Crystal structure of bis(trichlorodiethylamidotantalum(V) isopropylate), $[\text{TaCl}_3(\text{C}_4\text{H}_{10}\text{N})(\text{C}_3\text{H}_7\text{O})]_2$

Stephan Peter Blöß, Jürgen Nuss and Martin Jansen*

Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, 70569 Stuttgart, Germany

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similar to the tantalum pentachloride dimer with two chlorine atoms forming the common edge. One of the equatorial chlorine atoms is substituted by a diethyl amido group whereas the isopropylate group is replacing a chlorine atom from the apical position. The formation of the isopropylate anion can be explained by ether fission under formation of isopropyl chloride. The crystal structure shows no disorder. The Ta—N bond length (1.931(3) Å) is in good agreement with those found for other Ta compounds (e.g., 1.912 Å [2]). As expected, the bond length between tantalum and covalently bonded oxygen of the isopropylate anion (1.818(3) Å) is much shorter than in tantalum ether compound (2.265(4) Å), cf. (diethyl ether)-tetrachlorodimethylamido-tantalum(V), $\text{TaCl}_4(\text{C}_2\text{H}_5\text{N})(\text{C}_4\text{H}_{10}\text{O})$ [2]. The other distances and angles fit very well the expected values.

Table 1. Data collection and handling.

Crystal:	honey-yellow block, size 0.30 × 0.30 × 0.40 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	88.99 cm ⁻¹
Diffractometer, scan mode:	Bruker AXS, SMART APEX I, ω
$2\theta_{\text{max}}$:	68.4°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	19746, 5389
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5187
$N(\text{param})_{\text{refined}}$:	169
Programs:	SHELXS-97 [3], SHELXL-97 [4], ATOMS [5]

Abstract

$\text{C}_{14}\text{H}_{34}\text{Cl}_6\text{N}_2\text{O}_2\text{Ta}_2$, triclinic, $P\bar{1}$ (no. 2), $a = 8.0083(6)$ Å, $b = 8.8582(7)$ Å, $c = 10.7360(8)$ Å, $\alpha = 67.809(1)^\circ$, $\beta = 86.039(2)^\circ$, $\gamma = 69.812(1)^\circ$, $V = 660.2$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.035$, $wR_{\text{ref}}(F^2) = 0.079$, $T = 100$ K.

Source of material

Following the published prescription [1], 230 μL of $\text{Me}_3\text{SiNEt}_2$ were added dropwise to a solution of 0.42 g TaCl_5 in 20 mL diisopropyl ether. The yellow solution was allowed to stir at room temperature for 1 h. Afterwards the solution was stored at -30°C for 4 weeks. A piece of honey-yellow crystal was selected from the cold mother solution, transferred in a high viscous oil, and mounted onto a nylon loop for X-ray diffraction.

Discussion

The title compound was prepared in the course of our studies on single-source precursors for TaON [1,2]. The characteristic building unit of the molecular structure is a bi-octahedral dimer,

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(11)	2i	0.324(8)	−0.287(8)	0.889(6)	0.025
H(12A)	2i	0.27(1)	−0.181(9)	0.644(8)	0.045
H(12B)	2i	0.46(1)	−0.31(1)	0.694(7)	0.045
H(12C)	2i	0.43(1)	−0.13(1)	0.639(8)	0.045
H(13A)	2i	0.58(1)	−0.14(1)	0.844(8)	0.046
H(13B)	2i	0.62(1)	−0.34(1)	0.911(7)	0.046
H(13C)	2i	0.53(1)	−0.22(1)	0.987(8)	0.046
H(21A)	2i	0.389(8)	0.185(8)	0.792(6)	0.028
H(21B)	2i	0.349(8)	0.380(8)	0.744(6)	0.028
H(22A)	2i	0.37(1)	0.22(1)	0.558(8)	0.050
H(22B)	2i	0.52(1)	0.28(1)	0.586(8)	0.050
H(22C)	2i	0.35(1)	0.41(1)	0.514(8)	0.050
H(23A)	2i	−0.092(9)	0.546(8)	0.647(6)	0.030
H(23B)	2i	0.089(8)	0.564(8)	0.608(7)	0.030
H(24A)	2i	−0.05(1)	0.55(1)	0.861(9)	0.058
H(24B)	2i	−0.04(1)	0.70(1)	0.734(9)	0.058
H(24C)	2i	0.15(1)	0.56(1)	0.839(8)	0.058

* Correspondence author (e-mail: M.Jansen@fkf.mpg.de)

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Ta	2i	0.03163(2)	0.14174(2)	0.80886(1)	0.01461(7)	0.01340(7)	0.01230(6)	−0.00426(5)	0.00168(4)	−0.00487(5)
Cl(1)	2i	0.0935(1)	0.1120(1)	1.04334(9)	0.0199(4)	0.0184(3)	0.0151(3)	−0.0093(3)	0.0020(3)	−0.0071(3)
Cl(2)	2i	−0.0808(1)	0.1266(1)	0.61824(9)	0.0267(4)	0.0242(4)	0.0158(4)	−0.0091(3)	−0.0017(3)	−0.0073(3)
Cl(3)	2i	−0.2612(1)	0.3301(1)	0.8222(1)	0.0177(4)	0.0199(4)	0.0239(4)	−0.0022(3)	0.0011(3)	−0.0080(3)
O(1)	2i	0.2376(4)	−0.0371(4)	0.8224(3)	0.018(1)	0.017(1)	0.018(1)	−0.005(1)	0.0032(9)	−0.008(1)
C(11)	2i	0.3656(6)	−0.2039(5)	0.8330(4)	0.020(2)	0.020(2)	0.020(2)	−0.003(1)	0.001(1)	−0.010(1)
C(12)	2i	0.3801(7)	−0.2095(7)	0.6934(5)	0.037(2)	0.028(2)	0.021(2)	−0.003(2)	0.002(2)	−0.014(2)
C(13)	2i	0.5401(6)	−0.2271(7)	0.8968(6)	0.017(2)	0.037(2)	0.038(3)	−0.004(2)	−0.002(2)	−0.019(2)
N(2)	2i	0.1324(5)	0.3235(4)	0.7302(3)	0.023(2)	0.020(1)	0.016(1)	−0.008(1)	0.003(1)	−0.008(1)
C(21)	2i	0.3277(6)	0.2904(6)	0.7251(4)	0.022(2)	0.031(2)	0.023(2)	−0.015(2)	0.005(1)	−0.012(2)
C(22)	2i	0.3914(7)	0.2986(8)	0.5866(5)	0.024(2)	0.051(3)	0.030(2)	−0.019(2)	0.012(2)	−0.017(2)
C(23)	2i	0.0299(6)	0.5135(5)	0.6775(5)	0.028(2)	0.018(2)	0.023(2)	−0.008(2)	−0.002(2)	−0.002(1)
C(24)	2i	0.0165(9)	0.5910(8)	0.7843(7)	0.046(3)	0.033(2)	0.047(3)	−0.015(2)	0.012(3)	−0.025(2)

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