$\omega/2\theta$ scans
Absorption correction:
ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.521$, $T_{max} = 0.575$
3391 measured reflections
3241 independent reflections

$R_{int} = 0.007$
$\theta_{max} = 25°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 11$
$l = -18 \rightarrow 18$
3 standard reflections every 300 reflections intensity decay: 1.1%

Refinement

Refinement on F
$R = 0.040$
$wR = 0.048$
$S = 1.40$
2176 reflections
291 parameters
H-atom parameters refined
$w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{max} = 0.08$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}
Extinction coefficient: 3.57×10^{-6}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Ga—N(2)	2.173 (3)	N(1)—C(12)	1.396 (4)
Ga—C(16)	1.973 (5)	N(1)—C(13)	1.447 (4)
Ga—C(17)	1.972 (5)	N(2)—C(13)	1.489 (4)
Ga—C(18)	1.968 (5)	N(2)—C(14)	1.471 (5)
N(1)—C(1)	1.397 (4)	N(2)—C(15)	1.464 (5)
C(16)—Ga—N(2)	102.0 (2)	C(14)—N(2)—C(13)	110.7 (3)
C(17)—Ga—N(2)	102.0 (2)	C(15)—N(2)—C(14)	110.5 (4)
C(18)—Ga—N(2)	101.6 (2)	C(15)—N(2)—C(13)	110.8 (3)
C(17)—Ga—C(16)	116.9 (3)	C(13)—N(2)—Ga	108.1 (2)
C(18)—Ga—C(16)	114.2 (3)	C(14)—N(2)—Ga	108.2 (3)
C(18)—Ga—C(17)	116.6 (3)	C(15)—N(2)—Ga	108.5 (2)
C(12)—N(1)—C(1)	108.5 (3)	N(1)—C(12)—C(7)	108.6 (3)
C(12)—N(1)—C(13)	125.8 (3)	N(1)—C(13)—N(2)	115.9 (3)
C(1)—N(1)—C(13)	125.2 (3)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1987). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN LS*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BM1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetrachlorobis(*N*,*N*,*N*′,*N*′-tetramethylethylenediamine-*N*,*N*′)thorium(IV)

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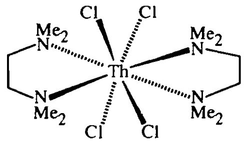
Abstract

The title compound, [$ThCl_4(C_6H_{16}N_2)_2$], displays an approximate dodecahedral geometry in the solid state, with the Th atom binding to four Cl ligands and to the four N atoms of two tetramethylethylenediamine ligands. The Th—Cl and Th—N distances are in the ranges 2.683 (3)–2.692 (3) and 2.800 (7)–2.825 (9) Å, respectively.

Comment

The preparation and characterization of the thorium and uranium complexes [$MCl_4(Me_2NCH_2CH_2NMe_2)_2$] ($M$ = Th, U) were reported by Edwards, Weydert, Petrie & Andersen (1994). The determination of the molecular structure of the uranium species (Zalkin, Edwards, Zhang & Andersen, 1986) is now compared with that of the thorium analog described herein. The eight-coordinate thorium complex [$ThCl_4(Me_2NCH_2CH_2NMe_2)_2$], (I), is isostructural with its uranium analog and consists of a Th atom surrounded by the four N atoms of two tetramethylethylenediamine



(I)

ligands and four Cl ligands, located in the *A* and *B* sites of an idealized dodecahedron, respectively (Hoard & Silverton, 1963).

The Th—Cl distances [range 2.683 (3)–2.692 (3) Å] are similar to those observed in other complexes containing terminal Th—Cl groups {*e.g.* 2.651 (5) Å in [$Cp^*_2Th\{OC(CO)SiR_3\}Cl$] (Radu, Engeler, Gerlach, Tilley & Rheingold, 1995), and 2.696 (3) and 2.743 (3) Å in [$ThCl_4\{OC(NMePh)_2\}_3$] (Al-Daher, Bagnall, Benetollo, Polo & Bombieri, 1986)}. Similarly, the Th—N bond lengths [2.800 (7)–2.825 (9) Å] are close to those observed in thorium complexes containing nitrogen-donor ligands {e.g. 2.66 (4)–2.83 (3) Å in [$ThCl_4(NMe_2)_3$] (Drew & Willey, 1984)}. The C—N bond distances range from 1.44 (2) to 1.52 (2) Å, while the C—C bond distances are 1.43 (2) and 1.50 (2) Å. These values are within the expected range for metal-coordinated tetramethylethylenediamine ligands (Zalkin, Edwards, Zhang & Andersen, 1986; Beyers, Pennington & Robinson, 1992). There are no unusual intermolecular contacts.

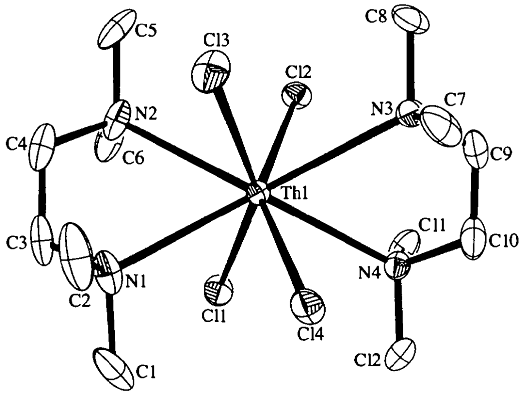


Fig. 1. The molecular structure of [$ThCl_4(Me_2NCH_2CH_2NMe_2)_2$], with H atoms omitted for clarity and displacement ellipsoids shown at the 35% probability level.

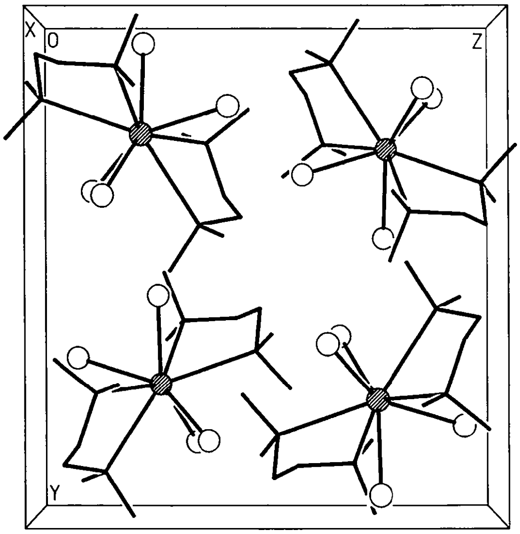


Fig. 2. A view of the unit cell of (I), with Th atoms striped, Cl atoms open and ligands without H atoms denoted as sticks.

Experimental

The preparation of (I) has been reported by Edwards, Weydert, Petrie & Andersen (1994). Crystals of the title compound used in this structure determination were obtained from a concentrated toluene solution at 243 K. The crystal was mounted with silicone onto the end of a glass fiber and placed in the nitrogen cold stream of the diffractometer.

Crystal data

[$ThCl_4(C_6H_{16}N_2)_2$]
$M_r = 606.3$
Orthorhombic
$P2_12_12_1$
$a = 13.098$ (3) Å
$b = 13.211$ (3) Å
$c = 12.578$ (3) Å
$V = 2176.5$ (9) Å^3
$Z = 4$
$D_x = 1.850$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 10.30$–$13.96°$
$\mu = 7.342$ mm^{-1}
$T = 150$ K
Polyhedron
0.12 × 0.12 × 0.07 mm
Colorless

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.356$, $T_{max} = 0.598$
4403 measured reflections
3828 independent reflections

3586 reflections with $F > 4\sigma(F)$
$R_{int} = 0.063$
$\theta_{max} = 25°$
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 15$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections intensity decay: 1.4%

Refinement

Refinement on *F*
$R = 0.0313$
$wR = 0.0356$
$S = 1.65$
3586 reflections
192 parameters
H atoms riding, overall *U* for H atoms refined: 0.078 (7) Å^2
$w = 1/[\sigma^2(F) + 0.008F^2]$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 1.75$ e Å^{-3}
$\Delta\rho_{min} = -2.29$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 0.000131 (8)
Scattering factors from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974)

Table 1. *Selected geometric parameters (Å, °)*

Th(1)—Cl(1)	2.688 (2)	Th(1)—N(1)	2.800 (7)
Th(1)—Cl(2)	2.690 (2)	Th(1)—N(2)	2.825 (9)
Th(1)—Cl(3)	2.692 (3)	Th(1)—N(3)	2.820 (7)
Th(1)—Cl(4)	2.683 (3)	Th(1)—N(4)	2.803 (7)
Cl(1)—Th(1)—Cl(2)	86.5 (1)	Cl(3)—Th(1)—N(1)	80.9 (2)
Cl(1)—Th(1)—Cl(3)	149.9 (1)	Cl(3)—Th(1)—N(2)	75.1 (2)
Cl(1)—Th(1)—Cl(4)	101.7 (1)	Cl(3)—Th(1)—N(3)	72.8 (2)

Cl(2)—Th(1)—Cl(3)	100.1 (1)	Cl(3)—Th(1)—N(4)	137.5 (2)
Cl(2)—Th(1)—Cl(4)	149.5 (1)	Cl(4)—Th(1)—N(1)	73.0 (2)
Cl(3)—Th(1)—Cl(4)	87.4 (1)	Cl(4)—Th(1)—N(2)	137.4 (2)
Cl(1)—Th(1)—N(1)	74.6 (2)	Cl(4)—Th(1)—N(3)	78.7 (2)
Cl(1)—Th(1)—N(2)	79.1 (2)	Cl(4)—Th(1)—N(4)	75.6 (2)
Cl(1)—Th(1)—N(3)	136.9 (2)	N(1)—Th(1)—N(2)	66.2 (3)
Cl(1)—Th(1)—N(4)	72.5 (2)	N(1)—Th(1)—N(3)	142.0 (2)
Cl(2)—Th(1)—N(1)	137.2 (2)	N(1)—Th(1)—N(4)	128.0 (3)
Cl(2)—Th(1)—N(2)	72.8 (2)	N(2)—Th(1)—N(3)	129.3 (3)
Cl(2)—Th(1)—N(3)	75.5 (2)	N(2)—Th(1)—N(4)	140.9 (2)
Cl(2)—Th(1)—N(4)	79.1 (1)	N(3)—Th(1)—N(4)	65.9 (2)

All non-H atomic displacement parameters were refined anisotropically. Orienting H atoms were located from difference Fourier maps and then the remaining H atoms were generated with ideal geometries (0.96 Å and 109.5°). All were subsequently refined as riding groups with a group displacement parameter. The maximum residual electron density is located 1.2 Å from C(2) and the minimum residual electron density is located 1.0 Å from Th(1). Structural refinements in the other enantiomorph of this non-centrosymmetric space group gave $R = 0.0569$ and $wR = 0.0645$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXTL-Plus* (Sheldrick, 1990). Molecular graphics: *SHELXTL-Plus*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BK1289). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1997). C**53**, 193–195

Metal–α,ω-Dicarboxylate Complexes. I. Aqua(2,2′-bipyridyl-*N*,*N*′)(malonato-*O*,*O*′)-copper(II) Monohydrate

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Abstract

The title compound, $[Cu(C_3H_2O_4)(C_{10}H_8N_2)(H_2O)]\cdot H_2O$, exists as discrete monomers. The Cu^{II} ion is square pyramidal, with two N atoms of the bipyridyl ligand and two O atoms, one from each of the carboxylates of the folded dibasic acid, providing the base and a water molecule forming a long apical bond [Cu—O5 2.523 (3) Å]. The 'scorpion-like' complex possesses a plane of symmetry bisecting the molecule lengthwise, the non-planarity arising from severe puckering of the six-membered chelate ring of the acid. Hydrogen-bonding interactions between the water molecules and the carboxylate O atoms stabilize the crystal structure.

Comment

In recent years, inorganic solids with novel polymeric networks have found applications in catalysis, separation, magnetism and optics. The extraordinary versatility of α,ω-dicarboxylic acids, forming different types of polymeric species upon coordination, is well known