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Key indicators

Single-crystal X-ray study
 T = 293 K
 Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
 R factor = 0.022
 wR factor = 0.051
 Data-to-parameter ratio = 20.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

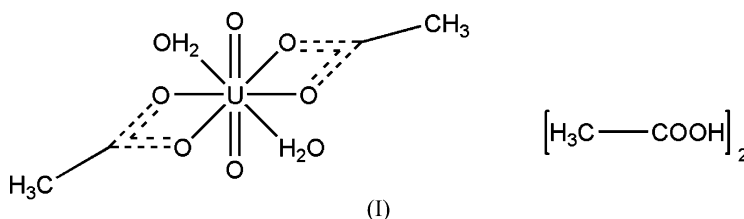
Diacetatodiaquadioxouranium(VI) acetic acid disolvate

The coordination polyhedron of the U atom in the title compound, $[\text{U}(\text{C}_2\text{H}_3\text{O}_2)_2\text{O}_2(\text{H}_2\text{O})_2] \cdot 2\text{CH}_3\text{COOH}$, is a distorted hexagonal bipyramid, with the equatorial positions occupied by four O atoms of two bidentate-chelating acetate anions and two O atoms of coordinated water molecules. The complex lies on an inversion centre. A two-dimensional network of hydrogen bonds is present in the crystal structure.

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Comment

Carboxylate compounds of the actinides play a significant role in laboratory practice and the radiochemical industry. Several uranyl compounds are known, in which both acetate anions and acetic acid molecules are present (Ramos Silva *et al.*, 1999; Grigor'ev *et al.*, 2004). Here, we present a further example of a uranyl acetate compound, (I), containing acetic acid as solvent.



The U atom in compound (I) lies on an inversion centre. Its coordination polyhedron is a distorted hexagonal bipyramid, with the equatorial positions occupied by four O atoms of two bidentate-chelating acetate anions and two O atoms of coordination water molecules (Fig. 1). Crystallographically independent bond lengths in the U-atom environment are given in Table 1.

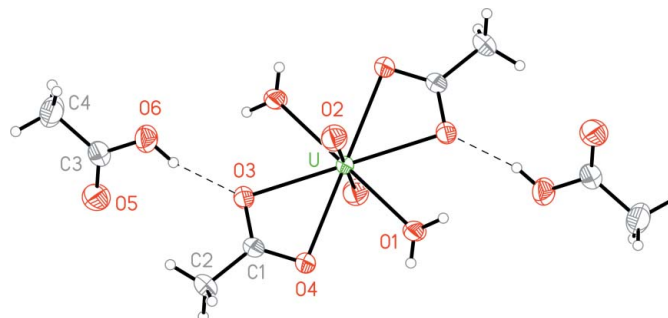


Figure 1
 A view of (I), showing the atom-labelling scheme for the asymmetric unit. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Dashed lines indicate the hydrogen-bonding interactions. [Symmetry code for unlabelled atoms: 1 - x; 1 - y; 1 - z.]

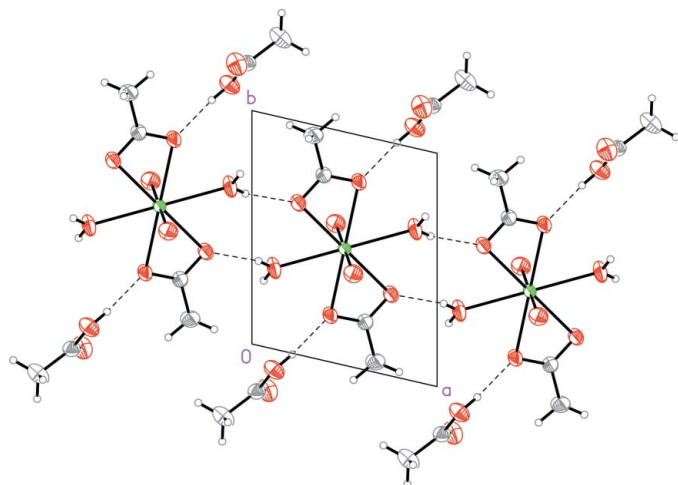


Figure 2
Part of an infinite chain in the crystal structure of (I). Dashed lines indicate hydrogen bonds.

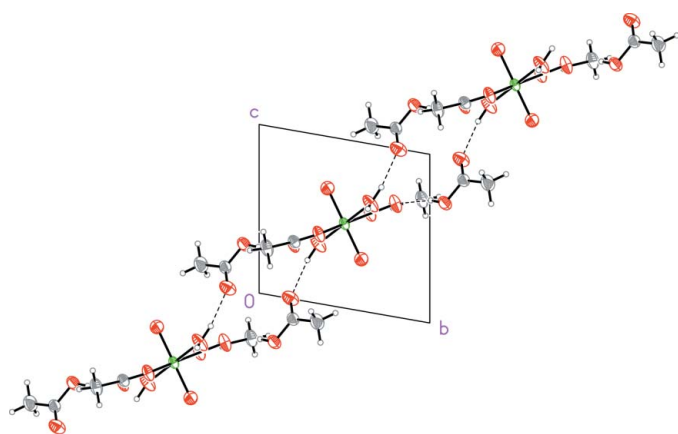


Figure 3
A layer in the crystal structure of (I), viewed along the direction of the chains. Dashed lines indicate hydrogen bonds.

The coordinated water and solvent acetic acid molecules act as H donors in several hydrogen bonds (Table 2). The acceptors in these bonds are O atoms of acetate anions and acetic acid. The hydrogen bonding results in the formation of infinite chains in the [100] direction (Fig. 2) and these chains are combined into layers parallel to the (01 $\bar{1}$) plane (Fig. 3).

Experimental

Crystals of (I) were obtained in an attempt to recrystallize anhydrous uranyl acetate from glacial acetic acid at 413 K. Possibly, a small admixture of water in the acid has resulted in the formation of (I)

Crystal data

[U(C ₂ H ₃ O ₂) ₂ O ₂ (H ₂ O) ₂] \cdot 2C ₂ H ₄ O ₂	$Z = 1$
$M_r = 544.25$	$D_x = 2.291 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 6.914 (2) \text{ \AA}$	Cell parameters from 24 reflections
$b = 7.822 (2) \text{ \AA}$	$\theta = 13.1\text{--}14.0^\circ$
$c = 8.211 (2) \text{ \AA}$	$\mu = 10.34 \text{ mm}^{-1}$
$\alpha = 95.00 (2)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 113.74 (2)^\circ$	Irregular fragment, yellow
$\gamma = 99.79 (2)^\circ$	$0.24 \times 0.18 \times 0.12 \text{ mm}$
$V = 394.6 (2) \text{ \AA}^3$	

Data collection

Siemens *P3/PC* diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.168$, $T_{\max} = 0.289$
 2494 measured reflections
 2316 independent reflections
 2316 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$
 $\theta_{\text{max}} = 30.1^\circ$
 $h = 0 \rightarrow 9$
 $k = -11 \rightarrow 10$
 $l = -11 \rightarrow 10$
 2 standard reflections
 every 98 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.022$
 $wR(F^2) = 0.051$
 $S = 1.05$
 2316 reflections
 111 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0313P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 1.36 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -1.55 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (\AA).

U—O2	1.760 (3)	U—O3	2.480 (3)
U—O1	2.441 (3)	U—O4	2.536 (3)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O6—H6 \cdots O3	0.86 (6)	1.84 (6)	2.672 (4)	163 (6)
O1—H1A \cdots O5 ⁱ	0.88 (7)	1.86 (7)	2.727 (4)	168 (6)
O1—H1B \cdots O4 ⁱⁱ	0.75 (9)	2.05 (9)	2.778 (4)	163 (9)

Symmetry codes: (i) $x + 1, y + 1, z + 1$; (ii) $-x + 2, -y + 1, -z + 1$.

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but each group was allowed to rotate freely about its C—C bond. The positions of H atoms bonded to O atoms were refined freely along with isotropic displacement parameters. The highest peak and the deepest hole on the final difference electron-density map are located 0.87 and 0.82 \AA , respectively, from atom U.

Data collection: *P3/PC* (Siemens, 1989); cell refinement: *P3/PC*; data reduction: *P3/PC*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXTL*.

References

- Grigor'ev, M. S., Antipin, M. Yu. & Krot, N. N. (2004). *Radiochemistry*, **46**, 224–231.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Ramos Silva, M., Matos Beja, A., Paixão, J. A., Alte da Veiga, L. & Martin-Gil, J. (1999). *Acta Cryst.* **C55**, 2039–2041.
- Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1989). *P3/PC*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.