C10—Cr—C13	77.7 (1)	C10′—Cr′—C13′	77.8 (1)
C10—Cr—C12	66.1 (1)	C10′—Cr′—C12′	66.4 (1)
C10—Cr—C11	36.8 (1)	C10′—Cr′—C11′	36.7 (1)
C9—Cr—C13	66.3 (1)	C9′—Cr′—C13′	66.2 (1)
C9—Cr—C12	78.6 (1)	C9′—Cr′—C12′	78.8 (1)
C9—Cr—C11	66.7 (1)	C9′—Cr′—C11′	66.5 (1)
C9—Cr—C10	37.0 (1)	C9′—Cr′—C10′	37.0 (1)
C8—Cr—C13	37.0 (1)	C8′—Cr′—C13′	37.0 (1)
C8—Cr—C12	66.3 (1)	C8′—Cr′—C12′	66.6 (1)
C8—Cr—C11	78.2 (1)	C8′—Cr′—C11′	78.1 (1)
C8—Cr—C10	65.9 (1)	C8′—Cr′—C10′	66.1 (1)
C8—Cr—C9	36.6 (1)	C8′—Cr′—C9′	36.5 (1)
C3—Cr—C13	98.1 (1)	C3′—Cr′—C13′	98.2 (1)
C3—Cr—C12	129.7 (1)	C3′—Cr′—C12′	129.2 (1)
C3—Cr—C11	163.7 (1)	C3′—Cr′—C11′	163.8 (1)
C3—Cr—C10	140.9 (1)	C3′—Cr′—C10′	142.3 (1)
C3—Cr—C9	105.4 (1)	C3′—Cr′—C9′	106.8 (1)
C3—Cr—C8	87.2 (1)	C3′—Cr′—C8′	88.0 (1)
C2—Cr—C13	163.0 (1)	C2′—Cr′—C13′	163.3 (1)
C2—Cr—C12	142.9 (1)	C2′—Cr′—C12′	142.8 (1)
C2—Cr—C11	107.4 (1)	C2′—Cr′—C11′	107.5 (1)
C2—Cr—C10	87.9 (1)	C2′—Cr′—C10′	88.0 (1)
C2—Cr—C9	96.8 (1)	C2′—Cr′—C9′	97.2 (1)
C2—Cr—C8	128.1 (1)	C2′—Cr′—C8′	128.4 (1)
C2—Cr—C3	87.3 (1)	C2′—Cr′—C3′	87.7 (1)
C1—Cr—C13	107.4 (1)	C1′—Cr′—C13′	107.7 (1)
C1—Cr—C12	88.0 (1)	C1′—Cr′—C12′	87.7 (1)
C1—Cr—C11	97.2 (1)	C1′—Cr′—C11′	97.2 (1)
C1—Cr—C10	128.6 (1)	C1′—Cr′—C10′	128.4 (1)
C1—Cr—C9	163.8 (1)	C1′—Cr′—C9′	163.6 (1)
C1—Cr—C8	143.0 (1)	C1′—Cr′—C8′	143.3 (1)
C1—Cr—C3	90.1 (1)	C1′—Cr′—C3′	88.8 (1)
C1—Cr—C2	88.6 (1)	C1′—Cr′—C2′	88.0 (1)
Cr—C1—O1	179.0 (3)	Cr′—C1′—O1′	179.3 (3)
Cr—C2—O2	178.3 (3)	Cr′—C2′—O2′	178.4 (3)
Cr—C3—O3	177.5 (3)	Cr′—C3′—O3′	179.1 (3)
O4—C4—C9	110.7 (2)	O4′—C4′—C9′	110.9 (2)
O4—C4—C5	110.8 (2)	O4′—C4′—C5′	111.0 (2)
C5—C4—C9	111.1 (2)	C5′—C4′—C9′	111.0 (2)
C4—C5—C6	108.6 (3)	C4′—C5′—C6′	109.0 (3)
C5—C6—C7	112.1 (3)	C5′—C6′—C7′	111.3 (3)
C6—C7—C8	114.1 (3)	C6′—C7′—C8′	114.1 (3)
Cr—C8—C7	130.1 (2)	Cr′—C8′—C7′	130.1 (2)
C7—C8—C13	119.4 (3)	C7′—C8′—C13′	119.5 (3)
C7—C8—C9	121.6 (3)	C7′—C8′—C9′	121.6 (3)
Cr—C8—C13	70.0 (2)	Cr′—C8′—C13′	70.0 (2)
Cr—C8—C9	71.3 (2)	Cr′—C8′—C9′	71.7 (2)
C4—C9—C8	120.8 (3)	C4′—C9′—C8′	121.0 (2)
Cr—C9—C8	72.1 (2)	Cr′—C9′—C8′	71.8 (2)
Cr—C9—C4	131.0 (2)	Cr′—C9′—C4′	130.6 (2)
C4—C9—C10	120.8 (2)	C4′—C9′—C10′	120.4 (2)
Cr—C9—C10	69.8 (2)	Cr′—C9′—C10′	69.8 (2)
Cr—C10—C9	73.2 (2)	Cr′—C10′—C9′	73.2 (2)
Cr—C10—C11	72.4 (2)	Cr′—C10′—C11′	72.2 (2)
Cr—C11—C10	70.9 (2)	Cr′—C11′—C10′	71.1 (2)
Cr—C11—C12	71.0 (2)	Cr′—C11′—C12′	70.8 (2)
Cr—C12—C11	72.2 (2)	Cr′—C12′—C11′	72.3 (2)
Cr—C12—C13	72.2 (2)	Cr′—C12′—C13′	72.2 (2)
Cr—C13—C12	71.4 (2)	Cr′—C13′—C12′	71.2 (2)
Cr—C13—C8	73.0 (2)	Cr′—C13′—C8′	72.9 (2)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *SDP* (B. A. Frenz & Associates Inc., 1985). Program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *MolEN* (Fair, 1990). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1200). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

B. A. Frenz & Associates Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

Davies, S. G. & Goodfellow, C. L. (1988). *J. Organomet. Chem.* **340**, 195–201.

Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.

Jaouen, G. & Meyer, A. (1975). *J. Am. Chem. Soc.* **97**, 4667–4672.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.

Muetterties, E. L., Bleeke, J. R., Wucherer, E. J. & Albright, T. A. (1982). *Chem. Rev.* **82**, 499–525.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Schmalz, H. G., Millies, B., Bats, J. W. & Dürner, G. (1992). *Angew. Chem. Int. Ed. Engl.* **31**, 631–633.

Solladie-Cavallo, A. (1989). *Advances in Metalorganic Chemistry*, Vol. 1, 99–133. London: JAI Press.

Acta Cryst. (1996). C**52**, 615–617

Diaquadioxobis(pyridine-3-carboxylato)-uranium(VI)

NATHANIEL W. ALCOCK,[a] WILLIAM ERRINGTON,[a] TERENCE J. KEMP[a] AND JANUSZ LECEIEJEWICZ[b]

[a]*Department of Chemistry, University of Warwick, Coventry CV4 7AL, England, and* [b]*Institute of Nuclear Chemistry and Technology, Dorodna 16, Warsaw, Poland. E-mail: msrbb@csv.warwick.ac.uk*

(*Received 30 August 1995; accepted 18 September 1995*)

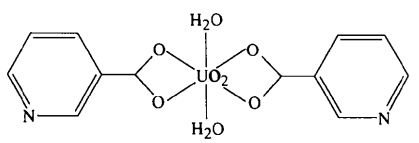
Abstract

The uranyl group in the title compound, $[UO_2(C_6H_4NO_2)_2(H_2O)_2]$, with U—O 1.782 (4) Å, is sixfold coordinated by two water molecules [U—O 2.431 (4) Å] and two bidentate nicotinic acid anions (pyridine-3-carboxylato ions) [U—O 2.459 (4)–2.486 (4) Å]. Individual molecules are linked into chains by hydrogen bonds between coordinated water molecules and pyridine N atoms.

Comment

Coordination of amino acids to uranyl ions takes place exclusively through the carboxylate function (Alcock, Flanders, Kemp & Shand, 1985; Bismondo, Casellato,

Sitran & Graziani, 1985). U—N bonding is found only when the N atom is in an aromatic ring, as in the complexes with dipicolinic acid (pyridine-2,6-dicarboxylic acid) (Immirzi, Bombieri, Degetto & Marangoni, 1975; Cousson, Proust & Rizkalla, 1991; Cousson, Nectoux, Pages & Rizkalla, 1993); in these complexes, the uranyl group is coordinated by a slightly distorted hexagon comprising four O and two N atoms of the dipicolinate ligands. We have also found U—N bonding in a uranyl complex with the 2-pyrazine carboxylate ion, in which the uranyl group is fivefold coordinated by two N atoms, two O atoms from the ligand and one water O atom (Alcock, Kemp & Leciejewicz, 1996). As part of our continuing study of uranyl coordination by N and O donors (Leciejewicz, Alcock & Kemp, 1995), we now report the structure of the complex, (I), with nicotinic acid (pyridine-3-carboxylic acid).



(I)

The uranyl group is located on an inversion centre, and is only coordinated by O atoms, four from the anion and two from water molecules. The geometry is identical to that of uranyl dinitrate dihydrate, (II) (Dalley, Mueller & Simonsen, 1971), as are the U—O distances; in both, U—O(water) is significantly shorter [2.431 (4) in (I); 2.450 (4) Å in (II)] than U—O(anion) [2.46–2.49 in (I); 2.48–2.51 Å in (II)]. The packing is dominated by pairs of hydrogen bonds between water H atoms and the pyridine N atoms of adjacent molecules, linking the complexes in chains along 011 (Fig. 2).

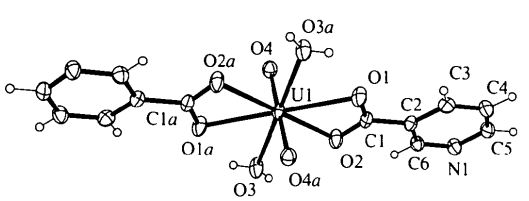


Fig. 1. View of the molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

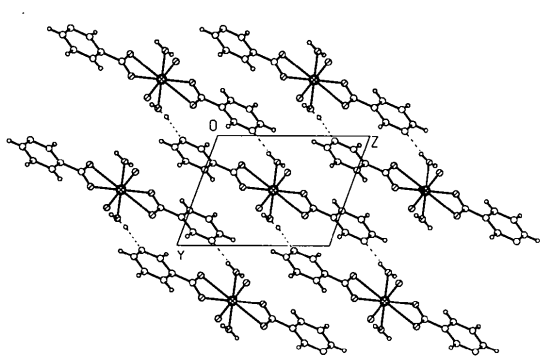


Fig. 2. Packing diagram viewed down the *a* axis, showing the hydrogen bonding.

Experimental

Crystals of the solid complex were obtained by evaporation of a 2:1 molar solution of uranyl nitrate and nicotinic acid in water. The density D_m was measured by flotation in bromoform/ethyl bromide.

Crystal data

$[UO_2(C_6H_4NO_2)_2(H_2O)_2]$
$M_r = 550.27$
Triclinic
$P\bar{1}$
$a = 5.645\ (4)$ Å
$b = 7.286\ (5)$ Å
$c = 9.784\ (7)$ Å
$\alpha = 106.33\ (6)°$
$\beta = 106.03\ (6)°$
$\gamma = 99.63\ (5)°$
$V = 357.8\ (4)$ Å^3
$Z = 1$
$D_x = 2.554$ Mg m^{-3}
$D_m = 2.50\ (2)$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 29 reflections
$\theta = 10–14°$
$\mu = 11.386$ mm^{-1}
$T = 210\ (2)$ K
Block
0.50 × 0.36 × 0.29 mm
Yellow–brown

Data collection

Siemens *R3m* four-circle diffractometer
Profile-fitted $2\theta/\omega$ scans
Absorption correction: analytical
$T_{min} = 0.26$, $T_{max} = 0.34$
1404 measured reflections
1270 independent reflections
1265 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0261$
$\theta_{max} = 25.03°$
$h = 0 \rightarrow 6$
$k = -8 \rightarrow 8$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 200 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0197$
$wR(F^2) = 0.0572$
$S = 1.098$
1265 reflections
114 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0460P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.160$ e Å^{-3}
$\Delta\rho_{min} = -1.659$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.040 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	*y*	*z*	U_{eq}
U1	1/2	1/2	1/2	0.01766 (15)
O1	0.6727 (7)	0.4721 (6)	0.2923 (4)	0.0283 (8)
O2	0.2963 (7)	0.2728 (6)	0.2324 (4)	0.0277 (8)
O3	0.1236 (7)	0.2428 (6)	0.4552 (4)	0.0290 (8)
O4	0.3158 (7)	0.6625 (5)	0.4503 (4)	0.0246 (7)
N1	0.2515 (8)	0.0447 (6)	−0.2179 (5)	0.0251 (8)
C1	0.4825 (9)	0.3378 (7)	0.1959 (5)	0.0216 (9)
C2	0.4797 (9)	0.2553 (7)	0.0377 (5)	0.0199 (9)
C3	0.6932 (9)	0.3145 (7)	−0.0002 (6)	0.0238 (9)
C4	0.6804 (10)	0.2377 (8)	−0.1477 (6)	0.0257 (10)
C5	0.4557 (11)	0.1041 (8)	−0.2530 (6)	0.0262 (10)
C6	0.2649 (9)	0.1217 (7)	−0.0739 (6)	0.0224 (9)

Table 2. *Selected geometric parameters (Å, °)*

U1—O4	1.782 (4)	U1—O2	2.486 (4)
U1—O3	2.431 (4)	O1—C1	1.253 (7)
U1—O1	2.459 (4)	O2—C1	1.262 (7)
O3—U1—O1^{i}	62.76 (14)	O1—U1—O2	52.39 (13)
O3—U1—O2	65.73 (14)		

Symmetry code: (i) $1-x, 1-y, 1-z$.

The temperature of the crystal was controlled using an Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). The H atoms attached to the coordinated water molecule were located from difference Fourier syntheses and their coordinates were refined. Others were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; each H atom was given an isotropic displacement parameter equal to 1.2 times the equivalent isotropic displacement parameter of the atom to which it is attached. The only significant difference density peaks were located close to the U atom and are considered to be diffraction ripples.

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1314). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alcock, N. W., Flanders, D. J., Kemp, T. J. & Shand, M. A. (1985). *J. Chem. Soc. Dalton Trans.* pp. 517–521.

Alcock, N. W., Kemp, T. J. & Leciejewicz, J. (1996). *Inorg. Chim. Acta.* In the press.

Bismondo, A., Casellato, U., Sitran. S. & Graziani. R. (1985). *Inorg. Chim. Acta*, **110**, 205–210.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Cousson, A., Nectoux, F., Pages, M. & Rizkalla, E. N. (1993). *Radiochim. Acta*, **61**, 177–180.

Cousson, A., Proust, P. & Rizkalla, E. N. (1991). *Acta Cryst.* C**47**, 2065–2069

Dalley, N. K., Mueller, M. H. & Simonsen, S. H. (1971). *Inorg. Chem.* **10**, 323–328.

Immirzi, A., Bombieri, G., Degetto, S. & Marangoni, G. (1975). *Acta Cryst.* B**31**, 1023–1028.

Leciejewicz, J., Alcock, N. W. & Kemp, T. J. (1995). *Struct. Bonding*, **82**, 44–83.

Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* University of Göttingen, Germany.

Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement.* University of Göttingen, Germany.

Acta Cryst. (1996). C**52**, 617–619

Tetrakis(triethylammonium) Hexakis(isothiocyanato-*N*)nickel(II)

PAUL E. KRUGER AND VICKIE MCKEE*

School of Chemistry, Queen's University, Belfast BT9 5AG, Northern Ireland. E-mail: v.mckee@qub.ac.uk

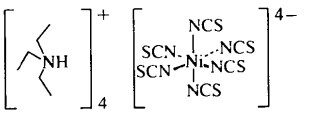
(Received 31 July 1995; accepted 9 October 1995)

Abstract

The unit cell of the title compound, $(C_6H_{16}N)_4[Ni(NCS)_6]$, contains two independent centrosymmetric $[Ni(NCS)_6]^{4-}$ anions and eight Et_3NH^+ cations. The Ni—N distances are in the range 2.072 (4)–2.097 (4) Å. In one anion, all six S atoms are hydrogen bonded to cations, but the second anion is involved in only two hydrogen bonds. There are no significant geometrical differences between the two anions.

Comment

Tetrakis(triethylammonium) hexakis(isothiocyanato-*N*)-nickel(II) is a potentially useful source of anhydrous nickel thiocyanate. Air-stable crystals of $(Et_3NH)_4[Ni(NCS)_6]$, (I), were readily obtained from water.



(I)

The unit cell of (I) contains two independent centrosymmetric $[Ni(NCS)_6]^{4-}$ anions and eight Et_3NH^+ cations. Fig. 1 shows the environments of the two complex anions about the Ni1 and Ni2 atoms. The anion containing Ni1 displays hydrogen bonding from all six S atoms to triethylammonium cations, whereas the second anion shows only two hydrogen bonds (details are given in Table 3). The geometry at each Ni atom is regular octahedral and all the thiocyanate groups are essentially linear [S—C—N 178.0 (4)–179.2 (5)°]. There is some variation, however, in the Ni—N—C angles,

ISSN 0108-2701