ЖУРНАЛ СТРУКТУРНОЙ ХИМИИ

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NEW NINE-COORDINATE $(NH_4)_3[Yb^{III}(TTHA)]\cdot 5H_2O$ AND EIGHT-COORDINATE $(NH_4)[Yb^{III}(PDTA)(H_2O)_2]\cdot 5H_2O$ COMPLEXES: STRUCTURAL DETERMINATION

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The $(NH_4)_3[Yb^{III}(ttha)]\cdot 5H_2O$ (**I**) (H_6ttha = triethylenetetramine-N,N,N′,N″,N‴,N‴-hexaacetic acid) and $(NH_4)[Yb^{III}(pdta)(H_2O)_2]\cdot 5H_2O$ (**II**) (H_4pdta = propylenediamine-N,N,N′,N′-tetraacetic acid) complexes are synthesized by heat-refluxing and acidity-adjusting methods, and their structures are determined by single crystal X-ray diffraction techniques. These two complexes are all mononuclear structures. The complex **I** crystallizes in the monoclinic crystal system with the $P2_1/c$ space group. The central Yb^{III} ion is nine-coordinated only by one ttha ligand, and one non-coordinate carboxyl group is left. The crystal data are as follows: $a = 10.321(4)$, $b = 12.744(5)$, $c = 23.203(9)$ Å, $\beta = 91.082(6)°$, $V = 3051(2)$ Å^3, $Z = 4$, $D_c = 1.754$ g/cm^3, $\mu = 3.150$ mm^{-1}, $F(000) = 1636$, $R = 0.0357$, and $wR = 0.0672$ for 6203 observed reflections with $I \geq 2\sigma(I)$. The YbN_4O_5 part in the $[Yb^{III}(ttha)]^{3-}$ complex anion forms a pseudo-monocapped square antiprismatic polyhedron. The complex **II** is coordinated with one pdta ligand and two water molecules, which form an eight-coordinate structure, and crystallizes in the triclinic crystal system with the $P\overline{1}$ space group. The YbN_2O_6 part in the $[Yb^{III}(pdta)(H_2O)_2]^-$ complex anion makes a pseudo-square antiprismatic polyhedron. The crystal data are as follows: $a = 9.8923(9)$, $b = 10.9627(10)$, $c = 12.2618(11)$ Å, $\alpha = 67.284(5)°$, $\beta = 70.956(6)°$, $\gamma = 68.741(5)°$, $V = 1115.97(18)$ Å^3, $Z = 2$, $D_c = 1.843$ g/cm^3, $\mu = 4.264$ mm^{-1}, $F(000) = 618$, $R = 0.0177$, and $wR = 0.0409$ for 4036 observed reflections with $I \geq 2\sigma(I)$.

Keywords: ytterbium(III), ttha, pdta, complex, synthesis, structure.

INTRODUCTION

Owing to their particular physical and chemical properties, lanthanide metals are usually selected and synthesized for metal-organic frameworks used in gas storage, adsorption, catalysis, and separation [1—8]. As an important member of the rare-earth family, ytterbium and its complexes are widely used for various applications [9, 10].

Lanthanide ions with large radii always tend to coordinate with O atoms. This is due to that there is a high affinity between lanthanide ions and hard donor atoms. So, ligands with either O or O—N atoms are suitable for lanthanide ions to form various complexes [11—16]. Animopolycarboxyl acids have usually been selected as this type of ligands. It is also easy to infer that the more O atoms they possess, the higher affinity to lanthanide ions aminopolycarboxyl acids have.

As known, because of their large radii rare earth metal complexes with aminopolycarboxyl acids generally adopt eight-, nine-, and ten-coordinate complex structures. the first several lanthanide elements have a chance of ten-coordinate complexes with aminopolycarboxyl acids due to their larger radii, and the others have little possibility of it. The Yb^{III} ion possesses a critical ionic radius of 0.1008 nm and the f^{13} electronic configuration, which provides eight- or nine-coordinate complex

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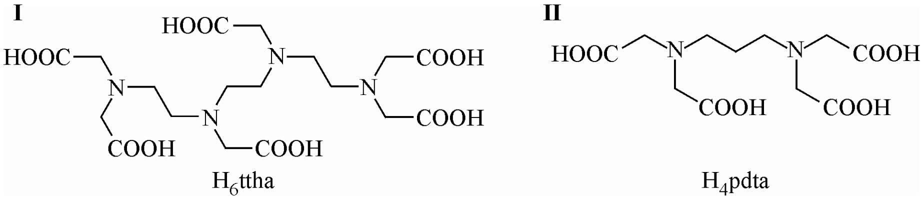
[17—20]. So, the ligand structure is an important factor that affects the coordination numbers of these complexes. Multidentate ligands with a strong chelating ability can form high-coordinate complexes with rare earth metals. H_6ttha (=triethylenetetramine-N,N,N′,N″,N‴,N‴-hexaacetic acid) is a decadentate ligand. Its intense chelating effect makes it coordinate with the central metal ion tightly and form a relatively high-coordinate complex. Therefore, the nine-coordinate structure may be the choice for the complex of the Yb^{III} ion with the ttha ligand. On the contrary, the hexadentate H_4pdta (=propylenediamine-N,N,N′,N′-tetraacetic acid) ligand, with a weaker chelating ability than the ttha ligand, seems to adopt an eight-coordinate structure with the Yb^{III} ion. Even more important, there would be a six-membered ring in the complex of the Yb^{III} ion with the pdta ligand, which takes much space so it is difficult to form a nine-coordinate complex.



Scheme 1. The structure of ligands **I** and **II**

In view of our prediction, the ttha and pdta ligands (their structures are shown in Scheme 1) have been selected to synthesize complexes with the Yb^{III} ion. Their molecule and crystal structures were determined. The results show that the complex **I** is nine-coordinate, while the complex **II** is eight-coordinate. So, it can validate that the structures of the complexes of rare earth metal ions with polyaminopolycarboxyl acids are determined by the radius of the central metal ion as well as the ligand structure.

EXPERIMENTAL

Synthesis of I. H_6ttha (=triethylenetetramine-N,N,N′,N″,N‴,N‴-hexaacetic acid) (A.R., Beijing SHLHT Science & Trade Co., Ltd., China) (2.4723 g, 5.0 mmol) was added to 100 ml of warm water and Yb_2O_3 (99.999 %, Beijing HENGYE ZHONGYUAN Chemical Co., Ltd., China) (0.9852 g, 2.5 mmol) powder was added slowly to the above solution. The solution became transparent after the mixture had been stirred and refluxed for 15.0 h, and then the pH value was adjusted to 6.0 with a dilute NH_3 aqueous solution. Finally, the solution was concentrated to 25 ml. A colorless crystal appeared after three weeks at room temperature.

Synthesis of II. 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 of warm water and Yb_2O_3 (99.999 %, Beijing HENGYE ZHONGYUAN Chemical Co., Ltd., China) (0.9852 g, 2.5 mmol) powder was added slowly to the above solution. The solution became transparent after the mixture had been stirred and refluxed for 18.0 h, and then the pH value was adjusted to 6.0 with a dilute NH_3 aqueous solution. Finally, the solution was concentrated to 25 ml. A colorless crystal appeared after two weeks at room temperature.

FT-IR determination. H_6ttha, H_4pdta, $(NH_4)_3[Yb^{III}(ttha)] \cdot 5H_2O$ and $(NH_4)[Yb^{III}(pdta)(H_2O)_2] \cdot \cdot 5H_2O$ were mulled and pressed into a pellet with KBr. Their FT-IR spectra were determined with a Shimadzu-IR 408 spectrograph.

X-ray structure determination. X-ray intensity data were collected on a Bruker SMART CCD-type X-ray diffractometer system with graphite-monochromatized MoK_α radiation ($\lambda = 0.71073$ Å). The structure was solved by direct methods. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares methods. All the calculations were performed by the SHELXTL-97 program on PDP11/44 and Pentium MMX/166 computers. Fig. 1 illustrates the perspective views of two complexes. Fig. 2 shows their coordination polyhedra. Fig. 3 show their molecular packings in a unit cell. The crystal data and structure refinement for two complexes are listed in Table 1. Selected bond distances of two complexes are listed in Table 2.

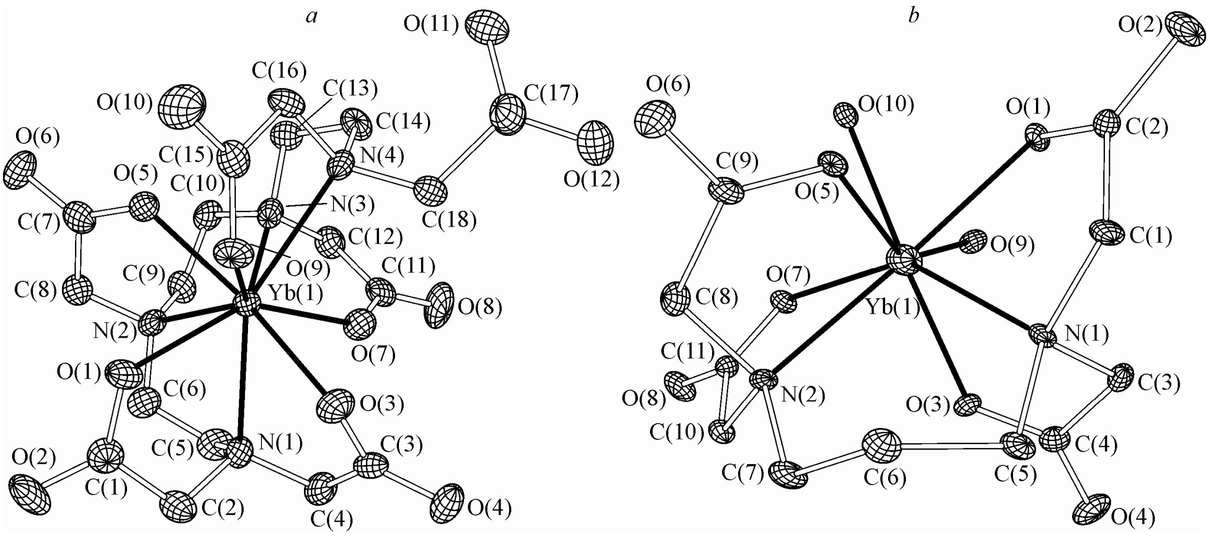


Fig. 1. Molecular structure of complex **I** (*a*) and **II** (*b*)

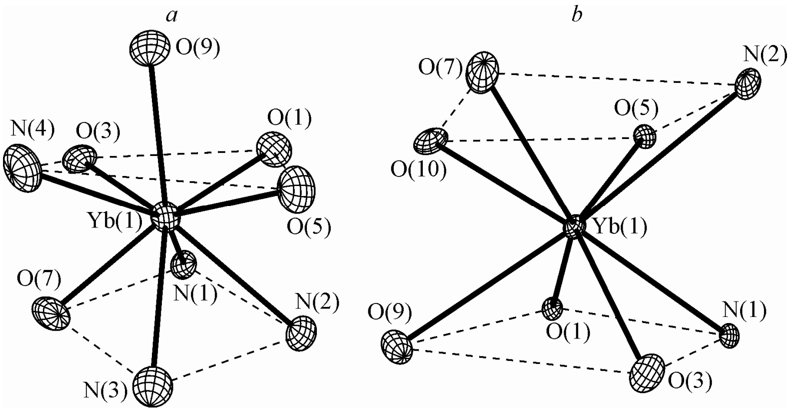


Fig. 2. Coordination polyhedron of complex **I** (*a*) and **II** (*b*)

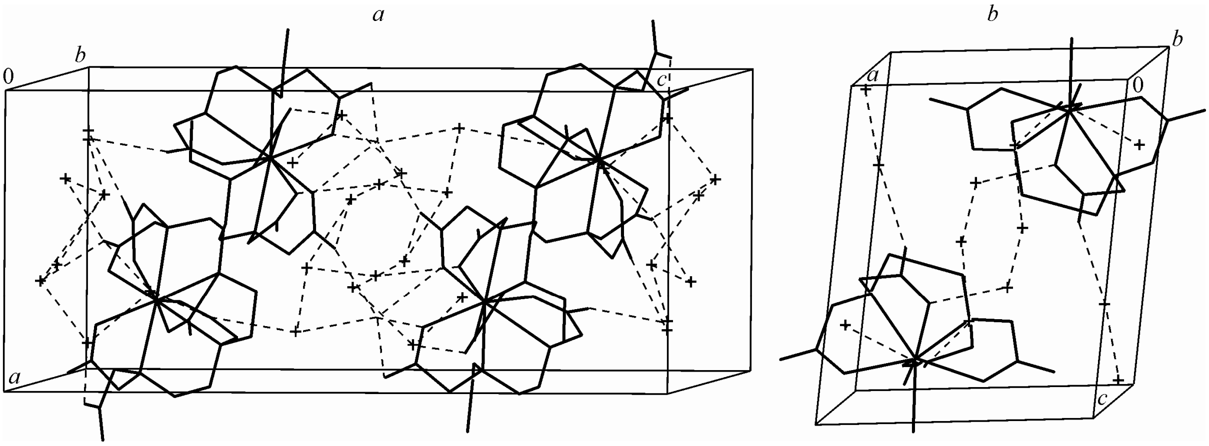


Fig. 3. Arrangement of complex **I** (*a*) and **II** (*b*) in unit cell (dashed lines represent intermolecular hydrogen bonds)

Table 1

Crystal data and structure refinement for $(NH_4)_3[Yb^{III}(ttha)]\cdot 5H_2O$ (**I**) *and* $(NH_4)[Yb^{III}(pdta)(H_2O)_2]\cdot 5H_2O$ (**II**)

Complex	**I**	**II**
Formula weight	805.63	619.42
Temperature, K	293(2)	93(2)
Wavelength, Å	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\bar{1}$
Unit cell dimensions		
a, *b*, *c*, Å	10.321(4), 12.744(5), 23.203(9)	9.8923(9), 10.9627(10), 12.2618(11)
α, β, γ, deg.	90, 91.082(6), 90	67.284(5), 70.956(6), 68.741(5)
Volume, $Å^3$	3051(2)	1115.97(18)
Z	4	2
ρ_{calcd}, mg/cm^3	1.754	1.843
Absorption coefficient, mm^{-1}	3.150	4.264
F(000)	1636	618
Crystal size, mm	0.30×0.30×0.24	0.23×0.23×0.17
θ range for data collection, deg.	1.76 to 26.44	3.18 to 25.50
Limiting indices	$-12 \le h \le 7$, $-12 \le k \le 15$, $-29 \le l \le 20$	$-11 \le h \le 11$, $-13 \le k \le 10$, $-14 \le l \le 14$
Reflections collected	14073	7805
Independent reflections	6203 [*R*(int) = 0.0434]	4036 [*R*(int) = 0.0203]
Completeness to θ_{max}, %	98.8	97.2
Max. and min. transmission	1.000000 and 0.862746	0.5362 and 0.4366
Goodness-of-fit on F^2	1.042	1.000
Final *R* indices $[I > 2\sigma(I)]$	$R_1 = 0.0357$, $wR_2 = 0.0672$	$R_1 = 0.0177$, $wR_2 = 0.0409$
R indices (all data)	$R_1 = 0.0623$, $wR_2 = 0.0736$	$R_1 = 0.0190$, $wR_2 = 0.0415$
Largest difference peak and hole, $Å^{-3}$	0.646 and –0.906	1.019 and –0.548
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	

Table 2

Selected bond distances d (Å) *for* **I** *and* **II**

Bond	*d*	Bond	*d*	Bond	*d*	Bond	*d*
I				**II**			
Yb(1)—O(1)	2.299(3)	Yb(1)—N(1)	2.588(4)	Yb(1)—O(1)	2.3017(18)	Yb(1)—O(9)	2.3097(19)
Yb(1)—O(3)	2.375(3)	Yb(1)—N(2)	2.667(4)	Yb(1)—O(3)	2.2821(19)	Yb(1)—O(10)	2.3067(19)
Yb(1)—O(5)	2.265(3)	Yb(1)—N(3)	2.721(4)	Yb(1)—O(5)	2.2863(18)	Yb(1)—N(1)	2.551(2)
Yb(1)—O(7)	2.273(3)	Yb(1)—N(4)	2.648(4)	Yb(1)—O(7)	2.2874(18)	Yb(1)—N(2)	2.573(2)
Yb(1)—O(9)	2.319(3)						

RESULTS AND DISCUSSION

FT-IR spectrum of I. It can be seen the ν(C—N) band of **I** is at 1083 cm^{-1} and is red-shifted by 19 cm^{-1} as compared with that (1102 cm^{-1}) of H_6ttha. This indicates that the nitrogen atoms of the ttha ligand are coordinated to the Yb^{III} ion. The ν_{as}(COOH) band of H_6ttha at 1737 cm^{-1} disappears in the FT-IR spectrum of the complex. The ν_{as}(COO) band of the complex is at 1604 cm^{-1} and is red-shifted by 40 cm^{-1} as compared with that at 1644 cm^{-1} in H_6ttha, and the ν_s(COO) of the complex is at 1415 cm^{-1}; it is blue-shifted by 17 cm^{-1} as compared with that at 1398 cm^{-1} in H_6ttha. These changes indicate that some oxygen atoms from the carboxyl groups are coordinated to the Yb^{III} ion, but a non-coordinate carboxyl groupe exists. There is a broad ν(OH) band of H_2O near 3432 cm^{-1}, which reveals the existence of H_2O in the complex.

FT-IR spectrum of II. It can be seen the ν(C—N) band of **II** is at 1249 cm^{-1} and is red-shifted by 41 cm^{-1} as compared with that (1290 cm^{-1}) of H_4pdta, which indicates that the nitrogen atoms of the ttha ligand are coordinated to the Yb^{III} ion. The ν_{as}(COOH) band of H_4pdta at 1735 cm^{-1} disappears in the FT-IR spectrum of the complex. The ν_{as}(COO) band of the complex is at 1623 cm^{-1} and is red-shifted by 32 cm^{-1} as compared with that at 1655 cm^{-1} in H_4pdta, and the ν_s(COO) of the complex is at 1405 cm^{-1}; it is red-shifted by 9 cm^{-1} as compared with that at 1414 cm^{-1} in H_4pdta. These changes indicate that oxygen atoms from the carboxyl groups are coordinated to the Yb^{III} ion. There is a broad ν(OH) band of H_2O near 3448 cm^{-1}, which reveals the existence of H_2O in the complex.

Molecular and crystal structure of I. Fig. 1, *a* shows that the Yb^{III} ion is coordinated with four N atoms and five O atoms, all from one ttha ligand. It should be noted that there is a free (uncoordinate) carboxyl group anion, [O(11)—C(17)—O(12)].

As can be seen from Fig. 2, the YbN_4O_5 part in the $[Yb^{III}(ttha)]^{3-}$ complex anion forms a pseudo-monocapped square antiprismatic polyhedron. The set of O(1), O(3), O(5), and N(4) and the set of O(7), N(1), N(2), and N(3) make two approximately parallel square planes. The torsion angle of two square planes is about 45°. The capped position is occupied by O(9), above the O(1), O(3), O(5). and N(4) plane. From Table 2, it can be seen that the Yb^{III}—O bond distances range from 2.299(3) Å (Yb^{III}—O(1)) to 2.375(3) Å (Yb^{III}—O(3)). Due to the repulsion between the capped atom O(9) and the top O(1), O(3), O(5) and N(4) plane, the Yb^{III}—O(9) bond distance is the second longest among the Yb^{III}—O bond distances. And the longest Yb^{III}—O bond distance is Yb^{III}—O(3), mainly due to the hydrogen bond between O(3) and N(5). The Yb^{III}—N bond distances range from 2.588(4) Å (Yb^{III}—N(1)) to 2.721(4) Å (Yb^{III}—N(3)); they are much longer than the Yb^{III}—O bond distances. This indicates that O atoms coordinate to the Yb^{III} ion much more stably than N atoms.

It can also be seen from Fig. 2, *a* that the geometry conformation is slightly distorted: it is not a standard monocapped square antiprismatic polyhedron. Evidently, the Yb^{III}—O(9) bond is not vertical to the top O(1), O(3), O(5), and N(4) plane; instead, it is close to the O(3)—N(4) bond. Probably, this is because O(9) is restricted to the five-membered ring of O(9)C(15)C(16)N(4)Yb and is unable to change its position. As a result, ∠O(3)—Yb^{III}—O(9) (72.80(11)°) and ∠N(4)—Yb^{III}—O(9) (64.77(11)°) angles are smaller than ∠O(1)—Yb^{III}—O(9) (73.55(12)°) and ∠O(5)—Yb^{III}—O(9) (76.28(11)°) angles, so that the average value of the dihedral angle between Δ(O(3)O(1)N(4)) and Δ(O(5)O(3)N(4)) triangles and between Δ(O(1)O(3)O(5)) and Δ(N(4)O(3)O(5)) triangles is 12.82(95)°. As for the bottom plane, the value of the dihedral angle between Δ(O(7)N(1)N(3)) and Δ(N(1)N(2)N(3)) triangles is 7°, and between Δ(O(7)N(1)N(2)) and Δ(O(7)N(2)N(3)) triangle it is 5.73°. The judgment about a monocapped square antiprismatic polyhedron, according to Guggenberger and Muetterties′ method [21], depends on the special bottom plane dihedral angle, which should be smaller than 26.4°. Therefore, although distorted, the $[Yb^{III}(ttha)]^{3-}$ complex anion still retains a monocapped square antiprismatic polyhedron.

The unit cell shown in Fig. 3 contains four $(NH_4)_3[Yb^{III}(ttha)]\cdot 5H_2O$ molecules. The molecules are connected with each other through the hydrogen bond and electrostatic bonding with NH_4^+ ions, and crystallize in the monoclinic system and the $P2_1/c$ space group. Each NH_4^+ ion is bonded to $[Yb^{III}(ttha)]^{3-}$ complex anions by electrostatic and hydrogen bonds. The crystallization water molecule

(O(15)) links two $[Yb^{III}(ttha)]^{3-}$ complex anions by a hydrogen bond, which contributes to the stabilization of the crystal structure. The hydrogen bonds in one unit cell link the crystallization water molecules, the NH_4^+ ion, and coordinate and non-coordinate carboxylic O atoms of the ttha ligands. Therefore, in the crystal, a network structure is formed through hydrogen bonds and electrostatic bonding.

Molecular and crystal structure of II. It can be seen from Fig. 1, *b* that the central Yb^{III} ion is coordinated with one pdta ligand by two N atoms and four O atoms and with two water molecules by two O atoms. In comparison with the complex **I**, two more water molecules are needed to form the complex in addition to the hexadentate pdta ligand. And as shown in Table 2, the bond distances between the central Yb^{III} ion and O(9) and O(10) from the water molecules are both longer than the other Yb^{III}—O bond distances, which are 2.3097(19) Å and 2.3067(19) Å respectively. However, the average value of the other Yb^{III}—O bond distances is 2.2893(93) Å. This indicates that the pdta ligand coordinates with the Yb^{III} ion more stably than water molecules. So, the complex **I** is more stable due to a non-coordinate water molecule. And it can also bring to the conclusion that O atoms coordinate to the Yb^{III} ion more stably than N atoms because the Yb^{III}—O bond distances are much shorter than the Yb^{III}—N bond distances (the average value of 2.562(2) Å) , as seen from Table 2.

As seen from Fig. 2, the O(5), O(7), O(10), and N(2) and O(1), O(3), O(9), and N(1) sets make two approximately parallel square planes. The torsion angle of two square planes is about 45°. Therefore, the YbN_2O_6 part in the $[Yb^{III}(pdta)(H_2O)_2]^-$ complex anion has a shape of a pseudo-square antiprismatic polyhedron. For the top plane, the average value of the dihedral angle between ΔO(5)O(7)O(10) and ΔO(5)O(7)N(2) triangles and between ΔO(7)O(10)N(2) and ΔO(5)O(10)N(2) triangles is 1.27(86)°. For the bottom plane, the average value of the dihedral angle between ΔO(1)O(3)O(9) and O(1)O(3)N(1) triangles and between ΔO(1)O(9)N(1) and ΔO(3)O(9)N(1) triangles is 3.94(97)°. From these data, also according to Guggenberger and Muetterties' method [21], it can be concluded that the conformation of the $[Yb^{III}(pdta)(H_2O)_2]^-$ complex anion keeps the shape of a square antiprismatic polyhedron, but it is slightly distorted.

In one unit cell shown in Fig. 3, *b*, there are two molecules of **II**. The molecules are connected with each other through the hydrogen bond and electrostatic bonding with NH_4^+ ions, and crystallize in the triclinic system and the $P\overline{1}$ space group. Each NH_4^+ ion links the $[Yb^{III}(pdta)(H_2O)_2]^-$ complex anion by electrostatic interactions. Three crystallization water molecules, O(11), O(15), and O(12) are connected in turn by hydrogen bonds. O(11) links a coordinate carboxylic O(3) atom of one pdta ligand and O(12) links a non-coordinate carboxylic O(6) atom of another pdta ligand. Through this action, two $[Yb^{III}(pdta)(H_2O)_2]^-$ complex anions are connected tightly, and it contributes to the stabilization of the crystal structure. In whole, hydrogen bonds link the crystallization water molecules, the NH_4^+ ion, and coordinate and non-coordinate carboxylic O atoms of the pdta ligands. So, a layered crystal structure is formed through hydrogen bonds and electrostatic bonding.

From a comparison of these two complexes, it can be easily found that due to different ligands, different complex structures form. The ttha ligand possesses ten coordination atoms, including four N and six O atoms, which is more than the pdta ligand has (two N and four O atoms). This indicates that the ttha ligand prefers to coordinate with the central metal ions and always forms nine-coordinate aminium salt complexes [22—26]. There is no exception for the complex **I**. Nine coordination atoms are all from one ttha ligand, which is of advantage to the stabilization of its molecular structure. In the complex of the pdta ligand with the Yb^{III} ion, there is a six-membered ring Yb(1)N(1)C(5)C(6)C(7)N(2). Being different from the other five rings in this complex, the six-membered ring certainly takes more space, which would hold back other coordination atoms close to the central Yb^{III} ion, such as the water molecule, the adjacent ligand, and so on, and decrease the opportunity to coordinate to the Yb^{III} ion. This induces that the complex **I** is eight-coordinate.

CONCLUSIONS

In conclusion, two novel rare earth metal complexes with aminopolycarboxylic acid (ttha and pdta) have been successfully synthesized. With a certain ionic radii and electronic configurations of the central Yb^{III} ion, as to different ligands, the complex **I** is a nine-coordinate pseudo-monocapped square antiprismatic structure, while the complex **II** has an eight-coordinate pseudo-square antiprismatic conformation. Therefore, the ligand structure plays an important role in the coordination number and structure of the complex.

Supplementary materials. CCDC 739569 $(NH_4)_3[Yb^{III}(ttha)]\cdot 5H_2O$, CCDC 739568 $(NH_4)[Yb^{III}(pdta)(H_2O)_2]\cdot 5H_2O$, 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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