Crystal structure of tetraaquamonosuberatonickel(II), $Ni(H_2O)_4(C_8H_{12}O_4)$

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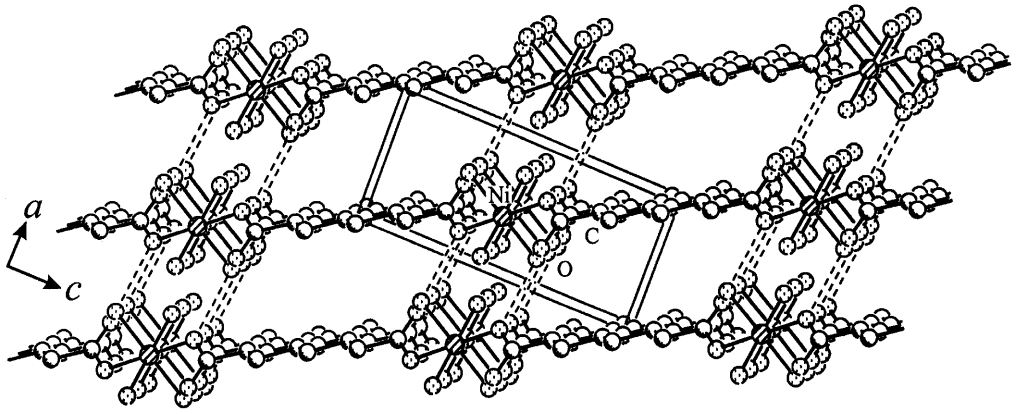
Received March 1, 2003, accepted and available on-line May 16, 2003; CCDC-No. 1267/1030



Abstract

$C_8H_{20}NiO_8$, triclinic, $P\bar{1}$ (No. 2), $a = 4.874(1)$ Å, $b = 6.329(1)$ Å, $c = 10.560(1)$ Å, $\alpha = 76.78(1)°$, $\beta = 87.79(1)°$, $\gamma = 76.97(1)°$, $V = 308.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.024$, $wR_{ref}(F^2) = 0.065$, $T = 293$ K.

Source of material

A methanolic solution of 0.44 g (2.50 mmol) suberic acid in 10 ml CH_3OH was added to a suspension of 0.30 g (2.50 mmol) $NiCO_3$ in 10 mml H_2O. The resulting mixture was then stirred for one hour. After filtration, the filtrate was allowed to stand at room temperature. Green well-shaped crystals were grown by slow evaporation for about 20 days.

Discussion

The Ni atoms in the title compound are each coordinated by four H_2O molecules and two suberato groups to complete slightly *trans* NiO_6 octahedra with d(Ni—O) = 2.044 Å – 2.080 Å. The acute cisoid O–Ni–O angles fall in the region 87.62(4)° – 89.72(5)° trivially less than 90° while the transoid O–Ni–O angles are equal to 180° due to imposition of the local $\bar{1}$ symmetry. The suberate anions function as bis-monodenate ligands with the middle C—C bond centered at the crystllographic 1*c* position. The C—O bond to the coordinating O(1) atom is 1.280(2) Å significantly longer than that of 1.245(2) Å to the uncoordinating O(2) atom and the terminal C—C bond is 1.512(2) Å considerably shorter than the remaining ones averaged to 1.522 Å. The bis-monodenate suberato groups bridge Ni atoms to generate $^1_\infty[Ni(H_2O)_4(C_8H_{12}O_4)_{2/2}]$ polymeric chains extending along [111] direction. The formed chain molecules display strong intramolecular hydrogen bonds between aqua O(3) atom and uncoordinating carboxylate O(2)$^{\#1}$ atom with d(O···O) = 2.623 Å and ∠O–H···O =157° (#1 = –*x*+1, –*y*, –*z*+1). The interchain hydrogen bonds with d(O···O) =2.763 Å – 2.942 Å and ∠O–H···O = 157° – 170° are responsible for the supramolecular assembly of the polymeric chains.

Table 1. Data collection and handling.

Crystal:	green block, size 0.178 × 0.244 × 0.267 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	15.97 cm^{-1}
Diffractometer, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{max}$:	59.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2375, 1795
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1722
$N(param)_{refined}$:	97
Programs:	SHELXS-97 [1], SHELXL-97 [2]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	2*i*	0.2422	–0.1148	0.1120	0.043
H(2B)	2*i*	–0.0130	–0.1572	0.2008	0.043
H(3A)	2*i*	0.1328	–0.5414	0.2105	0.043
H(3B)	2*i*	0.3866	–0.4972	0.1200	0.043
H(4A)	2*i*	–0.1824	–0.3195	0.0393	0.043
H(4B)	2*i*	0.0768	–0.2949	–0.0508	0.043
HO(4B)	2*i*	0.331(5)	–0.293(4)	0.671(2)	0.042(6)
HO(4A)	2*i*	0.143(5)	–0.113(4)	0.664(2)	0.044(7)
HO(3B)	2*i*	0.228(6)	0.348(5)	0.545(3)	0.064(8)
HO(3A)	2*i*	0.053(7)	0.255(6)	0.501(3)	0.08(1)

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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}
Ni	1*f*	1/2	0	1/2	0.0139(1)	0.0184(1)	0.0230(1)	–0.00080(8)	–0.00415(7)	–0.01152(8)
O(1)	2*i*	0.2680(2)	–0.0976(2)	0.3717(1)	0.0190(4)	0.0304(5)	0.0321(5)	–0.0019(4)	–0.0051(4)	–0.0205(4)
O(2)	2*i*	0.5786(2)	–0.3867(2)	0.3267(1)	0.0264(5)	0.0278(5)	0.0369(6)	0.0014(4)	–0.0099(4)	–0.0194(4)
O(3)	2*i*	0.1865(2)	0.2807(2)	0.4842(1)	0.0192(4)	0.0236(4)	0.0306(5)	0.0011(4)	–0.0048(4)	–0.0135(4)
O(4)	2*i*	0.2953(2)	–0.1598(2)	0.6569(1)	0.0231(5)	0.0244(5)	0.0338(6)	–0.0019(4)	0.0005(4)	–0.0081(4)
C(1)	2*i*	0.3571(3)	–0.2403(2)	0.3026(1)	0.0200(5)	0.0224(5)	0.0243(6)	–0.0068(4)	–0.0015(4)	–0.0119(5)
C(2)	2*i*	0.1817(3)	–0.2198(2)	0.1836(2)	0.0320(7)	0.0276(6)	0.0311(7)	0.0000(5)	–0.0115(5)	–0.0167(5)
C(3)	2*i*	0.1938(3)	–0.4346(2)	0.1402(1)	0.0324(7)	0.0269(6)	0.0249(6)	–0.0052(5)	–0.0086(5)	–0.0126(5)
C(4)	2*i*	0.0066(3)	–0.3938(2)	0.0208(1)	0.0351(7)	0.0267(6)	0.0279(7)	–0.0039(5)	–0.0108(5)	–0.0132(5)

Acknowledgments. The project was supported by the National Natural Science Foundation of China (20072022), the Excellent young Teachers Program of Moe, P. R. China (C982302), the Zhejiang Provincial Natural Science Foudation (C99034), the Ningbo Municipal Key Doctor's Funds (2003A61014), and the Ningbo Municipal Natural Science Foundation (01J201301-1). The authors also thank Mr. Jian-Li Lin for X-ray data collection.

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