

complex is much reduced compared with that in the chloro-bridged complex. This result is consistent with the spectral and conductivity data reported previously (Aoki, Hamaue, Kida, Yamashita, Takemura, Furuta & Kawamori, 1982).

Interestingly, the bond-distance ratios of the chloro- and bromo-bridged Pd complexes, [Pd(en)₂][PdX₂(en)₂](ClO₄)₄, are comparable to those of the chloro- and bromo-bridged Pt analogs, respectively, whereas the spectral and conductivity data of the chloro and bromo Pd complexes are nearly equal to those of the bromo and iodo Pt analogs, respectively (Aoki *et al.*, 1982).

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Structure of Di- μ -chloro-tetrachlorobis(tetrahydrofuran)-[μ -(dimethyl sulfide)]-diniobium(Nb–Nb), [Nb₂Cl₆(C₄H₈O)₂(C₂H₆S)], and Di- μ -chloro-tetrachlorobis(tetrahydrofuran)-[μ -(dimethyl sulfide)]-ditantalum(Ta–Ta), [Ta₂Cl₆(C₄H₈O)₂(C₂H₆S)]

BY F. ALBERT COTTON,* STAN A. DURAJ AND WIESLAW J. ROTH

Department of Chemistry and Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, Texas 77843, USA

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Abstract. [NbCl₂(THF)]₂(μ -Cl)₂(μ -SMe₂): M_r = 604.88, triclinic, $P\bar{1}$, a = 8.104 (1), b = 16.669 (5), c = 7.917 (4) Å, α = 101.28 (3), β = 95.21 (3), γ = 81.24 (2)°, V = 1034 (1) Å³, Z = 2, D_x = 1.942 Mg m⁻³, Mo $K\alpha$, λ = 0.71073 Å, μ = 1.94 mm⁻¹, $F(000)$ = 596, T = 298 K, R = 0.0575 for 1816 ($I \geq 3\sigma$) reflections. [TaCl₂(THF)]₂(μ -Cl)₂(μ -SMe₂): M_r = 780.96, triclinic, $P\bar{1}$, a = 8.122 (2), b = 16.663 (4), c = 7.932 (1) Å, α = 101.44 (2), β = 95.56 (2), γ = 81.18 (2)°, V = 1037 (1) Å³, Z = 2, D_x = 2.502 Mg m⁻³, Mo $K\alpha$, λ = 0.71073 Å, μ = 11.30 mm⁻¹, $F(000)$ = 724, T = 298 K, R = 0.0293 for 2886 ($I \geq 3\sigma$) reflections. The metal–metal bond lengths are: Nb=Nb 2.684 (2) and Ta=Ta 2.6695 (5) Å. The structures of these two confacial-bioctahedral complexes are isomorphous and correspond with the structures of similar Nb and Ta complexes previously studied. The metal–Cl and

metal–S bond lengths fall into a very narrow range, even when different *trans* ligands are present.

Introduction. Niobium and tantalum in their oxidation states of III exhibit a remarkable tendency to form dimeric complexes (Cotton & Walton, 1982; Rocklage, Turner, Fellmann & Schrock, 1982; Templeton, Dorman, Clardy & McCarley, 1978). There are two general types of geometry that these complexes adopt: edge-sharing bioctahedron or confacial bioctahedron (Cotton & Roth, 1983; Sattelberger, Wilson & Huffman, 1982; Cotton & Najjar, 1981; Templeton & McCarley, 1978; Cotton & Hall, 1980). There are indications that in the confacial-bioctahedral complexes, $M_2X_6(L_1)_2(\mu-L_2)$, M = Nb and Ta, X = Cl and Br and $L_1 = L_2$ = tetrahydrothiophene (THT) or SMe₂, the terminal ligands (L_1) are more labile than the bridging ones (L_2) and are easily replaced (Cotton, Falvello & Najjar, 1982; Cotton & Hall, 1980; Keen & Brown, 1982). We have further

* To whom correspondence should be addressed.

Table 1. *Experimental details of the X-ray diffraction studies*

	Nb ₂ Cl ₆ (SMe ₂)(THF) ₂	Ta ₂ Cl ₆ (SMe ₂)(THF) ₂
Crystal size (mm)	0.25 × 0.2 × 0.2	0.4 × 0.35 × 0.2
Instrument	Enraf-Nonius CAD-4	
Number of orientation reflections, range (°)	25, 17.4 ≤ 2θ ≤ 42.4	25, 16.7 ≤ 2θ ≤ 33.8
Scan method	ω-2θ	
Data collection range (°)	4 ≤ 2θ ≤ 50	
Max. (sinθ)/λ reached in intensity measurements (Å ⁻¹)	0.538	
Range of h, k and l	0 ≤ h ≤ 9, -19 ≤ k ≤ 19, -9 ≤ l ≤ 9	
Standard reflections, intensity variation throughout experiment (%)	3, 1-5	3, 6-1
Number of unique data	2687	3160
Number with F _o ² > 3σ(F _o ²)	1816	2886
Number of parameters refined	181	170
Empirical absorption correction max., min.	-	0.9991, 0.7839
R	0.0575	0.0293
wR, w = σ ⁻² (F _o) ²	0.0663	0.0396
Quality of fit indicator, S	1.554	1.125
Max. Δ/σ, final cycle	0.19	0.04
Largest peak (e Å ⁻³)	0.83	1.00

examined this chemical behavior and replaced two terminal ligands (SMe₂) of two M₂Cl₆(L)₃ complexes with weakly coordinating tetrahydrofuran molecules. Our study resulted in a pair of analogous complexes, [MCl₂(THF)]₂(μ-Cl)₂(μ-SMe₂), M = Nb and Ta, whose structures we have determined by single-crystal X-ray analysis. There is only one prior example of a similar pair that had been examined by X-ray crystallography (Templeton, Dorman, Clardy & McCarley, 1978). However, in this case bromine atoms and tetrahydrothiophene molecules were used as supporting ligands.

Experimental. All manipulations were performed under an atmosphere of argon using standard vacuum-line techniques. The M₂Cl₆(SMe₂)₃, M = Nb and Ta, starting materials were prepared by following the procedure used to synthesize Ta₂Br₆(THT)₃ (Templeton & McCarley, 1978). Crystals of the products, M₂Cl₆(μ-SMe₂)(THF)₂, M = Nb and Ta, red and brown, respectively, were grown by slow diffusion of hexane into the THF solution of the appropriate tris-SMe₂ complex. Structure determinations were carried out by general procedures fully described elsewhere (Bino, Cotton & Fanwick, 1979). Experimental details of X-ray diffraction studies are presented in Table 1. All calculations performed with Enraf-Nonius (1981) *Structure Determination Package* on VAX 11/80 and PDP 11/60 computers at the Department of Chemistry, Texas A&M Univ. Empirical absorption correction for Ta complex made using ψ scans for nine reflections with Eulerian angle near 90°, each reflection measured at 10° intervals from 0 to 360°. Analogous ψ scans for Nb compound showed <10% variation in intensity, correction not applied. F_o corrected for Lorentz and polarization effects. Atomic scattering factors and f', f'' values from *International Tables for X-ray Crystallography* (1974). Both structures solved by Patterson function. All non-hydrogen atoms located and refined,

using F_o, by series of least-squares refinements and difference Fourier syntheses. Some THF ligands were disordered and the following fractional occupancy factors were assigned by trial and error to give comparable B's for corresponding atoms: for the Nb complex, C(7) through C(10) - 0.6, C(7A) through C(10A) - 0.4; for the Ta complex: C(5) - 0.6, C(5A) - 0.4, C(7) through C(10) - 0.67, C(7A) through C(10A) - 0.33. All atoms in disordered rings refined isotropically. Most intense peaks in difference Fourier map after completion of refinement located in region around disordered THF ligands.

Discussion. The positional parameters are listed in Tables 2 and 3. Bond distances and bond angles between the atoms within the coordination sphere are presented in Tables 4 and 5.*

The compounds reported here belong to the class of Nb^{III} and Ta^{III} confacial-bioctahedral dimers with a double metal-to-metal bond. An ORTEP drawing of the niobium complex is shown in Fig. 1. Since the tantalum analog is isomorphous the same labeling scheme applies to both complexes.

Table 6 lists averaged M-M and M-L distances found in the structurally characterized representatives of this class. Based on the values of bond lengths presented in Table 4 the following conclusions can be drawn.

(a) Corresponding M-L bond lengths fall within a very narrow range, viz M-Cl_b 2.489-2.503, M-Cl_t 2.344-2.375, M-S_b 2.370-2.406, M-S_t 2.618-2.632 Å; there is no difference between Nb and Ta in this regard; the extent of M-Br bond elongation with respect to the M-Cl separation is consistent with the difference in the covalent radii [equal to 0.15 Å according to Pauling (1960)] of these two halogens.

* Full lists of bond distances, bond angles, structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42046 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

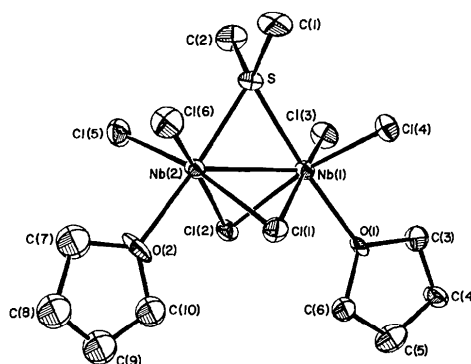


Fig. 1. A view of the [NbCl₂(THF)]₂(μ-Cl)₂(μ-SMe₂) molecule showing the atom-labeling scheme.

Table 2. Positional and equivalent isotropic thermal parameters for Nb₂Cl₆(SMe₂)(THF)₂

	x	y	z	B or B _{eq} (Å ²)
Nb(1)	0.2691 (2)	0.17812 (8)	0.1330 (2)	2.10 (3)
Nb(2)	0.3657 (2)	0.31699 (8)	0.0757 (2)	2.29 (3)
Cl(1)	0.3564 (5)	0.2873 (2)	0.3707 (5)	3.24 (9)
Cl(2)	0.0662 (4)	0.3002 (2)	0.0793 (5)	3.19 (9)
Cl(3)	0.1285 (5)	0.0902 (3)	-0.0854 (6)	4.0 (1)
Cl(4)	0.4586 (5)	0.0759 (2)	0.2510 (6)	3.70 (9)
Cl(5)	0.3121 (6)	0.3599 (3)	-0.1956 (5)	4.2 (1)
Cl(6)	0.6455 (5)	0.3446 (3)	0.1398 (6)	4.5 (1)
S	0.4712 (5)	0.1805 (2)	-0.0700 (5)	2.63 (8)
O(1)	0.085 (1)	0.1648 (6)	0.316 (1)	3.2 (2)
O(2)	0.282 (1)	0.4471 (6)	0.202 (1)	4.0 (3)
C(1)	0.687 (2)	0.135 (1)	-0.038 (2)	3.7 (4)
C(2)	0.432 (2)	0.147 (1)	-0.298 (2)	4.3 (4)
C(3)	0.048 (2)	0.0851 (9)	0.338 (2)	4.9 (4)
C(4)	-0.091 (2)	0.102 (1)	0.452 (2)	4.0 (4)
C(5)	-0.136 (2)	0.194 (1)	0.504 (3)	6.5 (5)
C(6)	-0.014 (2)	0.233 (1)	0.431 (2)	4.1 (4)*
C(7)	0.332 (5)	0.518 (2)	0.148 (5)	8 (1)*
C(8)	0.283 (4)	0.589 (2)	0.322 (4)	5.4 (7)*
C(9)	0.122 (5)	0.567 (3)	0.338 (6)	9 (1)*
C(10)	0.161 (4)	0.474 (2)	0.330 (5)	6.3 (8)*
C(7A)	0.148 (6)	0.499 (3)	0.128 (7)	6 (1)*
C(8A)	0.197 (5)	0.592 (2)	0.242 (5)	3.5 (8)*
C(9A)	0.200 (7)	0.561 (3)	0.416 (7)	7 (1)*
C(10A)	0.388 (7)	0.496 (3)	0.346 (8)	8 (1)*

* Atoms refined isotropically. For anisotropically refined atoms $B_{eq} = (4/3) [a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

Table 3. Positional and equivalent isotropic thermal parameters for Ta₂Cl₆(SMe₂)(THF)₂

	x	y	z	B or B _{eq} (Å ²)
Ta(1)	0.27058 (4)	0.17876 (2)	0.13083 (4)	2.331 (6)
Ta(2)	0.36531 (4)	0.31739 (2)	0.07595 (4)	2.460 (6)
Cl(1)	0.3542 (3)	0.2891 (1)	0.3713 (3)	3.25 (4)
Cl(2)	0.0657 (2)	0.3007 (1)	0.0792 (3)	3.36 (4)
Cl(3)	0.1281 (3)	0.0909 (1)	-0.0879 (3)	4.10 (5)
Cl(4)	0.4594 (3)	0.0782 (1)	0.2502 (3)	3.77 (5)
Cl(5)	0.3088 (3)	0.3616 (2)	-0.1922 (3)	4.37 (5)
Cl(6)	0.6425 (3)	0.3469 (2)	0.1434 (4)	4.40 (5)
S	0.4684 (2)	0.1829 (1)	-0.0674 (3)	2.77 (4)
O(1)	0.0854 (7)	0.1653 (3)	0.3115 (7)	3.1 (1)
O(2)	0.2813 (8)	0.4467 (3)	0.2010 (8)	3.7 (1)
C(1)	0.687 (1)	0.1375 (7)	-0.044 (1)	4.6 (2)
C(2)	0.433 (1)	0.1478 (6)	-0.301 (1)	4.0 (2)
C(3)	0.049 (1)	0.0840 (7)	0.330 (1)	4.9 (2)*
C(4)	-0.090 (1)	0.1012 (6)	0.450 (1)	4.4 (2)*
C(5)	-0.154 (2)	0.192 (1)	0.476 (2)	4.2 (3)*
C(5A)	-0.101 (3)	0.190 (1)	0.535 (3)	3.1 (4)*
C(6)	-0.009 (1)	0.2319 (6)	0.429 (1)	4.3 (2)*
C(7)	0.340 (2)	0.517 (1)	0.152 (2)	6.2 (4)*
C(8)	0.256 (3)	0.593 (1)	0.267 (3)	7.7 (5)*
C(9)	0.155 (2)	0.564 (1)	0.388 (2)	5.6 (4)*
C(10)	0.163 (3)	0.475 (1)	0.342 (3)	7.6 (5)*
C(7A)	0.168 (6)	0.518 (3)	0.124 (7)	10 (1)*
C(8A)	0.139 (5)	0.590 (2)	0.272 (5)	6.3 (8)*
C(9A)	0.288 (5)	0.581 (2)	0.382 (5)	6.7 (9)*
C(10A)	0.380 (5)	0.498 (3)	0.350 (6)	8 (1)*

* Atoms refined isotropically. For anisotropically refined atoms $B_{eq} = (4/3) [a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

Table 4. Important bond distances (Å) in the M₂Cl₆(SMe₂)(THF)₂ complexes

Numbers in parentheses are e.s.d.'s in the least significant digits.

	M = Nb	M = Ta
M(1)—M(2)	2.684 (2)	2.6695 (5)
Cl(1)	2.483 (4)	2.494 (2)
Cl(2)	2.494 (4)	2.495 (2)
Cl(3)	2.365 (4)	2.370 (2)
Cl(4)	2.387 (4)	2.370 (2)
S	2.406 (4)	2.372 (2)
O(1)	2.235 (11)	2.239 (6)
M(2)—Cl(1)	2.490 (4)	2.492 (2)
Cl(2)	2.489 (4)	2.494 (2)
Cl(5)	2.377 (5)	2.368 (2)
Cl(6)	2.372 (4)	2.366 (2)
S	2.408 (4)	2.369 (2)
O(2)	2.234 (9)	2.219 (5)

Table 5. Important bond angles (°) in the M₂Cl₆(SMe₂)(THF)₂ complexes

Numbers in parentheses are e.s.d.'s in the least significant digits.

	M = Nb	M = Ta
M(2)—M(1)—Cl(1)	57.5 (1)	57.59 (5)
Cl(2)	57.3 (1)	57.64 (5)
Cl(3)	121.8 (1)	121.98 (7)
Cl(4)	122.1 (1)	121.89 (6)
S	56.1 (1)	55.69 (5)
O(1)	128.5 (2)	128.3 (1)
Cl(1)—M(1)—Cl(2)	78.7 (1)	78.11 (7)
Cl(3)	167.8 (1)	166.67 (7)
Cl(4)	90.4 (1)	90.31 (7)
S	99.2 (1)	99.41 (7)
O(1)	84.2 (3)	83.9 (1)
Cl(2)—M(1)—Cl(3)	90.9 (1)	90.69 (7)
Cl(4)	167.1 (1)	166.22 (7)
S	99.3 (1)	99.31 (7)
O(1)	84.4 (3)	83.6 (1)
Cl(3)—M(1)—Cl(4)	99.0 (1)	99.73 (8)
S	88.7 (1)	89.43 (8)
O(1)	88.4 (3)	87.8 (1)
Cl(4)—M(1)—S	89.3 (2)	89.86 (8)
O(1)	87.6 (3)	87.8 (2)
S—M(1)—O(1)	175.4 (3)	176.0 (1)
M(1)—M(2)—Cl(1)	57.2 (1)	57.67 (5)
Cl(2)	57.5 (1)	57.67 (5)
Cl(5)	122.1 (1)	122.29 (6)
Cl(6)	121.6 (1)	121.96 (7)
S	56.1 (1)	55.77 (6)
O(2)	128.1 (3)	128.2 (2)
Cl(1)—M(2)—Cl(2)	78.6 (1)	78.16 (7)
Cl(5)	167.1 (2)	165.84 (8)
Cl(6)	90.3 (2)	90.35 (9)
S	98.9 (1)	99.53 (7)
O(2)	83.9 (3)	83.3 (2)
Cl(2)—M(2)—Cl(5)	90.5 (2)	90.19 (9)
Cl(6)	167.0 (2)	166.27 (8)
S	99.4 (1)	99.39 (7)
O(2)	84.2 (3)	83.9 (2)
Cl(5)—M(2)—Cl(6)	99.6 (2)	100.0 (1)
S	89.5 (1)	90.22 (8)
O(2)	88.3 (3)	87.5 (2)
Cl(6)—M(2)—S	88.9 (1)	89.82 (7)
O(2)	88.0 (3)	87.4 (2)
S—M(2)—O(2)	175.8 (3)	176.0 (2)
M(1)—Cl(1)—M(2)	65.3 (1)	64.74 (5)
M(1)—Cl(2)—M(2)	65.20 (9)	64.69 (5)
M(1)—S—M(2)	67.8 (1)	68.53 (5)

Table 6. Average values of selected interatomic distances in confacial-bioctahedral dimers of Nb and Ta (Å)

THT tetrahydrothiophene; <i>b</i> bridging; <i>t</i> terminal.						
	<i>M-M</i>	<i>M-X_b</i>	<i>M-X_t</i>	<i>M-L_b</i>	<i>M-L_t</i>	References
(I) Nb ₂ Br ₆ (THT) ₃	2.728 (5)	2.626 [10]*	2.534 [6]	2.487 [32]	2.632 [41]	Templeton <i>et al.</i> (1978)
(II) Ta ₂ Br ₆ (THT) ₃	2.710 (2)	2.633 [8]	2.516 [3]	2.393 [3]	2.624 [3]	Templeton <i>et al.</i> (1978)
(III) Ta ₂ Cl ₆ (THT) ₃	2.681 (1)	2.503 [5]	2.366 [2]	2.390 [11]	2.629 [7]	Cotton & Najjar (1981)
(IV)† Ta ₂ Cl ₆ (SMe ₂) ₃	2.691 (1)	2.498 [20]	2.370 [5]	2.378 (5)	2.618 (5)	Cotton & Najjar (1981)
(V) Ta ₂ Cl ₆ (μ-SMe ₂)(Me ₂ N) ₃ P ₁₂	2.704 (1)	2.500 [7]	2.347 [3]	2.400 [3]	2.726 [3]	Cotton, Falvello & Najjar (1982)
(VI)† Ta ₂ Cl ₆ (μ-(Bu ^t C) ₂)(THF) ₂	2.677 (1)	2.489 [7]	2.344 [17]	2.315 [10]‡	2.282 (8)	Cotton & Hall (1980)
(VII) Ta ₂ Cl ₆ (μ-SMe ₂)(THF) ₂	2.6695 (5)	2.494 [1]	2.368 [1]	2.370 [1]	2.229 [10]	This work
(VIII) Nb ₂ Cl ₆ (μ-SMe ₂)(THF) ₂	2.684 (2)	2.489 [2]	2.375 [5]	2.406 [1]	2.234 [1]	This work

* Numbers in square brackets are variances, obtained from the expression $[(\sum \Delta_i^2)/(n-1)]^{1/2}$, where Δ_i is the deviation of the *i*th value from the arithmetic mean and *n* is the total number of values averaged.

† Molecules of (IV) and (VI) reside on crystallographic symmetry elements *m* and *C₂*, respectively.

‡ Bridging C≡C group perpendicular to the *M-M* axis.

(b) Little variation in the *M-S_b* distances for different donors *trans* to *S_b* indicates a negligible *trans* effect.

(c) While the *M-X_b* distance is longer than the *M-X_t* one, the opposite is true for metal-sulfur bonds.

(d) In contrast to *M-L* bonds the *M-M* separation is more responsive to the nature of the ligands. It appears that Ta=Ta double bonds are shorter than those between niobium atoms in identical environments. This may arise from relativistic effects, which play a significant role with tantalum, but a more specific explanation can only be provided by theoretical MO calculations.

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Structure of Tetrakis[dimethyl(phenyl)phosphine]mercury(II) Decachloro-μ-oxo-ditantalate(V), [Hg{P(CH₃)₂(C₆H₅)₂}₄][Ta₂Cl₁₀O]

BY F. ALBERT COTTON,* STAN A. DURAJ AND WIESLAW J. ROTH

Department of Chemistry and Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, TX 77843, USA

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Abstract. *M_r* = 1485.65, orthorhombic, *Pbcn*, *a* = 4799.5 Å³, *Z* = 4, *D_x* = 2.056 g cm⁻³, λ(Mo Kα) = 0.71073 Å, μ = 84.077 cm⁻¹, *F*(000) = 2800.0, *T* = 295 K, *R* = 0.0472 for 1991 observed reflections. The compound consists of [Hg(PMe₂Ph)₄]²⁺ and

* To whom correspondence should be addressed.