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Crystal structure of two neodymium complexes with amino acids: $\{[Nd_2(cystine)_2(OH_2)_8](ClO_4)_6 \cdot 3H_2O\}_n$ and $[Nd_2(phenylalanine)_4(OH_2)_8](ClO_4)_6 \cdot H_2O$

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Abstract

The title complexes: $\{[Nd_2(Cys)_2(OH_2)_8](ClO_4)_6 \cdot 3H_2O\}_n$ (**1**) and $[Nd_2(Phe)_4(OH_2)_8](ClO_4)_6 \cdot H_2O$ (**2**) were prepared in aqueous solutions (where Cys = cystine, Phe = phenylalanine), and their crystal structures were determined by X-ray diffraction methods. For complex **1**, the crystal belongs to the orthorhombic space group $P2_12_12_1$, with cell parameters: $a = 13.674(3)$ Å, $b = 18.485(6)$ Å, $c = 19.334(7)$ Å, $V = 4887(3)$ Å^3, $Z = 4$; the final R value is 0.045; the crystal is built up from layer polymers; each neodymium ion is coordinated by four carboxylic oxygen atoms from four cystine molecules and four oxygen atoms from H_2O, forming a coordination polyhedron of square antiprism. In the case of complex **2**, the crystal belongs to the monoclinic space group $C2/c$, with unit cell parameters: $a = 10.835(11)$ Å, $b = 22.27(2)$ Å, $c = 25.85(3)$ Å, $\beta = 93\text{–}10(8)°$, $V = 6227(10)$ Å^3, $Z = 4$; the final R value is 0.069. The complex **2** is a binuclear molecule in which four carboxylic oxygen atoms of phenylalanine ligands and four oxygen atoms of water molecules form a distorted square antiprism around each neodymium ion.

1. Introduction

Lanthanide ions are often used as spectroscopic probes of calcium ions in studies of biological systems, as well as promoters in the textile dyeing industry, and diagnostic agents in clinical medicine [1–3]. Since lanthanide ions can substitute the calcium ions in living systems, studies of the bonding modes and structures of lanthanide complexes with some amino acids are of interest. Studies on lanthanide complexes with some amino acids in solution-phase have been done by NMR, luminescence and titration methods to determine the thermodynamic stability constants [4–6]. Crystallographic studies on these complexes in the solid state have also appeared recently. However, they are limited to glycine, alanine, glutamic acid and proline [7–10]. In this paper, two novel neodymium complexes with cystine and phenylalanine were synthesized and their crystal structures were determined by X-ray analysis. The structure data are discussed and compared with previous work on alanine and glutamic acid.

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2. Experimental details

2.1. Preparation

Both of the two title complexes were prepared by dissolving ligands (cystine (**1**), phenylalanine (**2**)) in an aqueous solution of $Nd(ClO_4)_3$. Crystals were obtained by slow evaporation of the aqueous solutions ((**1**) pH ≈ 4, (**2**) pH ≈ 1) at room temperature. Elemental analysis was used to determine the composition of the compounds. Formulas of the two complexes and elemental analysis data are listed in Table 1.

2.2. Crystal structure measurement

Suitable single crystals of the two complexes were selected and sealed in thin-walled capillaries. The reflection data were collected on a Siemens 3m/v diffractometer with graphite-monochromated Mo Kα radiation. The intensities for both complexes **1** and **2** were treated by Lorentz polarization and absorption

TABLE 1. Formula and elemental analysis data (calculated value in parentheses)

Compounds	N (%)	C (%)	H (%)
1. $Nd(Cys)(ClO_4)_3 \cdot 5.5H_2O$	3.15(3.35)	8.39(8.63)	3.10(2.75)
2 $Nd(Phe)_2(ClO_4)_3 \cdot 4.5H_2O$	2.70(3.09)	23.86(23.82)	3.26(3.63)

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TABLE 2. Crystal data, data collection and refinement

Formula	Complex **1** $[Nd_2(Cys)_2(OH_2)_8](ClO_4)_6 \cdot 3H_2O$	Complex **2** $[Nd_2(Phe)_4(OH_2)_8](ClO_4)_6 \cdot H_2O$
Colour	Reddish	Reddish
Crystal size (mm)	$0.30 \times 0.30 \times 0.25$	$0.30 \times 0.40 \times 0.35$
Crystal system	Orthorhombic	Monoclinic
Space group	$P2_12_12_1$	$C2/c$
a (Å)	13.674(3)	10.835(11)
b (Å)	18.485(6)	22.27(2)
c (Å)	19.334(7)	25.85(3)
β (°)		93.10(8)
V (Å^3)	4887(3)	6227(10)
Z	4	4
Formular weight	1563.9	1708.1
D_c (g cm^{-3})	2.126	1.822
$F(000)$	3104	3304
Absorption coefficient (mm^{-1})	2.719	2.017
Scan type	1	1
2θ range (°)	3.0–44.0	3.0–46.0
Standard reflections	1/100	1/100
Independent reflections	3396	4372
Observed reflections	$2862(F > 6.0\sigma\|F\|)$	$2495(F > 4.0\sigma\|F\|)$
Quantity minimized	$\Sigma\omega(F_o - F_c)^2$	$\Sigma\omega(F_o - F_c)^2$
No. of parameters refined	640	402
Final R	0.0449	0.0691
R_w	0.0503	0.0729
Goodness-of-fit	6.15	10.84
Max, min, residual ρ (e Å^{-3})	1.15, −1.52	1.40, −1.20

corrections. All calculations were performed using the Siemens shelxtl plus system (VMS). The structures were determined by direct methods and difference Fourier technique, and then refined by full-matrix least-squares methods. Crystal data, data collection and refinement are listed in Table 2.

The non-hydrogen atomic parameters for complexes **1** and **2** are given in Tables 3 and 4, respectively. For both complexes **1** and **2**, there are so many oxygen atoms in one asymmetric unit that we divided them into three series: O for the carboxylic group, Ow for the coordinated H_2O and Oq for the non-coordination water molecules. Selected bond distances and angles for complexes **1** and **2** are listed in Tables 5 and 6, respectively.

3. Description of the structure and discussion

3.1. Complex 1

The crystal of complex **1** is built up from layered polymers. The polymeric molecule is formed from binuclear units which connect with one another through the dicarboxylic ligands. An asymmetric unit of the complex is a binuclear unit, as shown in Fig. 1. Two neodymium ions are connected by four bridging carboxyl groups from four cystine molecules. Each neodymium ion is also coordinated by four oxygen atoms from water molecules, taking the coordination polyhedron of a distorted square antiprism with coordination number 8. Figure 2 shows the polyhedron around a neodymium ion. The four carboxylic oxygen atoms from the cystine ligands make a rough plane under the central ion and the four aqueous oxygens form the upper plane (Table 7).

TABLE 3. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients (Å$\times 10^3$) of complex **1**

Atom	x	y	z	U_{eq}[a]
Nd(1)	2493(1)	1894(1)	3554(1)	18(1)
Nd(2)	1813(1)	3105(1)	1609(1)	18(1)
Cl(1)	8018(5)	3241(4)	934(3)	52(2)
Cl(2)	3461(5)	6579(4)	1073(4)	55(2)
Cl(3)	5133(6)	4164(4)	230(4)	68(3)
Cl(4)	5658(5)	9289(4)	−71(4)	64(3)
Cl(5)	10864(6)	8625(4)	992(4)	62(3)
Cl(6)	5809(8)	1525(5)	1225(6)	100(4)
S(1)	−1697(7)	847(3)	1447(4)	67(3)
S(2)	−366(6)	830(4)	1929(4)	63(3)
S(3)	3247(5)	−417(3)	2291(4)	56(2)
S(4)	3485(5)	−1352(3)	1742(4)	55(3)

(continued)

TABLE 3. (*continued*)

Atom	x	y	z	U_{eq}[a]
O(1)	398(12)	2640(9)	2163(9)	45(6)
O(2)	1047(11)	2049(8)	3055(8)	42(6)
O(3)	1803(13)	3579(8)	2668(8)	43(6)
O(4)	2125(10)	3131(10)	3731(9)	49(5)
O(5)	2213(9)	1910(10)	1674(8)	49(5)
O(6)	2562(14)	1184(7)	2543(8)	42(6)
O(7)	3496(11)	3106(10)	1932(9)	52(6)
O(8)	3588(12)	2461(10)	2875(9)	49(6)
O(9)	7646(14)	3423(10)	1621(12)	77(8)
O(10)	7261(13)	3114(12)	437(9)	69(7)
O(11)	8616(17)	2582(12)	937(13)	90(10)
O(12)	8649(20)	3816(14)	647(13)	105(11)
O(13)	2988(25)	6841(17)	467(14)	136(14)
O(14)	3696(43)	5860(15)	853(15)	238(31)
O(15)	2977(24)	6686(21)	1691(13)	174(18)
O(16)	4296(23)	7044(33)	1071(36)	475(50)
O(17)	5625(22)	4123(14)	−406(11)	109(12)
O(18)	4823(14)	3444(11)	440(11)	71(8)
O(19)	5732(19)	4453(11)	771(10)	87(10)
O(20)	4302(23)	4619(14)	156(17)	148(15)
O(21)	6241(17)	9075(12)	514(12)	83(9)
O(22)	5393(19)	8642(13)	−460(12)	97(10)
O(23)	6254(24)	9746(14)	−506(15)	133(14)
O(24)	4788(21)	9702(17)	237(21)	161(18)
O(25)	10034(19)	8344(20)	626(18)	174(19)
O(26)	11644(17)	8212(20)	730(15)	147(15)
O(27)	10744(27)	8563(17)	1681(14)	148(16)
O(28)	10850(37)	9339(16)	825(24)	230(27)
O(29)	6191(30)	1436(24)	547(18)	198(23)
O(30)	5798(18)	864(14)	1583(24)	184(19)
O(31)	6418(18)	2017(12)	1670(18)	133(14)
O(32)	4831(16)	1819(16)	1180(20)	147(16)
OW(1)	2708(14)	3052(14)	527(8)	76(8)
OW(2)	2484(15)	4317(9)	1404(10)	73(7)
OW(3)	826(14)	2567(10)	725(10)	63(7)
OW(4)	567(15)	3937(10)	1291(13)	77(9)
OW(5)	3813(13)	1086(9)	3808(11)	63(7)
OW(6)	3564(13)	2426(9)	4432(10)	52(7)
OW(7)	1614(12)	1923(11)	4622(8)	54(6)
OW(8)	1783(13)	715(9)	3763(9)	57(6)
N(1)	−1440(14)	2300(11)	2505(12)	51(8)
N(2)	−1671(20)	−662(11)	590(10)	56(8)
N(3)	3012(23)	1059(12)	717(10)	70(8)
N(4)	4619(16)	−1615(12)	3140(12)	57(8)
C(1)	348(17)	2247(12)	2674(12)	31(7)
C(2)	−647(14)	1925(15)	2899(11)	38(7)
C(3)	−660(21)	1098(13)	2788(14)	52(10)
C(4)	−1881(17)	−1378(12)	1710(12)	38(8)
C(5)	−1564(19)	−648(11)	1368(14)	52(10)
C(6)	−2189(19)	−33(12)	1686(18)	60(11)
C(7)	2470(19)	1333(12)	1950(12)	37(8)
C(8)	2791(18)	733(11)	1409(14)	51(9)
C(9)	3615(17)	262(13)	1663(!5)	52(9)
C(10)	6079(19)	−2167(13)	2580(13)	39(8)
C(11)	4985(15)	−1960(15)	2520(11)	35(7)
C(12)	4769(16)	−1549(13)	1902(13)	44(9)
Oq(1)	9088(15)	4635(13)	2090(14)	96(10)
Oq(2)	7681(22)	5265(14)	−67(14)	124(13)
Oq(3)	4254(17)	4890(12)	2037(16)	104(11)

[a]Equivalent isotopic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

TABLE 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$) of complex **2**

Atom	x	y	z	U_{eq}[a]
Nd(1)	2956(1)	1138(1)	2565(1)	46(1)
Cl(1)	3746(17)	2293(6)	6082(6)	213(8)
Cl(2)	7264(13)	−1388(4)	2208(4)	129(5)
Cl(3)	−571(6)	−346(3)	3738(3)	69(2)
O(1)	4000(15)	1698(6)	1950(6)	67(6)
O(2)	4023(16)	1949(7)	3052(6)	65(6)
O(3)	4340(17)	585(7)	3122(6)	74(7)
O(4)	3729(17)	337(7)	2019(6)	76(7)
O(5)	4478(16)	1890(8)	5916(8)	89(8)
O(6)	3317(33)	2137(16)	6550(14)	233(21)
O(7)	2895(30)	2278(30)	5699(22)	468(42)
O(8)	4543(74)	2821(14)	6249(22)	825(56)
O(9)	6894(37)	−917(9)	2396(9)	260(20)
O(10)	7733(54)	−1140(20)	1745(22)	369(39)
O(11)	7621(45)	−1789(17)	2587(20)	317(33)
O(12)	6719(28)	−1789(16)	1948(13)	192(17)
O(13)	−1124(30)	−613(13)	3313(12)	202(17)
O(14)	75(25)	−758(8)	4019(9)	151(12)
O(15)	172(26)	116(11)	3555(11)	169(14)
O(16)	−1421(27)	−14(14)	3986(11)	177(15)
Ow(1)	2046(14)	1160(7)	3450(5)	75(6)
Ow(2)	1603(16)	255(7)	2660(7)	82(7)
Ow(3)	1446(17)	1128(9)	1782(7)	104(8)
Ow(4)	1483(15)	2021(17)	2568(7)	76(7)
Oq(1)	0	−1520(23)	2500	499(71)
N(1)	4603(18)	3035(7)	3466(7)	63(7)
N(2)	6014(21)	−756(8)	3402(9)	93(10)
C(1)	5132(25)	2026(9)	3170(7)	51(8)
C(2)	5488(20)	2531(8)	3543(8)	41(7)
C(3)	5691(22)	2335(9)	4080(8)	63(8)
C(4)	6141(20)	2796(8)	4477(8)	50(7)
C(5)	7299(22)	3036(10)	4451(10)	68(10)
C(6)	7703(25)	3468(11)	4791(10)	74(10)
C(7)	6942(26)	3684(12)	5150(9)	78(11)
C(8)	5778(25)	3462(12)	5172(9)	72(11)
C(9)	5396(23)	3036(11)	4837(8)	67(11)
C(10)	5282(26)	260(9)	3197(9)	62(9)
C(11)	5110(24)	−273(10)	3532(10)	71(10)
C(12)	5278(25)	−129(11)	4097(10)	88(11)
C(13)	4244(25)	244(9)	4315(7)	61(9)
C(14)	3115(26)	5(12)	4394(10)	88(11)
C(15)	2283(26)	359(15)	4655(11)	89(13)
C(16)	2589(26)	936(12)	4825(10)	78(11)
C(17)	3667(27)	1179(13)	4740(9)	78(10)
C(18)	4473(25)	812(11)	4497(9)	71(10)

[a]Equivalent isotopic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Moreover, the other four carboxyl groups of the four cystine molecules bridge four other pairs of neodymium ions and make the whole molecule an infinite layer. Although the complex molecule is a polymer, we prefer the title formula to express its composition and structure because the basic unit in this complex is a binuclear unit. In the majority of cases, no monomer molecule has been found in the structures of lanthanide complexes

TABLE 5. Selected bond lengths (Å) and angles (°) of complex **1**

Nd(1)–O(2)	2.218(16)	Nd(1)–O(4)	2.366(18)
Nd(1)–O(6)	2.357(14)	Nd(1)–O(8)	2.249(17)
Nd(1)–Ow(5)	2.395(17)	Nd(1)–Ow(6)	2.449(18)
Nd(1)–Ow(7)	2.390(16)	Nd(1)–Ow(8)	2.421(16)
Nd(2)–O(1)	2.372(17)	Nd(2)–O(3)	2.227(15)
Nd(2)–O(5)	2.279(18)	Nd(2)–O(7)	2.385(16)
Nd(2)–Ow(1)	2.426(17)	Nd(2)–Ow(2)	2.454(18)
Nd(2)–Ow(3)	2.394(19)	Nd(2)–Ow(4)	2.375(19)
O(1)–Nd(2)–O(3)	73.69(6)	O(1)–Nd(2)–O(5)	79.6(5)
O(1)–Nd(2)–O(7)	132.0(6)	O(2)–Nd(1)–O(4)	75.4(5)
O(2)–Nd(1)–O(6)	75.4(6)	O(2)–Nd(1)–O(8)	106.2(6)
O(3)–Nd(2)–O(5)	109.4(6)	O(3)–Nd(2)–O(7)	76.4(6)
O(4)–Nd(1)–O(6)	131.8(5)	O(4)–Nd(1)–O(8)	77.1(6)
O(5)–Nd(2)–O(7)	75.8(6)	O(6)–Nd(1)–O(8)	75.4(6)

TABLE 6. Bond lengths (Å) and angles (°) for complex **2**

Nd–O(1)	2.356(16)	Nd–O(2)	2.455(15)
Nd–O(3)	2.367(16)	Nd–O(4)	2.449(16)
Nd–Ow(1)	2.540(15)	Nd–Ow(2)	2.472(17)
Nd–Ow(3)	2.531(18)	Nd–Ow(4)	2.534(16)
O(1)–Nd–O(2)	74.1(5)	O(1)–Nd–O(3)	111.9(6)
O(2)–Nd–O(3)	78.8(5)	O(1)–Nd–O(4)	78.8(5)
O(2)–Nd–O(4)	131.7(6)	O(3)–Nd–O(4)	75.3(5)
O(1)–Nd–Ow(1)	145.2(5)	O(2)–Nd–Ow(1)	73.6(5)
O(3)–Nd–Ow(1)	74.1(5)	O(4)–Nd–Ow(1)	134.0(5)
O(1)–Nd–Ow(2)	142.9(5)	O(2)–Nd–Ow(2)	143.0(6)
O(3)–Nd–Ow(2)	83.4(6)	O(4)–Nd–Ow(2)	72.7(6)
Ow(1)–Nd–Ow(2)	70.4(5)	O(1)–Nd–Ow(3)	77.3(6)
O(2)–Nd–Ow(3)	133.0(6)	O(3)–Nd–Ow(3)	147.4(6)
O(4)–Nd–Ow(3)	76.1(6)	Ow(1)–Nd–Ow(3)	117.0(6)
Ow(2)–Nd–Ow(3)	73.3(6)	O(1)–Nd–Ow(4)	85.2(5)
O(2)–Nd–Ow(4)	72.9(5)	O(3)–Nd–Ow(4)	141.3(5)
O(4)–Nd–Ow(4)	143.3(5)	Ow(1)–Nd–Ow(4)	73.0(5)
Ow(2)–Nd–Ow(4)	103.9(5)	Ow(3)–Nd–Ow(4)	68.3(6)

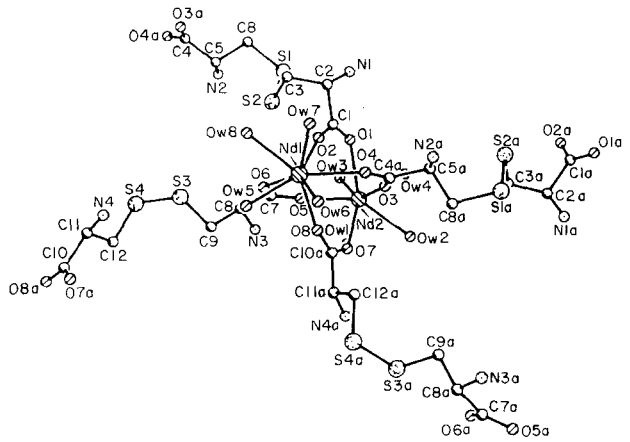

Fig. 1. The crystal structure of $[Nd_2(Cys)_2(H_2O)_8](ClO_4)_6 \cdot 3H_2O$.

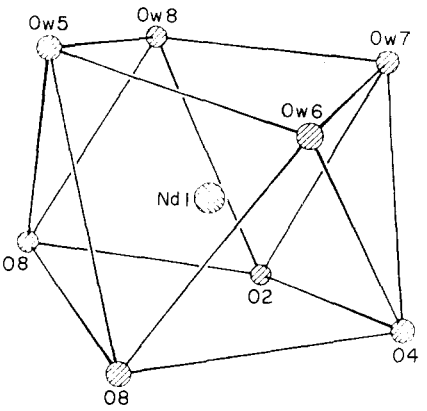

Fig. 2. The coordination polyhedron of $[Nd_2(Cys)_2(H_2O)_8](ClO_4)_6 \cdot 3H_2O$.

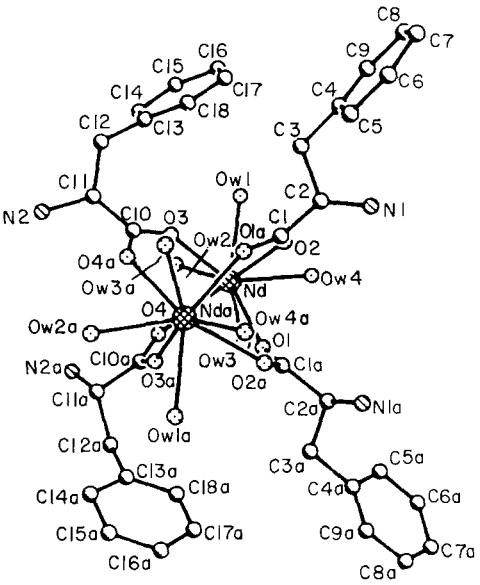

Fig. 3. The crystal structure of $[Nd_2(Phe)_4(H_2O)_8](ClO_4)_6 \cdot H_2O$.

with amino acids [7–10]. The reason is that the carboxyl groups of amino acids tend towards bridging lanthanide ions.

Compared with the similar polymeric layer structures of lanthanide complexes with glutamic acid, the cystine complex is different in bonding mode and coordination number. The carboxyl group far from the amino group in glutamic acid usually chelates two lanthanide ions, forming an oxygen bridge. Thus, the coordination number of glutamic acid complexes is usually 9 [9].

3.2. Complex **2**

The crystal of complex **2** is built up from dimeric molecules. Figure 3 shows the molecular structure of the complex. Two neodymium ions are connected by four bridging carboxyl groups from four phenylalanine molecules. Each central ion is also coordinated by four oxygen atoms from H_2O. The coordination number is

TABLE 7. Least square fitting equations of planes containing coplanar atoms

Complex 1	Plane (1)	Equation	$2.692x-9.333y+16.249z=3.5282$			
		Atoms	02	04	06	08
		P^* (Å)	−0.2921	0.1864	0.1901	−0.1844
	Plane (2)	Equation	$2.599x-9.133y+16.403z=6.1135$			
		Atoms	Ow5	Ow6	Ow7	Ow8
		P^* (Å)	−0.1320	−0.1325	0.1317	−0.1312
Complex 2	Plane (1)	Equation	$10.810x+0.030y-3.132z=3.5582$			
		Atoms	01	02	03	04
		P^* (Å)	0.1605	−0.1596	0.1571	−0.1580
	Plane (2)	Equation	$10.780x+0.525y-3.905z=0.8117$			
		Atoms	Ow1	Ow2	Ow3	Ow4
		P^* (Å)	0.1081	−0.1091	0.1108	−0.1098

P* is the distance of the atoms from the plane.

TABLE 8. Atom–atom distances (Å) related to H-bonding

Complex 1	Oq1–Ow4a	2.853	Oq1–Ow8a	2.849	Oq2–Ow8a	2.755
	Oq2–O24a	2.900	Oq2–N3a	2.787	Oq3–Ow2	2.912
	O10a–Ow1	2.914	O12a–N3	2.788	O13a–Ow7	2.860
	O14a–N2	2.858	O15a–N1	2.849	O18a–N4	2.852
	O20a–N2	2.749	O26a–Ow6	2.786		
Complex 2	Oq1–O11b	2.667	Oq1–O11c	2.667	Oq1–N1a	2.742
	Oq1–N1b	2.742	O6a–Ow4	2.950	O8a–Ow1	2.977
	O9–N2	2.841	O11a–Ow4	2.922	O15–Ow2	2.869
	O15a–Ow3	2.957				

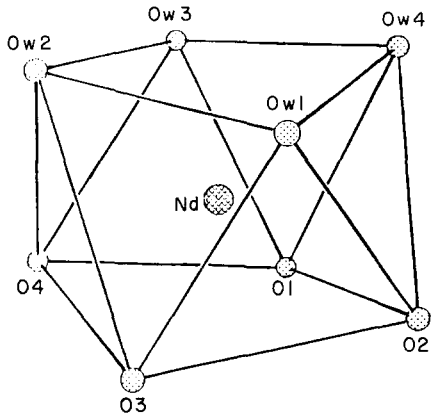

Fig. 4. The coordination polyhedron of $[Nd_2(Phe)_4(H_2O)_8](ClO_4)_6 \cdot H_2O$.

8, and the coordination polyhedron is a distorted square antiprism, as shown in Fig. 4. Like complex **1**, four oxygen atoms from water molecules form the upper plane and four carboxyl oxygen atoms form the lower plane (Table 8).

There is a C_2 symmetry axis in the dimeric structure, and two neodymium ions have the same stereochemistry.

The crystal structure of the phenylalanine complex has some similarities with those of alanine complexes reported previously [8]. For example, they are all dimer molecules, eight-coordinated, and have a C_2 symmetry axis.

As illustrated in Fig. 3, four phenyl rings lie outside the coordination sphere and are far from one another. Thus, the space hindrance is small. The minimal effect of phenyl rings on the coordination mode in phenylalanine has the same coordination properties with lanthanide ions as alanine.

The perchlorate anions and non-coordinating water molecules reside in the cavities of the crystal and stabilize the crystal packing by forming hydrogen bonds (Table 8).

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References

1 H.B. Brittain, F.S. Richardson and R.B. Martin, *J. Am. Chem. Soc., 98* (1978) 8255.
2 W.De.W. Horrocks Jr. and D.R. Sudnick, *J. Am. Chem. Soc., 101* (1979) 334.

3 H.G. Brittain, *Inorg. Chem., 18* (1979) 1740.
4 A. Aziz, S.J. Lyle and J.E. Newbery, *J. Inorg. Nucl. Chem., 33* (1979) 1975.
5 A.D. Sherry, C. Yoshida, E.R. Birnbaum and D.W. Darnall, *J. Am. Chem. Soc., 95* (1973) 3011.
6 J. Legendziewicz, E. Huskowska, G.Y. Argay and A. Waskowska, *Inorg. Chim. Acta, 95* (1984) 57.
7 T.Z. Jin, S. Gao, C.H. Huang, Y.Z. Han and G.X. Xu, *Chem. J. Chin. Univ., 10* (1989) 118.
8 T.Z. Jin, S. Gao, C.H. Huang, Y.Z. Han and G.X. Zu, *J. Chin. RE Soc., 5* (1987) 1.
9 I. Gsoregh. M. Gzugler, P. Kierkegaard, J. Legendziewicz and E. Huskowska, *Acta Chem. Scand., 43* (1989) 735.
10 J. Legendziewicz, T. Glowiak, E. Huskowska and D.C. Ngoan, *Polyhedron, 8* (1989) 2139.