Crystal structure of tetrabutylammonium hexachloroniobate(V), $N(C_4H_9)_4NbCl_6$

K. Stumpf, R. Blachnik, H. Reuter and H. Eickmeier

Universität Osnabrück, Institut für Chemie, Barbarastr. 7, D-49069 Osnabrück, Germany

Received February 19, 1997, CSD-No. 402873

Source of material: The title compound was prepared by reaction of niobium(V) chloride with tetrabutylammonium chloride in CH_2Cl_2 solution at room temperature. Thin needle-shaped crystals are yellow in color. Tetrabutylammonium hexachloroniobate(V) is sensitive towards moisture. An empirical absorption correction was carried out using ψ -scans.

The $NbCl_6^-$ anion shows almost regular O_h symmetry with an average $d_{Nb-Cl} = 2.3384 \text{ \AA}$, which is in good agreement with the Nb–Cl distance in the nonisotypic $N(C_2H_5)_4[NbCl_6]$ ($d_{Nb-Cl} = 2.3459 \text{ \AA}$, see ref. 1).

Table 1. Parameters used for the X-ray data collection

Crystal:	yellow needle, size 0.15 x 0.2 x 0.3 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	11.10 cm ⁻¹
Diffractometer:	Siemens P4
Scan mode:	$2\theta/\omega$
$T_{\text{measurement}}$:	293 K
$2\theta_{\text{max}}$:	48°
$N(hkl)_{\text{unique}}$:	3947
Criterion for I_o :	$I_o > 2 \sigma(I_o)$
$N(\text{param})_{\text{refined}}$:	219
Programs:	SHELXS-86, SHELXL-93

$C_{16}H_{36}Cl_6NNb$, triclinic, $P\bar{1}$ (No. 2), $a = 10.1612(6) \text{ \AA}$, $b = 10.5165(6) \text{ \AA}$, $c = 13.3486(7) \text{ \AA}$, $\alpha = 79.253(6)^\circ$, $\beta = 76.351(6)^\circ$, $\gamma = 66.701(7)^\circ$, $V = 1266.1 \text{ \AA}^3$, $Z = 2$, $R(F) = 0.038$, $R_w(F^2) = 0.095$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(111)	2i	0.2647(4)	0.3103(4)	0.6199(3)	0.109(3)
H(112)	2i	0.2255(4)	0.2057(4)	0.7101(3)	0.109(3)
H(121)	2i	0.0202(5)	0.4608(5)	0.6412(4)	0.109(3)
H(122)	2i	-0.0197(5)	0.3572(5)	0.7327(4)	0.109(3)
H(131)	2i	-0.0724(6)	0.3325(6)	0.5751(4)	0.109(3)
H(132)	2i	0.0930(6)	0.2896(6)	0.5262(4)	0.109(3)
H(141)	2i	0.0331(8)	0.0942(7)	0.5733(6)	0.109(3)
H(142)	2i	-0.0206(8)	0.1298(7)	0.6885(6)	0.109(3)
H(143)	2i	0.1451(8)	0.0868(7)	0.6395(6)	0.109(3)
H(211)	2i	0.1636(5)	0.4131(4)	0.9131(3)	0.109(3)
H(212)	2i	0.0379(5)	0.4216(4)	0.8608(3)	0.109(3)
H(221)	2i	0.2419(5)	0.1715(5)	0.9364(4)	0.109(3)
H(222)	2i	0.1198(5)	0.1773(5)	0.8800(4)	0.109(3)
H(231)	2i	0.0557(7)	0.3016(6)	1.0668(4)	0.109(3)
H(232)	2i	-0.0626(7)	0.2968(6)	1.0123(4)	0.109(3)
H(241)	2i	-0.0328(8)	0.1355(7)	1.1537(5)	0.109(3)
H(242)	2i	0.1330(8)	0.0645(7)	1.1077(5)	0.109(3)
H(243)	2i	0.0145(8)	0.0597(7)	1.0532(5)	0.109(3)
H(311)	2i	0.0991(4)	0.5723(4)	0.7240(3)	0.109(3)
H(312)	2i	0.2207(4)	0.5666(4)	0.7797(3)	0.109(3)
H(321)	2i	0.2751(5)	0.5175(4)	0.5707(3)	0.109(3)
H(322)	2i	0.3901(5)	0.5254(4)	0.6276(3)	0.109(3)
H(331)	2i	0.2427(5)	0.7610(5)	0.6510(3)	0.109(3)
H(332)	2i	0.1332(5)	0.7521(5)	0.5896(3)	0.109(3)
H(341)	2i	0.2846(6)	0.8585(6)	0.4803(4)	0.109(3)
H(342)	2i	0.3127(6)	0.7179(6)	0.4395(4)	0.109(3)
H(343)	2i	0.4222(6)	0.7268(6)	0.5008(4)	0.109(3)
H(411)	2i	0.4097(4)	0.1964(4)	0.7851(4)	0.109(3)
H(412)	2i	0.4491(4)	0.3044(4)	0.6985(4)	0.109(3)
H(421)	2i	0.4146(6)	0.3132(6)	0.9134(4)	0.109(3)
H(422)	2i	0.4306(6)	0.4380(6)	0.8321(4)	0.109(3)
H(431)	2i	0.6499(8)	0.1608(7)	0.8153(6)	0.109(3)
H(432)	2i	0.6685(8)	0.2972(7)	0.7544(6)	0.109(3)
H(441)	2i	0.7951(9)	0.2197(9)	0.8815(7)	0.109(3)
H(442)	2i	0.6529(9)	0.2286(9)	0.9624(7)	0.109(3)
H(443)	2i	0.6715(9)	0.3649(9)	0.9016(7)	0.109(3)

Table 3. Final 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	0.60799(4)	0.77549(4)	0.75448(3)	0.0497(2)	0.0500(2)	0.0540(2)	-0.0092(2)	-0.0143(2)	-0.0136(2)
Cl(1)	2i	0.5426(2)	0.9257(1)	0.88162(9)	0.0983(9)	0.0615(6)	0.0649(7)	-0.0171(6)	-0.0160(6)	-0.0236(5)
Cl(2)	2i	0.6733(2)	0.6151(1)	0.6328(1)	0.0951(9)	0.0761(8)	0.0812(8)	-0.0136(7)	-0.0211(7)	-0.0369(6)
Cl(3)	2i	0.7363(2)	0.5944(1)	0.8693(1)	0.107(1)	0.0709(8)	0.0866(9)	0.0070(7)	-0.0447(8)	-0.0075(6)
Cl(4)	2i	0.8207(2)	0.8207(2)	0.6874(1)	0.0797(9)	0.147(2)	0.102(1)	-0.061(1)	0.0084(8)	-0.037(1)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl(5)	2i	0.4878(2)	0.9546(2)	0.6396(1)	0.122(1)	0.0740(8)	0.0755(8)	0.0012(8)	-0.0430(8)	-0.0023(6)
Cl(6)	2i	0.3970(2)	0.7280(2)	0.8220(1)	0.0860(9)	0.152(2)	0.105(1)	-0.069(1)	-0.0005(8)	-0.035(1)
N(1)	2i	0.2330(3)	0.3726(3)	0.7624(2)	0.045(2)	0.046(2)	0.057(2)	-0.008(1)	-0.012(1)	-0.009(1)
C(11)	2i	0.2011(4)	0.3037(4)	0.6858(3)	0.059(2)	0.058(2)	0.062(2)	-0.017(2)	-0.008(2)	-0.018(2)
C(12)	2i	0.0454(5)	0.3633(5)	0.6675(4)	0.068(3)	0.077(3)	0.090(3)	-0.017(2)	-0.028(3)	-0.024(3)
C(13)	2i	0.0253(6)	0.2852(6)	0.5903(4)	0.084(3)	0.107(4)	0.084(3)	-0.036(3)	-0.022(3)	-0.028(3)
C(14)	2i	0.0478(8)	0.1356(7)	0.6261(6)	0.116(5)	0.097(5)	0.168(7)	-0.038(4)	-0.051(5)	-0.041(5)
C(21)	2i	0.1381(5)	0.3670(4)	0.8683(3)	0.060(2)	0.064(2)	0.054(2)	-0.020(2)	-0.011(2)	-0.007(2)
C(22)	2i	0.1441(5)	0.2264(5)	0.9233(4)	0.070(3)	0.064(3)	0.084(3)	-0.024(2)	-0.018(2)	0.002(2)
C(23)	2i	0.0349(7)	0.2456(6)	1.0271(4)	0.106(4)	0.086(4)	0.073(3)	-0.045(3)	-0.021(3)	0.006(3)
C(24)	2i	0.0376(8)	0.1150(7)	1.0910(5)	0.132(6)	0.114(5)	0.098(4)	-0.069(4)	-0.022(4)	0.011(4)
C(31)	2i	0.2020(4)	0.5267(4)	0.7271(3)	0.059(2)	0.045(2)	0.058(2)	-0.012(2)	-0.012(2)	-0.007(2)
C(32)	2i	0.2873(5)	0.5614(4)	0.6239(3)	0.064(3)	0.061(2)	0.056(2)	-0.017(2)	-0.008(2)	-0.007(2)
C(33)	2i	0.2351(5)	0.7176(5)	0.5957(3)	0.070(3)	0.063(3)	0.058(2)	-0.022(2)	-0.014(2)	0.000(2)
C(34)	2i	0.3216(6)	0.7590(6)	0.4948(4)	0.085(3)	0.098(4)	0.074(3)	-0.043(3)	-0.010(3)	0.008(3)
C(41)	2i	0.3936(4)	0.2945(4)	0.7673(4)	0.047(2)	0.057(2)	0.082(3)	-0.009(2)	-0.019(2)	0.000(2)
C(42)	2i	0.4548(6)	0.3381(6)	0.8425(4)	0.074(3)	0.108(4)	0.080(3)	-0.040(3)	-0.034(3)	0.014(3)
C(43)	2i	0.6277(8)	0.2582(7)	0.8203(6)	0.132(6)	0.104(5)	0.170(7)	-0.056(4)	-0.098(6)	0.030(5)
C(44)	2i	0.6917(9)	0.2686(9)	0.8973(7)	0.129(6)	0.134(7)	0.178(8)	-0.058(5)	-0.058(6)	0.018(6)

References

1. Siewert, B.; Koellner, G.; Ruhlandt-Senge, K.; Schmock, F.; Müller, U.: Thiohalogeno-Verbindungen von Niob und Tantal: NbSCl_3 , TaSCl_3 , $[\text{NbSCl}_5]^{2-}$, $[\text{TaSCl}_5]^{2-}$, $[\text{NbSBr}_4]^-$. Die Kristallstrukturen von $(\text{PPh}_4)_2[\text{NbSCl}_5] \cdot 2\text{CH}_2\text{Cl}_2$ und $\text{NEt}_4[\text{NbCl}_6]$. *Z. Anorg. Allg. Chem.* **593** (1991) 160-168.
2. Sheldrick, G. M.: SHELXS-86. Program for crystal structure determination. University of Göttingen, Germany 1986.
3. Sheldrick, G. M.: SHELXL-93. Program for crystal structure refinement. University of Göttingen, Germany 1993.