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Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713455674>

Syntheses and characterization of 2-D lanthanide complexes based on and isonicotinate *N*-oxide ligand

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First published on: 01 April 2010

To cite this Article Yin, Wen-Yu , Tang, Xiao-Yan , Yang, Jie , Ma, Yun-Sheng and Yuan, Rong-Xin(2010) 'Syntheses and characterization of 2-D lanthanide complexes based on and isonicotinate *N*-oxide ligand', *Journal of Coordination Chemistry*, 63: 7, 1157 – 1164, First published on: 01 April 2010 (iFirst)

To link to this Article: DOI: 10.1080/00958971003753823

URL: <http://dx.doi.org/10.1080/00958971003753823>

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Syntheses and characterization of 2-D lanthanide complexes based on SO_4^{2-} and isonicotinate *N*-oxide ligand

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(Received 14 July 2009; in final form 23 December 2009)

The lanthanide complexes $[\text{Ln}_2(\text{INO})_2(\text{SO}_4)_2(\text{H}_2\text{O})_4] \cdot \text{H}_2\text{O} \mathbf{1} \cdot \text{Ln}$ ($\text{Ln} = \text{Ce}, \text{Pr}, \text{and Nd}$; $\text{INO} = \text{isonicotinate } N\text{-oxide}$) were synthesized under hydrothermal conditions. The compounds are isostructural and crystallized in monoclinic space group $C2/c$. Ln(III) is nine-coordinate in a mono-capped square antiprism coordination environment. Ln(III) ions are linked by SO_4^{2-} , *N*-oxide groups and carboxylates from INO to give an inorganic chain. These chains are connected by INO ligands into a 2-D layer. These layers are joined through hydrogen bonding interactions into a 3-D crystal structure.

Keywords: Lanthanide; Isonicotinate *N*-oxide; Crystal structure

1. Introduction

The design and syntheses of metal–organic frameworks have drawn attention for potential applications as functional materials and also for their aesthetic structures [1–5]. Isonicotinate with the pyridine and carboxylate functional groups has proved to be a fascinating ligand to construct 3d/4f heterometallic coordination polymers [6–10]. Introducing a second ligand, 1,2-benzenedicarboxylic acid, helps form new heterometallic open frameworks [11]. Isonicotinate *N*-oxide (INO) has proved to be a good ligand to construct 2-D Mn, Co, and Cu complexes in the presence of N_3^- [12]. Reacting INO with Sm(III) or Tb(III) gives 1-D chain structures; the carboxylates of INO are only bidentate *syn–syn* bridging [13]. When SO_4^{2-} was used as coligand, three types of 3-D lanthanide complexes of INO were formed [14]. To synthesize Ln–Cu heterometallic coordination polymers based on INO , we combine Ln(III) ions ($\text{Ln} = \text{Ce}, \text{Pr}, \text{Nd}$) and Cu(II) sulfate. However, we were surprised to find that the copper was not present in the compound obtained. Here we report a new type of 2-D complex $[\text{Ln}_2(\text{INO})_2(\text{SO}_4)_2(\text{H}_2\text{O})_4] \cdot \text{H}_2\text{O} \mathbf{1} \cdot \text{Ln}$ ($\text{Ln} = \text{Ce}, \text{Pr}, \text{Nd}$) based on SO_4^{2-} and INO ligand.

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2. Experimental

2.1. Materials and methods

All the starting materials were of reagent grade used as purchased from Aldrich. Isonicotinic acid *N*-oxide (HINO) was prepared according to the literature [15]. Elemental analyses were performed on a PE 240 elemental analyzer. IR spectra were recorded on a NICOLET 380 spectrometer with pressed KBr pellets.

2.2. Syntheses of $[Ln_2(INO)_2(SO_4)_2(H_2O)_4] \cdot H_2O \cdot Ln$ ($Ln = Ce, Pr, Nd$)

These compounds were prepared by a similar experimental procedure except that appropriate lanthanide(III) nitrate hydrates were used. A typical procedure for the preparation of **1** · Ce is described. A mixture of HINO (0.0139 g, 0.1 mmol), $Ce(NO_3)_3 \cdot 6H_2O$ (0.0434 g, 0.1 mmol), $CuSO_4 \cdot 5H_2O$ (0.025, 0.1 mmol), and H_2O (5 mL) was transferred to a Teflon-lined autoclave and heated at 130°C for 5 days. The colorless block crystals were collected, washed with H_2O , and dried in air. Yield: 21 mg (50% based on Ce). Elemental Anal. $C_{12}H_{18}N_2Ce_2O_{19}S_2$ Calcd (%): C 17.19, H 2.16, N 3.34; found (%): C 17.30, H 2.30, N 3.11; IR (KBr): The strong and broad stretching bands centered at 3386 cm^{-1} are attributed to $\nu(O-H)$ of coordinated H_2O . The two very strong bands centered at 1605 and 1388 cm^{-1} , attributed to the $\nu_{as}(COO)$ and $\nu_s(COO)$ of INO with $\Delta = 217\text{ cm}^{-1}$ confirm coordination to Ce(III) by bidentate-bridging carboxylates [16]. The strong band at 1200 cm^{-1} was assigned to the characteristic absorption of N–O. The strong bands centered at 1118 and 1047 cm^{-1} were assigned to SO_4^{2-} .

2.2.1. 1 · Pr. Yield 18.6 mg (44% based on Pr). Elemental Anal. $C_{12}H_{18}N_2Pr_2O_{19}S_2$ Calcd (%): C 17.02, H 2.14, N 3.31; found (%): C 17.30, H 2.35, N 3.05; IR (KBr): 3396 (br, O–H), 1602 (s, C–O), 1391 (vs, C–O), 1191 (s, N–O), 1120 (s, SO_4^{2-}), 1049 (vs, SO_4^{2-}).

2.2.2. 1 · Nd. Yield 18.9 mg (45% based on Nd). Elemental Anal. $C_{12}H_{18}N_2Nd_2O_{19}S_2$ Calcd (%): C 17.15, H 2.16, N 3.33; found (%): C 16.90, H 2.32, N 3.08; IR (KBr): 3379 (br, O–H), 1605 (vs, C–O), 1398 (vs, C–O), 1199 (s, N–O), 1118 (vs, SO_4^{2-}), 1044 (s, SO_4^{2-}).

2.3. X-ray crystallography

Single crystals with dimensions of $0.30\text{ mm} \times 0.20\text{ mm} \times 0.10\text{ mm}$ for **1** · Ce, $0.30\text{ mm} \times 0.20\text{ mm} \times 0.10\text{ mm}$ for **1** · Pr and $0.40\text{ mm} \times 0.20\text{ mm} \times 0.20\text{ mm}$ for **1** · Nd were selected for indexing and intensity data collection at 293(2) K on a Rigaku SCX mini diffractometer using graphite-monochromated Mo-K α radiation ($\lambda = 0.71073\text{ \AA}$) at room temperature. A hemisphere of data was collected in the θ range $3.02\text{--}27.48^\circ$ for **1** · Ce, $3.03\text{--}27.48^\circ$ for **1** · Pr, and $3.03\text{--}27.48^\circ$ for **1** · Nd using a narrow-frame method with scan widths of 0.30° in ω and an exposure time of 5 s per frame. The numbers of observed and unique reflections are 11378 and 2587 ($R_{int} = 0.0534$) for **1** · Ce, 11275

and 2573 ($R_{\text{int}} = 0.0345$) for $\mathbf{1} \cdot \mathbf{Pr}$, and 11236 and 2562 ($R_{\text{int}} = 0.0303$) for $\mathbf{1} \cdot \mathbf{Nd}$. Cell parameters were refined using *CrystalClear* [17] on all observed reflections. The collected data were reduced using *CrystalClear*, and an absorption correction (multiscan) was applied. The reflection data were also corrected for Lorentz and polarization effects. The structures were solved by direct methods and refined on F^2 by full-matrix least-squares using SHELXTL [18]. All non-hydrogen atoms were located from the Fourier maps and were refined anisotropically. All hydrogens were refined isotropically, with the isotropic vibration parameters related to the atom to which they are bonded. The crystallographic and refinement details of $\mathbf{1} \cdot \mathbf{Ln}$ are listed in table 1. Selected bond lengths and angles are given in table 2 for $\mathbf{1} \cdot \mathbf{Ln}$.

3. Results and discussion

3.1. Syntheses

Compounds $\mathbf{1} \cdot \mathbf{Ln}$ were obtained through hydrothermal reactions of INO, $\text{Ln}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ at 130°C instead of Cu–Ln heterometallic coordination polymers. This polymer shows that INO affinity is greater for lanthanide than for copper ions. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ provides the SO_4^{2-} in the reaction system. Temperature is important in producing the single phase of title compound. When the reaction temperature is increased to 150°C , the title compound formed together with an unknown blue precipitate which was not characterized. Upon lowering the reaction temperature to 110°C , pale precipitate was formed in the system. The molar ratio of the starting materials is also important in the formation of pure product. The increase of the amount of INO [$\text{INO} : \text{Ln}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O} : \text{CuSO}_4 \cdot 5\text{H}_2\text{O} = 2 : 1 : 1$] favors the

Table 1. Crystal data and structure refinement details for $\mathbf{1} \cdot \mathbf{Ln}$.

Compound	$\mathbf{1} \cdot \mathbf{Ce}$	$\mathbf{1} \cdot \mathbf{Pr}$	$\mathbf{1} \cdot \mathbf{Nd}$
Empirical formula	$\text{C}_{12}\text{H}_{18}\text{N}_2\text{Ce}_2\text{O}_{19}\text{S}_2$	$\text{C}_{12}\text{H}_{18}\text{N}_2\text{Pr}_2\text{O}_{19}\text{S}_2$	$\text{C}_{12}\text{H}_{18}\text{N}_2\text{Nd}_2\text{O}_{19}\text{S}_2$
Formula weight	838.64	840.22	846.90
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$C2/c$	$C2/c$	$C2/c$
Unit cell dimensions (\AA , $^\circ$)			
a	19.441(4)	19.398(4)	19.384(4)
b	7.5036(15)	7.4836(15)	7.4734(15)
c	17.222(3)	17.203(3)	17.150(3)
β	116.38(3)	116.18(3)	116.04(3)
Volume (\AA^3), Z	2250.7(10), 4	2241.1(9), 4	2232.2(9), 4
Calculated density (g cm^{-3})	2.475	2.490	2.520
$F(000)$	1616	1624	1632
$\mu(\text{Mo-K}\alpha)$ (cm^{-1})	42.78	45.82	48.87
Reflections collected	11378	11275	11236
Independent/observed reflections	2587/2205	2573/2365	2562/2356
Data/parameters	2587/168	2573/168	2562/177
Goodness-of-fit on F^2	1.03	1.13	1.14
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0392$, $wR_2^a = 0.0760$	$R_1 = 0.0483$, $wR_2^a = 0.1551$	$R_1 = 0.0207$, $wR_2^a = 0.0417$
$(\Delta\rho)_{\text{max}}$ and $(\Delta\rho)_{\text{min}}$ (e \AA^{-3})	0.647 and -1.282	1.831 and -1.422	0.538 and -0.655

^a $R_1 = \sum \|F_o\| - |F_c| / \sum \|F_o\|$; $wR_2 = [\sum w(F_o^2 - F_c^2)^2 / \sum w(F_o^2)]^{1/2}$.

Table 2. Selected bond lengths and angles for $\mathbf{1} \cdot \text{Ln}$.

	$\mathbf{1} \cdot \text{Ce}$	$\mathbf{1} \cdot \text{Pr}$	$\mathbf{1} \cdot \text{Nd}$
Ln(1)–O(1)	2.606(4)	2.584(8)	2.580(2)
Ln(1)–O(6)	2.566(4)	2.547(8)	2.525(2)
Ln(1)–O(8)	2.684(4)	2.669(8)	2.668(2)
Ln(1)–O(1W)	2.471(5)	2.463(10)	2.445(3)
Ln(1)–O(2W)	2.579(6)	2.574(10)	2.554(3)
Ln(1)–O(8C)	2.430(5)	2.413(8)	2.401(2)
Ln(1)–O(2A)	2.459(4)	2.437(9)	2.433(3)
Ln(1)–O(3B)	2.463(4)	2.448(9)	2.438(3)
Ln(1)–O(1D)	2.589(4)	2.577(8)	2.564(2)
O(1)–Ln(1)–O(1W)	74.69(15)	74.7(3)	74.74(9)
O(1)–Ln(1)–O(1D)	60.54(14)	60.1(3)	60.27(8)
O(1)–Ln(1)–O(2A)	75.02(14)	75.2(3)	75.20(8)
O(1)–Ln(1)–O(8C)	78.75(14)	79.1(3)	79.06(8)
O(1W)–Ln(1)–O(1D)	94.14(15)	93.7(3)	93.15(9)
O(1W)–Ln(1)–O(2A)	81.58(17)	81.6(3)	81.64(10)
O(1D)–Ln(1)–O(8C)	88.87(14)	89.3(3)	89.90(8)
O(8C)–Ln(1)–O(2A)	73.53(15)	73.7(3)	73.67(9)
O(6)–Ln(1)–O(8)	53.24(14)	53.5(2)	53.63(7)
O(6)–Ln(1)–O(2W)	70.00(17)	69.6(3)	69.94(9)
O(2W)–Ln(1)–O(3B)	70.59(17)	69.9(3)	69.42(9)
O(8)–Ln(1)–O(3B)	67.64(14)	67.5(3)	67.53(8)

Symmetry codes: A: $-x, -y, -z$; B: $x+1/2, y-1/2, z$; C: $-x+1/2, -y-1/2, -z$; D: $-x+1/2, -y+1/2, -z$.

formation of the uncharacterized pale blue precipitate, while reduction of the amount of INO [INO: Ln(NO₃)₃ · 6H₂O: CuSO₄ · 5H₂O = 0.5: 1: 1] yields only clear solution.

3.2. Crystal structures

Compounds [Ln₂(INO)₂(SO₄)₂(H₂O)₄] · H₂O $\mathbf{1} \cdot \text{Ln}$ (Ln = Ce, Pr, Nd) are isostructural and crystallized in monoclinic space group *C2/c*; $\mathbf{1} \cdot \text{Nd}$ will serve as an example. The asymmetric unit contains half of the [Nd₂(INO)₂(SO₄)₂(H₂O)₄] · H₂O molecule, with O3W on the *C2* axis. As shown in figure 1, Nd(III) is nine-coordinate, mono-capped square antiprism formed by two *N*-oxide groups [O1, O1D] from two INO, two carboxylate oxygens [O2A, O3B] from the other two INO, three oxygens [O6, O8, O8C] from two SO₄²⁻ anions and two coordinated waters [O1W, O2W]. The Nd–O distances range from 2.401(2) to 2.668(2) Å and are in the range of 2.299(3)–2.712(3) Å observed in [Nd(INO)(SO₄)(H₂O)] [14]. The O–Nd–O angle ranges from 60.27(8)° to 147.74°. In the inorganic moiety, two Nd ions are bridged by a pair of SO₄²⁻ anions in the 2.2100 coordination mode (Harris notation) [19] in a discrete dimer (scheme 1). The dimer itself has two additional Nd–O–C–O–Nd connectivities by two other INO ligands with two *syn–syn* bridging carboxylate groups. The neighboring dimers are connected by two INO ligands with their *N*-oxide groups as μ_2 -bridges binding two Nd ions to give an infinite chain running along the *b*-direction. The Nd ··· Nd separations are 4.115(1) Å within the dimer and 4.448(1) Å between the dimers. The chains are further linked along *a*-direction through the coordination connections of INO and Nd ions between the chains; ligands with bidentate carboxylate *syn–syn* bridges in one chain are coordinated

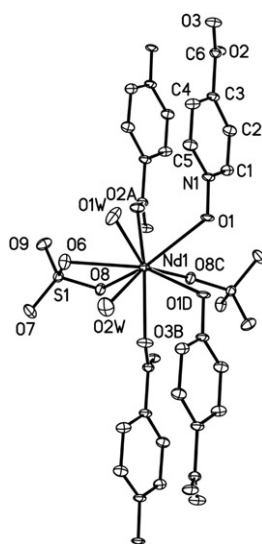
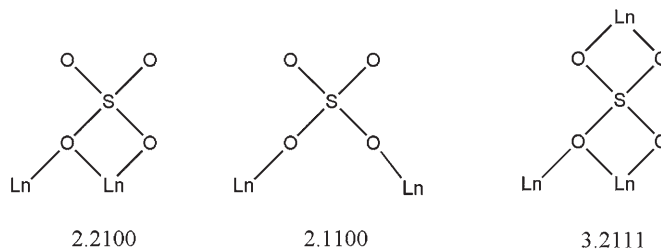


Figure 1. Building unit of structure **1·Nd** with the atom labeling scheme (thermal ellipsoids at 50% probability). Symmetry codes are the same as those in table 2.



Harris notation describes the binding mode as $[X.Y_1Y_2...Y_n]$, where X is the overall number of metals bound by the whole ligand, and each value of Y refers to the number of metal atoms attached to the different donor atoms.

Scheme 1. Coordination modes of the SO_4^{2-} ligand.

to pairs of Nd ions and in the adjacent chains *via* their *N*-oxide groups as monodentate μ_2 -bridges. Therefore, each INO connects four Nd ions, two in one chain through its monodentate *N*-oxide and two in the other chain through its *syn-syn* bridging carboxylate, creating a 2-D coordination sheet (figure 2). The layer structure is remarkably different from $[\text{Nd}(\text{INO})(\text{SO}_4)(\text{H}_2\text{O})]$ with a two-fold interpenetrated “3-D herringbone” network [8], where SO_4^{2-} adopts 3.2111 coordination (scheme 1). The structure may be compared with that of a 2-D layered compound $[\text{Gd}(\text{NNO})(\text{SO}_4)(\text{H}_2\text{O})]$ (NNO = nicotinate *N*-oxide), which contains inorganic chains that are composed of SO_4^{2-} , carboxylate, and *N*-oxide bridged Gd(III) ions [20]. However, in $[\text{Gd}(\text{NNO})(\text{SO}_4)(\text{H}_2\text{O})]$ the SO_4^{2-} is 2.1100 coordination mode (scheme 1).

In **1·Nd**, four intramolecular hydrogen bonds, $\text{O1W-H1A}\cdots\text{O9}$ [$x, 1+y, z$], 2.716(3) Å, 177(5)°; $\text{O2W-H2B}\cdots\text{O7}$ [$x, 1+y, z$], 3.323(4) Å, 179°; $\text{C5-H5}\cdots\text{O9}$ [$x, 1+y, z$], 3.233(4) Å, 162°; $\text{C1-H1}\cdots\text{O7}$ [$-x+1/2, -y-1/2, -z$], 2.310(4) Å, 167°,

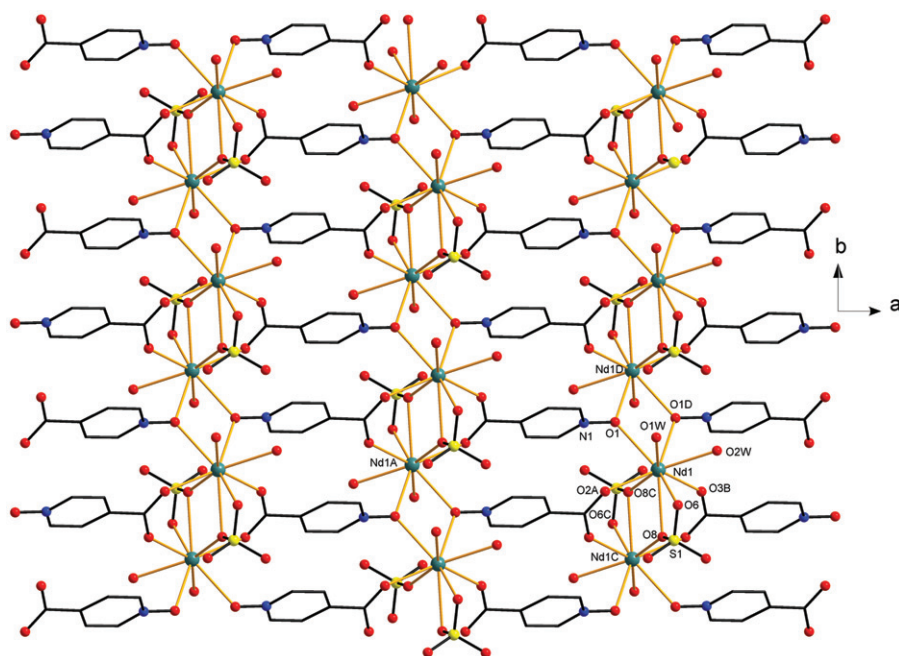


Figure 2. 2-D structure of $1 \cdot \text{Nd}$ viewed in the ab plane.

were observed between layers. Additionally, intermolecular hydrogen bonding contacts $\text{O3W-H3A} \cdots \text{O6}$ [$x, 1+y, z$], 3.146(3) Å, 126(4)°; $\text{O3W-H3A} \cdots \text{O7}$ [$x, 1+y, z$], 2.815(4) Å, 163(6)°; $\text{O1W-H1B} \cdots \text{O9}$ [$-x+1/2, y+1/2, -z+1/2$], 2.688(4) Å, 169(4)°; $\text{O2W-H2A} \cdots \text{O3W}$ [$x, y-1, z$], 2.780(5) Å, 171°, link the layers to form a 3-D supramolecular network (figure 3).

The structures of $1 \cdot \text{Ln}$ (Ln = Ce, Pr) are identical to that of $1 \cdot \text{Nd}$ except that the cell volumes decrease in the sequence $1 \cdot \text{Ce} > 1 \cdot \text{Pr} > 1 \cdot \text{Nd}$, as a result of lanthanide contraction. The Ln \cdots Ln distances over the $\mu\text{-O8}$ bridge are 4.143(1) and 4.124(1) Å for $1 \cdot \text{Ce}$ and $1 \cdot \text{Pr}$, while Ln \cdots Ln distances over the $\mu\text{-O1}$ bridge are 4.487(1) and 4.467(1) Å for $1 \cdot \text{Ce}$ and $1 \cdot \text{Pr}$, respectively.

In summary, we report three new 2-D lanthanide INO coordination polymers $1 \cdot \text{Ce}$, $1 \cdot \text{Pr}$, and $1 \cdot \text{Nd}$. The crystal structure is significantly different from the reported lanthanide INO complex $[\text{Nd}(\text{INO})(\text{SO}_4)(\text{H}_2\text{O})]$ that has 3-D structure [14]. Further work is in progress to study the lanthanide INO complexes under different reaction conditions.

Supplementary material

Electronic supplementary information (ESI) available: figure S1, figure S2, and figure S3 IR spectra of $1 \cdot \text{Ln}$ (Ln = Ce, Pr, Nd). Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 739473, 739474, and 739475 for $1 \cdot \text{Ce}$, $1 \cdot \text{Pr}$, and $1 \cdot \text{Nd}$, respectively.

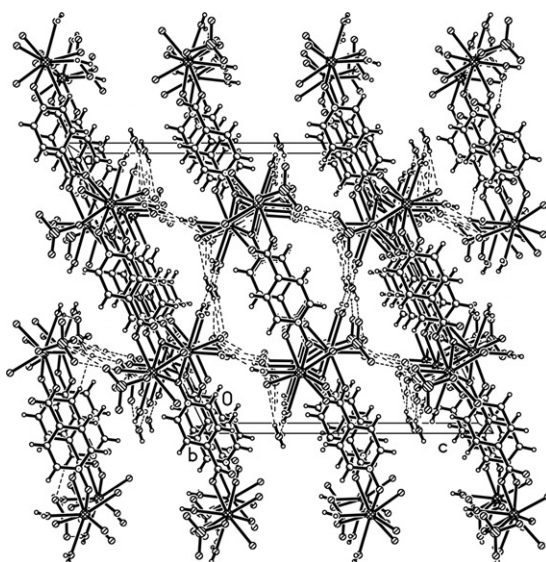


Figure 3. Hydrogen bond strengthened 3-D cell packing diagram of $1 \cdot \text{Nd}$, view down the *b*-axis.

The copies of this information can be had free of charge from CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44-1223-336-033; E-mail: deposit@ccdc.ac.uk).

Acknowledgments

This work was supported by the Natural Science Fund of Jiangsu Province of China (No. 08KJB150001, No. BK2006044, No. 06KJA43014) and NSFC (No. 20971017).

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