Crystal structure of pentakis(piperidyl)tantal(V), Ta(NC₅H₁₀)₅

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Abstract

C₂₅H₅₀N₅Ta, monoclinic, *P*12₁/*c*1 (No. 14), *a* = 15.3542(9) Å, *b* = 9.9150(6) Å, *c* = 18.141(1) Å, β = 108.109(1)°, *V* = 2624.9 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.053, *wR*_{ref}(*F*²) = 0.142, *T* = 100 K.

Source of material

Pentakis(piperidyl)tantal(V) was obtained by reacting tantalum pentachloride with lithiated piperidine in hexane [1]. Transparent red crystals were grown from hexane solution at 278 K. The compound is sensitive to air and moisture, so that it is to be handled under dry and oxygen-free argon.

Experimental details

All non-hydrogen atoms were refined anisotropically, hydrogen atoms were calculated isotropically with fixed positions.

Discussion

The coordination sphere of tantalum in Ta(NC₅H₁₀)₅ is square pyramidal as it has been observed in Ta[N(CH₃)₂]₅ [2] and Nb(NC₅H₁₀)₅ [3,4], the shortest distances are *d*(Ta—N1) = 197.5(4) pm, *d*(Ta—N2–5) in the range from 202.3(5) pm to 203.8(5) pm. The piperidine rings are in the common *armchair* conformation. Because of nitrogen–tantalum backbond interactions the nitrogen seeks for a trigonal planar coordination. Bond lengths are in good agreement with literature values [2,4].

Table 1. Data collection and handling.

Crystal:	red transparent block, size 0.1 × 0.2 × 0.4 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	42.08 cm ^{−1}
Diffractionmeter, scan mode:	Smart APEX (Bruker AXS), ω
2θ _{max} :	70.42°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	40945, 11119
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 9332
<i>N</i> (<i>param</i>) _{refined} :	280
Programs:	SHELXS-97 [5], SHELXL-97 [6], DIAMOND [7]

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(11A)	4e	0.8262	0.7485	−0.1002	0.156
H(11B)	4e	0.7620	0.6803	−0.0579	0.156
H(12A)	4e	0.9120	0.6363	0.0529	0.110
H(12B)	4e	0.8940	0.5392	−0.0198	0.110
H(13A)	4e	0.9609	0.7298	−0.0772	0.156
H(13B)	4e	1.0335	0.6428	−0.0130	0.156
H(14A)	4e	1.0487	0.8556	0.0469	0.201
H(14B)	4e	0.9804	0.7805	0.0825	0.201
H(15A)	4e	0.9152	1.0048	0.0192	0.078
H(15B)	4e	0.9063	0.9357	−0.0616	0.078
H(21A)	4e	0.6873	1.2249	0.0589	0.113
H(21B)	4e	0.6108	1.2444	−0.0208	0.113
H(22A)	4e	0.7045	1.4346	0.0023	0.127
H(22B)	4e	0.7923	1.3452	0.0151	0.127
H(23A)	4e	0.7561	1.4328	−0.1064	0.129
H(23B)	4e	0.6542	1.3836	−0.1262	0.129
H(24A)	4e	0.7302	1.2318	−0.1829	0.075
H(24B)	4e	0.8081	1.2111	−0.1036	0.075
H(25A)	4e	0.6233	1.1298	−0.1327	0.097
H(25B)	4e	0.7048	1.0298	−0.1243	0.097
H(31A)	4e	0.5346	1.0244	−0.0748	0.033
H(31B)	4e	0.4779	0.8979	−0.0651	0.033
H(32A)	4e	0.4362	0.9532	−0.1970	0.034
H(32B)	4e	0.5388	0.9461	−0.1957	0.034
H(33A)	4e	0.4704	0.7465	−0.2488	0.068
H(33B)	4e	0.4319	0.7230	−0.1790	0.068
H(34A)	4e	0.6199	0.7160	−0.1680	0.106
H(34B)	4e	0.5621	0.5897	−0.1594	0.106
H(35A)	4e	0.5437	0.6838	−0.0447	0.096
H(35B)	4e	0.6481	0.6627	−0.0349	0.096
H(41A)	4e	0.5690	0.7792	0.0732	0.078
H(41B)	4e	0.6125	0.7909	0.1635	0.078
H(42A)	4e	0.5560	0.5713	0.1364	0.096
H(42B)	4e	0.6086	0.5513	0.0757	0.096
H(43A)	4e	0.6911	0.5658	0.2383	0.142
H(43B)	4e	0.6905	0.4382	0.1868	0.142
H(44A)	4e	0.7854	0.5414	0.1292	0.144
H(44B)	4e	0.8311	0.5508	0.2194	0.144
H(45A)	4e	0.7828	0.7747	0.2191	0.164
H(45B)	4e	0.8402	0.7609	0.1615	0.164

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Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(51A)	4e	0.7398	0.9827	0.2066	0.072
H(51B)	4e	0.7594	1.1374	0.2045	0.072
H(52A)	4e	0.8560	1.0468	0.3190	0.074
H(52B)	4e	0.8940	0.9410	0.2723	0.074
H(53A)	4e	0.9996	1.1145	0.3086	0.204

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(53B)	4e	0.9232	1.2223	0.2714	0.204
H(54A)	4e	0.9805	1.0194	0.1838	0.298
H(54B)	4e	1.0045	1.1733	0.1818	0.298
H(55A)	4e	0.8425	1.2231	0.1294	0.236
H(55B)	4e	0.833	1.1310	0.0725	0.236

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Ta(1)	4e	0.72872(1)	0.92399(2)	0.032239(9)	0.01708(8)	0.01652(8)	0.01783(8)	0.00133(5)	0.00358(5)	−0.00361(5)
N(1)	4e	0.8256(3)	0.8438(5)	−0.0041(3)	0.029(2)	0.042(3)	0.029(2)	0.013(2)	0.009(2)	−0.006(2)
N(2)	4e	0.7016(3)	1.1046(4)	−0.0235(3)	0.020(2)	0.014(2)	0.056(3)	−0.000(1)	−0.006(2)	−0.001(2)
N(3)	4e	0.6105(3)	0.8576(4)	−0.0462(2)	0.037(2)	0.014(2)	0.021(2)	−0.004(1)	−0.009(2)	0.002(1)
N(4)	4e	0.7059(4)	0.7873(5)	0.1074(3)	0.052(3)	0.028(2)	0.025(2)	−0.018(2)	−0.016(2)	0.011(2)
N(5)	4e	0.7998(5)	1.0320(7)	0.1275(3)	0.090(5)	0.065(4)	0.025(2)	−0.056(4)	0.028(3)	−0.023(2)
C(11)	4e	0.8201(6)	0.726(1)	−0.0500(8)	0.044(5)	0.15(1)	0.20(2)	−0.030(6)	0.042(7)	−0.14(1)
C(12)	4e	0.9026(7)	0.6322(9)	−0.0025(8)	0.117(9)	0.038(4)	0.16(1)	0.033(5)	0.106(9)	0.029(6)
C(13)	4e	0.9791(8)	0.699(1)	−0.024(1)	0.107(9)	0.058(6)	0.29(2)	0.041(6)	0.15(1)	0.034(9)
C(14)	4e	0.9888(7)	0.814(2)	0.0349(7)	0.051(6)	0.38(3)	0.061(6)	0.10(1)	0.008(5)	0.00(1)
C(15)	4e	0.9083(7)	0.9196(9)	−0.0083(7)	0.059(5)	0.053(5)	0.082(7)	0.003(4)	0.021(5)	0.005(4)
C(21)	4e	0.6760(7)	1.2305(6)	0.0034(9)	0.070(5)	0.014(2)	0.24(2)	−0.003(3)	0.105(8)	−0.007(5)
C(22)	4e	0.7278(8)	1.3528(7)	−0.014(1)	0.115(8)	0.015(3)	0.25(2)	−0.017(4)	0.14(1)	−0.021(5)
C(23)	4e	0.7172(5)	1.3615(7)	−0.098(1)	0.032(3)	0.026(3)	0.26(2)	0.004(3)	0.042(6)	0.051(6)
C(24)	4e	0.7431(6)	1.2280(7)	−0.1271(5)	0.066(5)	0.035(3)	0.054(4)	−0.024(3)	−0.029(4)	0.022(3)
C(25)	4e	0.6878(7)	1.1148(8)	−0.1060(5)	0.090(6)	0.043(4)	0.056(4)	−0.043(4)	−0.053(4)	0.034(3)
C(31)	4e	0.5258(3)	0.9287(5)	−0.0857(3)	0.021(2)	0.035(2)	0.022(2)	−0.007(2)	−0.001(2)	0.002(2)
C(32)	4e	0.4941(4)	0.9074(5)	−0.1740(3)	0.028(2)	0.027(2)	0.021(2)	−0.005(2)	−0.004(2)	0.002(2)
C(33)	4e	0.4830(6)	0.7592(7)	−0.1934(4)	0.075(5)	0.030(3)	0.036(3)	−0.008(3)	−0.027(3)	−0.004(2)
C(34)	4e	0.5701(8)	0.6859(8)	−0.1498(5)	0.113(8)	0.032(3)	0.065(5)	0.025(4)	−0.051(5)	−0.029(3)
C(35)	4e	0.5938(8)	0.7135(6)	−0.0627(5)	0.116(7)	0.015(2)	0.053(4)	−0.009(3)	−0.054(5)	0.004(2)
C(41)	4e	0.6188(8)	0.7480(7)	0.1173(5)	0.127(8)	0.031(3)	0.068(5)	−0.025(4)	0.075(6)	−0.012(3)
C(42)	4e	0.6119(9)	0.5931(8)	0.1248(8)	0.126(9)	0.035(4)	0.121(9)	−0.028(5)	0.099(9)	−0.012(4)
C(43)	4e	0.693(1)	0.5360(9)	0.1880(6)	0.28(2)	0.032(4)	0.043(4)	−0.023(7)	0.055(8)	0.011(3)
C(44)	4e	0.780(1)	0.5814(8)	0.1763(7)	0.16(1)	0.036(4)	0.084(7)	−0.041(5)	−0.083(8)	0.036(4)
C(45)	4e	0.783(1)	0.7349(9)	0.1704(7)	0.18(1)	0.039(4)	0.092(7)	−0.044(6)	−0.108(8)	0.037(5)
C(51)	4e	0.7848(8)	1.0485(7)	0.2023(4)	0.129(8)	0.029(3)	0.028(3)	0.012(4)	0.033(4)	−0.004(2)
C(52)	4e	0.8708(7)	1.0322(9)	0.2713(4)	0.109(7)	0.057(4)	0.026(3)	−0.029(5)	0.030(4)	−0.016(3)
C(53)	4e	0.943(1)	1.131(2)	0.2669(5)	0.26(2)	0.20(2)	0.023(3)	−0.20(2)	0.009(7)	−0.015(6)
C(54)	4e	0.959(1)	1.110(3)	0.1876(5)	0.22(2)	0.48(4)	0.024(4)	−0.30(2)	0.009(7)	−0.015(9)
C(55)	4e	0.866(1)	1.134(2)	0.1230(5)	0.26(2)	0.28(2)	0.022(3)	−0.25(2)	0.011(7)	−0.005(7)

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