

# Monopentamethylcyclopentadienyl isocyanide, amine and imido tantalum(V) complexes. X-ray crystal structure of $[\text{TaCp}^*\text{Cl}_4(\text{CN-2,6-Me}_2\text{C}_6\text{H}_3)]$

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## Abstract

$[\text{TaCp}^*\text{Cl}_4]$  ( $\text{Cp}^* = \eta^5\text{-C}_5\text{Me}_5$ ) reacts with isocyanides and amines to give the pseudo-octahedral adducts  $[\text{TaCp}^*\text{Cl}_4\text{L}]$  ( $\text{L} = 2,6\text{-Me}_2\text{C}_6\text{H}_3\text{NC}$ , **1**;  $2,4,6\text{-Me}_3\text{C}_6\text{H}_2\text{NC}$ , **2**;  $^t\text{BuNC}$ , **3**; or  $\text{C}_6\text{H}_5\text{NH}_2$ , **4**). Reduction of **1**, or alternatively of  $[\text{TaCp}^*\text{Cl}_4]$  in the presence of stoichiometric amounts of isocyanide, with two equivalents of Na/Hg gives the diamagnetic pseudo-octahedral tantalum(III) complex,  $[\text{TaCp}^*\text{Cl}_2(\text{CN-2,6-Me}_2\text{C}_6\text{H}_3)_3]$ , **5**. Reaction of **4** with two equivalents of  $\text{LiNEt}_2$  affords the pseudo-octahedral imido complex  $[\text{TaCp}^*\text{Cl}_2(\text{N-C}_6\text{H}_5)]$ , **6**. All the complexes were characterized by usual IR and NMR spectroscopic methods and the molecular structure of **1** was confirmed by X-ray diffraction methods.

**Keywords:** Tantalum; Pentamethylcyclopentadienyl; Isonitrile complexes; X-ray diffraction; Amine complexes; Imido complexes

## 1. Introduction

The Lewis acidity of niobium(V) and tantalum(V) compounds has been extensively studied, and a large number of adducts of niobium and tantalum halides and halo(alkyls) has been isolated [1]. The chemistry of cyclopentadienyl-niobium and tantalum complexes has attracted growing attention and many adducts of  $[\text{M-CpCl}_4\text{L}]$  have been reported [2], ( $\text{M} = \text{Nb}$  or  $\text{Ta}$ ;  $\text{Cp} = \eta^5\text{-C}_5\text{H}_5$  or  $\eta^5\text{-C}_5\text{Me}_5$ ;  $\text{L} = \text{THF}$ ,  $\text{DME}$ ,  $\text{P(OMe)}_3$ ,  $\text{PCy}_3$ , 2,2'-bipyridine,  $\text{MeCN}$ , or  $\text{PhCN}$ ). We reported [3] reactions with phosphorus ylides to give the neutral derivatives  $[\text{TaCp}^*\text{Cl}_4(\text{CH}_2\text{PR}_2\text{R}')]_n$ . When the base is  $\text{H}_2\text{O}$ , the reaction with  $[\text{MCp}^*\text{Cl}_4]$  ( $\text{M} = \text{Nb}$  [4] or  $\text{Ta}$  [2f–5]) takes place with immediate hydrolysis, leading to the mononuclear hydroxo complex and di- and trinuclear  $\mu$ -oxo metal derivatives.

As part of our interest in the chemistry of cyclopentadienyl derivatives, we report here the synthesis of new tantalum(V) complexes containing isocyanide,

amine and imido ligands, the reduction to the tantalum(III) derivative  $[\text{TaCp}^*\text{Cl}_2\{\text{CN}(2,6\text{-Me}_2\text{C}_6\text{H}_3)_3\}]$ , characterization of the new complexes by IR and NMR spectroscopy, and the molecular structure of  $[\text{TaCp}^*\text{Cl}_4(\text{CN-2,6-Me}_2\text{C}_6\text{H}_3)]$  determined by X-ray diffraction methods.

## 2. Results and discussion

Pseudo-octahedral adducts  $[\text{TaCp}^*\text{Cl}_4\text{L}]$ , ( $\text{L} = \text{CN-2,6-Me}_2\text{C}_6\text{H}_3$ , **1**;  $\text{CN-2,4,6-Me}_3\text{C}_6\text{H}_2$ , **2**;  $\text{CN}^t\text{Bu}$ , **3**;  $\text{NH}_2\text{C}_6\text{H}_5$ , **4**) can be easily prepared when  $\text{L}$  are added to a toluene suspension of  $[\text{TaCp}^*\text{Cl}_4]$  as shown in Scheme 1. Reduction of  $[\text{TaCp}^*\text{Cl}_4]$  or of complex **1**, previously isolated in the presence of the appropriate amount of the isocyanide with two equivalents of 10% sodium amalgam or  $\text{KC}_8$  [6] leads to the diamagnetic, pseudo-octahedral, 18-electron tantalum(III) complex,  $[\text{TaCp}^*\text{Cl}_2\{\text{CN}(2,6\text{-Me}_2\text{C}_6\text{H}_3)_3\}]$ , **5**. The reaction of **4** with two equivalents of  $\text{LiNEt}_2$  in toluene takes place with simultaneous elimination of  $\text{LiCl}$  and  $\text{NHEt}_2$ , giving the imidocomplex **6**,  $[\text{TaCp}^*\text{Cl}_2(\text{NC}_6\text{H}_5)]$ .

All the complexes **1–6** are extremely air- and mois-

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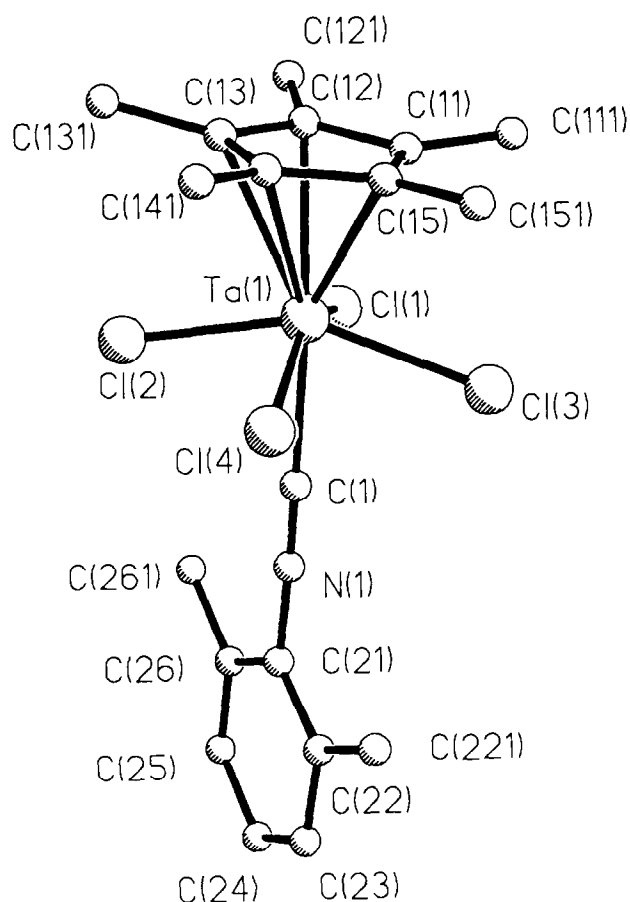


Fig. 1. Crystal structure of  $[\text{TaCp}^*\text{Cl}_4(\text{CN}-2,6\text{-Me}_2\text{C}_6\text{H}_3)]$  **1**, with the atom-numbering scheme.

gen atoms are displayed in Table 2. Selected bond distances and angles are given in Table 3.

The structure shows that the tantalum atom has a pseudo-octahedral coordination, similar to that found for the analogous niobium complex [8]. If the centroid of the Cp\* ring is considered as occupying one coordination site, the angles between the Cp centroid and the four chlorine atoms range from  $104.5^\circ$  to  $105.0^\circ$ , and the angles between C(1) and the chlorine atoms range from  $74.4^\circ$  to  $76.3^\circ$ . The chlorine atoms define a plane and the tantalum atom is located  $0.609 \text{ \AA}$  from it, towards the Cp\* ring.

The pentamethylcyclopentadienyl ring is pentahapto, the distance from tantalum to the centroid of the ring being  $2.166 \text{ \AA}$ , the distance to the plane defined by the cyclopentadienyl ring. This plane is parallel to the equatorial plane defined by the chlorine atoms.

The isocyanide is linear with Ta(1)–C(1)–N(1)  $178.8(6)^\circ$  and C(1)–N(1)–C(21)  $177.7(7)^\circ$  angles and Ta–C(1)  $2.234(7) \text{ \AA}$  and C(1)–N(1)  $1.139(9) \text{ \AA}$  bond distances, that confirm the C–N triple-bond character.

The phenyl ring is planar and perpendicular to the pentamethylcyclopentadienyl and chlorine planes, and

Table 2  
Table of positional parameters and their estimated standard deviations

Atom	x	y	z	B (Å <sup>2</sup> )
TA1	0.31439(3)	0.14392(1)	0.75055(3)	2.283(5)
CL1	0.0391(2)	0.14700(6)	0.4853(2)	4.46(4)
CL2	0.2088(2)	0.08204(5)	0.8113(2)	4.40(4)
CL3	0.4401(2)	0.18215(5)	0.5927(2)	4.47(4)
CL4	0.6169(2)	0.11904(6)	0.9162(2)	4.39(4)
C1	0.3544(8)	0.0998(2)	0.5766(7)	3.1(1)
N1	0.3727(7)	0.0769(2)	0.4883(6)	3.3(1)
C11	0.2434(8)	0.2121(2)	0.8073(7)	3.4(1)
C12	0.1187(7)	0.1867(2)	0.8336(7)	3.3(1)
C13	0.2154(8)	0.1623(2)	0.9766(7)	3.6(1)
C14	0.3967(9)	0.1717(2)	1.0412(7)	4.1(2)
C15	0.4173(8)	0.2025(2)	0.9381(8)	4.2(1)
C111	0.190(1)	0.2451(3)	0.679(1)	7.9(2)
C121	−0.085(1)	0.1885(3)	0.740(1)	6.9(3)
C131	0.136(1)	0.1350(3)	1.063(1)	7.3(2)
C141	0.542(1)	0.1556(3)	1.209(1)	8.3(3)
C151	0.588(1)	0.2250(3)	0.975(1)	8.0(2)
C21	0.3903(8)	0.0478(2)	0.3807(6)	2.8(1)
C22	0.5546(8)	0.0444(2)	0.3751(7)	3.5(1)
C23	0.5667(9)	0.0147(2)	0.2669(8)	4.5(2)
C24	0.420(1)	−0.0089(2)	0.1697(8)	4.6(2)
C25	0.258(1)	−0.0045(2)	0.1784(8)	4.3(2)
C26	0.2384(8)	0.0239(2)	0.2832(7)	3.3(1)
C221	0.7137(9)	0.0700(3)	0.485(1)	5.4(2)
C261	0.0647(9)	0.0292(3)	0.295(1)	5.3(2)

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as:  $(4/3)[a^2 B(1, 1) + b^2 B(2, 2) + c^2 B(3, 3) + ab(\cos \gamma) B(1, 2) + ac(\cos \beta) B(1, 3) + bc(\cos \alpha) B(2, 3)]$

is located in a staggered position with respect to the chlorine atoms.

### 3. Experimental details

Standard Schlenk procedures were used for all syntheses and sample manipulations. The solvents were dried by standard methods (hexane over Na/K alloy, and toluene over sodium) and distilled under argon

Table 3  
Selected bond distances (Å) and angles ( $^\circ$ )

Ta(1)–Cl(1)	2.405(1)	Cl(1)–Ta(1)–Cl(2)	87.81(6)
Ta(1)–Cl(2)	2.391(2)	Cl(1)–Ta(1)–Cl(3)	84.53(6)
Ta(1)–Cl(3)	2.424(2)	Cl(1)–Ta(1)–Cl(4)	150.54(7)
Ta(1)–Cl(4)	2.411(2)	Cl(1)–Ta(1)–C(1)	75.5(1)
Ta(1)–C(1)	2.234(7)	Cl(2)–Ta(1)–Cl(3)	150.70(7)
C(1)–N(1)	1.139(9)	Cl(2)–Ta(1)–Cl(4)	87.73(6)
N(1)–C(21)	1.399(8)	Cl(2)–Ta(1)–C(1)	76.3(2)
Ta(1)–Ce <sup>a</sup>	2.166	Cl(3)–Ta(1)–Cl(4)	85.26(6)
		Cl(3)–Ta(1)–C(1)	74.4(2)
		Cl(4)–Ta(1)–C(1)	75.1(1)
		Ta(1)–C(1)–N(1)	178.8(6)
		C(1)–N(1)–C(21)	177.7(7)

<sup>a</sup> CE is the centroid of the C(11)–C(15) ring. Numbers in parentheses are estimated standard deviations in the least significant digits.

before use. Reagent-grade chemicals purchased from commercial sources and used without further purification were as follows: carbon and aniline (Aldrich), sodium (Panreac), potassium and *tert*-butyl isocyanide (Fluka).  $[\text{TaCp}^*\text{Cl}_4]$  [10], 2,6- $\text{Me}_2\text{C}_6\text{H}_3\text{NC}$  and 2,4,6- $\text{Me}_3\text{C}_6\text{H}_2\text{NC}$  [11] and  $\text{LiNEt}_2$  [12] were prepared as described previously.

IR spectra were recorded on a Perkin-Elmer 583 spectrophotometer (4000–200  $\text{cm}^{-1}$ ) as Nujol mulls between CsI pellets.  $^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra were recorded on a Varian VXR-300 Unity instrument and chemical shifts are reported in  $\delta$  units (positive chemical shifts to a higher frequency) relative to a TMS standard. C, H and N analyses were performed with a Perkin-Elmer 240C microanalyzer.

### 3.1. Preparation of $[\text{TaCp}^*\text{Cl}_4]\text{L}$ , ( $\text{L} = 2,6\text{-Me}_2\text{C}_6\text{H}_3\text{NC}$ , **1**; 2,4,6- $\text{Me}_3\text{C}_6\text{H}_2\text{NC}$ , **2**; $^i\text{BuNC}$ , **3**; $\text{NH}_2\text{C}_6\text{H}_5$ , **4**)

A toluene solution (15 ml) of **L** (2.18 mmol) was added to a toluene suspension of  $[\text{TaCp}^*\text{Cl}_4]$  (1.00 g, 2.18 mmol) and the mixture was vigorously stirred for 30 min. at room temperature. The yellow or orange ( $\text{L} = \text{aniline}$ ) suspension was filtered and the solid residue washed twice with *n*-hexane ( $2 \times 10$  ml), dried in vacuo, and identified as **1–3** (yellow) or **4** (orange).

The data for the compounds are as follows. **1**: Yield 95% (1.22 g). Anal. Found: C, 38.56; H, 3.96; N, 2.40.  $\text{C}_{19}\text{H}_{24}\text{Cl}_4\text{NTa}$  (589.2). Calc.: C, 38.70; H, 4.00; N, 2.37%. IR: 2225m, 1650m, 1173w, 1020m, 800w, 776s, 722s, 640w, 600w, 470w, 420w, 360m  $\text{cm}^{-1}$ .

**2**: Yield 90% (1.18 g). Anal. Found: C, 39.71; H, 4.27; N, 2.08.  $\text{C}_{20}\text{H}_{26}\text{Cl}_4\text{NTa}$  (602.75). Calc.: C, 39.78; H, 4.47; N, 2.32%. IR: 2218m, 1603m, 1461s, 1196w, 1019m, 864m, 647w, 601w, 467s, 359m, 327m, 298s  $\text{cm}^{-1}$ .

**3**: Yield 80% (0.94 g). Anal. Found: C, 33.10; H, 4.18; N, 2.23.  $\text{C}_{15}\text{H}_{24}\text{Cl}_4\text{NTa}$  (540.75). Calc.: C, 33.25; H, 4.43; N, 2.58%. IR: 2241m, 1625m, 1460s, 1240m, 1200s, 1017m, 850m, 720m, 530m, 440w, 400m, 326vs, 298vs  $\text{cm}^{-1}$ .

**4**: Yield 90% (1.08 g). Anal. Found: C, 34.74; H, 3.93; N, 2.76.  $\text{C}_{16}\text{H}_{22}\text{Cl}_4\text{NTa}$  (551.33). Calc.: C, 34.82; H, 3.99; N, 2.64%. IR: 3308m, 3244m, 1598m, 1564m, 1269m, 1125s, 1018m, 805m, 475m, 397w, 363m, 307vs  $\text{cm}^{-1}$ .

### 3.2. Preparation of $[\text{TaCp}^*\text{Cl}_2\{\text{CN}(2,6\text{-Me}_2\text{C}_6\text{H}_3)\}_3]$ , **5**.

A toluene solution (50 ml) of 2,6- $\text{Me}_2\text{C}_6\text{H}_3\text{NC}$  (0.26 g, 1.96 mmol) was added to a mixture of  $[\text{TaCp}^*\text{Cl}_4]$  (0.30 g, 0.654 mmol) and 10% sodium amalgam (0.031 g, 1.31 mmol of Na). The mixture was stirred for 2 h at room temperature. The resulting red suspension was decanted and filtered through Celite, and the filtrate

Table 4

Crystal data, experimental data and structure refinement procedures for compound **1**

Formula	$\text{C}_{19}\text{H}_{24}\text{N}_1\text{Cl}_4\text{Ta}_1$
Crystal habit	Prismatic
Color	Yellow
Crystal size	$0.20 \times 0.12 \times 0.15$
Symmetry	Monoclinic $P2_1/c$
Unit cell determination	Least-squares fit from 25 reflections
Unit cell dimensions:	
$a, b, c, \text{Å}$	8.313(4), 33.322(4), 8.666(3)
$\beta$ deg	116.8(2)
$V, \text{Å}^3$	2142(2)
$Z$	4
$D_{\text{calc}}, \text{g cm}^{-3}$	1.827
$M_w$	589.17
$F(000)$	1144
$\mu \text{cm}^{-1}$	55.78
Scan mode	$\omega/2\theta$ scans; $\theta_{\text{max}} = 30^\circ$
No. of reflections: Measured	4130
Independent observed	3246 $I > 2\sigma(I)$ criterion
Range of $hkl$	$h$ 0–9; $k$ 0–39; $l$ –10–10
Standard reflections	2 reflections every 120 min, no variation
$R$	0.037
$R_w$	0.050
Max. peak in final diff. map, $\text{e Å}^{-3}$	1.81
Min. peak in final diff. map, $\text{e Å}^{-3}$	–1.17

was concentrated to ca. 15 ml, cooled to  $-40^\circ\text{C}$  to give **5** as red crystals. Yield 50% (0.26 g). Anal. Found: C, 57.66; H, 5.47; N, 5.00.  $\text{C}_{37}\text{H}_{42}\text{Cl}_2\text{N}_3\text{Ta}$  (779.85). Calc.: C, 56.93; H, 5.38; N, 5.38%. IR: 2089vs, 2022s, 1583m, 1302m, 1161m, 1081m, 1026m, 781s, 487m, 312w  $\text{cm}^{-1}$ .

### 3.3. Preparation of $[\text{TaCp}^*\text{Cl}_2(\text{N-C}_6\text{H}_5)]_6$ .

Toluene (50 ml) was added to a mixture of **4** (1.00 g, 1.8 mmol) and  $\text{LiNEt}_2$  (0.283 g, 3.6 mmol). After stirring overnight, the orange suspension was decanted and filtered through Celite. The filtrate was concentrated to ca. 20 ml and cooled to  $-40^\circ\text{C}$  overnight to give **6** as orange crystals. Yield 60% (0.51 g). Anal. Found: C, 39.99; H, 4.47; N, 2.76.  $\text{C}_{16}\text{H}_{20}\text{Cl}_2\text{NTa}$  (477.85). Calc.: C, 40.18; H, 4.18; N, 2.93%. IR: 1583m, 1348s, 1162m, 1024m, 975m, 890w, 688m, 502m, 434w, 378m, 341m  $\text{cm}^{-1}$ .

### 3.4. Crystallographic structural determination

Crystallographic and experimental details of X-ray crystal structure determination are given in Table 4. A suitable crystal of **1** was mounted on an Enraf-Nonius CAD-4 automatic four-circle diffractometer with bisecting geometry and using graphite-oriented monochromator with  $\text{Mo-K}\alpha$  radiation ( $\lambda_{\text{Mo-K}\alpha} = 0.71073 \text{ Å}$ ). Data were collected at room temperature.

Intensities were corrected for Lorentz and polarization effects in the usual manner. Absorption correction was made by the DIFABS method [13] and no extinction correction was made. The structure was solved by a combination of direct methods and Fourier synthesis and refined (on  $F$ ) by full-matrix least-squares calculations. All the non-hydrogen atoms were refined anisotropically. In the last cycle of refinement, the hydrogen atoms were introduced from geometrical calculations with thermal parameters equivalent to the one of the carbon to which they are attached.

Final values of  $R = 0.037$  and  $R_w = 0.050$  were obtained with  $R_w = [\sum \omega \{ |F_o| - |F_c| \}^2 / \omega |F_o|^2]^{1/2}$  and  $\omega = 4F_o^2 / [\sigma |F_o|^2]^2$ .

Anomalous dispersion corrections and atomic scattering factors were taken from international tables [14]. Calculations were performed with the SDP package [15], and the programs MULTAN [16] and DIRDIF [17] on a Microvax II computer. A full list of atomic coordinates, thermal parameters, bond lengths and angles have been deposited with the Cambridge Crystallographic Data Centre.

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