研究简报

水合羟基高氯酸镍(Ⅱ)的晶体结构

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Crystal Structure of the [nickel(Ⅱ)-(μ_3-hydroxide)-(μ_3-perchlorate)-aqua]$_n$ Complex

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Green single crystal of the title compound was accidentally obtained by the reaction of N,N′-bis(pyridylcarbonyl)-4,4′-diaminodiphenyl ether (L) with nickel perchlorate. In the structure of [Ni(μ_3-OH)(μ_3-ClO_4)(H_2O)]$_n$, the local coordinating geometry around Ni center displays a distorted octahedron in which one coordination site of each metal atom was occupied by a water molecule. Each perchlorate anion and hydroxyl group acts as a tridentate bridging spacer to bind three Ni atoms, thus resulting in the formation of two-dimensional layered network. CSD: 414263.

Keywords: **crystal structure** **nickel** **hydroxyl group** **perchlorate**

Comment

The structural in the inorganonickel perchlorate is well documented[1,2]. Among these, the structures in which perchlorate anion acts as a tridentate ligand are rather rare. To our best knowledge, inorganonickel perchlorate compound containing tridentate perchlorate and tridentate hydroxyl group together has not been reported. Here we report on the crystal structure of the tile compound [Ni(μ_3-OH)(μ_3-ClO_4)(H_2O)]$_n$. In the structure of [Ni(μ_3-OH)(μ_3-ClO_4)(H_2O)]$_n$, the local coordinating geometry around Ni center displays a distorted octahedron. As shown in Fig.1, the structure features two tridentate perchlorate ligands that μ_3-bridge via two oxygen atoms each to two nickel atoms leading to the formation of an eight-membered ring. The coordination hydroxyl group also tridentate to three nickel atoms. Thus the tridentate perchlorate and hydroxyl group result in the formation of 2D network. In Fig.2. We can see that the 2D network based on corner-sharing between octahedron and tetrahedron and edge-sharing between octahedrons. The Ni-O (perchlorate group, hydroxyl group and water molecule)

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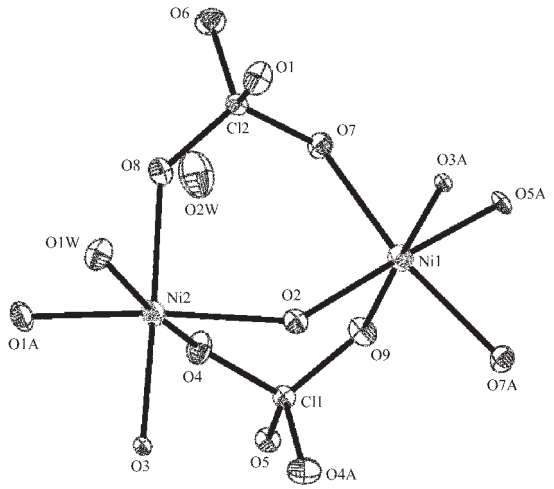

Fig.1 Molecular structure of $[Ni(\mu_3\text{-}OH)(\mu_3\text{-}ClO_4)(H_2O)]_{1.5}$ with hydrogen atoms omitted

Key geometric parameters:

Ni(1)-O(7) 0.209 5(4) nm, Ni(2)-O(3) 0.211 4(4) nm, Ni(1)-O(2) 0.147 9(6) nm, Ni(2)-O(4) 0.214 1(5) nm; O(7)-Ni(1)-O(2) 96.31(13)°, O(2)-Ni(1)-O(9) 95.10(3)°, O(3)-Ni(2)-O(4) O(3)-Ni(2)-O(1W) 88.2(2)°.

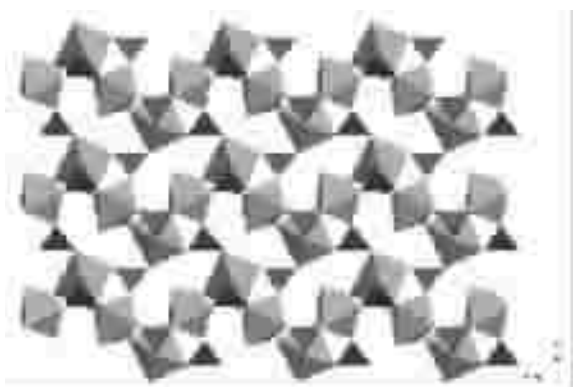

Fig.2 2D network representation of $[Ni(\mu_3\text{-}OH)(\mu\text{-}ClO_4)(H_2O)]_n$ highlighting Ni octahedron and Cl tetrahedron

bond distances (0.208 7~0.222 0 nm) are a little longer than normal Ni-O distances 0.205 9 nm[3]. Ni-OH bond length (0.214 4 nm) is quite similar to normal Ni-OH bond distance (0.214 0 nm)[4]. The intermolecular Ni-Ni bond distances range from 0.306 7 to 0.647 3 nm and Cl-O from 0.146 0 to 0.147 9 nm.

Experimental Details

Hydrothermal treatment of Ni $(ClO_4)_2 \cdot 6H_2O$ (1.0 mmol) and N,N′-bis (pyridylcarbonyl)-4,4′-diaminodiphenyl ether (1.0 mmol) over one day at 90 ℃ gave green crystals. Intensity data were collected at 293(2) K on a Bruker AXS SMART CCD for a dimension of 0.10 mm × 0.15 mm × 0.18 mm. $H_9Cl_3Ni_3O_{18}$, M= 579.55, orhorhombic space group $Cmc2_1$ with a= 1.819 5(4) nm, b=0.766 60(15) nm, c=0.989 2(2) nm, V=1.379 5(5) nm^3, Z=8, R_1 [$I>2\sigma(I)$]=0.028 6, wR_2 (all) =0.084 3. Programs used: SAINT, SADABS, SHELX-97, ORTEP.

CSD: 414263.

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