Crystal structure of tetraaquamonosuberatonickel(II), $\text{Ni}(\text{H}_2\text{O})_4(\text{C}_8\text{H}_{12}\text{O}_4)$

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Abstract

$\text{C}_8\text{H}_{20}\text{NiO}_8$, triclinic, $P\bar{1}$ (No. 2), $a = 4.874(1) \text{ \AA}$, $b = 6.329(1) \text{ \AA}$, $c = 10.560(1) \text{ \AA}$, $\alpha = 76.78(1)^\circ$, $\beta = 87.79(1)^\circ$, $\gamma = 76.97(1)^\circ$, $V = 308.9 \text{ \AA}^3$, $Z = 2$, $R_{\text{gt}}(F) = 0.024$, $wR_{\text{ref}}(F^2) = 0.065$, $T = 293 \text{ K}$.

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 ml 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 suberate groups to complete slightly *trans* NiO_6 octahedra with $d(\text{Ni}—\text{O}) = 2.044 \text{ \AA} - 2.080 \text{ \AA}$. The acute cisoid $\text{O}—\text{Ni}—\text{O}$ angles fall in the region $87.62(4)^\circ - 89.72(5)^\circ$ trivially less than 90° while the transoid $\text{O}—\text{Ni}—\text{O}$ 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 $\text{C}—\text{C}$ bond centered at the crystallographic $1c$ position. The $\text{C}—\text{O}$ bond to the coordinating $\text{O}(1)$ atom is $1.280(2) \text{ \AA}$ significantly longer than that of $1.245(2) \text{ \AA}$ to the uncoordinating $\text{O}(2)$ atom and the terminal $\text{C}—\text{C}$ bond is $1.512(2) \text{ \AA}$ considerably shorter than the remaining ones averaged to $1.522 \text{ \AA}$. The bis-monodenate suberate groups bridge Ni atoms to generate $[\text{Ni}(\text{H}_2\text{O})_4(\text{C}_8\text{H}_{12}\text{O}_4)_{2/2}]$ polymeric chains extending along $[111]$ direction. The formed chain molecules display strong intramolecular hydrogen bonds between aqua $\text{O}(3)$ atom and uncoordinating carboxylate $\text{O}(2)^{\#1}$ atom with $d(\text{O} \cdots \text{O}) = 2.623 \text{ \AA}$ and $\angle \text{O}—\text{H} \cdots \text{O} = 157^\circ$ ($\#1 = -x+1, -y, -z+1$). The interchain hydrogen bonds with $d(\text{O} \cdots \text{O}) = 2.763 \text{ \AA} - 2.942 \text{ \AA}$ and $\angle \text{O}—\text{H} \cdots \text{O} = 157^\circ - 170^\circ$ are responsible for the supramolecular assembly of the polymeric chains.

Table 1. Data collection and handling.

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

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

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

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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
Ni	1 <i>f</i>	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>i</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>i</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>i</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>i</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>i</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>i</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>i</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>i</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)

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