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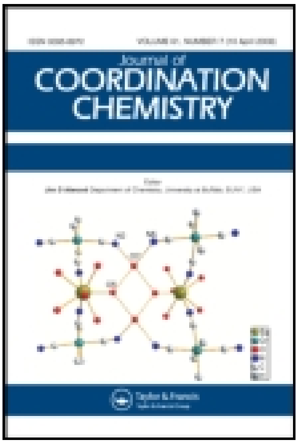

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Synthesis and crystal structure of a 2-D yttrium coordination network with propylenediamine-N,N,N′,N′-tetraacetic acid

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Synthesis and crystal structure of a 2-D yttrium coordination network with propylenediamine-*N,N,N′,N′*-tetraacetic acid

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A 2-D binuclear coordination polymer, $\{[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O\}_n$ ($H_4pdta =$ propylenediamine-*N,N,N′,N′*-tetraacetic acid), has been synthesized through direct hydrothermal reaction and characterized by infrared spectrum and thermal analysis. Single-crystal X-ray diffraction reveals that Y(III) is eight-coordinate of almost standard square antiprismatic polyhedron.

Keywords: Hydrothermal synthesis; Y(III); pdta ($H_4pdta =$ propylenediamine-*N,N,N′,N′*-tetraacetic acid); 2-D structure

1. Introduction

Lanthanide coordination chemistry has attracted considerable attention due to its intriguing frameworks and applications [1, 2] as shift reagents for nuclear magnetic resonance (NMR) spectroscopy [3], magnetic resonance imaging (MRI) contrast agent [4, 5], luminescent chemosensors, and probes for medical diagnostics [6–9]. The lanthanide-like ^{90}Y, with a short half life of 64.1 h, high-energy particle emitter ($E_{\max\beta-} = 2.27\,\text{MeV}$ and $\beta^- = 100\%$) and low toxicity, makes it an excellent candidate for therapeutic applications [10, 11]. Since ^{90}Y is a pure β-emitter with no γ-radiation [12], ^{90}Y-labeled complexes are available in clinical practice, making patient treatment feasible [13]. The high-energy β-emitter of Y(III) is superior in treatment of larger tumors [14, 15]. ^{90}Y-DOTA-HBP shows favorable biodistribution as a bone-seeking agent for the palliation of metastatic bone pain [16] and ^{90}Y-DOTA-Tyr^3-octreotide has been particularly useful in the therapy of somatostatin receptor-positive tumors [17, 18]. The characteristics and useful applications of the complexes largely originate from the geometric configuration and coordinate structures. Therefore, research on crystal and molecular structures of Y(III) complexes is needed. Lanthanide ions with high coordination flexibility and variable coordination geometries compared with transition metals have better affinity to hard donors, and ligands containing oxygen or nitrogen have been widely exploited to construct stable and high-dimensional

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lanthanide complexes. Pdta (=propylenediamine-*N,N,N′,N′*-tetraacetic acid), a hexadentate ligand, containing two nitrogens and four COOH groups is good for the synthesis of lanthanide complexes because of its versatile coordination. In this manuscript, we report a complex of Y(III) with pdta and its distinct crystal and molecular structure.

A series of lanthanide complexes with pdta have been reported by our laboratory, such as $K_2[Sm^{III}(pdta)(H_2O)_2]_2 \cdot 4.5H_2O$ [19] and $(NH_4)_2[Eu_2^{III}(pdta)_2(H_2O)_2] \cdot 2H_2O$ [20]. In this study, we select hexadentate pdta and obtain a coordination polymer through the reaction of Y_2O_3 powder with pdta at a molar ratio of 1 : 2. A striking feature is that the polymer exhibits a 2-D layer structure along the *ab* plane.

2. Experimental

2.1. *Synthesis*

H_4pdta (=propylenediamine-*N,N,N′,N′*-tetraacetic acid) (A.R., Beijing SHLHT Science & Trade Co., Ltd., China) (1.5313 g, 5.0 mmol) was added to 100 mL warm water, and during stirring Y_2O_3 powder (99.999%, Yuelong Rare Earth Co., Ltd., China) (0.5645 g, 2.5 mmol) was added slowly to the warm solution. The solution became transparent after the mixture had been stirred and refluxed for 18.0 h, and then was concentrated to 25 mL and left for static cultivation. Colorless crystals appeared after 2 weeks at room temperature.

2.2. *Infrared spectrum*

The H_4pdta and $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ samples were skived and pressed to slices with KBr and their infrared (IR) spectra were recorded by a Shimadza-IR 408 spectrograph. The spectra are shown in "Supplementary material."

2.3. *TG-DTA*

TG-DTA curve of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ was recorded by a Mettler-Toledo 851° thermogravimetric analyzer in the presence of air ($20\,mL\,min^{-1}$) from room temperature to 800°C at a heating rate of $20^\circ C\,min^{-1}$ (Supplementary material).

2.4. *^{1}H-NMR*

^{1}H-NMR spectra of H_4pdta and $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ samples using D_2O solvent were recorded on a Mercury Plus Varian-300 MHz NMR spectrometer operating at 300.07 MHz. The spectra are shown in "Supplementary material."

2.5. *X-ray structure determination*

X-ray intensity data were collected on a Bruker SMART CCD X-ray diffractometer system with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). The structure

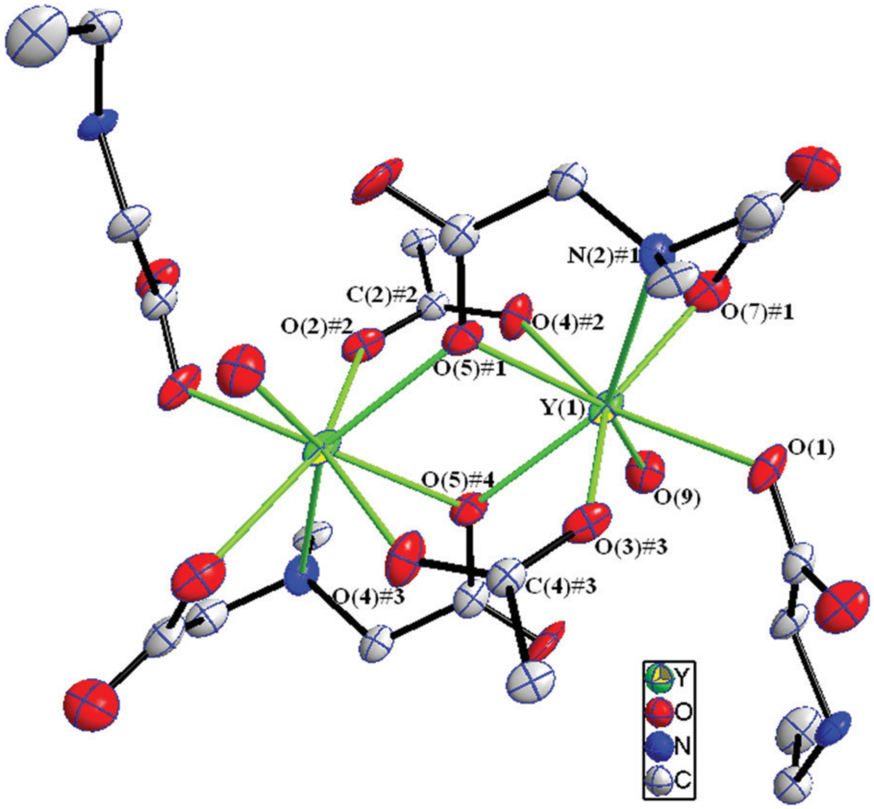

Figure 1. The structure of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$.

was solved by direct methods. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares methods. All the calculations were performed with the SHELXTL-97 program on PDP11/44 and Pentium MMX/166 computers. Figure 1 illustrates the perspective view of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$, figure 2 shows the coordination polyhedron of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$, figure 3 gives the coordination mode of one pdta ligand, and figure 4 displays the 2-D layered network of $\{[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O\}_n$. The crystal and structure refinement data for $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ are listed in table 1. Selected bond distances (Å) and angles (°) of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ are listed in table 2. Final atomic coordinates and equivalent isotropic displacement parameters for all the non-hydrogen fractional coordinates are presented in "Supplementary material" for $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$.

3. Results and discussion

3.1. *FT-IR spectra*

A comparison of IR spectra between H_4pdta and $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ shows that ν(C–N) of the complex at 866 cm^{-1} has a blue-shift (19 cm^{-1}) compared with that (847 cm^{-1}) of H_4pdta, indicating that the amine nitrogens of H_4pdta are coordinated

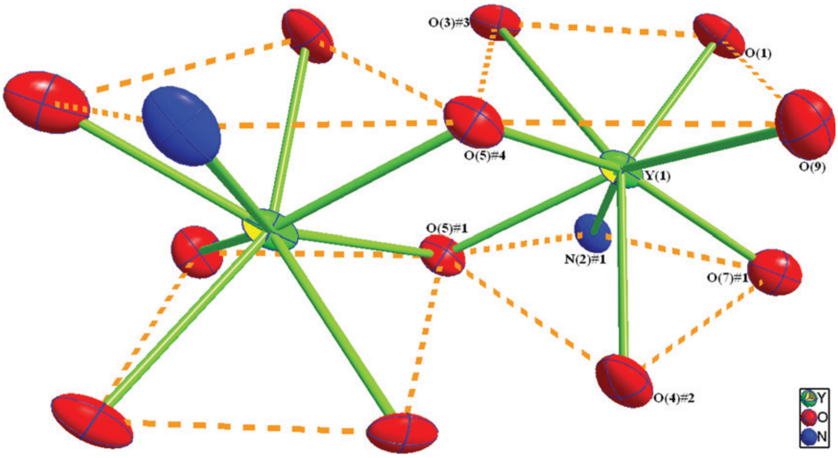

Figure 2. Coordination polyhedron around Y(1) in $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$.

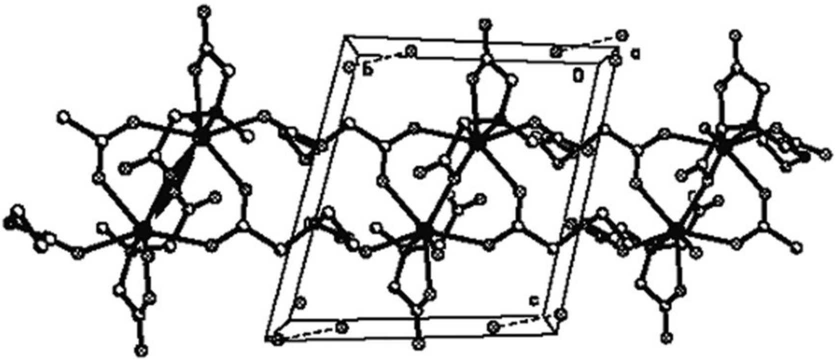

Figure 3. Arrangement of $Na_4[Eu^{III}(dtpa)(H_2O)]_2 \cdot 11.5H_2O$ in the unit cell.

to Y(III). The $\nu_{as}(COOH)$ band of H_4pdta at 1733 cm^{-1} disappears in the FT-IR spectrum of the complex, the $\nu_{as}(COO)$ band of complex at 1624 cm^{-1} shows a blue-shift (67 cm^{-1}) compared with 1557 cm^{-1} of H_4pdta ligand, and the $\nu_s(COO)$ of complex at 1403 cm^{-1} reveals a red-shift (41 cm^{-1}) compared with 1444 cm^{-1} of H_4pdta ligand. These changes confirm that oxygens from carboxyl groups of H_4pdta ligand are also coordinated to Y(III). A broad $\nu(OH)$ band near 3448 cm^{-1} indicates H_2O in the complex.

3.2. *Thermal analysis*

The TG-DTA curve of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ has a three-stage decomposition and the DTA curve shows two endothermic peaks and a series of exothermal peaks corresponding to three stages of mass loss. The weight of the complex decreases gradually as the temperature rises. The first stage weight loss is 13.36% from room

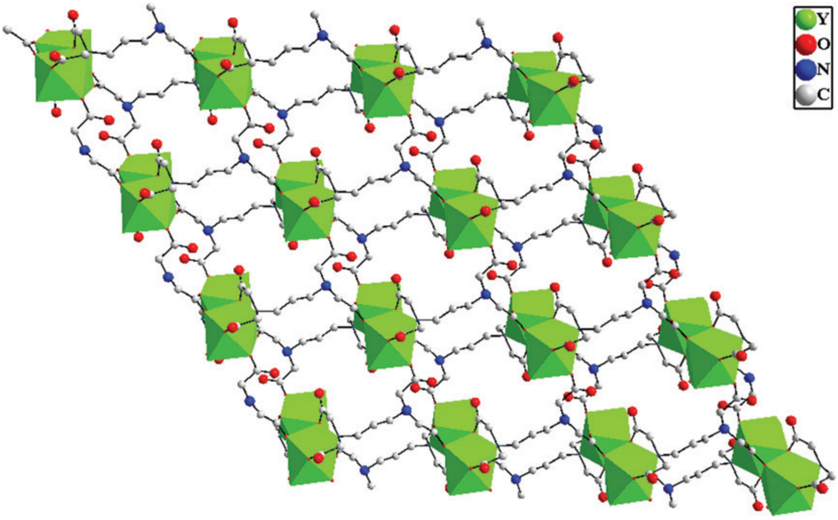

Figure 4. Polyhedral view of the 2-D layered network of $\{[\mathrm{Y^{III}(Hpdta)(H_2O)}]_2\cdot 6\mathrm{H_2O}\}_n$.

Table 1. Crystal data and structure refinement for $[\mathrm{Y^{III}(Hpdta)(H_2O)}]_2\cdot 6\mathrm{H_2O}$.

Formula weight	464.22
Temperature (K)	298(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	
a	10.0901(12)
b	10.5899(13)
c	10.9402(15)
α	71.280(2)
β	72.040(2)
γ	65.82
Volume (Å^3), Z	988.6(2), 2
Calculated density ρ_{calcd} (Mg m^{-3})	1.559
Absorption coefficient (mm^{-1})	3.008
$F(000)$	476
Crystal size (mm^3)	$0.26\times0.14\times0.06$
θ range for data collection (°)	2.26–25.00
Limiting indices	$-12\le h\le 11$; $-10\le k\le 12$; $-9\le l\le 13$
Reflections collected	4676
Independent reflections	3302 [$R(\mathrm{int})=0.1047$]
Completeness to $\theta_{\max}$ (%)	95.0
Max. and min. transmission	0.8401 and 0.5085
Goodness-of-fit on F^2	1.086
Final R indices [$I>2\sigma(I)$]	$R_1=0.1447$, $wR_2=0.3344$
R indices (all data)	$R_1=0.2058$, $wR_2=0.3659$
Largest difference peak and hole (e Å^{-3})	3.612 and −2.311
Absorption correction	Semi-empirical
Refinement method	Full-matrix least-squares on F^2

temperature to 268°C, which corresponds to expulsion of six crystal water molecules. Weight loss of 8.97% from 268°C to 365°C corresponds to two coordinated water molecules, with the endothermic peak at 310°C in the DTA curve. The third weight loss of 38.34% from 365°C to 800°C corresponds to decomposition and combustion of

Table 2. Selected bond distances (Å) and angles (°)) of $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$.

Bond	d (Å)	Bond	d (Å)	Bond	d (Å)
Y(1)–O(1)	2.327(12)	Y(1)–O(5)#1	2.400(11)	Y(1)–O(9)	2.345(13)
Y(1)–O(3)#3	2.419(12)	Y(1)–O(5)#4	2.435(12)	Y(1)–N(2)#1	2.605(14)
Y(1)–O(4)#2	2.405(12)	Y(1)–O(7)#1	2.348(14)	–	–
Angle	ω (°)	Angle	ω (°)	Angle	ω (°)
O(1)–Y(1)–O(3)#3	71.2(4)	O(3)#3–Y(1)–O(7)#1	141.4(4)	O(5)#1–Y(1)–O(9)	143.4(4)
O(1)–Y(1)–O(4)#2	147.8(5)	O(3)#3–Y(1)–O(9)	119.0(4)	O(5)#1–Y(1)–N(2)#1	65.0(4)
O(1)–Y(1)–O(5)#1	133.3(4)	O(3)#3–Y(1)–N(2)#1	86.1(4)	O(5)#4–Y(1)–O(7)#1	144.0(5)
O(1)–Y(1)–O(5)#4	123.8(4)	O(4)#2–Y(1)–O(5)#1	73.0(4)	O(5)#4–Y(1)–O(9)	77.8(4)
O(1)–Y(1)–O(7)#1	78.2(5)	O(4)#2–Y(1)–O(5)#4	76.7(4)	O(5)#4–Y(1)–N(2)#1	137.4(4)
O(1)–Y(1)–O(9)	81.9(4)	O(4)#2–Y(1)–O(7)#1	72.8(5)	O(7)#1–Y(1)–O(9)	78.0(4)
O(1)–Y(1)–N(2)#1	82.4(4)	O(4)#2–Y(1)–O(9)	78.8(4)	O(7)#1–Y(1)–N(2)#1	66.7(5)
O(3)#3–Y(1)–O(4)#2	140.9(4)	O(4)#2–Y(1)–N(2)#1	98.4(5)	O(9)–Y(1)–N(2)#1	143.6(5)
O(3)#3–Y(1)–O(5)#1	74.1(4)	O(5)#1–Y(1)–O(5)#4	73.4(4)	–	–
O(3)#3–Y(1)–O(5)#4	74.2(4)	O(5)#1–Y(1)–O(7)#1	114.1(4)	–	–

Symmetrical code: #1: $x+1, y, z$; #2: $x, y+1, z$; #3: $-x+1, -y, -z+1$; #4: $-x, -y+1, -z+1$.

carboxylate, with the endothermic peak at 386°C and a series of exothermal peaks at 495°C, 535°C, 560°C, 615°C, 695°C, and 730°C in the DTA curve. The total weight loss is 60.52%.

3.3. *^{1}H-NMR*

^{1}H-NMR spectra of H_4pdta and $[Y^{III}(Hpdta)(H_2O)]_2 \cdot 6H_2O$ dissolved in D_2O at 298 K were recorded. A narrow singlet signal of carboxyl CH_2 of free pdta appears at 4.058 ppm. Due to coordination of pdta to Y(III), the singlet of carboxyl CH_2 shifts upfield (3.842 ppm). A triplet of free pdta observed at 3.407, 3.433, and 3.459 ppm split into a quartet at 3.284, 3.337, 3.379, and 3.433 ppm after coordination, caused by asymmetric protonated nitrogen. The broad singlet of middle propylene CH_2, at 2.228 to 2.259 ppm, significantly shifts downfield between 2.796 and 2.807 ppm. All these characteristics are in accord with the single-crystal X-ray diffraction.

3.4. *Molecular and crystal structures*

Single-crystal X-ray analysis suggests that the complex crystallizes in the triclinic $P\bar{1}$ space group (table 1). The structure consists of binuclear $[YNO_6Ow]_2$ moieties (Ow stands for water) formed from two metals, six carboxyl oxygens, and a non-protonated amino nitrogen of pdta and a coordinated water molecule. The central yttriums are eight-coordinate in a square antiprism. The metal coordination can be described as built-up of two square antiprisms that share one edge, two symmetry related O(5) and O(5)#4 form the edge (as shown in figure 1).

As shown in figure 2, the upper square plane is formed by three carboxyl oxygens (O(1), O(3)#3, and O(5)#4) and one water (O(9)), while the bottom plane is formed by one amine nitrogen (N(2)#1) and three carboxyl oxygens (O(4)#2, O(5)#1, and O(7)#1). The Y(1)–O bond lengths range from 2.435(12) Å (Y(1)–O(5)#4) to 2.327(12) Å

(Y(1)–O(1)), significantly shorter than the Y(1)–N bond length (2.605(14) Å). O–Y(1)–O bond angles vary from 147.8(5)° (∠O(1)–Y(1)–O(4)#2) to 71.2(4)° (∠O(1)–Y(1)–O(3)#3) (shown in table 2). For the top plane, the average value of dihedral angle is 5.72° between Δ(O(1)O(5)#4O(3)#3) and Δ(O(1)O(5)#4O(9)), and 5.58° between Δ(O(3)#3O(9)O(1)) and Δ(O(3)#3O(9)O(5)#5). The average value of dihedral angle for the bottom plane is 21.77° between Δ(N(2)#1O(5)#1O(7)#1) and Δ(O(4)#2O(5)#1O(7)#1) and 20.69° between Δ(N(2)#2O(4)#2O(7)#1) and Δ(N(2)#2O(4)#2O(5)#1).

Every two adjacent Y(III) ions are interconnected by two bridging carboxyl oxygens (O(5) and O(5)#4 from two pdta ligands) and two carboxyl groups (O(3)#2–C(4)#2–O(4)#2 and O(3)#3–C(4)#3–O(4)#3) from two pdta ligands, forming a basic binuclear unit, i.e. $[Y^{III}(Hpdta)(H_2O)]_2$. The Y(1)·····Y(1)#5 separation is 3.878(3) Å (as shown in figure 1). There are four molecules in a unit cell (shown in figure 3). The two neighboring $[Y^{III}(Hpdta)(H_2O)]_2$ are connected together along the *a*-axis by pdta ligands *via* the amine nitrogen (N(1)) and its carboxylate, resulting in the formation of an infinite 1-D chain. Two adjacent chains are further linked through sharing of propanediamine (N(1)–C(5)–C(6)–C(7)–N(2)) along the *a*-axis, forming a 2-D layered network (figure 4). The 2-D layered networks are further consolidated *via* strong hydrogen bonds between coordinated water (O(9)) and non-coordinated carboxyl oxygen (O(8)) to extend into a 3-D supramolecular structure, in which O(9)–H ··· O(8), O(10)–H ··· O(1), and O(10)–H ··· O(7) distances are 2.698, 2.944, 2.949 Å, respectively. This structure is in striking contrast with the structures of rare earth metal complexes reported previously, such as $(NH_4)_3[Y^{III}(nta)_2]$, $Na[Y^{III}(edta)(H_2O)_3]\cdot 5H_2O$, $(NH_4)[Eu^{III}(pdta)(H_2O)]\cdot H_2O$, and $(enH_2)[Tb^{III}(pdta)(H_2O)]_2\cdot 8H_2O$ [21–24].

4. Conclusions

A coordination polymer, $\{[Y^{III}(Hpdta)(H_2O)]_2\cdot 6H_2O\}_n$, with 2-D layered network, has been synthesized. The central Y(III) ions are eight-coordinate in an antiprismatic polyhedron with six carboxyl oxygens and a non-protonated amino nitrogen from pdta and a coordinated water. The IR spectrum and thermal analyses conform to the results determined by single-crystal X-ray diffraction analysis. On the basis of this work, further research of lanthanide complexes with various aminopolycarboxylic acid ligands are in progress in our laboratory.

Supplementary material

CCDC 774818 $[Y^{III}(Hpdta)(H_2O)]_2\cdot 6H_2O$ contain the supplementary crystallographic data for this article. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif by e-mailing data_request@ccdc.cam.ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223-336033.

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