Crystal structure of hexakis(acetonitrile)sodium hexachlorotantalate(V), $[Na(NCCH_3)_6][TaCl_6]$

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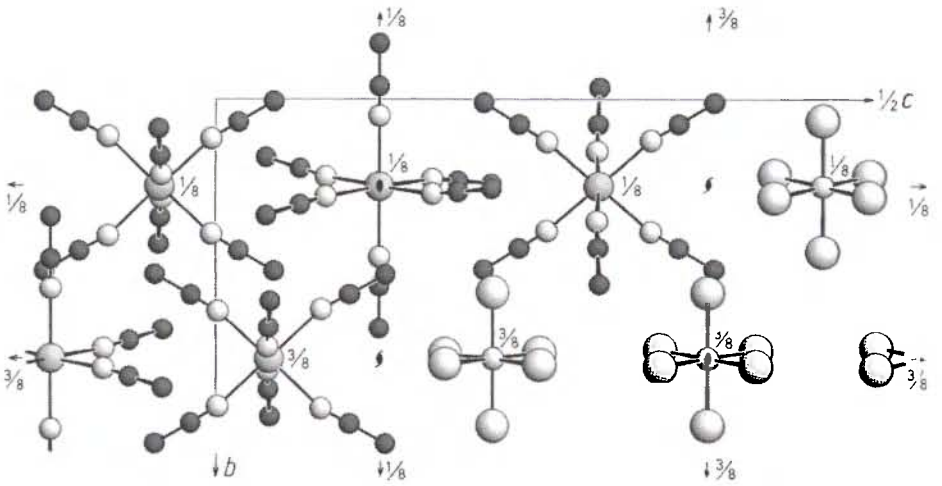

Abstract

$C_{12}H_{18}Cl_6N_6NaTa$, orthorhombic, *Fddd* (No. 70), $a = 15.602(2)$ Å, $b = 21.668(2)$ Å, $c = 45.865(6)$ Å, $V = 15505$ Å^3, $Z = 24$, $R_{gt}(F) = 0.038$, $wR(F^2) = 0.097$, $T = 173$ K.

Source of material

0.4 g Na_2S_4, 1.0 g $TaCl_5$ and 0.29 g 15-crown-5 ether were stirred for 50 h in 40 ml of acetonitrile. A residue was filtered off. At 281 K [Na-15-crown-5][$TaCl_6$] crystallized first. After a few days at 281 K $[Na(NCCH_3)_6][TaCl_6]$ was obtained. The crystals were not dried, but set on the diffractometer with adhering mother liquor and cooled in a dry nitrogen stream.

Discussion

Groups of three consecutive $Na(NCCH_3)_6^+$ ions and three $TaCl_6^-$ ions alternate in rows along *c* as shown in the figure. The central $Na(NCCH_3)_6^+$ ion within one of the groups is rotated by approximately 90° in respect to the other two ions, thus permitting an interweaving of acetonitrile ligands of adjacent ions.

The acetonitrile ligand N(1)–C(11)–C(12) was forced to remain on a twofold rotation axis parallel to *b*, which inplies a bond angle Na–N(1)–C(11) of 180°. However, just as for all other acetonitrile groups, this angle probably should be between 150° and 170°; then the C(11) and especially C(12) should be disordered in two positions with half occupancy close to the twofold axis. In fact, this shows up in the rather large vaules for U_{11} and U_{33}. The attempted refinement with split positions was unstable.

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Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.125 × 0.25 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	48.99 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, ω
$2\theta_{max}$:	49.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2570, 2565
Criterion for I_{obs}:	$I_{obs} > 2\ \sigma(I_{obs})$, 1781
$N(param)_{refined}$:	181
Programs:	XCAD4 [1], SHELXL-97 [2], ATOMS [3]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(12A)	32*h*	0.5	0.0738	–0.1224	0.1343	0.473
H(12B)	32*h*	0.5	0.1742	–0.1224	0.1354	0.473
H(12C)	32*h*	0.5	0.1270	–0.1224	0.1053	0.473
H(22A)	32*h*		0.3162	0.1174	0.0247	0.078
H(22B)	32*h*		0.3888	0.1437	0.0448	0.078
H(22C)	32*h*		0.3179	0.1879	0.0325	0.078
H(32A)	32*h*		0.3190	0.2471	–0.0576	0.093
H(32B)	32*h*		0.3675	0.1942	–0.0411	0.093
H(32C)	32*h*		0.4192	0.2488	–0.0551	0.093
H(42A)	32*h*		0.4373	0.5084	0.1403	0.086
H(42B)	32*h*		0.3388	0.4940	0.1407	0.086
H(42C)	32*h*		0.3727	0.5540	0.1254	0.086
H(52A)	32*h*		0.6716	0.2535	0.0591	0.081
H(52B)	32*h*		0.6676	0.2475	0.0251	0.081
H(52C)	32*h*		0.6035	0.2101	0.0446	0.081

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}
Ta(1)	8*b*	5/8	1/8	1/8	0.0379(4)	0.0261(4)	0.0343(4)	0	0	0
Ta(2)	16*g*	3/8	7/8	0.038065(9)	0.0374(3)	0.0335(3)	0.0348(3)	0.0062(2)	0	0
Cl(1)	32*h*	0.5190(1)	0.1258(1)	0.08880(4)	0.047(1)	0.055(1)	0.043(1)	0.001(1)	–0.0093(9)	0.004(1)
Cl(2)	16*f*	5/8	0.0169(1)	1/8	0.077(2)	0.029(2)	0.056(2)	0	–0.010(2)	0
Cl(3)	32*h*	0.4820(1)	–0.1213(1)	0.00265(4)	0.042(1)	0.058(1)	0.052(1)	0.006(1)	0.012(1)	–0.001(1)
Cl(4)	32*h*	0.3834(2)	–0.2329(1)	0.03830(5)	0.068(1)	0.036(1)	0.058(1)	0.007(1)	–0.004(1)	0.0024(9)
Cl(5)	32*h*	0.4791(1)	–0.1196(1)	0.07474(5)	0.064(1)	0.066(1)	0.056(1)	0.010(1)	–0.023(1)	–0.006(1)
Na(1)	8*a*	1/8	1/8	1/8	0.033(3)	0.040(3)	0.033(3)	0	0	0
N(1)	16*f*	1/8	0.0099(5)	1/8	0.065(7)	0.033(6)	0.059(7)	0	–0.006(7)	0
C(11)	16*f*	1/8	–0.0394(8)	1/8	0.18(2)	0.05(1)	0.09(1)	0	0.09(1)	0
C(12)	16*f*	1/8	–0.1076(8)	1/8	0.50(6)	0.018(9)	0.42(5)	0	0.37(5)	0
N(2)	32*h*	0.2350(4)	0.1271(3)	0.0848(2)	0.048(4)	0.045(4)	0.055(4)	0.010(4)	0.007(4)	0.002(5)
C(21)	32*h*	0.2765(5)	0.1351(4)	0.0652(2)	0.037(4)	0.041(5)	0.039(5)	0.006(4)	0.005(4)	0.007(4)
C(22)	32*h*	0.3294(6)	0.1470(4)	0.0396(2)	0.055(5)	0.050(5)	0.052(6)	0.008(4)	0.002(4)	0.013(4)
Na(2)	16*g*	3/8	3/8	0.04195(8)	0.041(2)	0.036(3)	0.043(3)	–0.003(2)	0	0
N(3)	32*h*	0.3545(6)	0.2992(4)	0.0021(2)	0.095(7)	0.061(5)	0.065(5)	0.005(5)	–0.007(5)	–0.016(5)
C(31)	32*h*	0.3601(6)	0.2715(4)	–0.0187(2)	0.067(6)	0.029(5)	0.055(6)	0.006(4)	–0.009(5)	0.000(4)
C(32)	32*h*	0.3670(6)	0.2376(5)	–0.0453(2)	0.068(7)	0.056(6)	0.062(6)	0.012(5)	–0.009(6)	–0.012(5)
N(4)	32*h*	0.4011(6)	0.4526(4)	0.0810(2)	0.104(7)	0.066(5)	0.056(5)	–0.005(5)	0.002(5)	–0.023(5)
C(41)	32*h*	0.3933(6)	0.4781(4)	0.1023(2)	0.062(6)	0.033(5)	0.055(6)	–0.007(4)	0.004(5)	0.000(5)
C(42)	32*h*	0.3848(6)	0.5114(5)	0.1295(2)	0.075(7)	0.058(6)	0.039(6)	–0.001(5)	–0.004(6)	–0.004(4)
N(5)	32*h*	0.5256(6)	0.3366(4)	0.0409(2)	0.055(5)	0.091(7)	0.109(8)	0.019(5)	–0.002(5)	–0.011(6)
C(51)	32*h*	0.5741(6)	0.2990(4)	0.0415(2)	0.047(5)	0.048(6)	0.059(7)	–0.002(5)	–0.006(5)	–0.006(5)
C(52)	32*h*	0.6342(5)	0.2483(4)	0.0427(2)	0.061(6)	0.048(6)	0.053(6)	0.000(4)	0.001(5)	–0.003(4)

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