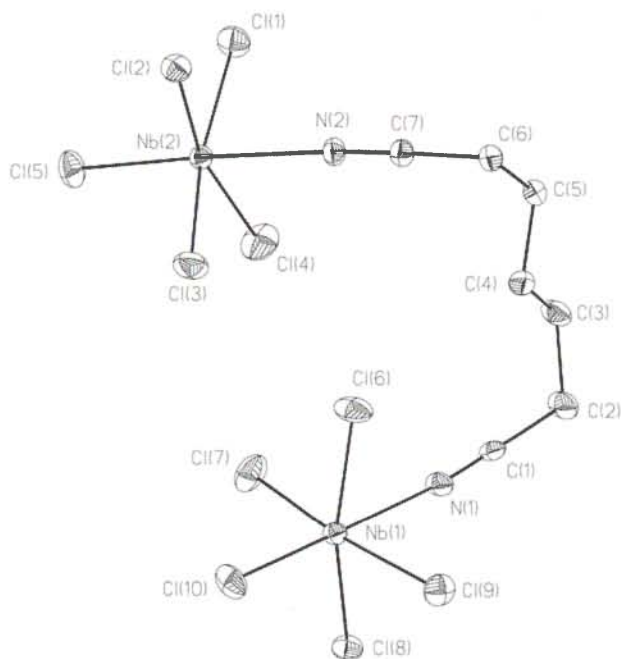


Crystal structure of pimelonitrile-*N,N*-bis[pentachloroniobium(V)], $\text{Cl}_5\text{Nb}(\text{NC}(\text{CH}_2)_5\text{CN})\text{NbCl}_5$

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Abstract

$\text{C}_7\text{H}_{10}\text{Cl}_{10}\text{N}_2\text{Nb}_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 10.887(1) \text{ \AA}$, $b = 16.896(2) \text{ \AA}$, $c = 12.380(2) \text{ \AA}$, $\beta = 110.44(1)^\circ$, $V = 2133.9 \text{ \AA}^3$, $Z = 4$, $R_g(F) = 0.034$, $R_w(F^2) = 0.066$, $T = 293 \text{ K}$.

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 $\text{Cl}_5\text{Nb}(\text{NC}(\text{CH}_2)_5\text{CN})\text{NbCl}_5$ 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 $d_{\text{Nb-Cl}} = 2.25 \text{ \AA}$ and four long bonds $d_{\text{Nb-Cl}} = 2.31 \text{ \AA}$. The plane of the long equatorial Nb-Cl bonds is bent towards the Nb-N bonds $d_{\text{Nb-N}} = 2.26 \text{ \AA}$. Bond distances and angles were similar to bond distances found in $\text{NbCl}_5(\text{CH}_3\text{CN})$ [1], $\text{NbCl}_5(\text{Cl}_3\text{CCN})$ [2] and $\text{NbCl}_5((\text{CH}_3)_3\text{CCN})$ [3]. These groups are connected by a pimelonitrile bridge via nitrogen ($d_{\text{Nb-N}} = 2.249 \text{ \AA}$) 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 $K\alpha$ radiation (0.71073 Å)
μ :	23.18 cm^{-1}
Diffractometer, scan mode:	Siemens P4, $\omega/2\theta$
$2\theta_{\text{max}}$:	44°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	3320, 2581
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1937
$N(\text{param})_{\text{refined}}$:	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)	4e	0.6897(7)	0.4270(4)	0.2525(8)	0.062(7)
H(2B)	4e	0.7429(7)	0.4297(4)	0.3875(8)	0.062(7)
H(3A)	4e	0.5128(7)	0.3586(4)	0.2438(7)	0.062(7)
H(3B)	4e	0.5279(7)	0.4176(4)	0.3449(7)	0.062(7)
H(4A)	4e	0.5825(7)	0.2551(4)	0.3656(6)	0.062(7)
H(4B)	4e	0.6351(7)	0.3106(4)	0.4733(6)	0.062(7)
H(5A)	4e	0.3666(7)	0.2955(4)	0.3462(7)	0.062(7)
H(5B)	4e	0.4263(7)	0.3404(4)	0.4635(7)	0.062(7)
H(6A)	4e	0.5023(7)	0.2154(4)	0.5530(6)	0.062(7)
H(6B)	4e	0.3495(7)	0.2255(4)	0.5078(6)	0.062(7)

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Table 3. 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
Nb(1)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)	4e	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)

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