Hydrogen-bonded network structures in dipyridinium, bis(2-methylpyridinium), bis(3-methylpyridinium) and bis(4-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI)

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Four complexes containing the $[\text{UO}_2(\text{oda})_2]^{2-}$ anion (oda is oxydiacetate) are reported, namely dipyridinium dioxido-bis(oxydiacetato)uranate(VI), $(\text{C}_5\text{H}_6\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$, (I), bis(2-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI), $(\text{C}_8\text{H}_8\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$, (II), bis(3-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI), $(\text{C}_8\text{H}_8\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$, (III), and bis(4-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI), $(\text{C}_8\text{H}_8\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$, (IV). The anions are achiral and are located on a mirror plane in (I) and on inversion centres in (II)–(IV). The four complexes are assembled into three-dimensional structures *via* N–H...O and C–H...O interactions. Compounds (III) and (IV) are isomorphous; the $[\text{UO}_2(\text{oda})_2]^{2-}$ anions form a porous matrix which is nearly identical in the two structures, and the cations are located in channels formed in this matrix. Compounds (I) and (II) are very different from (III) and (IV): (I) forms a layered structure, while (II) forms ribbons.

Comment

We recently published a study of complexes featuring the nine-coordinate chiral $[\text{Ln}(\text{oda})_3]^{3-}$ anion (where Ln = Pr, Eu, Gd or Dy, and oda = oxydiacetate) (Lennartson & Håkansson, 2009*a*). In the case of $\text{Na}_5[\text{Er}(\text{oda})_3](\text{H}_2\text{O})_6(\text{BF}_4)_2$, which crystallizes in the Sohncke space group (Flack, 2003) *R*32, it was possible to crystallize a whole sample as one enantiomerically pure single crystal. This represents the first example of the preparation of enantiomerically pure bulk quantities of a nine-coordinate complex displaying only achiral ligands. Since all precursors [diglycolic acid, erbium(III) chloride hexahydrate, sodium hydroxide, sodium bicarbonate, sodium tetrafluoroborate and water] were achiral, the overall synthesis may be regarded as a case of absolute asymmetric

synthesis (Feringa & van Delden, 1999; Mislow, 2003). Progressing from the oxydiacetate lanthanide complexes we have examined the corresponding actinide complexes, and four complexes containing the dioxido-bis(oxydiacetato)uranate(VI) anion, $[\text{UO}_2(\text{oda})_2]^{2-}$, are presented in this paper.

Only six crystal structures of uranyl complexes containing the oxydiacetate ligand are listed in the Cambridge Structural Database (CSD, Version 5.30 of May 2009; Allen, 2002). In the absence of coordinating ligands, uranyl oxydiacetate forms a coordination polymer (Bombieri *et al.*, 1974), which undergoes spontaneous resolution (Jacques *et al.*, 1984; Perez-Garcia & Amabilino, 2007) on crystallization. The other structures published are oxodiacetatodi(pyridine oxide)dioxouranium(VI) (Bombieri *et al.*, 1973), di(1,3,5,7-tetraazaadamant-1-ium)-di(μ_2 -hydroxo)di(oxodiacetato)tetraoxodiuuranium(VI) dihydrate (Jiang *et al.*, 2002) and three structures containing the $[\text{UO}_2(\text{oda})_2]^{2-}$ anion (Bombieri *et al.*, 1973; Jiang *et al.*, 2002).

Dipyridinium dioxido-bis(oxydiacetato)uranate(VI), (I), bis(2-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI), (II), bis(3-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI), (III), and bis(4-methylpyridinium) dioxido-bis(oxydiacetato)uranate(VI), (IV), all form yellow crystals from aqueous solution. None of the crystal structures includes water, neither coordinated to the U atom nor as co-crystallized water.

Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]

Figure 2

The molecular structure of (II), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (ii) $-x, -y, -z$.]

Figure 3

The molecular structure of (III), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (ii) $-x, -y, -z$.]

Figure 4

The molecular structure of (IV), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (ii) $-x, -y, -z$.]

Figure 5

The $N-H \cdots O$ and $C-H \cdots O$ bonded layer in (I). Hydrogen bonds are shown as dashed lines. All H atoms not involved in these interactions have been omitted for clarity.

Figure 6

Diagram showing how the layers in (I) are assembled into a network structure by $C-H \cdots O$ interactions (dashed lines). Three layers are depicted and run horizontally in the figure. All H atoms not involved in these interactions have been omitted for clarity.

The uranyl moieties in compounds (I)–(IV) are linear, as expected, and are coordinated by two oxydiacetate ligands, giving rise to complex $[UO_2(oda)_2]^{2-}$ anions. The anions differ somewhat between the four compounds. In (I), the $[UO_2(oda)_2]^{2-}$ anion is located on a mirror plane bisecting both oxydiacetate ligands (Fig. 1). The two oxydiacetate ligands are coordinated differently to the central U atom. One is virtually planar, and atoms O3 and O5 are both coordinated to the central U atom. The other ligand deviates considerably from planarity and, since the $U1-O8$ distance is probably too long to be considered a $U-O$ bond, it is best described as a bidentate ligand. The $[UO_2(oda)_2]^{2-}$ anions in (II), (III) and (IV) are very similar (Figs. 2–4), with the central U atoms located on crystallographic inversion centres and with the oxydiacetate ligands virtually planar. Both types of coordi-

Figure 7

Diagram showing how the N—H...O and C—H...O interactions in (II) (dashed lines) give rise to ribbons. All H atoms not involved in these interactions have been omitted for clarity.

Figure 8

Schematic drawing of the ribbons in the structure of (II). The ribbons form layers which are stacked into a three-dimensional structure; three such layers are depicted.

nation mode have been reported previously (Jiang *et al.*, 2002). Selected geometric parameters for (I)–(IV) are compared in Table 1. The $[\text{UO}_2(\text{oda})_2]^{2-}$ anions are achiral, in contrast with the propeller-shaped $[\text{Ln}(\text{oda})_3]^{3-}$ anions, but this does not exclude the possibility of a chiral crystal structure, since achiral molecules may assemble into chiral supramolecular structures (Matsuura & Koshima, 2005; Lennartson & Håkansson, 2009b). However, compounds (I)–(IV) form centrosymmetric crystals.

The ions in (I) are associated by N—H...O and C—H...O interactions (Table 2). Classical N—H...O interactions form a short contact between atoms H1 and O7 within the asymmetric unit. Due to symmetry, each $[\text{UO}_2(\text{oda})_2]^{2-}$ anion will interact with two pyridinium cations. The C—H...O interactions involving H5...O4($1-x, -y, 1-z$), H7...O4($1-x, -y, 2-z$) and H8...O7($x, y, 1+z$) give rise to layers in the bc plane (Fig. 5). These layers are further associated into a network structure (Fig. 6) by two sets of C—H...O interactions, *viz.* H2A...O2($-1+x, y, z$) and H4A...O1($1+x, y, z$).

Introducing a methyl group in the 2-position on the pyridinium cation, *i.e.* on going from (I) to (II), dramatically alters

Figure 9

The C—H...O interactions in (III) (dashed lines), involving the cation (top) and the anion (bottom).

Figure 10

Diagram showing how the cations in (III) are located in channels running through the unit cell. All H atoms have been omitted.

the crystal packing (Table 3). The 2-picolinium cation in (II) binds two $[\text{UO}_2(\text{oda})_2]^{2-}$ anions through N—H...O and C—H...O interactions. Two sets of interactions, *viz.* H1...O3($-x, -y, -z$) and H10...O5, connect the 2-picolinium cation to one anion, and a third interaction, H8...O6($1-x, 1-y, -z$), introduces connections to a second anion. As seen in Fig. 7, these interactions give rise to infinite ribbons.

Figure 11
C—H...O interactions in (IV) (dashed lines), involving the cation (top) and the anion (bottom).

Sets of ribbons are partly stacked in a similar fashion to the strakes in a ship's hull, giving rise to layers. The layers are stacked into a three-dimensional structure, where ribbons in adjacent layers are orthogonal; a schematic drawing is presented in Fig. 8.

The crystal structure of the analogous 3-picolinium complex, (III), is different from both (I) and (II) (Table 4). N—H...O and C—H...O interactions in (III) give rise to a three-dimensional network structure (Fig. 9). The $[\text{UO}_2(\text{oda})_2]^{2-}$ anions in (III) form a porous matrix with channels running parallel to the crystallographic *a* and *c* axes. These channels are occupied by the 3-picolinium cations. A view along the *a* axis is presented in Fig. 10.

Compound (IV) is isomorphous with (III). The matrices formed by the anions are almost identical, forming the same type of channels. The orientations of the cations occupying these channels differ between the two structures, and the intermolecular interactions in (IV) (Table 5) are of course different from those in (III), as depicted in Fig. 11.

In the case of the $[\text{Ln}(\text{oda})_3]^{3-}$ complexes, spontaneous resolution did not occur for $\text{Na}_3[\text{Ln}(\text{oda})_3](\text{H}_2\text{O})_6$, which crystallized in the polar space group *Cc*. Addition of certain salts led to more complex structures, of which $\text{Na}_3\text{NH}_4[\text{Ln}(\text{oda})_3](\text{SCN})(\text{H}_2\text{O})_4$ is racemic and $\text{Na}_5[\text{Ln}(\text{oda})_3](\text{H}_2\text{O})_6(\text{BF}_4)_2$ undergoes spontaneous resolution. It appears that the presence of BF_4^- is essential for spontaneous resolution to occur in this system. Preliminary studies show that recrystallization of (I) from water in the presence of inorganic salts leads to cocrystallization in certain cases, and the formation of a chiral supramolecular structure may be observed at a future date.

Experimental

For the preparation of uranyl oxydiacetate, diglycolic acid (0.13 g, 1.0 mmol) and sodium bicarbonate (0.17 g, 2.0 mmol) were dissolved in water (5 ml). A solution of uranyl nitrate hexahydrate (0.50 g, 1 mmol) in water (5 ml) was added. The solution was heated to reflux and a yellow precipitate formed. The mixture was cooled to ambient temperature and the precipitate collected by filtration, washed with water (3×5 ml) and acetone (3×5 ml), and dried by suction (yield 0.35 g, 87%). For the preparation of (I), pyridine (0.3 ml) and water (1.0 ml) were added to a mixture of uranyl oxydiacetate (0.35 g, 0.82 mmol) and diglycolic acid (0.11 g, 0.82 mmol). The mixture was heated until a clear solution was obtained. Yellow crystals of (I) formed on cooling to ambient temperature (yield 0.33 g, 54%). Compounds (II)–(IV) were prepared in an analogous manner, substituting pyridine by 2-, 3- and 4-picoline, respectively.

Compound (I)

Crystal data

$(\text{C}_5\text{H}_6\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$	$V = 1076.5 (5) \text{ \AA}^3$
$M_r = 694.39$	$Z = 2$
Monoclinic, $P2_1/m$	Mo $K\alpha$ radiation
$a = 6.675 (2) \text{ \AA}$	$\mu = 7.61 \text{ mm}^{-1}$
$b = 23.025 (5) \text{ \AA}$	$T = 100 \text{ K}$
$c = 7.500 (2) \text{ \AA}$	$0.2 \times 0.2 \times 0.1 \text{ mm}$
$\beta = 110.946 (11)^\circ$	

Data collection

Rigaku R-Axis IIC image-plate diffractometer	5883 measured reflections
Absorption correction: multi-scan (<i>CrystalClear</i> ; Rigaku, 2000)	1853 independent reflections
$T_{\min} = 0.240$, $T_{\max} = 0.470$	1763 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.032$

Table 1

Selected geometric parameters ($\text{\AA}$, $^\circ$) for (I)–(IV).

	(I)	(II)	(III)	(IV)
U1—O1	1.781 (3)	1.760 (3)	1.756 (4)	1.760 (4)
U1—O2	1.777 (3)	2.443 (3)	2.364 (4)	2.414 (4)
U1—O4	2.348 (2)	2.690 (3)	2.716 (4)	2.653 (3)
U1—O5	2.561 (3)	2.391 (3)	2.426 (4)	2.445 (4)
U1—O6	2.357 (2)			
O1—U1—O2	178.59 (12)	92.63 (13)	90.1 (2)	87.66 (17)
O1—U1—O3	89.38 (7)			
O1—U1—O5	94.06 (11)	92.24 (14)	89.33 (18)	88.15 (18)
O1—U1—O6	96.12 (8)			
O2—U1—O4		57.89 (10)	57.79 (13)	58.72 (11)
O2—U1—O5		114.84 (10)	114.65 (14)	116.69 (12)
O3—U1—O5	61.48 (5)			
O3—U1—O6	73.72 (7)			
O4—U1—O5		57.73 (10)	56.95 (12)	58.24 (12)

Table 2

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$) for (I).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O7	0.87 (3)	1.82 (3)	2.688 (3)	179 (6)
C2—H2A...O2 ⁱ	0.97	2.61	3.446 (5)	144
C4—H4A...O1 ⁱⁱ	0.97	2.55	3.482 (5)	161
C5—H5...O4 ⁱⁱⁱ	0.93	2.28	3.168 (4)	161
C7—H7...O4 ^{iv}	0.93	2.28	3.195 (4)	167
C8—H8...O7 ^v	0.93	2.47	3.354 (4)	159

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

Table 3

Hydrogen-bond geometry (Å, °) for (II).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O3 ⁱ	0.90 (6)	1.77 (6)	2.666 (5)	170 (6)
C7—H7...O1 ⁱⁱ	0.93	2.63	3.409 (6)	141
C8—H8...O6 ⁱⁱⁱ	0.93	2.32	3.242 (7)	171
C10—H10...O5	0.93	2.41	3.328 (6)	170

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x, -y + 1, -z$; (iii) $-x + 1, -y + 1, -z$.**Refinement** $R[F^2 > 2\sigma(F^2)] = 0.016$ $wR(F^2) = 0.038$ $S = 1.06$

1853 reflections

161 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 0.81 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.96 \text{ e } \text{\AA}^{-3}$ **Compound (II)****Crystal data** $(\text{C}_6\text{H}_8\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$ $M_r = 722.44$ Monoclinic, $P2_1/c$ $a = 9.183 \text{ (2) } \text{\AA}$ $b = 11.104 \text{ (3) } \text{\AA}$ $c = 12.826 \text{ (4) } \text{\AA}$ $\beta = 111.490 \text{ (7)}^\circ$ $V = 1217.0 \text{ (6) } \text{\AA}^3$ $Z = 2$ Mo $K\alpha$ radiation $\mu = 6.73 \text{ mm}^{-1}$ $T = 295 \text{ K}$ $0.2 \times 0.2 \times 0.1 \text{ mm}$ **Data collection**

Rigaku R-AXIS IIC image-plate diffractometer

Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000) $T_{\min} = 0.304, T_{\max} = 0.510$

7415 measured reflections

2106 independent reflections

1694 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.027$ **Refinement** $R[F^2 > 2\sigma(F^2)] = 0.022$ $wR(F^2) = 0.059$ $S = 0.92$

2106 reflections

165 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 1.15 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -1.38 \text{ e } \text{\AA}^{-3}$ **Compound (III)****Crystal data** $(\text{C}_6\text{H}_8\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$ $M_r = 722.44$ Monoclinic, $P2_1/c$ $a = 7.415 \text{ (2) } \text{\AA}$ $b = 13.312 \text{ (5) } \text{\AA}$ $c = 11.806 \text{ (4) } \text{\AA}$ $\beta = 91.224 \text{ (15)}^\circ$ $V = 1165.1 \text{ (7) } \text{\AA}^3$ $Z = 2$ Mo $K\alpha$ radiation $\mu = 7.03 \text{ mm}^{-1}$ $T = 295 \text{ K}$ $0.4 \times 0.2 \times 0.1 \text{ mm}$ **Data collection**

Rigaku R-AXIS IIC image-plate diffractometer

Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000) $T_{\min} = 0.088, T_{\max} = 0.500$

7099 measured reflections

1885 independent reflections

1544 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.104$ **Refinement** $R[F^2 > 2\sigma(F^2)] = 0.050$ $wR(F^2) = 0.130$ $S = 1.03$

1885 reflections

165 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 2.20 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -3.34 \text{ e } \text{\AA}^{-3}$ **Table 4**

Hydrogen-bond geometry (Å, °) for (III).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O6	0.94 (7)	1.79 (7)	2.682 (7)	157 (6)
C3—H3A...O3 ⁱ	0.97	2.57	3.441 (8)	150
C5—H5...O2 ⁱⁱ	0.93	2.32	3.233 (8)	169
C6—H6...O6 ⁱⁱⁱ	0.93	2.55	3.309 (7)	139
C7—H7...O3 ^{iv}	0.93	2.38	3.291 (7)	165

Symmetry codes: (i) $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x, -y, -z$; (iii) $x - 1, y, z$; (iv) $x - 1, y, z + 1$.**Table 5**

Hydrogen-bond geometry (Å, °) for (IV).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1...O5	0.90 (6)	1.97 (6)	2.835 (6)	165 (7)
C2—H2B...O6 ⁱ	0.97	2.59	3.451 (6)	147
C5—H5...O3 ⁱⁱ	0.93	2.27	3.115 (6)	152
C6—H6...O6 ⁱⁱⁱ	0.93	2.50	3.387 (7)	160
C8—H8...O6 ^{iv}	0.93	2.49	3.279 (7)	143
C9—H9...O3 ^v	0.93	2.18	3.099 (7)	169

Symmetry codes: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + 1, -y, -z$; (iii) $-x + 1, -y, -z + 1$; (iv) $-x, -y, -z + 1$; (v) $-x, -y, -z$.**Compound (IV)****Crystal data** $(\text{C}_6\text{H}_8\text{N})_2[\text{U}(\text{C}_4\text{H}_4\text{O}_5)_2\text{O}_2]$ $M_r = 722.24$ Monoclinic, $P2_1/c$ $a = 7.3439 \text{ (13) } \text{\AA}$ $b = 12.999 \text{ (2) } \text{\AA}$ $c = 12.194 \text{ (2) } \text{\AA}$ $\beta = 92.438 \text{ (9)}^\circ$ $V = 1163.0 \text{ (3) } \text{\AA}^3$ $Z = 2$ Mo $K\alpha$ radiation $\mu = 7.05 \text{ mm}^{-1}$ $T = 295 \text{ K}$ $0.2 \times 0.2 \times 0.1 \text{ mm}$ **Data collection**

Rigaku R-AXIS IIC image-plate diffractometer

Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000) $T_{\min} = 0.231, T_{\max} = 0.490$

6992 measured reflections

2002 independent reflections

1752 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.061$ **Refinement** $R[F^2 > 2\sigma(F^2)] = 0.033$ $wR(F^2) = 0.086$ $S = 1.14$

2002 reflections

165 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\max} = 2.28 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -1.99 \text{ e } \text{\AA}^{-3}$

The N-bound H atoms were located in difference Fourier maps and refined isotropically, with the N—H distances in (I) and (IV) restrained to 0.90 (2) Å. The C-bound H atoms were included in calculated positions, with C—H = 0.93 (aromatic), 0.96 (methyl) or 0.97 Å (methylene), and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl groups and $1.5U_{\text{eq}}(\text{C})$ for the remainder. A few strong low-angle reflections were excluded since these caused saturation of the image plate.

For all compounds, data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLUTON* (Spek,

2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GG3221). Services for accessing these data are described at the back of the journal.

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