

## Structure of *catena*-Poly{[aqua(benzoato)dioxouranium(VI)]- $\mu$ -(benzoato)} Ethanol Solvate

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**Abstract.**  $[U(C_7H_5O_2)_2(O)_2(H_2O)] \cdot C_2H_5O$ ,  $M_r = 576.35$ , monoclinic,  $Cc$ ,  $a = 11.659$  (5),  $b = 13.864$  (4),  $c = 11.586$  (8) Å,  $\beta = 94.09$  (4)°  $V = 1868$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.049$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71073$  Å,  $\mu = 82.75$  cm<sup>-1</sup>,  $F(000) = 1080$ ,  $T = 295$  K,  $R = 0.033$  for 1305 unique reflections. The structure is of polymeric type. The linear  $UO_2^{2+}$  is equatorially surrounded by five O atoms from two bidentate benzoate ligands and one water molecule. The pentagon is not exactly planar: the maximum deviations of the atoms from this plane are  $\pm 0.14$  Å. One benzoate ligand is bidentate on one U atom with its two O atoms adjacent in the equatorial plane while the other is shared by two U atoms. Two pentagons are then linked by bridging benzoate to form a polymer chain.

**Experimental.** Yellow crystal obtained on leaving a mixture of silver benzoate–ethanol and uranyl

chloride–water for several days at 294 K; crystal  $0.15 \times 0.15 \times 0.40$  mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo  $K\alpha$  radiation;  $\omega/2\theta$  scan technique; cell parameters obtained from least-squares procedure on 25 reflections ( $8 < \theta < 12^\circ$ ); three standard reflections ( $\bar{2}24$ , 006,  $\bar{2}06$ ) monitored every hour, no significant decay was observed, 0.6% in 24 h; data corrected for Lorentz, polarization and empirical absorption based on  $\psi$  scans,  $0.46 < T < 1.00$  (North, Phillips & Mathews, 1968); 1883 reflections collected with  $4 < 2\theta < 50^\circ$ , 1645 unique ( $R_{int} = 2.8\%$ ), 1305 with  $I > 3\sigma(I)$ ;  $h(-13,13)$   $k(0,16)$   $l(0,13)$ ; structure solved by the heavy-atom method and refined by full-matrix least squares on  $F$ ; anisotropic thermal parameters for U

Table 1. Atomic coordinates and equivalent isotropic thermal parameters

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (Å <sup>2</sup> )
U	0.000	0.96427 (4)	0.000	2.288 (8)*
O(1)	0.148 (1)	1.006 (1)	0.031 (1)	3.9 (3)*
O(2)	-0.137 (1)	0.926 (1)	-0.028 (1)	3.6 (3)*
O(3)	0.086 (1)	0.799 (1)	-0.018 (1)	3.9 (3)*
O(4)	0.054 (1)	0.887 (1)	-0.182 (1)	4.3 (3)*
O(5)	-0.005 (1)	0.884 (1)	0.176 (1)	4.4 (3)*
O(6)	-0.023 (1)	1.085 (1)	-0.132 (1)	4.1 (3)*
O(7)	-0.062 (1)	1.093 (1)	0.120 (1)	3.0 (3)*
O(21)	-0.249 (2)	1.202 (1)	0.136 (1)	6.3 (4)*
C(1)	-0.028 (2)	0.864 (2)	0.281 (2)	3.2 (4)
C(2)	-0.071 (2)	0.764 (2)	0.289 (2)	3.3 (4)
C(3)	-0.088 (2)	0.726 (2)	0.396 (2)	5.2 (6)
C(4)	-0.122 (3)	0.634 (3)	0.410 (3)	7.3 (8)
C(5)	-0.144 (2)	0.572 (2)	0.330 (2)	5.6 (6)
C(6)	-0.130 (3)	0.619 (2)	0.194 (3)	6.4 (7)
C(7)	-0.086 (2)	0.708 (2)	0.197 (3)	5.7 (6)
C(8)	0.084 (2)	0.802 (2)	-0.124 (2)	3.0 (4)
C(9)	0.108 (2)	0.715 (1)	-0.184 (2)	2.5 (3)
C(10)	0.118 (2)	0.626 (2)	-0.135 (2)	4.6 (5)
C(11)	0.142 (2)	0.545 (2)	-0.200 (2)	5.5 (6)
C(12)	0.158 (3)	0.561 (2)	-0.292 (3)	6.6 (7)
C(13)	0.154 (2)	0.637 (2)	-0.372 (2)	4.1 (5)
C(14)	0.126 (2)	0.720 (2)	-0.313 (2)	4.4 (5)
C(22)	-0.270 (3)	1.200 (3)	0.268 (3)	8.4 (9)
C(23)	-0.225 (4)	1.295 (3)	0.293 (4)	10 (1)

$$*B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j.$$

Table 2. Selected bond distances (Å) and angles (°)

U environment			
U—O(1)	1.831 (7)	U—O(5)	2.33 (1)
U—O(2)	1.692 (7)	U—O(6)	2.266 (7)
U—O(3)	2.513 (7)	U—O(7)	2.401 (6)
U—O(4)	2.48 (1)		
O(1)—U—O(2)	179.5 (4)	O(3)—U—O(4)	53.9 (3)
O(1)—U—O(3)	85.8 (3)	O(3)—U—O(5)	70.9 (4)
O(1)—U—O(4)	90.6 (3)	O(4)—U—O(6)	76.7 (3)
O(1)—U—O(5)	93.2 (5)	O(5)—U—O(7)	79.7 (3)
O(1)—U—O(6)	88.2 (3)	O(6)—U—O(7)	79.3 (2)
O(1)—U—O(7)	88.2 (4)		
Benzoate ligands			
O(4)—C(8)	1.40 (2)	O(5)—C(1)	1.29 (2)
O(3)—C(8)	1.22 (1)	O(6)—C(1)	1.24 (1)
C(8)—C(9)	1.43 (2)	C(1)—C(2)	1.48 (1)
C(9)—C(10)	1.37 (2)	C(2)—C(3)	1.38 (2)
C(10)—C(11)	1.39 (2)	C(3)—C(4)	1.36 (2)
C(11)—C(12)	1.11 (2)	C(4)—C(5)	1.27 (2)
C(12)—C(13)	1.40 (2)	C(5)—C(6)	1.73 (2)
C(13)—C(14)	1.40 (2)	C(6)—C(7)	1.34 (2)
C(14)—C(9)	1.52 (2)	C(7)—C(2)	1.31 (2)
C(7)—C(2)—C(3)	120 (1)	C(14)—C(9)—C(10)	116 (1)
C(2)—C(3)—C(4)	122 (1)	C(9)—C(10)—C(11)	121 (1)
C(3)—C(4)—C(5)	127 (2)	C(10)—C(11)—C(12)	115 (2)
C(4)—C(5)—C(6)	113 (2)	C(11)—C(12)—C(13)	141 (2)
C(5)—C(6)—C(7)	112 (2)	C(12)—C(13)—C(14)	107 (1)
C(6)—C(7)—C(2)	126 (2)	C(13)—C(14)—C(9)	120 (1)
Ethanol			
O(21)—C(22)	1.57 (2)	O(21)—C(22)—C(23)	96 (2)
C(22)—C(23)	1.43 (2)		

Symmetry code: (i)  $x, -y, \frac{1}{2} + z$ .

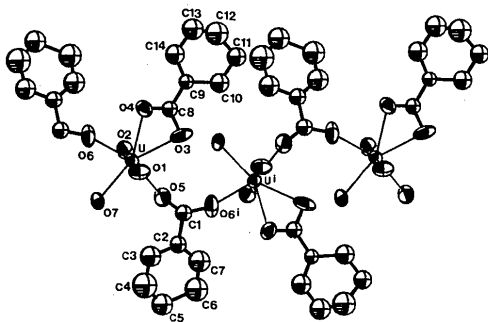


Fig. 1. ORTEP (Johnson, 1976) drawing of the polymer [symmetry code: (i)  $x, -y, \frac{1}{2} + z$ ].

and O; scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974 Vol. IV, Tables 2.2B and 2.3.1); H atoms of benzoate groups introduced at calculated positions, not refined but constrained to ride on their C atoms (C—H = 0.95 Å and  $B = 6 \text{ Å}^2$ ),  $R = 0.033$ ,  $wR = 0.0411$  ( $w = 1$ ),  $(\Delta/\sigma)_{\max} = 0.10$ ,  $S = 5.02$ . In final difference Fourier synthesis, no peak greater than  $0.72 \text{ e Å}^{-3}$  except for two peaks ( $1.3$  and  $1.03 \text{ e Å}^{-3}$ ) in the vicinity of the U atom; calculations carried out on a Digital Computer MicroVAX II using the SDP chain of programs

(Frenz, 1985). The structure belongs clearly to the non-centrosymmetric group and the absolute configuration has been obtained unambiguously.

Atomic coordinates are given in Table 1, selected bond lengths and angles in Table 2.\* Fig. 1 shows the polymeric structure and the labelling scheme.

**Related literature.** The title compound was obtained during the preparation of diaqua(benzoato)chlorodioxouranium(VI) (Charpin, Keller, Lance & Vigner, 1989).

\* 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 53189 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

#### References

- CHARPIN, P., KELLER, N., LANCE, M. & VIGNER, D. (1989). *Acta Cryst.* C45, 954–956.  
 FRENZ, B. A. (1985). *Enraf-Nonius SDP-Plus Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.  
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.  
 NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. D. (1968). *Acta Cryst.* A24, 351–359.

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## Structure of Di- $\mu$ -oxo-bis[(cyclopentadienyl)(heptafluoropropyl)oxotungsten]

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**Abstract.** C<sub>8</sub>H<sub>5</sub>F<sub>7</sub>O<sub>2</sub>W,  $M_r = 449.96$ , triclinic,  $P\bar{1}$ ,  $a = 6.294$  (4),  $b = 12.737$  (9),  $c = 14.871$  (11) Å,  $\alpha = 67.83$  (5),  $\beta = 78.97$  (5),  $\gamma = 83.74$  (5)°,  $V = 1083$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 2.760 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71073$  Å,  $\mu = 10.99 \text{ mm}^{-1}$ ,  $F(000) = 824$ ,  $T = 291$  (1) K, final  $R = 0.074$  for 2840 unique observed [ $F \geq 4.0\sigma(F)$ ] diffractometer data. The crystal contains two independent centrosymmetric molecular dimers. The central part of these molecules is a planar four-membered W—O—W—O ring [W—O

1.95 (2) Å, O—W—O 74.4 (6)°, W...W 3.102 (3), O...O 2.35 (2) Å]. A cyclopentadienyl ring is attached to each W, with W—C distances in the range 2.33 (2)–2.49 (2) Å and these rings are *trans* to each other with respect to the central ring. If one takes the centers of the four-membered W—O—W—O ring and the cyclopentene ring as coordination points, these two coordination points and the atoms C(*i*1) and O(*i*1) ( $i = 1, 2$ ) bound to W form a distorted tetrahedron around W.