Crystal structure of bis(μ-oxo)hexakis(2,6-diisopropylphenoxy)-ditantalum(V), $Ta_2O_2(C_{12}H_{17}O)_6$

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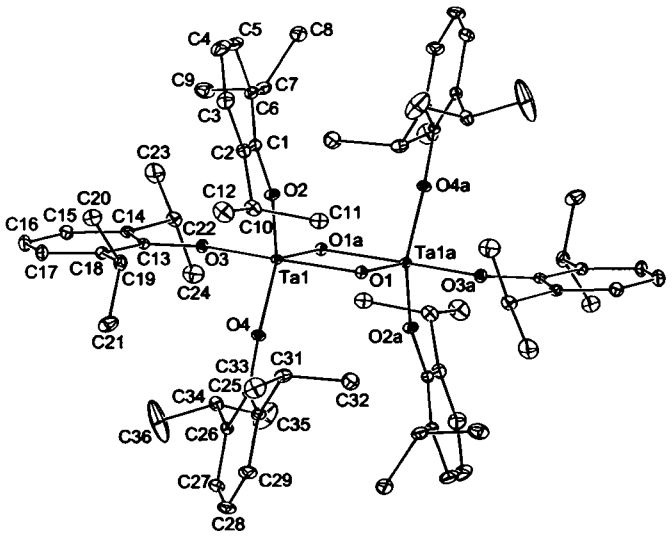

Abstract

$C_{72}H_{102}O_8Ta_2$, triclinic, $P\bar{1}$ (no. 2), a = 11.8613(1) Å, b = 13.2907(2) Å, c = 13.3311(2) Å, α = 113.2461(7)°, β = 97.4903(9)°, γ = 111.0447(8)°, V = 1709.1 Å^3, Z = 1, $R_{gt}(F)$ = 0.029, $wR_{ref}(F^2)$ = 0.066, T = 200 K.

Source of material

(η^4-2,3-Dimethylbutadiene)tris(2,6-diisopropylphenoxy)tantalum [1] in toluene was exposed to nitrogen gas which had been unknowingly contaminated from an outside source with air. If the exposure takes place slowly and without stirring, single crystals of the product form readily.

Discussion

The structural result reveals that the original Ta(III) aryloxide complex has picked up an oxygen atom and dimerized, leading to a planar four-membered Ta_2O_2 ring, located on a crystallographic center of inversion. As a result of the O1–Ta–O1′ angle being 78.80(8)°, the coordination geometry is somewhat irregular, but might best be described as trigonal bipyramidal, with O1 and O3 occupying the axial sites (∠O1–Ta–O3 = 175.15(7)°). The O–Ta–O′ angles in the equatorial plane are then 115.78(9)° (O1′, O2), 117.94(9)° (O1′, O4), and 123.97(9)° (O2, O4). However, there is a noticeable tilt of these equatorial ligands down from O3 toward O1, as indicated by the O3–Ta–O* angles of 96.35(8)°, 94.34(9)°, and 94.59(9)°, respectively, for O* = O1′, O2, and O4. This can be considered as a distortion toward a face-monocapped tetrahedral geometry, with O1 formally capping the tetrahedral face defined by O1′, O2, and O4. As might be expected from such a geometry, the Ta—O1 distance of 2.065(2) Å is particularly long relative to the other Ta—O distances of 1.850(2) Å, 1.872(2) Å, 1.890(2) Å, and 1.877(2) Å, respectively, for O1′, O2, O3, and O4.

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Table 1. Data collection and handling.

Crystal:	pale yellow prism, size 0.20 × 0.25 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	32.50 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	60.06°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13501, 9527
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 8726
$N(param)_{refined}$:	382
Programs:	SIR97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	−0.2580	0.1642	0.4402	0.071
H(4)	2*i*	−0.3027	−0.0061	0.4668	0.084
H(5)	2*i*	−0.2393	−0.1523	0.3672	0.074
H(7)	2*i*	−0.0796	−0.1489	0.1567	0.060
H(8A)	2*i*	−0.2190	−0.3085	0.2379	0.100
H(8B)	2*i*	−0.1679	−0.3483	0.1303	0.100
H(8C)	2*i*	−0.2791	−0.3074	0.1239	0.100
H(9A)	2*i*	0.0847	−0.0497	0.3311	0.102
H(9B)	2*i*	0.0526	−0.1915	0.2642	0.102
H(9C)	2*i*	0.0023	−0.1433	0.3707	0.102
H(10)	2*i*	−0.0479	0.2652	0.3021	0.057
H(11A)	2*i*	−0.2057	0.1240	0.1236	0.078
H(11B)	2*i*	−0.2046	0.2550	0.1647	0.078
H(11C)	2*i*	−0.3126	0.1493	0.1760	0.078
H(12A)	2*i*	−0.2460	0.3068	0.3851	0.112
H(12B)	2*i*	−0.1294	0.4045	0.3729	0.112
H(12C)	2*i*	−0.1041	0.3620	0.4669	0.112
H(15)	2*i*	0.5157	0.0912	0.4390	0.053
H(16)	2*i*	0.5647	0.2739	0.5996	0.063
H(17)	2*i*	0.4397	0.3740	0.6020	0.057
H(19)	2*i*	0.1637	0.2703	0.3674	0.053
H(20A)	2*i*	0.1246	0.2527	0.5278	0.088
H(20B)	2*i*	0.1237	0.3760	0.5366	0.088
H(20C)	2*i*	0.2449	0.3825	0.6125	0.088
H(21A)	2*i*	0.3752	0.5011	0.5200	0.095
H(21B)	2*i*	0.2480	0.4795	0.4381	0.095
H(21C)	2*i*	0.3438	0.4336	0.3832	0.095
H(22)	2*i*	0.2383	−0.0701	0.1913	0.051
H(23A)	2*i*	0.4392	−0.1008	0.2980	0.118
H(23B)	2*i*	0.3157	−0.2076	0.1909	0.118
H(23C)	2*i*	0.3017	−0.1418	0.3149	0.118
H(24A)	2*i*	0.3859	0.0418	0.1298	0.113
H(24B)	2*i*	0.3775	−0.0924	0.0844	0.113

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(24C)	2*i*	0.4953	0.0226	0.1921	0.113
H(27)	2*i*	0.5190	0.4898	0.1229	0.062
H(28)	2*i*	0.4411	0.6282	0.1321	0.074
H(29)	2*i*	0.2386	0.5904	0.1443	0.067
H(31)	2*i*	0.0310	0.3404	0.1770	0.055
H(32A)	2*i*	–0.0096	0.3976	–0.0057	0.095
H(32B)	2*i*	–0.1213	0.3241	0.0309	0.095
H(32C)	2*i*	–0.0330	0.2649	–0.0222	0.095
H(33A)	2*i*	0.1127	0.5543	0.3047	0.118

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(33B)	2*i*	–0.0362	0.4919	0.2371	0.118
H(33C)	2*i*	0.0656	0.5728	0.1980	0.118
H(34)	2*i*	0.3558	0.2178	0.1463	0.052
H(35A)	2*i*	0.4548	0.2415	–0.0271	0.184
H(35B)	2*i*	0.3110	0.1530	–0.0468	0.184
H(35C)	2*i*	0.4251	0.1295	0.0003	0.184
H(36A)	2*i*	0.5614	0.2680	0.2092	0.292
H(36B)	2*i*	0.5494	0.3917	0.2756	0.292
H(36C)	2*i*	0.6041	0.3711	0.1694	0.292

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)	2*i*	0.071070(9)	0.085730(8)	0.127084(8)	0.02702(6)	0.01770(5)	0.02462(6)	0.01104(4)	0.00979(4)	0.00968(4)
O(1)	2*i*	–0.0672(2)	0.0512(2)	–0.0102(2)	0.0323(9)	0.0247(9)	0.0286(9)	0.0183(8)	0.0093(7)	0.0106(8)
O(2)	2*i*	–0.0546(2)	0.0550(2)	0.1976(2)	0.043(1)	0.037(1)	0.039(1)	0.0193(9)	0.0233(9)	0.0218(9)
O(3)	2*i*	0.1990(2)	0.1058(2)	0.2441(2)	0.039(1)	0.028(1)	0.029(1)	0.0169(9)	0.0039(8)	0.0116(8)
O(4)	2*i*	0.1655(2)	0.2390(2)	0.1374(2)	0.036(1)	0.0235(9)	0.044(1)	0.0109(8)	0.0104(9)	0.0191(9)
C(1)	2*i*	–0.1227(3)	0.0364(3)	0.2695(2)	0.028(1)	0.041(2)	0.029(1)	0.010(1)	0.011(1)	0.016(1)
C(2)	2*i*	–0.1626(3)	0.1245(3)	0.3256(3)	0.033(2)	0.048(2)	0.038(2)	0.017(1)	0.016(1)	0.016(1)
C(3)	2*i*	–0.2301(4)	0.1058(4)	0.3997(3)	0.053(2)	0.082(3)	0.052(2)	0.035(2)	0.034(2)	0.030(2)
C(4)	2*i*	–0.2570(4)	0.0046(5)	0.4153(4)	0.068(3)	0.110(4)	0.065(3)	0.045(3)	0.048(2)	0.058(3)
C(5)	2*i*	–0.2183(4)	–0.0819(4)	0.3567(4)	0.066(2)	0.081(3)	0.065(2)	0.032(2)	0.038(2)	0.054(2)
C(6)	2*i*	–0.1487(3)	–0.0677(3)	0.2824(3)	0.040(2)	0.051(2)	0.037(2)	0.017(1)	0.015(1)	0.026(2)
C(7)	2*i*	–0.1012(4)	–0.1601(3)	0.2228(3)	0.063(2)	0.047(2)	0.053(2)	0.023(2)	0.024(2)	0.034(2)
C(8)	2*i*	–0.2007(4)	–0.2930(4)	0.1744(4)	0.081(3)	0.047(2)	0.055(2)	0.013(2)	0.012(2)	0.026(2)
C(9)	2*i*	0.0204(4)	–0.1338(4)	0.3045(4)	0.060(2)	0.061(3)	0.097(3)	0.028(2)	0.026(2)	0.049(3)
C(10)	2*i*	–0.1385(3)	0.2309(3)	0.3010(3)	0.040(2)	0.046(2)	0.056(2)	0.023(2)	0.023(2)	0.017(2)
C(11)	2*i*	–0.2229(4)	0.1858(4)	0.1805(3)	0.051(2)	0.054(2)	0.068(2)	0.032(2)	0.028(2)	0.033(2)
C(12)	2*i*	–0.1561(5)	0.3355(4)	0.3894(4)	0.080(3)	0.057(3)	0.080(3)	0.041(2)	0.037(3)	0.013(2)
C(13)	2*i*	0.2944(3)	0.1491(2)	0.3413(2)	0.031(1)	0.024(1)	0.029(1)	0.008(1)	0.007(1)	0.014(1)
C(14)	2*i*	0.3648(3)	0.0838(3)	0.3396(3)	0.034(1)	0.031(1)	0.039(2)	0.015(1)	0.010(1)	0.020(1)
C(15)	2*i*	0.4655(3)	0.1329(3)	0.4379(3)	0.037(2)	0.047(2)	0.051(2)	0.018(1)	0.007(1)	0.028(2)
C(16)	2*i*	0.4941(3)	0.2408(3)	0.5339(3)	0.047(2)	0.051(2)	0.043(2)	0.014(2)	–0.004(2)	0.019(2)
C(17)	2*i*	0.4202(3)	0.3008(3)	0.5346(3)	0.054(2)	0.038(2)	0.032(2)	0.015(2)	–0.002(1)	0.010(1)
C(18)	2*i*	0.3175(3)	0.2565(3)	0.4387(3)	0.043(2)	0.028(1)	0.032(1)	0.013(1)	0.009(1)	0.013(1)
C(19)	2*i*	0.2358(3)	0.3226(3)	0.4414(3)	0.057(2)	0.033(2)	0.031(2)	0.022(2)	0.007(1)	0.007(1)
C(20)	2*i*	0.1770(4)	0.3345(4)	0.5382(4)	0.069(2)	0.055(2)	0.062(2)	0.035(2)	0.030(2)	0.029(2)
C(21)	2*i*	0.3069(5)	0.4449(4)	0.4461(4)	0.099(3)	0.053(2)	0.064(2)	0.046(2)	0.038(2)	0.037(2)
C(22)	2*i*	0.3307(3)	–0.0347(3)	0.2336(3)	0.043(2)	0.032(2)	0.052(2)	0.021(1)	0.011(1)	0.018(1)
C(23)	2*i*	0.3484(5)	–0.1297(4)	0.2619(4)	0.099(3)	0.039(2)	0.093(3)	0.037(2)	0.011(3)	0.027(2)
C(24)	2*i*	0.4038(5)	–0.0139(4)	0.1528(4)	0.097(4)	0.059(3)	0.070(3)	0.039(3)	0.048(3)	0.020(2)
C(25)	2*i*	0.2400(3)	0.3434(2)	0.1349(2)	0.036(1)	0.019(1)	0.027(1)	0.005(1)	0.007(1)	0.010(1)
C(26)	2*i*	0.3623(3)	0.3642(3)	0.1279(2)	0.039(2)	0.032(2)	0.030(1)	0.008(1)	0.009(1)	0.011(1)
C(27)	2*i*	0.4354(3)	0.4726(3)	0.1273(3)	0.045(2)	0.043(2)	0.052(2)	0.005(2)	0.016(2)	0.022(2)
C(28)	2*i*	0.3896(4)	0.5552(3)	0.1331(4)	0.072(3)	0.033(2)	0.066(2)	0.004(2)	0.022(2)	0.027(2)
C(29)	2*i*	0.2692(4)	0.5324(3)	0.1403(3)	0.076(3)	0.028(2)	0.066(2)	0.020(2)	0.025(2)	0.028(2)
C(30)	2*i*	0.1910(3)	0.4263(3)	0.1420(3)	0.053(2)	0.028(1)	0.038(2)	0.018(1)	0.014(1)	0.018(1)
C(31)	2*i*	0.0573(3)	0.4009(3)	0.1474(3)	0.057(2)	0.040(2)	0.060(2)	0.030(2)	0.026(2)	0.032(2)
C(32)	2*i*	–0.0349(4)	0.3416(4)	0.0268(4)	0.058(2)	0.061(2)	0.071(3)	0.027(2)	0.013(2)	0.035(2)
C(33)	2*i*	0.0491(5)	0.5152(5)	0.2291(4)	0.100(4)	0.073(3)	0.080(3)	0.058(3)	0.045(3)	0.030(3)
C(34)	2*i*	0.4119(3)	0.2723(3)	0.1204(3)	0.034(2)	0.044(2)	0.045(2)	0.016(1)	0.010(1)	0.016(2)
C(35)	2*i*	0.3997(9)	0.1925(6)	0.0019(5)	0.254(9)	0.093(4)	0.063(3)	0.122(6)	0.062(4)	0.034(3)
C(36)	2*i*	0.5428(7)	0.3307(6)	0.2004(8)	0.124(6)	0.092(5)	0.216(9)	0.075(4)	–0.100(6)	–0.040(5)

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