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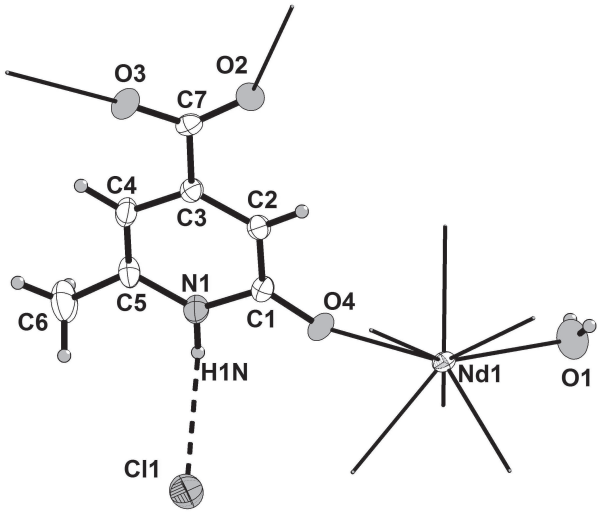
Crystal structure of diaqua-bis(μ_3-2-methyl-6-oxidopyridinium-4-carboxylato-$\kappa^3O:O':O''$) neodymium(III) chloride, $C_{14}H_{16}ClN_2O_8Nd$



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Abstract

$C_{14}H_{14}ClN_2O_8Nd$, Tetragonal, $I4_1/a$, $a = 10.3597(15)$ Å, $b = 10.3597(15)$ Å, $c = 32.729(10)$ Å, $V = 3512.6(12)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0243$, $wR_{ref}(F^2) = 0.0620$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The mixture of $NdCl_3$ (0.050 g, 0.2 mmol) and H_2minca (represents 2-(hydroxyl)-6-methyl-isonicotinic acid, 0.039 g, 0.2 mmol) is mixed with an aqueous solution (10 mL). After stirring for 20 min in air, the pH value was adjusted to 4.5, and the mixture was placed into 25 mL Teflon-lined autoclave under autogenous pressure being heated at 160 °C for 72 h, then the autoclave was cooled over a period of 28 h at a rate 5 °C/h to room temperature. After filtration and washing with H_2O, the pink prism crystals of title complex were obtained suitable for X-ray diffraction analysis. For title complex, yield: 0.060 g (58%) based on neodymium(III) element. Elemental analysis (%): calcd for $C_{14}H_{14}ClN_2O_8Nd$: C 32.46, H 2.72, N 5.41, found: C 32.38, H 3.21, N 5.42. IR (KBr pellet, cm^{-1}): 3182 br, 2930s, 2037s, 1705s, 1640vs, 1091m, 918s, 792s, 770m, 668s, 605s.

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.28×0.35×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	31.54 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω scans
$2\theta_{max}$:	50.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9267, 1642
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1358
$N(param)_{refined}$:	120
Programs:	SHELX [10]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{iso}
H(1W)	16f	1.2432	0.6768	0.5108	0.056
H(2W)	16f	1.1937	0.5555	0.5233	0.056
H(1N)	16f	0.584(4)	0.593(4)	0.465(1)	0.05(1)
H(2)	16f	0.8637	0.6841	0.3888	0.023
H(4)	16f	0.5111	0.5928	0.3466	0.029
H(6A)	16f	0.3667	0.5854	0.4427	0.065
H(6B)	16f	0.3964	0.4477	0.4252	0.065
H(6C)	16f	0.3365	0.5539	0.3969	0.065

Discussion

The crystal engineering of supramolecular architectures based on metal and organic building blocks has been rapidly expanding in recent years owing to their novel and diverse topologies and potential applications [1, 2]. Isonicotinic acid based ligands are very important building blocks to form

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Nd(1)	8*e*	1	0.75	0.49184(2)	0.0187(2)	0.0174(2)	0.0113(1)	−0.0013(1)	0.0	0.0
O(2)	16*f*	0.8587(2)	0.6820(2)	0.31301(7)	0.025(2)	0.034(2)	0.020(1)	0.001(1)	0.003(1)	0.004(1)
O(1)	16*f*	1.2007(3)	0.6425(3)	0.52073(8)	0.034(2)	0.032(2)	0.046(2)	0.002(1)	−0.004(1)	0.012(1)
C(7)	16*f*	0.7410(3)	0.6749(3)	0.3187(1)	0.026(2)	0.012(2)	0.017(2)	0.003(1)	−0.002(2)	−0.001(1)
O(4)	16*f*	0.8069(2)	0.6402(2)	0.46601(6)	0.026(1)	0.037(2)	0.015(1)	−0.007(1)	−0.003(1)	0.001(1)
Cl(1)	8*e*	0.5	0.75	0.51749(5)	0.0380(9)	0.070(1)	0.0330(7)	0.0081(8)	0.0	0.0
N(1)	16*f*	0.6119(3)	0.6000(3)	0.43859(9)	0.021(2)	0.035(2)	0.018(1)	−0.002(1)	0.000(1)	0.005(1)
C(1)	16*f*	0.7385(3)	0.6342(3)	0.4341(1)	0.020(2)	0.019(2)	0.020(2)	−0.002(1)	−0.003(1)	−0.000(1)
C(2)	16*f*	0.7791(3)	0.6587(3)	0.3937(1)	0.017(2)	0.020(2)	0.020(2)	−0.002(1)	0.001(1)	0.000(1)
C(3)	16*f*	0.6945(3)	0.6453(3)	0.3617(1)	0.020(2)	0.016(2)	0.018(2)	0.003(1)	−0.003(1)	0.001(1)
O(3)	16*f*	0.6563(2)	0.6903(2)	0.29192(7)	0.033(2)	0.038(2)	0.017(1)	0.000(1)	−0.007(1)	0.006(1)
C(4)	16*f*	0.5673(3)	0.6043(4)	0.3685(1)	0.018(2)	0.031(2)	0.023(2)	−0.000(2)	−0.006(2)	0.001(2)
C(5)	16*f*	0.5271(3)	0.5815(3)	0.4074(1)	0.016(2)	0.030(2)	0.029(2)	−0.002(2)	−0.003(2)	0.004(2)
C(6)	16*f*	0.3949(4)	0.5383(5)	0.4191(1)	0.021(2)	0.064(3)	0.044(2)	−0.008(2)	−0.000(2)	0.013(2)

many novel compounds widely used in medicine, agriculture, industry, and coordination chemistry [3, 4]. In addition, isonicotinic acid has been intensively employed to provide a topological architecture due to its remarkable versatile coordination modes as well as the noncovalent bond, and the electrical properties depend heavily on the intermolecular interactions [5, 6]. In recent years, a variety of multi-carboxylate pyridimine/pyridine-based ligands have been extensively employed for exhibiting various coordination fashions [7–9]. In order to further study the coordination behavior and role of neodymium(III) cation in the self-assembly processes in the presence of nitrogen-heterocyclic dicarboxylate, the title complex has been synthesized and characterized. The single crystal analysis reveals that the basic unit of title compound consists of $[Nd(H_2O)_2(minca)_2]^+$, and one chloride anion. The hydrogens from hydroxyl and carboxylic group of H_2minca have been fully deprotonated and one of them is shifted to the nitrogen of the pyridineyl moiety. The Nd(III) ion is coordinated by eight oxygen atoms. Four oxygen atoms are from four different carboxylate groups, two oxygen atoms from two hydroxy groups of two different Hminca ligands, and two oxygen atoms from two water molecules. The Nd–O bond lengths range from 2.386(2) Å for the Nd1–O3 to 2.541(3) Å Nd1–O1 distance of the water ligands. The bond angles around Nd are between 70.05(8)° and 139.76(8)°. Each Hminca-ligand coordinates to three adjacent Nd metal centers to form a three dimensional polymer. The shortes Nd$\cdots$Nd distances are 5.21 Å.

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