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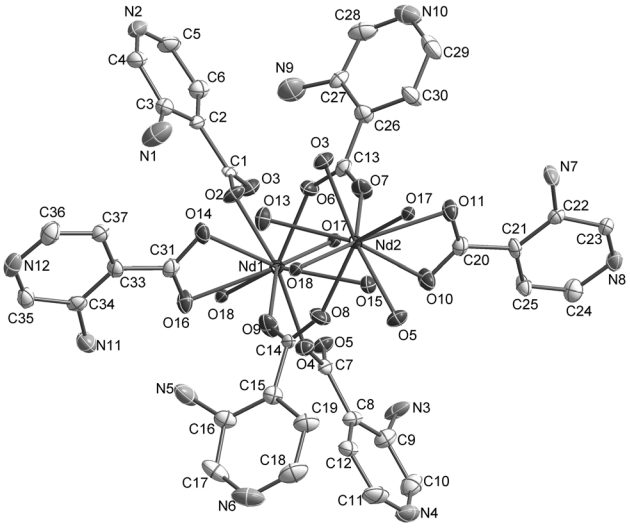
The crystal structure of poly[di(μ_2-aqua)-diaqua-bis(3-aminopyridine-4-carboxylate-κ^2O: O')-tetra(μ_2-3-aminopyridine-4-carboxylate-κ^2O: O')-dineodymium(III), $[Nd_2(C_6H_5N_2O_2)_6(H_2O)_4]_n$



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Abstract

$[Nd_2(C_6H_5N_2O_2)_6(H_2O)_4]_n$, monoclinic, $P2_1/c$ (no. 14), a = 9.383(3) Å, b = 22.741(6) Å, c = 18.775(5) Å, β = 90.231(5)°, V = 4006(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0427, $wR_{ref}(F^2)$ = 0.0862, T = 296 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.18 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.66 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	27.6°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	24,277, 9122, 0.063
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6092
$N(param)_{refined}$:	605
Programs:	Bruker [1], Diamond [2], Olex2 [3], SHELX [4, 5]

1 Source of materials

3-Aminoisonicotinic acid (0.6 mmol 0.083 g), Nd_2O_3 (0.2 mmol 0.067 g), and 0.4 mol L^{-1} NaOH solution (1 mL), were dissolved in deionized water (7 mL). The mixture was sealed in a 25-mL Teflon-lined steel autoclave, and heated at 453 K for 8 days, then slowly cooled to room temperature. Colorless crystals of title complex could be found in the product.

2 Experimental details

All hydrogen atoms were placed in calculated positions and refined as riding atoms. The values were set to be 1.2U_{eq} or 1.5U_{eq} of the parent atoms.

3 Comment

Lanthanide complexes of 3-aminoisonicotinic acid have attracted some attentions during recent decades [6–11]. However, transition metal complexes for this ligand are relatively rare. A complex ($[Nd_2(C_6H_5N_2O_2)_6(H_2O)_4]_n$) of 3-aminoisonicotinic acid has been prepared under solvothermal reaction conditions. The asymmetric unit of title complex contains one $Nd1^{3+}$ ion, one $Nd2^{3+}$ ion, six L^- (HL = 3-aminoisonicotinic acid) ligands and four coordinated aqua molecules. Each Nd1(III) ion is nine-coordinated by

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

Atom	x	y	z	U_{iso}*/U_{eq}
Nd1	0.49211 (3)	0.35460 (2)	0.25256 (2)	0.01435 (8)
Nd2	1.04449 (3)	0.39830 (2)	0.26271 (2)	0.01375 (8)
O17	0.3219 (3)	0.43644 (14)	0.28824 (19)	0.0190 (8)
H17A	0.332005	0.444117	0.332611	0.028*
H17B	0.336265	0.465857	0.263161	0.028*
O2	0.3641 (4)	0.32326 (16)	0.3573 (2)	0.0227 (9)
O3	0.1471 (4)	0.36036 (16)	0.3721 (2)	0.0223 (9)
O4	0.3895 (4)	0.38804 (16)	0.14489 (19)	0.0219 (9)
O5	0.1729 (4)	0.42775 (16)	0.1527 (2)	0.0240 (9)
O6	0.6358 (4)	0.38627 (16)	0.3514 (2)	0.0248 (9)
O7	0.8481 (4)	0.42707 (17)	0.3392 (2)	0.0288 (10)
O8	0.8946 (4)	0.36458 (17)	0.1701 (2)	0.0280 (10)
O9	0.6796 (4)	0.32430 (17)	0.1688 (2)	0.0305 (10)
O10	0.9188 (4)	0.48624 (16)	0.2102 (2)	0.0278 (10)
O11	1.0712 (4)	0.50565 (15)	0.2961 (2)	0.0239(10)
O18	1.2009 (3)	0.31453 (14)	0.22682 (19)	0.0196 (8)
H18A	1.190447	0.306198	0.183953	0.029*
H18B	1.183987	0.282718	0.252823	0.029*
O13	0.9118 (4)	0.30711 (15)	0.3059 (2)	0.0242 (9)
H13A	0.966630	0.278136	0.303797	0.036*
H13B	0.827120	0.305276	0.287157	0.036*
O14	0.6150 (4)	0.26242 (15)	0.3004 (2)	0.0250 (9)
O15	0.6233 (3)	0.44527 (15)	0.2172 (2)	0.0222 (9)
H15A	0.663738	0.471007	0.240164	0.033*
H15B	0.611437	0.451907	0.171814	0.033*
N1	0.4474 (5)	0.2346 (2)	0.4431 (3)	0.0498 (17)
H1A	0.500895	0.206943	0.459449	0.060*
H1B	0.463952	0.249337	0.401782	0.060*
N2	0.2119 (5)	0.2420 (2)	0.5932 (3)	0.0292 (12)
N3[a]	0.1029 (9)	0.5117 (4)	0.0600 (4)	0.036 (3)
H3A[a]	0.055540	0.540824	0.042599	0.044*
H3B[a]	0.082424	0.498311	0.101494	0.044*
N4	0.3488 (5)	0.4930 (2)	−0.0866 (3)	0.0318 (13)
N5	0.6720 (5)	0.2251 (2)	0.0826 (3)	0.0416 (15)
H5A	0.653680	0.234067	0.126133	0.050*
H5B	0.633608	0.194446	0.063716	0.050*
N6	0.8905 (6)	0.2677 (3)	−0.0666 (3)	0.0455 (16)
N7	1.1154 (5)	0.61980 (19)	0.3327 (3)	0.0278 (12)
H7A	1.135478	0.584279	0.344883	0.033*
H7B	1.156977	0.648773	0.353677	0.033*
N8	0.8775 (5)	0.70374 (19)	0.2196 (3)	0.0258 (12)
N9	0.5324 (5)	0.3836 (3)	0.4886 (3)	0.0489 (16)
H9A	0.474985	0.374649	0.522530	0.059*
H9B	0.533580	0.362783	0.450327	0.059*
N10	0.6961 (6)	0.5074 (2)	0.5742 (3)	0.0402 (15)
N11	0.3938 (5)	0.1409 (2)	0.1704 (3)	0.0329 (13)
H11A	0.349967	0.114278	0.146312	0.039*
H11B	0.375878	0.177462	0.162787	0.039*
N12	0.6206 (5)	0.0448 (2)	0.2743 (3)	0.0293 (12)
C1	0.2514 (5)	0.3300 (2)	0.3918 (3)	0.0157 (12)
C2	0.2404 (6)	0.2990 (2)	0.4618 (3)	0.0185 (12)
C3	0.3371 (6)	0.2548 (2)	0.4824 (3)	0.0209 (13)
C4	0.3159 (6)	0.2291 (3)	0.5487 (3)	0.0269 (14)
H4	0.380522	0.200365	0.562882	0.032*

Table 2: (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C5	0.1210 (6)	0.2839 (3)	0.5719 (3)	0.0296 (15)
H5	0.046426	0.293797	0.602089	0.036*
C6	0.1314 (6)	0.3127 (3)	0.5087 (3)	0.0253 (14)
H6	0.065431	0.341665	0.496927	0.030*
C7	0.2865 (6)	0.4183 (2)	0.1197 (3)	0.0179 (12)
C8	0.3049 (6)	0.4428 (2)	0.0468 (3)	0.0204 (13)
C9	0.2134 (6)	0.4862 (3)	0.0205 (3)	0.0272 (14)
H9[b]	0.135727	0.499106	0.046892	0.033*
C10	0.2420 (7)	0.5094 (3)	−0.0459 (4)	0.0351 (16)
H10	0.181564	0.538634	−0.063087	0.042*
C11	0.4343 (6)	0.4511 (3)	−0.0616 (3)	0.0348 (16)
H11	0.509144	0.438386	−0.090008	0.042*
C12	0.4173 (6)	0.4258 (3)	0.0041 (3)	0.0243 (14)
H12[a]	0.481224	0.397333	0.019876	0.029*
C13	0.7369 (6)	0.4174 (2)	0.3733 (3)	0.0190 (12)
C14	0.7974 (6)	0.3347 (2)	0.1421 (3)	0.0174 (12)
C15	0.8287 (5)	0.3098 (2)	0.0700 (3)	0.0200 (12)
C16	0.7620 (6)	0.2595 (3)	0.0435 (3)	0.0243 (13)
C17	0.7982 (7)	0.2409 (3)	−0.0258 (4)	0.0373 (17)
H17	0.753572	0.207506	−0.043628	0.045*
C18	0.9557 (7)	0.3152 (3)	−0.0399 (4)	0.0413 (17)
H18	1.022357	0.334542	−0.067942	0.050*
C19	0.9289 (6)	0.3366 (3)	0.0267 (3)	0.0323 (15)
H19	0.978297	0.369447	0.043111	0.039*
C20	0.9793 (6)	0.5221 (2)	0.2500 (3)	0.0197 (13)
C21	0.9451 (5)	0.5865 (2)	0.2433 (3)	0.0192 (13)
C22	1.0182 (6)	0.6301 (2)	0.2805 (3)	0.0206 (13)
C23	0.9805 (6)	0.6886 (2)	0.2635 (3)	0.0234 (13)
H23	1.032371	0.718655	0.284970	0.028*
C24	0.8056 (6)	0.6609 (3)	0.1858 (3)	0.0281 (14)
H24	0.731770	0.670806	0.154862	0.034*
C25	0.8390 (5)	0.6025 (2)	0.1960 (3)	0.0221 (13)
H25	0.789617	0.573695	0.170927	0.027*
C26	0.7216 (5)	0.4468 (2)	0.4443 (3)	0.0221 (13)
C27	0.6210 (5)	0.4304 (2)	0.4954 (3)	0.0197 (13)
C28	0.6141 (7)	0.4621 (3)	0.5581 (3)	0.0361 (16)
H28	0.546609	0.450637	0.591448	0.043*
C29	0.7917 (8)	0.5230 (3)	0.5259 (4)	0.0449 (19)
H29	0.851187	0.554587	0.536135	0.054*
C30	0.8077 (6)	0.4950 (3)	0.4614 (3)	0.0343 (16)
H30	0.875733	0.508203	0.429238	0.041*
C31	0.5475 (6)	0.2296 (2)	0.2586 (3)	0.0200 (13)
O16	0.4587 (4)	0.25024 (15)	0.2148 (2)	0.0241 (10)
C33	0.5704 (5)	0.1648 (2)	0.2622 (3)	0.0190 (12)
C34	0.4915 (6)	0.1252 (2)	0.2208 (3)	0.0199 (13)
C35	0.5183 (6)	0.0655 (3)	0.2320 (3)	0.0278 (14)
H35	0.460816	0.038411	0.208341	0.033*
C36	0.6973 (6)	0.0833 (3)	0.3112 (4)	0.0305 (15)
H36	0.769784	0.069407	0.340718	0.037*
C37	0.6739 (6)	0.1422 (2)	0.3075 (3)	0.0221 (13)
H37	0.727830	0.167549	0.335649	0.027*
N3A[b]	0.5295 (12)	0.3863 (5)	0.0206 (6)	0.030 (4)
H3AA[b]	0.597268	0.380750	−0.009397	0.036*
H3AB[b]	0.529864	0.367833	0.060574	0.036*

[a]Occupancy: 0.596(9), [b]Occupancy: 0.404(9).

four oxygen atoms of four bridging L^- ligands, two chelating oxygen atoms of one L^- ligand and three oxygen atoms of three coordinated aqua molecules. The environment of the $Nd2^{3+}$ ion is similar to $Nd1^{3+}$. Each of Nd2(III) ions is surrounded by two chelating oxygen atoms of a L^- ligand, four oxygen atoms of four μ_2-bridging L^- ligands and three water oxygen atoms, forming a nine-coordination geometry, too. The Nd–O distances vary from 2.359(4) to 2.918(3) Å [12–14]. The O–Nd1–O angles are in the range 51.35(12)–145.45(13)°. Among the six L^- ligands of the asymmetric unit, four act as μ_2-bridges, linking Nd1(III) and Nd2(III) ions, and two chelate to Nd(III) ions. While two of the four coordinated aqua molecules also play the roles of bridges, connecting Nd1(III) and Nd2(III) ions, and the other two act as monodentate ligands.

Each Nd1(III) ion is connected to one Nd2(III) ion via two μ_2–L^- and two water molecules, and to another Nd2(III) ion by two μ_2–L^-–L ligands, forming the 1–D zigzag-chain structure of the title complex with nonbonding Nd···Nd separations of 4.32–5.28 Å and Nd–Nd–Nd angles of 155.408(11)°. The zigzag chains stack parallel along the *b* axis, constructing the 3–D architecture of the title complex.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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