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

Cd—O(1)	2.333 (6)	Nb—F(2)	1.936 (3)
Cd—N(1)	2.335 (4)	Nb—F(3)	2.095 (4)
Cd—N(2)	2.359 (8)	Nb—O(1)	1.750 (6)
Nb—F(1)	1.932 (3)		
O(1)—Cd—N(1)	90.0	F(1)—Nb—O(1)	95.3 (1)
O(1)—Cd—N(2)	90.00 (1)	F(2)—Nb—F(2^{i})	166.7 (2)
N(1)—Cd—N(2)	90.0	F(2)—Nb—F(3)	83.3 (1)
F(1)—Nb—F(1^{i})	169.3 (2)	F(2)—Nb—O(1)	96.6 (1)
F(1)—Nb—F(2)	89.0 (1)	F(3)—Nb—O(1)	180.0
F(1)—Nb—F(2^{i})	89.8 (1)	Cd—O(1)—Nb	180.0
F(1)—Nb—F(3)	84.7 (1)		

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

Neutral-atom scattering factors were taken from Cromer & Waber (1974). The coordinates and isotropic U values for the disordered N(3) and C(9) sites were constrained to be equal in the refinement.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *DIRDIF* (Beurskens *et al.*, 1992). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1219). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$[(CpTi)_6(\mu_3\text{-}Te)_6(\mu_3\text{-}O)_2]$

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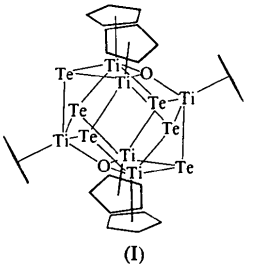
(Received 23 August 1994; accepted 2 April 1996)

Abstract

The X-ray crystal structure of the title cluster, hexakis[1,2,3,4,5,6(η^5)-cyclopentadienyl]-di-μ_3-oxo-1:2:3κ^3O;4:5:6κ^3O-hexatelluriumhexatitanium(18 *Te—Ti*), $[\{Ti(C_5H_5)\}_6(\mu_3\text{-}Te)_6(\mu_3\text{-}O)_2]$, has been determined. The metric parameters for this cluster are compared with those of the closely related oxo cluster and other relevant titanium compounds.

Comment

The cluster $[(CpTi)_6(\mu_3\text{-}Te)_6(\mu_3\text{-}O)_2]$, (1), where Cp is cyclopentadienyl, was obtained from the reaction of $Cp_2Ti(TeSiPh_3)_2$ with PPh_3. Although we have little information as to how this compound is formed, it is most likely that the oxo ligands are introduced by trace amounts of water in the toluene solvent used.



(I)

An *ORTEP*II (Johnson, 1976) diagram of the molecular structure of (1) viewed down the O—O vector is shown in Fig. 1. The structure of (1) is similar to the structure of $[(CpTi)_6(\mu_3\text{-}O)_6]$, (2), reported some time ago by Huffman, Stone, Krusell & Caulton (1977). The six Ti atoms form an octahedron with each of the faces capped by a chalcogen. Each individual Ti atom is coordinated in a distorted square-pyramidal geometry. The *L*—Ti—*L* angles range from 75.1 (1) to 86.8 (2)° for *cis* ligands and from 121.8 (2) to 138.0 (2)° for *trans* ligands. Unlike cluster (2), the Ti···Ti distances [3.273 (2)–3.990 (2) Å] in (1) do not suggest any degree of interaction between metal centres. The Ti—C distances (average 2.374 Å) are identical to values found in both (2) and related titanocene derivatives. The Te1···Te3 and Te2···Te3 distances [3.442 (1) and 4.048 (1) Å, respectively] lie outside the range of bond lengths found for Te—Te covalent bonds (Du

Mont, Lange, Karsch, Peters & von Schnering, 1987). The Ti—Te distances [2.798 (2)–2.844 (2) Å] are close to those found in both the $[(MeCp)_5Ti_5Te_6]$ cluster (Fenske & Grissinger, 1990) and the $[(MeCp)_2Ti_2Te_2]$ dimer, but longer than the Ti—Te distances found in $[(MeCp)_2Ti\{TeSi(SiMe_3)_3\}_2]$ (average 2.788 Å; Christou, Wuller & Arnold, 1993).

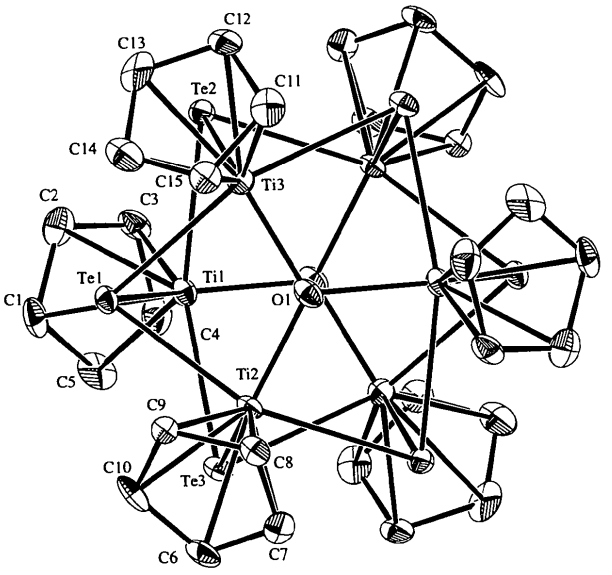


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the molecular structure of $[(CpTi)_6(\mu_3\text{-}Te)_6(\mu_3\text{-}O)_2]$ showing 50% probability displacement ellipsoids and the atom-numbering scheme. The H atoms have been omitted for clarity.

Experimental

The title cluster, $[(CpTi)_6(\mu_3\text{-}Te)_6(\mu_3\text{-}O)_2]$, was obtained in low yield as black plate-like crystals from the reaction of $Cp_2Ti(TeSiPh_3)_2$ with five equivalents of PPh_3 in toluene over a period of several days. A portion of one plate was cleaved off and used for the structure determination.

Crystal data

$[\{Ti(C_5H_5)\}_6Te_6(O)_2]$
M_r = 1475.58
Monoclinic
$P2_1/a$
a = 10.388 (2) Å
b = 16.781 (7) Å
c = 11.234 (2) Å
β = 115.60 (2)°
V = 1757 (2) Å^3
Z = 2
D_x = 2.785 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 22–24°
μ = 6.184 mm^{-1}
T = 138 K
Plate
0.30 × 0.20 × 0.20 mm
Black

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans
Absorption correction: empirical *via* ψ scan (*MolEN*; Fair, 1990)
T_{min} = 0.25, T_{max} = 0.29
2572 measured reflections
2289 independent reflections
1841 observed reflections [$F > 3\sigma(F^2)$]
θ_{max} = 45°
h = 0 → 11
k = 0 → 18
l = −12 → 12
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
R = 0.0318
wR = 0.0373
S = 1.543
1841 reflections
199 parameters
H-atom parameters not refined
$w = 1/\sigma(F)^2$; non-Poisson contribution
$(\Delta/\sigma)_{max}$ = 0.000
$\Delta\rho_{max}$ = 1.71 e Å^{-3}
$\Delta\rho_{min}$ = −0.93 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *MolEN* (Fair, 1990)

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

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

	x	y	z	B_{eq}
Te1	0.21928 (1)	0.61974 (1)	0.47769 (1)	1.22 (1)
Te2	0.31576 (1)	0.44074 (1)	0.64436 (1)	1.28 (1)
Te3	−0.04249 (1)	0.67822 (1)	0.56794 (1)	1.28 (1)
Ti1	0.1875 (2)	0.5757 (1)	0.7041 (2)	1.22 (4)
Ti2	−0.0774 (2)	0.6145 (1)	0.3239 (2)	1.10 (3)
Ti3	0.1482 (2)	0.4632 (1)	0.3738 (2)	1.30 (4)
O1	0.0470 (6)	0.4971 (4)	0.6982 (5)	1.4 (1)
C1	0.362 (1)	0.6755 (6)	0.8233 (9)	2.0 (2)
C2	0.426 (1)	0.6005 (7)	0.8606 (9)	2.0 (2)
C3	0.354 (1)	0.5590 (6)	0.9233 (9)	1.8 (2)
C4	0.248 (1)	0.6093 (7)	0.9255 (9)	2.4 (2)
C5	0.252 (1)	0.6803 (6)	0.8657 (9)	2.3 (2)
C6	−0.178 (1)	0.7403 (6)	0.2260 (9)	1.8 (2)
C7	−0.2618 (9)	0.6796 (6)	0.1406 (9)	1.7 (2)
C8	−0.1743 (9)	0.6388 (6)	0.0933 (8)	1.4 (2)
C9	−0.0385 (9)	0.6741 (6)	0.1524 (8)	1.4 (2)
C10	−0.042 (1)	0.7368 (6)	0.2318 (9)	1.9 (2)
C11	0.142 (1)	0.3952 (6)	0.1905 (9)	2.0 (2)
C12	0.2741 (9)	0.3745 (6)	0.2953 (9)	1.8 (2)
C13	0.3574 (9)	0.4431 (7)	0.3357 (9)	2.3 (2)
C14	0.279 (1)	0.5063 (6)	0.2550 (9)	2.5 (2)
C15	0.146 (1)	0.4771 (6)	0.1651 (8)	1.9 (2)

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

Te1—Ti1	2.802 (2)	Te3—Ti2	2.815 (2)
Te1—Ti2	2.809 (2)	Te3—Ti3^{i}	2.801 (2)
Te1—Ti3	2.830 (2)	Ti3—O1^{i}	1.947 (6)
Ti1—O1	1.943 (6)	Ti1—*Cp*1†	2.050 (2)
Te2—Ti1	2.844 (2)	Ti1—C (Cp1 average)	2.372
Te2—Ti2	2.806 (2)	Ti2—*Cp*2†	2.060 (2)
Te2—Ti3	2.799 (2)	Ti2—C (Cp2 average)	2.379
Ti2—O1	1.925 (6)	Ti3—*Cp*3†	2.050 (2)
Te3—Ti1	2.798 (2)	Ti3—C (Cp3 average)	2.373
Te1—Ti1—Te2	75.1 (1)	Te1—Ti2—O1	85.5 (2)
Te1—Ti1—Te3	75.5 (1)	Te2—Ti2—O1	84.3 (2)
Te2—Ti1—Te3	137.1 (1)	Te3^{ii}—Ti2—O1	122.2 (2)
Te1—Ti1—O1	121.8 (2)	Te1—Ti3—Te2	75.3 (1)
Te2—Ti1—O1	82.9 (2)	Te1—Ti3—Te3	138.0 (2)
Te3—Ti1—O1	86.8 (2)	Te2—Ti3—Te3	75.9 (1)
Te1—Ti2—Te2^{ii}	136.6 (2)	Te1—Ti3—O1	85.0 (2)
Te1—Ti2—Te3	75.1 (1)	Te2—Ti3—O1	122.9 (2)
Te2^{ii}—Ti2—Te3^{ii}	75.5 (1)	Te3—Ti3—O1	86.7 (2)

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, -y, 1-z$.

† Cp1, Cp2 and Cp3 denote cyclopentadienyl rings C1–C5, C6–C10 and C11–C15, respectively, with *Cp*1, *Cp*2 and *Cp*3 being their midpoints.

H atoms were included with isotropic displacement parameters.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Program(s) used to solve structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SZ1028). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sodium Cholate Methanolate and Sodium Cholate 2-Propanolate

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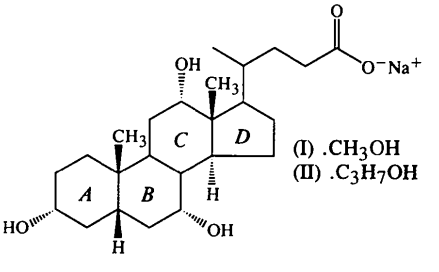
Abstract

The crystal structures of sodium cholate methanolate (sodium $3\alpha,7\alpha,12\alpha$-trihydroxy-5β-cholan-24-oate methanol solvate, $Na^+.C_{24}H_{39}O_5^-.CH_4O$) recrystallized from absolute methanol by diffusion of acetone, and sodium cholate 2-propanolate (sodium $3\alpha,7\alpha,12\alpha$-trihydroxy-5β-cholan-24-oate 2-propanol solvate, $Na^+.C_{24}H_{39}O_5^-.C_3H_8O$), recrystallized from 2-propanol by diffusion of acetone, have been determined. These are the second and third reported sodium cholate structures solely solvated by an organic molecule.

Comment

Our research into methyl rotation in solid-state pseudo-polymorphs led us to a number of compounds containing multiple methyl groups, including the two bile acids, cholic acid ($3\alpha,7\alpha,12\alpha$-trihydroxy-5β-cholan-24-oic acid) and deoxycholic acid ($3\alpha,12\alpha$-dihydroxy-5β-cholan-24-oic acid). Many solvated structures have been reported for both cholic acid (Lessinger, 1982; Lessinger & Low, 1993; Miki *et al.*, 1988; Miki, Kasai, Shibakami, Takemoto & Miyata, 1991; Nakano, Sada & Miyata, 1994; Caira, Nassimbeni & Scott, 1993, 1994*a*,*b*; Shibakami & Sekiya, 1994; Scott, 1995) and deoxycholic acid (Giglio, 1984). Of special note are the isostructural forms of cholic acid when solvated with methanol, ethanol and 1-propanol (Johnson & Schaefer, 1972; Jones & Nassimbeni, 1990). However, less attention has been given to cholic acid salt complexes. Until now, only four cholic acid salt structures have been reported in the literature, one with calcium (Hogan, Ealick, Bugg & Barnes, 1984), the other three with sodium (Norton & Haner, 1965; Cobbledick & Einstein, 1980; Wahle, Stowell & Byrn, 1996). One of the sodium salts is reported to be anhydrous, while another sodium salt and the calcium salt are both hydrated structures. The third sodium salt is solvated with ethanol. We have previously reported (Wahle, Stowell & Byrn, 1996) that a search of the Cambridge Structural Database (Allen *et al.*, 1979) revealed only five sodium salt steroid complexes. Four of these sodium salts are hydrates and the fifth is an acetone/water solvate. Recently, we reported the first cholic acid salt structure solvated solely with an organic molecule (Wahle, Stowell & Byrn, 1996). Here we continue our examination of sodium cholate structures solvated solely with organic molecules by reporting two new solvates, namely, sodium cholate methanolate (NACAMEOH), (I), and sodium cholate 2-propanolate (NACA2PROH), (II).



(I)

*ORTEP*II (Johnson, 1976) diagrams of the cholate salt structures of (I) and (II) are given in Figs. 1 and 2, respectively. The rings in (I) have a geometry similar to the other cholic acid structures reported to date, with a *cis* ring junction for the *A*/*B* rings and *trans* ring junctions for the *B*/*C* and *C*/*D* rings. When the methanol-solvated and ethanol-solvated sodium salts are overlaid using a least-squares fit, the *A*, *B* and *C*

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