Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

catena-Poly[[(isonicotinato)uranyl(VI)]-μ-oxalato]

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Received 25 September 2006
Accepted 13 December 2006

The repeat unit of the title compound, $[U(C_6H_5NO_2)(C_2O_4)O_2]_n$, consists of a uranyl unit, an oxalate anion and a zwitterionic isonicotinic acid. The U atoms and the isonicotinic acid ligand are located on a crystallographic twofold rotation axis, while the oxalate lies on an inversion centre. Each UO_2 unit is coordinated by two oxalate ligands and one isonicotinic ligand. In the crystal structure, the UO_8 hexagonal bipyramids are connected by oxalate groups to form chains, and adjacent chains are linked into two-dimensional sheets by bifurcated N—H···O hydrogen bonds.

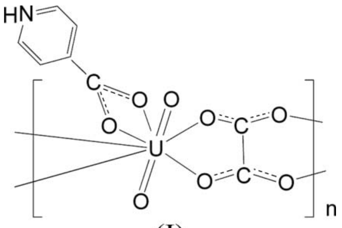
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.008 Å
R factor = 0.026
wR factor = 0.058
Data-to-parameter ratio = 15.6

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

Comment

In the past decade, metal-organic coordination polymers have attracted considerable attention due to their potential applications and intriguing architectures (Rosi *et al.*, 2003; Batten & Robson, 1998; Férey *et al.*, 2005; James, 2003). Among these compounds, reports of actinide-organic coordination polymers are less common. The uranyl ion is the most extensively studied unit in actinide chemistry because uranyl complexes display structural diversity and a wide range of physicochemical properties such as optical, magnetic and catalytic (Zheng *et al.*, 2005; Frisch & Cahill, 2005; Bean *et al.*, 2001; McCleskey *et al.*, 1999). Only six uranyl complexes containing the isonicotinic system have been found in the Cambridge Structure Database (CSD; Version 5.27, updated August, 2006; Allen, 2002). We report here the first metal-organic polymeric uranyl complex, (I), consisting of an oxalate–isonicotinic acid mixed ligand system where oxalate acts as a bridging ligand to connect two metal centres.



(I)

Compound (I) crystallizes in space group $C2/c$. U1, N1, H1, C2 and C3 lie on a crystallographic twofold rotation axis. Chains made up of UO_8 hexagonal bipyramids are connected by centrosymmetric oxalate groups in the crystal structure. The U^{VI} centre is bound axially to two O atoms, forming a nearly linear uranyl unit [O=U=O = 175.0 (2)°] with a U=O bond length of 1.736 (4) Å. Equatorially, the uranyl (UO_2) unit is coordinated by four O atoms from two different oxalate ligands and is bidentate-chelated by two O atoms from an

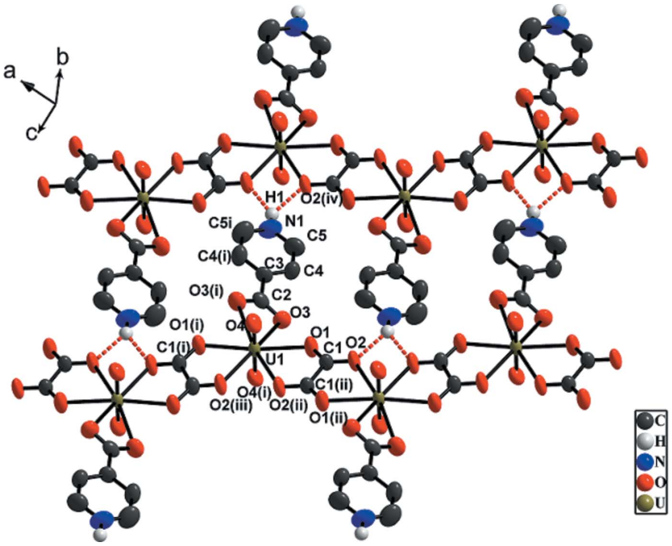


Figure 1
The two-dimensional sheet built from N—H···O hydrogen bonding (dashed lines) between two adjacent chains shown with 80% probability displacement ellipsoids. For clarity, only H atoms involved in hydrogen bonding have been included. [symmetry codes: (i) $-x, y, \frac{1}{2} - z$; (ii) $-\frac{1}{2} - x, \frac{1}{2} - y, -z$; (iii) $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $-x - \frac{1}{2}, -y + \frac{3}{2}, -z$.]

isonicotinic acid ligand to give a hexagonal–bipyramidal geometry as shown in Fig. 1. The equatorial U—O bond lengths range from 2.463 (4) to 2.522 (4) Å (Table 1). The UO_8 hexagonal bipyramids are connected by oxalate groups to form chains in the crystal structure. Bifurcated N···H—O hydrogen-bonding interactions (Table 2) between the isonicotinic acid and oxalate ligands in adjacent chains lead to the formation of two-dimensional sheets (Fig. 1).

Experimental

$UO_2(NO_3)_2\cdot6H_2O$ (0.1720 g, 0.34 mmol), isonicotinic acid (0.0330 g, 0.3 mmol) and oxalic acid (0.0276 g, 0.30 mmol) were mixed with distilled water (5 ml) and ethanol (2 ml). To the solution etheylenediamine (0.033 ml, 0.5 mmol) was added dropwise with quick stirring until completely homogenized. The reaction mixture was sealed in a 23 ml Teflon-lined stainless steel autoclave, heated for 72 h at 423 K and then cooled slowly to room temperature overnight. Needle-like yellow crystals of (I) were obtained.

Crystal data

$[U(C_6H_5NO_2)(C_2O_4)O_2]$
M_r = 481.16
Monoclinic, $C2/c$
a = 10.6836 (12) Å
b = 11.8752 (14) Å
c = 9.0111 (11) Å
β = 109.460 (2)°
V = 1077.9 (2) Å^3
Z = 4
D_x = 2.965 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 15.09 mm^{-1}
T = 298 (2) K
Needle, yellow
0.18 × 0.07 × 0.06 mm

Data collection

Bruker SMART APEX-II CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{\min}$ = 0.172, $T_{\max}$ = 0.465 (expected range = 0.150–0.404)
3946 measured reflections
1314 independent reflections
1075 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.036
$\theta_{\max}$ = 28.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.058$
S = 0.99
1314 reflections
84 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 2.24 e Å^{-3}
$\Delta\rho_{\min}$ = −1.65 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

U1—O4	1.736 (4)	U1—O2$^{\rm i}$	2.497 (4)
U1—O1	2.463 (4)	U1—O3	2.522 (4)
O4—U1—O4$^{\rm ii}$	175.0 (2)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2$^{\rm iii}$	0.86	2.20	2.946 (7)	145

Symmetry code: (iii) $-x - \frac{1}{2}, -y + \frac{3}{2}, -z$.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å, N—H = 0.86 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N). The highest residual electron density peak is located 0.96 Å from atom U1 and the deepest hole 0.72 Å from U1.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *PLATON* (Spek, 2003).

We gratefully acknowledge the financial support of the Natural Science Foundation of JiangXi Province (grant Nos. 0320024 and 0520036). We thank the Key Laboratory of Coordination Chemistry of Jiangxi Province for collecting the crystal data, and Professor Huang Changcang for his assistance in the crystal structure analysis.

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