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The Structure of Tetrakis(acetonitrile)tetrachlorouranium(IV), $[UCl_4(C_2H_3N)_4]$

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Abstract. $M_r = 544\cdot05$, monoclinic, $C2/c$, $a = 14\cdot709\,(4)$, $b = 8\cdot459\,(3)$, $c = 13\cdot938\,(4)$ Å, $\beta = 91\cdot79\,(2)^\circ$, $V = 1733\cdot2\,(9)$ Å^3, $Z = 4$, $D_x = 2\cdot085$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 94\cdot919$ cm^{-1}, $F(000) = 992\cdot0$, $T = 278$ K, $R = 0\cdot040$ for 1051 ($I \geq 3\sigma$) reflections. The molecules are discrete monomeric units with eight ligands coordinated in an almost ideal dodecahedral arrangement. The four nitrogen atoms form an elongated tetrahedron and the four chlorine atoms a flattened tetrahedron. Important bond distances are: U–Cl(1) $2\cdot627\,(4)$; U–Cl(2) $2\cdot620\,(4)$; U–N(1) $2\cdot601\,(13)$; U–N(2) $2\cdot577\,(14)$ Å.

Introduction. Because of their large radii, actinide-metal ions have been observed in complexes in which the coordination number has been as large as 14. Of the many possible geometries for an eight-coordinate species, the dodecahedron and the square antiprism are the most common forms (Hoard & Silverton, 1963; Blight & Kepert, 1968; Kepert, 1965). Four less frequently encountered geometries are the hexagonal bipyramid, the puckered hexagonal bipyramid, the bicapped trigonal prism and the cube.

The configurations of most of the eight-coordinate compounds of uranium are based on the dodecahedron or the square antiprism, but there are very few compounds in which the geometry is precisely either of the ideal models. Rather, intermediate forms between the two models are commonly encountered. We report here that $UCl_4(CH_3CN)_4$ very closely approximates the idealized geometry of a dodecahedron.

Experimental. In an effort to form a metal–metal-bonded uranium trimer of the type $[M_3(C_6Me_6)Cl_6]^{n+}$, analogous to those of niobium and zirconium (Goldberg, Spivack, Stanley, Eisenberg, Braitsch, Miller & Abkowitz, 1977; Stollmaier & Thewalt, 1981), a mixture of UCl_4 (760 mg), Al (42 mg), $AlCl_3$ (194 mg) and C_6Me_6 (1 g) was heated in a sealed glass tube for 4 h at 413 K. The reaction product was first extracted

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Table 1. *Positional parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $\frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$.

	x	y	z	B(Å^2)
U	0·000	0·18155 (9)	0·250	3·00 (1)
Cl(1)	0·3484 (2)	0·4072 (4)	0·6611 (3)	4·94 (8)
Cl(2)	0·5776 (2)	0·2285 (4)	0·5924 (3)	4·66 (8)
C(1)	0·6310 (9)	0·036 (2)	0·330 (1)	4·4 (3)
N(1)	0·4133 (7)	0·063 (1)	0·6971 (8)	4·4 (3)
C(2)	0·687 (1)	0·171 (2)	0·365 (1)	6·5 (4)
N(2)	0·4534 (8)	0·428 (1)	0·3389 (8)	4·6 (3)
C(3)	0·4231 (9)	0·323 (2)	0·380 (1)	4·3 (3)
C(4)	0·384 (1)	0·192 (2)	0·429 (1)	4·9 (3)

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

U–Cl(1)	2·627 (4)	C(1)–N(1)	1·12 (2)
–Cl(2)	2·620 (4)	–C(2)	1·48 (2)
–N(1)	2·601 (13)	C(3)–N(2)	1·15 (2)
–N(2)	2·577 (14)	–C(4)	1·43 (2)
Cl(1)–U–Cl(1′)	146·8 (2)	Cl(2)–U–N(2)	140·7 (3)
–Cl(2)	94·2 (1)	–N(2′)	73·1 (3)
–Cl(2′)	95·3 (1)	N(1)–U–N(1′)	67·9 (5)
Cl(1)–U–N(1)	72·6 (3)	–N(2)	133·5 (4)
–N(1′)	140·6 (3)	–N(2′)	133·6 (4)
Cl(1)–U–N(2)	76·7 (3)	N(2)–U–N(2′)	67·7 (6)
–N(2′)	75·8 (3)	N(1)–C(1)–C(2)	178·(2)
Cl(2)–U–Cl(2′)	146·2 (2)	N(2)–C(3)–C(4)	179·(2)
Cl(2)–U–N(1)	75·2 (3)		
–N(1′)	76·9 (3)		

with hexane to remove any unreacted C_6Me_6. A second extraction with CH_2Cl_2 yielded a small amount of an as yet uncharacterized red material. A final extraction with CH_3CN gave a green solution from which X-ray quality crystals of the title complex grew at room temperature within 24 h. We presume that the compound can be prepared in a more deliberate and simpler way, but have not endeavoured to do so. The compound is not recorded in the literature, to the best of our knowledge.

Crystal 0·3 x 0·3 x 0·4 mm mounted on Syntex $P\bar{1}$ diffractometer. Graphite-monochromated Mo $K\alpha$ radiation; unit-cell dimensions from exact goniometer settings of 15 reflections, $15 \leq 2\theta \leq 28°$; systematic absences $hkl, h + k \neq 2n$, and $h0l, l \neq 2n$, indicated Cc or $C2/c$; centrosymmetric choice confirmed by successful refinement; 1314 unique reflections, $4 \leq 2\theta \leq 50°$; 1051 with $I \geq 3\sigma$, $0 \leq h \leq 17$, $0 \leq k \leq 11$, $-17 \leq l \leq 17$; 3 intensity standards checked every 100 reflections showed 13·43% decay over 27·4 h of X-ray exposure; empirical absorption correction using ψ scans for each of 9 reflections with Eulerian angle near 90°, each reflection measured at 10° intervals from 0 to 360°; normalized transmission factors from 1·00 to 0·68; position of metal atom derived from three-dimensional Patterson map, remaining atoms found by iterative application of least-squares refinement and difference Fourier maps; $F_{\rm obs}$, corrected for absorption, Lorentz and polarization, used for refinement of positions, anisotropic temperature factors and scale factor; $R = 0·040$, $R_w = 0·049$, $w = 1/\sigma^2(|F_o|)$, $S = 0·92$, $\Delta_{\rm max}/\sigma = 0·09$, max. $\Delta\rho$ excursion in final difference map 0·612 e Å^{-3}; atomic scattering factors and f', f'' values from *International Tables for X-ray Crystallography*. All calculations performed with the Enraf–Nonius (1981) *Structure Determination Package* on the VAX 11/80 computer at the Department of Chemistry, Texas A&M University, College Station, TX 77843.

Discussion. The positional parameters and equivalent isotropic thermal parameters are given in Table 1, while selected interatomic distances and angles are listed in Table 2. Fig. 1 depicts the molecule and defines the atomic labelling scheme. The unit-cell packing is shown in Fig. 2.*

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* Lists of structure factors, anisotropic thermal parameters, least-squares planes, and non-essential bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39365 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

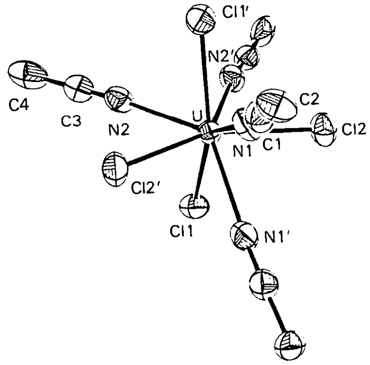

Fig. 1. A computer-drawn view of the molecule in which atoms are represented by their ellipsoids of thermal vibration drawn at 40% probability level.

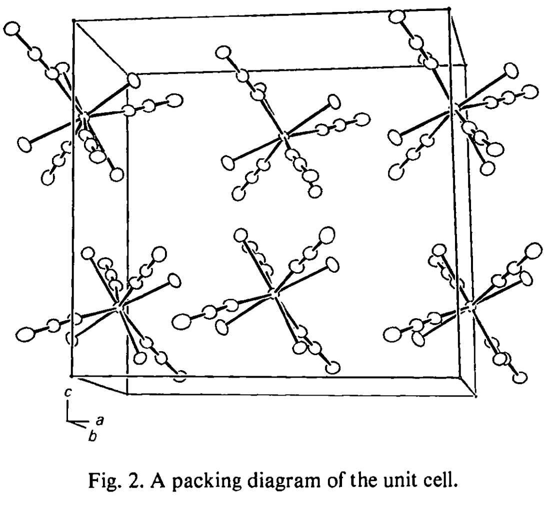

Fig. 2. A packing diagram of the unit cell.

In the molecule, there are two separate trapezoids (not precisely planar), one defined by atoms N(1), N(1′), Cl(1), Cl(1′) [dihedral angle 1·7 (5)°] and the other defined by N(2), N(2′), Cl(2), Cl(2′) [dihedral angle 1·6 (8)°]. The two best planes are essentially orthogonal to each other [90·5 (9)°]. The four nitrogen atoms form an elongated tetrahedron, while the four chlorine atoms form a flattened tetrahedron. This is as would be expected since the most crowded positions of the polyhedra are at the corners of the elongated tetrahedron and therefore are more suitable for the smaller nitrogen atoms. The average angle between the crystallographically imposed twofold axis and the uranium–nitrogen bonds is 33·9 [1]° while the average angle between the axis and the uranium–chloride bonds is 106·8 [2]°. These structural values place the $UCl_4(CH_3CN)_4$ structure near that which has been calculated for the minimum repulsion energy of a dodecahedron (Kepert, 1978). The structure of this complex comes very close to being an ideal dodecahedron.

We thank the Robert A. Welch Foundation for support.

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Structure of (+)-(7*S*,11*S*)-11-Hydroxy-10-methylbicyclo[5.3.1]dec-1(10)-en-7-ylmethyl (+)-10-Camphorsulfonate,* $C_{23}H_{36}O_5S$

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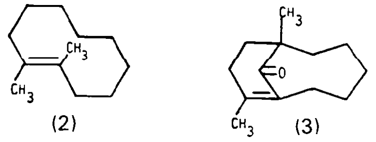
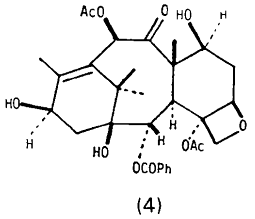
Abstract. $M_r = 424{\cdot}6$, tetragonal, $P4_1$, $a = 9{\cdot}833$ (4), $c = 23{\cdot}46$ (1) Å, $V = 2267$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}24$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}64$ cm^{-1}, $F(000) = 920$, $T = 291$ K, final $R = 0{\cdot}065$ for 1168 observed reflections. The eight-carbon ring is in a conformation corresponding to that of minimum energy for cyclooctane with an approximate D_2 symmetry. The best planes through it and the condensed cyclohexene form an angle of 101·7 (6)°.

Introduction. In connection with studies on optically active *trans*-cycloalkenes, we recently resolved racemic 7-hydroxymethyl-10-methylbicyclo[5.3.1]dec-1(10)-en-11-ol *via* the (+)-10-camphorsulfonate (1) and converted the highly crystalline (+)-diastereomer to (−)-*trans*-1,2-dimethylcyclodecene (2) by a six-step sequence (Marshall, Konicek & Flynn, 1980). Our assignment of configuration of (+)-(1) and (−)-(2) was based upon the optical rotatory dispersion curve analysis of enone (3),





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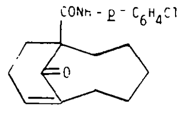

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* 10-Camphorsulfonic acid is 7,7-dimethyl-2-oxobicyclo[2.2.1]-hept-1-ylmethanesulfonic acid.

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