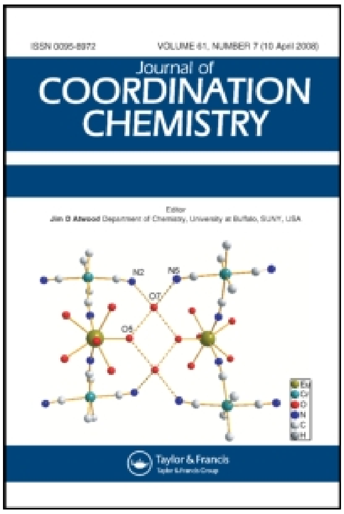

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Synthesis and crystal structure of two praseodymium 2-iodobenzoic acid complexes

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Two new complexes, $\{[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-}bipy]_2 \cdot [Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-}bipy]_2 \cdot 0.5C_2H_5OH \cdot H_2O\}$ (**1**) and $[Pr(2\text{-}IBA)_3 \cdot phen]_2$ (**2**) (2-IBA = 2-iodobenzoate; 2,2′-bipy = 2,2′-bipyridine; phen = 1,10-phenanthroline) were synthesized, and their crystal structures were determined by X-ray diffraction. Complex **1** consists of two binuclear molecules $[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-}bipy]_2$ (a) and $[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-}bipy]_2$ (b), half uncoordinated ethanol and one uncoordinated water. In the two molecules (a) and (b), the coordination environment of central ions is similar. The $Pr1^{3+}$ ion in molecule (a) and $Pr2^{3+}$ ion in molecule (b) are nine-coordinate with seven oxygen atoms from five 2-IBA ligands and two nitrogen atoms from one 2,2′-bipy molecule. The crystal structure of complex **2** is similar to that of binuclear $[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-}bipy]_2$ in complex **1**.

Keywords: Praseodymium complexes; 2-Iodobenzoic acid; Crystal structure

1. Introduction

Lanthanide carboxylate complexes have been extensively studied due to special structures and applications in optics, electronics, magnetics etc. We are interested in the crystal structures of such complexes. Carboxylate groups coordinate to lanthanide ions in different ways, chelating bidentate, bridging bidentate and bridging tridentate, leading to various structures of lanthanide carboxylate complexes. $Dy(4\text{-}CH_3C_6H_4COO)_3phen$ [1], $[Tb(o\text{-}CH_3C_6H_4COO)_3(phen)]$ [2], and $[Eu_2(SA)_6(phen)_2] \cdot 6H_2O$ (SA = succinamate, phen = 1,10-phenanthroline) [3] contain two independent binuclear molecules in the asymmetric unit. The terbium 2-fluorobenzoate (2-FBA) complex with phen contains two dimers with different compositions [4]. $Tb(2\text{-}FBA)_3 \cdot 2,2'$-bipy contains three independent molecules in the asymmetric unit [4]. Complexes containing several different molecules are relatively

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less reported in literature. Two new praseodymium 2-iodobenzoic acid complexes, $\{[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-bipy}]_2 \cdot [Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-bipy}]_2 \cdot 0.5C_2H_5OH \cdot H_2O\}$ (**1**) and $[Pr(2\text{-}IBA)_3 \cdot \text{phen}]_2$ (**2**) (2-IBA = 2-iodobenzoate; 2,2′-bipy = 2,2′-bipyridine; phen = 1,10-phenanthroline) were synthesized. Complex **1** contains two different binuclear molecules, $[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-bipy}]_2$ (a) and $[Pr(2\text{-}IBA)_3 \cdot 2,2'\text{-bipy}]_2$ (b). We report the crystal structures of the complexes in this article.

2. Experimental

2.1. *Materials and methods*

$PrCl_3 \cdot 6H_2O$ was prepared by dissolving its oxides in hydrochloric acid, and then drying the solution. Elemental analysis was performed on an Elementar Vario EL analyzer. The IR spectra were recorded with a Bruker EQUINOX-55 using the KBr pellet technique. The UV-VIS spectra were measured on a TU-1810 spectrophotometer.

2.2. *Synthesis of complexes*

1.05 mmol of 2-iodobenzoic acid was dissolved in 25 mL 95% ethanol solution. The pH was adjusted to 6 ~ 7 with 2 M NaOH solution. Then 0.35 mmol 2,2′-bipyridine or 1,10-phenanthroline and 0.35 mmol $PrCl_3 \cdot 6H_2O$ were added to the solution successively. The mixture was heated under reflux with stirring for 0.5–1 h and then filtered. Light green crystals were obtained from the mother liquor after 4 days. For complex **1**, the yield, 0.19 g, 52%. Calcd (%) for $C_{63}H_{45}I_6N_4O_{13.50}Pr_2$: C, 35.74; H, 2.14; N, 2.64; found (%): C, 35.54; H, 2.43; N, 2.79. Main IR (KBr pellet, cm^{-1}): 3429w, 1607vs, 1531s, 1434s, 1399vs, 1012s, 850m, 756s, 745s, 690m, 639m, 423w. For complex **2**, yield, 0.11 g, 30%. Calcd (%) for $C_{66}H_{40}I_6N_4O_{12}Pr_2$: C, 37.32; H, 1.90; N, 2.64. Found (%): C, 37.01; H: 2.12; N, 2.87. Selected IR (KBr pellet, cm^{-1}): 1609 vs, 1537s, 1468 m, 1398 vs, 1013 s, 849 m, 754 s, 729 m, 690 m, 639 m, 419 m.

2.3. *Single-crystal X-ray diffraction*

X-ray crystal data collection for the complexes was on a Bruker Smart 1000 CCD diffratometer with monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å) at 293 K. Semi-empirical absorption corrections were applied using the SADABS program. All calculations were carried out on a computer with use of SHELXS-97 and SHELXL-97 programs [5, 6]. The structures were solved by direct methods and refinement on $|F|^2$ used the full-matrix least-squares methods. A summary of the crystallographic data and details of the structure refinements are listed in table 1; selected bond distances and angles in tables 2 and 3.

Table 1. Crystal data and structure refinement for complexes.

Complex	**1**	**2**
Chemical formula	$C_{63}H_{45}I_6N_4O_{13.50}Pr_2$	$C_{66}H_{40}I_6N_4O_{12}Pr_2$
Formula weight	2117.25	2124.24
Temperature (K)	294(2)	294(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	11.8062(13)	11.6991(14)
b (Å)	14.0132(14)	13.2387(15)
c (Å)	21.261(2)	13.3054(16)
α (°)	86.005(2)	115.188(2)
β (°)	87.573(2)	114.647(2)
γ (°)	76.332(2)	91.113(2)
Volume (Å^3)	3408.3(6)	1645.6(3)
Z	1	1
D_{Calcd} (Mg m^{-3})	2.063	2.143
μ (mm^{-1})	4.193	4.341
F(000)	1990	996
Crystal size (mm^3)	0.18 × 0.10 × 0.08	0.26 × 0.22 × 0.14
Theta range for data collection (°)	0.96–25.01	1.88–25.01
Limiting indices	$-14 \leq h \leq 9$, $-16 \leq k \leq 16$–$25 \leq l \leq 24$	$-13 \leq h \leq 13$, $-15 \leq l \leq 11$–$14 \leq l \leq 15$
Reflections collected/unique	817401/11926 [R(int) = 0.0263]	8246/5676[R(int) = 0.0208]
Data/restraints/parameters	11926/50/820	5676/0/406
Goodness-of-fit on F^2	1.017	1.023
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0523$, $wR_2 = 0.1286$	$R_1 = 0.0351$, $wR_2 = 0.0838$
R indices (all data)	$R_1 = 0.0799$, $wR_2 = 0.1491$	$R_1 = 0.0442$, $wR_2 = 0.0915$
Largest diff. peak and hole (e Å^{-3})	2.386 and −2.081	1.650 and −1.387

3. Results and discussion

3.1. *Structural description of complex*

{[Pr(2-IBA)$_3$ · 2,2′-bipy]$_2$ · [Pr(2-IBA)$_3$ · 2,2′-bipy]$_2$ · 0.5C$_2$H$_5$OH · H$_2$O} (1)

Complex **1** {[Pr(2-IBA)$_3$ · 2,2′-bipy]$_2$ · [Pr(2-IBA)$_3$ · 2,2′-bipy]$_2$ · 0.5C_2H_5OH · H_2O} consists of two binuclear molecules [Pr(2-IBA)$_3$ · 2,2′-bipy]$_2$ (a) and [Pr(2-IBA)$_3$ · 2,2′-bipy]$_2$ (b), half uncoordinated ethanol molecule and one uncoordinated water molecule, as shown in figure 1(a). In (a), two Pr1^{3+} ions are linked together by four bridging 2-IBA groups to form a centrosymmetric dimer. Each central Pr1^{3+} ion is also chelated by one 2-IBA group and one 2,2′-bipy molecule. The Pr1^{3+} ion is nine-coordinate with seven oxygen atoms from five 2-IBA ligands and two nitrogen atoms from one 2,2′-bipy. The coordination environment of Pr1^{3+} may be describes as a distorted monocapped square-antiprism (figure 1b). The four apical positions of the upper square are occupied by O1, O4A, O6A and N1, and those of the lower square by O3, O4, O5 and N2 with mean deviations of 0.2105 and 0.10433 Å, respectively, and the dihedral angle between the two squares is 5.3°. O2 caps the upper plane. Molecule (b) is almost the same as (a). In molecule (b), the Pr2^{3+} ion is coordinated by nine atoms in a distorted monocapped square-antiprism (figure 1c). Atoms O7, O8A, O9, O10A and O11, O12, N3, N4 formed upper and lower square planes with mean deviations of 0.1294 and 0.0202 Å, respectively, with a dihedral angle of 5.3° between the two planes. Atom O8 caps the upper plane. In molecule (a), Pr1–O distances range from 2.436(6) ~ 2.513(6) Å with

Table 2. Selected bond lengths (Å) and angles (°) for **1**.

Pr(1)–O(5)	2.436(6)	Pr(1)–O(4)#1	2.439(6)
Pr(1)–O(6)#1	2.448(6)	Pr(1)–O(3)	2.498(6)
Pr(1)–O(2)	2.508(6)	Pr(1)–O(1)	2.513(6)
Pr(1)–O(4)	2.882(6)	Pr(1)–N(1)	2.653(7)
Pr(1)–N(2)	2.697(7)	Pr(2)–O(10)#2	2.428(7)
Pr(2)–O(8)#2	2.440(7)	Pr(2)–O(9)	2.441(8)
Pr(2)–O(11)	2.475(9)	Pr(2)–O(7)	2.518(9)
Pr(2)–O(12)	2.520(8)	Pr(2)–O(8)	2.679(8)
Pr(2)–N(3)	2.667(9)	Pr(2)–N(4)	2.661(10)
O(5)–Pr(1)–O(4)#1	73.6(2)	O(5)–Pr(1)–O(6)#1	132.9(2)
O(4)#1–Pr(1)–O(6)#1	77.2(2)	O(5)–Pr(1)–O(3)	89.6(2)
O(4)#1–Pr(1)–O(3)	125.1(2)	O(6)#1–Pr(1)–O(3)	77.9(2)
O(5)–Pr(1)–O(2)	133.5(2)	O(4)#1–Pr(1)–O(2)	80.8(2)
O(6)#1–Pr(1)–O(2)	75.1(2)	O(3)–Pr(1)–O(2)	136.6(2)
O(5)–Pr(1)–O(1)	91.7(2)	O(4)#1–Pr(1)–O(1)	94.5(2)
O(6)#1–Pr(1)–O(1)	127.1(2)	O(3)–Pr(1)–O(1)	138.9(2)
O(2)–Pr(1)–O(1)	52.0(2)	N(1)–Pr(1)–N(2)	60.9(3)
O(5)–Pr(1)–O(4)	69.6(2)	O(4)#1–Pr(1)–O(4)	77.8(2)
O(6)#1–Pr(1)–O(4)	68.6(2)	O(3)–Pr(1)–O(4)	47.58(18)
O(2)–Pr(1)–O(4)	141.0(2)	O(1)–Pr(1)–O(4)	161.0(2)
N(1)–Pr(1)–O(4)	115.5(2)	N(2)–Pr(1)–O(4)	105.3(2)
O(10)#2–Pr(2)–O(8)#2	74.0(3)	O(10)#2–Pr(2)–O(9)	134.7(3)
O(8)#2–Pr(2)–O(9)	75.3(3)	O(10)#2–Pr(2)–O(11)	127.9(3)
O(8)#2–Pr(2)–O(11)	82.9(3)	O(9)–Pr(2)–O(11)	79.4(3)
O(10)#2–Pr(2)–O(7)	81.7(3)	O(8)#2–Pr(2)–O(7)	123.6(3)
O(9)–Pr(2)–O(7)	88.5(3)	O(11)–Pr(2)–O(7)	147.1(3)
O(10)#2–Pr(2)–O(12)	79.1(3)	O(8)#2–Pr(2)–O(12)	82.9(3)
O(9)–Pr(2)–O(12)	128.6(3)	O(11)–Pr(2)–O(12)	51.8(3)
O(7)–Pr(2)–O(12)	140.9(3)	N(4)–Pr(2)–N(3)	60.2(4)
O(10)#2–Pr(2)–O(8)	69.7(2)	O(8)#2–Pr(2)–O(8)	74.3(3)
O(9)–Pr(2)–O(8)	70.4(2)	O(11)–Pr(2)–O(8)	145.7(3)
O(7)–Pr(2)–O(8)	49.6(3)	O(12)–Pr(2)–O(8)	145.3(3)

Symmetry transformations used to generate equivalent atoms: #1 $-x$, $-y+1$, $-z$; #2 $-x+1$, $-y$, $-z+1$.

a mean bond length of 2.532 Å. The average Pr1–N distance is 2.675 Å. The distance between two $Pr1^{3+}$ ions is 4.151(12) Å. In molecule (b), Pr2–O distances range from 2.428(7) ~ 2.679(8) Å with a mean bond length of 2.501 Å. The average Pr2–N distance is 2.664 Å. The distance between two $Pr2^{3+}$ ions is 4.0845(12) Å.

The crystal structure of complex **1** is similar to that of $[Tb(o\text{-}CH_3C_6H_4COO)_3(phen)]$ [2] and $[Eu_2(SA)_6(phen)_2]6H_2O$ [3]. The latter two complexes also contain two binuclear molecules in the asymmetric unit, and in the two molecules of each complex, the coordination environment of the central ions is similar, but the corresponding bond lengths and bond angles are a little different. This is unlike $Dy(4\text{-}CH_3C_6H_4COO)_3phen$ [1], which contains two binuclear molecules in the asymmetric unit owing to different coordination modes of carboxylate groups. In one, two Dy^{3+} ions were linked by four $4\text{-}CH_3C_6H_4COO^-$ groups, in the other, two Dy^{3+} ions were linked by two $4\text{-}CH_3C_6H_4COO^-$ groups. The above mentioned complexes are different from the terbium complex with 2-fluorobenzoate (2-FBA) and phen, which contains two binuclear molecules with different compositions, namely, $[Tb(2\text{-}FBA)_3 \cdot phen \cdot CH_3CH_2OH]_2$ and $[Tb(2\text{-}FBA)_3 \cdot phen]_2$ [4]. Complex **1** is also different from complex $Tb(2\text{-}FBA)_3 \cdot 2,2'$-bipy [4], which contains three independent binuclear molecules in the asymmetric unit. In two of the molecules, two Tb^{3+} ions are held together by four 2-FBA groups in bidentate bridging and chelating-bridging two modes. In another one, two Tb^{3+} ions are held

Table 3. Selected bond lengths (Å) and angles (°) for **2**.

Pr(1)–O(4)#1	2.403(4)	Pr(1)–O(3)	2.412(4)
Pr(1)–O(5)#1	2.433(3)	Pr(1)–O(1)	2.502(4)
Pr(1)–O(2)	2.516(4)	Pr(1)–O(6)	2.557(4)
Pr(1)–O(5)	2.704(4)	Pr(1)–N(1)	2.699(4)
Pr(1)–N(2)	2.675(5)		
O(4)#1–Pr(1)–O(3)	133.59(13)	O(4)#1–Pr(1)–O(5)#1	74.69(13)
O(3)–Pr(1)–O(5)#1	74.31(13)	O(4)#1–Pr(1)–O(1)	89.42(13)
O(3)–Pr(1)–O(1)	131.83(13)	O(5)#1–Pr(1)–O(1)	150.38(13)
O(4)#1–Pr(1)–O(2)	138.23(13)	O(3)–Pr(1)–O(2)	80.50(13)
O(5)#1–Pr(1)–O(2)	146.70(13)	O(1)–Pr(1)–O(2)	51.60(13)
O(4)#1–Pr(1)–O(6)	86.72(15)	O(3)–Pr(1)–O(6)	82.39(15)
O(5)#1–Pr(1)–O(6)	123.32(13)	O(1)–Pr(1)–O(6)	79.39(14)
O(2)–Pr(1)–O(6)	73.23(14)	O(4)#1–Pr(1)–O(5)	68.27(12)
O(3)–Pr(1)–O(5)	70.71(12)	O(5)#1–Pr(1)–O(5)	74.53(13)
O(1)–Pr(1)–O(5)	122.99(13)	O(2)–Pr(1)–O(5)	117.13(12)
O(6)–Pr(1)–O(5)	48.96(12)	N(2)–Pr(1)–N(1)	60.86(14)

Symmetry transformations used to generate equivalent atoms: #1 $-x$, $-y$, $-z$.

together by four 2-FBA groups in the bidentate bridging mode. Many lanthanide mono-carboxylate complexes with phen or 2,2′-bipy contain only one binuclear molecule in the asymmetric unit, such as $Sm_2(C_8H_7O_2)_6(C_{10}H_8N_2)_2$ ($C_8H_7O_2$ = 4-methylbenzoate, $C_{10}H_8N_2$ = 2,2′-bipy) [7], [Eu(2,3-DMOBA)$_3$2,2′-bipy]$_2$ (2,3-DMOBA = 2,3-dimethoxybenzoate) [8], Eu_2(phen)$_2$ $(C_6H_5COO)_6$ [9] and Eu_2(3,4-DMBA)$_6$(phen)$_2$ (3,4-DMBA = 3,4-dimethylbenzoate) [10].

In complex **1**, half uncoordinated ethanol and one uncoordinated water exist in the asymmetric unit. The distances of the uncoordinated ethanol molecule between $Pr1^{3+}$ and $Pr2^{3+}$ are $d(O13 \cdots Pr1^{3+}) = 8.582(8)$ Å and $d(O13 \cdots Pr2^{3+}) = 6.820(8)$ Å, respectively. The distances of water to $Pr1^{3+}$ and $Pr2^{3+}$ are $d(O14 \cdots Pr1^{3+}) = 10.568(8)$ Å and $d(O14 \cdots Pr2^{3+}) = 4.889(8)$ Å, respectively. The uncoordinated ethanol molecule and uncoordinated water molecule form hydrogen bonds, O13 ··· O14, 2.743(9) Å, 110.8° and water also forms hydrogen bonds with carboxylate groups coordinated to $Pr2^{3+}$, O14 ··· O12, 2.750(9) Å, 20.9°. Therefore, molecules (b) form an infinite one-dimensional chain by these hydrogen bonds viewed down the *c*-axis as shown in figure 1(d).

3.2. *Structural description of (2) [Pr(2-IBA)$_3$ • phen]$_2$*

The crystal structure of **2** is shown in figure 2(a) as a dimer with an inversion center. The Pr1 ··· Pr1A distance is 4.0918(7) Å. Two $Pr1^{3+}$ ions are connected by four bridging 2-IBA groups, two bidentate-bridging and two tridentate-bridging; each metal is also chelated by one 2-IBA group and one phen. The structure of complex **2** is common for nine-coordinate lanthanide carboxylate complexes containing phen, such as Eu_2(phen)$_2$$(C_6H_5COO)_6$ [9] and Eu_2(3,4-DMBA)$_6$(phen)$_2$ (3,4-DMBA = 3,4-dimethylbenzoate) [10]. The Pr^{3+} ion is in a distorted monocapped square-antiprism comprised of seven O atoms from five 2-IBA ligands and two N atoms from phen (figure 2b). Atoms O3, O4A, O5A, O6, and O1, O2, N1, N2 form lower and upper square planes with mean deviations of 0.1291 and 0.0832 Å, respectively; the dihedral angle between the two squares is 2.2°. O5 caps the upper plane. The O–Pr bond distances range from 2.403(4) to 2.704(4) Å and those of the N–Pr from 2.699(4) to 2.675(5) Å (table 3).

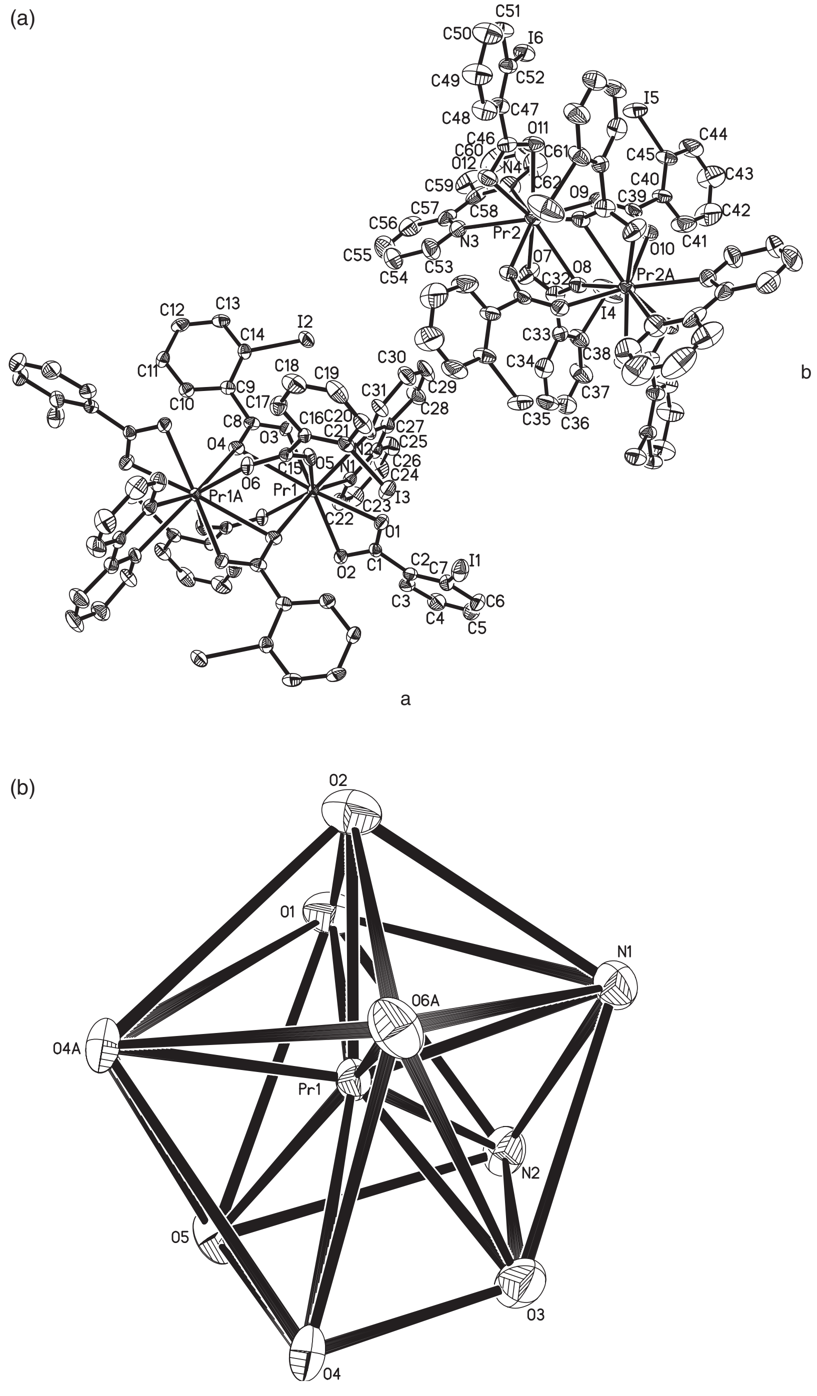

Figure 1. (a) Molecular structure of **1**. (b) The coordination polyhedron of $Pr1^{3+}$ in molecule (a). (c) The coordination polyhedron of $Pr2^{3+}$ in molecule (b). (d) One-dimensional polymer chain structure by hydrogen bonds viewed down the c-axis (All hydrogen atoms have been omitted for clarity; the thermal ellipsoids are shown at the 30% probability level).

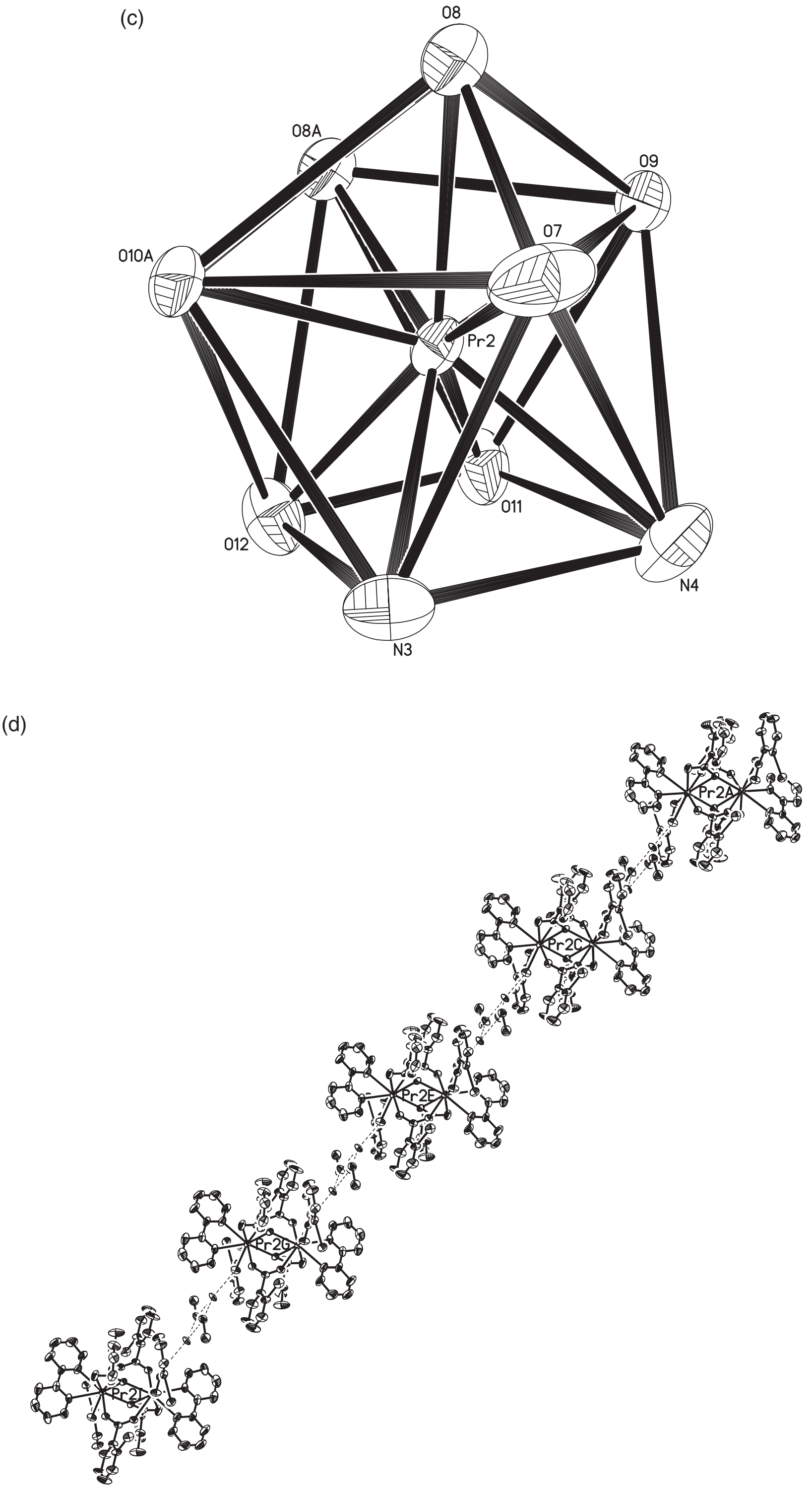

Figure 1. Continued.

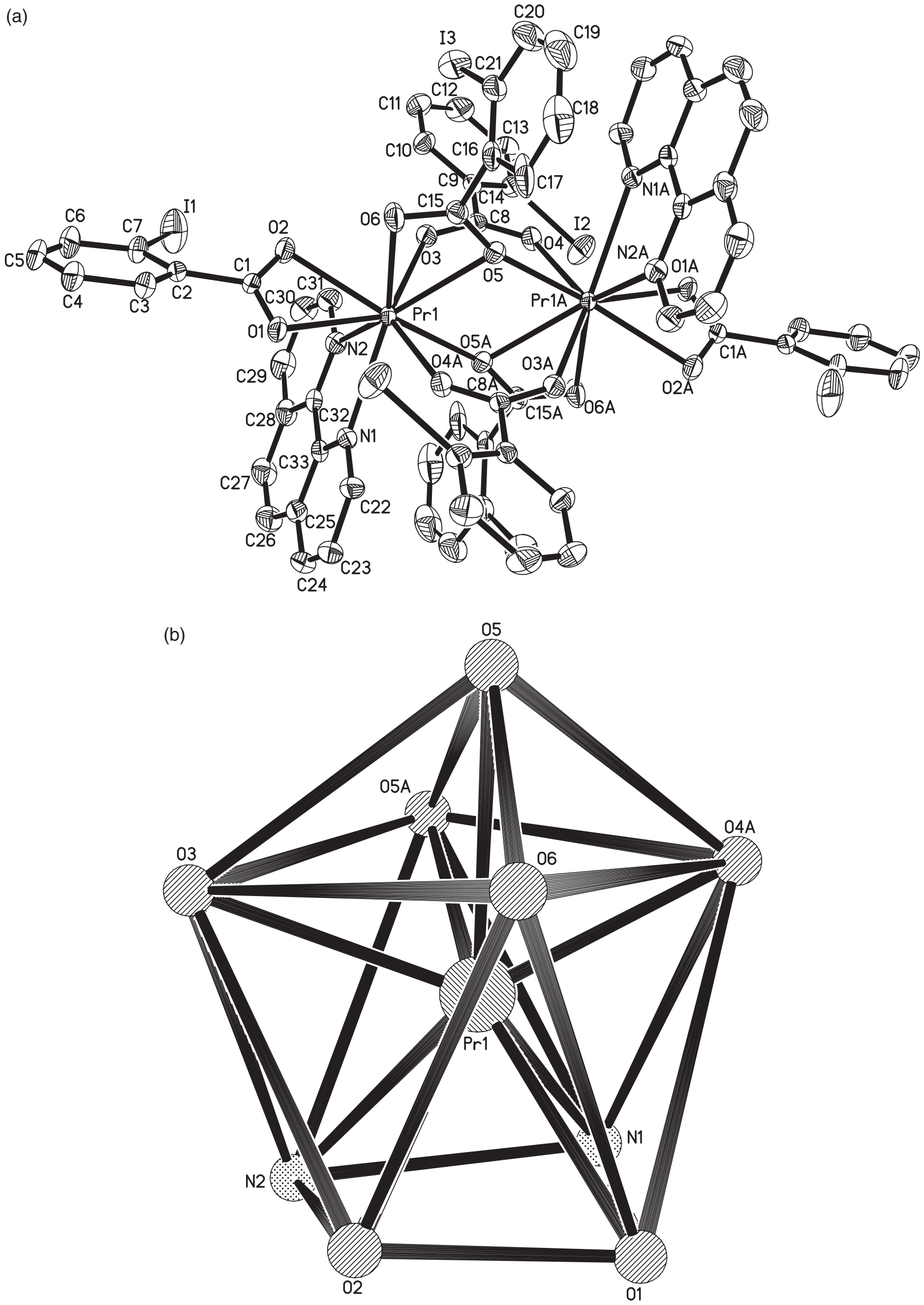

Figure 2. (a) Molecular structure of **2**. (b) The coordination polyhedron of Pr^{3+} in **2**. (All hydrogen atoms have been omitted for clarity; the thermal ellipsoids are shown at the 30% probability level.)

The crystal structure of **2** is very similar to that of the molecule (a) or (b) of complex **1** while **2** contains one independent binuclear molecule, **1** contains two independent binuclear molecules, perhaps because 2,2′-bipy is more flexible than phen.

3.3. *UV absorption spectra*

UV absorption spectra for $1.0 \times 10^{-4}\,\mathrm{mol\,L^{-1}}$ DMF solutions were determined. The broad absorption band at 283 nm for **1** and 278 nm for **2** correspond to the π–π^* transition of the ligands.

Supplementary data

The crystallographic data have been deposited at Cambridge Crystallographic Data Centre, CCDC-286080 for complex **1** and 294226 for complex **2** contains the supplementary crystallographic data for this article. These data can be obtained free of charge from CCDC, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223-336033; Email: deposit@ccdc.cam.ac.uk.

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