Crystal structure of bis(2,6-di(phenylamino)pyridinium) hexachlorotantalate(V) chloride—toluene (1/1), $(C_{17}H_{16}N_3)_2(TaCl_6)Cl \cdot C_7H_8$

M. Polamo* and M. Hämäläinen

University of Helsinki, Department of Chemistry, Inorganic Chemistry Laboratory, P.O. Box 55 (A. I. Virtasen aukio 1), FIN-00014, Helsinki, Finland

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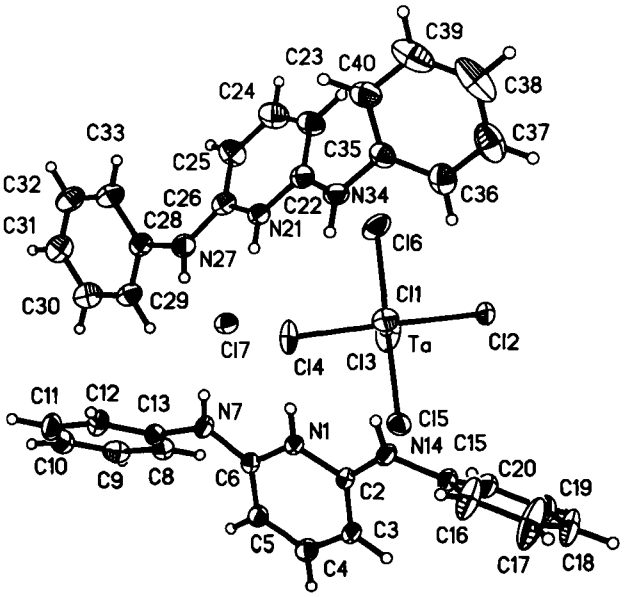

Abstract

$C_{41}H_{40}Cl_7N_6Ta$, triclinic, $P\bar{1}$ (No. 2), $a = 14.175(9)$ Å, $b = 14.376(6)$ Å, $c = 12.095(6)$ Å, $\alpha = 100.84(4)°$, $\beta = 110.62(4)°$, $\gamma = 102.01(5)°$, $V = 2162.5$ Å^3, $Z = 2$, $R_{gt}(F) = 0.034$, $wR_{ref}(F^2) = 0.100$, $T = 193$ K.

Source of material

The title compound was formed in the reaction of 2,6-di(phenylamino)pyridine and tantalum(V)chloride under reflux in toluene. Because of insufficient reaction time trichlorobis[2,6-di(phenylamino)pyridinato-*N*,*N*']tantalum [1] was obtained with bis(2,6-di(phenylamino)pyridinium) hexachlorotantalate(V) chloride (title compound). The title compound crystallised from a reaction mixture when toluene solution cooled to ambient temperature.

* Correspondence author (e-mail: 0505877924@mobiili.net)

Table 1. Data collection and handling.

Crystal:	red prism, size 0.20 × 0.35 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	30.11 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC7S, $\omega/2\theta$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	15888, 7605
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6814
$N(param)_{refined}$:	496
Programs:	SHELXS-86 [2], SHELXL-97 [3], SHELXTL-NT [4], teXsan [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.3190	0.4838	0.5140	0.042
H(3)	2*i*	0.0353	0.3248	0.2616	0.052
H(4)	2*i*	−0.0267	0.3391	0.4162	0.057
H(5)	2*i*	0.0837	0.4237	0.6189	0.053
H(7)	2*i*	0.3601	0.5252	0.7073	0.048
H(9)	2*i*	0.4076	0.4861	0.9096	0.060
H(10)	2*i*	0.3971	0.5268	1.0991	0.077
H(11)	2*i*	0.2629	0.5914	1.1165	0.073
H(12)	2*i*	0.1428	0.6169	0.9461	0.068
H(13)	2*i*	0.1534	0.5785	0.7550	0.055
H(14)	2*i*	0.3050	0.4226	0.3258	0.049
H(16)	2*i*	0.2556	0.2604	0.1459	0.095
H(17)	2*i*	0.1654	0.1973	−0.0673	0.134
H(18)	2*i*	0.0436	0.2664	−0.1758	0.093
H(19)	2*i*	−0.0020	0.3918	−0.0695	0.069
H(20)	2*i*	0.0880	0.4552	0.1439	0.061
H(21)	2*i*	0.5460	0.7050	0.6791	0.053
H(23)	2*i*	0.7026	0.9232	0.5958	0.072
H(24)	2*i*	0.7036	1.0319	0.7675	0.091
H(25)	2*i*	0.6307	0.9772	0.8942	0.078
H(27)	2*i*	0.5041	0.7208	0.8327	0.067
H(29)	2*i*	0.3798	0.7446	0.9269	0.069
H(30)	2*i*	0.3704	0.8027	1.1147	0.083
H(31)	2*i*	0.5118	0.9276	1.2732	0.083
H(32)	2*i*	0.6633	0.9909	1.2452	0.086
H(33)	2*i*	0.6794	0.9261	1.0632	0.076
H(34)	2*i*	0.5739	0.6652	0.5126	0.060
H(36)	2*i*	0.5276	0.6839	0.2880	0.081
H(37)	2*i*	0.6085	0.7033	0.1537	0.112
H(38)	2*i*	0.7927	0.7708	0.2329	0.141
H(39)	2*i*	0.8940	0.8235	0.4440	0.123
H(40)	2*i*	0.8126	0.8028	0.5802	0.082
H(42)	2*i*	0.0476	0.0633	0.1199	0.145
H(43)	2*i*	−0.0403	0.1494	0.0016	0.180
H(44)	2*i*	−0.1825	0.1944	0.0328	0.147
H(45)	2*i*	−0.2214	0.1651	0.1860	0.181
H(46)	2*i*	−0.1359	0.0630	0.3017	0.157
H(471)	2*i*	0.0634	−0.0201	0.2593	0.322
H(472)	2*i*	−0.0430	−0.0669	0.2735	0.322
H(473)	2*i*	0.0431	0.0312	0.3748	0.322

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	2*i*	0.24880(1)	0.75586(1)	0.42934(1)	0.0436(1)	0.0393(1)	0.0286(1)	0.02030(8)	0.01030(9)	0.00881(8)
Cl(1)	2*i*	0.1449(2)	0.8550(1)	0.4609(2)	0.122(1)	0.113(1)	0.0612(9)	0.093(1)	0.037(1)	0.0218(9)
Cl(2)	2*i*	0.2098(1)	0.77388(8)	0.2308(1)	0.0548(7)	0.0498(6)	0.0349(6)	0.0101(5)	0.0134(5)	0.0170(5)
Cl(3)	2*i*	0.34889(9)	0.65107(8)	0.3952(1)	0.0389(6)	0.0426(5)	0.0563(7)	0.0179(4)	0.0181(5)	0.0120(5)
Cl(4)	2*i*	0.2863(1)	0.7330(1)	0.6260(1)	0.089(1)	0.098(1)	0.0341(6)	0.0472(8)	0.0211(7)	0.0285(7)
Cl(5)	2*i*	0.09973(9)	0.6145(1)	0.3470(1)	0.0391(6)	0.0653(7)	0.0604(8)	0.0135(5)	0.0222(6)	0.0273(6)
Cl(6)	2*i*	0.3977(1)	0.89302(9)	0.5072(2)	0.076(1)	0.0400(6)	0.0665(9)	0.0014(6)	–0.0062(7)	0.0035(6)
Cl(7)	2*i*	0.50869(8)	0.54197(7)	0.6611(1)	0.0325(5)	0.0362(5)	0.0466(6)	0.0087(4)	0.0129(4)	0.0113(4)
N(1)	2*i*	0.2522(2)	0.4541(2)	0.4952(3)	0.026(2)	0.037(2)	0.029(2)	0.006(1)	0.008(1)	0.009(1)
C(2)	2*i*	0.1884(3)	0.4042(3)	0.3749(4)	0.033(2)	0.037(2)	0.027(2)	0.009(2)	0.008(2)	0.010(2)
C(3)	2*i*	0.0822(3)	0.3602(3)	0.3444(4)	0.029(2)	0.049(2)	0.032(2)	0.004(2)	0.005(2)	0.009(2)
C(4)	2*i*	0.0461(3)	0.3688(3)	0.4370(4)	0.028(2)	0.057(3)	0.044(3)	0.009(2)	0.013(2)	0.015(2)
C(5)	2*i*	0.1110(3)	0.4182(3)	0.5574(4)	0.033(2)	0.052(2)	0.036(2)	0.009(2)	0.015(2)	0.010(2)
C(6)	2*i*	0.2182(3)	0.4605(3)	0.5884(4)	0.035(2)	0.035(2)	0.030(2)	0.012(2)	0.012(2)	0.011(2)
N(7)	2*i*	0.2951(3)	0.5065(3)	0.7021(3)	0.031(2)	0.046(2)	0.029(2)	0.011(1)	0.009(1)	0.008(2)
C(8)	2*i*	0.2817(3)	0.5279(3)	0.8141(4)	0.043(2)	0.034(2)	0.032(2)	0.010(2)	0.015(2)	0.007(2)
C(9)	2*i*	0.3535(4)	0.5130(3)	0.9169(4)	0.052(3)	0.045(2)	0.035(2)	0.019(2)	0.009(2)	0.009(2)
C(10)	2*i*	0.3473(5)	0.5368(4)	1.0291(5)	0.078(4)	0.050(3)	0.032(2)	0.013(3)	0.008(2)	0.009(2)
C(11)	2*i*	0.2680(5)	0.5754(4)	1.0394(5)	0.078(4)	0.047(3)	0.041(3)	0.008(2)	0.031(3)	0.004(2)
C(12)	2*i*	0.1969(4)	0.5904(3)	0.9382(5)	0.056(3)	0.044(2)	0.060(3)	0.010(2)	0.035(3)	0.005(2)
C(13)	2*i*	0.2026(4)	0.5674(3)	0.8245(4)	0.047(2)	0.040(2)	0.042(2)	0.015(2)	0.018(2)	0.011(2)
N(14)	2*i*	0.2356(3)	0.4004(3)	0.2953(3)	0.027(2)	0.049(2)	0.026(2)	0.005(1)	0.005(1)	0.005(2)
C(15)	2*i*	0.1794(3)	0.3623(3)	0.1647(4)	0.031(2)	0.054(2)	0.027(2)	0.009(2)	0.009(2)	0.009(2)
C(16)	2*i*	0.2030(5)	0.2873(5)	0.1023(5)	0.061(3)	0.122(5)	0.033(3)	0.058(4)	0.008(2)	0.003(3)
C(17)	2*i*	0.1502(6)	0.2507(7)	–0.0244(6)	0.084(5)	0.180(8)	0.037(3)	0.082(5)	0.012(3)	–0.006(4)
C(18)	2*i*	0.0771(4)	0.2902(5)	–0.0884(5)	0.054(3)	0.126(5)	0.024(2)	0.031(3)	0.007(2)	0.011(3)
C(19)	2*i*	0.0513(4)	0.3658(4)	–0.0256(5)	0.050(3)	0.066(3)	0.039(3)	0.018(2)	0.010(2)	0.022(2)
C(20)	2*i*	0.1037(4)	0.4024(3)	0.1006(4)	0.046(3)	0.045(2)	0.040(2)	0.011(2)	0.008(2)	0.014(2)
N(21)	2*i*	0.5775(3)	0.7688(2)	0.6966(3)	0.039(2)	0.030(2)	0.046(2)	0.009(1)	0.008(2)	0.013(2)
C(22)	2*i*	0.6206(3)	0.7985(3)	0.6201(5)	0.037(2)	0.042(2)	0.053(3)	0.016(2)	0.009(2)	0.021(2)
C(23)	2*i*	0.6705(4)	0.8990(3)	0.6459(5)	0.058(3)	0.040(2)	0.075(4)	0.016(2)	0.029(3)	0.027(2)
C(24)	2*i*	0.6712(5)	0.9628(4)	0.7491(6)	0.084(4)	0.033(2)	0.091(5)	0.015(3)	0.036(4)	0.017(3)
C(25)	2*i*	0.6279(5)	0.9312(3)	0.8246(6)	0.079(4)	0.033(2)	0.075(4)	0.021(2)	0.036(3)	0.018(2)
C(26)	2*i*	0.5794(4)	0.8303(3)	0.7979(5)	0.044(3)	0.035(2)	0.055(3)	0.014(2)	0.011(2)	0.010(2)
N(27)	2*i*	0.5343(3)	0.7853(3)	0.8635(4)	0.060(3)	0.037(2)	0.046(2)	0.007(2)	0.014(2)	0.008(2)
C(28)	2*i*	0.5300(4)	0.8300(3)	0.9765(5)	0.049(3)	0.036(2)	0.050(3)	0.012(2)	0.012(2)	0.008(2)
C(29)	2*i*	0.4385(4)	0.7939(3)	0.9925(5)	0.046(3)	0.043(2)	0.058(3)	0.011(2)	0.013(2)	0.007(2)
C(30)	2*i*	0.4325(5)	0.8291(4)	1.1033(6)	0.060(3)	0.056(3)	0.076(4)	0.022(3)	0.030(3)	0.010(3)
C(31)	2*i*	0.5162(5)	0.9025(4)	1.1973(6)	0.072(4)	0.053(3)	0.062(4)	0.025(3)	0.025(3)	0.000(3)
C(32)	2*i*	0.6062(5)	0.9392(4)	1.1807(6)	0.062(3)	0.049(3)	0.063(4)	0.008(2)	0.013(3)	–0.007(3)
C(33)	2*i*	0.6155(4)	0.9023(4)	1.0722(5)	0.049(3)	0.049(3)	0.062(3)	0.011(2)	0.014(3)	0.004(2)
N(34)	2*i*	0.6124(3)	0.7258(3)	0.5251(4)	0.040(2)	0.038(2)	0.053(2)	0.006(2)	0.012(2)	0.015(2)
C(35)	2*i*	0.6629(4)	0.7422(3)	0.4442(5)	0.052(3)	0.041(2)	0.059(3)	0.020(2)	0.025(2)	0.025(2)
C(36)	2*i*	0.6020(5)	0.7121(4)	0.3188(5)	0.082(4)	0.048(3)	0.063(3)	0.026(3)	0.030(3)	0.022(2)
C(37)	2*i*	0.6494(7)	0.7231(5)	0.2401(7)	0.138(7)	0.069(4)	0.086(5)	0.047(4)	0.068(5)	0.039(4)
C(38)	2*i*	0.7593(9)	0.7640(6)	0.288(1)	0.17(1)	0.094(5)	0.152(9)	0.075(6)	0.132(9)	0.076(6)
C(39)	2*i*	0.8197(7)	0.7946(6)	0.413(1)	0.090(5)	0.090(5)	0.157(9)	0.043(4)	0.085(6)	0.069(6)
C(40)	2*i*	0.7721(4)	0.7832(4)	0.4937(6)	0.052(3)	0.057(3)	0.088(4)	0.017(2)	0.029(3)	0.033(3)
C(41)	2*i*	–0.0403(9)	0.0541(6)	0.219(1)	0.116(8)	0.066(5)	0.119(8)	–0.017(5)	–0.024(6)	0.035(5)
C(42)	2*i*	–0.0098(9)	0.0810(7)	0.132(1)	0.139(8)	0.084(5)	0.105(7)	–0.002(5)	0.075(7)	0.003(5)
C(43)	2*i*	–0.060(1)	0.1319(9)	0.064(1)	0.19(1)	0.117(8)	0.087(7)	–0.022(8)	0.073(8)	0.005(6)
C(44)	2*i*	–0.1445(9)	0.1604(6)	0.084(1)	0.107(7)	0.068(4)	0.107(8)	0.015(5)	–0.004(6)	0.005(5)
C(45)	2*i*	–0.1690(8)	0.141(1)	0.168(1)	0.076(6)	0.14(1)	0.14(1)	0.008(6)	0.037(7)	–0.061(8)
C(46)	2*i*	–0.1156(8)	0.0815(9)	0.2401(8)	0.071(5)	0.16(1)	0.070(5)	–0.039(6)	0.024(5)	–0.022(6)
C(47)	2*i*	0.010(1)	–0.0050(7)	0.287(1)	0.25(2)	0.068(5)	0.20(1)	–0.015(7)	–0.14(1)	0.049(7)

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