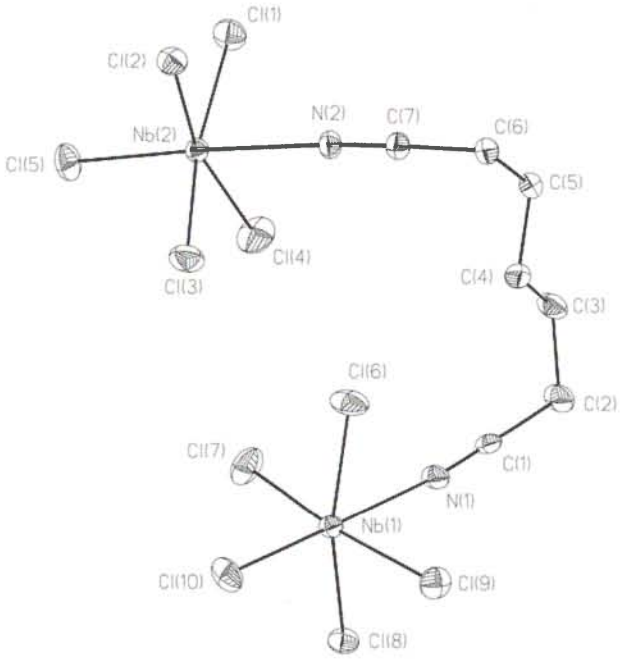
Crystal structure of pimelonitrile-*N*,*N*-bis[pentachloroniobium(V)], $Cl_5Nb(NC(CH_2)_5CN)NbCl_5$

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

Universität Osnabrück, Institut für Chemie, Barbarastraße 7, D-49069 Osnabrück, Germany

Received November 16, 1998, CCDC-No. 1267/62



Abstract

$C_7H_{10}Cl_{10}N_2Nb_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 10.887(1)$ Å, $b = 16.896(2)$ Å, $c = 12.380(2)$ Å, $\beta = 110.44(1)°$, $V = 2133.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.034$, $R_w(F^2) = 0.066$, $T = 293$ K.

Source of material

The title compound was prepared by reaction of niobium(V) chloride with pimelonitrile in stoichiometric ratio in an evacuated ampoule at 423 K. Thin needle-shaped crystals of Cl_5Nb-$NC(CH_2)_5CN$-$NbCl_5$ are colorless and sensitive towards moisture.

Discussion

The molecule consists two distorted square pyramids, each formed by a central Nb atom bonded to five chlorine atoms with one short $d_{Nb-Cl} = 2.25$ Å and four long bonds $\bar{d}_{Nb-Cl} = 2.31$ Å. The plane of the long equatorial Nb-Cl bonds is bent towards the Nb–N bonds $d_{Nb-N} = 2.26$ Å. Bond distances and angles were similar to bond distances found in $NbCl_5(CH_3CN)$ [1], $NbCl_5(Cl_3CCN$ [2] and $NbCl_5((CH_3)_3CCN)$ [3]. These groups are connected by a pimelonitrile bridge via nitrogen ($d_{Nb-N} = 2.249$ Å) of the nitrile group in *cis*-configuration. In this way the coordination sphere of the central niobium atoms is completed to an octahedral arrangement.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.2 x 0.2 x 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	23.18 cm^{-1}
Diffractometer, scan mode:	Siemens P4, $\omega/2\theta$
$2\theta_{max}$:	44°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3320, 2581
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1937
$N(param)_{refined}$:	192
Program:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.6897(7)	0.4270(4)	0.2525(8)	0.062(7)
H(2B)	4*e*	0.7429(7)	0.4297(4)	0.3875(8)	0.062(7)
H(3A)	4*e*	0.5128(7)	0.3586(4)	0.2438(7)	0.062(7)
H(3B)	4*e*	0.5279(7)	0.4176(4)	0.3449(7)	0.062(7)
H(4A)	4*e*	0.5825(7)	0.2551(4)	0.3656(6)	0.062(7)
H(4B)	4*e*	0.6351(7)	0.3106(4)	0.4733(6)	0.062(7)
H(5A)	4*e*	0.3666(7)	0.2955(4)	0.3462(7)	0.062(7)
H(5B)	4*e*	0.4263(7)	0.3404(4)	0.4635(7)	0.062(7)
H(6A)	4*e*	0.5023(7)	0.2154(4)	0.5530(6)	0.062(7)
H(6B)	4*e*	0.3495(7)	0.2255(4)	0.5078(6)	0.062(7)

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

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}
Nb(1)	4*e*	0.96769(6)	0.16107(3)	0.36840(5)	0.0303(4)	0.0312(3)	0.0386(4)	0.0031(3)	0.0095(3)	0.0063(3)
Nb(2)	4*e*	0.38910(6)	0.00298(4)	0.22149(5)	0.0388(4)	0.0368(3)	0.0355(4)	–0.0037(3)	0.0151(3)	–0.0036(3)
Cl(1)	4*e*	0.1747(2)	0.0469(1)	0.1335(2)	0.052(1)	0.066(1)	0.064(1)	0.013(1)	0.013(1)	0.010(1)
Cl(2)	4*e*	0.3127(2)	–0.0778(1)	0.3359(2)	0.059(1)	0.050(1)	0.060(1)	0.000(1)	0.030(1)	0.013(1)
Cl(3)	4*e*	0.6000(2)	–0.0293(1)	0.3408(2)	0.039(1)	0.098(2)	0.071(1)	0.004(1)	0.011(1)	–0.014(1)
Cl(4)	4*e*	0.4649(3)	0.1072(1)	0.1416(2)	0.112(2)	0.081(2)	0.080(2)	–0.034(2)	0.057(2)	0.007(1)
Cl(5)	4*e*	0.3818(2)	–0.0847(1)	0.0828(2)	0.084(2)	0.069(1)	0.049(1)	0.009(1)	0.021(1)	–0.019(1)
Cl(6)	4*e*	0.8156(2)	0.1303(1)	0.4519(2)	0.059(1)	0.066(1)	0.112(2)	0.007(1)	0.053(1)	0.037(1)
Cl(7)	4*e*	0.8257(2)	0.1218(1)	0.1901(2)	0.069(2)	0.068(1)	0.065(1)	–0.011(1)	–0.012(1)	–0.010(1)
Cl(8)	4*e*	1.0936(2)	0.2252(1)	0.2769(2)	0.046(1)	0.056(1)	0.049(1)	0.001(1)	0.0218(9)	0.0136(9)
Cl(9)	4*e*	1.0944(2)	0.2236(1)	0.5384(2)	0.060(1)	0.071(1)	0.041(1)	–0.004(1)	0.010(1)	–0.006(1)
Cl(10)	4*e*	1.0814(2)	0.0468(1)	0.4074(2)	0.074(1)	0.049(1)	0.080(1)	0.028(1)	0.034(1)	0.021(1)
N(1)	4*e*	0.8522(5)	0.2751(3)	0.3315(5)	0.035(4)	0.035(3)	0.060(4)	0.008(3)	0.017(3)	0.012(3)
C(1)	4*e*	0.7868(6)	0.3279(4)	0.3244(6)	0.029(4)	0.042(4)	0.048(4)	–0.009(4)	0.014(3)	0.001(4)
C(2)	4*e*	0.7007(7)	0.3961(4)	0.3213(8)	0.054(5)	0.036(4)	0.110(7)	0.015(4)	0.039(5)	0.015(4)
C(3)	4*e*	0.5674(7)	0.3721(4)	0.3220(7)	0.044(5)	0.050(5)	0.079(6)	0.015(4)	0.028(4)	0.010(4)
C(4)	4*e*	0.5651(7)	0.3036(4)	0.3997(6)	0.037(4)	0.045(4)	0.067(5)	0.000(4)	0.014(4)	–0.007(4)
C(5)	4*e*	0.4375(7)	0.2948(4)	0.4202(7)	0.047(5)	0.044(4)	0.071(5)	–0.001(4)	0.028(4)	–0.012(4)
C(6)	4*e*	0.4251(7)	0.2206(4)	0.4841(6)	0.046(5)	0.048(4)	0.051(5)	–0.002(4)	0.020(4)	–0.013(4)
C(7)	4*e*	0.4111(7)	0.1494(4)	0.4134(6)	0.045(4)	0.040(4)	0.040(4)	0.002(4)	0.012(4)	–0.003(4)
N(2)	4*e*	0.4024(6)	0.0970(3)	0.3554(5)	0.055(4)	0.037(3)	0.044(4)	–0.003(3)	0.022(3)	–0.009(3)

References

1. Willey, G. R.; Woodman, T. J.; Drew, M. G. B.: Structural characterization of the acetonitrile complex $NbCl_5(MeCN)$. Polyhedron **16** (1997) 351.
2. Stumpf, K.; Blachnik, R.; Roth, G.: Crystal structure of pentachloro(trichloroacetonitrile)niobium(V), $NbCl_5(Cl_3CCN)$. Z. Kristallogr. NCS **213** (1998) 151.
3. Blachnik, R.; Stumpf, K.; Reuter, H.; Pawlak, R.: Crystal structure of pentachloro(pivalonitrile)niobium(V), $NbCl_5((CH_3)_3CCN)$. Z. Kristallogr. NCS **213** (1998) 152.
4. Sheldrick, G. M.: SHELXS-97. Program for crystal structure determination. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.