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Synthesis and Crystal Structure of $[Y(C_6H_4NO_2)_2(H_2O)_4]_n.nCl$

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A novel yttirium complex with the general formula $[Y(C_6H_4NO_2)_2(H_2O)_4]_n.nCl$ (**1**) has been prepared *via* a hydrothermal synthesis method and structurally characterized by X-ray diffractions. Complex **1** crystallizes in the space group Pbcn of the orthorhombic system with four formula units in a cell: a = 8.9401(14), b = 19.576(3), c = 10.0063(13) Å, V = 1751.2(4) $Å^3$, $C_{12}H_{16}N_2O_8ClY$, M_r = 440.63, D_c = 1.671 g/cm^3, T = 293.15 K, F(000) = 888 and R1/wR^2 = 0.0421/0.0991 for 1256 observed reflections [$I > 2\sigma(I)$] and 1539 unique reflections. Complex **1** features a novel one-dimensional (1-D) chain-like structure.

Key Words: Crystal, Hydrothermal, Yttirium, Isonicotinic, Supramolecule.

INTRODUCTION

In recent years, lanthanide materials have attracted increasing interest due to their potential applications as biological materials, zeolite-like materials, catalysts, magnetic functional materials and so forth. Many investigations have been conducted on lanthanide materials with conjugated ligands so far.[1-6] As one of conjugated ligands, isonicotinate anion is a very interesting ligand in building extended structures because it has an unsymmetrical divergent motif with one nitrogen atom at one end and two oxygen atoms of carboxylato group at the other end. Therefore, isonicotinate anion may coordinate to two or more metal ions with the nitrogen atom and one or two oxygen atoms from the carboxylato group[7,8]. Thus, we recently interested in the area of lanthanide materials with isonicotinic acid as a ligand. We report herein the synthesis and crystal structure of $[Y(C_6H_4NO_2)_2(H_2O)_4]_n.nCl$ (**1**) with a novel one-dimensional chain-like structure, which is obtained from a hydrothermal reaction.

EXPERIMENTAL

All reactants of A.R. grade were obtained commercially and used without further purification.

Synthesis of $[Y(C_6H_4NO_2)_2(H_2O)_4]_n.nCl$ (1): This complex was prepared by mixing $YCl_3.6H_2O$ (1 mmol, 304 mg), isonicotinic acid (2 mmol, 246 mg) and 10 mL distilled water in a 25 mL Teflon-lined stainless steel autoclave and heated at 453 K for 10 days. After being slowly cooled to room temperature at 6 K/h, colourless crystals suitable for X-ray analysis were obtained. Yield: 43 % (based on yttrium).

X-ray structure determination: X-ray diffraction data set was collected on a Rigaku Mercury CCD X-ray diffractometer with graphite monochromated MoK_α radiation (λ = 0.71073 Å) using a ω scan technique. Crystal clear software was used for data reduction and empirical absorption correction. The structure was solved by the direct methods using the Siemens SHELXTL™ version 5 package of crystallographic software. The difference Fourier maps based on the atomic positions yield all non-hydrogen atoms. The structure was refined using a full-matrix least-squares refinement on F^2. All atoms were refined anisotropically. The summary of crystallographic data and structure analysis is given in Table- 1. The selected bond lengths and bond angles are listed in Table-2.

Crystallographic data for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 883747 for **1**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (44) 1223 336-033; e-mail: deposit@ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

X-ray diffraction analysis reveals that the structure of complex **1** consists of cationic $[Y(C_6H_4NO_2)_2(H_2O)_4]_n^{n+}$ 1-D chains and chloride anions, as shown in Fig. 1. All the crystallographically independent atoms are in general positions except for Y(1) and Cl(1) atoms. There is only one crystallographically independent yttrium atom and its occupancy must be set to 0.5 to get a rational structure model and thermal

TABLE-1
SUMMARY OF CRYSTALLOGRAPHIC DATA AND STRUCTURE ANALYSIS OF $[Y(C_6H_4NO_2)_2(H_2O)_4]_n \cdot nCl$

Formula	$C_{12}H_{16}N_2O_8ClY$
Formula weight	440.63
Colour	Colourless
Crystal size/mm^3	0.32 0.21 0.20
Crystal system	Orthorhombic
Space group	Pbcn
a (Å)	8.9401(14)
c (Å)	19.576(3)
V (Å^3)	10.0063(13)
Z	4
$2\theta_{max}$ (°)	50
Index ranges	$-10 \le h \le 8, -23 \le k \le 23, -11 \le l \le 11$
Reflections collected	10289
Independent, observed reflections (R_{int})	1539, 1256 (0.0759)
$d_{calcd.}$ (g/cm^3)	1.671
μ (mm^{-1})	3.523
T (K)	293.15
F(000)	888
R1, wR2	0.0421, 0.0991
S	1.002
Largest and mean Δ/σ	0, 0
Δρ(max, min) (e/Å^3)	0.511, -0.540

TABLE-2
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (°)

Y(1)-O(2)#1	2.288(3)
Y(1)-O(2)	2.288(3)
Y(1)-O(1)#2	2.309(3)
Y(1)-O(1)#3	2.309(3)
Y(1)-O(2W)#1	2.400(3)
Y(1)-O(2W)	2.400(3)
Y(1)-O(1W)#1	2.452(3)
Y(1)-O(1W)	2.452(3)
O(2)#1-Y(1)-O(2)	96.82(14)
O(2)#1-Y(1)-O(1)#2	98.78(11)
O(2)-Y(1)-O(1)#2	147.18(11)
O(2)#1-Y(1)-O(1)#3	147.18(11)
O(2)-Y(1)-O(1)#3	98.78(11)
O(1)#2-Y(1)-O(1)#3	83.16(15)
O(2)#1-Y(1)-O(2W)#1	77.95(12)
O(2)-Y(1)-O(2W)#1	70.16(12)
O(1)#2-Y(1)-O(2W)#1	141.53(12)
O(1)#3-Y(1)-O(2W)#1	80.49(11)
O(2W)#1-Y(1)-O(2W)	131.22(18)
O(2)#1-Y(1)-O(1W)#1	141.14(11)
O(2)-Y(1)-O(1W)#1	69.33(11)
O(1)#2-Y(1)-O(1W)#1	80.51(11)
O(1)#3-Y(1)-O(1W)#1	71.65(12)
O(2W)#1-Y(1)-O(1W)#1	125.67(12)
O(2W)-Y(1)-O(1W)#1	71.44(12)
O(2)#1-Y(1)-O(1W)	69.33(11)
O(2)-Y(1)-O(1W)	141.14(11)
O(1)#2-Y(1)-O(1W)	71.65(12)
O(1)#3-Y(1)-O(1W)	80.51(11)
O(2W)#1-Y(1)-O(1W)	71.44(12)
O(2W)-Y(1)-O(1W)	125.67(12)
O(1W)#1-Y(1)-O(1W)	142.59(18)
O(2)#1-Y(1)-O(2W)	70.16(12)
O(2)-Y(1)-O(2W)	77.95(12)
O(1)#2-Y(1)-O(2W)	80.49(11)
O(1)#3-Y(1)-O(2W)	141.53(12)

Symmetry codes: #1 -x+1, y, -z+3/2; #2 x, -y, z-1/2; #3 -x+1, -y, -z+2

displacement parameter. The Y(1) atom is in an eight coordination environment, bound by eight oxygen atoms, of which four are from four water molecules and others are from four isonicotinate anions, yielding a distorted square anti-prism with the top and bottom planes defined by O(1W), O(1)(x, -y, -1/2+z), O(2W), O(2)(1-x, y, 3/2-z) and O(2W)(1-x, y, 3/2-z), O(1)(1-x, -y, 2-z), O(1W)(1-x, y, 3/2-z), O(2) atoms, respectively. The bond lengths of Y-$O_{isonicotinic\ acid}$ range from 2.288(3) Å to 2.309(3) Å with an average value of 2.299(3) Å, which is obviously shorter than that of Y-O_{water} being 2.400(3) Å and 2.452(3) Å, suggesting that isonicotinate anions have a stronger affinity to Y(III) ion than that of water. The Y^{III} ions are linked by two μ_2-isonicotinate anions to construct a 1-D chain running along the c axis with the Y···Y distance of *ca.* 5.024 Å. In **1**, there is no π…π stacking interactions established between the adjacent isonicotinate anions. The 1-D $[Y(C_6H_4NO_2)_2(H_2O)_4]_n^{n+}$ chains are interconnected together by chloride anions, yielding a 2-D layer extending along the ac plane (Fig. 2). The 2-D layers are further held together *via* hydrogen bonds to form a 3-D framework, as shown in Fig. 3.

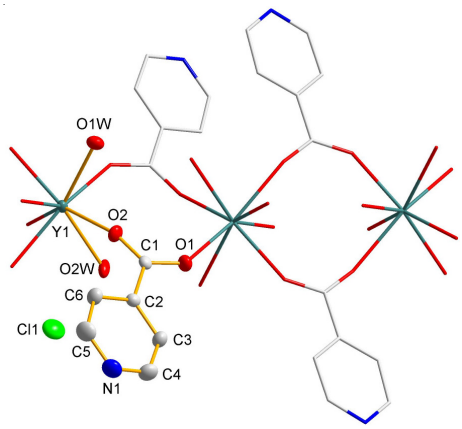

Fig. 1. Molecular structure of $[Y(C_6H_4NO_2)_2(H_2O)_4]_n \cdot nCl$ with 40 % thermal ellipsoids

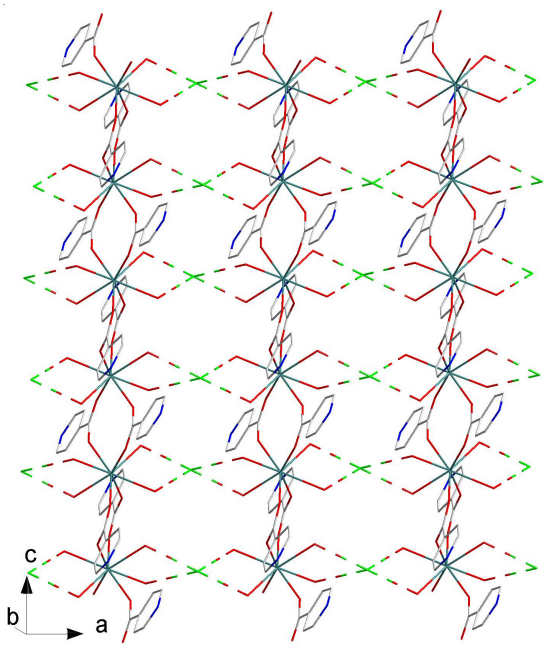

Fig. 2. 2-D layer constructed by Cl···O hydrogen bonds of $[Y(C_6H_4NO_2)_2(H_2O)_4]_n \cdot nCl$ in wires representation

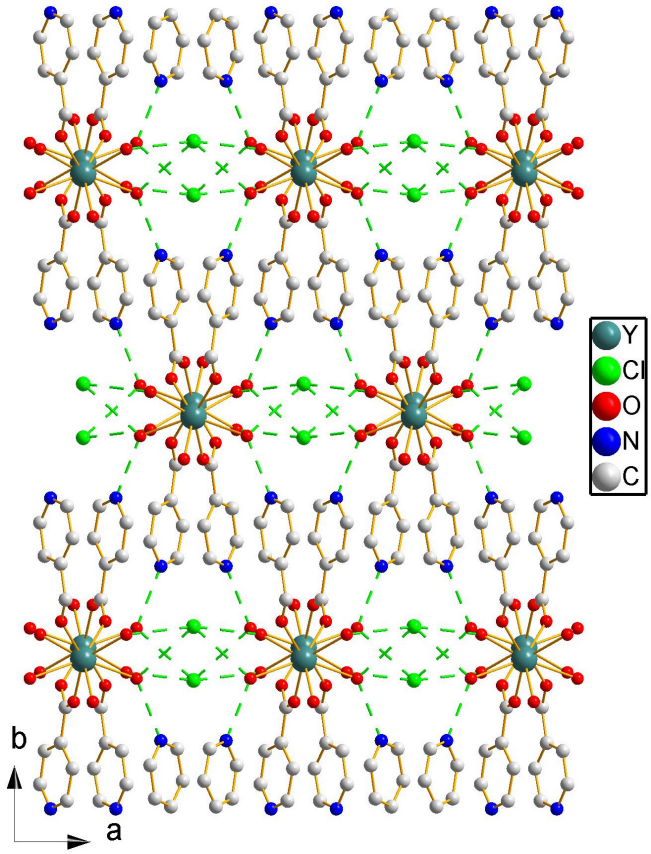

Fig. 3. Packing diagram of $[Y(C_6H_4NO_2)_2(H_2O)_4]_n \cdot nCl$ with the dashed lines representing hydrogen bonds (Å): Cl1···O1W(-1+x, y, z) 3.235(2), Cl1···O1W(1-x, y, 3/2-z) 3.235(2), Cl1···O2W 3.042(2), Cl1···O2W(-x, y, 3/2-z) 3.042(2), O1W···N1(1/2+x, -1/2+y, 3/2-z) 2.7145(3)

In conclusion, we successfully synthesized a novel lanthanide complex $[Y(C_6H_4NO_2)_2(H_2O)_4]_n \cdot nCl$ *via* a hydrothermal reaction. The crystal structure of the title complex is characterized by a 1-D chain-like motif. The 1-D chains are interconnected *via* hydrogen bonds to yield a 3-D supramolecular framework.

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