Crystal structures of acetonitrile solvates of bis(tetraphenylphosphonium) tetrachlorooxovanadate(IV), hexachlorostannate(IV) and -molybdate(IV), $[\text{P}(\text{C}_6\text{H}_5)_4]_2[\text{VOCl}_4] \cdot 4\text{CH}_3\text{CN}$, $[\text{P}(\text{C}_6\text{H}_5)_4]_2[\text{MCl}_6] \cdot 4\text{CH}_3\text{CN}$ ($M = \text{Sn}, \text{Mo}$)

Susanne Rabe^I, Wilfried Bubenheim^I and Ulrich Müller^{*,II}

^I Universität Kassel, Fachbereich Biologie-Chemie, D-34109 Kassel, Germany

^{II} Universität Marburg, Fachbereich Chemie, D-35032 Marburg, Germany

Received January 30, 2004, accepted and available on-line March 24, 2004; CSD no. 409762, 409763, 409764

Abstract

$\text{C}_{56}\text{H}_{52}\text{Cl}_4\text{N}_4\text{OP}_2\text{V}$, monoclinic, $P12_1/c1$ (no. 14), $a = 9.473(2)$ Å, $b = 19.832(6)$ Å, $c = 14.461(2)$ Å, $\beta = 99.00(2)^\circ$, $V = 2683.3$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.035$, $wR_{\text{ref}}(F^2) = 0.096$, $T = 193$ K.

$\text{C}_{56}\text{H}_{52}\text{Cl}_6\text{N}_4\text{P}_2\text{Sn}$, monoclinic, $P12_1/c1$ (no. 14), $a = 9.606(7)$ Å, $b = 19.842(9)$ Å, $c = 15.129(9)$ Å, $\beta = 98.91(2)^\circ$, $V = 2848.8$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.038$, $wR_{\text{ref}}(F^2) = 0.089$, $T = 300$ K.

$\text{C}_{56}\text{H}_{52}\text{Cl}_6\text{MoN}_4\text{P}_2$, monoclinic, $P12_1/c1$ (no. 14), $a = 9.505(3)$ Å, $b = 19.624(2)$ Å, $c = 14.759(2)$ Å, $\beta = 98.72(2)^\circ$, $V = 2721.1$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.062$, $wR_{\text{ref}}(F^2) = 0.175$, $T = 193$ K.

Source of materials

1.0 g of VS_4 and 0.5 mL of S_2Cl_2 were heated for 18 d to 673 K in a sealed glass ampoule. The obtained solid was stirred with a solution of 1.4 g of $\text{P}(\text{C}_6\text{H}_5)_4\text{Cl}$ in 50 mL of dry acetonitrile for 48 h at 343 K. After filtration, the filtrate was cooled first to 278 K (precipitation of sulfur) and then to 255 K, yielding green crystals of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{VOCl}_4] \cdot 4\text{CH}_3\text{CN}$.

1.5 g of $\text{P}(\text{C}_6\text{H}_5)_4\text{Cl}$ (4.0 mmol) and 0.52 g of SnCl_4 (2.0 mmol) were dissolved in 50 mL of dry acetonitrile. Upon evaporation of the solvent colorless crystals of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{SnCl}_6] \cdot 4\text{CH}_3\text{CN}$ were obtained.

For preparation of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{MoCl}_6] \cdot 4\text{CH}_3\text{CN}$ see [1].

Discussion

The square-pyramidal VOCl_4^{2-} ions are disordered in two orientations, the V atom being located close to an inversion center (split positions with half occupation for V and O). The superposition of the two orientations results in a pseudo-octahedral arrangement.

The octahedral arrangement is observed for the MCl_6^{2-} ions in the isotypical structures of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{MCl}_6] \cdot 4\text{CH}_3\text{CN}$ ($M = \text{Sn}, \text{Mo}$; with M atoms at 0,0,0 and Cl atoms at both O positions). The bond lengths are $\text{Sn}-\text{Cl}$ 2.438(2) Å, 2.440(2) Å, 2.418(2) Å and $\text{Mo}-\text{Cl}$ 2.382(2) Å, 2.395(2) Å, 2.270(2) Å. The same structure is also known for the corresponding hexachloro complexes of zirconium [2], tungsten [3], and rhenium [4]. The chemical formula given in [4] is not correct (with 2 instead of 4 CH_3CN molecules per formula unit).

When a crystal of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{VOCl}_4] \cdot 4\text{CH}_3\text{CN}$ (Pearson symbol $mP240$) is slowly (2 h) heated to 333 K, it loses acetonitrile. In a topotactic reaction involving a volume contraction of 10.3 % a triclinic crystal of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{VOCl}_4] \cdot 2\text{CH}_3\text{CN}$ [5] is formed. $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{VOCl}_4] \cdot 4\text{CH}_3\text{CN}$ also forms a triclinic modification $aP240$ [5].

* Correspondence author (e-mail: mueller@chemie.uni-marburg.de)

**1. Bis(tetraphenylphosphonium) tetrachlorooxovanadate(IV)
tetraacetonitrile solvate, [P(C₆H₅)₄]₂[VOCl₄] · 4CH₃CN**

Table 1. Data collection and handling.

Crystal:	green needle, size 0.36 × 0.52 × 1.16 mm
Wavelength:	Mo K _α radiation (0.71073 Å)
μ:	4.86 cm ⁻¹
Diffraction, scan mode:	Nonius CAD4, ω
2θ _{max} :	50.02°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	4902, 4703
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 4201
<i>N</i> (<i>param</i>) _{refined} :	311
Programs:	SHELX-97 [6], DIAMOND [7]

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(22)	4e	0.7598	0.3110	0.5498	0.044
H(23)	4e	0.6690	0.4144	0.5845	0.054
H(24)	4e	0.4371	0.4436	0.5234	0.054
H(25)	4e	0.2979	0.3715	0.4227	0.049
H(26)	4e	0.3888	0.2693	0.3822	0.039
H(32)	4e	0.5713	0.2000	0.6161	0.048
H(33)	4e	0.6459	0.1466	0.7573	0.059
H(34)	4e	0.8420	0.0773	0.7743	0.064
H(35)	4e	0.9611	0.0568	0.6497	0.061
H(36)	4e	0.8827	0.1066	0.5047	0.046
H(42)	4e	0.7648	0.1130	0.3080	0.046
H(43)	4e	0.9481	0.1196	0.2205	0.054
H(44)	4e	1.0863	0.2160	0.2262	0.052
H(45)	4e	1.0450	0.3050	0.3204	0.047
H(46)	4e	0.8587	0.2999	0.4074	0.038
H(2A)	4e	0.3536	0.4970	0.2897	0.131(8)
H(2B)	4e	0.4897	0.4912	0.2410	0.131
H(2C)	4e	0.4111	0.4258	0.2665	0.131
H(4A)	4e	0.2001	0.3163	-0.0145	0.16(1)
H(4B)	4e	0.0680	0.3084	0.0376	0.16
H(4C)	4e	0.1561	0.2447	0.0171	0.16

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(12)	4e	0.5155	0.1913	0.2464	0.040
H(13)	4e	0.3214	0.1332	0.1677	0.045
H(14)	4e	0.2009	0.0566	0.2458	0.048
H(15)	4e	0.2734	0.0367	0.4035	0.052
H(16)	4e	0.4688	0.0936	0.4839	0.043

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

Atom	Site	Occ.	<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
V	4e	0.5	0.05169(6)	-0.00582(3)	0.01773(4)	0.0209(3)	0.0265(3)	0.0196(3)	0.0017(2)	0.0018(2)	0.0021(2)
Cl(1)	4e		0.09694(5)	0.01232(2)	-0.13536(3)	0.0436(3)	0.0417(3)	0.0316(3)	-0.0005(2)	0.0144(2)	0.0032(1)
Cl(2)	4e		0.04386(5)	0.11165(2)	0.03457(3)	0.0411(3)	0.0311(3)	0.0420(3)	-0.0034(2)	0.0108(2)	-0.0021(1)
O	4e	0.5	0.2061(3)	-0.0247(1)	0.0750(2)	0.0251(2)	0.044(2)	0.027(1)	0.003(1)	0.001(1)	-0.001(1)
P	4e		0.65307(5)	0.19845(2)	0.43650(3)	0.0223(2)	0.0283(3)	0.0255(2)	-0.0014(2)	0.0027(2)	-0.0016(2)
C(11)	4e		0.5111(2)	0.1487(1)	0.3735(1)	0.0233(8)	0.0272(9)	0.0307(9)	0.0000(7)	0.0028(7)	-0.0050(7)
C(12)	4e		0.4670(2)	0.1603(1)	0.2782(1)	0.032(1)	0.036(1)	0.032(1)	0.0018(8)	0.0027(8)	-0.0006(8)
C(13)	4e		0.3510(2)	0.1256(1)	0.2312(1)	0.033(1)	0.042(1)	0.034(1)	0.0075(8)	-0.0046(8)	-0.0080(8)
C(14)	4e		0.2790(2)	0.0797(1)	0.2779(2)	0.029(1)	0.040(1)	0.049(1)	-0.0022(8)	-0.0019(9)	-0.0159(9)
C(15)	4e		0.3225(2)	0.0678(1)	0.3723(2)	0.039(1)	0.044(1)	0.048(1)	-0.0144(9)	0.0084(9)	-0.008(1)
C(16)	4e		0.4390(2)	0.1019(1)	0.4205(1)	0.037(1)	0.037(1)	0.032(1)	-0.0068(8)	0.0042(8)	-0.0032(8)
C(21)	4e		0.5834(2)	0.2795(1)	0.4624(1)	0.0270(9)	0.0322(9)	0.0287(9)	-0.0021(7)	0.0079(7)	-0.0022(7)
C(22)	4e		0.6674(2)	0.3232(1)	0.5236(1)	0.030(1)	0.040(1)	0.041(1)	-0.0052(8)	0.0055(8)	-0.0101(9)
C(23)	4e		0.6126(2)	0.3847(1)	0.5449(2)	0.049(1)	0.040(1)	0.049(1)	-0.0113(9)	0.014(1)	-0.015(1)
C(24)	4e		0.4741(2)	0.4024(1)	0.5077(2)	0.055(1)	0.032(1)	0.053(1)	0.0029(9)	0.023(1)	-0.0030(9)
C(25)	4e		0.3909(2)	0.3593(1)	0.4476(2)	0.038(1)	0.040(1)	0.045(1)	0.0084(9)	0.0102(9)	0.0034(9)
C(26)	4e		0.4448(2)	0.2980(1)	0.4238(1)	0.0294(9)	0.035(1)	0.034(1)	-0.0016(8)	0.0053(8)	-0.0014(8)
C(31)	4e		0.7196(2)	0.1589(1)	0.5463(1)	0.0292(9)	0.0314(9)	0.0291(9)	-0.0073(7)	-0.0023(7)	0.0011(7)
C(32)	4e		0.6490(2)	0.1708(1)	0.6224(1)	0.041(1)	0.046(1)	0.032(1)	-0.0090(9)	0.0030(8)	-0.0021(9)
C(33)	4e		0.6944(3)	0.1394(1)	0.7070(2)	0.062(1)	0.051(1)	0.031(1)	-0.017(1)	0.001(1)	0.0009(9)
C(34)	4e		0.8106(3)	0.0977(1)	0.7168(2)	0.074(2)	0.044(1)	0.037(1)	-0.019(1)	-0.011(1)	0.009(1)
C(35)	4e		0.8822(3)	0.0853(1)	0.6421(2)	0.049(1)	0.033(1)	0.063(2)	-0.0030(9)	-0.016(1)	0.008(1)
C(36)	4e		0.8363(2)	0.1154(1)	0.5554(2)	0.036(1)	0.032(1)	0.044(1)	-0.0041(8)	-0.0019(9)	0.0017(8)
C(41)	4e		0.7932(2)	0.20583(9)	0.3666(1)	0.0224(8)	0.034(1)	0.0285(9)	0.0014(7)	0.0031(7)	0.0027(7)
C(42)	4e		0.8205(2)	0.1518(1)	0.3103(1)	0.034(1)	0.038(1)	0.044(1)	-0.0001(8)	0.0102(8)	-0.0064(9)
C(43)	4e		0.9301(2)	0.1557(1)	0.2581(2)	0.039(1)	0.055(1)	0.043(1)	0.006(1)	0.0127(9)	-0.008(1)
C(44)	4e		1.0129(2)	0.2133(1)	0.2618(2)	0.032(1)	0.059(1)	0.041(1)	0.0077(9)	0.0142(9)	0.010(1)
C(45)	4e		0.9877(2)	0.2667(1)	0.3177(1)	0.029(1)	0.045(1)	0.044(1)	-0.0022(8)	0.0056(8)	0.0118(9)
C(46)	4e		0.8767(2)	0.2636(1)	0.3702(1)	0.0278(9)	0.034(1)	0.0324(9)	0.0010(8)	0.0032(7)	0.0025(8)
N(1)	4e		0.2381(5)	0.4749(2)	0.0849(2)	0.203(4)	0.067(2)	0.065(2)	0.021(2)	0.037(2)	0.010(2)
C(1)	4e		0.3096(4)	0.4734(1)	0.1556(2)	0.115(3)	0.037(1)	0.066(2)	0.005(1)	0.051(2)	0.004(1)
C(2)	4e		0.3983(2)	0.47173(6)	0.2456(1)	0.066(2)	0.056(2)	0.087(2)	-0.009(1)	0.040(2)	-0.009(1)
N(2)	4e		0.3259(2)	0.30601(6)	0.1904(1)	0.080(2)	0.060(1)	0.069(2)	-0.019(1)	-0.016(1)	0.015(1)
C(3)	4e		0.2532(2)	0.30034(6)	0.1214(1)	0.041(1)	0.037(1)	0.058(1)	-0.0054(9)	0.006(1)	0.005(1)
C(4)	4e		0.1618(2)	0.29171(6)	0.0331(1)	0.052(1)	0.067(2)	0.059(2)	0.004(1)	-0.002(1)	-0.008(1)

2. Bis(tetraphenylphosphonium) hexachlorostannate(IV) tetraacetonitrile solvate, [P(C₆H₅)₄]₂[SnCl₆] · 4CH₃CN

Table 4. Data collection and handling.

Crystal:	colorless needle, size 0.30 × 0.44 × 0.96 mm
Wavelength:	Mo K α radiation (0.71071 Å)
μ :	8.25 cm ⁻¹
Diffractionmeter, scan mode:	Nonius CAD4, ω
$2\theta_{\max}$:	47.96°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	4750, 4457
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2684
$N(\text{param})_{\text{refined}}$:	317
Program:	SHELX-97 [6]

Table 5. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(12)	4e	0.5146	0.1884	0.2573	0.093
H(13)	4e	0.3188	0.1308	0.1874	0.106
H(14)	4e	0.2052	0.0556	0.2654	0.108
H(15)	4e	0.2870	0.0346	0.4147	0.111
H(16)	4e	0.4826	0.0908	0.4851	0.089

Table 5. Continued.

Atom	Site	x	y	z	U_{iso}
H(22)	4e	0.7562	0.2993	0.5627	0.096
H(23)	4e	0.6635	0.3989	0.6048	0.122
H(24)	4e	0.4443	0.4332	0.5399	0.126
H(25)	4e	0.3119	0.3684	0.4331	0.120
H(26)	4e	0.4050	0.2695	0.3857	0.099
H(32)	4e	0.6043	0.1860	0.6163	0.113
H(33)	4e	0.7093	0.1287	0.7440	0.152
H(34)	4e	0.9050	0.0656	0.7393	0.161
H(35)	4e	0.9964	0.0517	0.6101	0.151
H(36)	4e	0.8906	0.1058	0.4797	0.109
H(42)	4e	0.7653	0.1182	0.3036	0.100
H(43)	4e	0.9499	0.1303	0.2231	0.127
H(44)	4e	1.0784	0.2280	0.2348	0.132
H(45)	4e	1.0343	0.3129	0.3308	0.116
H(46)	4e	0.8464	0.3017	0.4103	0.085
H(2A)	4e	0.4595	0.4146	0.2675	0.22(3)
H(2B)	4e	0.3725	0.4710	0.3067	0.22
H(2C)	4e	0.5095	0.4898	0.2677	0.22
H(4A)	4e	0.1819	0.3364	0.0057	0.22(3)
H(4B)	4e	0.0866	0.2865	0.0499	0.22
H(4C)	4e	0.2038	0.2584	-0.0017	0.22

Table 6. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn	2a	0	0	0	0.0353(2)	0.0483(3)	0.0320(2)	0.0015(3)	0.0014(2)	0.0043(3)
Cl(1)	4e	0.1197(1)	0.01407(7)	-0.12960(7)	0.0593(8)	0.0908(9)	0.0456(7)	0.0048(7)	0.0179(6)	0.0137(7)
Cl(2)	4e	0.0513(1)	0.11677(6)	0.04396(9)	0.0654(9)	0.0526(8)	0.0694(8)	-0.0050(7)	0.0044(7)	-0.0053(7)
Cl(3)	4e	0.2191(1)	-0.03456(7)	0.08940(8)	0.0446(7)	0.0812(9)	0.0491(7)	0.0125(7)	-0.0064(6)	0.0056(7)
P	4e	0.6587(1)	0.19527(6)	0.43684(8)	0.0414(7)	0.0507(8)	0.0423(7)	-0.0014(6)	0.0008(6)	0.0031(6)
C(11)	4e	0.5175(5)	0.1459(2)	0.3799(3)	0.040(3)	0.045(3)	0.049(3)	0.000(2)	-0.002(2)	-0.005(2)
C(12)	4e	0.4688(5)	0.1577(3)	0.2893(3)	0.059(3)	0.067(4)	0.056(3)	0.000(3)	-0.004(3)	0.001(3)
C(13)	4e	0.3518(6)	0.1232(3)	0.2476(4)	0.064(4)	0.080(4)	0.061(4)	0.002(3)	-0.013(3)	-0.013(3)
C(14)	4e	0.2844(6)	0.0782(3)	0.2940(4)	0.054(4)	0.067(4)	0.090(5)	-0.009(3)	-0.008(3)	-0.023(4)
C(15)	4e	0.3329(6)	0.0657(3)	0.3833(4)	0.071(4)	0.067(4)	0.083(4)	-0.021(3)	0.006(3)	-0.006(3)
C(16)	4e	0.4493(5)	0.0994(3)	0.4252(3)	0.059(3)	0.060(4)	0.057(3)	-0.012(3)	0.000(3)	-0.004(3)
C(21)	4e	0.5895(5)	0.2733(2)	0.4700(3)	0.047(3)	0.054(3)	0.048(3)	-0.006(3)	0.014(2)	0.001(2)
C(22)	4e	0.6670(6)	0.3130(3)	0.5359(3)	0.059(3)	0.069(4)	0.065(3)	-0.011(3)	0.016(3)	-0.010(3)
C(23)	4e	0.6115(8)	0.3723(3)	0.5610(4)	0.101(6)	0.064(4)	0.084(5)	-0.012(4)	0.035(4)	-0.024(3)
C(24)	4e	0.4804(9)	0.3927(3)	0.5223(4)	0.111(6)	0.058(4)	0.093(5)	0.011(4)	0.049(5)	0.001(4)
C(25)	4e	0.4018(7)	0.3546(3)	0.4582(4)	0.068(4)	0.074(5)	0.102(5)	0.014(4)	0.026(4)	-0.002(4)
C(26)	4e	0.4570(6)	0.2950(3)	0.4307(4)	0.065(4)	0.066(4)	0.066(3)	0.005(3)	0.008(3)	-0.002(3)
C(31)	4e	0.7396(5)	0.1519(2)	0.5359(3)	0.057(3)	0.053(3)	0.045(3)	-0.014(3)	-0.006(3)	0.012(2)
C(32)	4e	0.6831(6)	0.1590(3)	0.6147(3)	0.086(4)	0.088(4)	0.049(3)	-0.024(4)	0.001(3)	0.013(3)
C(33)	4e	0.7463(9)	0.1251(4)	0.6909(4)	0.134(7)	0.097(6)	0.061(4)	-0.054(5)	-0.026(5)	0.017(4)
C(34)	4e	0.862(1)	0.0870(4)	0.6874(5)	0.124(8)	0.085(6)	0.090(6)	-0.043(5)	-0.056(6)	0.034(5)
C(35)	4e	0.9175(7)	0.0788(3)	0.6107(6)	0.083(5)	0.081(5)	0.122(6)	-0.011(4)	-0.032(5)	0.035(5)
C(36)	4e	0.8549(6)	0.1114(3)	0.5330(4)	0.062(4)	0.064(4)	0.084(4)	-0.010(3)	-0.011(3)	0.021(3)
C(41)	4e	0.7885(5)	0.2082(3)	0.3657(3)	0.043(3)	0.057(3)	0.044(3)	0.002(3)	0.002(2)	-0.001(2)
C(42)	4e	0.8190(6)	0.1574(3)	0.3091(3)	0.056(4)	0.077(4)	0.068(4)	0.003(3)	0.010(3)	-0.002(3)
C(43)	4e	0.9287(7)	0.1647(4)	0.2606(4)	0.073(4)	0.108(6)	0.076(4)	0.016(4)	0.026(4)	-0.008(4)
C(44)	4e	1.0059(7)	0.2228(4)	0.2684(4)	0.061(4)	0.133(7)	0.075(5)	0.015(4)	0.026(4)	0.027(4)
C(45)	4e	0.9790(6)	0.2742(3)	0.3250(4)	0.066(4)	0.095(5)	0.075(4)	-0.008(4)	0.021(3)	0.015(4)
C(46)	4e	0.8677(5)	0.2671(3)	0.3732(3)	0.050(3)	0.065(4)	0.057(3)	0.002(3)	0.012(3)	0.010(3)
N(1)	4e	0.2827(9)	0.4763(4)	0.1075(5)	0.159(7)	0.135(6)	0.135(6)	-0.020(5)	-0.001(5)	0.029(5)
C(1)	4e	0.3472(8)	0.4702(4)	0.1752(6)	0.091(6)	0.076(4)	0.113(6)	-0.011(4)	0.026(5)	0.008(5)
C(2)	4e	0.4288(7)	0.4606(4)	0.2613(5)	0.090(5)	0.142(7)	0.092(5)	-0.008(5)	0.013(5)	-0.006(5)
N(2)	4e	0.3555(9)	0.2963(4)	0.1764(6)	0.170(8)	0.136(7)	0.189(9)	-0.034(6)	-0.055(6)	0.047(6)
C(3)	4e	0.2761(9)	0.2953(4)	0.1152(7)	0.086(6)	0.066(5)	0.161(9)	-0.005(4)	0.007(6)	0.014(5)
C(4)	4e	0.1796(8)	0.2941(4)	0.0361(6)	0.090(6)	0.126(7)	0.147(8)	0.011(5)	0.003(6)	-0.013(6)

3. Bis(tetraphenylphosphonium) hexachloromolybdate(IV) tetraacetonitrile solvate, [P(C₆H₅)₄]₂[MoCl₆] · 4CH₃CN

Table 7. Data collection and handling.

Crystal:	yellow needle, size 0.36 × 0.84 × 1.52 mm
Wavelength:	Mo K _α radiation (0.71070 Å)
μ:	6.35 cm ⁻¹
Diffractometer, scan mode:	Nonius CAD4, ω
2θ _{max} :	50.14°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	5100, 4801
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 3785
<i>N</i> (<i>param</i>) _{refined} :	297
Program:	SHELX-97 [6]

Table 8. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(22)	4e	0.7566	0.3046	0.5600	0.049
H(23)	4e	0.6653	0.4076	0.5988	0.061
H(24)	4e	0.4373	0.4391	0.5367	0.059
H(25)	4e	0.3040	0.3717	0.4283	0.052
H(26)	4e	0.3984	0.2705	0.3828	0.045
H(32)	4e	0.5889	0.1911	0.6176	0.062
H(33)	4e	0.6855	0.1350	0.7526	0.091
H(34)	4e	0.8850	0.0706	0.7569	0.102
H(35)	4e	0.9873	0.0544	0.6268	0.090
H(36)	4e	0.8919	0.1064	0.4898	0.060
H(42)	4e	0.7660	0.1155	0.3043	0.050
H(43)	4e	0.9515	0.1252	0.2208	0.061
H(44)	4e	1.0892	0.2224	0.2325	0.061
H(45)	4e	1.0444	0.3099	0.3280	0.054
H(46)	4e	0.8500	0.3028	0.4066	0.039
H(2A)	4e	0.4453	0.5046	0.2872	0.20(3)
H(2B)	4e	0.5048	0.4366	0.2521	0.20
H(2C)	4e	0.3660	0.4356	0.2975	0.20
H(4A)	4e	0.1564	0.3437	0.0122	0.17(3)
H(4B)	4e	0.0849	0.2778	0.0442	0.17
H(4C)	4e	0.2109	0.2716	-0.0126	0.17

Table 8. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(12)	4e	0.5156	0.1886	0.2524	0.040
H(13)	4e	0.3189	0.1316	0.1789	0.044
H(14)	4e	0.2014	0.0544	0.2577	0.047
H(15)	4e	0.2795	0.0337	0.4112	0.051
H(16)	4e	0.4777	0.0903	0.4863	0.041

Table 9. 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
Mo	2a	0	0	0	0.0671(5)	0.0310(4)	0.0407(4)	-0.0011(3)	0.0212(3)	-0.0023(3)
Cl(1)	4e	0.1110(2)	0.01251(7)	-0.13331(8)	0.0453(7)	0.0481(7)	0.0379(6)	0.0033(5)	0.0151(5)	0.0069(5)
Cl(2)	4e	0.0518(1)	0.11648(6)	0.04066(8)	0.0415(6)	0.0307(6)	0.0423(7)	0.0000(5)	0.0073(5)	-0.0016(5)
Cl(3)	4e	0.2101(2)	-0.03244(9)	0.08299(9)	0.0675(9)	0.0651(9)	0.0563(9)	-0.0008(8)	0.0122(7)	-0.0038(7)
P	4e	0.6570(1)	0.19654(6)	0.43684(7)	0.0253(5)	0.0264(6)	0.0251(5)	-0.0021(4)	0.0010(4)	0.0018(4)
C(11)	4e	0.5142(5)	0.1463(2)	0.3770(3)	0.029(2)	0.025(2)	0.029(2)	-0.001(2)	0.000(2)	-0.003(2)
C(12)	4e	0.4677(5)	0.1577(2)	0.2846(3)	0.032(2)	0.032(2)	0.035(2)	0.002(2)	0.002(2)	0.001(2)
C(13)	4e	0.3507(5)	0.1235(3)	0.2407(3)	0.036(2)	0.041(3)	0.031(2)	0.005(2)	-0.005(2)	-0.008(2)
C(14)	4e	0.2806(5)	0.0774(3)	0.2877(3)	0.031(2)	0.039(3)	0.047(3)	-0.002(2)	0.001(2)	-0.015(2)
C(15)	4e	0.3274(5)	0.0650(3)	0.3796(4)	0.041(3)	0.038(3)	0.050(3)	-0.011(2)	0.010(2)	-0.007(2)
C(16)	4e	0.4452(5)	0.0990(2)	0.4248(3)	0.037(2)	0.033(2)	0.033(2)	-0.005(2)	0.003(2)	-0.003(2)
C(21)	4e	0.5861(5)	0.2768(2)	0.4677(3)	0.030(2)	0.030(2)	0.028(2)	-0.003(2)	0.009(2)	-0.001(2)
C(22)	4e	0.6662(5)	0.3182(3)	0.5329(3)	0.040(3)	0.040(3)	0.043(3)	-0.010(2)	0.010(2)	-0.011(2)
C(23)	4e	0.6110(6)	0.3792(3)	0.5567(4)	0.059(4)	0.045(3)	0.052(3)	-0.014(3)	0.018(3)	-0.019(3)
C(24)	4e	0.4751(6)	0.3985(3)	0.5186(4)	0.062(4)	0.033(3)	0.061(3)	0.000(2)	0.033(3)	-0.005(2)
C(25)	4e	0.3951(6)	0.3582(3)	0.4540(4)	0.043(3)	0.041(3)	0.049(3)	0.010(2)	0.016(2)	0.003(2)
C(26)	4e	0.4511(5)	0.2974(2)	0.4275(3)	0.038(3)	0.036(3)	0.039(3)	-0.001(2)	0.008(2)	0.001(2)
C(31)	4e	0.7331(5)	0.1546(2)	0.5404(3)	0.034(2)	0.034(2)	0.035(2)	-0.013(2)	-0.011(2)	0.011(2)
C(32)	4e	0.6689(7)	0.1636(3)	0.6191(3)	0.062(4)	0.055(3)	0.035(3)	-0.029(3)	-0.004(2)	0.006(2)
C(33)	4e	0.7278(9)	0.1303(4)	0.7001(4)	0.120(6)	0.073(5)	0.028(3)	-0.055(5)	-0.013(3)	0.015(3)
C(34)	4e	0.8453(9)	0.0913(4)	0.7023(5)	0.117(7)	0.058(4)	0.060(5)	-0.049(5)	-0.051(5)	0.031(4)
C(35)	4e	0.9072(8)	0.0819(3)	0.6244(6)	0.067(4)	0.039(3)	0.101(6)	-0.016(3)	-0.043(4)	0.025(4)
C(36)	4e	0.8508(6)	0.1132(3)	0.5424(4)	0.040(3)	0.035(3)	0.065(4)	-0.010(2)	-0.018(3)	0.016(2)
C(41)	4e	0.7922(4)	0.2077(2)	0.3656(3)	0.024(2)	0.033(2)	0.030(2)	0.001(2)	0.003(2)	0.003(2)
C(42)	4e	0.8213(5)	0.1548(3)	0.3088(4)	0.035(3)	0.040(3)	0.052(3)	0.002(2)	0.010(2)	-0.004(2)
C(43)	4e	0.9321(6)	0.1605(3)	0.2591(4)	0.040(3)	0.064(4)	0.051(3)	0.011(3)	0.013(2)	-0.005(3)
C(44)	4e	1.0144(6)	0.2187(3)	0.2661(4)	0.040(3)	0.075(4)	0.039(3)	0.010(3)	0.016(2)	0.014(3)
C(45)	4e	0.9869(5)	0.2713(3)	0.3223(4)	0.036(3)	0.053(3)	0.047(3)	-0.006(2)	0.006(2)	0.015(3)
C(46)	4e	0.8723(3)	0.2665(1)	0.3708(2)	0.032(2)	0.037(3)	0.030(2)	-0.002(2)	0.005(2)	0.005(2)
N(1)	4e	0.2698(3)	0.4789(1)	0.1067(2)	0.145(7)	0.052(4)	0.091(5)	0.008(4)	0.032(5)	0.012(4)
C(1)	4e	0.3377(3)	0.4714(1)	0.1731(2)	0.087(5)	0.039(3)	0.060(4)	0.001(3)	0.029(4)	0.006(3)
C(2)	4e	0.4199(3)	0.4613(1)	0.2592(2)	0.068(4)	0.076(5)	0.067(4)	-0.015(4)	0.022(3)	-0.013(4)
N(2)	4e	0.3471(3)	0.2972(1)	0.1777(2)	0.109(5)	0.066(4)	0.106(5)	-0.026(4)	-0.043(4)	0.033(4)
C(3)	4e	0.2732(3)	0.2976(1)	0.1171(2)	0.057(4)	0.034(3)	0.079(4)	-0.004(3)	-0.006(3)	0.011(3)
C(4)	4e	0.1730(3)	0.2977(1)	0.0332(2)	0.051(3)	0.073(4)	0.069(4)	0.006(3)	0.000(3)	-0.011(3)

References

1. Rabe, S.; Müller, U.: Kaliumheptachlorodioxodimolybdat, $\text{KM}_2\text{O}_2\text{Cl}_7$. *Z. Anorg. Allg. Chem.* **626** (2000) 12-13.
2. Linfeng, C.; Cotton, A. F.: Synthesis, structure and reactivity of $[\text{Zr}_6\text{Cl}_8\text{H}_5]^{2-}$, the first paramagnetic species of its class. *Inorg. Chem.* **35** (1996) 7364-7369.
3. Lau, C.; Dietrich, A.; Plate, M.; Dierkes, B.; Wocadlo, S.; Massa, W.; Harms, K.; Dehnicke, K.: Die Kristallstrukturen der Hexachlorometallate NH_4SbCl_6 , NH_4WCl_6 , $[\text{K}(18\text{-Krone-6})(\text{CH}_2\text{Cl}_2)]_2\text{WCl}_6 \cdot 6\text{CH}_2\text{Cl}_2$ und $(\text{PPh}_4)_2[\text{WCl}_6] \cdot 4\text{CH}_3\text{CN}$. *Z. Anorg. Allg. Chem.* **629** (2003) 473-478.
4. Malecka, J.; Jäger, L.; Wagner, C.; Mrozinski, J.: Preparation, crystal structure and properties of $(\text{P}(\text{C}_6\text{H}_5)_4)_2[\text{ReCl}_6] \cdot 2\text{CH}_3\text{CN}$. *Pol. J. Chem.* **72** (1998) 1879-1885.
5. Noll, A.; Rabe, S.; Müller, U.: Die Oxochlorovanadate $\text{PPh}_4[\text{VOCl}_3\text{OH}]$, $\text{PPh}_4[\text{VOCl}_4]$, $(\text{PPh}_4)_2[\text{VOCl}_4] \cdot 2\text{CH}_3\text{CN}$ und $(\text{PPh}_4)_2[\text{VOCl}_4] \cdot 4\text{CH}_3\text{CN}$ mit auffälligen Abweichungen von der quadratisch-pyramidalen Struktur. *Z. Naturforsch.* **54b** (1999) 591-596.
6. Sheldrick, G. M.: SHELX-97. A program package for crystal structure solution and refinement. University of Göttingen, Germany 1997.
7. Brandenburg, K.: DIAMOND. Visual Crystal Structure Information System. Version 2.1.e. Crystal Impact GbR, Bonn, Germany 2001.