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Di-μ-aqua-bis[bis(nitrato-*O*,*O'*)dioxouranium(VI)] Bis(2-dimethylaminoethanol)

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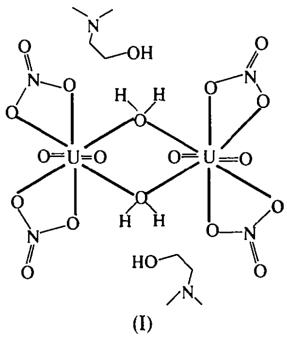
Abstract

Each linear uranyl group in the title compound, $[(UO_2)_2(NO_3)_4(H_2O)_2].2C_4H_{11}NO$, is perpendicular to the equatorial plane formed by six O atoms, of which two belong to the bridging water molecules and four to the bidentate nitrate groups. The coordination polyhedron has a slightly distorted hexagonal bipyramidal arrangement. The non-coordinated 2-dimethylaminoethanol molecules are hydrogen bonded to the bridging water molecules with an O···O distance of 2.77 (3) Å.

Comment

Most uranium complexes have been found to be di-, tetra- or polynuclear. For example, uranyl acetate dihydrate has a dimeric structure with both bridging and terminal acetate groups (Howatson, Grew & Morosin, 1975) while the recently prepared uranium complex including both acetate and 2-dimethylaminoethanol units is a tetramer (Turpeinen, Hämäläinen, Mutikainen & Orama, 1995). In the present dimeric uranyl compound, (I), the amino alcohol molecules act as secondary ligands since they are only hydrogen bonded to the bridging water molecules.



(I)

Two neutral $UO_2(NO_3)_2$ units are bridged by two water O atoms. The bridging angle U(1)—O(2)—U(1*a*) is 112.1 (6)°. The bridging U—O(water) distances are typically about 0.2 Å shorter than the terminal U—O(nitrate) distances, as observed in related compounds (Cragg, Bott & Atwood, 1988; Åberg, 1969). The U···U distance [3.931 (4) Å] and other geometric parameters of the dimer unit are similar to those of related structures (Perry, Ruben, Templeton & Zalkin, 1980; Perrin, 1976; Dalley, Mueller & Simonsen, 1971; Taylor & Mueller, 1965).

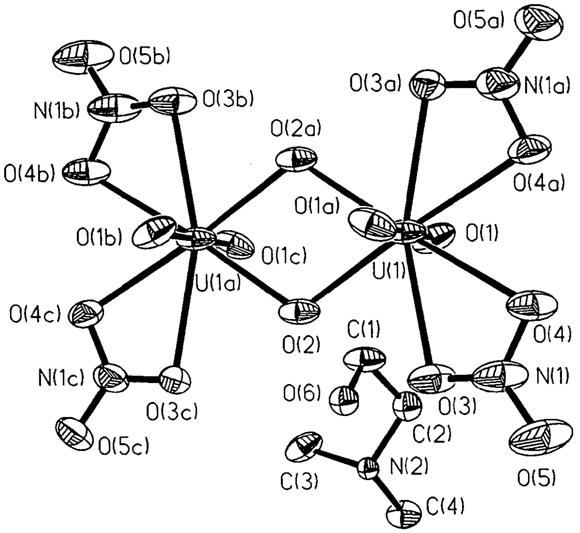


Fig. 1. View of the dinuclear complex and organic molecule with 30% probability ellipsoids. Only one of the two possible positions for each of C(2), C(3) and N(2) is shown.

Experimental

The title compound was prepared by slow evaporation a 1:1 molar ratio of uranyl nitrate hexahydrate and 2-dimethylaminoethanol in methanol solution at room temperature.

Crystal data

$[U_2O_4(NO_3)_4(H_2O)_2]$.-
  $2C_4H_{11}NO$
$M_r = 1002.38$
Monoclinic
$C2/m$
$a = 12.799\,(5)$ Å
$b = 12.969\,(9)$ Å
$c = 7.885\,(4)$ Å
$\beta = 99.01\,(4)°$
$V = 1293\,(1)$ Å^3
$Z = 2$
$D_x = 2.575$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 18 reflections
$\theta = 7–14°$
$\mu = 12.61$ mm^{-1}
$T = 293$ K
Cubiform
0.2 × 0.2 × 0.15 mm
Yellow

Data collection

Nicolet $P3F$ diffractometer
ω scans

$R_{\rm int} = 0.018$
$\theta_{\rm max} = 27°$

Absorption correction:
ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.74$, $T_{\max} = 1.00$
1721 measured reflections
1484 independent reflections
949 observed reflections
$[I \geq 3\sigma(I)]$

$h = 0 \rightarrow 16$
$k = 0 \rightarrow 16$
$l = -10 \rightarrow 9$
3 standard reflections
monitored every 97 reflections
intensity decay: 2%

Refinement

Refinement on F
$R = 0.057$
$wR = 0.062$
$S = 2.04$
949 reflections
77 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F) + 0.0006F^2]$

$(\Delta/\sigma)_{\max} = 0.214$
$\Delta\rho_{\max} = 1.00$ e Å^{-3}
$\Delta\rho_{\min} = -2.73$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
U(1)	0	0.1515 (1)	0	0.075 (1)
O(1)	0.1310 (9)	0.1518 (12)	0.0996 (15)	0.095 (5)
O(2)	−0.0314 (10)	0	0.1518 (16)	0.075 (6)
O(3)	−0.0610 (15)	0.1860 (16)	0.2830 (19)	0.117 (7)
O(4)	−0.0327 (12)	0.3238 (13)	0.1453 (16)	0.110 (7)
O(5)	−0.0997 (19)	0.3348 (18)	0.380 (3)	0.175 (11)
O(6)	0.1043 (11)	0	0.4625 (19)	0.070 (4)
N(1)	−0.0668 (16)	0.281 (3)	0.274 (2)	0.131 (11)
N(2)†	0.2537 (13)	−0.0292 (12)	0.7602 (19)	0.045 (5)
C(1)	0.2141 (17)	0	0.446 (2)	0.090 (11)
C(2)†	0.279 (2)	0.037 (2)	0.617 (3)	0.081 (10)
C(3)†	0.281 (3)	−0.141 (2)	0.727 (4)	0.098 (16)
C(4)	0.3124 (17)	0	0.932 (2)	0.081 (9)

† Occupancy = 0.5.

Table 2. *Selected geometric parameters (Å)*

U(1)—O(1)	1.736 (11)	U(1)—O(2)	2.368 (8)
U(1)—O(3)	2.517 (17)	U(1)—O(4)	2.575 (17)
U(1)···U(1^{i})	3.930 (4)	O(3)—N(1)	1.23 (4)
O(4)—N(1)	1.29 (3)	O(5)—N(1)	1.22 (3)
O(6)—C(1)	1.43 (3)	N(2)—C(2)	1.50 (3)
N(2)—C(3)	1.52 (3)	N(2)—C(4)	1.49 (2)
C(1)—C(2)	1.54 (3)		

Symmetry code: (i) $x, -y, z$.

Data collection and cell refinement were performed using Nicolet *P*3 software (Nicolet XRD Corporation, 1980). The structure was solved by the heavy-atom method and refined using the *SHELXTL-Plus* program system (Sheldrick, 1990). H atoms except those belonging to the alcohol group and the water molecule were introduced in calculated positions and refined with fixed geometry with respect to their carrier atoms. The 2-dimethylaminoalcohol appears to be disordered about the mirror plane through O(6), C(1) and C(4). A population parameter of 0.5 was assigned to atoms C(2), C(3) and N(2).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1283). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Piperazine Metavanadate

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Abstract

The structure of the title compound {piperazinediium poly[trioxovanadate(2−)], $(C_4H_{12}N_2)(VO_3)_2$} is monoclinic, space group $P2_1/c$, and contains infinite $(VO_3^-)_n$ chains running parallel to the c axis. These chains form layers running parallel to the (100) plane. The protonated piperazine molecules are located between the $(VO_3^-)_n$ chains and form hydrogen bonds which contribute to the stabilization of the structure.

Comment

Anhydrous metavanadates typically adopt structures containing infinite chains of corner-sharing VO_4 tetrahedra, as found in the organic cation metavanadates $[(CH_2)_2(NH_3)_2](VO_3)_2$ (Bensch, Hug, Reller & Oswald, 1987), $[(CH_3)(CH_2)_5(NH_3)]VO_3$ (Roman, Aranzabe, Luque & Gutiérrez-Zorrilla, 1991), $[(CH_2)_6(NH_3)_2](VO_3)_2$ (Tyršelová & Pavelčík, 1992) and $[(CH_2)_4(NH_3)_2(NH)](VO_3)_2$ (Roman, Macias, Luque & Gutiérrez-Zorrilla, 1992). Structures containing a V_4O_{12} group based on a ring of four VO_4 tetrahedra have been reported for $[N(C_4H_9)_4]_3[HV_4O_{12}]$