms were

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