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## Structure of Dibromodioxouranium(VI)–Tris(tetrahydrofuran)

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**Abstract.** [UO<sub>2</sub>Br<sub>2</sub>].3C<sub>4</sub>H<sub>8</sub>O,  $M_r = 646.17$ , monoclinic,  $P2_1/c$ ,  $a = 6.931(3)$ ,  $b = 17.032(4)$ ,  $c = 16.257(4)$  Å,  $\beta = 94.25(5)^\circ$ ,  $V = 1914(2)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.242$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 121.920$  cm<sup>-1</sup>,  $F(000) = 1192$ ,  $T = 295(1)$  K,  $R = 0.037$  for 965 observed reflections. The coordination polyhedron of the U atom is a bipyramid with a non-planar pentagonal base made up of two Br<sup>-</sup> ions and three THF O atoms. The two uranyl O atoms are

in the apical positions. The stacking in the crystal is dictated by van der Waals interactions.

**Introduction.** The title compound has been prepared as part of our investigations on the bonding and coordination geometry in actinide complexes. We report here its characterization and structure analysis by X-ray diffraction.

**Experimental.** Light-brown single crystals obtained by reaction of a solution of [U{1,4,7-(CH<sub>3</sub>)<sub>3</sub>C<sub>9</sub>H<sub>4</sub>}]Br<sub>3</sub>.2THF in THF with dry oxygen (5 µl per week)

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at room temperature and sealed inside thin-walled glass capillaries under an inert atmosphere. Crystal  $0.15 \times 0.25 \times 0.30$  mm. Enraf-Nonius CAD-4 X-ray diffractometer, graphite-monochromated Mo  $K\alpha$  radiation. Cell parameters refined by least squares from angle data of 25 reflections in range  $20-40^\circ$  ( $2\theta$ ). Space group unequivocally established from systematic absences. 5529 reflections collected ( $\theta-2\theta$  scan mode) in range  $4 \leq 2\theta \leq 46^\circ$ ,  $h -7/7$ ,  $k -18/0$ ,  $l -17/17$ ; 1900 reflections considered observed [ $I \geq 2\sigma(I)$ ], averaged to 1601 unique reflections ( $R_{\text{int}} = 0.08$ ), 965 reflections with  $I > 2\sigma(I)$  used in refinement. Three reflections monitored periodically showed no significant deviations from mean. Intensities corrected for Lorentz-polarization effects; empirical absorption corrections (transmission

factors range from 58.93 to 99.54%). Structure solved by direct methods and Fourier techniques; full-matrix least-squares refinement [minimizing  $\sum w(\Delta F)^2$ ;  $w = 1/[\sigma(F_o)]^2$  with  $\sigma(F_o) = \sigma(F_o^2)/2F_o$  and  $\sigma(F_o^2) = [\sigma^2(I) + (A/I)^2]^{1/2}/L_p$  where  $A$ , the ignorance factor, is 0.06]. Anisotropic thermal parameters for non-H atoms; H atoms in calculated positions, included in final structure-factor calculation with  $B_{\text{iso}} = 5.0 \text{ \AA}^2$ .  $R = 0.037$ ,  $wR = 0.039$ ,  $S = 1.002$ ,  $(\Delta/\sigma)_{\text{max}} = 0.01$ ,  $\Delta\rho$  in final difference Fourier map within 0.823 and  $-0.622 \text{ e \AA}^{-3}$ . Atomic scattering factors and anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974). Enraf-Nonius (1981) SDP programs.

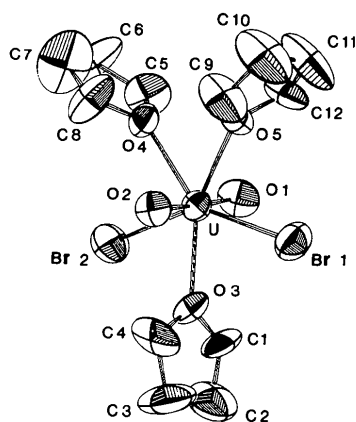


Fig. 1. The molecular structure.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (e.s.d.'s in parentheses)

	x	y	z	$B_{\text{eq}}^*(\text{\AA}^2)$
U	0.0601 (1)	0.22052 (5)	0.91043 (5)	4.34 (2)
Br(1)	0.2026 (4)	0.3404 (2)	1.0207 (2)	7.30 (8)
Br(2)	0.1209 (4)	0.0625 (2)	0.8596 (2)	8.08 (8)
O(1)	0.238 (2)	0.2488 (8)	0.8457 (8)	6.5 (4)
O(2)	-0.131 (2)	0.1944 (7)	0.9712 (7)	5.4 (4)
O(3)	0.255 (2)	0.1544 (8)	1.0216 (8)	5.6 (4)
O(4)	-0.161 (2)	0.1964 (7)	0.7868 (8)	5.5 (4)
O(5)	-0.129 (2)	0.3386 (7)	0.8713 (7)	5.4 (4)
C(1)	0.461 (3)	0.152 (1)	1.022 (1)	7.8 (7)
C(2)	0.523 (4)	0.116 (2)	1.105 (2)	10.8 (9)
C(3)	0.353 (3)	0.082 (2)	1.140 (2)	13 (1)
C(4)	0.174 (3)	0.113 (1)	1.089 (1)	9.2 (7)
C(5)	-0.099 (3)	0.192 (1)	0.702 (1)	7.4 (7)
C(6)	-0.268 (4)	0.146 (2)	0.660 (1)	12.1 (9)
C(7)	-0.420 (5)	0.136 (2)	0.711 (1)	14 (1)
C(8)	-0.358 (3)	0.160 (2)	0.788 (1)	8.6 (8)
C(9)	-0.310 (3)	0.361 (1)	0.910 (2)	9.6 (8)
C(10)	-0.344 (4)	0.440 (2)	0.884 (2)	12.4 (9)
C(11)	-0.173 (4)	0.471 (1)	0.852 (2)	12 (1)
C(12)	-0.050 (3)	0.405 (1)	0.831 (1)	7.2 (7)

$$* B_{\text{eq}} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + abc\cos\gamma B(1,2) + accos\beta B(1,3) + bccos\alpha B(2,3)].$$

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

(1) U coordination sphere			
U—O(1)	1.75 (1)	O(1)—U—O(2)	176.3 (7)
U—O(2)	1.77 (1)		
U—Br(1)	2.845 (3)	Br(1)—U—O(3)	73.6 (4)
U—Br(2)	2.856 (3)	O(3)—U—Br(2)	72.3 (4)
U—O(3)	2.45 (2)	Br(2)—U—O(4)	72.8 (4)
U—O(4)	2.47 (1)	O(4)—U—O(5)	68.8 (5)
U—O(5)	2.46 (2)	O(5)—U—Br(1)	74.3 (4)
(2) THF			
O(3)—C(1)	1.43 (3)	C(7)—C(8)	1.35 (4)
C(1)—C(2)	1.51 (4)	C(8)—O(4)	1.50 (3)
C(2)—C(3)	1.46 (4)	O(5)—C(9)	1.49 (3)
C(3)—C(4)	1.54 (4)	C(9)—C(10)	1.42 (4)
C(4)—O(3)	1.45 (3)	C(10)—C(11)	1.43 (4)
O(4)—C(5)	1.47 (2)	C(11)—C(12)	1.47 (4)
C(5)—C(6)	1.54 (4)	C(12)—O(5)	1.43 (3)
C(6)—C(7)	1.41 (4)		
U—O(3)—C(1)	121 (2)	C(5)—C(6)—C(7)	112 (3)
U—O(3)—C(4)	124 (2)	C(6)—C(7)—C(8)	108 (4)
C(4)—O(3)—C(1)	115 (2)	C(7)—C(8)—O(4)	110 (3)
O(3)—C(1)—C(2)	104 (2)	U—O(5)—C(9)	123 (2)
C(1)—C(2)—C(3)	109 (3)	U—O(5)—C(12)	123 (2)
C(2)—C(3)—C(4)	107 (3)	C(12)—O(5)—C(9)	111 (2)
C(3)—C(4)—O(3)	104 (2)	O(5)—C(9)—C(10)	104 (3)
U—O(4)—C(5)	124 (2)	C(9)—C(10)—C(11)	110 (3)
U—O(4)—C(8)	125 (2)	C(10)—C(11)—C(12)	107 (3)
C(8)—O(4)—C(5)	108 (2)	C(11)—C(12)—O(5)	105 (2)
O(4)—C(5)—C(6)	100 (2)		

Table 3. Short intramolecular contacts ( $\text{\AA}$ ) with e.s.d.'s in parentheses

Br(1)···O(1)	3.27 (1)	Br(2)···O(1)	3.29 (2)
Br(1)···O(3)	3.19 (2)	Br(2)···O(3)	3.14 (2)
Br(1)···O(5)	3.22 (2)	Br(2)···O(4)	3.17 (1)
Br(1)···C(1)	3.67 (3)	Br(2)···C(1)	3.74 (3)
Br(1)···C(4)	4.03 (3)	Br(2)···C(4)	3.81 (3)
Br(1)···C(9)	3.87 (3)	Br(2)···C(5)	3.63 (3)
Br(1)···C(12)	3.60 (3)	Br(2)···C(8)	3.81 (3)
O(1)···C(5)	3.32 (3)	O(2)···C(4)	3.07 (3)
O(1)···C(12)	3.32 (3)	O(2)···C(8)	3.32 (3)
		O(2)···C(9)	3.22 (3)
O(4)···O(5)	2.79 (2)	O(2)···O(3)	2.82 (2)

**Discussion.** The atomic nomenclature and the configuration of the molecule are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Final atomic coordinates for non-H atoms are given in Table 1;\* selected interatomic distances, angles and short intramolecular contacts are reported in Tables 2 and 3.

The U–O bond lengths within the  $\text{UO}_2$  unit compare with the mean value of 1.764 Å for fivefold equatorial coordination (*Gmelin's Handbook of Inorganic Chemistry*, 1983). The equatorial U–O(THF) bond distances at 2.45 (2)–2.47 (1) Å are in the range observed for uranyl and uranium complexes. In  $[\text{UO}_2\text{Cl}(\text{C}_6\text{H}_5\text{CONOHC}_6\text{H}_5)] \cdot 2\text{C}_4\text{H}_8\text{O}$  (Smith & Raymond, 1979) the corresponding distances were 2.42 (3) and 2.47 (3) Å; in  $[\text{UO}_2(\text{NO}_3)_2] \cdot 2\text{C}_4\text{H}_8\text{O}$  (Reynolds, Zalkin & Templeton, 1977) 2.42 (2) Å; in  $[\text{U}(\text{BH}_4)_4] \cdot 2\text{C}_4\text{H}_8\text{O}$  (Rietz, Edelstein, Ruben, Templeton & Zalkin, 1978) 2.47 (1) Å; and in  $[\text{U}(\text{C}_9\text{H}_7)]\text{Br}_3 \cdot 2\text{C}_4\text{H}_8\text{O}$  (Rebizant, Spirlet & Goffart, 1985) 2.466 (8) Å.

The lengthening of the U–Br bond distances [2.845 (3), 2.856 (3) Å] in comparison with the values observed in  $[\text{NH}_4]_2[\text{UO}_2\text{Br}_4] \cdot 2\text{H}_2\text{O}$  (Van den Bossche, Spirlet, Rebizant & Goffart, 1987), 2.812 (1) and 2.815 (1) Å, and in  $[\text{U}(\text{C}_9\text{H}_7)]\text{Br}_3 \cdot 2\text{C}_4\text{H}_8\text{O}$  (Rebizant *et al.*, 1985), 2.759 (1), 2.775 (2) and 2.791 (1) Å, is a

\*Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43870 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

result of steric interference with the three equatorial THF rings. Table 3 shows evidence of interligand compressions which have resulted in distortions of the molecule from the ideal geometry of a precisely planar equatorial set of ligands. Indeed the coordinating ligand atoms are substantially displaced out of the mean equatorial plane (by up to 0.3 Å). A significant bending of 15.6 and 15.8° respectively of the two neighbouring THF ligands towards Br is also observed. As expected the outermost C atoms of the THF molecules exhibit large thermal motions, principally perpendicular to the rings, and some C–C bond lengths are therefore foreshortened. The stacking in the crystal is dictated by van der Waals interactions.

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## Structure of Dichlorobis(2,2-dimethoxy-2-phenylethylamine)palladium(II)

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**Abstract.**  $[\text{PdCl}_2(\text{C}_{10}\text{H}_{15}\text{NO}_2)_2]$ ,  $M_r = 539.4$ , triclinic,  $P\bar{1}$ ,  $a = 10.391$  (2),  $b = 10.457$  (1),  $c = 5.7833$  (4) Å,  $\alpha = 105.875$  (7),  $\beta = 103.875$  (8),  $\gamma = 87.21$  (1)° (standardized parameters are:  $a = 5.7833$ ,  $b = 10.391$ ,  $c = 10.457$  Å,  $\alpha = 87.21$ ,  $\beta = 74.125$ ,  $\gamma = 76.125^\circ$ ),  $U = 586.7$  Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.53$  Mg m<sup>−3</sup>, Mo  $K\alpha$ ,  $\lambda$

$= 0.71069$  Å,  $\mu = 1.03$  mm<sup>−1</sup>,  $F(000) = 276$ ,  $T = 291$  K, final  $R = 0.025$  for 4187 unique observed reflections. The Pd atom exhibits a square-planar coordination geometry with the amine groups occupying *trans* positions. Bond lengths and angles fall within normal ranges.

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