Crystal structure of pentachloro(triphenylphosphinesulfide)niobium(V), $\text{NbCl}_5(\text{Ph}_3\text{PS})$

K. Stumpf^I, R. Blachnik^{*I,II}, G. Roth^{II} and G. Kastner^I^I Universität Osnabrück, Institut für Chemie, Barbarastraße 7, D-49069 Osnabrück, Germany^{II} RWTH Aachen, Institut für Kristallographie, Jägerstraße 17-19, D-52056 Aachen, Germany

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Abstract

$\text{C}_{18}\text{H}_{15}\text{Cl}_5\text{NbPS}$, triclinic, $P\bar{1}$ (No. 2), $a = 9.944(2)$ Å, $b = 10.179(2)$ Å, $c = 11.893(2)$ Å, $\alpha = 88.9(8)^\circ$, $\beta = 70.25(9)^\circ$, $\gamma = 72.96(9)^\circ$, $V = 1079.0$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.029$, $wR_{\text{ref}}(F^2) = 0.056$, $T = 293$ K.

Source of material

Red crystals of $\text{NbCl}_5(\text{Ph}_3\text{PS})$ were obtained from mixing solutions of niobium pentachloride and triphenylphosphinesulfide in dichloromethane in equimolar ratios. $\text{NbCl}_5(\text{Ph}_3\text{PS})$ is sensitive towards moisture.

Discussion

The neutral complex consists of approximately regular octahedra centred by niobium atoms. Nb—Cl bond lengths are in the range 2.29 Å – 2.35 Å, in good agreement with values of 2.25 Å and 2.35 Å in $\text{NbCl}_5(\text{POCl}_3)$ [1] and show the usual *trans*-effect. Nb—S length in $\text{NbCl}_5(\text{Ph}_3\text{PS})$ ($d_{\text{Nb-S}} = 2.62$ Å) is close to that in $\text{NbSCl}_3(\text{Ph}_3\text{PS})$ ($d_{\text{Nb-S}} = 2.58$ Å) [2].

Table 1. Data collection and handling.

Crystal:	red needle, size $0.2 \times 0.2 \times 0.3$ mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ :	13.49 cm^{-1}
Diffractometer, scan mode:	Stoe-IPDS, 159 exposures, $\Delta\phi = 1.7^\circ$
$2\theta_{\text{max}}$:	56.36°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	13833, 4780
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3085
$N(\text{param})_{\text{refined}}$:	226
Programs:	SHELXS-97 [3], SHELXL-97 [4]

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

Atom	Site	x	y	z	U_{iso}
H(2)	2i	0.3160	0.7639	0.7184	0.051
H(3)	2i	0.1016	0.9451	0.8074	0.055
H(4)	2i	0.0901	1.0927	0.9571	0.047
C(5)	2i	0.2950(3)	0.9973(3)	0.9616(3)	0.0401(7)
H(5)	2i	0.2889	1.0555	1.0237	0.048
H(6)	2i	0.5057	0.8748	0.9342	0.041
H(8)	2i	0.6506	0.8981	0.6234	0.051
H(9)	2i	0.7123	0.9578	0.4275	0.062
C(10)	2i	0.7199(4)	0.7803(3)	0.3547(3)	0.0494(8)
H(10)	2i	0.7420	0.8062	0.2767	0.059
H(11)	2i	0.7210	0.5892	0.3136	0.056
H(12)	2i	0.6623	0.5251	0.5087	0.042
H(14)	2i	0.6357	0.6071	0.9625	0.046
H(15)	2i	0.8172	0.6057	1.0446	0.061
H(16)	2i	1.0332	0.6527	0.9313	0.068
H(17)	2i	1.0702	0.7046	0.7358	0.065
H(18)	2i	0.8876	0.7132	0.6543	0.050

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Nb(1)	2i	0.75872(3)	0.25522(2)	0.69951(3)	0.0361(2)	0.0264(1)	0.0338(2)	−0.0095(1)	−0.0145(1)	0.0047(1)
Cl(1)	2i	0.9214(1)	0.03484(7)	0.65677(9)	0.0691(6)	0.0328(4)	0.0748(7)	0.0040(4)	−0.0357(6)	−0.0011(4)
Cl(3)	2i	0.8063(1)	0.27998(8)	0.87496(8)	0.0817(7)	0.0505(4)	0.0496(6)	−0.0241(4)	−0.0417(5)	0.0110(4)
Cl(2)	2i	0.91983(9)	0.37788(7)	0.60401(8)	0.0337(4)	0.0421(4)	0.0521(5)	−0.0137(3)	−0.0058(4)	0.0064(3)

* Correspondence author (e-mail: rblachni@rz.uni-osnabrueck.de)

Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Cl(4)	2i	0.5479(1)	0.18552(7)	0.79963(8)	0.0561(5)	0.0500(4)	0.0485(5)	−0.0316(4)	−0.0160(5)	0.0127(4)
Cl(5)	2i	0.7129(1)	0.23504(7)	0.51956(7)	0.0648(6)	0.0461(4)	0.0390(5)	−0.0122(4)	−0.0253(5)	−0.0023(3)
S(1)	2i	0.53908(8)	0.48707(6)	0.76366(7)	0.0299(4)	0.0286(3)	0.0371(4)	−0.0110(3)	−0.0078(4)	0.0023(3)
P(1)	2i	0.60057(8)	0.66202(6)	0.73850(6)	0.0273(4)	0.0265(3)	0.0260(4)	−0.0084(3)	−0.0093(4)	0.0011(3)
C(1)	2i	0.4336(3)	0.8011(2)	0.8173(2)	0.027(2)	0.029(1)	0.027(2)	−0.010(1)	−0.007(1)	0.006(1)
C(2)	2i	0.3113(3)	0.8227(3)	0.7795(3)	0.039(2)	0.052(2)	0.038(2)	−0.008(1)	−0.019(2)	−0.009(1)
C(3)	2i	0.1834(3)	0.9310(3)	0.8325(3)	0.031(2)	0.054(2)	0.049(2)	−0.005(1)	−0.019(2)	0.002(2)
C(4)	2i	0.1761(3)	1.0187(3)	0.9226(3)	0.031(2)	0.030(1)	0.047(2)	−0.005(1)	−0.004(2)	0.005(1)
C(6)	2i	0.4245(3)	0.8886(2)	0.9084(3)	0.032(2)	0.031(1)	0.042(2)	−0.007(1)	−0.017(2)	−0.001(1)
C(7)	2i	0.6534(3)	0.7038(2)	0.5853(2)	0.024(2)	0.031(1)	0.026(2)	−0.007(1)	−0.005(1)	0.003(1)
C(8)	2i	0.6659(4)	0.8351(3)	0.5613(3)	0.050(2)	0.035(1)	0.039(2)	−0.014(1)	−0.011(2)	0.004(1)
C(9)	2i	0.7013(4)	0.8711(3)	0.4444(3)	0.058(2)	0.043(2)	0.053(2)	−0.022(2)	−0.015(2)	0.022(2)
C(11)	2i	0.7069(4)	0.6506(3)	0.3764(3)	0.054(2)	0.057(2)	0.031(2)	−0.018(2)	−0.017(2)	0.002(1)
C(12)	2i	0.6725(3)	0.6122(3)	0.4929(3)	0.040(2)	0.031(1)	0.038(2)	−0.011(1)	−0.019(2)	0.007(1)
C(13)	2i	0.7436(3)	0.6615(2)	0.7996(3)	0.030(2)	0.026(1)	0.034(2)	−0.006(1)	−0.015(2)	−0.002(1)
C(14)	2i	0.7222(4)	0.6280(2)	0.9171(3)	0.045(2)	0.030(1)	0.040(2)	−0.007(1)	−0.019(2)	0.002(1)
C(15)	2i	0.8314(4)	0.6263(3)	0.9657(3)	0.074(3)	0.035(2)	0.052(2)	−0.002(2)	−0.044(2)	−0.001(1)
C(16)	2i	0.9603(4)	0.6547(3)	0.8980(4)	0.055(2)	0.038(2)	0.090(3)	−0.002(2)	−0.051(3)	−0.009(2)
C(17)	2i	0.9824(4)	0.6863(3)	0.7815(4)	0.037(2)	0.049(2)	0.081(3)	−0.013(2)	−0.025(2)	−0.009(2)
C(18)	2i	0.8734(3)	0.6906(3)	0.7328(3)	0.035(2)	0.043(2)	0.048(2)	−0.012(1)	−0.015(2)	−0.002(1)

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