Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71420 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1055]

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Acta Cryst. (1994). C**50**, 52–54

Inclusion Complex Between Uranyl and an Azacrown; Structure of $[UO_2(18\text{-azacrown-}6)]^{2+}.2CF_3SO_3^-$

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(*Received* 3 *February* 1993; *accepted* 28 *June* 1993)

Abstract

The title complex, (1,4,7,10,13,16-hexaazacyclooctadecane)dioxouranium bis(trifluoromethylsulfonate), $[UO_2(C_{12}H_{30}N_6)]^{2+}.2CF_3SO_3^-$, is composed of $[UO_2(18\text{-azacrown-}6)]^{2+}$ and $CF_3SO_3^-$ ions. In the cation, the uranyl is bonded to the six N atoms of the crown in a hexagonal bipyramidal coordination. This is the first reported uranyl hexaazacrown inclusion complex.

Comment

Complexation of the dioxouranium by macrocycles, particularly by crown ethers, has been widely studied (Folcher, Charpin, Costes, Keller & de Villardi, 1979; Fux, Lagrange & Lagrange, 1984; Brighli, Fux, Lagrange & Lagrange, 1985). Of particular interest has been the inclusion of the uranyl ion in macrocycles; however, the majority of structural studies concern complexes in which the crown ligand is linked to the uranyl ion *via* hydrogen bonds (Rogers, Bond, Hipple, Rollins & Henry, 1991; Deshayes, Keller, Lance, Nierlich & Vigner, 1993; Gutberlet, Dreissig, Luger, Bechthold, Maung & Knochel, 1989) and few structural studies show a direct uranyl–crown coordination (Navaza, Villain & Charpin, 1984; Dejean, Charpin, Folcher, Rigny, Navaza & Tsoucaris, 1987; Deshayes, Keller, Lance, Navaza, Nierlich & Vigner, 1993). The probability of the uranyl complexing in the crown cavity is increased by a judicious choice of the solvent–anion pair, which has to be poorly coordinating with respect to the uranyl. As part of our studies of uranyl–macrocycle complexes, we report here the structure of a uranyl–azacrown inclusion complex (18-azacrown-6; 1,4,7,10,13,16-hexaazacyclooctadecane).

As shown in Figs. 1 and 2, the structure consists of $[UO_2(\text{azacrown-}6)]^{2+}$ cations, in which the uranyl group is hexacoordinated by the six N atoms of the azacrown giving a hexagonal bipyramidal geometry about the U atom, and $(CF_3SO_3)^-$ anions. The uranyl bond length is normal [average U—O 1.75 (1) Å] but the O—U—O angle of 174.9 (5)° deviates significantly from linearity. The six N atoms are in a more or less puckered plane; the deviations from the mean least-squares plane are 0.04 (1), −0.24 (1), 0.59 (2), −0.78 (2), 0.52 (2), −0.13 (2) Å for N(1), N(4), N(7), N(10), N(13), N(16), respectively, with the U atom at 0.066 (1) Å from this plane. The average U—N bond length of 2.66 (6) Å is longer than the average U—N distance of 2.54 (4) Å in a uranyl pentaporphyrin complex with five N atoms equatorially bonded to the U atom (Burrel, Hemmi, Lynch & Sessler, 1991), but compares well with the average U—N distance of 2.63 (8) Å in a pyrrole-derived uranyl Shiff base complex where six atoms are equatorially bonded (Sessler, Mody & Lynch, 1992). The average U—N bond length, which is longer than the analogous U—O distances in similar crown ether complexes [2.51 (2) Å (Navaza, Villian & Charpin, 1984) and 2.58 (7) Å (Deshayes, Keller, Lance, Navaza, Nierlich & Vigner, 1993)], indicates the relative weakness of the U—N bond compared with the U—O bond.

The macrocycle adopts a distorted $g-g+g+g-g+g+$ conformation, one of the six sequences which commonly occurs in solid-state 18-crown-6 complexes (Fyles & Gandour, 1992). A distortion in two N—C—C—N units [N(10)—C(11)—C(12)—N(13) −38 (3) and N(13)—C(14)—C(15)—N(16) 54 (2)°] provokes deviations in three C—N—C—C units [C(6)—N(7)—C(8)—C(9) 101 (2), C(11)—N(10)—C(9)—C(8) 69 (3), C(9)—N(10)—C(11)—C(12) −131 (2)°].

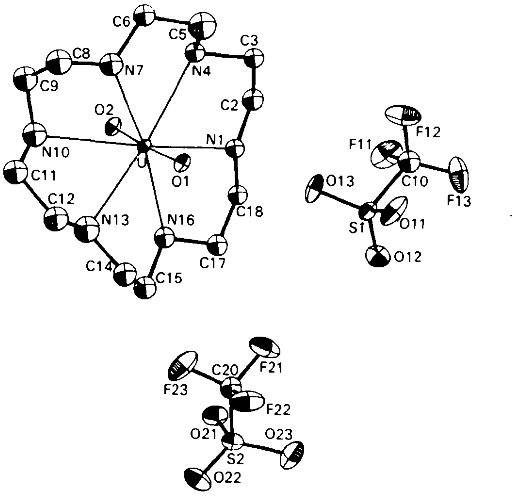

Fig. 1. *ORTEP* (Johnson, 1976) drawing of the $[UO_2(18\text{-aza-crown-6})]^{2+}$ and $(CF_3SO_3)^-$ ions with atom labelling.

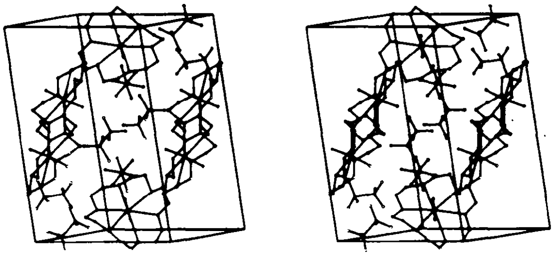

Fig. 2. Stereoview of the crystal packing.

Experimental

Crystal data

$[UO_2(C_{12}H_{30}N_6)]$.-
$2CF_3SO_3$
M_r = 826.58
Monoclinic
$P2_1/n$
a = 13.233 (6) Å
b = 11.551 (9) Å
c = 17.416 (9) Å
β = 96.78 (5)°
V = 2643 (4) Å^3
Z = 4

D_x = 2.077 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–12°
μ = 6.06 mm^{-1}
T = 213 K
Irregular
0.40 × 0.25 × 0.20 mm
Red-brown

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.873, T_{max} = 1.118
2963 measured reflections
2450 independent reflections
1744 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.01
θ_{max} = 20°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 11$
$l = -16 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity variation: −4.9% in 36 h (linearly corrected)

Refinement

Refinement on F
R = 0.034
wR = 0.039
S = 4.3
1744 reflections
234 parameters
H-atom parameters not introduced

Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.685 e Å^{-3}
$\Delta\rho_{min}$ = −0.456 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Data collection: Enraf-Nonius CAD-4 software. Programs used: *MolEN* (Fair, 1990). The structure was solved by the heavy-atom method and refined by full-matrix least-squares procedures with anisotropic thermal parameters for U, O, S and F atoms. All calculations were performed on a VAX4200 computer.

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

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
U	0.12882 (4)	0.11990 (5)	0.39186 (3)	2.94 (1)
S(1)	0.2576 (4)	0.1141 (5)	0.0889 (3)	6.7 (1)
S(2)	0.6858 (4)	0.0455 (5)	0.3169 (3)	6.4 (1)
F(11)	0.110 (1)	−0.025 (1)	0.0571 (9)	11.4 (4)
F(12)	0.0788 (9)	0.155 (1)	0.0241 (9)	11.2 (4)
F(13)	0.167 (1)	0.053 (1)	−0.0434 (7)	11.7 (5)
F(21)	0.535 (1)	0.176 (1)	0.2660 (9)	12.4 (5)
F(22)	0.674 (1)	0.267 (1)	0.2965 (9)	10.7 (4)
F(23)	0.589 (1)	0.203 (1)	0.3831 (8)	12.9 (5)
O(1)	0.1935 (8)	0.2229 (9)	0.3436 (6)	4.8 (3)
O(2)	0.0585 (8)	0.0112 (9)	0.4325 (6)	4.8 (3)
O(11)	0.288 (1)	0.220 (1)	0.0502 (8)	9.0 (4)
O(12)	0.323 (1)	0.016 (1)	0.0848 (9)	8.4 (4)
O(13)	0.220 (1)	0.130 (1)	0.1611 (6)	8.1 (3)
O(21)	0.612 (1)	−0.036 (1)	0.3391 (9)	8.1 (4)
O(22)	0.774 (1)	0.060 (1)	0.3726 (8)	7.8 (4)
O(23)	0.708 (1)	0.037 (1)	0.2380 (8)	9.5 (4)
N(1)	0.105 (1)	0.008 (1)	0.2619 (9)	6.4 (4)
N(4)	−0.042 (1)	0.160 (1)	0.3042 (9)	6.6 (4)
N(7)	0.027 (1)	0.314 (2)	0.422 (1)	8.8 (5)
N(10)	0.119 (1)	0.179 (2)	0.544 (1)	8.4 (5)
N(13)	0.291 (1)	0.123 (2)	0.495 (1)	10.1 (5)
N(16)	0.264 (1)	−0.050 (1)	0.3797 (9)	6.6 (4)
C(2)	0.002 (2)	−0.011 (2)	0.231 (1)	7.4 (5)
C(3)	−0.046 (1)	0.104 (2)	0.223 (1)	6.8 (5)
C(5)	−0.069 (2)	0.279 (2)	0.294 (1)	10.0 (7)
C(6)	−0.075 (2)	0.320 (2)	0.374 (1)	7.9 (6)
C(8)	0.029 (2)	0.347 (2)	0.501 (1)	9.4 (7)
C(9)	0.030 (2)	0.246 (2)	0.548 (1)	9.4 (7)
C(10)	0.151 (2)	0.075 (2)	0.025 (1)	8.2 (6)
C(11)	0.209 (2)	0.233 (2)	0.580 (1)	10.4 (7)

C(12)	0.293 (2)	0.234 (2)	0.535 (1)	8.9 (6)
C(14)	0.376 (2)	0.109 (2)	0.447 (1)	9.8 (6)
C(15)	0.371 (2)	−0.024 (2)	0.419 (1)	7.8 (6)
C(17)	0.273 (2)	−0.087 (2)	0.302 (1)	7.5 (5)
C(18)	0.164 (1)	−0.110 (2)	0.265 (1)	6.8 (5)
C(20)	0.618 (2)	0.178 (2)	0.314 (1)	7.6 (5)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

U environment			
U—O(1)	1.74 (1)	U—O(2)	1.76 (1)
U—N(1)	2.59 (2)	U—N(4)	2.61 (2)
U—N(7)	2.70 (2)	U—N(10)	2.76 (2)
U—N(13)	2.63 (2)	U—N(16)	2.68 (2)
O(1)—U—O(2)	174.9 (5)	O(1)—U—N(1)	86.2 (5)
N(1)—U—N(4)	64.7 (5)	N(1)—U—N(16)	65.0 (5)
N(4)—U—N(7)	63.4 (6)	N(7)—U—N(10)	61.7 (6)
N(10)—U—N(13)	57.7 (6)	N(13)—U—N(16)	64.3 (6)
Triflate			
S(1)—O(11)	1.48 (1)	S(2)—O(21)	1.44 (1)
S(1)—O(12)	1.44 (1)	S(2)—O(22)	1.44 (1)
S(1)—O(13)	1.42 (2)	S(2)—O(23)	1.44 (1)
S(1)—C(10)	1.74 (3)	S(2)—C(20)	1.77 (2)
C(10)—F(11)	1.42 (3)	C(20)—F(21)	1.30 (3)
C(10)—F(12)	1.33 (3)	C(20)—F(22)	1.33 (2)
C(10)—F(13)	1.27 (3)	C(20)—F(23)	1.34 (3)
O(11)—S(1)—O(12)	115.6 (8)	O(21)—S(2)—O(22)	114.8 (9)
O(11)—S(1)—O(13)	116.1 (9)	O(21)—S(2)—O(23)	115 (1)
O(11)—S(1)—C(10)	100 (1)	O(21)—S(2)—C(20)	102 (1)
O(12)—S(1)—O(13)	115 (1)	O(22)—S(2)—O(23)	114 (1)
O(12)—S(1)—C(10)	102 (1)	O(22)—S(2)—C(20)	107 (1)
O(13)—S(1)—C(10)	105 (1)	O(23)—S(2)—C(20)	101 (1)
S(1)—C(10)—F(11)	106 (2)	S(2)—C(20)—F(21)	113 (2)
S(1)—C(10)—F(12)	111 (2)	S(2)—C(20)—F(22)	113 (2)
S(1)—C(10)—F(13)	116 (2)	S(2)—C(20)—F(23)	111 (2)
F(11)—C(10)—F(12)	105 (2)	F(21)—C(20)—F(22)	109 (2)
F(11)—C(10)—F(13)	108 (2)	F(21)—C(20)—F(23)	106 (2)
F(12)—C(10)—F(13)	109 (2)	F(22)—C(20)—F(23)	105 (2)
Hexaaza-18-crown-6			
N(1)—C(2)	1.43 (2)	N(1)—C(18)	1.56 (2)
N(4)—C(3)	1.56 (2)	N(4)—C(5)	1.43 (3)
N(7)—C(6)	1.52 (3)	N(7)—C(8)	1.43 (3)
N(10)—C(9)	1.41 (3)	N(10)—C(11)	1.42 (3)
N(13)—C(12)	1.45 (3)	N(13)—C(14)	1.49 (3)
N(16)—C(15)	1.53 (3)	N(16)—C(17)	1.43 (2)
C(2)—C(3)	1.47 (3)	C(5)—C(6)	1.48 (3)
C(8)—C(9)	1.42 (3)	C(11)—C(12)	1.43 (3)
C(14)—C(15)	1.62 (3)	C(17)—C(18)	1.54 (3)
C(2)—N(1)—C(18)	109 (2)	C(3)—N(4)—C(5)	108 (2)
C(6)—N(7)—C(8)	116 (2)	C(9)—N(10)—C(11)	114 (2)
C(12)—N(13)—C(14)	113 (2)	C(15)—N(16)—C(17)	108 (2)
N(1)—C(2)—C(3)	106 (2)	C(2)—C(3)—N(4)	109 (2)
N(4)—C(5)—C(6)	104 (2)	C(5)—C(6)—N(7)	111 (2)
N(7)—C(8)—C(9)	110 (2)	C(8)—C(9)—N(10)	112 (2)
N(10)—C(11)—C(12)	115 (2)	C(11)—C(12)—N(13)	106 (2)
N(13)—C(14)—C(15)	105 (2)	C(14)—C(15)—N(16)	109 (2)
N(16)—C(17)—C(18)	106 (2)	C(17)—C(18)—N(1)	108 (2)

The complex was prepared as described in a previous paper (Folcher, Charpin, Costes, Keller & de Villardi, 1979). An excess of UO_3 was added to bring the pH within the range 4-5 and undissolved oxide was removed by filtration. The water was evaporated under vacuum and the resulting solid was redissolved in acetonitrile; crystals suitable for X-ray analysis were obtained by slow cooling.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71447 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1044]

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Acta Cryst. (1994). C**50**, 54–56

Molecular Adduct of Dimethyltin Dichloride with Methyl *N*-(2-Hydroxybenzylidene)-alaninate (1:2), $[SnCl_2(C_{11}H_{13}NO_3)_2(CH_3)_2]$

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(*Received* 19 *February* 1993; *accepted* 7 *June* 1993)

Abstract

The structure of the 2:1 complex of methyl *N*-(2-hydroxybenzylidene)alaninate with dimethyltin dichloride, dichlorobis(2-{[1-(methoxycarbonyl)-ethyl]iminiomethyl}phenolato-*O*)dimethyltin, has been determined. The coordination of the Sn atom is octahedral, all *trans*, with bond distances Sn—Cl = 2.599 (1), Sn—O = 2.244 (3) and Sn—C = 2.111 (4) Å. The ligands are coordinated to Sn