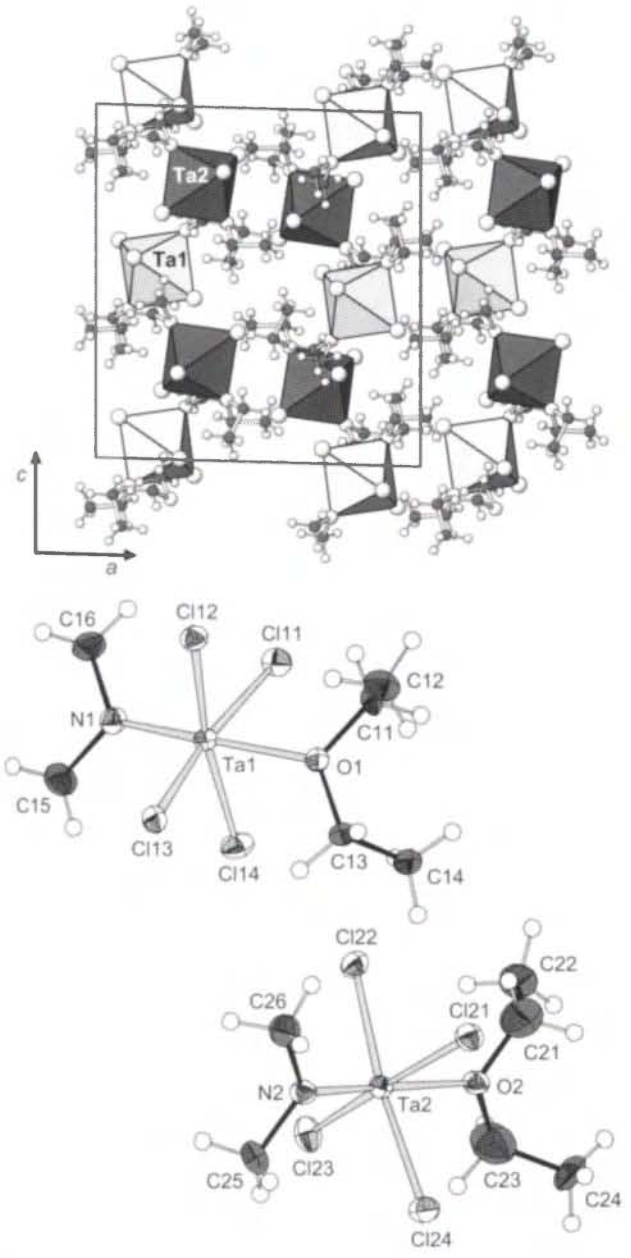
Z. Kristallogr. NCS 221 (2006) 209-210 / DOI 10.1524/ncrs.2006.0041

Crystal structure of (diethyl ether)-tetrachloro-dimethylamido-tantalum(V), $TaCl_4(C_2H_6N)(C_4H_{10}O)$

S. P. Bloß, J. Nuss and M. Jansen*

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

Received April 25, 2006, accepted and available on-line May 22, 2006; CCDC no. 1267/1772



Abstract

$C_6H_{16}Cl_4NOTa$, monoclinic, $P12_1/c1$ (no. 14), a = 13.975(2) Å, b = 12.494(2) Å, c = 15.318(2) Å, β = 91.717(3)°, V = 2673.4 Å^3, Z = 8, $R_{gt}(F)$ = 0.070, $wR_{ref}(F^2)$ = 0.171, T = 100 K.

* Correspondence author (e-mail: m.jansen@fkf.mpg.de)

Source of material

Following the published prescription [1], 0.380 mL of Me_3SiNMe_2 were added dropwise to a solution of 0.84 g $TaCl_5$ in 40 mL diethyl ether. The yellow solution was allowed to stir at room temperature for 2 h. Afterwards the solution was stored at –30 °C for 24 h. A broken piece of an orange, needle-shaped crystal, which was selected from the cold mother solution and dispersed in a high viscous oil, was mounted onto a nylon loop for X-ray diffraction measurement.

Experimental details

The studied crystal was systematically twinned: Rotational twin, rotation axis <001> or <100> (180°). The integration of the diffraction data was performed with two orientation matrices. The use of the approximately doubled data set in the refinement procedure led to enlarged numbers of total and observed reflections compared with a common structure determination (N(twin part 1) = 15858, N(twin part 2) = 15810, N(overlap twin parts 1+ 2) = 9526). The absorption correction was made with TWINABS [2]. The relationship of the two twin components is 0.6435(5)/ 0.3565.

Discussion

The title compound was prepared in the course of our investigations on single source precursors for TaON.

Due to packing effects, the asymmetric unit contains two crystallographically independent $TaCl_4(C_2H_6N)(C_4H_{10}O)$ molecules being nearly equal. Both show a characteristic octahedral surrounding of tantalum by four chlorine atoms, one dimethyl amido ligand, and one diethyl ether as conventional σ-donor ligand. The crystal structure does not show disorder, in contrast to the compound bearing a diethyl amido group instead of the dimethyl amido ligand [3]. The Ta—N and Ta—O bond lengths of 1.912(5) Å and 2.265(4) Å, respectively, for Ta1 and 1.889(5) Å and 2.273(4) Å, respectively, for Ta2 are in good agreement with those found for other Ta compounds (e.g. 1.895 Å and 2.213 Å, respectively, [4]). The remaining distances and angles fit very well to the expected values.

Table 1. Data collection and handling.

Crystal:	orange fragment, size 0.25 × 0.30 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	89.90 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, ω
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{total}$:	41194, 41194
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 31668
$N(param)_{refined}$:	244
Programs:	TWINABS [2], SHELXTL [5], ATOMS [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(11A)	4e	0.4013	0.6703	0.0516	0.033
H(11B)	4e	0.3749	0.5858	0.1254	0.033
H(12A)	4e	0.5633	0.6576	0.0880	0.053
H(12B)	4e	0.5241	0.5377	0.0786	0.053
H(12C)	4e	0.5421	0.5889	0.1734	0.053
H(13A)	4e	0.5220	0.8151	0.1397	0.033
H(13B)	4e	0.4502	0.8835	0.1971	0.033
H(14A)	4e	0.4243	0.8355	0.0162	0.059
H(14B)	4e	0.4394	0.9521	0.0576	0.059
H(14C)	4e	0.3416	0.8887	0.0718	0.059
H(15A)	4e	0.1343	0.8157	0.4341	0.057
H(15B)	4e	0.2418	0.8597	0.4357	0.057
H(15C)	4e	0.2095	0.7754	0.5077	0.057
H(16A)	4e	0.1949	0.5852	0.4690	0.052
H(16B)	4e	0.1863	0.5544	0.3676	0.052
H(16C)	4e	0.1063	0.6279	0.4102	0.052

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(21A)	4e	0.8832	0.8754	0.1117	0.085
H(21B)	4e	0.9009	0.7956	0.1915	0.085
H(22A)	4e	1.0505	0.8071	0.1863	0.059
H(22B)	4e	1.0181	0.9246	0.1561	0.059
H(22C)	4e	1.0487	0.8383	0.0849	0.059
H(23A)	4e	0.9328	0.5699	0.0609	0.079
H(23B)	4e	1.0176	0.6401	0.1034	0.079
H(24A)	4e	0.9596	0.6290	0.2356	0.059
H(24B)	4e	0.9404	0.5121	0.1961	0.059
H(24C)	4e	0.8549	0.5970	0.1993	0.059
H(25A)	4e	0.6333	0.6367	−0.1983	0.046
H(25B)	4e	0.7426	0.5982	−0.1943	0.046
H(25C)	4e	0.7068	0.6815	−0.2676	0.046
H(26A)	4e	0.6841	0.8600	−0.2391	0.051
H(26B)	4e	0.6984	0.9086	−0.1430	0.051
H(26C)	4e	0.6047	0.8401	−0.1678	0.051

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ta(1)	4e	0.31130(2)	0.71973(2)	0.29099(1)	0.0227(1)	0.0154(1)	0.0183(1)	−0.00006(8)	0.00067(8)	0.00075(8)
Cl(11)	4e	0.2038(1)	0.6304(1)	0.19475(9)	0.0237(7)	0.0267(7)	0.0287(7)	−0.0002(5)	−0.0045(6)	−0.0003(5)
Cl(12)	4e	0.3921(1)	0.5575(1)	0.31622(9)	0.0257(7)	0.0197(6)	0.0240(7)	0.0010(5)	−0.0026(5)	0.0022(5)
Cl(13)	4e	0.4365(1)	0.8106(1)	0.36640(8)	0.0331(8)	0.0271(7)	0.0190(6)	−0.0056(6)	−0.0008(6)	−0.0014(5)
Cl(14)	4e	0.2520(1)	0.8862(1)	0.2450(1)	0.0331(8)	0.0204(7)	0.0377(8)	0.0043(6)	0.0020(7)	0.0059(6)
O(1)	4e	0.4035(3)	0.7328(3)	0.1723(2)	0.027(2)	0.020(2)	0.019(2)	−0.006(2)	0.001(2)	−0.001(1)
C(11)	4e	0.4185(4)	0.6454(5)	0.1114(4)	0.041(4)	0.029(3)	0.014(3)	−0.007(3)	−0.002(3)	−0.005(2)
C(12)	4e	0.5212(4)	0.6037(5)	0.1130(5)	0.029(3)	0.031(3)	0.046(4)	0.002(3)	0.009(3)	−0.007(3)
C(13)	4e	0.4537(4)	0.8315(4)	0.1485(4)	0.035(3)	0.020(3)	0.026(3)	−0.008(2)	0.005(3)	0.000(2)
C(14)	4e	0.4110(5)	0.8813(5)	0.0663(4)	0.068(5)	0.025(3)	0.025(3)	−0.014(3)	−0.003(3)	0.001(2)
N(1)	4e	0.2300(3)	0.7075(4)	0.3887(3)	0.022(2)	0.023(2)	0.026(2)	0.000(2)	0.007(2)	0.001(2)
C(15)	4e	0.2014(5)	0.7973(5)	0.4465(4)	0.040(4)	0.034(4)	0.040(4)	0.005(3)	0.009(3)	−0.007(3)
C(16)	4e	0.1747(5)	0.6106(5)	0.4107(4)	0.036(4)	0.029(3)	0.040(4)	−0.009(3)	0.010(3)	0.002(3)
Ta(2)	4e	0.80414(2)	0.73800(2)	−0.04681(1)	0.0207(1)	0.0175(1)	0.0189(1)	0.00021(9)	−0.00002(8)	−0.00060(8)
Cl(21)	4e	0.6977(1)	0.8307(1)	0.04221(9)	0.0263(7)	0.0248(7)	0.0278(7)	0.0025(6)	0.0043(6)	−0.0022(5)
Cl(22)	4e	0.8843(1)	0.9010(1)	−0.06853(9)	0.0312(8)	0.0263(7)	0.0240(7)	−0.0070(6)	0.0007(6)	0.0034(5)
Cl(23)	4e	0.9290(1)	0.6465(1)	−0.1151(1)	0.0268(8)	0.0392(8)	0.0250(7)	0.0072(6)	0.0024(6)	−0.0047(6)
Cl(24)	4e	0.7419(1)	0.5731(1)	−0.0027(1)	0.0313(8)	0.0218(7)	0.0322(8)	−0.0013(6)	−0.0004(6)	0.0016(5)
O(2)	4e	0.8978(3)	0.7249(3)	0.0767(2)	0.024(2)	0.017(2)	0.024(2)	0.003(2)	−0.002(2)	−0.000(2)
C(21)	4e	0.9221(7)	0.8136(7)	0.1322(6)	0.105(8)	0.050(5)	0.057(6)	0.001(5)	−0.007(6)	0.011(4)
C(22)	4e	1.0169(5)	0.8485(6)	0.1405(5)	0.028(4)	0.043(4)	0.045(4)	−0.011(3)	−0.009(3)	−0.002(3)
C(23)	4e	0.9478(7)	0.6269(7)	0.1041(6)	0.078(6)	0.051(5)	0.067(6)	0.016(5)	−0.010(5)	−0.006(4)
C(24)	4e	0.9237(5)	0.5880(5)	0.1909(4)	0.048(4)	0.036(4)	0.035(4)	0.008(3)	0.009(3)	0.018(3)
N(2)	4e	0.7242(3)	0.7476(4)	−0.1481(3)	0.022(2)	0.021(2)	0.023(2)	−0.001(2)	−0.001(2)	−0.000(2)
C(25)	4e	0.6997(4)	0.6586(5)	−0.2069(4)	0.031(3)	0.036(3)	0.024(3)	0.001(3)	−0.001(3)	−0.009(3)
C(26)	4e	0.6735(5)	0.8477(5)	−0.1770(4)	0.037(4)	0.035(3)	0.031(3)	0.001(3)	−0.003(3)	0.001(3)

References

1. Chao, Y.-W.; Polson, S.; Wigley, D. E.: Preparation and properties of amido halide complexes of niobium and tantalum and their reactions with alkynes. Polyhedron **9** (1990) 2709-2716.
2. Sheldrick, G. M.: TWINABS. Bruker Nonius scaling and corrections for twinned crystals. Version 1.05. Bruker AXS, Madison, Wisconsin, USA 2004.
3. Blöß, S. P.; Nuss, J.; Jansen, M.: Crystal structure of diethyl ether-tetrachloro-diethylamido-tantalum(V), $TaCl_4(C_4H_{10}N)(C_4H_{10}O)$. Z. Kristallogr. NCS **220** (2005) 215-216.
4. Rivard, E.; Honeyman, C. H.; McWilliams, A.R.; Lough, A. J., Manners, I.: Synthesis and Reactivity of Perhalogenated Acyclic and Metallacyclic Tantalum(V) Phosphoraniminato Complexes: Discovery of an Unexpected Ligand Coupling Reaction to form the novel Phosphazenium Salt $[N(PCl_2NH_2)_2][TaCl_6]$. Inorg. Chem. **40** (2001) 1489-1495.
5. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.
6. Dowty, E.: ATOMS. A Complete Program for Displaying Atomic Structures. Version 6.2. Shape Software, Kingsport, Tennessee, USA 2005.