reflected in enlarged N—P—C angles (average 111·5°) for axial phenyl groups, compared with an average N—P—C angle of 106·1° for equatorial phenyl groups. A similar effect occurs in one of the chelate rings of the Fe complex.

The S—Cu—S angles within the three chelate rings [average 123·6 (2)°] are significantly larger than interligand S—Cu—S angles (average 118·5°). This feature is also evident, although to a lesser extent, in the Mn complex, where the average intrachelate S—Mn—S angle is 111·9° and the average interligand S—Mn—S angle is 108·3°; this is in contrast to the situation in the Ni and Fe complexes where distortions from tetrahedral coordination are mostly small and random.

Packing of the crystal structure is determined largely by van der Waals forces; there are no particularly short contacts. The cation charge is effectively buried inside the shell of phenyl groups, and the closest contacts of the $CuCl_2^-$ group are all to phenyl-group H atoms.

OS acknowledges support by the National Institutes of Health through USPHS Grant GM-23072.

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Acta Cryst. (1978). B**34**, 2632–2634

Bis(hydroxylamido)(1,2-ethanediol-*O*,*O*)dioxouranium(VI)

BY H. W. W. ADRIAN AND A. VAN TETS

*Atomic Energy Board, Private Bag X*256, *Pretoria* 0001, *Republic of South Africa*

(*Received* 10 *March* 1978; *accepted* 13 *April* 1978)

Abstract. $UO_2(NH_2O)_2.HOCH_2CH_2OH$, monoclinic, $C2/c$, $a = 9{\cdot}3891$ (9), $b = 8{\cdot}7671$ (8), $c = 9{\cdot}8766$ (8) Å, $\beta = 104{\cdot}400$ (8)°, $V = 787{\cdot}8$ (1) Å^3, $Z = 4$, $D_c = 3{\cdot}42$ Mg m^{-3}. The structure was solved by the heavy-atom method and refined to $R_w = 0{\cdot}052$ for 500 unique diffractometer data. All three ligands are coordinated to the U atom through bidentate bonds.

Introduction. Remarkably stable compounds with structural formulae $UO_2(NH_2O)_2.xL$ or $UO_2(NH_2O)_2.xL_1.yL_2$ can be formed, where L_1 and L_2 are neutral ligands (*e.g.* H_2O or NH_2OH). It has been found that in all such structures six coordinating O or N bonds are formed to the U atom, including two from the neutral ligands (Van Tets & Adrian, 1977; Adrian & Van Tets, 1977, 1978*a*,*b*). The new compound described here is no exception. It was prepared from a solution containing 3·5 g $NH_2OH.HCl$, 50 cm^3 HOC_2H_4OH and 3 cm^3 0·5 *M* aqueous UO_2Cl_2 by slow absorption of neutralizing ammonia vapour from a dilute aqueous solution of ammonium carbonate.

Accurate *d* values were obtained from a powder diffractogram taken on a Huber Guinier camera (114·6 mm diameter) with an internal Si standard and quartz-monochromated Cu $K\alpha_1$ radiation. By the use of an analytical method (Azaroff & Buerger, 1958), together with an automatic powder-indexing routine (Visser, 1969), a monoclinic cell was found from the first forty lines. The whole powder pattern was indexed using this cell. A least-squares refinement on 53 non-overlapping reflections gave the final cell constants. The extinctions indicate space group Cc or $C2/c$. Z must be 4 by analogy with the volumes occupied by similar molecules (Adrian & Van Tets, 1977, 1978*a*,*b*). $C2/c$ with its eight general positions is the more likely space group. No extinctions due to special positions were observed. The U atom was therefore assumed to occupy the special position $0,y,\frac{1}{4}$. The y coordinate was determined by calculating a Patterson map from 45 Lp-corrected powder intensities (Hellner, 1954) and from a strong peak in the Harker section (line $0,v,\frac{1}{2}$) y was found to be 0·17. Refinement of the U atom using these reflections resulted in $R = 0{\cdot}128$ with $y = 0{\cdot}166$ (2).

When material of larger crystalline composition became available, a small irregularly-shaped specimen (about 0·05 × 0·1 × 0·2 mm) was mounted on a Philips PW 1100 automatic diffractometer, and 590 reflections were measured to a maximum $\sin\theta/\lambda$ of 0·55 Å^{-1} with graphite-monochromated Mo $K\alpha_1$ radiation; 500 reflections were significant [$I > 3\sigma(I)$]. Absorption (μ_l = 2·02 m^{-1}) was disregarded, but Lp corrections were applied and a dispersion correction for U was included (Cromer & Liberman, 1970).

A structure factor calculation with U at 0,0·166,$\frac{1}{4}$ gave R = 0·162 and a difference Fourier map revealed all the non-H atoms. The O, N and C atoms were recognized by their typical chemical and coordinating bond distances. The temperature factor of the U atom was refined anisotropically, and those of all other atoms isotropically. With 26 parameters, final values of R = 0·042 and R_w = 0·052 were reached in the full-matrix refinement [$w = 1/\sigma(I)$]. The final difference Fourier map showed no significant peaks and no H atoms were detected.

Atomic parameters are given in Table 1.* The program system XRAY 72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) was used for the calculations, the drawing being produced by *ORTEP* (Johnson, 1965).

Discussion. Interatomic distances and angles are given in Table 2; Fig. 1 is a view of the molecule showing the atomic nomenclature.

The chelating bonds of the hydroxylamido ligands are essentially similar to those described previously (Van Tets & Adrian, 1977; Adrian & Van Tets, 1977, 1978*a*,*b*).

A rotation axis lies in the equatorial plane of the uranyl group and bisects the N—U—N angle and also the C—C bond of the glycol ligand. All the atoms coordinated to U lie essentially in the equatorial plane. The C atoms are 0·31 Å from this plane.

The neutral ligand forms bidentate O bonds to the U atom, and no clear signs of angular distortion in the chelate ring were observed. The C—O and C—C distances are normal.

Table 1. *Atomic parameters* (×10^4, U_{ij} ×10^3)

	x	y	z	U (Å^2)
U	0	1695 (1)	2500	Anisotropic*
O(1)	9068 (13)	1696 (15)	3899 (12)	25 (3)
O(2)	7825 (14)	1066 (15)	933 (14)	30 (3)
O(3)	6323 (13)	933 (14)	8384 (12)	22 (3)
N	8444 (17)	366 (18)	6402 (16)	27 (4)
C	4491 (20)	504 (22)	1780 (19)	26 (4)

* U_{11} = 137 (5), U_{22} = 258 (6), U_{33} = 124 (5), U_{13} = 18 (3).

Table 2. *Interatomic distances* (Å) *and angles* (°)

U—O(1)	1·81 (1)	U···O(3)	2·47 (1)
U···O(2)	2·30 (1)	O(3)—C	1·46 (2)
U···N	2·41 (2)	C—C	1·50 (2)
O(2)—N	1·41 (2)	U···C	3·42 (3)
O(1)—U—O(2)	89·9 (5)	N—U—N	82·7 (5)
O(1)—U—N	90·6 (6)	O(3)—U—O(2)	71·3 (4)
O(1)—U—O(3)	88·5 (5)	O(3)—U—O(3)	65·2 (4)
O(2)—U—N	34·8 (5)	U—O(3)—C	118·9 (9)
U—O(2)—N	76·6 (8)	O(3)—C—C	107 (1)
U—N—O(2)	68·6 (8)		

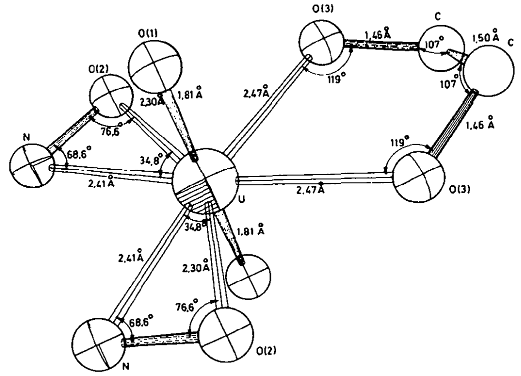

Fig. 1. A view of the molecule with the atomic nomenclature.

No intramolecular hydrogen bonding was found in similar compounds (Van Tets & Adrian, 1977; Adrian & Van Tets, 1977, 1978*a*,*b*). There is no reason to expect it here.

Strong hydrogen bonding involving OH groups is evident from IR spectra and an intermolecular O(3)···O(2) distance of 2·78 (2) Å.

The length of the uranyl group also indicates hydrogen bonding to its O atoms. This is confirmed by IR spectra which show a characteristic shift of the $\nu_{as}(UO_2)$ stretch frequency. The indications are that this distortion is caused by an O(1)···H—N bridge [intermolecular O···N distance 2·92 (2) Å] because the H atom of O(3) is already assigned to its nearest intermolecular neighbour, and the closest intermolecular O(1)···O(3) distance is 3·40 (2) Å. A remaining N—H···O(3) bridge [intermolecular N···O distance 3·15 (3) Å] may be formed, so that all H atoms, except those of the nonpolar CH_2 groups, are involved in hydrogen bonding.

We thank Dr Gafner of the CSIR for making the X-ray diffractometer available, and the Atomic Energy Board for permission to publish this paper.

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33549 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1978). B**34**, 2634–2636

1,3,5,7-Tetraselenocane

BY G. VALLE, G. ZANOTTI AND M. MAMMI

Centro Studi sui Biopolimeri, Università di Padova, Via Marzolo 1, 35100 *Padova, Italy*

(*Received* 22 *December* 1977; *accepted* 31 *March* 1978)

Abstract. $(CH_2Se)_4$, orthorhombic, $Pna2_1$, a = 10·713 (8), b = 6·904 (4), c = 11·662 (3) Å, Z = 4, D_c = 2·86 g cm^{-3}, V = 862·55 Å^3, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 182·0 cm^{-1}. The structure, solved by Patterson–Fourier methods, was refined to R = 0·041. The molecule assumes a twist-chair conformation.

Introduction. The compound was prepared by Russo, Mortillaro, Credali & De Checchi (1966) and the crystals were grown from light petroleum.

The intensities were collected from a crystal of approximate dimensions 0·1 × 0·25 × 0·4 mm, on a Philips PW 1100 four-circle diffractometer operating in the $\theta/2\theta$ scan mode (scan width = 1·2°, scan speed = 0·04° s^{-1}) with Mo $K\alpha$ radiation monochromatized by a graphite crystal. 795 independent reflections up to θ = 25° were measured, of which 604 had $I \geq 3\sigma(I)$, $\sigma(I)$ being calculated from counting statistics. During the data collection two standard reflections were measured every 90 min to check the stability of the crystal and electronics. Intensities were corrected for Lorentz and polarization factors; an experimental absorption correction was applied following the method suggested by North, Phillips & Mathews (1968).

Approximate positions of the four Se atoms were deduced from two-dimensional Patterson maps. Structure factor calculations based on refined positions of these heavy atoms yielded a conventional R factor of 0·12. A three-dimensional difference electron density map showed the positions of all C atoms, which were included in the subsequent cycles of full-matrix least-squares refinement. The coordinates of the H atoms were calculated and included in the last cycle of refinement, but not varied. The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $1/w = 0{\cdot}55 \times \{1 + [(F_o - 43)/15]^2\}$. The correction for the real and imaginary parts of the anomalous dispersion was applied for Se only. The final R and R_w values for all observed reflections were 0·041 and 0·052 respectively.

The scattering factors were obtained from *International Tables for X-ray Crystallography* (1968). All the calculations were carried out on the Cyber 76 computer of the 'Centro di Calcolo Interuniversitario dell'Italia Nord Orientale' with the XRAY system of crystallographic programs (Stewart, Kundell & Baldwin, 1970).

Discussion. Polyselenomethylene can be obtained in two crystalline forms, hexagonal and orthorhombic, by cationic polymerization in bulk of triselenane and tetra-

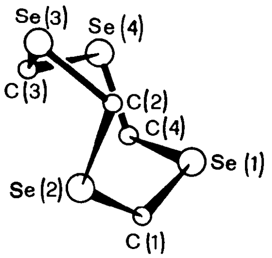

Fig. 1. A perspective view of 1,3,5,7-tetraselenocane.