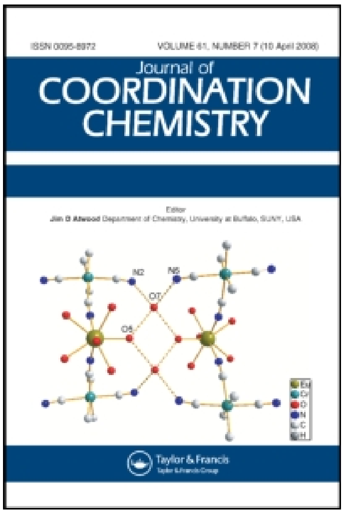

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Synthesis, structure, and magnetic properties of a self-assembled salicylaldimine neodymium(III) nitrate coordination polymer

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To cite this Article Li, Han , Lu, Weimin , Chen, Fang , Dong, Xue and Dai, Yihu(2010) 'Synthesis, structure, and magnetic properties of a self-assembled salicylaldimine neodymium(III) nitrate coordination polymer', Journal of Coordination Chemistry, 63: 1, 106 — 114, First published on: 05 October 2009 (iFirst)

To link to this Article: DOI: 10.1080/00958970903302699

URL: http://dx.doi.org/10.1080/00958970903302699

Journal of Coordination Chemistry
Vol. 63, No. 1, 10 January 2010, 106–114

Synthesis, structure, and magnetic properties of a self-assembled salicylaldimine neodymium(III) nitrate coordination polymer

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(*Received 4 February 2009; in final form 10 July 2009*)

Complex formed by the reaction of neodymium nitrate with Schiff base has been synthesized and characterized by elemental analysis and IR spectroscopy. The crystal structure and magnetic properties of the self-assembled complex, $[Nd_2(H_2salen)_3(NO_3)_6]_n$ ($H_2salen =$ N,N′-ethylene*bis*(salicylideneimine)), are reported. The central Nd(III) ion is nine-coordinate with a typical tricapped trigonal prismatic coordination geometry with an infinite 2-D net structure, in which every Schiff-base ligand links two Nd(III) ions, and Nd(III) ions as nodes make up hexagons with $\bar{3}$ axes passing through the centers. The susceptibility data of the complex are well-fitted to the Curie–Weiss law and anti-ferromagnetic interactions take place between intermolecular adjacent paramagnetic ions.

Keywords: Coordination polymer; Neodymium; X-ray crystal structure; Magnetic property; Self-assembly

1. Introduction

Crystal engineering of the structures with various topologies has attracted attention because of its applications in numerous fields, e.g., catalysis, electrical conductivity, molecular magnets, and host-guest chemistry [1–5]. Therefore, the synthesis of 1-D, 2-D, or 3-D coordination polymers is especially an interesting research area.

Transition metals as the coordination center have been studied broadly due to their significance as effective catalysts in oxygenation and asymmetric catalysis [6–9] and other applications as multifunctional materials [10–15]. N,N′-Ethylene*bis*(salicylidenimine) (H_2salen) complexes with d-block metals are widely used for asymmetric catalytic reactions [16–19]. Relative to d-electron elements, the use of lanthanide ions in crystal engineering is less developed, but their potential applications attract us to search for new complexes. Although numerous lanthanide derivatives of salen ligands have been reported, most are not well characterized with structures proposed on the basis of elemental composition and spectroscopic data [20–23], except for some complexes of known structures [24–27]. The problem of protonated or deprotonated phenolic hydroxyl groups in H_2salen is still argumentative.

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Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2010 Taylor & Francis
DOI: 10.1080/00958970903302699

Herein a self-assembled, 2-D salicylaldimine neodymium(III) nitrate coordination polymer with high symmetry is reported and some of its properties are discussed. To the best of our knowledge, it is the first documented report of such a structure in salicylaldimine lanthanide complexes and is a rare example of exclusive oxygen-donor coordination of salen-type ligands.

2. Experimental

2.1. *General*

All chemicals of reagent grade were obtained from commercial sources and used without purification. $Nd(NO_3)_3 \cdot 6H_2O$ was also obtained from commercial sources, but the compound had slightly deliquesced before use. H_2salen was prepared from the reaction of salicylaldehyde and ethylenediamine by routine methods. IR spectra were recorded using KBr pellets from 4000 to 400 cm^{-1} on a Nicolet 560 spectrophotometer and elemental analyses were performed on a Cobra-Erba 1110 instrument.

2.2. *Synthesis of the title complex*

A total of 0.09 mmol (24.9 mg) of $Nd(NO_3)_3$ hydrate in 5 mL of ethanol was slowly added to an ethanol solution (25 mL) of H_2salen (0.18 mmol, 79.8 mg) with stirring at 338 K. The initial bright yellow solution turned light green upon the addition of $Nd(NO_3)_3$ hydrate. After filtration, the filtrate was maintained at 338 K for 2 h and then cooled to room temperature. Green single crystals suitable for X-ray diffraction were obtained after 24 h in 69% yield.

Anal. Calcd for $Nd_2(C_{16}H_{16}N_2O_2)_3(NO_3)_6$ (%): C, 39.33; H, 3.30; N, 11.47. Found (%): C, 38.62; H, 3.51; N, 10.12. IR (KBr/cm^{-1}): ν 3440–3480 (OH), ν 2360 (O–H–N), ν 1650 (C=N), ν 1602, 1450 (C=C, phen ring), ν 1280 (C–O), ν 1470–1430, 1360–1310 (NO_3), ν 754 (C–H, out of phen ring bend), ν 483 (Nd–O).

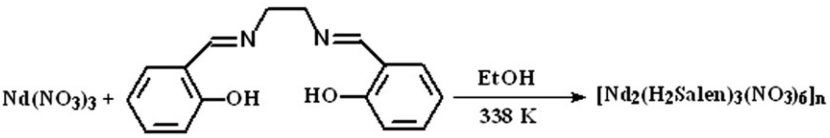
2.3. *X-ray structural determination*

A green needle of the title complex with approximate dimensions $0.25 \times 0.05 \times 0.03\,mm^3$ was mounted on a glass fiber and X-ray intensity data was collected on a Rigaku RAXIS-RAPID diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71069$ Å) by the $\omega - 2\theta$ scan technique. Usual Lp and adsorption corrections were applied.

The structure was solved by direct methods and expanded using the Fourier technique. Structure refinement was carried out by full-matrix least-squares using the SHELXTL/PC program package [28]. H atoms were located from the difference Fourier map and refined isotropically, where H1 was fixed. Anisotropic refinement including all non-H atoms converged to agreement factors $R = 0.0426$ and $wR = 0.1277$, where $w = 1/[0.0001F_o^2 + 0.6000\sigma(F_o^2)]/(4F_o^2)$. Atomic scattering factors were taken from International Tables for X-ray crystallography [29]. Crystal data collection and refinement parameters are summarized in table 1.

Table 1. Crystallographic data for the title complex.

Empirical formula	$C_{48}H_{48}N_{12}O_{24}Nd_2$
Formula weight	1454.79
Temperature (K)	293
Crystal system	Trigonal
Space group	*P-3c1*
Symmetry International Tables number	165
Unit cell dimensions (Å, °)	
a	16.106(4)
b	16.106(4)
c	16.405(4)
α	90
β	90
γ	120
V (Å^3)	3685.2(15)
Z	2
Calculated density (g cm^{-3})	1.305
θ range for data collection (°)	1.064–27.44
Limiting indices	$-20 \le h \le 20$; $-19 \le k \le 20$; $-20 \le l \le 21$
Reflections collected	34,741
Unique reflections	2802
$F(000)$	1440.00
Goodness-of-fit on F^2	1.002
Maximum shift (Δ/σ)	0.001
R indices	$R_1 = 0.0426$, $wR_2 = 0.1277$
Maximum/minimum $\Delta\rho$ (e Å^{-3})	0.75/−0.68



Scheme 1. The reaction of $Nd(NO_3)_3$ hydrate with H_2salen in EtOH.

3. Results and discussion

3.1. *Formation of [$Nd_2(H_2salen)_3(NO_3)_6$]$_n$*

Formation of the Nd-H_2salen complex was readily accomplished by the reaction of $Nd(NO_3)_3$ hydrate with H_2salen (1 : 2) in EtOH (scheme 1). The immediate color change upon the addition of $Nd(NO_3)_3$ solution revealed the coordination.

3.2. *Characterization and spectroscopic studies*

The complex was shown, by IR and elemental analysis, to have an empirical formula of $[Nd_2(H_2salen)_3(NO_3)_6]_n$, which was later confirmed by X-ray crystallography. In the IR spectra, broad bands at 3440–3480 cm^{-1} and ~2360 cm^{-1} indicate that six-membered rings were formed by intramolecular hydrogen bonds between the phenolic hydroxyl and nitrogen of azomethine groups [30]. Hydroxyl absorption frequency of the conjugate-chelate ring involving $OH\cdots N{=}C$ hydrogen bond extends from approximately 3300 to 2300 cm^{-1} [30]. The value observed for our complex (2360 cm^{-1}) was less than those of corresponding complexes (2650 and 2576 cm^{-1}), indicating that the bond between hydrogen and oxygen was quite weak. Compared with free ligand, a shift of the C–O stretching frequency and appearance of a new peak at 483 cm^{-1} afforded evidence for the participation of phenolic oxygen in metal–ligand bonding. The strong band attributable to ν(C=N) at 1632 cm^{-1} in the free ligand shifted to 1650 cm^{-1} in the complex, as for corresponding complex [27]. Due to the complicated absorptions around 1350–1640 cm^{-1}, it is hard to judge from the IR spectrum whether there is coordination to nitrogen of the imine groups.

3.3. *Crystal structure of the complex*

The single crystal X-ray structure determination is consistent with spectroscopic characterization and the coordination is explicitly confirmed. Each Nd(III) is nine-coordinate with three phenolic oxygens from three different H_2salen molecules and six oxygens from three bidentate nitrates, as shown in figure 1, in a tricapped trigonal prism. Unlike previously reported halide complexes [31], no coordination took place between nitrogen and Nd(III). Instead, one phenolic oxygen from a H_2salen

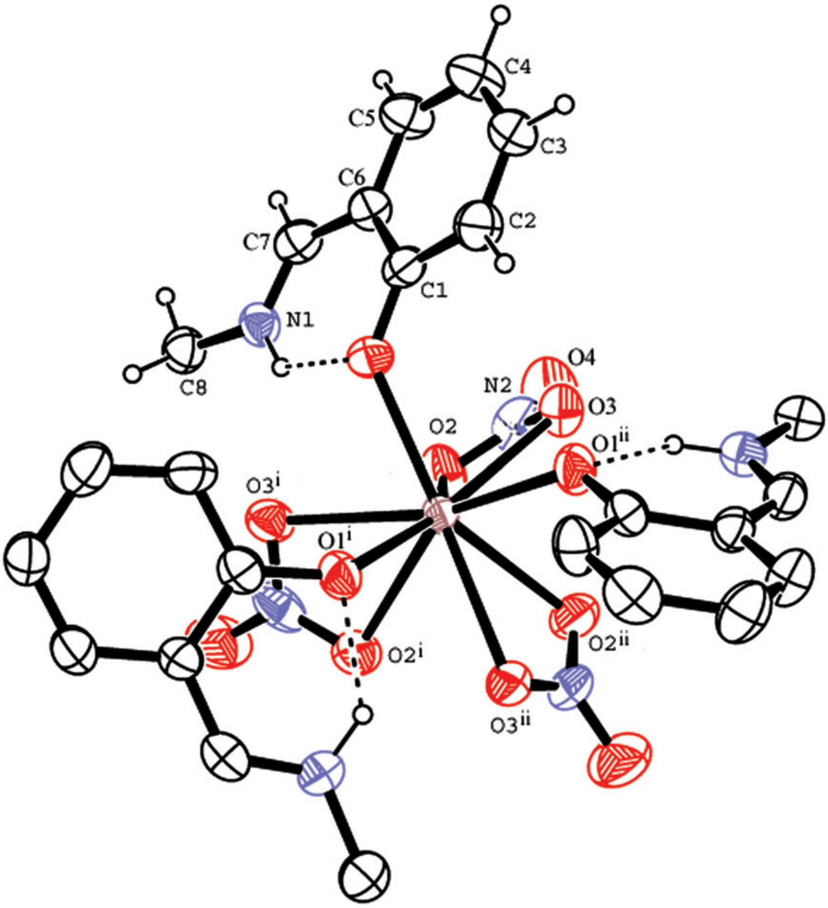


Figure 1. Anisotropic displacement ellipsoid representation of the coordination sphere of Nb. The ellipsoids are drawn at 30% probability level and hydrogens are represented by spheres of arbitrary radii.

coordinates with Nd(III), and another phenolic oxygen links neighboring Nd(III) ions. Thus, each H_2salen ligand is a bridge between two Nd(III) ions. As every Nd(III) links three H_2salen ligands and every H_2salen joins two Nd(III) ions, the complex forms a self-assembled, 2-D net structure as shown in figure 2(a), in which Nd(III) as nodes make up hexagons along *a* and *b* axes with the diameter of the pore about 9.2 Å and

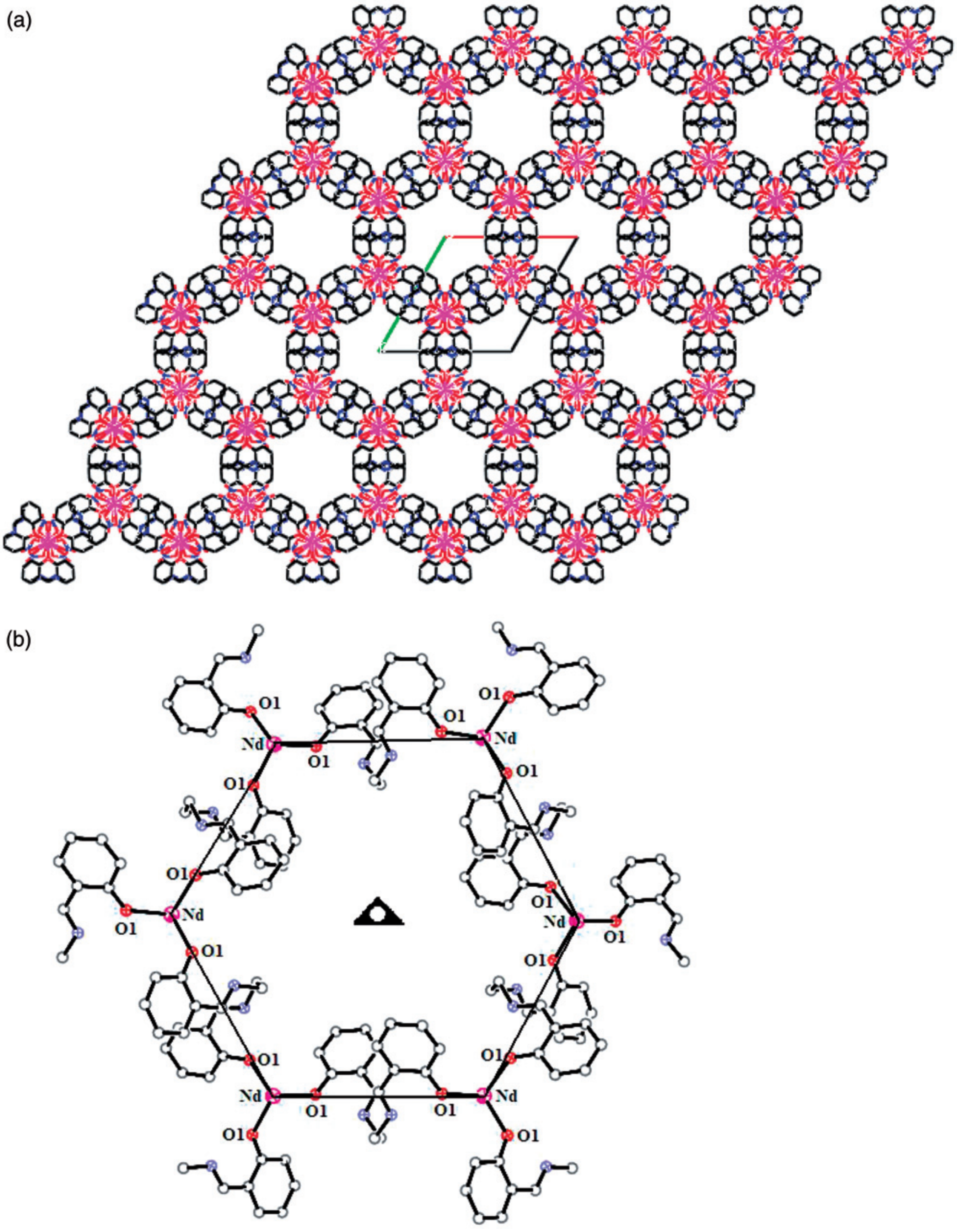


Figure 2. View of the packing of the title complex along the *c*-axis showing (a) the 2-D polymeric net structure of the complex (H atoms have been omitted for clarity). (b) Nd(III) ions as nodes making up hexagon with $\bar{3}$ axis. H atoms and (NO_3) groups have been omitted for clarity.

with $\bar{3}$ axes passing through the centers as shown in figure 2(b). The structure is unique and completely different from the corresponding polymeric lanthanide H_2salen complexes [25, 27]. The Nd(III)'s in the coordination polymer are assembling and templating joints for the salen-type ligand.

Selected bond lengths and angles are listed in table 2. The Nd–O bond lengths can be divided into two groups due to the nature of the oxygen. As the nitrate groups have the same coordination formation, two Nd–O bond lengths are similar with 2.549(5) and 2.586(6) Å, respectively. The distances between Nd and phenolic oxygens are all the same with 2.344(5) Å and shorter than average Nd–$O_{phenolic}$ distances in similar complexes (table 3), in which phenolic hydroxyls exist as ionized forms [32, 33]. In the title complex, the Schiff-base ligands are neutral rather than ionized phenolic hydroxyl groups, like other lanthanide(III) complexes $[La(H_2L)(NO_3)_3(MeOH)_2]_n$ (H_2L = N,N′-ethylene*bis*(salicylideneimine) [25], $[La_2(H_2L)_4(NO_3)_6]_n$ (H_2L = N,N′-*bis*(-salicylidene)-1,4-butanediamine) [27], and $[La(H_2L)(H_2O)_4]Cl_3$ (H_2L = N,N′-ethylene-3,3-(3,6-dioxactane-1,8-diyldioxy)*bis*(2-hydroxybenzylideneimine) [34].

Table 2. Selected bond lengths and angles of the title complex (Å, °).

	Distance		Distance		Angles		Angles
Nd–O1	2.344(4)	Nd–$O3^{ii}$	2.586(4)	O1–Nd–$O1^{i}$	94.00(17)	O2–Nd–$O2^{ii}$	74.4(2)
Nd–$O1^{i}$	2.345(5)	C1–O1	1.325(8)	O1–Nd–$O1^{ii}$	94.00(18)	$O2^{i}$–Nd–$O2^{ii}$	74.4(2)
Nd–$O1^{ii}$	2.344(6)	N2–O2	1.279(12)	$O1^{i}$–Nd–$O1^{ii}$	94.00(19)	O2–Nd–O3	50.8(2)
Nd–O2	2.549(5)	N2–O3	1.280(10)	O1–Nd–O2	79.67(16)	O2–Nd–$O3^{i}$	68.7(2)
Nd–$O2^{i}$	2.549(6)	N2–O4	1.217(10)	O1–Nd–$O2^{i}$	120.9(2)	O2–Nd–$O3^{ii}$	119.57(16)
Nd–$O2^{ii}$	2.549(6)	C7–N1	1.297(10)	O1–Nd–$O2^{ii}$	144.69(18)	O3–Nd–$O3^{i}$	114.73(17)
Nd–O3	2.586(5)	C8–N1	1.473(12)	O1–Nd–O3	76.42(19)	O3–Nd–$O3^{ii}$	114.73(19)
Nd–$O3^{i}$	2.586(7)			O1–Nd–$O3^{i}$	70.46(19)	$O3^{i}$–Nd–$O3^{ii}$	114.73(19)
				O1–Nd–$O3^{ii}$	160.76(14)	Nd–O1–C1	135.1(6)
				O2–Nd–$O2^{i}$	74.36(17)	Nd–O2–N2	96.0(4)
				O2–Nd–$O2^{i}$	74.4(2)	Nd–O3–N2	94.2(5)

Hydrogen bond data

D–H	A	H···A	*d*(D···A)	∠DHA
N1–H1	O1	1.864	2.617	133.84

Symmetry code: (i) $1+x, y, z$; (ii) $1+x, 1+y, z$.

Table 3. Comparison of bond lengths of the title complex with average values of corresponding salicylaldimine lanthanide(III) complexes (Å).

M	M–$O_{phenolic}$	M–$O_{nitrate}$	C–O	C–N	C=N	Reference
Nd	2.344(5)	2.568	1.325(8)	1.473(12)	1.297(10)	
Nd	2.428					[32]
Nd	2.354					[32]
Nd	2.289					[32]
Nd	2.416					[32]
Nd	2.429					[33]
La	2.43	2.672	1.293		1.300(7)	[25]
La	2.46(2)	2.68	1.299	1.461	1.271	[27]
L^{i}			1.348	1.466	1.274	[27]
L^{ii}			1.350	1.426	1.280	[33]
L^{iii}			1.338	1.464	1.288	[36]

L^{i} = $[La_2(H_2L)_4(NO_3)_6]_n$ (H_2L = N,N′-*bis*(salicylidene)-1,4-butanediamine).
L^{ii} = N,N′-*bis*(5-bromo-3-methoxysalicylidene)-1,2-phenylenediamine.
L^{iii} = N,N′-*bis*(3-hydroxysalicylidene)-2,2′-dimethylpropenyldiamine.

Whether the hydrogens of phenolic hydroxyl in lanthanide complexes exist deprotonated or protonated is controversial because there are deprotonated intramolecular N···OH hydrogen bonds in the free ligands. In the title complex, a hydrogen near the nitrogens of imine groups was found from the Fourier map when the structure was solved. The O···H and O···N distances are 1.864 and 2.617 Å, respectively, showing that intramolecular hydrogen bonds exist and that the hydrogens in the phenolic hydroxyls are protonated. Upon coordination to Nd^{3+}, the phenolic hydroxyl has dramatically increased the acidity and the protons, after ionization, resided on the imine nitrogens, leaving the whole Schiff-base ligand neutral. The positive and negative charges reside on azomethine and on the metal coordination sphere, respectively [35]. This tautomeric form of the phenolic hydroxyl group in the title complex may result from the reaction of the strong Lewis acid neodymium(III) trinitrate with the phenolic oxygen. The existence of a proton near the imine nitrogens explains the lack of coordination of nitrogen to the lanthanide ion.

The length of phenolic C–O bond (table 3), 1.325(8) Å, is notably shorter than those in the related free ligands [27, 33, 36] from the coordination of the phenolic oxygen with Nd(III). Compared with those in La complexes, which also have polymeric structures, the C–O bond is slightly longer. The distances of C–N and C=N in the complex are longer than those in the related free ligands [27, 33, 36]. The elongation of bonds between carbon and nitrogen, together with the notably short phenolic C–O distance, may be caused by the tautomeric form of the phenolic hydroxyl group.

3.4. *Magnetic properties*

The temperature dependence of the magnetic susceptibilities was examined for the pure crystal in the 5–300 K range in an applied field of 20 kOe. The magnetic properties of the complex in the form of $1/\chi_M$ *versus* T and $\chi_M T$ *versus* T plots are shown in figure 3. The χ_M is $2.21\,cm^3\,mol^{-1}\,K$ at 301.55 K and increases slightly as the temperature decreases to its maximum value of $2.39\,cm^3\,mol^{-1}\,K$ at 185.14 K, and then the value decreases as the temperature decreases. The observed $\chi_M T$ at room temperature is $2.22\,cm^3\,mol^{-1}\,K$, higher than the theoretical value of $1.64\,cm^3\,mol^{-1}\,K$ for free Nd(III), indicating that the tautomeric form of the phenolic hydroxyl group could be used to affect the magnetism of the compound through changes in the charge near the Nd(III) centers.

In the $1/\chi_M$ *versus* T plot, the observed susceptibility data are well-fitted to the Curie–Weiss law ($\chi_M = C/(T-\theta)$), with Weiss constant $\theta = -6.7383$ K. The observed behavior points to anti-ferromagnetic interactions in the complex. Because the closest interatomic Nd···Nd distance is more than 7 Å, the possibility of exchange between the Nd(III) ions is slight. It is difficult to distinguish, but the anti-ferromagnetic interaction may be attributed to weak lattice interactions or the ligands which are protonated.

4. Conclusion

Molecular design of Schiff-base complexes, including salicylideneimine complexes, has concentrated on directed variation of the binding of azomethine derivatives to allow buildup of different chelate ring size and combinations [37–39]. In this article, a self-assembled salicylaldimine neodymium(III) nitrate coordination complex with an

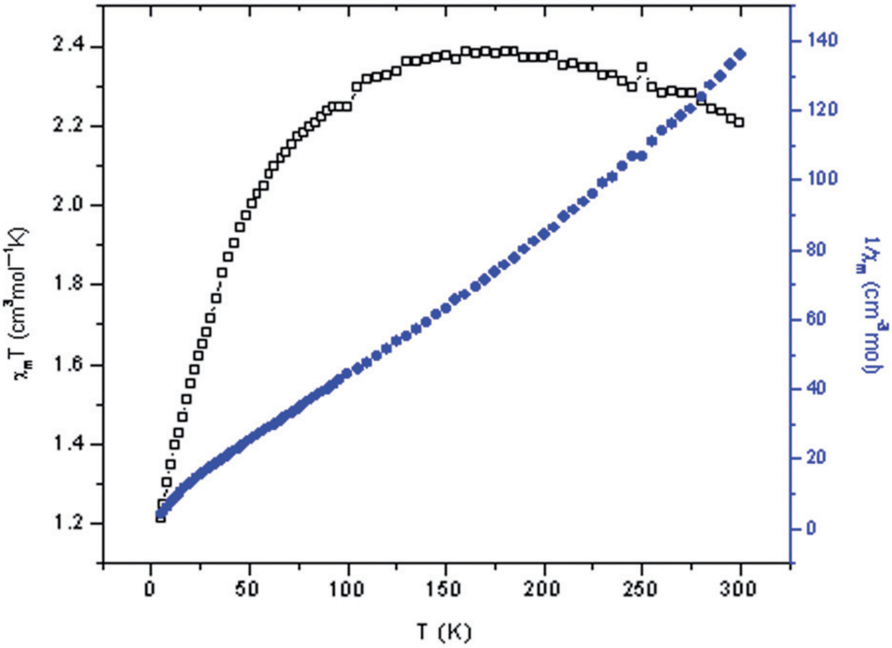


Figure 3. Plots of $\chi_M T$ (hollow dot) and $1/\chi_M$ (solid dot) *vs.* T for the complex.

unusual 2-D hexagonal net polymeric structure extends the coordination chemistry of Schiff bases with different coordination of the chelating ligands. The protonated but neutral tautomeric form of the phenolic hydroxyl in H_2salen may play an important role for the formation of the net structure, structural stability, and variation of salicylaldimine lanthanide complexes. The coordination pattern of H_2salen revealed by X-ray analysis provides valuable information for structural studies of other related lanthanide complexes. The observed magnetic behavior of the complex, caused through changes in the charge near the Nd(III) centers or the ligands, is our further investigation and confirmation.

Supplementary material

Crystallographic data (excluding structure factors) for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 659973. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK, Fax: +44 1223 336 033, Email: deposit@ccdc.cam.ac.uk, or www: http://www.ccdc.cam.ac.uk.

Acknowledgements

The authors would like to thank the Key Laboratory of Zhejiang University for X-ray data collection and the Education Committee of Zhejiang Province, China, for funds.

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